General $CuBr_2$-Catalyzed Highly Enantioselective Approach for Optically Active Allenols from Terminal Alkynols

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Supporting Information

The Photo of Apparatus Used for Typical Procedure I (130 ^{o}C).

The Photo of Apparatus Used for Typical Procedure II (70 ^{o}C).

The Photo of Apparatus Used for the Large Scale Reaction

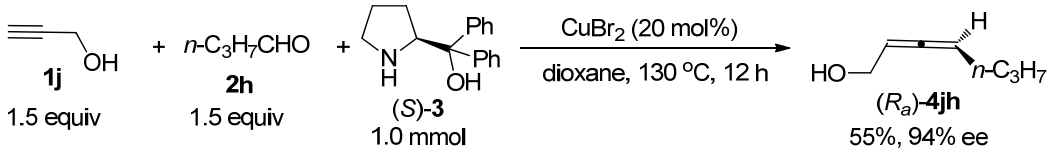
General Experimental Methods

^{1}H and ^{13}C nuclear magnetic resonance spectra were recorded on an instrument operated at 300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR spectra. Infrared spectra were recorded from the films of pure samples on sodium chloride plates for liquid or in the form of KBr discs for the solid samples. Mass and HRMS spectra were carried out in EI or ESI mode. Thin layer chromatography was performed on pre-coated glass-back plates and visualized with UV light at 254 nm. Flash column chromatography was performed on silica gel. $CuBr_2$ and CuBr were purchased from J&K. (*S*)-α,α-Diphenylprolinol and (*R*)-α,α-diphenylprolinol were purchased from Shanghai Darui Fine Chemicals. Aldehydes were distilled right before use. Dioxane was dried over sodium wire with benzophenone as the indicator and distilled freshly before use. All the temperatures are referred to the oil baths used.

Synthesis of axially chiral allenols.

1. Preparation of (R_a)-2,3-heptadien-1-ol (R_a)-4jh. Typical Procedure I.

(tangxj-8-112)

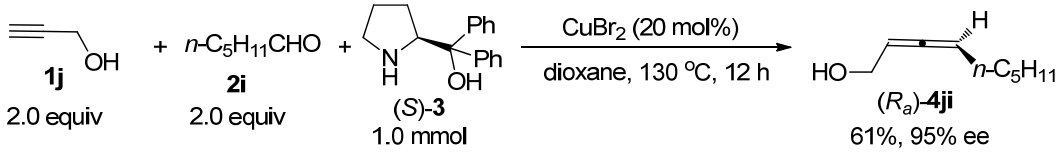


Typical Procedure I: To a flame-dried Schlenk tube with a polytetrafluoroethylene plug were added $CuBr_2$ (44.7 mg, 0.2 mmol), (*S*)-**3** (253.5 mg, 1.0 mmol), **1j** (84.7 mg, 1.5 mmol)/dioxane (1.5 mL), and **2h** (108.5 mg, 1.5 mmol)/dioxane (1.5 mL) sequentially under nitrogen atmosphere. The Schlenk tube was then sealed by screwing the polytetrafluoroethylene plug tightly with the outlet connected to the vacuum line and the nitrogen flow being closed (For an apparatus, see page S2 in SI). The reaction was complete after being stirred in an oil bath preheated at 130 ^{o}C for 12 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 10/1). Then the resulting mixture was diluted with ether (30 mL) and washed with an aqueous solution of hydrochloric acid (3 M, 20 mL). The organic layer was separated and the aqueous layer was extracted with ether (20 mL). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography (eluent: petroleum ether/ethyl acetate = 8/1) on silica gel to afford (R_a)-**4jh** (61.7 mg, 55%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 94% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 500/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 19.0 min, t_R(minor) =

20.7 min); $[\alpha]_D^{25.3}$ = -83.5 (*c* = 1.07, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.37-5.24 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.6 Hz, J_2 = 2.8 Hz, 2 H, OCH_2), 2.06-1.97 (m, 2 H, CH_2), 1.82 (s, 1 H, OH), 1.44 (sext, *J* = 7.3 Hz, 2 H, CH_2), 0.93 (t, *J* = 7.2 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.1, 93.6, 91.6, 60.7, 0.7, 22.3, 3.5; IR (neat) ν (cm^{-1}) 3324, 2959, 2930, 2871, 1963, 1460 1419, 1379, 1339, 1292, 1258, 1213, 1136, 1102, 1061, 1009, MS (EI): *m/z* (%) 112 (M^+, 0.29), 94 ($(M-H_2O)^+$, 58.82), 79 (100); HRMS calcd for $C_7H_{10}O$ [$(M-H_2O)^+$]: 94.0783, found: 94.0782.

The following compounds (R_a)-**4ji** ~ (R_a)-**4ne** were prepared at 130 °C according to **Typical Procedure I** or at 70 °C according to **Typical Procedure II**. Their corresponding racemic enantiomers were prepared by following the same procedure using racemic diphenylprolinol ***rac*-3.**

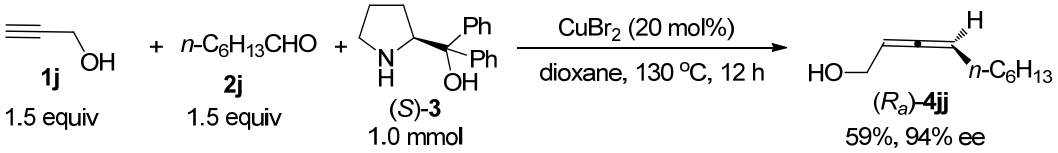
2. Preparation of (R_a)-2,3-nonadien-1-ol (R_a)-4ji. (ct-6-98)



The reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1j** (112.5 mg, 2.0 mmol), (*S*)-**3** (253.1 mg, 1.0 mmol), and **2i** (201.2 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ji** (85.1 mg, 61%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid:[1] 95% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 100/1, 0.7 mL/min, λ = 214 nm, t_R(major) = 14.2 min, t_R(minor) = 15.5 min); $[\alpha]_D^{26}$ = -77.7 (*c* = 1.02, $CHCl_3$) (reported value: 98% ee; $[\alpha]_D^{21}$ = -78.4 (c = 1.03, $CHCl_3$)); ^{1}H NMR (400 MHz,

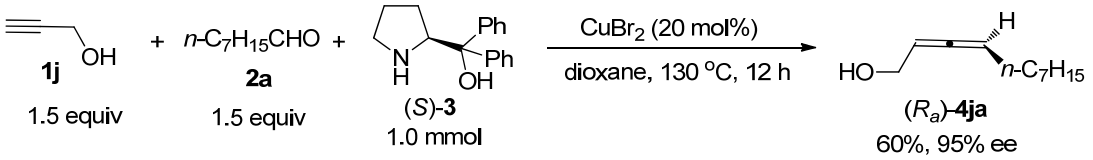
$CDCl_3$) δ 5.36-5.23 (m, 2 H, CH=C=CH), 4.14-4.07 (m, 2 H, OCH_2), 2.08-1.96 (m, 2 H, CH_2), 1.77 (br s, 1 H, OH), 1.44-1.18 (m, 6 H, CH_2 × 3), 0.89 (t, J = 6.8 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.9, 93.9, 91.6, 60.7, 31.2, 28.7, 28.6, 22.4, 14.0; IR (neat) ν (cm^{-1}) 3337, 2957, 2927, 2857, 1963, 1466; MS (EI): *m/z* (%) 122 ($(M-H_2O)^+$, 0.68), 55 (100).

3. Preparation of (R_a)-deca-2,3-dien-1-ol (R_a)-4jj. (Hx-12-51)



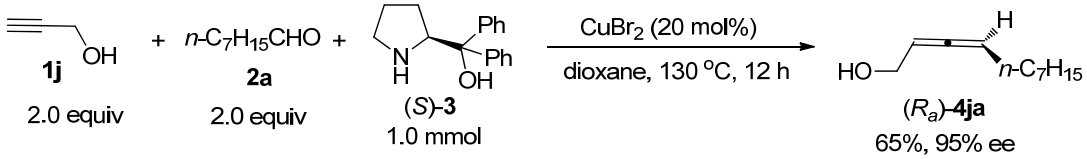
The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1j** (84.8 mg, 1.5 mmol), (*S*)-**3** (253.7 mg, 1.0 mmol), and **2j** (171.6 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jj** (90.6 mg, 59%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 94% ee (HPLC conditions: Chiralcel As-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 30.9 min, t_R(minor) = 32.9 min); $[\alpha]_D^{20.0}$ = -72.5 (c = 1.02, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 5.40-5.18 (m, 2 H, CH=C=CH), 4.11 (br s, 2 H, OCH_2), 2.10-1.96 (m, 2 H, CH_2), 1.78 (br s, 1 H, OH), 1.49-1.12 (m, 8 H, CH_2 × 4), 0.89 (t, J = 6.8 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.0, 93.9, 91.6, 60.7, 31.6, 29.0, 28.7, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3321, 2957, 2924, 2855, 1963, 1462, 1418, 1378, 1352, 1261, 1213, 1060, 1010; MS (EI) m/z (%): 154 (M^+, 0.11), 133 ($(M-H_2O)^+$, 0.26), 55 (100); HRMS calcd for $C_{10}H_{18}O$ [M^+]: 154.1358, found: 154.1354.

4. Preparation of (R_a)-2,3-undecadien-1-ol (R_a)-4ja. (hx-11-198)



The reaction of $CuBr_2$ (45.1 mg, 0.2 mmol), (*S*)-**3** (253.6 mg, 1.0 mmol), **1j** (84.5 mg, 1.5 mmol), and **2a** (192.5 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ja** (101.3 mg, 60%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid:[1] 95% ee (HPLC conditions: Chiralcel As-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 20.1 min, t_R(minor) = 21.3 min); $[\alpha]_D^{20.0}$ = -65.9 (*c* = 1.215, $CHCl_3$); (reported value: 97% ee; $[\alpha]_D^{20}$ = -66.1 (c = 1.03, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.38-5.18 (m, 2 H, CH=C=CH), 4.17-3.98 (m, 2 H, OCH_2), 2.21-1.87 (m, 3 H,OH + CH_2), 1.49-1.12 (m, 10 H, CH_2 × 5), 0.88 (t, *J* = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.0, 93.9, 91.6, 60.7, 31.8, 29.07, 29.05, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3336, 2956, 2926, 2855, 1963, 1465, 1376, 1013; MS (EI): *m/z* (%) 168 (M^+, 0.04), 55 (100).

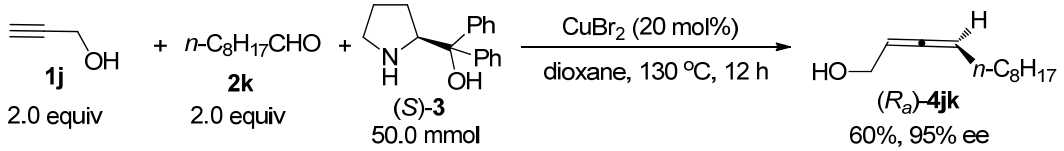
5. Preparation of (R_a)-2,3-undecadien-1-ol (R_a)-4ja. (hx-12-59)



The reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1j** (113.7 mg, 2.0 mmol), (*S*)-**3** (254.0 mg, 1.0 mmol), and **2a** (255.6 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ja** (109.8 mg, 65%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 95% ee (HPLC conditions: Chiralcel As-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 26.7 min, t_R(minor) = 28.4 min); $[\alpha]_D^{20.0}$ = -65.6 (*c* = 0.905, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.42-5.13 (m, 2 H, CH=C=CH), 4.21-4.03 (m,

2 H, OCH_2), 2.10-1.95 (m, 2 H, CH_2), 1.79 (br s, 1 H, OH), 1.49-1.14 (m, 10 H, CH_2 × 5), 0.88 (t, J = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.0, 93.9, 91.6, 60.7, 31.8, 29.06, 29.04, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3336, 2956, 2926, 2855, 1963, 1465, 1376, 1013; MS (EI): *m/z* (%) 168 (M^+, 0.04), 55 (100).

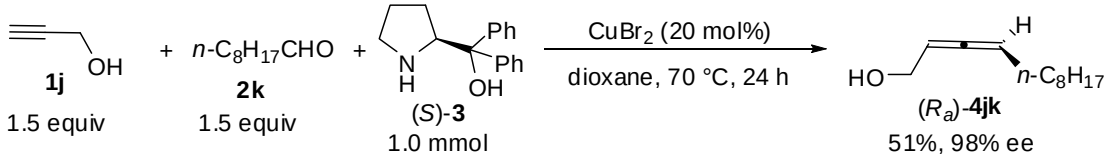
6. Preparation of (R_a)-dodeca-2,3-dien-1-ol (R_a)-4jk. (hx-12-62)



To a flame-dried three-neck flask with a reflux condenser were added $CuBr_2$ (2.2406 g, 10.0 mmol), (*S*)-**3** (12.6504 g, 50.0 mmol), **1j** (5.6047 g, 100.0 mmol)/dioxane (25 mL), and **2k** (14.2570 g, 100.0 mmol)/dioxane (10 mL) sequentially under nitrogen atmosphere. The reaction was complete after being stirred in an oil bath preheated at 130 °C for 12 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 5/1). After cooling to room temperature, the resulting mixture was diluted with ether (200 mL) and washed with an aqueous solution of hydrochloric acid (3 M, 200 mL). The organic layer was separated, and the aqueous layer was extracted with ether (200 mL × 2). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography on silica gel to afford afforded (R_a)-**4jk** (5.4270 g, 60%) (eluent: petroleum ether/ethyl acetate = 20/1 to 10/1) as a liquid: 95% ee (HPLC conditions: Chiralcel As-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 24.0 min, t_R(minor) = 25.6 min); $[\alpha]_D^{26.1}$ = -61.7 (*c* = 0.98, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 5.40-5.20 (m, 2 H, CH=C=CH), 4.21-4.02 (m, 2 H,

OCH_2), 2.09-1.92 (m, 2 H, CH_2), 1.69 (br s, 1 H, OH), 1.49-1.12 (m, 12 H, $CH_2 \times 6$), 0.88 (t, J = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.0, 93.8, 91.6, 60.7, 31.8, 29.3, 29.2, 29.1, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3328, 2924, 2856, 1964, 1462, 1378, 1263, 1211, 1057, 1014; MS (EI) m/z 182 (M^+, 0.52), 55 (100); HRMS calcd. for $C_{12}H_{22}O$ [M^+]: 182.1671, Found:182.1667.

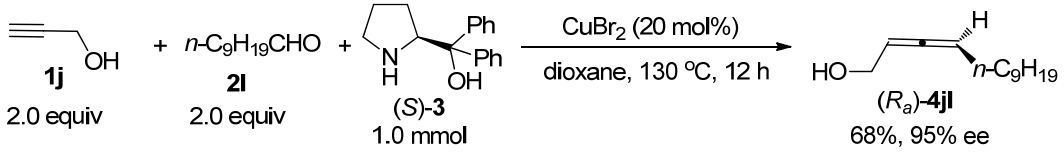
7. Preparation of (R_a)-dodeca-2,3-dien-1-ol (R_a)-4jk. (ct-6-79)



Typical Procedure II: To a flame-dried Schlenk tube with a rubber plug were added $CuBr_2$ (45.7 mg, 0.2 mmol), (*S*)-**3** (253.8 mg, 1.0 mmol), **1j** (84.6 mg, 1.5 mmol)/dioxane (1.5 mL), and **2k** (213.1 mg, 1.5 mmol)/dioxane (1.5 mL) sequentially under nitrogen atmosphere (For an apparatus, see page S3 in SI). The reaction mixture was then stirred in an oil bath preheated at 70 ^oC for 24 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 10/1). The resulting mixture was diluted with ether (30 mL), and washed with an aqueous solution of hydrochloric acid (3 M, 20 mL). The organic layer was separated, and the aqueous layer was extracted with ether (20 mL). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography (eluent: petroleum ether/ethyl acetate = 8/1) on silica gel to afford (R_a)-**4jk** (93.1 mg, 51%) as a liquid: 98% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 300/1, 1 mL/min, λ = 214 nm, t_R(major) = 11.9 min, t_R(minor) = 12.5 min); $[\alpha]_D^{27}$ = -59.5 (c = 0.98, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$)

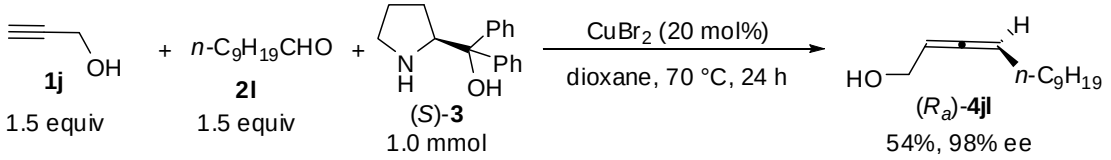
δ 5.34-5.24 (m, 2 H, CH=C=CH), 4.13-4.07 (m, 2 H, OCH_2), 2.06-1.98 (m, 2 H, CH_2), 1.88 (br s, 1 H, OH), 1.45-1.20 (m, 12 H, $CH_2 \times 6$), 0.88 (t, J = 6.2 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 93.8, 91.6, 60.7, 31.8, 29.3, 29.2, 29.1, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3328, 2924, 2856, 1964, 1462, 1378, 1263, 1211, 1057, 1014; MS (EI) m/z 182 (M^+, 0.52), 55 (100); HRMS calcd. for $C_{12}H_{22}O$ [M^+]: 182.1671, Found:182.1667.

8. Preparation of (R_a)-2,3-tridecadien-1-ol (R_a)-4jl. (lwl-5-118)



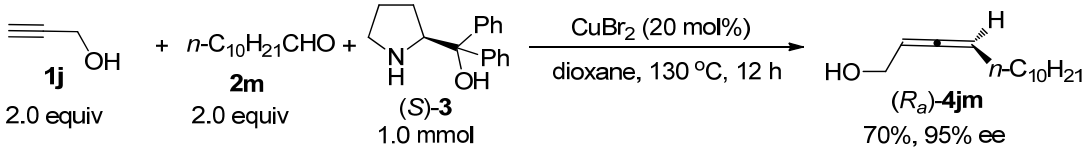
The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1j** (112.2 mg, 2.0 mmol), (*S*)-**3** (253.3 mg, 1.0 mmol), and **2l** (312.4 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jl** (133.5 mg, 68%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 95% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 300/1, 0.7 mL/min, λ = 214 nm, t_R(major) = 16.9 min, t_R(minor) = 17.7 min); $[\alpha]_D^{26}$ = -57.7 (c = 1.10, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 5.35-5.25 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.2 Hz, J_2 = 2.8 Hz, 2 H, OCH_2), 2.05-1.98 (m, 2 H, CH_2), 1.67 (br s, 1 H, OH), 1.45-1.20 (m, 14 H, $CH_2 \times 6$), 0.88 (t, J = 6.8 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.9, 94.0, 91.7, 60.8, 31.9, 29.5, 29.4, 29.3, 29.1, 29.0, 28.6, 22.6, 14.1; IR (neat) ν (cm^{-1}) 3317, 2955, 2922, 2853, 1963, 1463, 1056, 1011; MS (EI) m/z (%): 196 (M^+, 0.18), 178 ($(M-H_2O)^+$, 0.95), 84 (100); HRMS calcd for $C_{13}H_{24}O$ [M^+]: 196.1827, found: 196.1830.

9. Preparation of (R_a)-2,3-tridecadien-1-ol (R_a)-4jl. (ct-6-81)



Following **Typical Procedure II**, the reaction of $CuBr_2$ (43.8 mg, 0.2 mmol), **1j** (84.4 mg, 1.5 mmol), (*S*)-**3** (253.3 mg, 1.0 mmol), and **2l** (233.7 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jl** (105.8 mg, 54%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 98% ee (HPLC conditions: Chiralcel IA-H column, hexane/*i*-PrOH = 300/1, 1 mL/min, λ = 214 nm, t_R(major) = 17.7 min, t_R(minor) = 19.0 min); $[\alpha]_D^{27}$ = -57.5 (*c* = 1.045, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.35-5.23 (m, 2 H, CH=C=CH), 4.14-4.06 (m, 2 H, OCH_2), 2.06-1.98 (m, 2 H, CH_2), 1.88 (br s, 1 H, OH), 1.44-1.21 (m, 14 H, CH_2 × 7), 0.88 (t, *J* = 6.4 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 93.8, 91.6, 60.7, 31.8, 29.5, 29.4, 29.3, 29.1, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3317, 2955, 2922, 2853, 1963, 1463, 1056, 1011; MS (EI) m/z (%): 196 (M^+, 0.18), 178 ($(M-H_2O)^+$, 0.95), 84 (100); HRMS calcd for $C_{13}H_{24}O$ [M^+]: 196.1827, found: 196.1830.

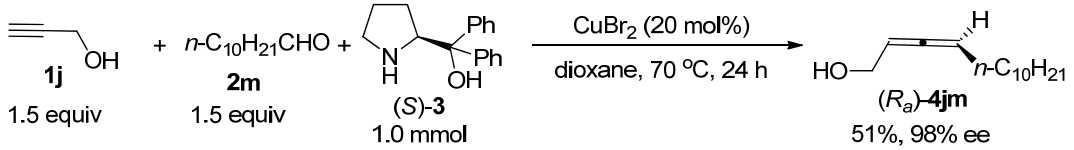
10. Preparation of (R_a)-2,3- tetradecdien-1-ol (R_a)-4jm. (hanyl-3-156)



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1j** (111.8 mg, 2.0 mmol), (*S*)-**3** (257.9 mg, 1.0 mmol), and **2m** (339.8 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jm** (146.6 mg, 70%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 95% ee (HPLC conditions: Chiralcel IA column, hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 20.3 min, t_R(minor) = 22.0 min); $[\alpha]_D^{29.6}$ = -52.8 (*c* = 1.01,

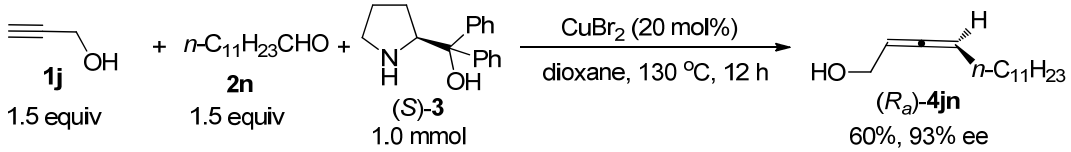
$CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 5.38-5.24 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.4 Hz, J_2 = 3.0 Hz, 2 H, OCH_2), 2.09-1.96 (m, 2 H, CH_2), 1.82 (br s, 1 H, OH), 1.46-1.20 (m, 16 H, CH_2), 0.88 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.0, 93.8, 91.6, 60.7, 31.9, 29.58, 29.56, 29.4, 29.3, 29.1, 29.0, 28.6, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3319, 2922, 2853, 1963, 1463, 1057, 1011; MS (EI) m/z (%): 210 (M^+, 0.21), 192 ($(M-H_2O)^+$, 1.09), 84 (100); HRMS calcd for $C_{14}H_{26}O$ [M^+]: 210.1984, found: 210.1987.

11. Preparation of (R_a)-2,3- tetradecdien-1-ol (R_a)-4jm. (lwl-5-103)



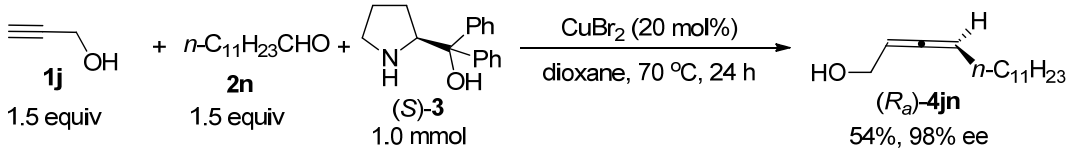
Following **Typical Procedure II**, the reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1j** (84.2 mg, 1.5 mmol), (*S*)-**3** (253.3 mg, 1.0 mmol), and **2m** (255.5 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jm** (107.1 mg, 51%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 98% ee (HPLC conditions: Chiralcel IA column, hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 17.7 min, t_R(minor) = 19.1 min); $[\alpha]_D^{27}$ = -56.6 (c = 1.02, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 5.36-5.25 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.6 Hz, J_2 = 3.2 Hz, 2 H, OCH_2), 2.06-1.98 (m, 2 H, CH_2), 1.59 (br s, 1 H, OH), 1.45-1.21 (m, 16 H, CH_2 × 8), 0.88 (t, J = 7.0 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.9, 94.0, 91.7, 60.8, 31.9, 29.60, 29.59, 29.4, 29.3, 29.11, 29.06, 28.6, 22.7, 14.1; IR (neat) ν (cm^{-1}) 3319, 2922, 2853, 1963, 1463, 1057, 1011; MS (EI) m/z (%): 210 (M^+, 0.21), 192 ($(M-H_2O)^+$, 1.09), 84 (100); HRMS calcd for $C_{14}H_{26}O$ [M^+]: 210.1984, found: 210.1987.

12. Preparation of (R_a)-pentadeca-2,3-dien-1-ol (R_a)-4jn. (jxg-1-41)



The reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1j** (84.9 mg, 1.5 mmol), (*S*)-**3** (253.8 mg, 1.0 mmol), and **2n** (291.2 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jn** (133.7 mg, 60%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 93% ee (HPLC conditions: Chiralcel IA-H column, hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 24.4 min, t_R(minor) = 27.7 min); $[\alpha]_D^{25.9}$ = -52.1 (*c* = 0.99, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.36-5.25 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.4 Hz, J_2 = 3.0 Hz, 2 H, OCH_2), 2.07-1.97 (m, 2 H, CH_2), 1.77 (s, 1 H, OH), 1.45-1.21 (m, 18 H, CH_2 × 9), 0.88 (t, *J* = 7.0 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ202.9, 93.9, 91.6, 60.7, 31.9, 29.62, 29.59. 29.4, 29.3. 29.1, 29.0, 28.6, 22.6, 14.1; IR (neat) ν (cm^{-1}) 3328, 2922, 2853, 1963, 1462, 1377, 1353, 1275, 1212, 1058, 1012; MS (EI) *m/z* 224 (M^+, 0.13), 84 (100); HRMS calcd. for $C_{15}H_{28}O$ [M^+]: 224.2140, Found: 224.2142.

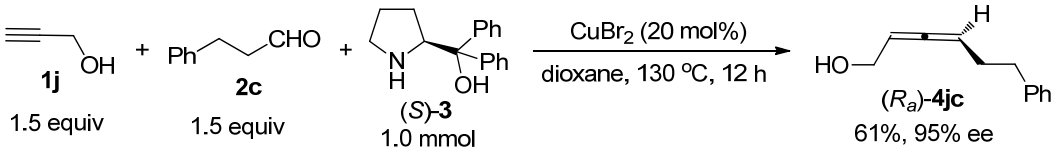
13. Preparation of (R_a)-pentadeca-2,3-dien-1-ol (R_a)-4jn. (lwl-5-104)



Following **Typical Procedure II**, the reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1j** (84.0 mg, 1.5 mmol), (*S*)-**3** (253.2 mg, 1.0 mmol), and **2n** (276.4 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jn** (121.1 mg, 54%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 98% ee (HPLC conditions: Chiralcel IA-H column,

hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 26.8 min, t_R(minor) = 31.0 min); $[\alpha]_D^{27}$ = -55.3 (*c* = 1.06, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.36-5.25 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.8 Hz, J_2 = 3.0 Hz, 2 H, OCH_2), 2.06-1.98 (m, 2 H, CH_2), 1.61 (br s, 1 H, OH), 1.45-1.21 (m, 18 H, CH_2 × 9), 0.88 (t, *J* = 7.0 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.9, 94.0, 91.7, 60.8, 31.9, 29.63, 29.60. 29.4, 29.3, 29.10, 29.06, 28.6, 22.7, 14.1; IR (neat) ν (cm^{-1}) 3328, 2922, 2853, 1963, 1462, 1377, 1353, 1275, 1212, 1058, 1012; MS (EI) *m/z* 224 (M^+, 0.13), 84 (100); HRMS calcd. for $C_{15}H_{28}O$ [M^+]: 224.2140, Found: 224.2142.

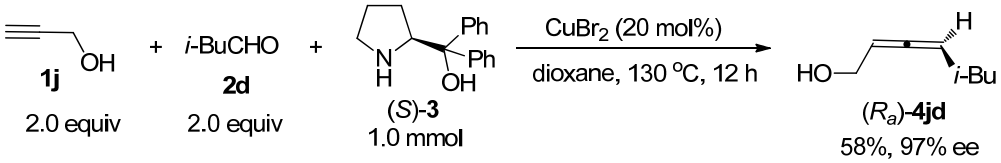
14. Preparation of (R_a)-6-phenyl-2,3-hexadien-1-ol (R_a)-4jc. (hx-10-166, hx-10-167)



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1j** (84.2 mg, 1.5 mmol), (*S*)-**3** (253.1 mg, 1.0 mmol), and **2c** (201.4 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jc** (106.1 mg, 61%) (eluent: petroleum ether/ethyl acetate = 8/1 to petroleum ether/ethyl acetate = 5/1) as a liquid:[1] 95% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 22.4 min, t_R(minor) = 24.4 min); $[\alpha]_D^{20}$ = -38.0 (*c* = 0.89, $CHCl_3$) (reported value: 96% ee; $[\alpha]_D^{20}$ = -38.7 (c = 1.05, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.13 (m, 5 H, ArH), 5.35-5.21 (m, 2 H, CH=C=CH), 4.04-3.90 (m, 2 H, OCH_2), 2.83-2.64 (m, 2 H, CH_2), 2.46-2.24 (m, 2 H, CH_2), 1.62 (s, 1 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.2, 141.4, 128.4, 128.2, 125.9, 92.9, 92.1, 60.4, 35.0, 29.9; IR (neat) ν (cm^{-1}) 3366, 3084, 3062, 3026,

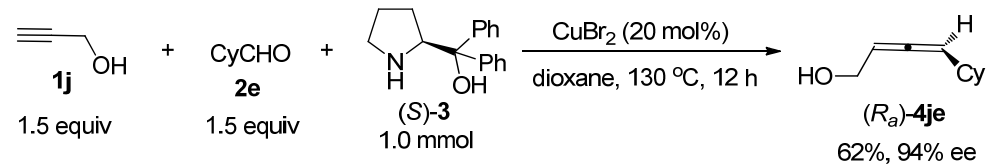
2923, 2856, 1962, 1603, 1496, 1453, 1062, 1011; MS (EI) m/z (%): 174 (M^+, 0.03), 156 ($(M-H_2O)^+$, 41.67), 91 (100).

15. Preparation of (R_a)-6-methyl-2,3-heptadien-1-ol (R_a)-4jd. (jxg-1-93)



The reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1j** (113.0 mg, 2.0 mmol), (*S*)-**3** (253.6 mg, 1.0 mmol), and **2d** (172.8 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jd** (73.9 mg, 58%) (eluent: petroleum ether/ethyl acetate = 15/1) as a liquid:[1] 97% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 15.1 min, t_R(minor) = 16.7 min); $[\alpha]_D^{29.5}$ = -79.9 (*c* = 0.970, $CHCl_3$) (reported value: 98% ee; $[\alpha]_D^{22}$ = -80.3 (c = 1.01, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.34-5.21 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 6.0 Hz, J_2 = 2.8 Hz, 2 H, OCH_2), 1.96-1.89 (m, 2 H, CH_2), 1.75 (br s, 1 H, OH), 1.73-1.62 (m, 1 H, CH), 0.93 (d, *J* = 6.4 Hz, 6 H, Me × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.6, 92.3, 91.0, 60.8, 38.1, 28.3, 22.11, 22.09; IR (neat) ν (cm^{-1}) 3338, 2956, 2926, 2893, 2870, 1962, 1466, 1384, 1367, 1056, 1014; MS (EI) m/z (%): 126 (M^+, 0.10), 108 ($(M-H_2O)^+$, 31.12), 55 (100).

16. Preparation of (R_a)-4-cyclohexyl-2,3-butadien-1-ol (R_a)-4je. (hx-10-161, hx-10-162)

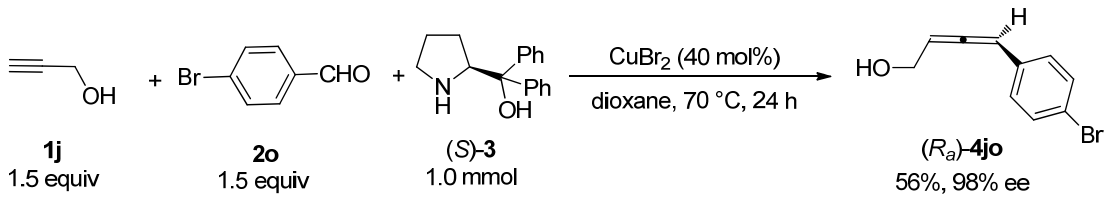


The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1j** (84.8 mg, 1.5 mmol), (*S*)-**3** (252.2

mg, 1.0 mmol), and **2e** (168.2 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4je** (94.2 mg, 62%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid:[1] 94% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 98/2, 0.6 mL/min, λ = 214 nm, t_R(major) = 15.7 min, t_R(minor) = 18.6 min); $[\alpha]_D^{20}$ = -98.1 (*c* = 1.045, $CHCl_3$) (reported value: 99% ee; $[\alpha]_D^{22}$ = -100.3 (c = 1.00, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.41-5.25 (m, 2 H, CH=C=CH), 4.10 (dd, J_1 = 5.7 Hz, J_2 = 3.0 Hz, 2 H, OCH_2), 2.08-1.93 (m, 1 H, CH from Cy), 1.88-1.58 (m, 6 H, OH and five protons from Cy), 1.37-1.00 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 99.9, 92.6, 60.8, 37.0, 33.0, 32.9, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3331, 2924, 2851, 1961, 1448, 1412, 1011; MS (EI) m/z (%): 152 (M^+, 0.38), 134 ($(M-H_2O)^+$, 6.78), 55 (100).

17. Preparation of (R_a)-4-(4-bromophenyl)-2,3-butadien-1-ol (R_a)-4jo.

(hanyl-3-077)

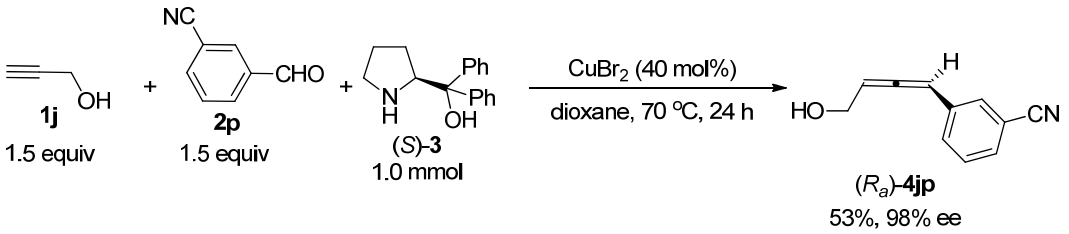


1j 1.5 equiv | **2o** 1.5 equiv | (*S*)-**3** 1.0 mmol | (R_a)-**4jo** 56%, 98% ee

Following **Typical Procedure II**, the reaction of $CuBr_2$ (88.8 mg, 0.4 mmol), **1j** (84.5 mg, 1.5 mmol), (*S*)-**3** (253.5 mg, 1.0 mmol), and **2o** (279.3 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jo** (125.3 mg, 56%) (eluent: petroleum ether/ethyl acetate = 8/1) as a solid: 98% ee (HPLC conditions: Chiralcel IB column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 12.4 min, t_R(minor) = 13.2 min); $[\alpha]_D^{25.3}$ = -220.1 (*c* = 1.01, $CHCl_3$); ^{1}H NMR (400M Hz, $CDCl_3$) δ 7.40 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.14 (d, *J* = 8.4 Hz, 2 H, Ar-H), 6.28-6.18 (m, 1 H, C=CH),

5.77 (dd, J = 6 Hz, 1 H, C=CH), 4.29-4.19 (m, 2 H, C=CCH_2), 2.22-2.07 (s, 1 H, OH); ^{13}C NMR (100M Hz, $CDCl_3$) δ 204.3, 132.8, 131.6, 128.3, 120.8, 96.15, 96.13, 60.1; IR (neat) ν = 3334, 3242, 2946, 2923, 2852, 1947, 1893, 1710, 1644, 1587, 1570, 1484, 1422, 1386, 1343, 1213, 1109, 1068, 1048, 1026, 1007 cm^{-1}; MS (EI): *m/z* (%) 226 (M^+(^{81}Br), 32.41), 224 (M^+(^{79}Br), 34.15), 115 (100); Anal. Calcd. for $C_{10}H_9BrO$: C 53.27, H 4.04; Found: C 53.36, H 4.03.

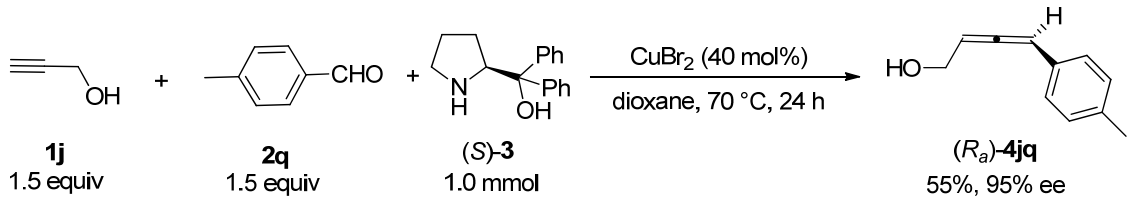
18. Preparation of (R_a)-4-(4-cyanphenyl)-2,3-butadien-1-ol (R_a)-4jp. (lwl-5-114)



Following **Typical Procedure II**, the reaction of $CuBr_2$ (89.4 mg, 0.4 mmol), **1j** (84.2 mg, 1.5 mmol), (*S*)-**3** (253.2 mg, 1.0 mmol), and **2p** (196.6 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jp** (90.7 mg, 53%) (isolated via column chromatography at 0 °C; eluent: petroleum ether/ethyl acetate = 5/1) as a liquid: 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.2 mL/min, λ = 214 nm, t_R(major) = 32.1 min, t_R(minor) = 29.8 min); $[\alpha]_D^{26}$ = -245.5 (c = 0.95, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (s, 1 H, ArH), 7.54-7.49 (m, 1 H, ArH), 7.49-7.44 (m, 2 H, ArH), 7.39 (t, J = 7.8 Hz, 2 H, ArH), 6.31-6.25 (m, 1 H, CH=), 5.86 (q, J_1 = 6.0 Hz, 1 H, =CH), 4.35-4.24 (m, 2 H, OCH_2), 2.37 (br s, 1 H, OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 135.5, 130.9, 130.3, 130.0, 129.3, 118.6, 112.5, 96.8, 95.3, 59.8; IR (neat) ν (cm^{-1}) 3380, 2930, 2872, 2230, 1950, 1598, 1578, 1482, 1009; MS (EI) m/z (%): 171 (M^+, 43.09), 140 (100); HRMS calcd for $C_{11}H_9NO$ [M^+]:

171.0684, found: 171.0683.

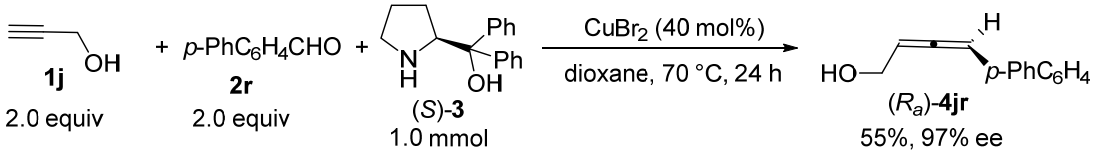
19. Preparation of (R_a)-4-(4-methylphenyl)-2,3-butadien-1-ol (R_a)-4jq. (hanyl-3-144)



1j 1.5 equiv | **2q** 1.5 equiv | (*S*)-**3** 1.0 mmol | (R_a)-**4jq** 55%, 95% ee

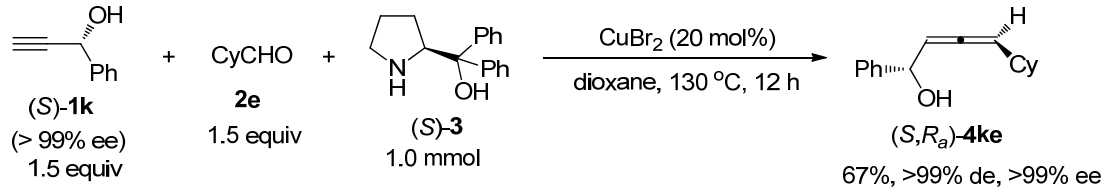
Following **Typical Procedure II**, the reaction of $CuBr_2$ (89.9 mg, 0.2 mmol), **1j** (84.6 mg, 1.5 mmol), (*S*)-**3** (258.1 mg, 1.0 mmol), and **2q** (181.3 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jq** (87.7 mg, 55%) (isolated via column chromatography at 0 ^oC; eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 95% ee (HPLC conditions: Chiralcel AY-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 16.6 min, t_R(minor) = 15.4 min); $[\alpha]_D^{26.8}$ = -159.6 (*c* = 0.995, $CHCl_3$); ^1H NMR (400M Hz, $CDCl_3$): δ 7.18 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.10 (d, *J* = 8.0 Hz, 2 H, Ar-H), 6.31-6.24 (m, 1 H, =CH), 5.74 (q, *J* = 6.0 Hz, 1 H, =CH), 4.26-4.18 (m, 2 H, $=CCH_2$), 2.32 (s, 3 H, $ArCH_3$), 1.96 (brs, 1 H, OH); ^{13}C NMR (100M Hz, $CDCl_3$) δ 203.9, 137.0, 130.7, 129.3, 126.7, 96.9, 95.6, 60.4, 21.1; IR (neat) ν= 3316, 3022, 2985, 2912, 2856, 2723, 2014, 1945, 1912, 1803, 1667, 1573, 1509, 1464, 1434, 1392, 1344, 1311, 1265, 1228, 1102, 1049, 1012 cm^{-1}; MS (EI) m/z (%): 160 (M^+, 51.03), 129 (100); HRMS calcd for $C_{11}H_{12}O$ [M^+]: 160.0888, found: 160.0891.

20. Preparation of (R_a)-4-(4-phenyl)phenyl-2,3-butadien-1-ol (R_a)-4jr. (zjs-3-162)



Following **Typical Procedure II**, the reaction of $CuBr_2$ (90.2 mg, 0.4 mmol), **1j** (113.4 mg, 2.0 mmol), (*S*)-**3** (258.2 mg, 1.0 mmol), and **2r** (364.0 mg, 2.0 mmol) in dioxane (3.0 mL) afforded (R_a)-**4jr** (123.9 mg, 55%) (eluent: petroleum ether/ethyl acetate = 8/1) as a solid, m.p. 124-125 ^{o}C (ethyl acetate/*n*-hexane) 97% ee (HPLC conditions: Chiralcel AY-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R(minor) = 25.3 min, t_R(major) = 27.0 min); $[\alpha]_D^{26}$ = -257.0 (*c* = 1.00, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.51 (m, 4 H, ArH), 7.45-7.30 (m, 5 H, ArH), 6.38-6.32 (m, 1 H, =CH), 5.80 (q, *J* = 6.1 Hz, 1 H, =CH), 4.33-4.14 (m, 2 H, CH_2), 1.76 (br s, 1 H, OH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 140.6, 140.0, 132.8, 128.7, 127.32, 127.25, 127.2, 126.9, 96.7, 95.9, 60.3; IR (neat) ν (cm^{-1}) 3196, 3056, 3032, 2928, 2869, 1944, 1597, 1562, 1483, 1424, 1395, 1350, 1257, 1211, 1106, 1047, 1017; MS (EI) m/z (%): 222 (M^+, 64.36), 191 (100); HRMS calcd for $C_{16}H_{14}O$ [M^+]: 222.1045, found: 222.1050.

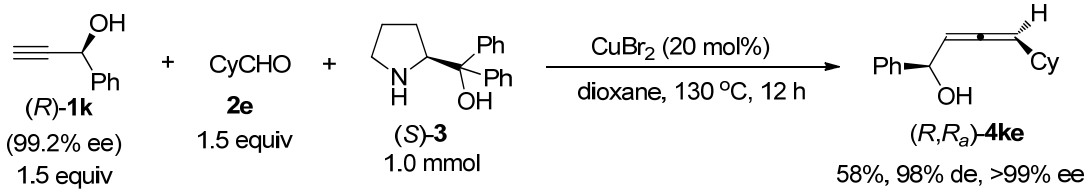
21. Preparation of (S,R_a)-4ke (hx-10-194)



The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), (*S*)-**1k** (197.6 mg, 1.5 mmol), (*S*)-**3** (252.7 mg, 1.0 mmol), and **2e** (168.3 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (S,R_a)-**4ke** (152.0 mg, 67%) (eluent: petroleum ether/ethyl acetate = 12/1) as a liquid:[2] >99% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H

column, hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R(major) = 19.4 min; $[\alpha]_D^{20}$ = -69.7 (*c* = 1.24, $CHCl_3$) (reported value: 92% de, >99% ee; $[\alpha]_D^{20}$ = -60.7 (c = 1.02, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.21 (m, 5 H, ArH), 5.47-5.38 (m, 1 H, one proton from HC=C=CH), 5.38-5.30 (m, 1 H, one proton from HC=C=CH), 5.21 (d, *J* = 5.4 Hz, 1 H, PhCH), 2.33 (s, 1 H, OH), 2.07-1.91 (m, 1 H, CH from Cy), 1.80-1.56 (m, 5 H, five protons from Cy), 1.35-0.96 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 143.2, 128.3, 127.5, 126.0, 100.9, 96.9, 72.3, 37.1, 32.9, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3365, 3063, 3029, 2924, 2850, 1960, 1599, 1489, 1449, 1015; MS (EI) m/z (%): 228 (M^+, 2.53), 107 (100).

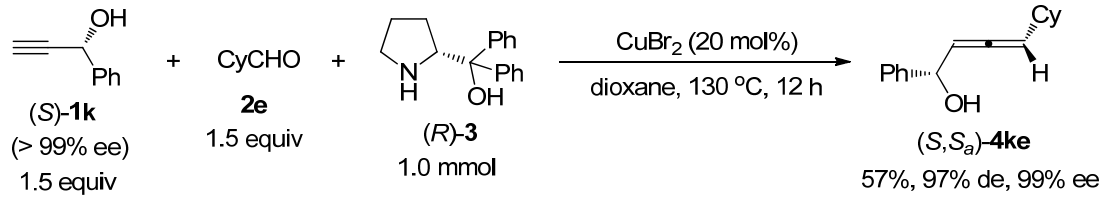
22. Preparation of (*R*,*R*$_a$)-4ke (hx-11-6)



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), (*R*)-**1k** (198.5 mg, 1.5 mmol), (*S*)-**3** (253.7 mg, 1.0 mmol), and **2e** (168.1 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (*R*,*R*$_a$)-**4ke** (133.3 mg, 58%) (eluent: petroleum ether/ethyl acetate = 12/1) as a liquid:[2] 98% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R(major) = 11.1 min; $[\alpha]_D^{20}$ = -52.6 (*c* = 0.98, $CHCl_3$) (reported value: 94% de, 97% ee; $[\alpha]_D^{20}$ = -56.8 (c = 0.98, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.29 (m, 4 H, ArH), 7.29-7.21 (m, 1 H, ArH), 5.47-5.38 (m, 1 H, one proton from HC=C=CH), 5.37-5.30 (m, 1 H, one proton from HC=C=CH), 5.18 (dd, J_1 = 5.9 Hz, J_2 = 2.6 Hz, 1 H, PhCH), 2.30 (s, 1 H, OH), 2.07-1.93 (m, 1 H, CH from Cy), 1.80-1.56 (m, 5 H, five protons from Cy), 1.36-0.98

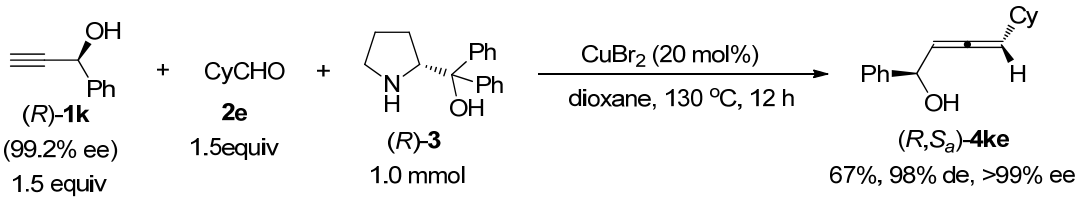
(m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.9, 143.1, 128.3, 127.5, 126.1, 101.2, 97.0, 72.1, 37.1, 32.9, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3373, 3063, 3029, 2924, 2850, 1961, 1599, 1493, 1449, 1014; MS (EI) m/z (%): 228 (M^+, 2.17), 107 (100).

23. Preparation of (*S*,*S*$_a$)-4ke (hx-11-12)



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), (*S*)-**1k** (198.2 mg, 1.5 mmol), (*R*)-**3** (253.8 mg, 1.0 mmol), and **2e** (167.9 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (*S*,*S*$_a$)-**4ke** (131.5 mg, 57%) (eluent: petroleum ether/ethyl acetate = 12/1) as a liquid:[2] 97% de, 99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R(minor) = 11.7 min, t_R(major) = 16.1 min; $[\alpha]_D^{20}$ = +56.6 (*c* = 1.34, $CHCl_3$) (reported value: 89% de, >99% ee; $[\alpha]_D^{20}$ = +54.7 (c = 1.17, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.21 (m, 5 H, ArH), 5.47-5.38 (m, 1 H, one proton from HC=C=CH), 5.37-5.30 (m, 1 H, one proton from HC=C=CH), 5.18 (dd, J_1 = 5.7 Hz, J_2 = 2.4 Hz, 1 H, PhCH), 2.33 (s, 1 H, OH), 2.08-1.93 (m, 1 H, CH from Cy), 1.80-1.56 (m, 5 H, five protons from Cy), 1.36-0.98 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.9, 143.1, 128.3, 127.5, 126.1, 101.2, 97.0, 72.1, 37.0, 32.9, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3365, 3062, 3029, 2924, 2850, 1961, 1602, 1492, 1449, 1014; MS (EI) m/z (%): 228 (M^+, 1.93), 107 (100).

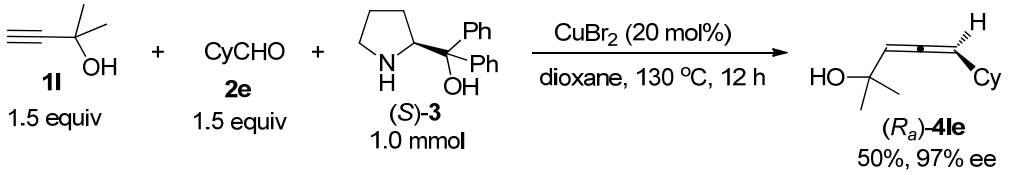
24. Preparation of (*R*,*S*$_a$)-4ke (hx-11-13)



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), (*R*)-**1k** (197.6 mg, 1.5 mmol), (*R*)-**3** (254.0 mg, 1.0 mmol), and **2e** (169.2 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (*R*,S_a)-**4ke** (153.4 mg, 67%) (eluent: petroleum ether/ethyl acetate = 12/1) as a liquid:[2] 98% de, >99% ee (major isomer) (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R(major) = 13.4 min; $[\alpha]_D^{20}$ = +73.0 (*c* = 1.165, $CHCl_3$) (reported value: 93% de, >99% ee; $[\alpha]_D^{20}$ = +60.8 (c = 0.62, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.21 (m, 5 H, ArH), 5.47-5.38 (m, 1 H, one proton from HC=C=CH), 5.37-5.30 (m, 1 H, one proton from HC=C=CH), 5.19 (d, *J* = 5.7 Hz, 1 H, PhCH), 2.40 (s, 1 H, OH), 2.07-1.91 (m, 1 H, CH from Cy), 1.80-1.56 (m, 5 H, five protons from Cy), 1.35-0.97 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 143.2, 128.3, 127.5, 126.0, 100.8, 96.9, 72.3, 37.1, 32.87, 32.85, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3358, 3062, 3029, 2924, 2850, 1961, 1602, 1493, 1449, 1015; MS (EI) m/z (%): 228 (M^+, 2.23), 107 (100).

25. Preparation of (R_a)-5-Cyclohexyl-2-methyl-3,4-pentadien-2-ol (R_a)-4le.

(hx-10-172)

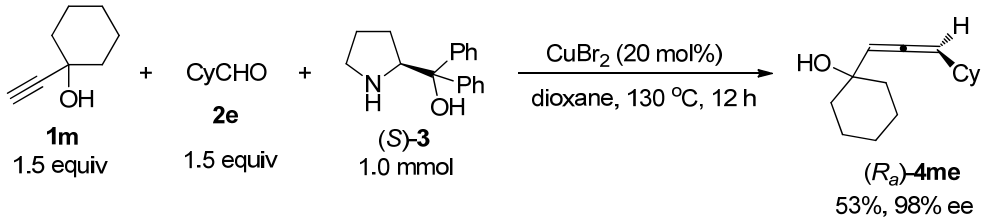


The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1l** (126.8 mg, 1.5 mmol), (*S*)-**3** (253.2 mg, 1.0 mmol), and **2e** (168.6 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4le**

(89.3 mg, 50%) (eluent: petroleum ether/ethyl acetate = 12/1) as a liquid:[2] 97% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.6 mL/min, λ = 214 nm, t_R(major) = 8.9 min, t_R(minor) = 10.2 min); $[\alpha]_D^{20}$ = -99.2 (*c* = 0.97, $CHCl_3$) (reported value: 97% ee; $[\alpha]_D^{20}$ = -99.5 (c = 1.15, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.38-5.26 (m, 2 H, CH=C=CH), 2.07-1.92 (m, 1 H, CH from Cy), 1.84-1.59 (m, 6 H, OH and five protons from Cy), 1.34 (s, 6 H, Me × 2), 1.37-0.98 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.1, 102.1, 101.0, 69.5, 37.2, 33.02, 32.99, 30.0, 29.9, 26.03, 26.00; IR (neat) ν (cm^{-1}) 3358, 2974, 2925, 2851, 1960, 1448, 1373, 1361, 1228, 1149; MS (EI) m/z (%): 180 (M^+, 0.29), 165 $(M\text{-}Me)^+$, 3.81), 59 (100).

26. Preparation of (R_a)-1-(3-Cyclohexylpropa-1,2-dienyl)cyclohexanol (R_a)-4me.

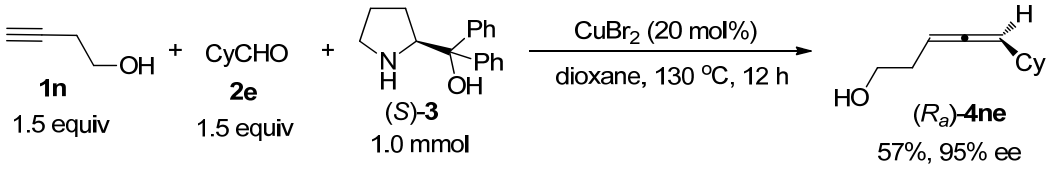
(hx-10-180, hx-10-181)



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1m** (186.8 mg, 1.5 mmol), (*S*)-**3** (252.7 mg, 1.0 mmol), and **2e** (168.2 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4me** (115.6 mg, 53%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid:[2] 98% ee (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 100/1, 0.5 mL/min, λ = 214 nm, t_R(major) = 23.5 min, t_R(minor) = 26.2 min); $[\alpha]_D^{20}$ = -106.6 (*c* = 1.21, $CHCl_3$) (reported value: 96% ee; $[\alpha]_D^{20}$ = -108.6 (c = 0.98, $CHCl_3$)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.31 (d, *J* = 4.2 Hz, 2 H, CH=C=CH), 2.08-1.90 (m, 1 H, CH from

Cy), 1.87-1.40 (m, 15 H), 1.40-1.00 (m, 6 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.9, 101.3, 100.8, 70.4, 38.4, 38.2, 37.2, 33.1, 33.0, 26.00, 25.97, 25.5, 22.4; IR (neat) ν (cm^{-1}) 3373, 2926, 2851, 1960, 1448, 1347, 1262, 1242, 1146, 1056, 1034; MS (EI) m/z (%): 220 (M^+, 0.69), 99 (100).

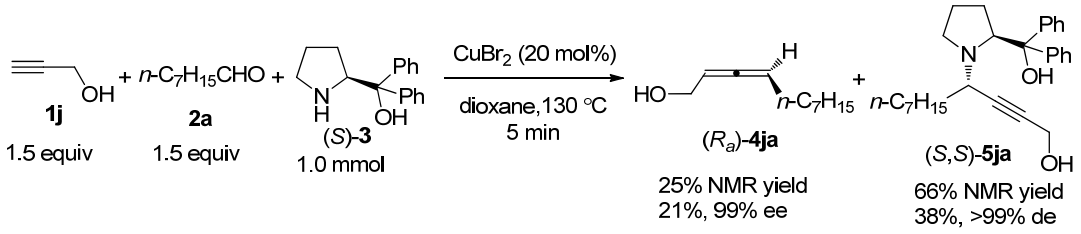
27. Preparation of (R_a)-5-cyclohexylpenta-3,4-dien-1-ol (R_a)-4ne. (hx-12-49)



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1n** (107.2 mg, 1.5 mmol), (*S*)-**3** (253.2 mg, 1.0 mmol), and **2e** (169.4 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ne** (94.4 mg, 57%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid: 95% ee (HPLC conditions: Chiralcel IC column, hexane/*i*-PrOH = 100/1, 0.6 mL/min, λ = 214 nm, t_R(minor) = 33.8 min, t_R(major) = 36.4 min); $[\alpha]_D^{20}$ = -86.7 (*c* = 0.925, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.23-4.96 (m, 2 H, CH=C=CH), 3.69 (t, *J* = 5.9 Hz, 2 H, OCH_2), 2.31-2.06 (m, 3 H, OH + CH_2), 2.05-1.87 (m, 1 H, CH from Cy), 1.82-1.52 (m, 5 H, five protons from Cy), 1.39-0.90 (m, 5 H, five protons from Cy); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.3, 97.6, 88.0, 61.9, 37.0, 33.0, 32.9, 32.3, 26.0, 25.9; IR (neat) ν (cm^{-1}) 3340, 2924, 2851, 1961, 1448, 1049; MS (EI) m/z (%): 166 (M^+, 6.05), 67 (100); HRMS calcd for $C_{11}H_{18}O$ [M^+]: 166.1358, found: 166.1365.

Control experiments in the main text.

1. Preparation of (R_a)-4ja and (S,S)-5ja. hx-11-64



To a flame-dried Schlenk tube equipped with a polytetrafluoroethylene plug were added $CuBr_2$ (44.6 mg, 0.2 mmol), (*S*)-**3** (253.7 mg, 1.0 mmol), **1j** (85.3 mg, 1.5 mmol)/dioxane (1.5 mL), and **2a** (192.1 mg, 1.5 mmol)/dioxane (1.5 mL) sequentially under nitrogen atmosphere. The Schlenk tube was then sealed by screwing the polytetrafluoroethylene plug tightly with the outlet connected to the vacuum line and the nitrogen flow being closed (For an apparatus, see page S2). The Schlenk tube was then stirred in an oil bath preheated at 130 ^{o}C for 5 min. After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with EtOAc (30 mL). After evaporation, the NMR yields for (R_a)-**4ja** (25%) and (*S*,*S*)-**5ja** (66%) were determined by ^{1}H NMR of the crude product with mesitylene (46 μL) as the internal standard. Chromatography on silica gel (eluent: petroleum ether/ethyl acetate (8/1 to 5/1)) afforded pure (*S*,*S*)-**5ja** (162.4 mg, 38%) and impure (R_a)-**4ja** (48.7 mg).

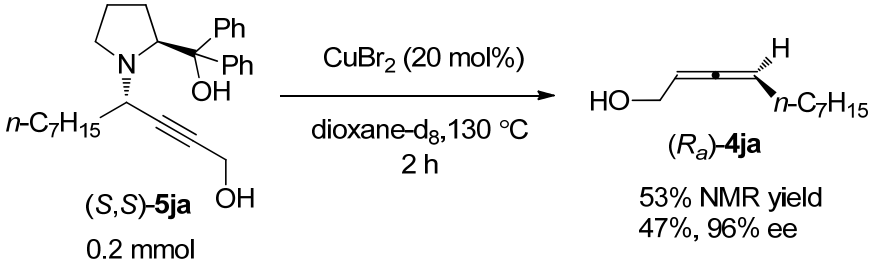
The impure (R_a)-**4ja** obtained was dissolved in ether (30 mL) and washed with an aqueous solution of hydrochloric acid (3 M, 20 mL). The organic layer was separated, and the aqueous layer was extracted with ether (20 mL). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and

evaporation, the residue was purified by chromatography (eluent: petroleum ether/ethyl acetate = 8/1) on silica gel to afford pure (R_a)-**4ja** (35.3 mg, 21%).

(R_a)-**4ja**: 99% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 100/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 12.9 min, t_R(minor) = 13.5 min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.37-5.24 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.4 Hz, J_2 = 3.3 Hz, 2 H, OCH_2), 2.08-1.96 (m, 2 H, CH_2), 1.64 (s, 1 H, OH), 1.48-1.20 (m, 10 H, CH_2 × 5), 0.88 (t, J = 6.8 Hz, 3 H, Me).

(*S*,*S*)-**5ja**: liquid; $[\alpha]_D^{20}$ = -104.1 (c = 1.05, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68-7.58 (m, 2 H, ArH), 7.57-7.50 (m, 2 H, ArH), 7.30-7.17 (m, 4 H, ArH), 7.17-7.06 (m, 2 H, ArH), 4.62 (bs, 1 H, OH), 4.28 (d, J = 1.5 Hz, 2 H, OCH_2), 4.15 (dd, J = 8.6 Hz, J = 4.4 Hz, 1 H, CH), 2.99-2.74 (m, 2 H, CH_2), 2.64 (t, J = 7.7 Hz, 1 H, CH), 2.07 (bs, 1 H, OH), 1.90-1.56 (m, 4 H, CH_2 × 2), 1.41-0.79 (m, 15 H, CH_2 × 6 and Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 146.3, 128.0, 127.9, 126.3, 126.1, 125.5, 125.3, 84.6, 82.6, 77.7, 68.6, 53.6, 51.1, 48.5, 35.0, 31.6, 29.4, 29.0, 28.7, 26.1, 24.4, 22.5, 14.0; IR (neat) ν (cm^{-1}) 3381, 3084, 3059, 3031, 2927, 2856, 2248, 1596, 1492, 1449, 1377, 1314, 1184, 1123, 1033; MS (ESI, m/z) 420 ($M+H^+$); HRMS calcd. for $C_{28}H_{38}NO_2$ ($M+H^+$): 420.2897; Found: 420.2887.

2. Conversion of (*S*,*S*)-5ja with 20 mol% of $CuBr_2$. (hx-11-80)



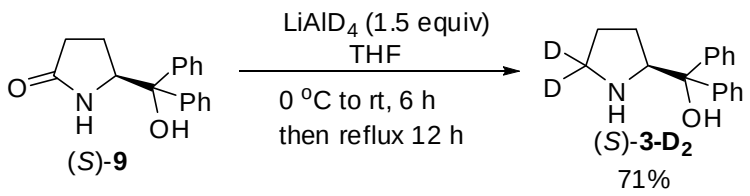
To a dried NMR-tube were added $CuBr_2$ (9.0 mg, 0.04 mmol), (S,S)-**5ja** (83.3 mg,

0.2 mmol)/dioxane-d_8 (0.6 mL), and mesitylene (18.4 μL, 0.4 mmol, the internal standard) sequentially. Then the NMR tube was sealed with a plastic cap and wrapped tightly with polytetrafluoroethylene tape followed by the first ^{1}H NMR test. Then the tube was placed into a pre-heated oil bath at 130 ^{o}C and shook with hand for every 5 min followed by the second ^{1}H NMR test. The same operations were carried out for another 5 min, 5 min, 5 min, 5 min, 5 min, 10 min, 10 min, 10 min, 30 min, 30 min respectively.

The resulting mixture was diluted with ether (20 mL) and washed with an aqueous solution of hydrochloric acid (3 M, 20 mL). The organic layer was separated and the aqueous layer was extracted with ether (20 mL). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography (eluent: petroleum ether/ethyl acetate = 8/1) on silica gel to afford (R_a)-**4ja** (15.8 mg, 47%) as a liquid: 96% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 21.2 min, t_R(minor) = 22.3 min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.37-5.24 (m, 2 H, CH=C=CH), 4.11 (dd, J_1 = 5.4 Hz, J_2 = 3.3 Hz, 2 H, OCH_2), 2.08-1.96 (m, 2 H, CH_2), 1.57 (bs, 1 H, OH), 1.48-1.20 (m, 10 H, CH_2 × 5), 0.88 (t, J = 6.8 Hz, 3 H, Me).

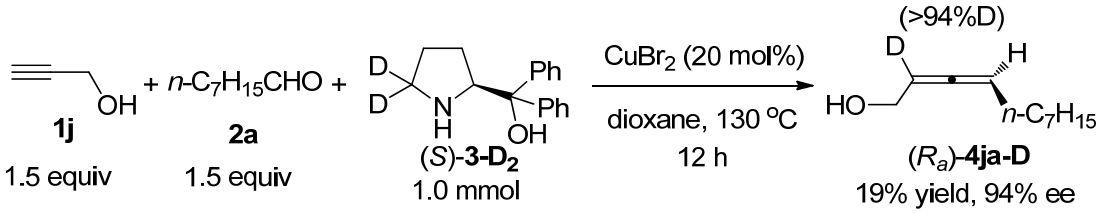
Deuterium-labeling experiment

1. Preparation of (*S*)-diphenyl(pyrrolidin-2-yl)methanol (*S*)-3-D_2. (hx-12-23)



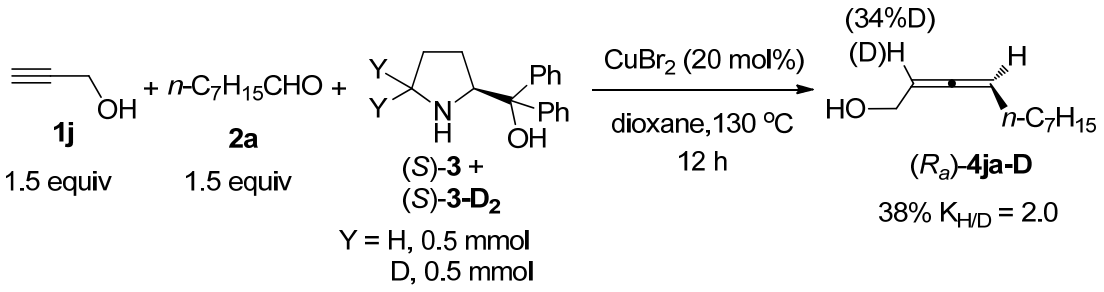
To a flame-dried three-necked bottle were added $LiAlD_4$ (0.5047 g, 12.0 mmol) and THF (30.0 mL) in a N_2 atmosphere. Then the solution was cooled to 0 °C and (*S*)-**9** (2.1365 g, 8.0 mmol) was added slowly at 0 °C. The mixture was stirred at room temperature for 6 hours and then refluxed at 80 °C for 12 hours. The reaction was quenched with 30 mL of Na_2SO_4 (aq., sat.) carefully and a lot of jelly appeared. Then 30 mL of THF were added and the resulting mixture was stirred vigorously. After standing, the upper clean liquid was separated and the remaining jelly was washed with ethyl ether (30×2 mL). The combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography (eluent: ethyl acetate to ethyl acetate/MeOH = 10/1 to dichloromethane/MeOH = 5/1) on silica gel to afford (*S*)-**3-D$_2$** (1.4460 g, 71%) as a white solid; m.p. 135-137 °C (MeOH/*n*-hexane); ^{1}H NMR (300 MHz, MeOD) δ 7.67-7.59 (m, 2 H, Ar-H), 7.55-7.49 (m, 2 H, Ar-H), 7.41-7.29 (m, 4 H, Ar-H), 7.28-7.17 (m, 2 H, Ar-H), 4.65-4.54 (m, 1 H, CHN), 1.98-1.81 (m, 4 H, 2×CH_2); ^{13}C NMR (75 MHz, MeOD) δ 146.7, 146.3, 130.6, 130.4, 129.5, 129.4, 127.7, 127.6, 79.3, 68.8, 28.0, 26.1; IR (neat) ν (cm^{-1}) 3345, 3257, 3059, 3025, 2937, 2739, 2566, 1560, 1493, 1449, 1386, 1165, 1066; MS (EI) m/z (%): 255 (M^+, 0.01), 72 (100); HRMS calcd for $C_{17}H_{17}D_2NO$ [M^+]: 255.1592, found: 255.1592.

2. Preparation of (R_a)-2,3-undecadien-1-ol (R_a)-4ja-D. (hx-12-32)



Following **Typical Procedure I:** The reaction of $CuBr_2$ (44.5 mg, 0.2 mmol), **1j** (84.9 mg, 1.5 mmol), (*S*)-**3-D$_2$** (258.3 mg, 1.0 mmol), and **2a** (192.1 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ja-D** (32.5 mg, 19%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 94% ee (HPLC conditions: Chiralcel As-H column, hexane/*i*-PrOH = 200/1, 0.6 mL/min, λ = 214 nm, t_R(major) = 29.1 min, t_R(minor) = 30.8 min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.38-5.23 (m, 1.06 H, CH=C=CH), 4.19-4.01 (m, 2 H, OCH_2), 2.11-1.92 (m, 2 H, CH_2), 1.52-1.16 (m, 11 H, OH + CH_2 × 6), 0.88 (t, J = 6.8 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) for (R_a)-**4ja-D** δ 203.0, 94.2, 91.5 (t, $J_{C\text{-}D}$ = 24.8 Hz), 60.7, 31.8, 29.1, 29.0, 28.6, 22.6, 14.1; ^{13}C NMR (75 MHz, $CDCl_3$) the following signal is discernible for (R_a)-**4ja** δ 94.1, 91.7, 60.8; IR (neat) ν (cm^{-1}) 3342, 2959, 2926, 2855, 1950, 1466, 1403, 1376, 1015; MS (EI): *m/z* (%) 169 (M^+, 0.11), 56 (100); HRMS calcd for $C_{11}H_{19}DO$ [M^+]: 169.1577, found: 169.1583.

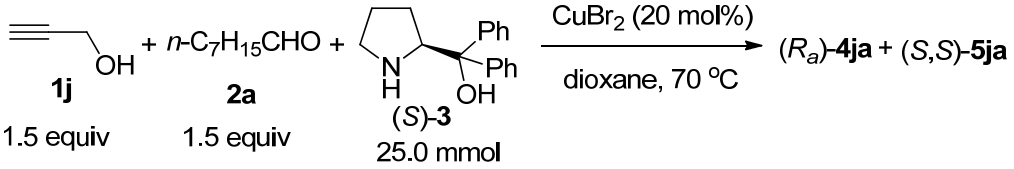
KIE study of (R_a)-2,3-undecadien-1-ol (R_a)-4ja-D. (hx-12-47)



Following **Typical Procedure I:** The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1j** (87.0 mg, 1.5 mmol), (*S*)-**3-D$_2$** (127.6 mg, 0.5 mmol), (*S*)-**3** (127.4 mg, 0.5 mmol) and **2a** (193.9 mg, 1.5 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ja-D** (63.5 mg, 19%) (eluent: petroleum ether/ethyl acetate = 8/1) as a liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.39-5.19 (m, 1.66 H, CH=C=CH), 4.19-3.90 (m, 2 H, OCH_2), 2.11-1.86 (m, 3 H, OH + CH_2), 1.50-1.16 (m, 10 H, CH_2 × 5), 0.88 (t, J = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) for **(R_a)-4ja-D** δ 202.9, 93.83, 91.34 (t, $J_{C\text{-}D}$ = 25.2 Hz), 60.67, 29.06, 29.05, 29.0, 28.6; ^{13}C NMR (75 MHz, $CDCl_3$) the following signal is discernible for **(R_a)-4ja** δ 93.77, 91.6, 60.73.

Kinetic experiments

(1) The kinetic experiment of **1j**, **2a** and *S*-**3** at 70 °C

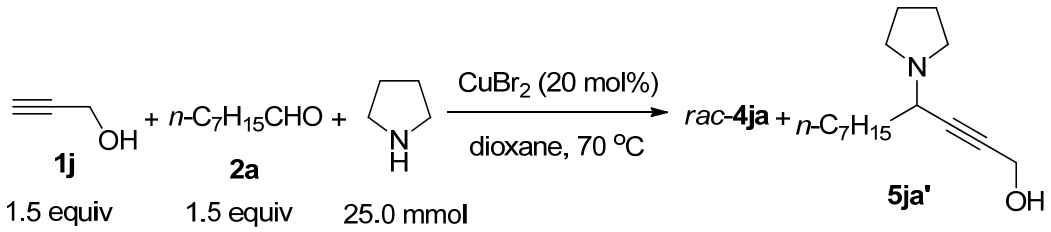


To a flame dried three-necked flask were added $CuBr_2$ (1.1208 g, 5.0 mmol), (*S*)-**3** (6.3255 g, 25 mmol), **1j** (2.1056 g, 37.5 mmol)/dioxane (35.0 mL), and **2a** (4.8072 g, 37.5 mmol)/dioxane (40.0 mL) sequentially under nitrogen atmosphere. Then the flask was placed in an oil bath preheated at 70 °C with stirring vigorously. 0.6 mL each of reaction mixture was taken after 0.17 h, 0.83 h, 1.67 h, 2.67 h, 8 h, respectively, with a syringe (1.0 mL), which was filtrated through a pipette fitted with a cotton ball eluted with EtOAc (20 mL). After evaporation, the five samples were tested with 18.4 uL of mesitylene as the internal standard by ^{1}H NMR analysis with the results being summarized in Table S1.

Table S1: The kinetic experiment of **1j**, **2a** and *S*-**3** at 70 °C

Entry	Time (h)	Yield of (R_a)-**4ja**	Yield of (*S*,*S*)-**5ja**
1	0.17	8.3	67.6
2	0.83	19.5	49.2
3	1.67	24.4	46.5
4	2.67	28	42.6
5	8	33.7	32.1

(2) The kinetic experiment of **1j**, **2a** and tetrahydropyrrole at 70 °C



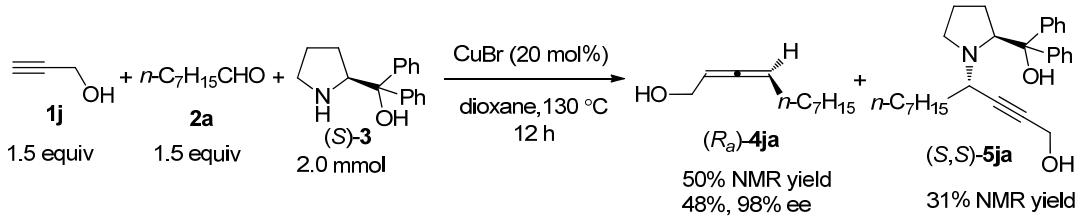
To a flame dried three-necked flask were added $CuBr_2$ (1.1218 g, 5.0 mmol), dioxane (30.0 mL), tetrahydropyrrole (2.06 mL, d = 0.86 g/mL, 25 mmol)/dioxane (15.0 mL), **1j** (2.1065 g, 37.5 mmol)/dioxane (15.0 mL) and **2a** (4.8072 g, 37.5 mmol)/dioxane (15.0 mL) sequentially under nitrogen atmosphere. Then the flask was placed in an oil bath preheated at 70 °C with stirring rapidly. 0.6 mL each of reaction mixture was taken after 0.17 h, 0.83 h, 1.67 h, 2.67 h, 8 h, respectively, with a syringe (1.0 mL), which was filtrated through a pipette fitted with a cotton ball eluted with

EtOAc (20 mL). After evaporation, the five samples were tested with 18.4 uL of mesitylene as the internal standard by ^{1}H NMR analysis with the results being summarized in Table S2.

Table S2: The kinetic experiment of **1j**, **2a** and tetrahydropyrrole at 70 ^{o}C

Entry	Time (h)	Yield of *rac*-**4ja**	Yield of **5ja'**
1	0.17	0	0
2	0.83	0	0
3	1.67	0	0
4	2.67	0	0
5	8	0	0

Control experiment with CuBr under the standard conditions. hx-13-4



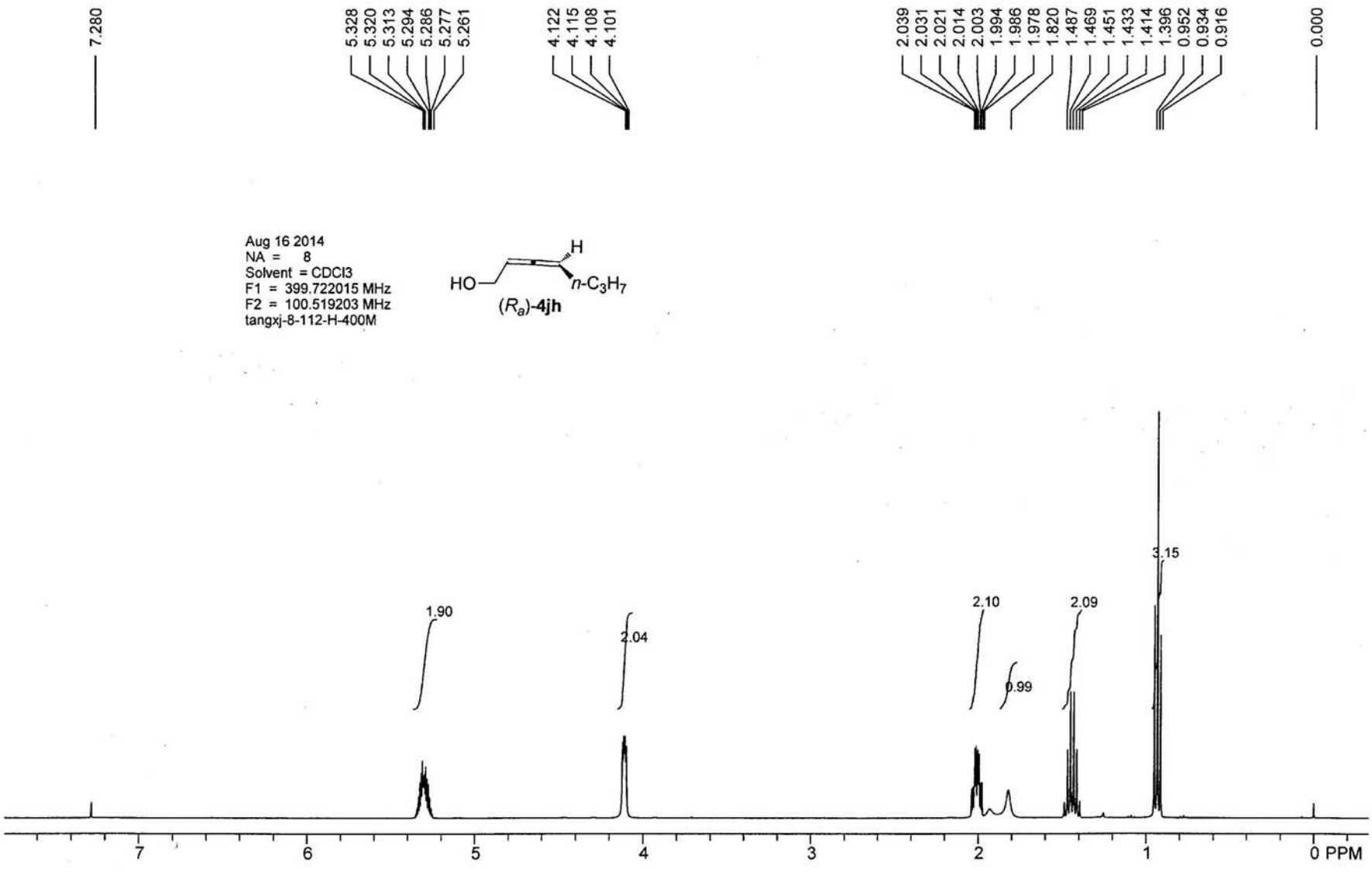
To a flame-dried Schlenk tube equipped with a polytetrafluoroethylene plug were added CuBr (57.7 mg, 0.4 mmol), (*S*)-**3** (506.0 mg, 2.0 mmol), **1j** (168.6 mg, 3.0 mmol)/dioxane (3.0 mL), and **2a** (384.2 mg, 3.0 mmol)/dioxane (3.0 mL) sequentially under nitrogen atmosphere. The Schlenk tube was then sealed by screwing the polytetrafluoroethylene plug tightly with the outlet connected to the vacuum line and the nitrogen flow being closed (For an apparatus, see page S2). The Schlenk tube was then stirred in an oil bath preheated at 130 ^{o}C for 12 h. After

cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with EtOAc (30 mL). After evaporation, the NMR yields for (R_a)-**4ja** (50%) and (*S*,*S*)-**5ja** (31%) were determined by ^{1}H NMR of the crude product with mesitylene (92 μL) as the internal standard. Chromatography on silica gel (eluent: petroleum ether/ethyl acetate (10/1 to 5/1)) afforded impure (R_a)-**4ja,** which was then dissolved in ether (30 mL), and washed with an aqueous solution of hydrochloric acid (*v*/*v* = 10%, 20 mL). The organic layer was separated and the aqueous layer was extracted with ether (20 mL). The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. After filtration and evaporation, the residue was purified by chromatography (eluent: petroleum ether/ethyl acetate = 8/1) on silica gel to afford (R_a)-**4ja** (159.9 mg, 48%): 98% ee (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 28.8 min, t_R(minor) = 32.0 min); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.40-5.24 (m, 2 H, CH=C=CH), 4.12 (dd, J_1 = 5.3 Hz, J_2 = 3.2 Hz, 2 H, OCH_2), 2.08-1.96 (m, 2 H, CH_2), 1.58 (s, 1 H, OH), 1.48-1.20 (m, 10 H, CH_2 × 5), 0.88 (t, *J* = 6.8 Hz, 3 H, Me).

References

1. J. Ye, W. Fan, S. Ma, *Chem. Eur. J.*, 2013, **19**, 716.

2. J. Ye, S. Li, B. Chen, W. Fan, J. Kuang, J. Liu, Y. Liu, B. Miao, B. Wan, Y. Wang, X. Xie, Q. Yu, W. Yuan, S. Ma, *Org. Lett.*, 2012, **14**, 1346.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Aug 16 2014
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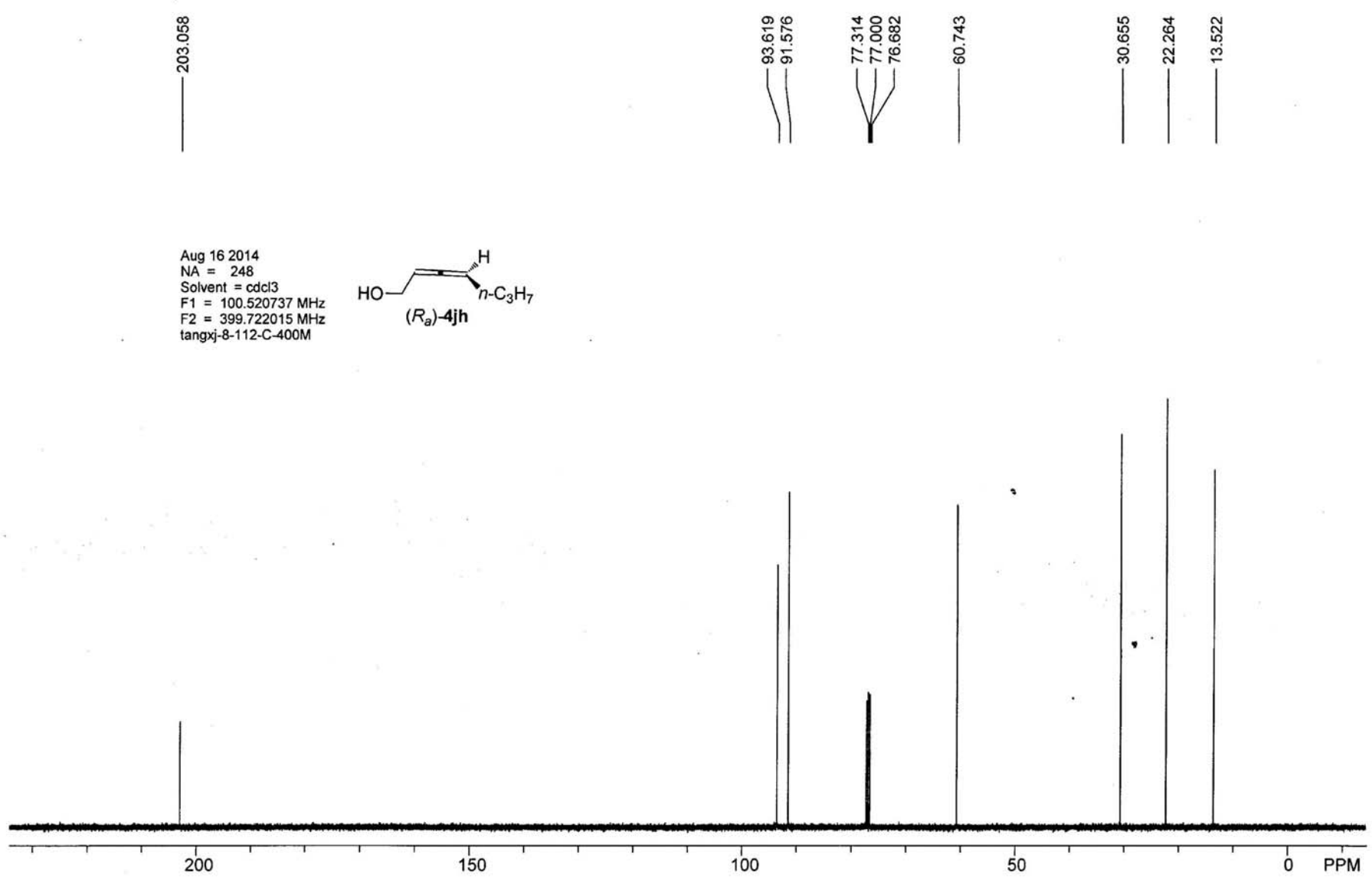
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txj-8-112

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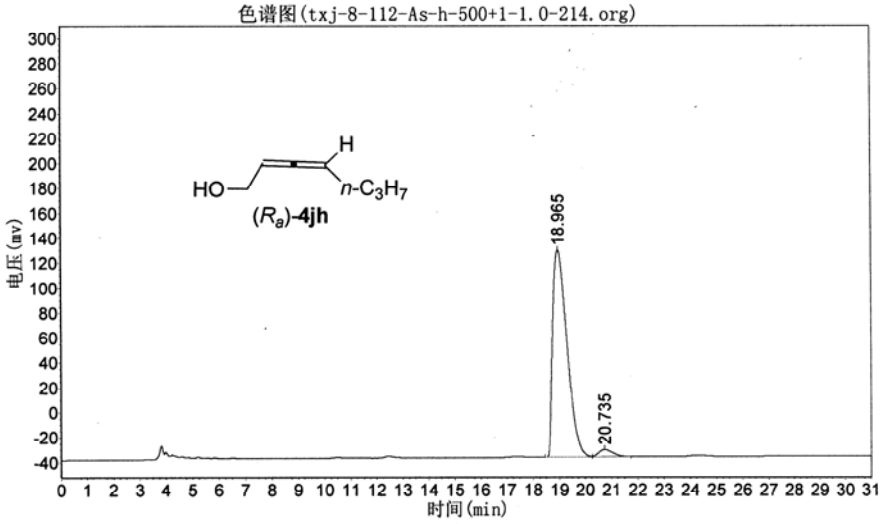

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txj-8-111

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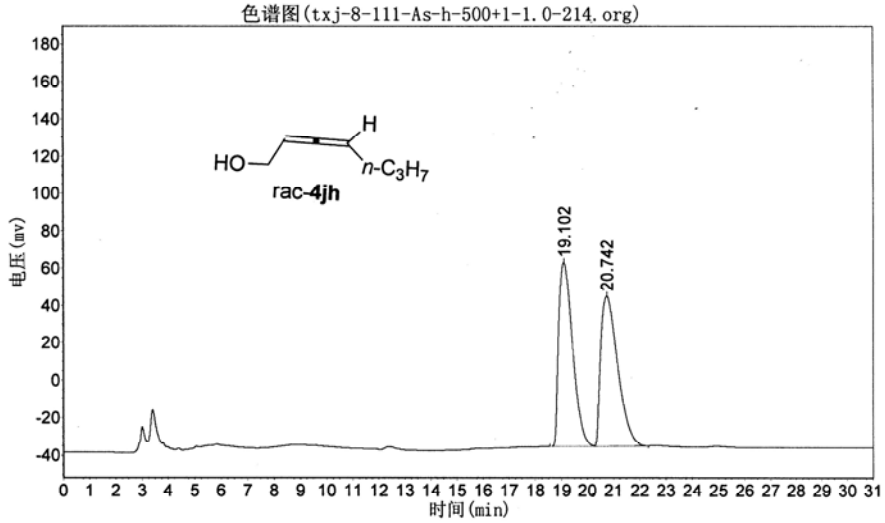
214nm　1.0ml/min

色谱图(txj-8-111-As-h-500+1-1.0-214.org)



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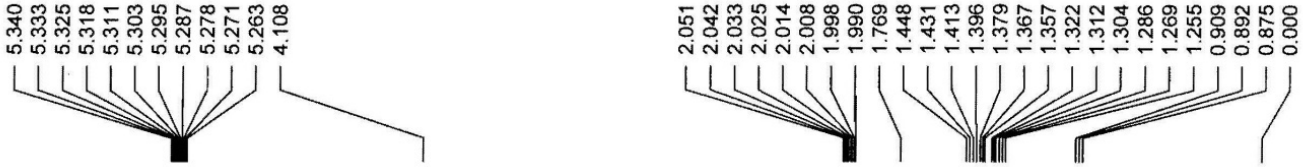

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Oct 28 2014
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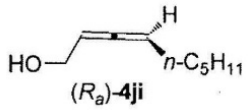
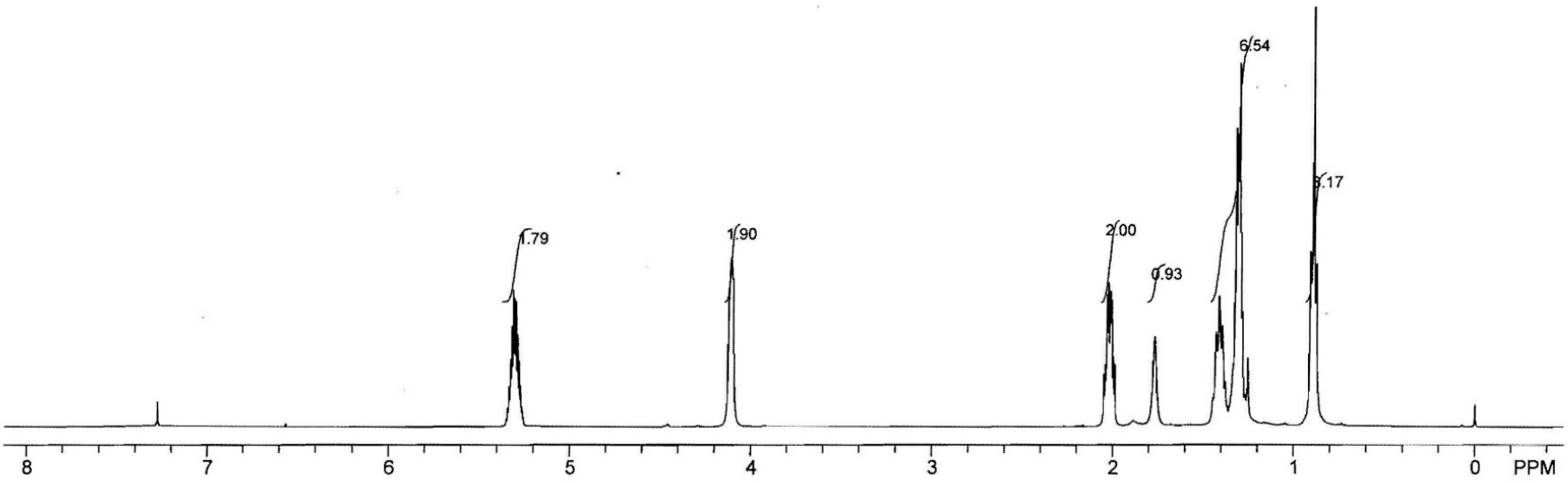
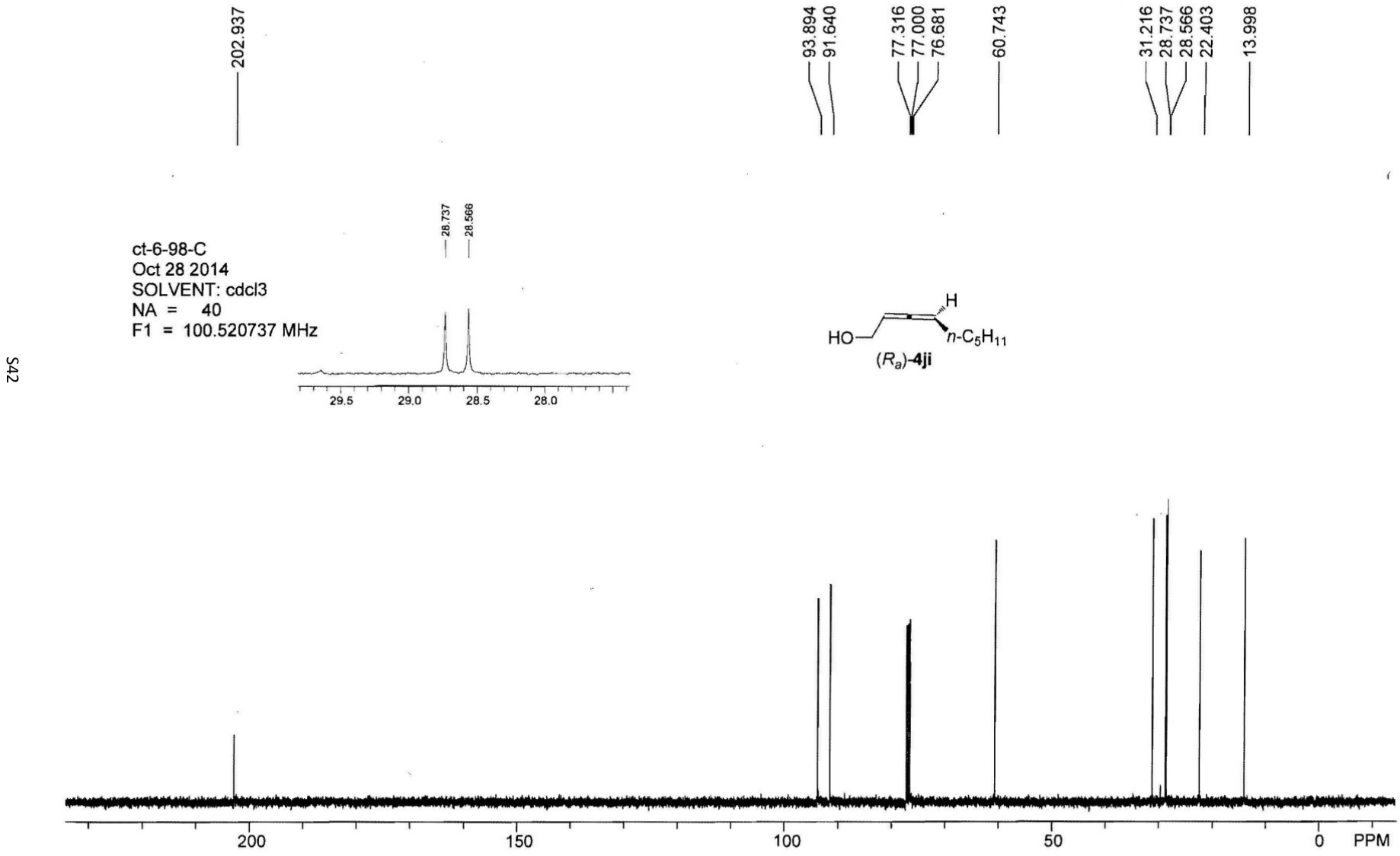


(R_a)-**4ji**

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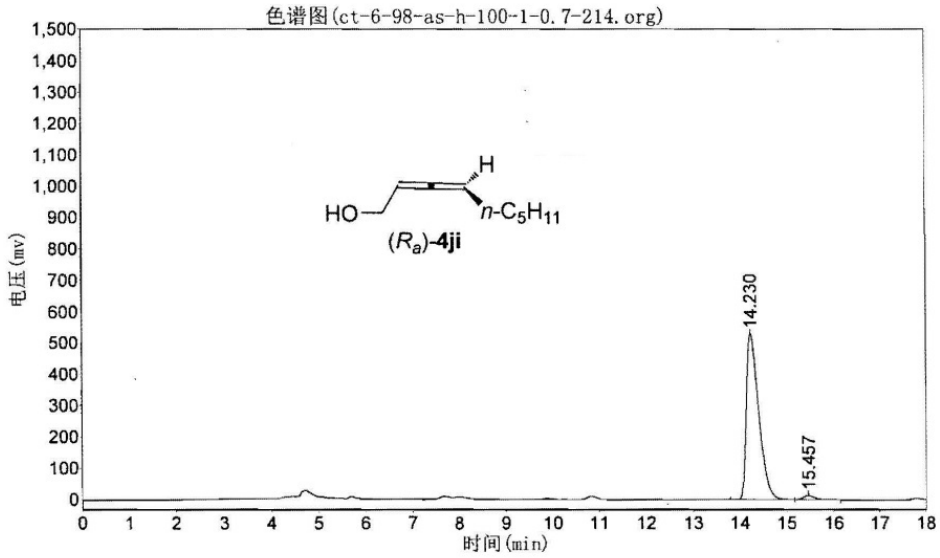

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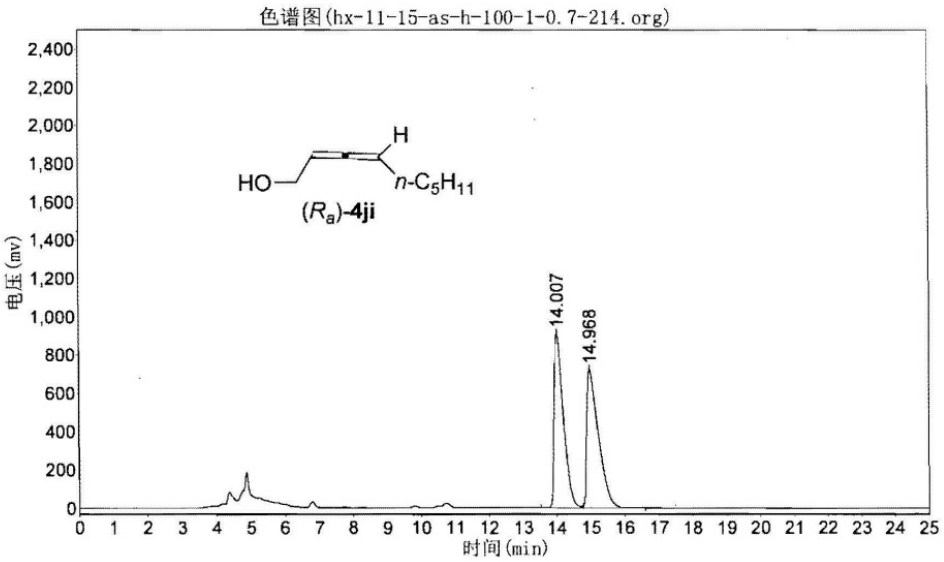

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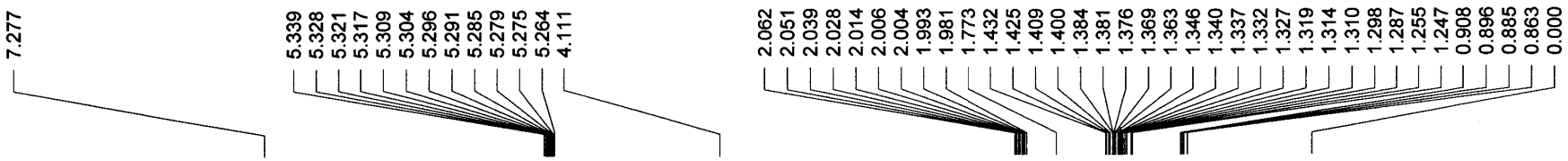

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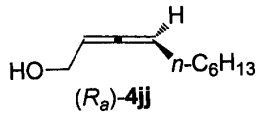

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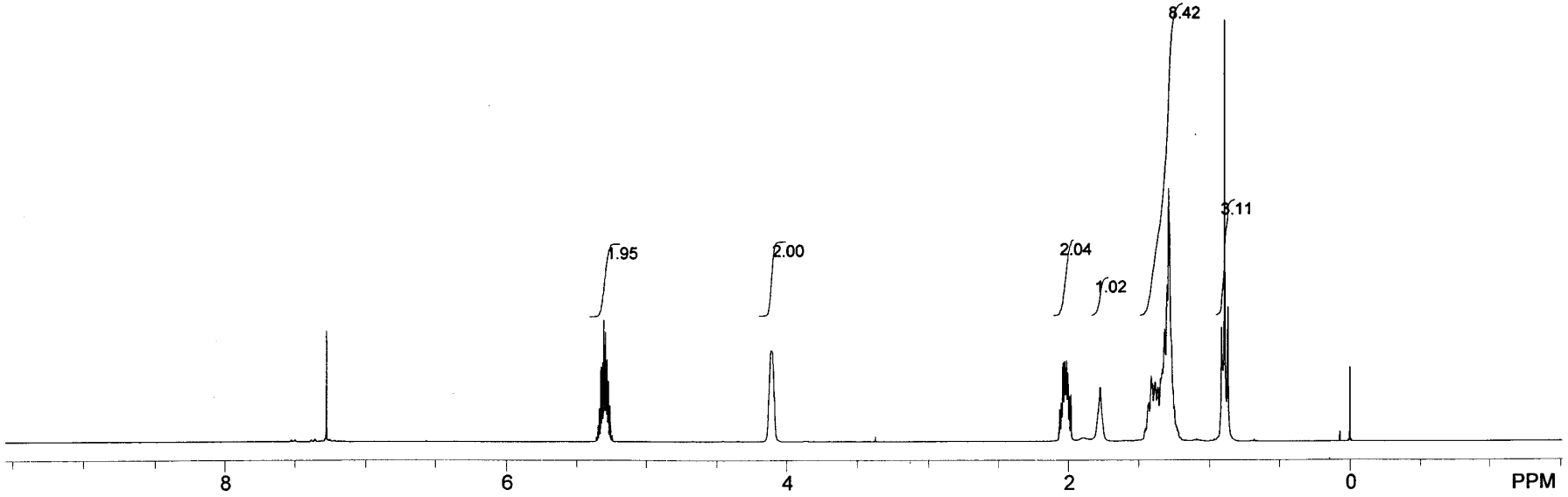

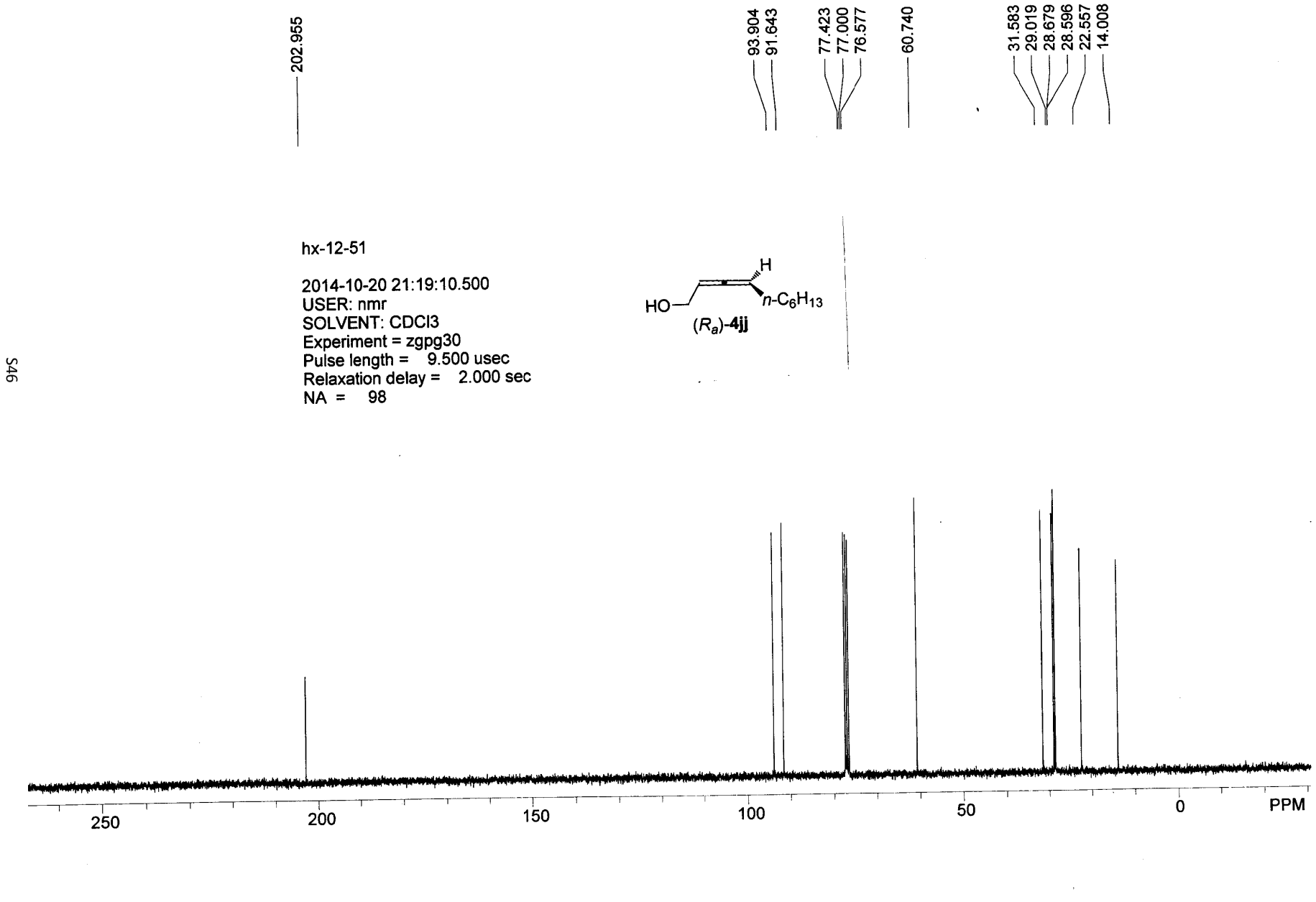
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hx-12-51

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实验者：hx
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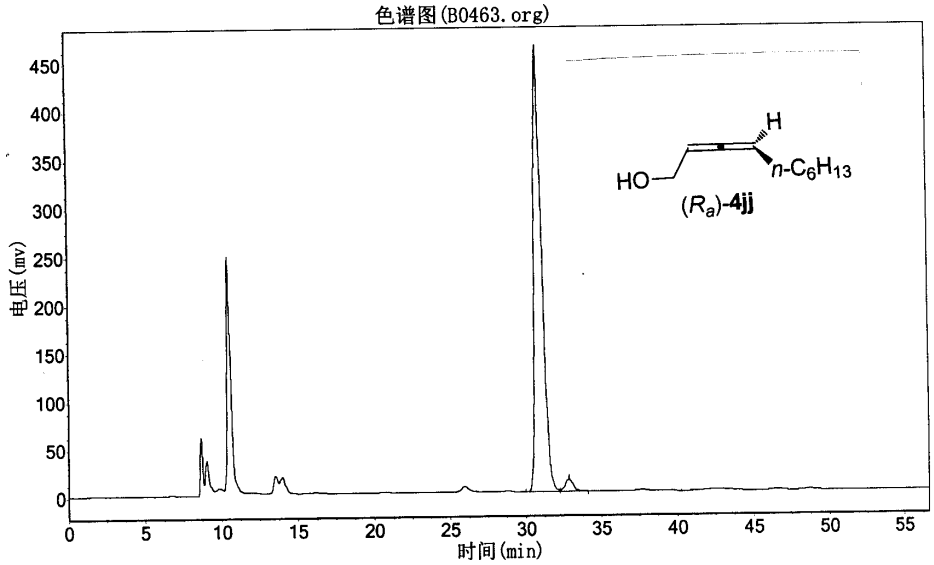

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2014-10-20

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hx-12-28

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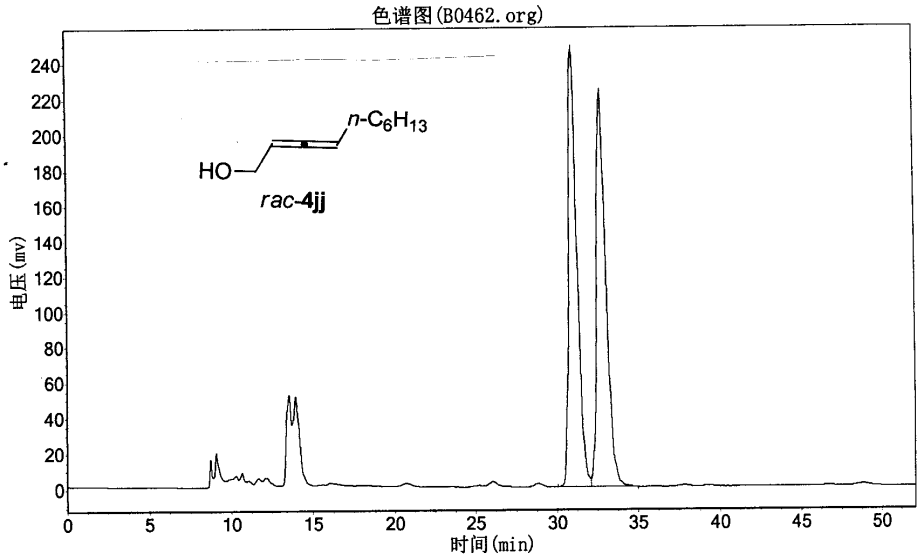

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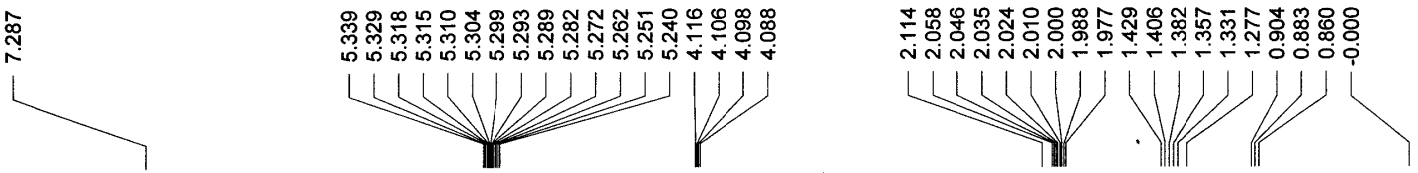
2014-10-20

浙江大学智能信息研究所



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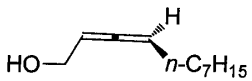
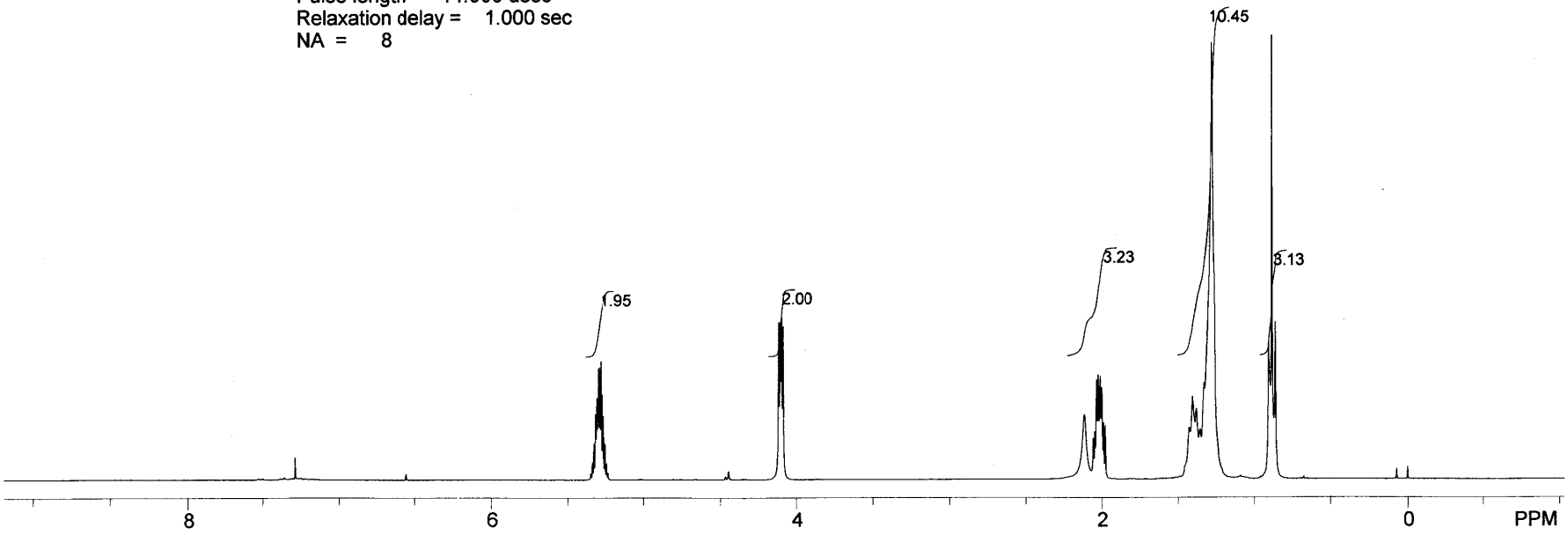
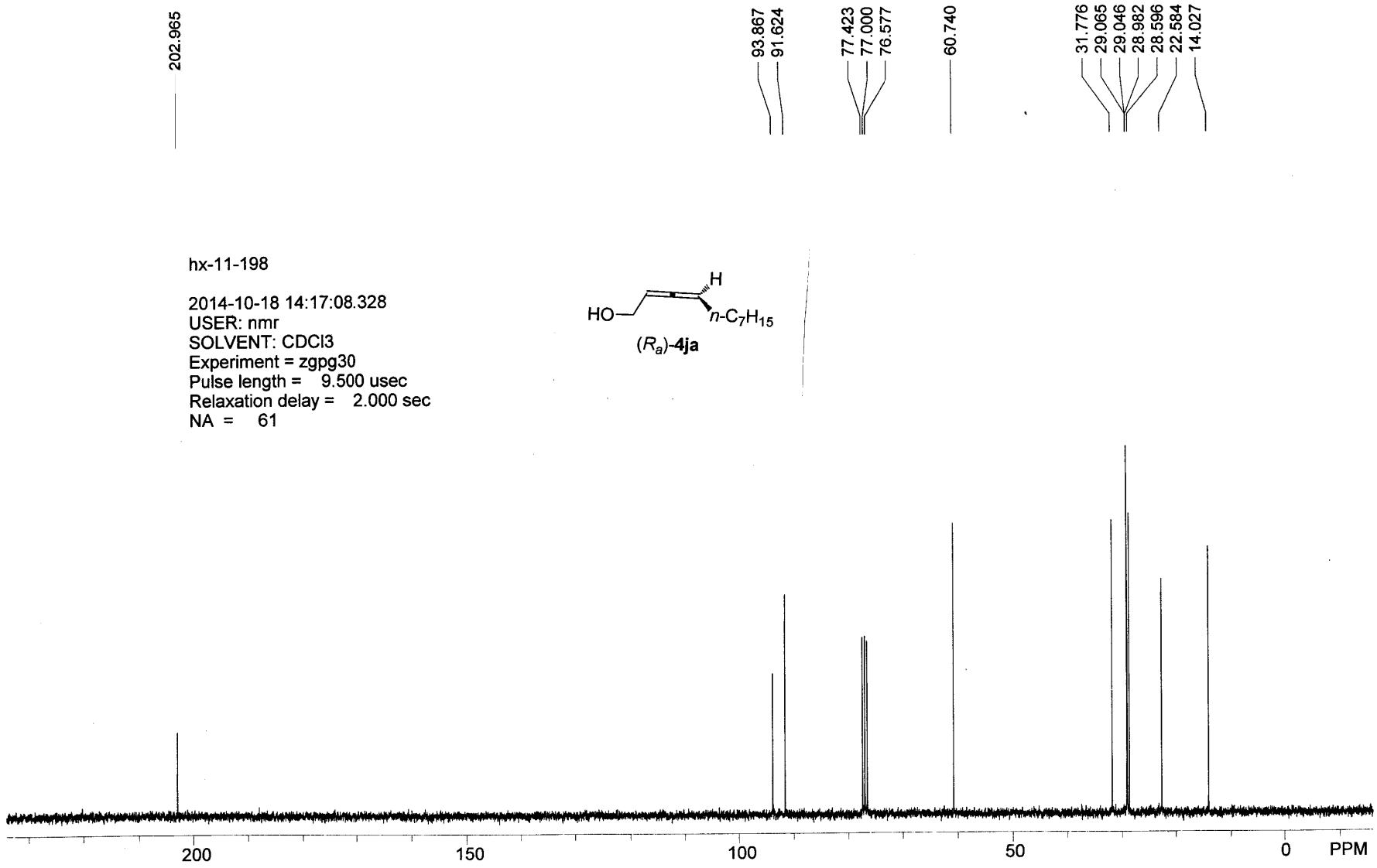


(R_a)-**4ja**

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hx-11-198

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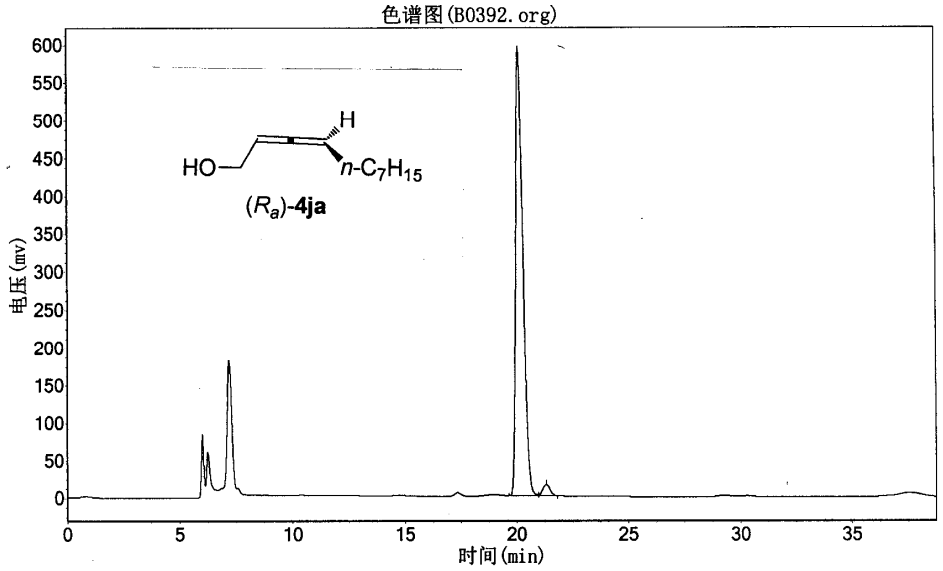

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2014-10-01 浙江大学智能信息研究所

hx-11-185

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实验者：hx
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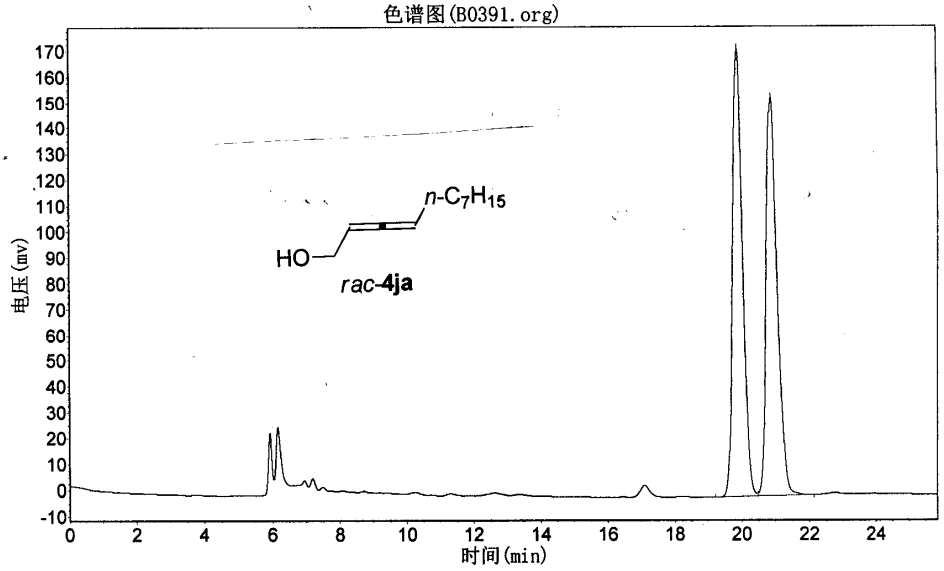

分析结果表

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2		20.887	154004.016	3509724.500	49.6832
总计			327141.391	7064205.500	100.0000

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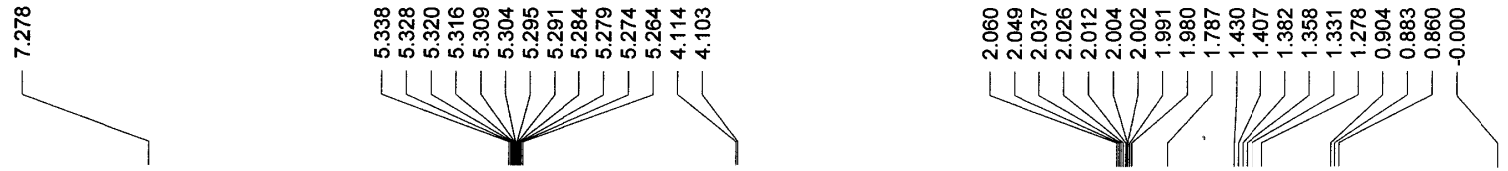
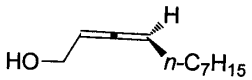
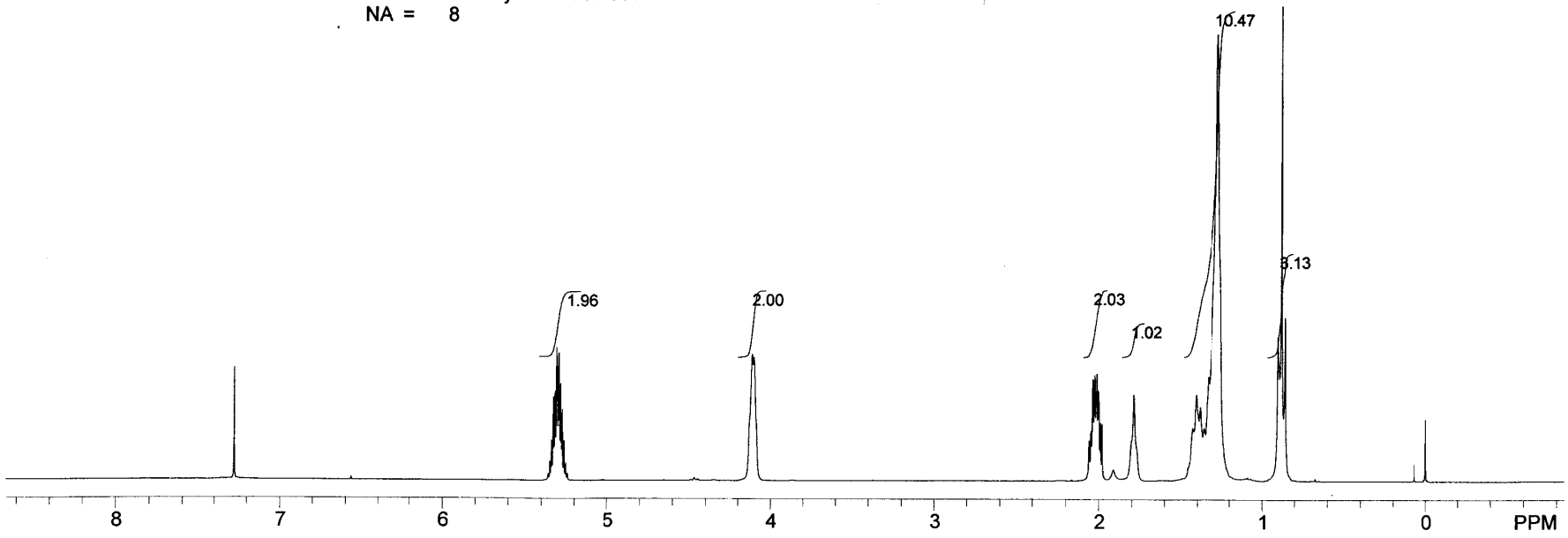


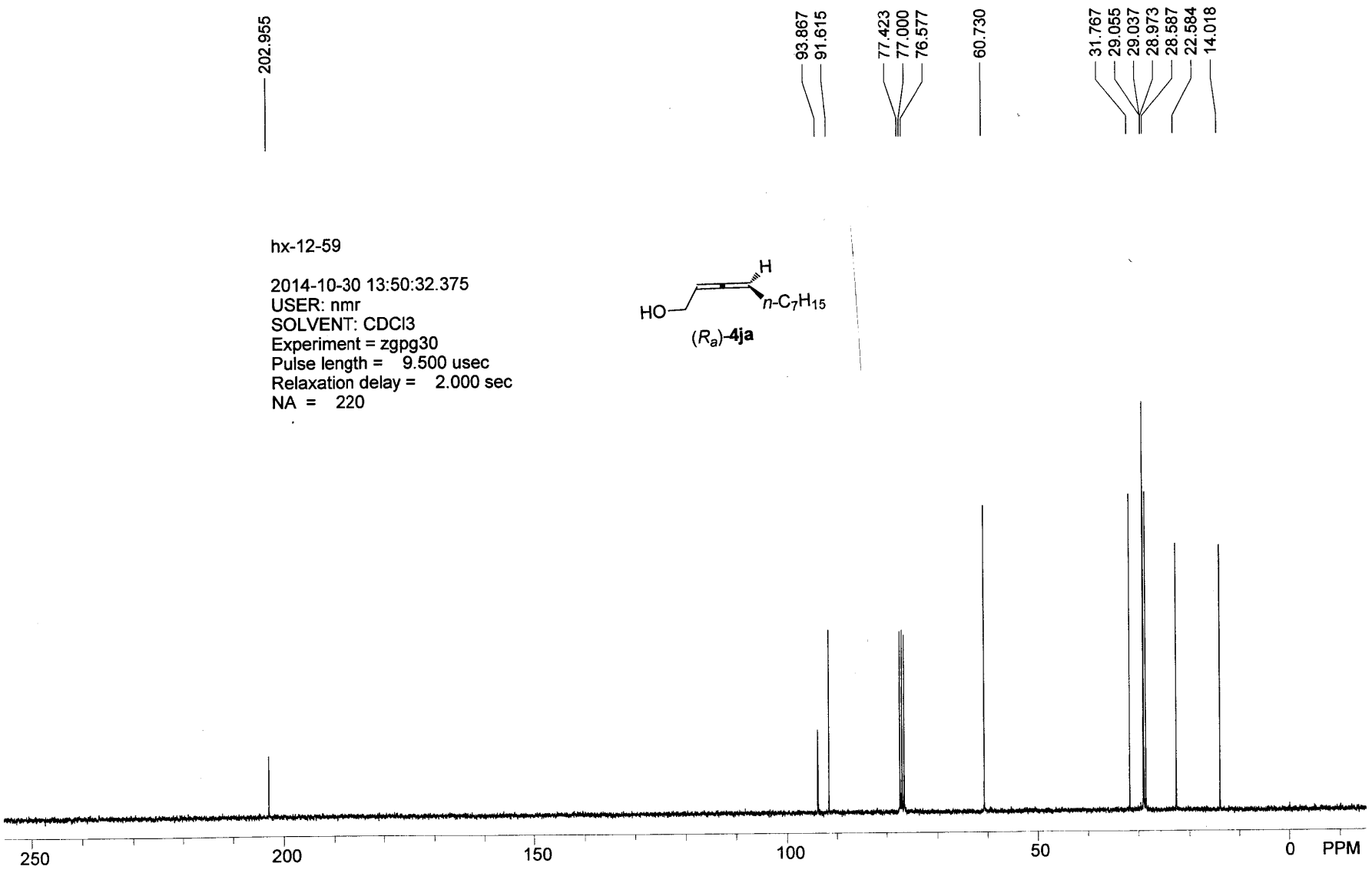
hx-12-59

2014-10-23 20:37:08.593
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ja**




202.955
93.867
91.615
77.423
77.000
76.577
60.730
31.767
29.055
29.037
28.973
28.587
22.584
14.018
hx-12-59
2014-10-30 13:50:32.375
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 220
HO
H
n-C7H15
(Ra)-4ja
250
200
150
100
50
0
PPM

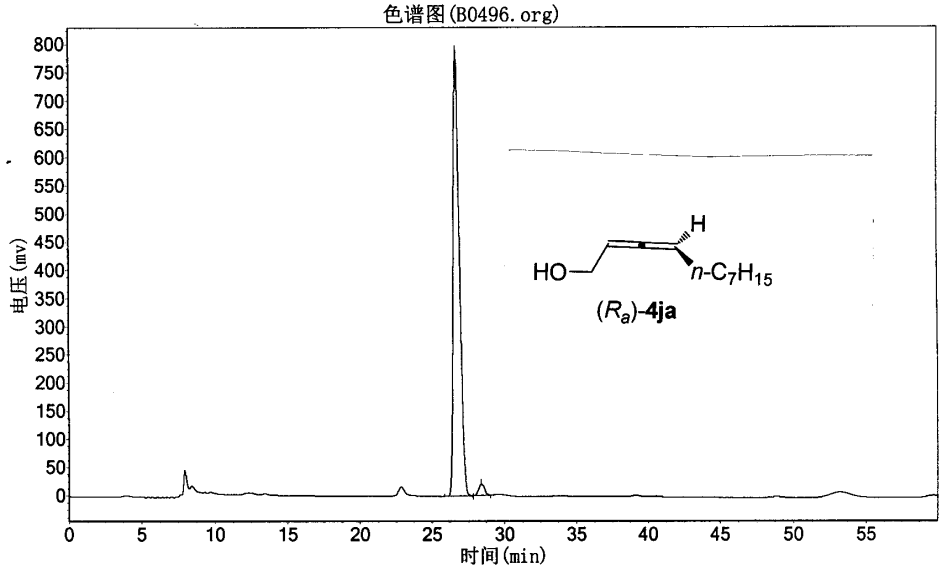
hx-12-59

实验单位：zju
实验时间：2014-10-24,9:48:35
谱图文件:D:\浙大智达\N2000\样品\B0496.org

实验者：hx
报告时间：2014-10-24,10:53:51
积分方法:面积归一法

实验内容简介：
AS-H, hexane/i-PrOH = 200/1, 0.6 ml/min, 214 nm

色谱图(B0496.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		26.668	790162.625	23602220.000	97.6886
2		28.415	19864.154	558444.500	2.3114
总计			810026.779	24160664.500	100.0000

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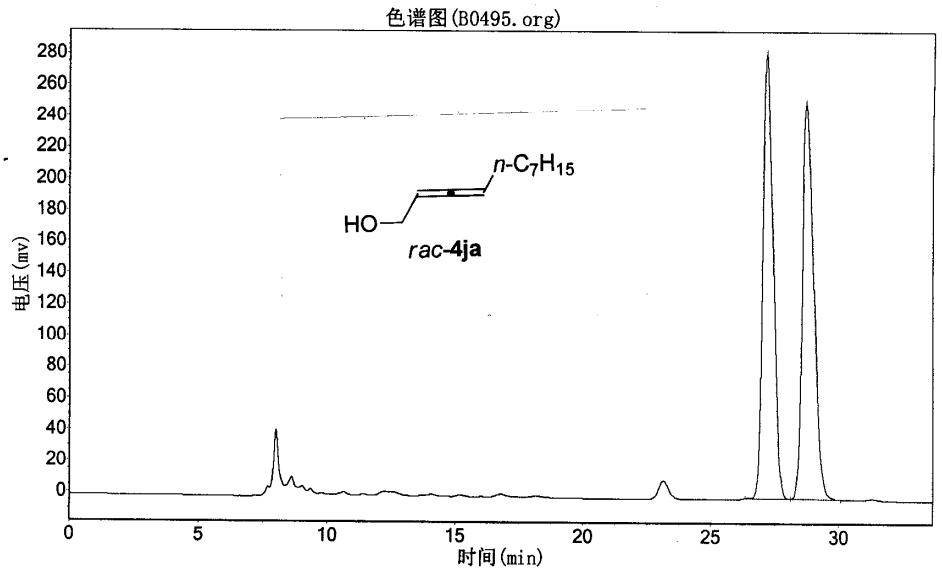
hx-11-185

实验单位：zju
实验时间：2014-10-24,9:11:01
谱图文件:D:\浙大智达\N2000\样品\B0495.org

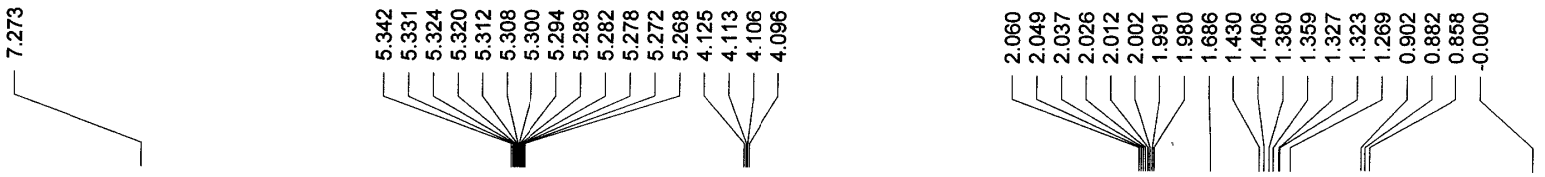
实验者：hx
报告时间：2014-10-24,9:45:43
积分方法:面积归一法

实验内容简介：
AS-H, hexane/i-PrOH = 200/1, 0.6 ml/min, 214 nm



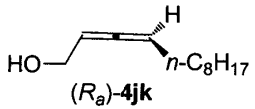
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.200	283049.375	7860046.000	50.1559
2		28.750	251405.078	7811197.000	49.8441
总计			534454.453	15671243.000	100.0000

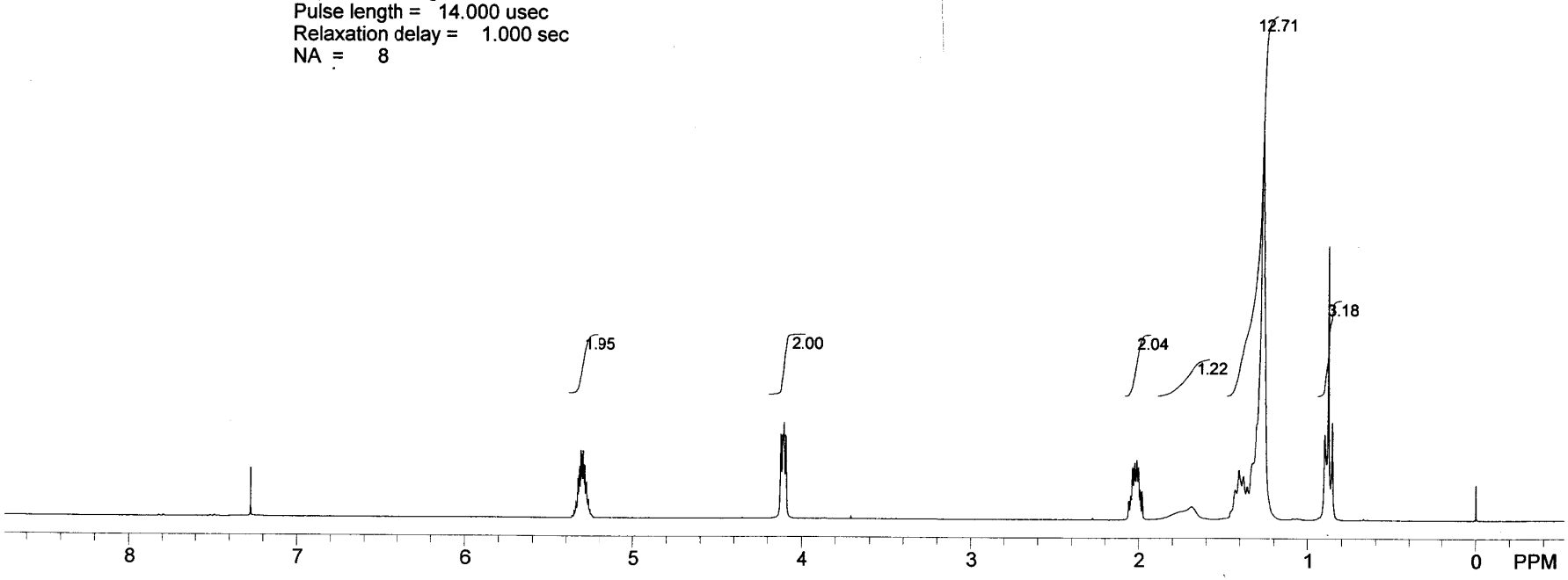


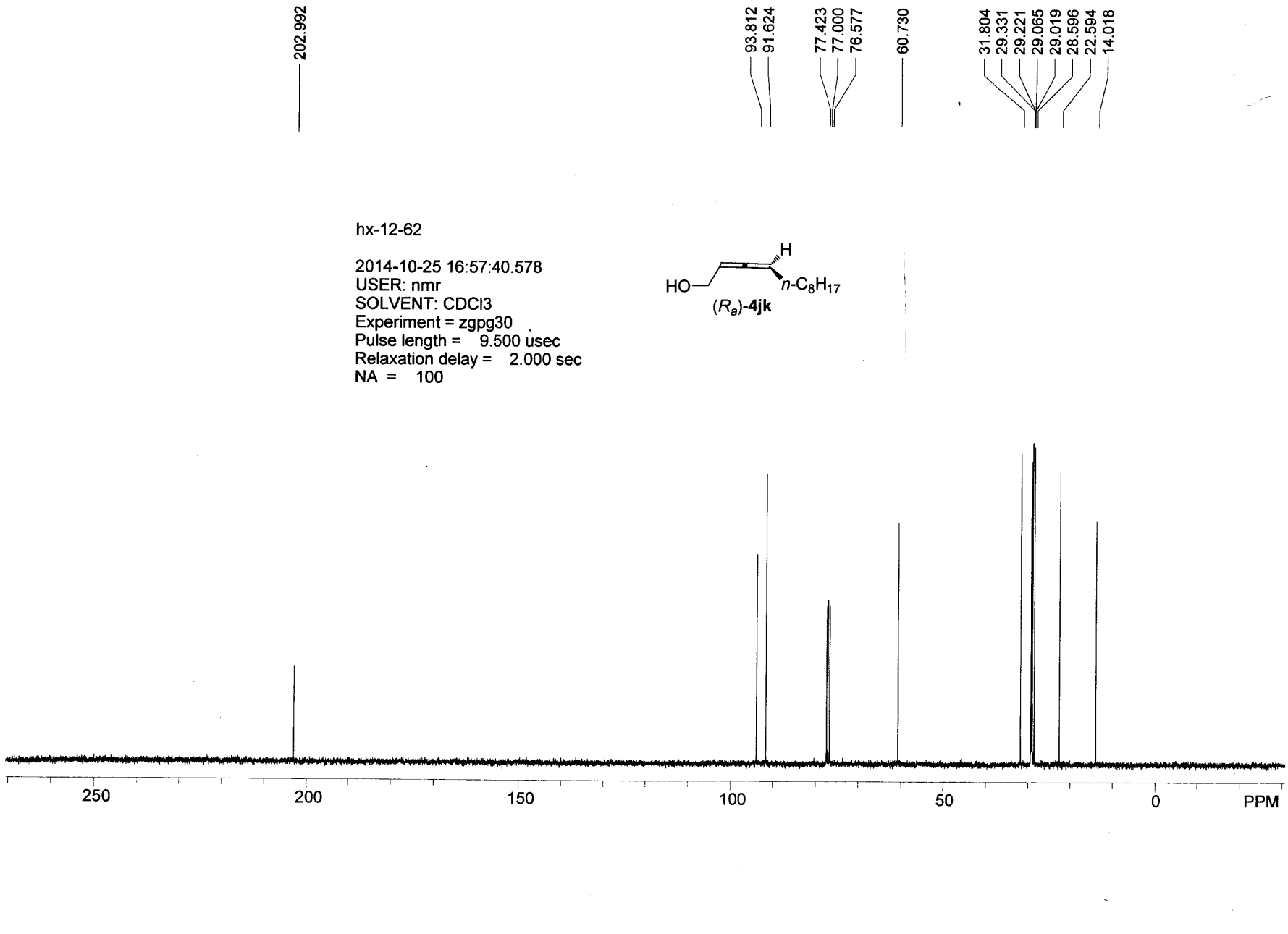
hx-12-62

2014-10-28 21:31:44.000
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4jk**



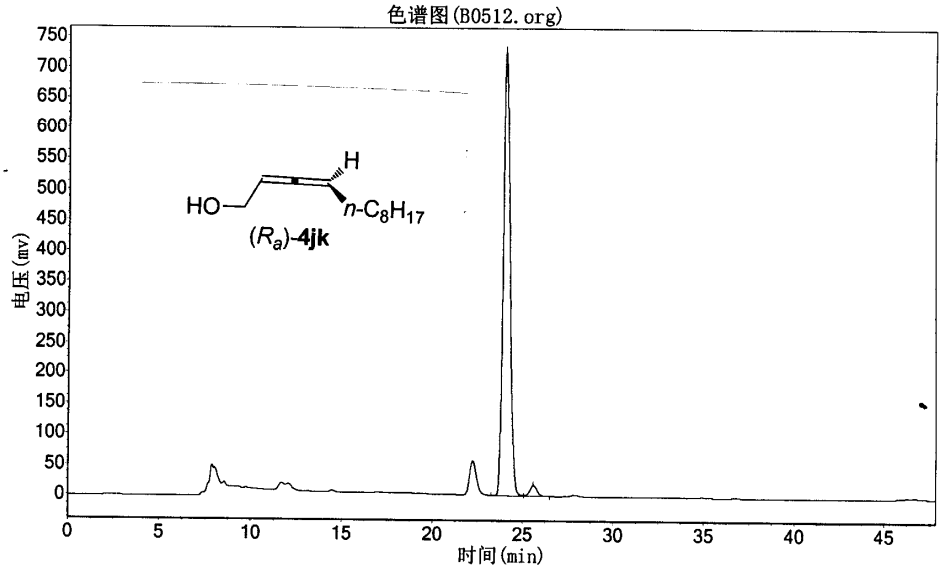

202.992
93.812
91.624
77.423
77.000
76.577
60.730
31.804
29.331
29.221
29.065
29.019
28.596
22.594
14.018
hx-12-62
2014-10-25 16:57:40.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 100
HO
H
n-C8H17
(Ra)-4jk
250
200
150
100
50
0
PPM

hx-12-62

实验单位：zju
实验时间：2014-10-25,21:31:23
谱图文件:D:\浙大智达\N2000\样品\B0512.org

实验者：hx
报告时间：2014-10-25,22:20:30
积分方法:面积归一法

实验内容简介：
AS-H, n-hexane/iPrOH = 200/1, 214 nm, 0.6 ml/min



分析结果表

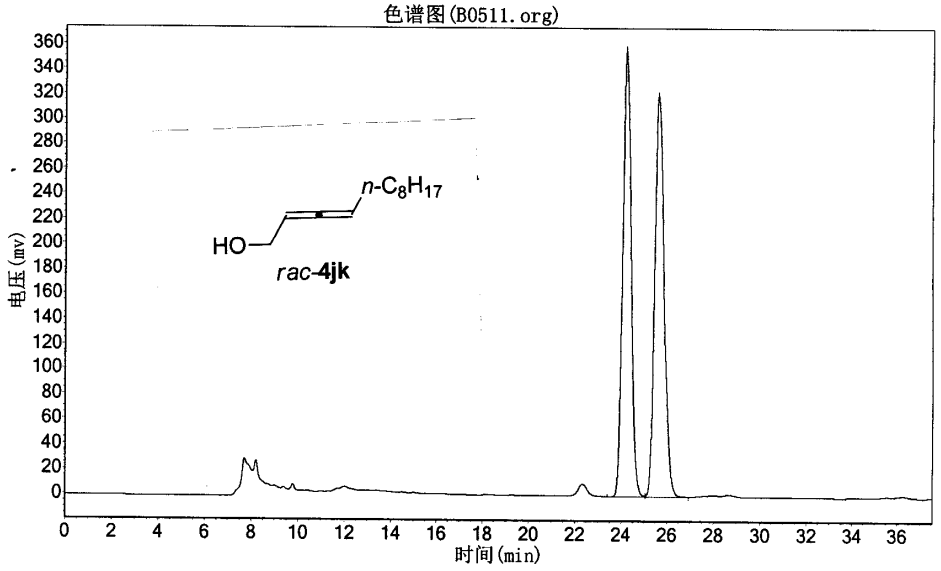
峰号	峰名	保留时间	峰高	峰面积	含量
1		24.018	727514.063	19066170.000	97.5350
2		25.600	17350.953	481857.625	2.4650
总计			744865.016	19548027.625	100.0000

hx-12-48

实验单位：zju
实验时间：2014-10-25,20:49:50
谱图文件:D:\浙大智达\N2000\样品\B0511.org

实验者：hx
报告时间：2014-10-25,21:29:37
积分方法:面积归一法

实验内容简介：
AS-H, n-hexane/iPrOH = 200/1, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		24.208	356075.813	8969040.000	49.7334
2		25.595	319364.969	9065183.000	50.2666
总计			675440.781	18034223.000	100.0000

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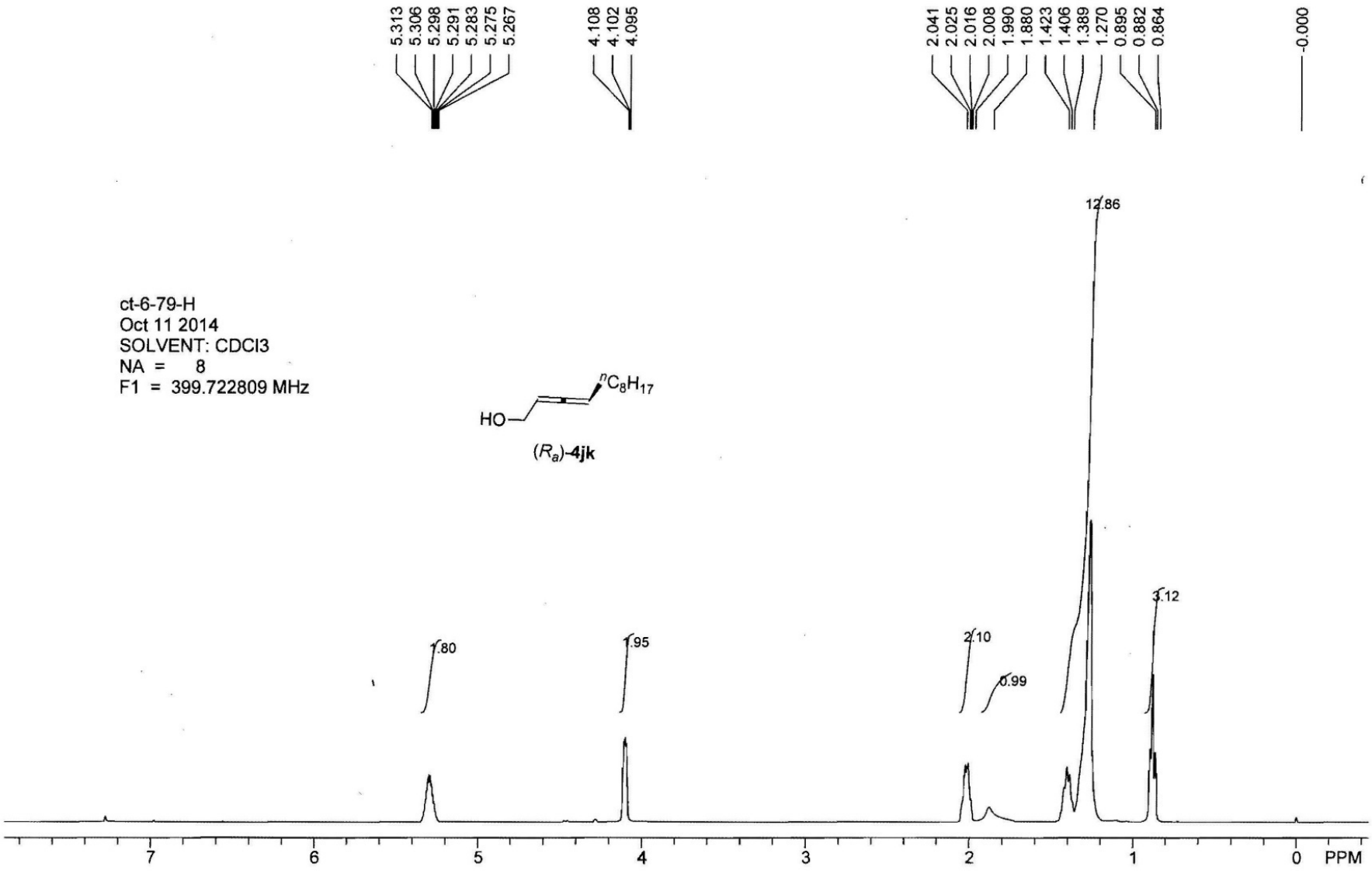
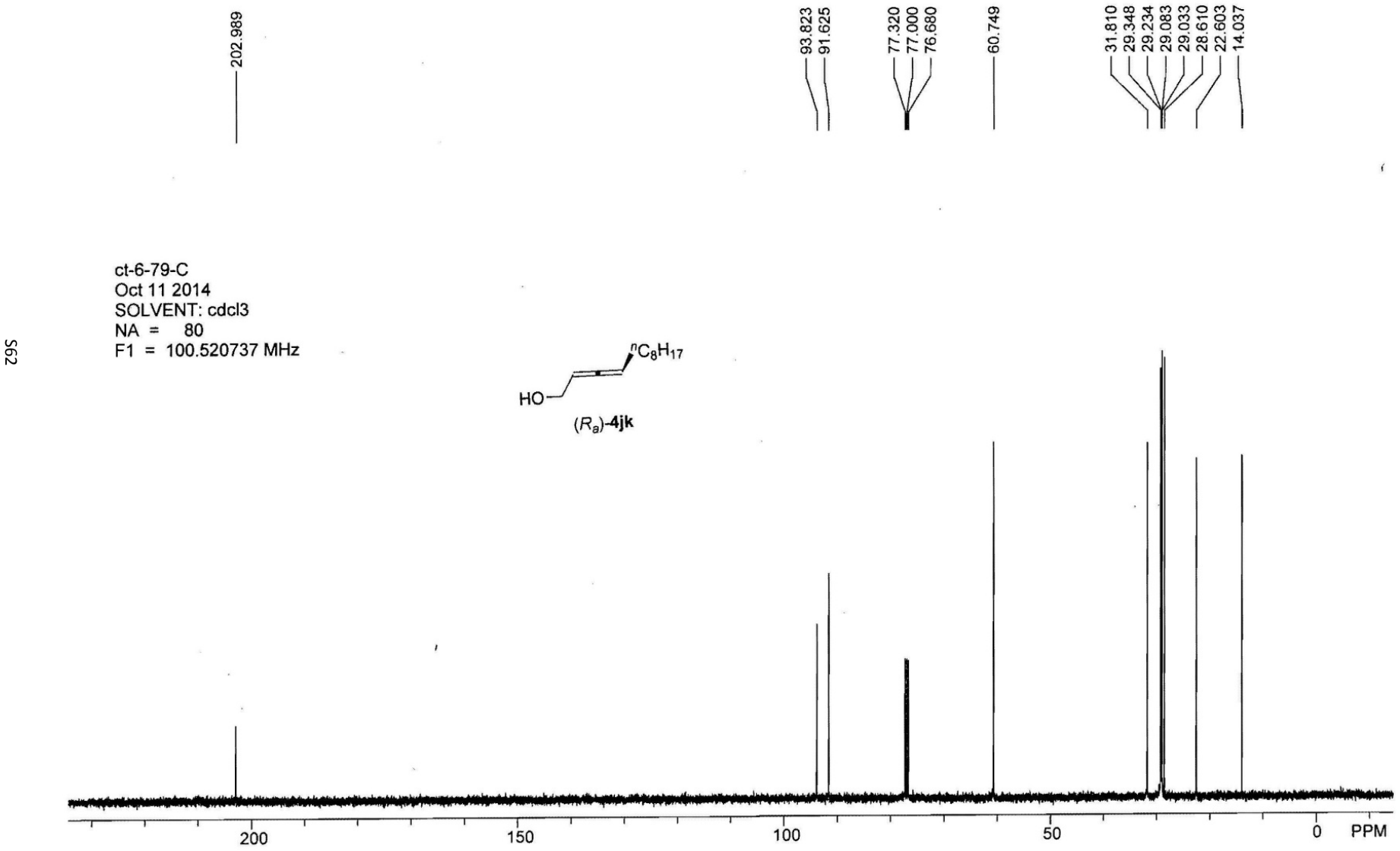
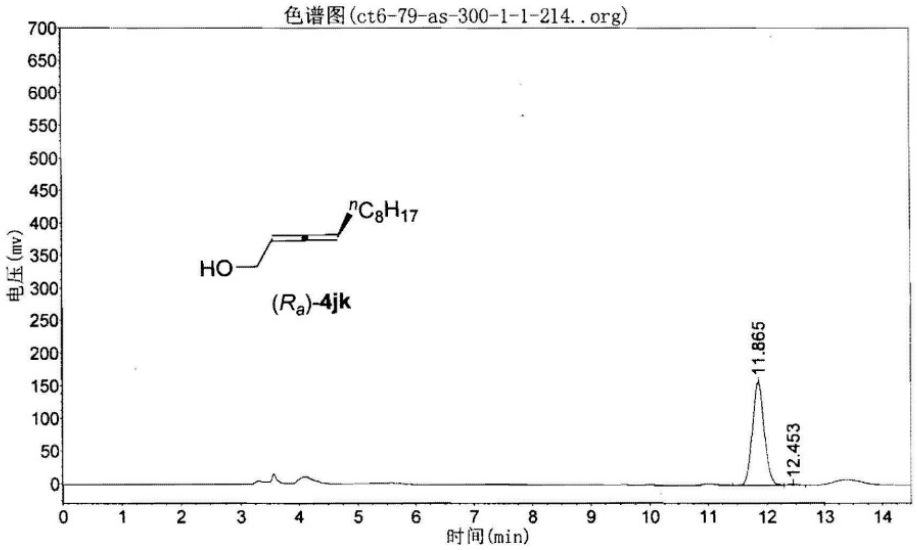

5.313
5.306
5.298
5.291
5.283
5.275
5.267
4.108
4.102
4.095
2.041
2.025
2.016
2.008
1.990
1.880
1.423
1.406
1.389
1.270
0.895
0.882
0.864
-0.000
ct-6-79-H
Oct 11 2014
SOLVENT: CDCl3
NA = 8
F1 = 399.722809 MHz
HO
^{n}C$_8$H$_{17}$
(R$_a$)-4jk
12.86
3.12
1.80
1.95
2.10
0.99
7
6
5
4
3
2
1
0
PPM


202.989
93.823
91.625
77.320
77.000
76.680
60.749
31.810
29.348
29.234
29.083
29.033
28.610
22.603
14.037
ct-6-79-C
Oct 11 2014
SOLVENT: cdcl3
NA = 80
F1 = 100.520737 MHz
HO
$^{n}C_8H_{17}$
(R_a)-4jk
200
150
100
50
0
PPM

ct-6-79-as-300-1-1-214

实验时间：2014-10-10,11:46:59　　报告时间：2014-10-10,12:38:32
谱图文件:D:\zhuguangjiong\jxg\20141009\ct6-79-as-300-1-1-214..org

实验内容简介：



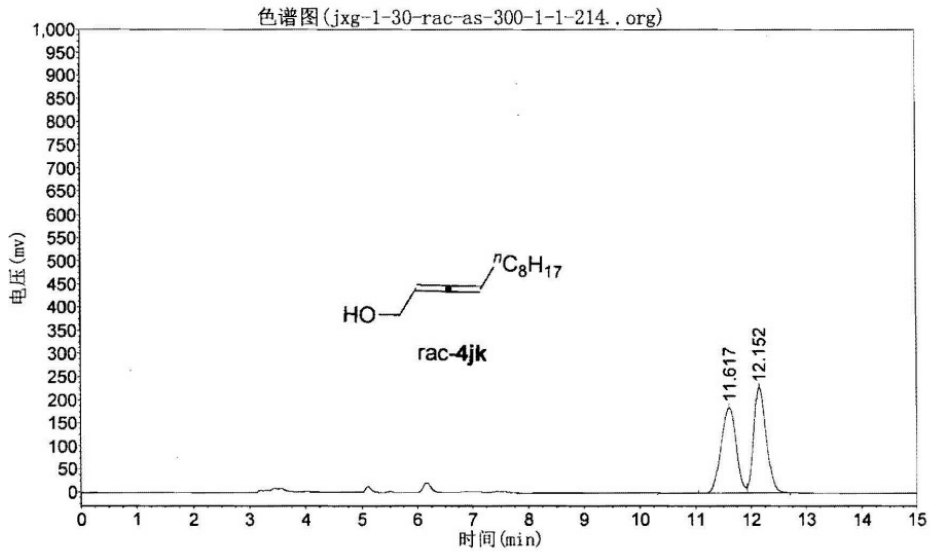
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.865	157957.859	2228711.750	99.1462
2		12.453	1556.399	19192.471	0.8538
总计			159514.258	2247904.221	100.0000

jxg-1-30-rac-as-300-1-1-214

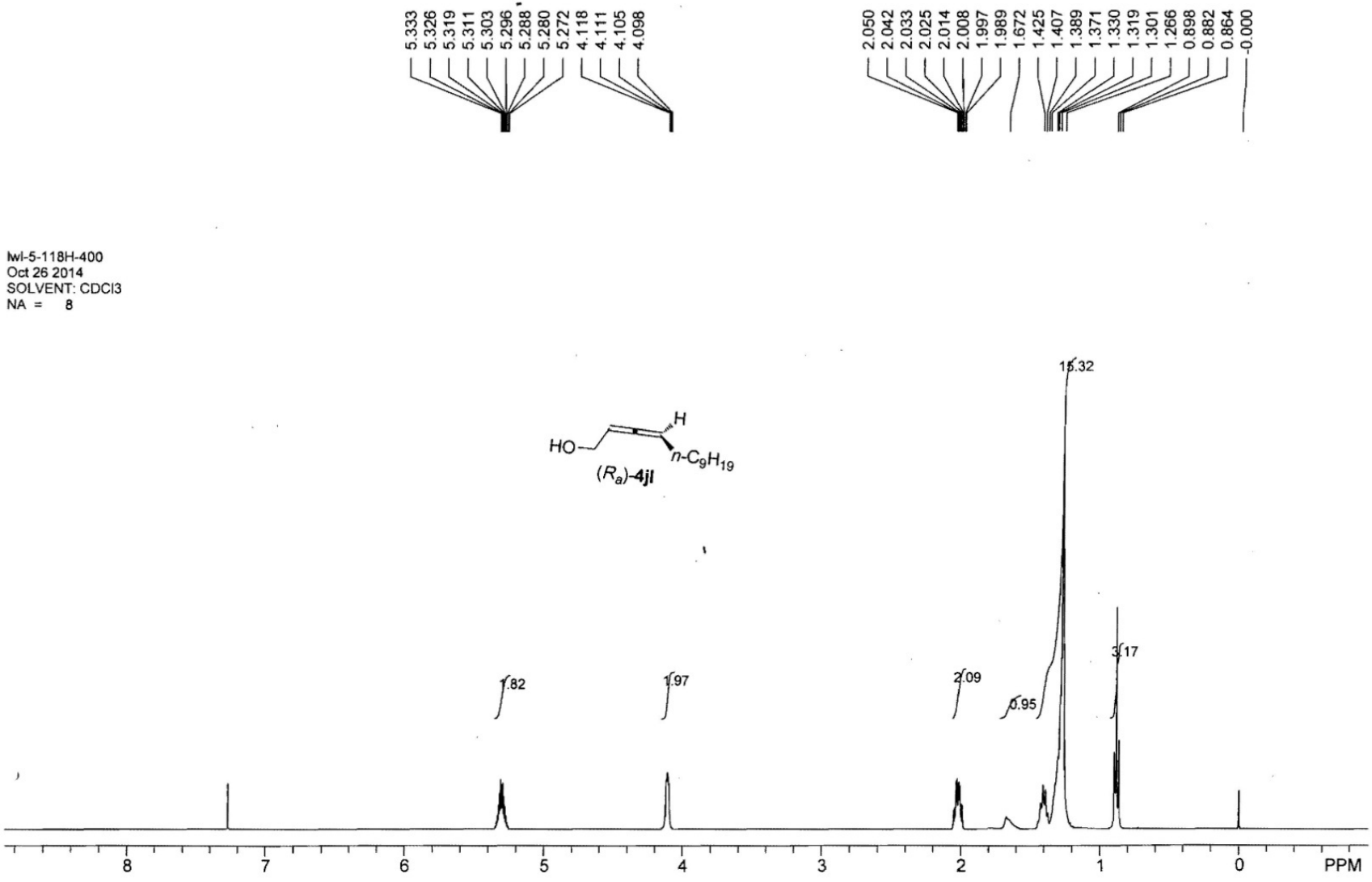
实验时间：2014-10-10,12:03:29　　报告时间：2014-10-10,12:36:57
谱图文件:D:\zhuguangjiong\jxg\20141009\jxg-1-30-rac-as-300-1-1-214..org

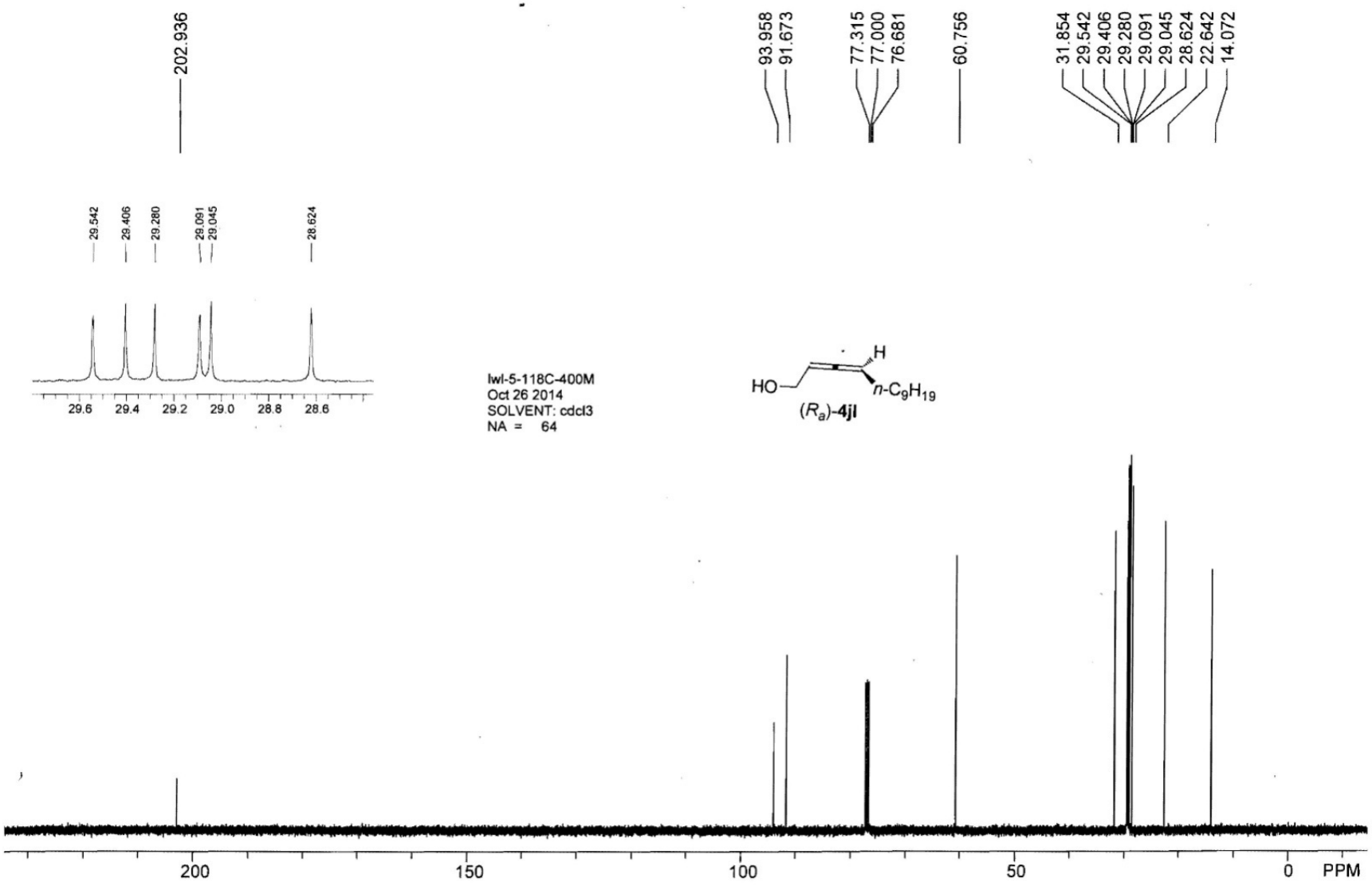
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.617	185454.031	3513074.000	49.5782
2		12.152	229043.313	3572853.000	50.4218
总计			414497.344	7085927.000	100.0000

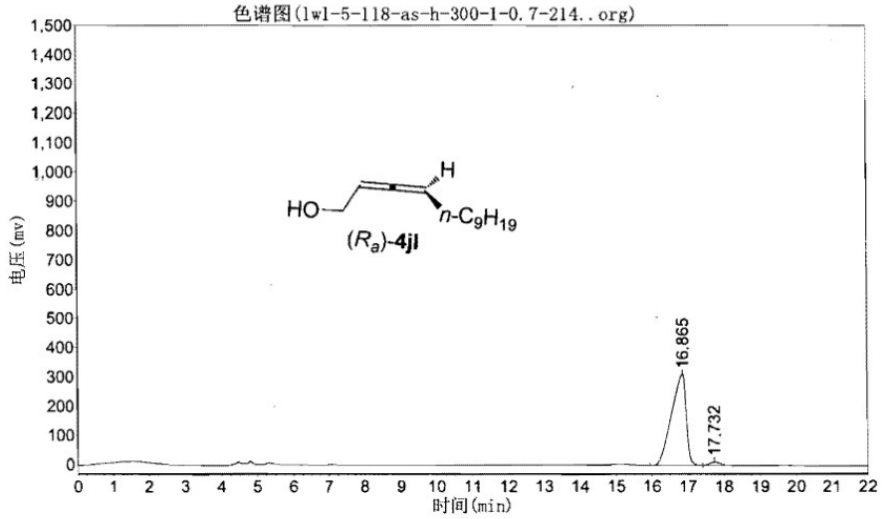

5.333
5.326
5.319
5.311
5.303
5.296
5.288
5.280
5.272
4.118
4.111
4.105
4.098
2.050
2.042
2.033
2.025
2.014
2.008
1.997
1.989
1.672
1.425
1.407
1.389
1.371
1.330
1.319
1.301
1.266
0.898
0.882
0.864
-0.000
lwl-5-118H-400
Oct 26 2014
SOLVENT: CDCl3
NA = 8
HO
H
n-C9H19
(Ra)-4jl
15.32
3.17
1.82
1.97
2.09
0.95
8
7
6
5
4
3
2
1
0
PPM


202.936
93.958
91.673
77.315
77.000
76.681
60.756
31.854
29.542
29.406
29.280
29.091
29.045
28.624
22.642
14.072
29.542
29.406
29.280
29.091
29.045
28.624
29.6
29.4
29.2
29.0
28.8
28.6
lwl-5-118C-400M
Oct 26 2014
SOLVENT: cdcl3
NA = 64
HO
H
n-C9H19
(Ra)-4jl
200
150
100
50
0
PPM

lwl-5-118-as-h-300-1-0.7-214

实验时间：2014-10-27,12:23:38　　报告时间：2014-10-27,16:47:52
谱图文件:D:\zhuguangjiong\ct\20141027\lwl-5-118-as-h-300-1-0.7-214..org

实验内容简介：



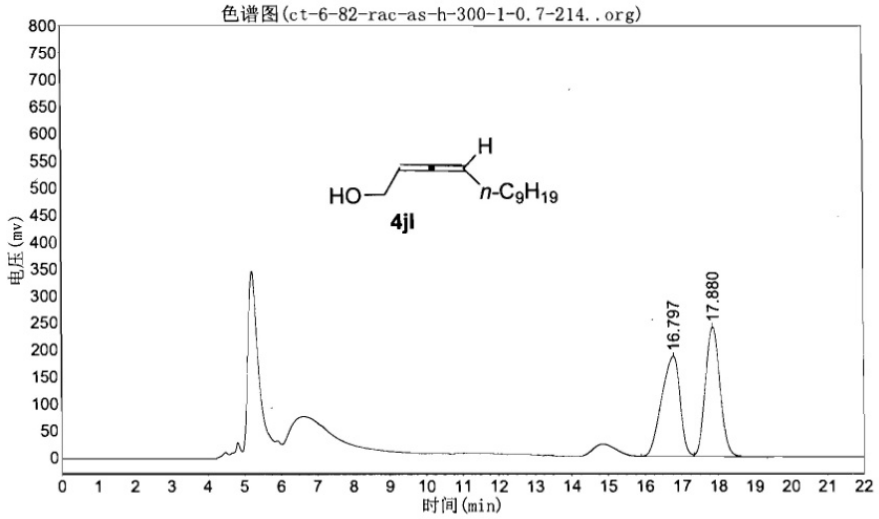
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.865	311774.219	8830430.000	97.3501
2		17.732	11937.540	240369.516	2.6499
总计			323711.759	9070799.516	100.0000

ct-6-82-rac-as-h-300-1-0.7-214

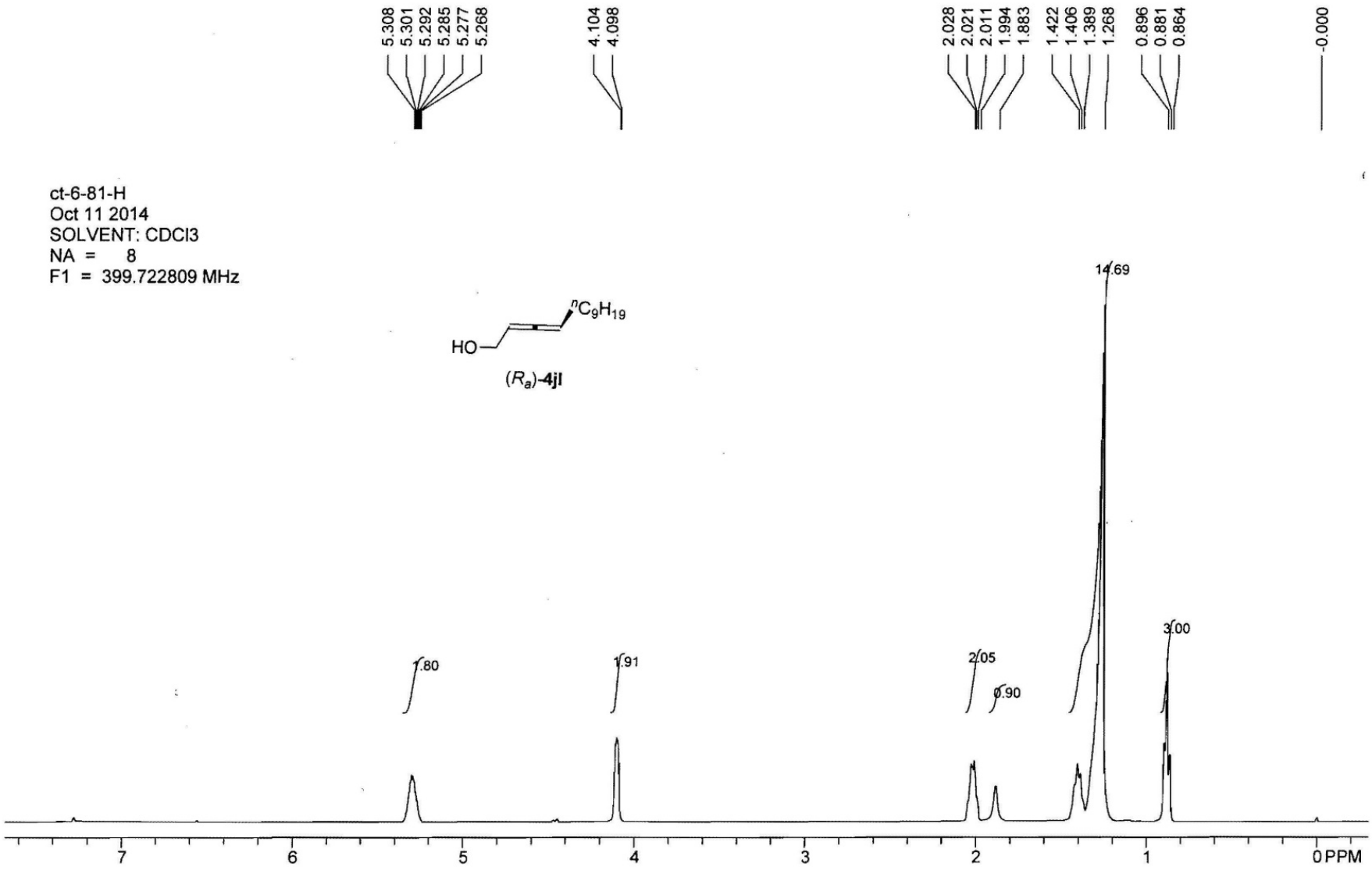
实验时间：2014-10-27,11:15:56　　报告时间：2014-10-27,16:46:38
谱图文件:D:\zhuguangjiong\ct\20141027\ct-6-82-rac-as-h-300-1-0.7-214..org

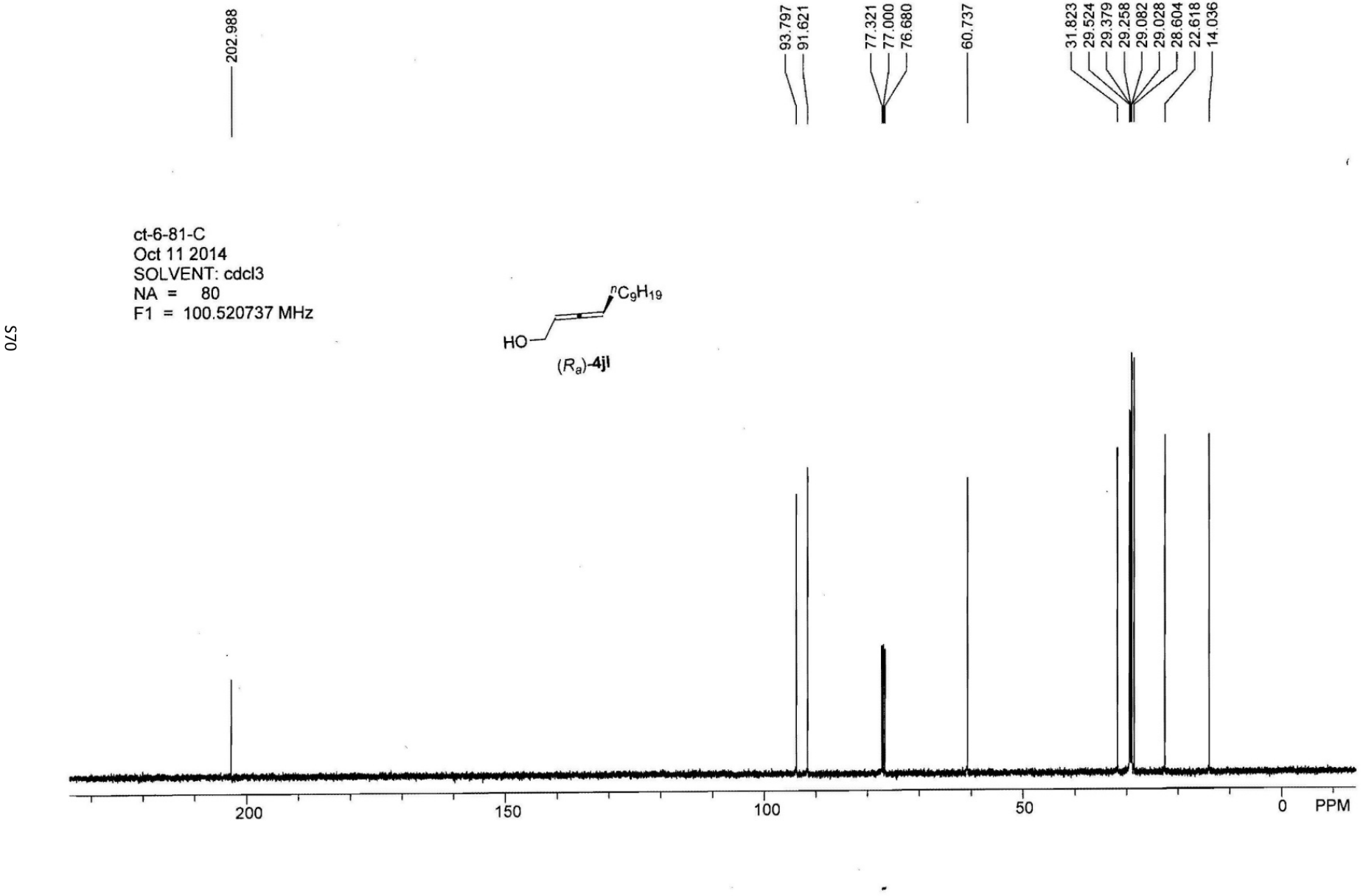
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.797	185313.500	6524928.500	49.4554
2		17.880	240394.469	6668629.500	50.5446
总计			425707.969	13193558.000	100.0000

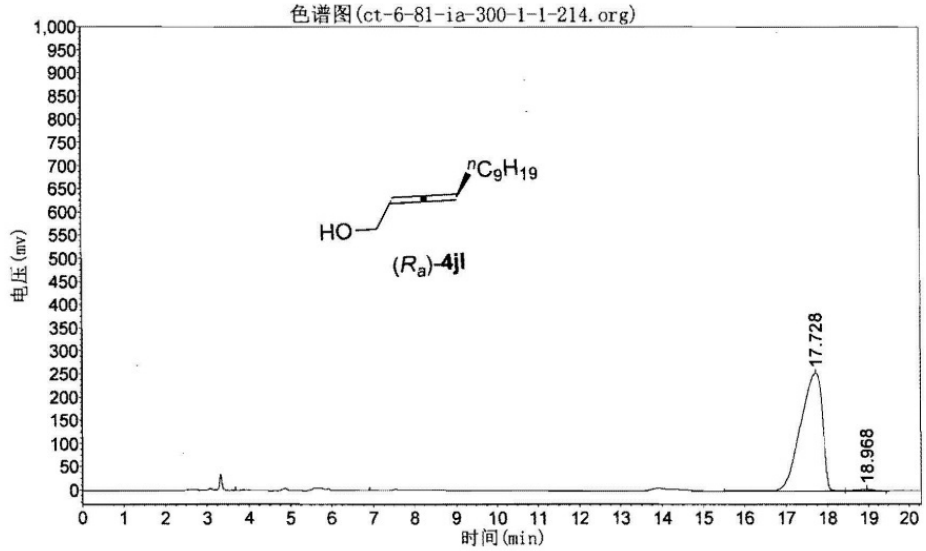

5.308
5.301
5.292
5.285
5.277
5.268
4.104
4.098
2.028
2.021
2.011
1.994
1.883
1.422
1.406
1.389
1.268
0.896
0.881
0.864
-0.000
ct-6-81-H
Oct 11 2014
SOLVENT: CDCl3
NA = 8
F1 = 399.722809 MHz
HO
$^{n}C_9H_{19}$
(R_a)-4jl
14.69
3.00
1.80
1.91
2.05
0.90
7
6
5
4
3
2
1
0 PPM


202.988
93.797
91.621
77.321
77.000
76.680
60.737
31.823
29.524
29.379
29.258
29.082
29.028
28.604
22.618
14.036
ct-6-81-C
Oct 11 2014
SOLVENT: cdcl3
NA = 80
F1 = 100.520737 MHz
HO
$^{n}C_9H_{19}$
(R_a)-4jl
200
150
100
50
0
PPM

ct-6-81-ia-300-1-1-214

实验时间：2014-10-11,14:43:11　　报告时间：2014-10-11,15:29:39
谱图文件:D:\zhuguangjiong\lwl\20141011\ct-6-81-ia-300-1-1-214.org

实验内容简介：



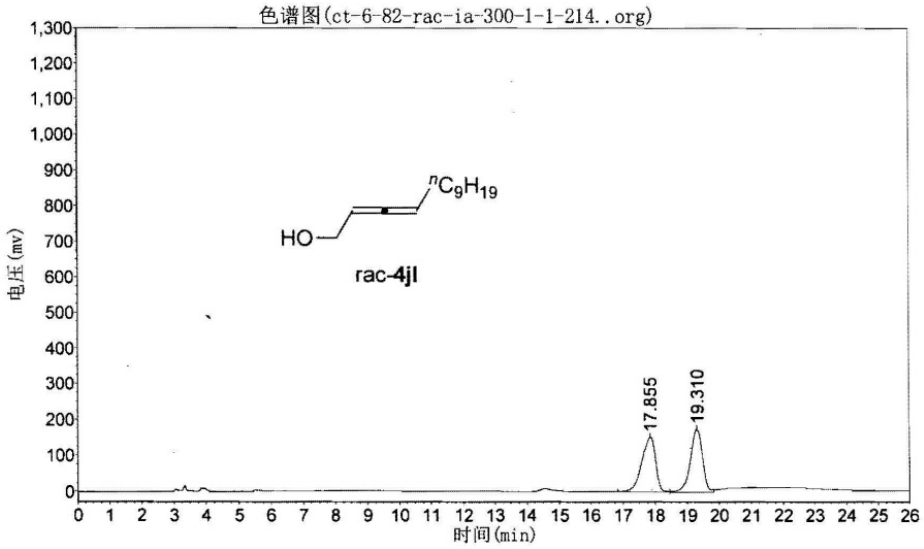
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.728	253951.594	9382286.000	98.9350
2		18.968	3633.985	100993.758	1.0650
总计			257585.578	9483279.758	100.0000

ct-6-82-rac-ia-300-1-1-214

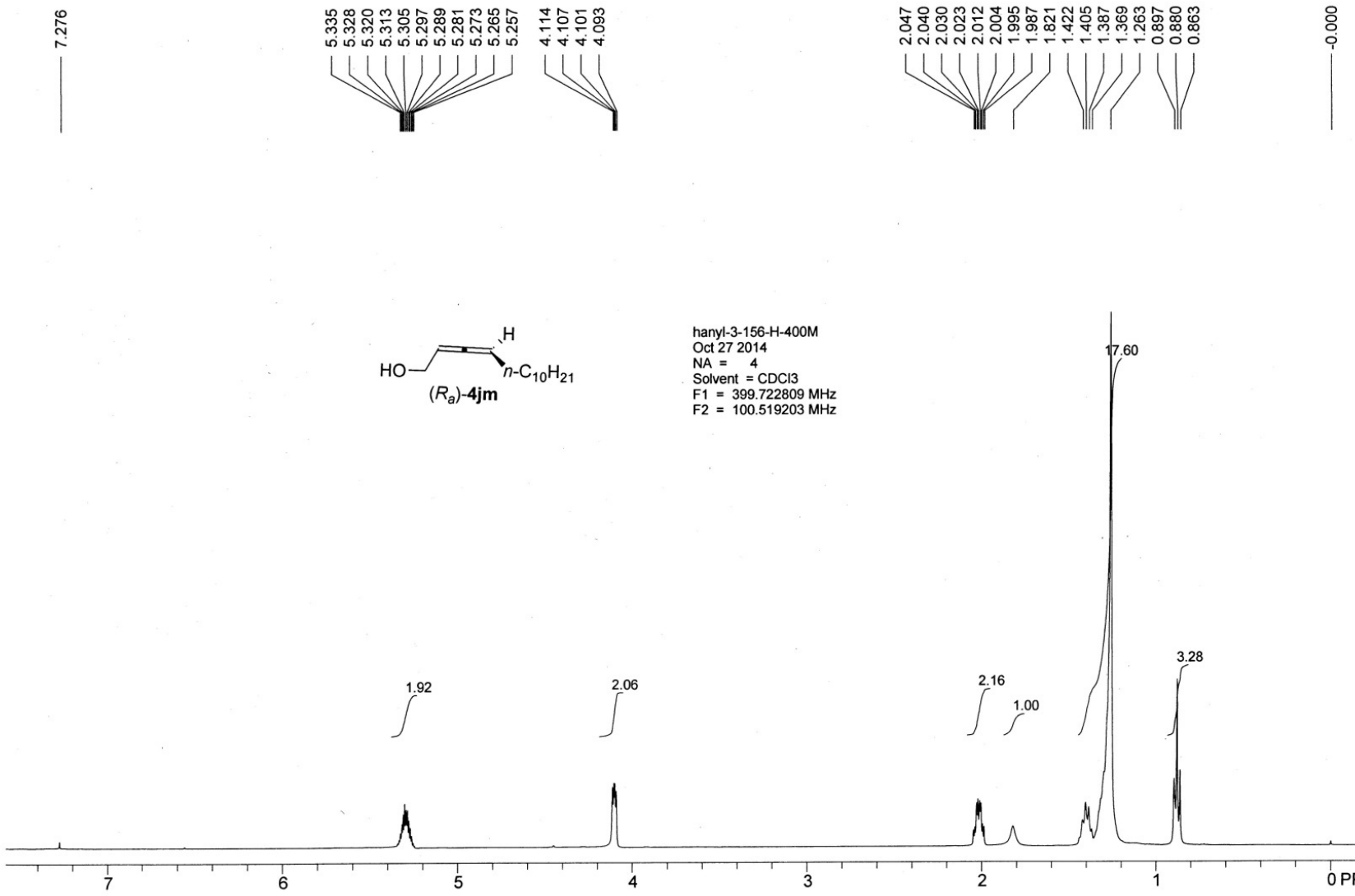
实验时间：2014-10-11,12:48:26　　报告时间：2014-10-11,15:36:08
谱图文件:D:\zhuguangjiong\lwl\20141011\ct-6-82-rac-ia-300-1-1-214..org

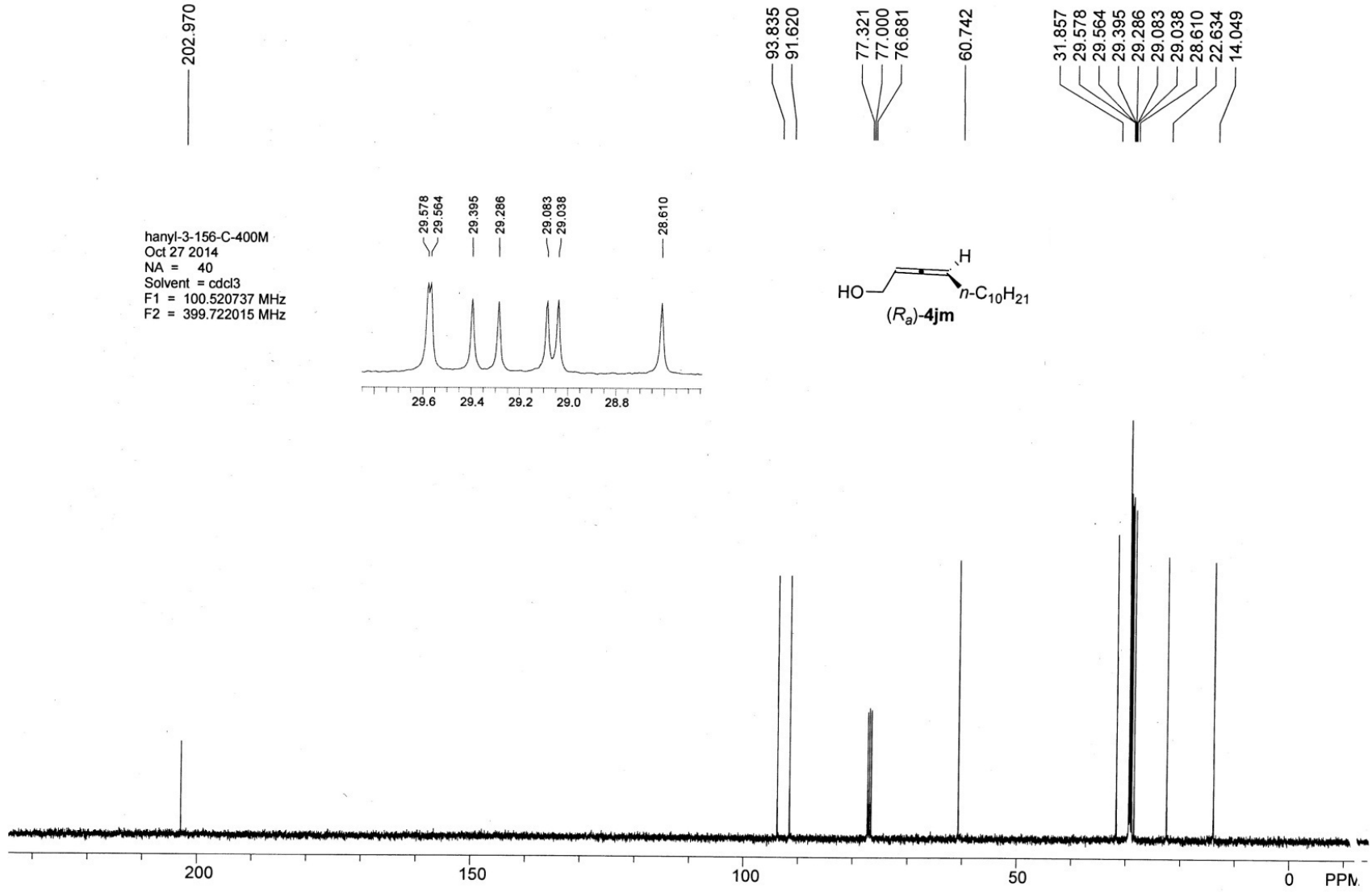
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.855	151675.219	4641426.500	50.0624
2		19.310	175117.688	4629859.500	49.9376
总计			326792.906	9271286.000	100.0000

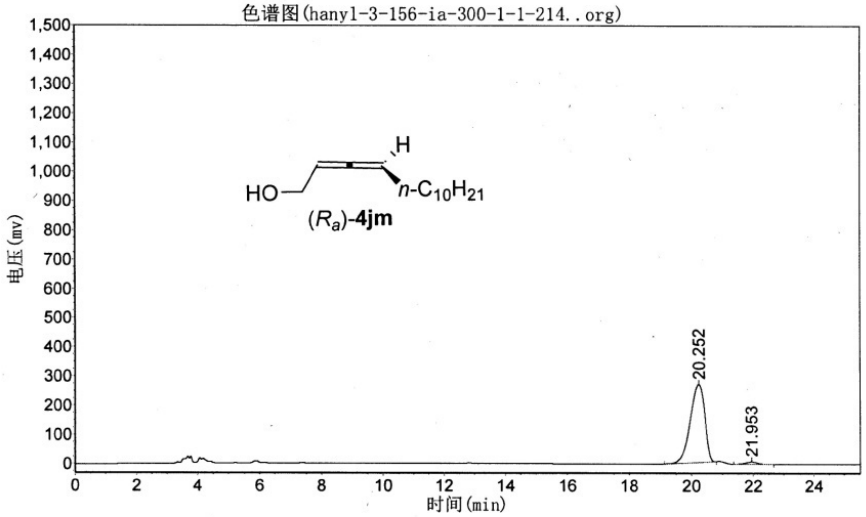

7.276
5.335
5.328
5.320
5.313
5.305
5.297
5.289
5.281
5.273
5.265
5.257
4.114
4.107
4.101
4.093
2.047
2.040
2.030
2.023
2.012
2.004
1.995
1.987
1.821
1.422
1.405
1.387
1.369
1.263
0.897
0.880
0.863
-0.000
HO
H
n-C10H21
(Ra)-4jm
hanyl-3-156-H-400M
Oct 27 2014
NA = 4
Solvent = CDCl3
F1 = 399.722809 MHz
F2 = 100.519203 MHz
1.92
2.06
2.16
1.00
17.60
3.28
7
6
5
4
3
2
1
0 PPM


202.970
93.835
91.620
77.321
77.000
76.681
60.742
31.857
29.578
29.564
29.395
29.286
29.083
29.038
28.610
22.634
14.049
hanyl-3-156-C-400M
Oct 27 2014
NA = 40
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
29.6
29.4
29.2
29.0
28.8
HO
H
n-C10H21
(Ra)-4jm
200
150
100
50
0
PPM

hanyl-3-156-ia-300-1-1-214

实验时间：2014-10-27,17:33:03　　报告时间：2014-10-27,18:00:50
谱图文件:D:\zhuguangjiong\hyl\20141027\hanyl-3-156-ia-300-1-1-214..org

实验内容简介：



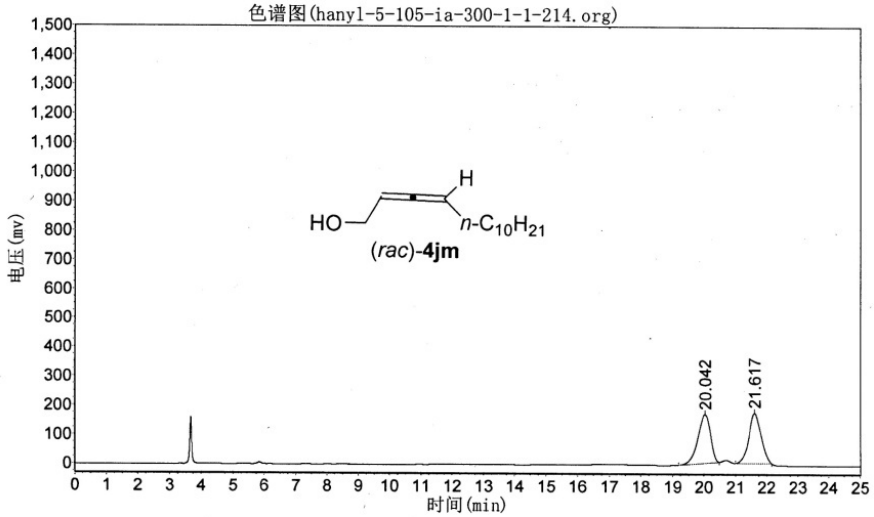
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.252	267351.750	8665986.000	97.4259
2		21.953	8677.536	228966.609	2.5741
总计			276029.286	8894952.609	100.0000

hanyl-5-105-ia-300-1-1-214

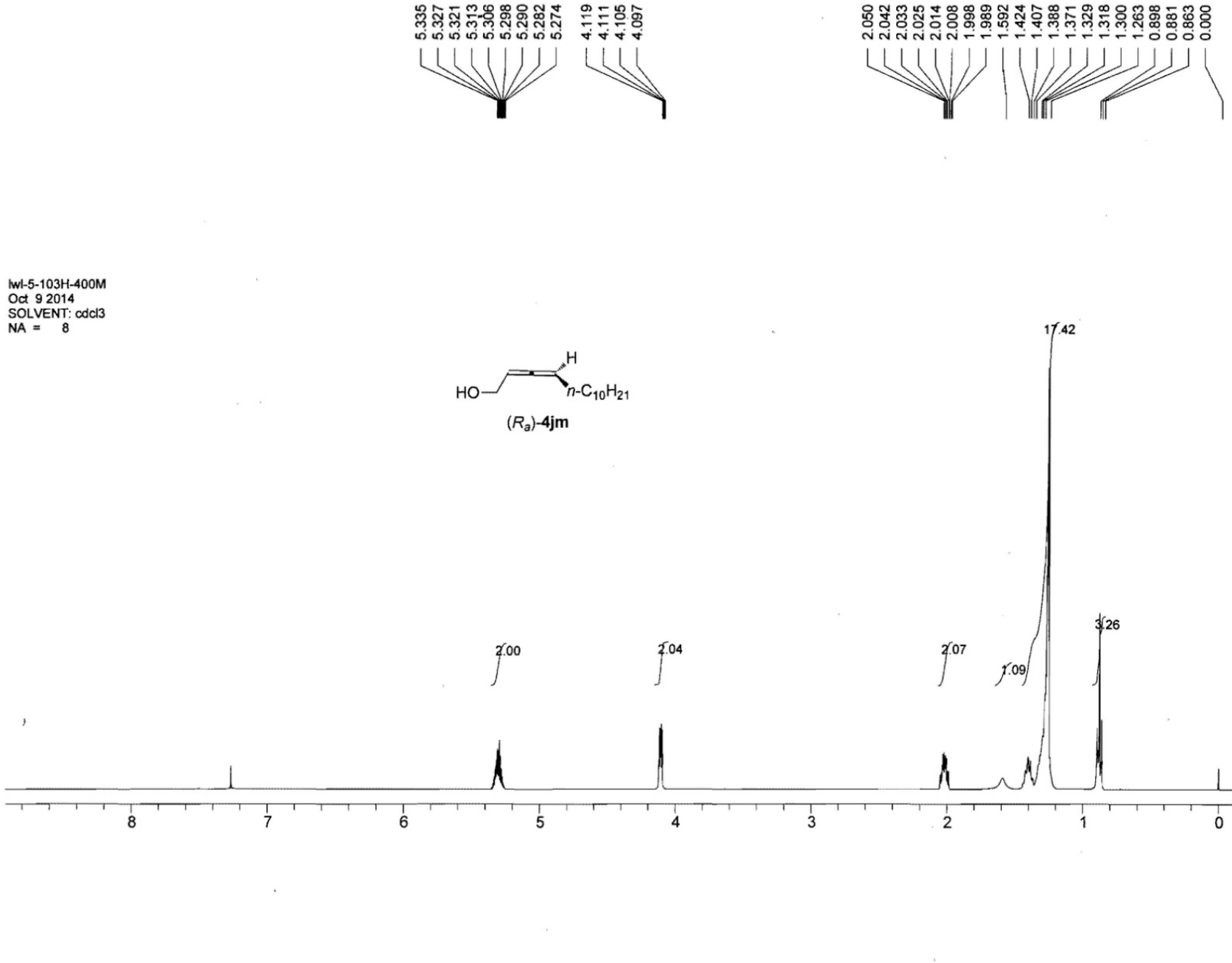
实验时间：2014-10-27,16:40:37　　报告时间：2014-10-27,17:37:05
谱图文件:D:\zhuguangjiong\hyl\20141027\hanyl-5-105-ia-300-1-1-214.org

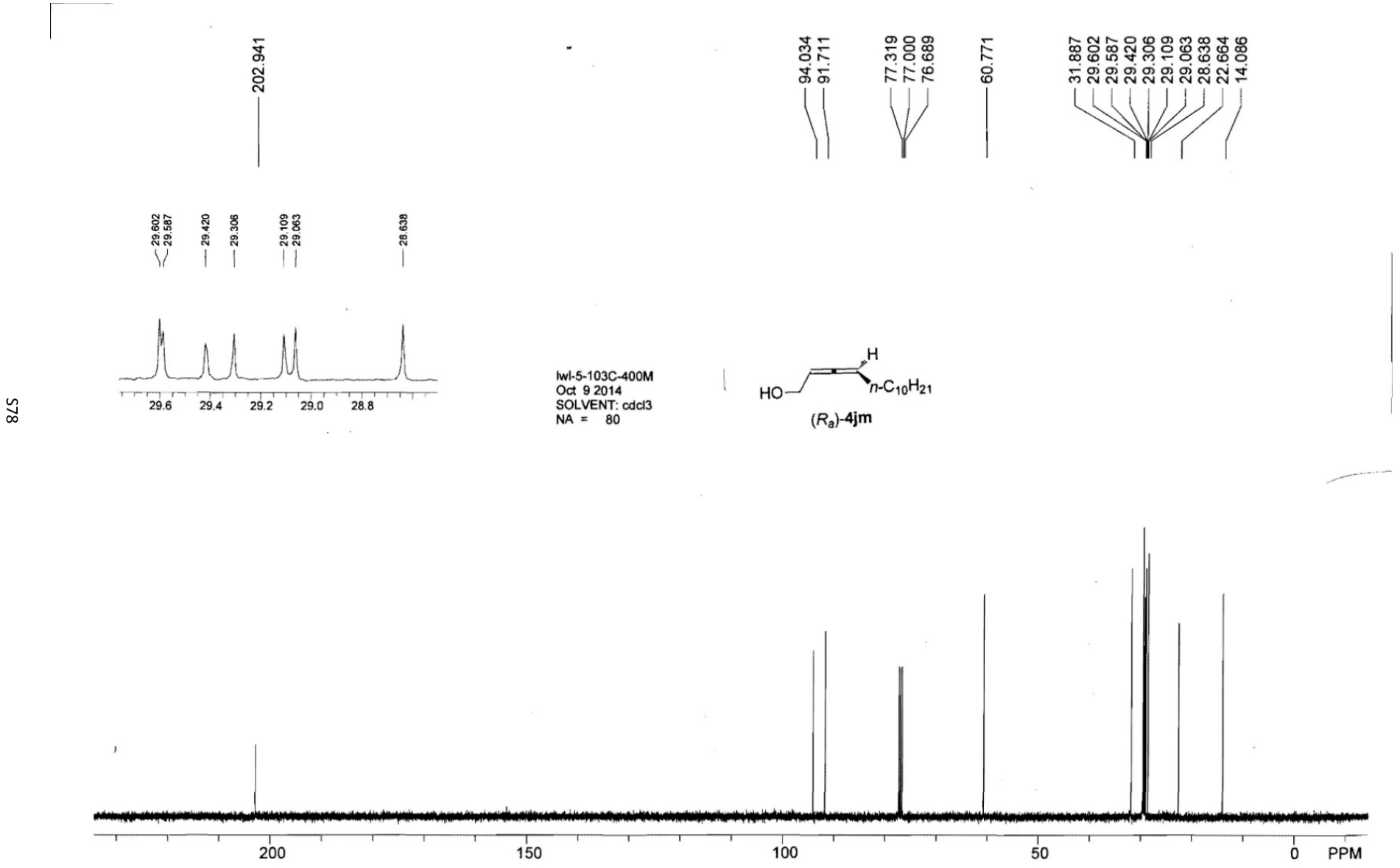
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.042	166376.344	4677892.500	49.7013
2		21.617	171770.469	4734112.000	50.2987
总计			338146.813	9412004.500	100.0000

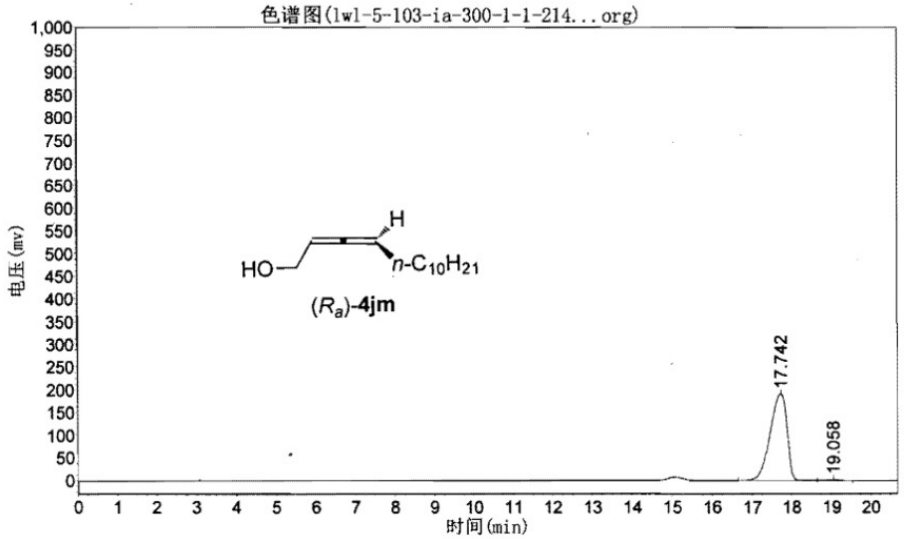

5.335
5.327
5.321
5.313
5.306
5.298
5.290
5.282
5.274
4.119
4.111
4.105
4.097
2.050
2.042
2.033
2.025
2.014
2.008
1.998
1.989
1.592
1.424
1.407
1.388
1.371
1.329
1.318
1.300
1.263
0.898
0.881
0.863
0.000
lwl-5-103H-400M
Oct 9 2014
SOLVENT: cdcl3
NA = 8
HO
H
n-C10H21
(Ra)-4jm
17.42
3.26
2.00
2.04
2.07
1.09
8
7
6
5
4
3
2
1
0
PPM


202.941
94.034
91.711
77.319
77.000
76.689
60.771
31.887
29.602
29.587
29.420
29.306
29.109
29.063
28.638
22.664
14.086
29.602
29.587
29.420
29.306
29.109
29.063
28.638
29.6
29.4
29.2
29.0
28.8
lwl-5-103C-400M
Oct 9 2014
SOLVENT: cdcl3
NA = 80
HO
H
n-C10H21
(Ra)-4jm
200
150
100
50
0
PPM

lwl-5-103-ia-300-1-1-214

实验时间：2014-10-11,11:55:51　　报告时间：2014-10-11,15:41:28
谱图文件:D:\zhuguangjiong\lwl\20141011\lwl-5-103-ia-300-1-1-214...org

实验内容简介：



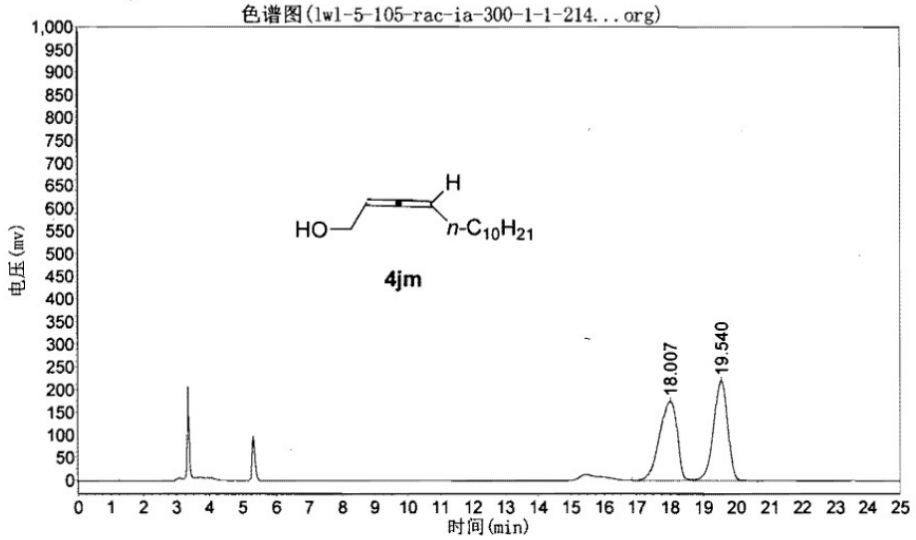
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.742	190325.438	5636665.000	99.0375
2		19.058	2362.942	54778.160	0.9625
总计			192688.380	5691443.160	100.0000

lwl-5-105-rac-ia-300-1-1-214

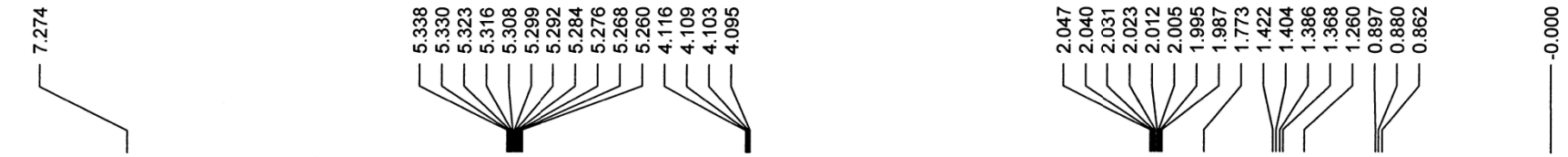
实验时间：2014-10-11,11:01:09　　报告时间：2014-10-11,15:38:06
谱图文件:D:\zhuguangjiong\lwl\20141011\lwl-5-105-rac-ia-300-1-1-214...org

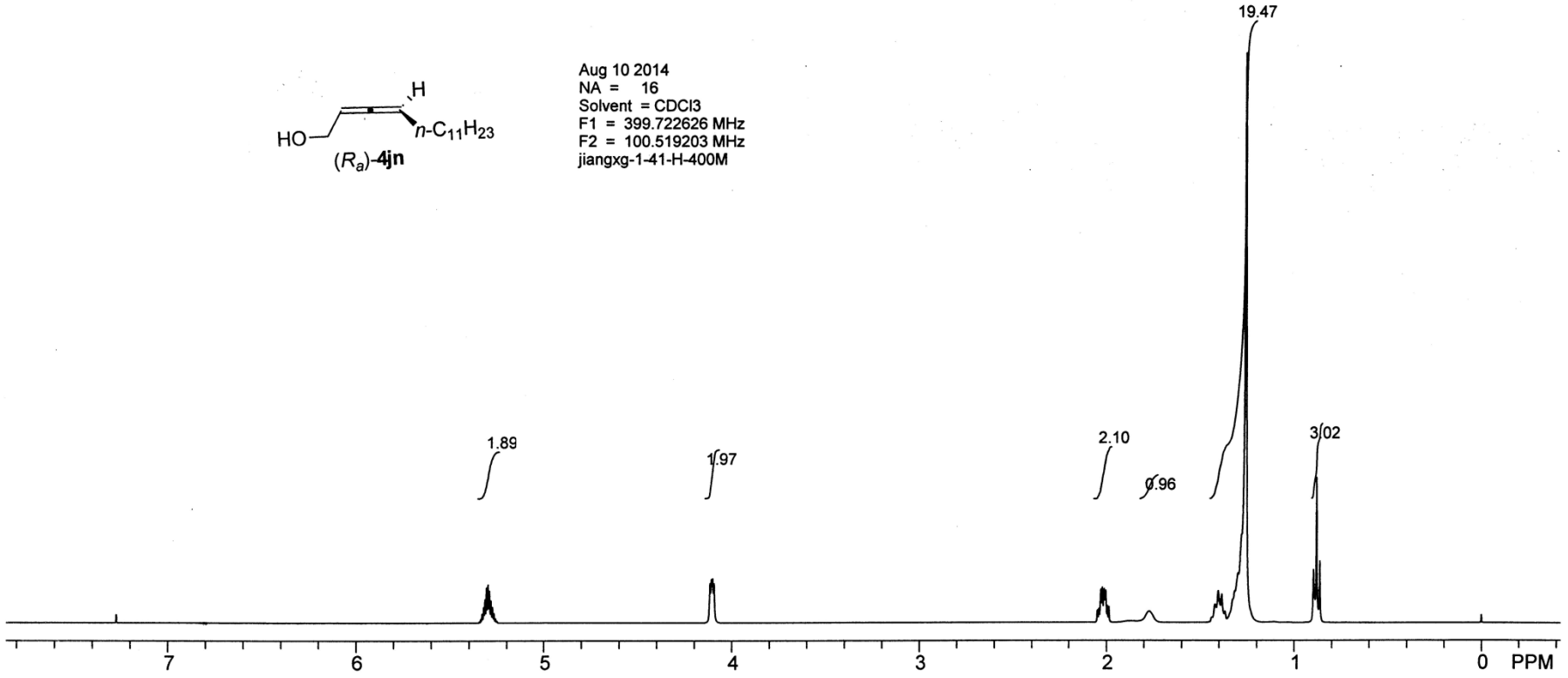
实验内容简介：

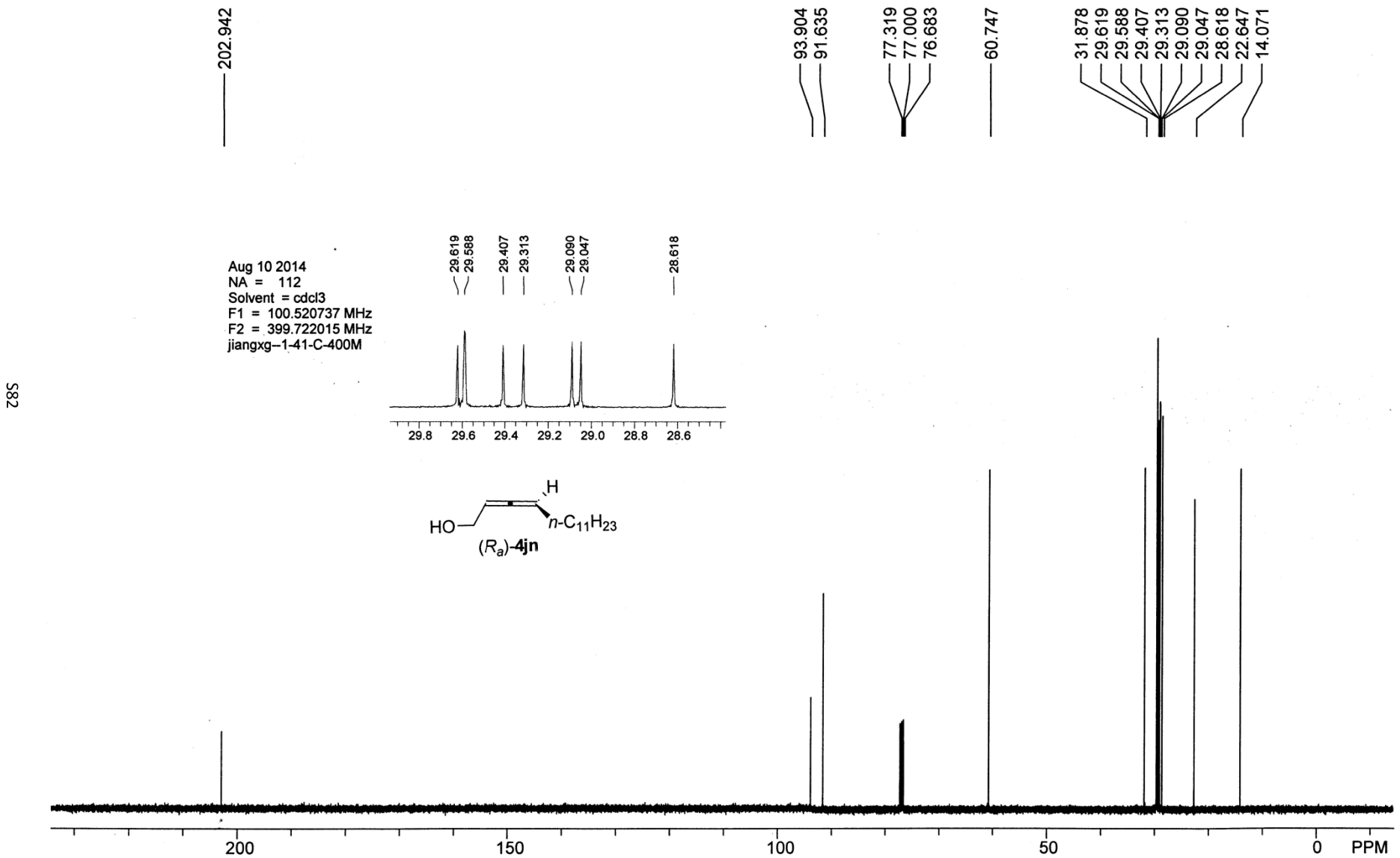


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		18.007	174036.000	6481541.000	49.6361
2		19.540	219611.266	6576580.500	50.3639
总计			393647.266	13058121.500	100.0000


7.274
5.338
5.330
5.323
5.316
5.308
5.299
5.292
5.284
5.276
5.268
5.260
4.116
4.109
4.103
4.095
2.047
2.040
2.031
2.023
2.012
2.005
1.995
1.987
1.773
1.422
1.404
1.386
1.368
1.260
0.897
0.880
0.862
-0.000

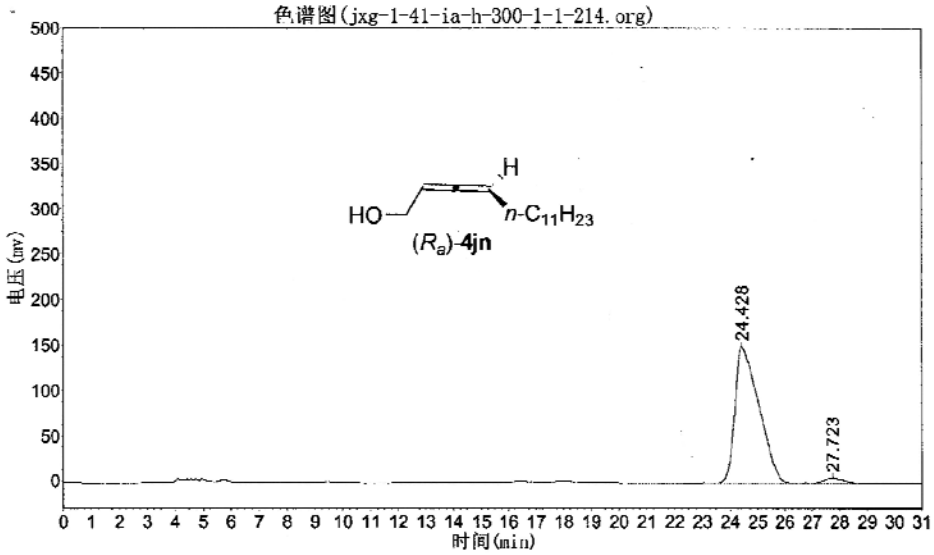

HO
H
n-C11H23
(Ra)-4jn
Aug 10 2014
NA = 16
Solvent = CDCl3
F1 = 399.722626 MHz
F2 = 100.519203 MHz
jiangxg-1-41-H-400M
19.47
1.89
1.97
2.10
0.96
3.02
7
6
5
4
3
2
1
0
PPM


202.942
93.904
91.635
77.319
77.000
76.683
60.747
31.878
29.619
29.588
29.407
29.313
29.090
29.047
28.618
22.647
14.071
Aug 10 2014
NA = 112
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
jiangxg-1-41-C-400M
29.8
29.6
29.4
29.2
29.0
28.8
28.6
HO
H
n-C11H23
(Ra)-4jn
200
150
100
50
0
PPM

jxg-1-41-ia-300-1-1-214

实验时间：2014-08-11,18:12:34　　报告时间：2014-08-11,18:46:02
谱图文件:D:\zhuguangjiong\jxg\20140811\jxg-1-41-ia-h-300-1-1-214.org

实验内容简介：



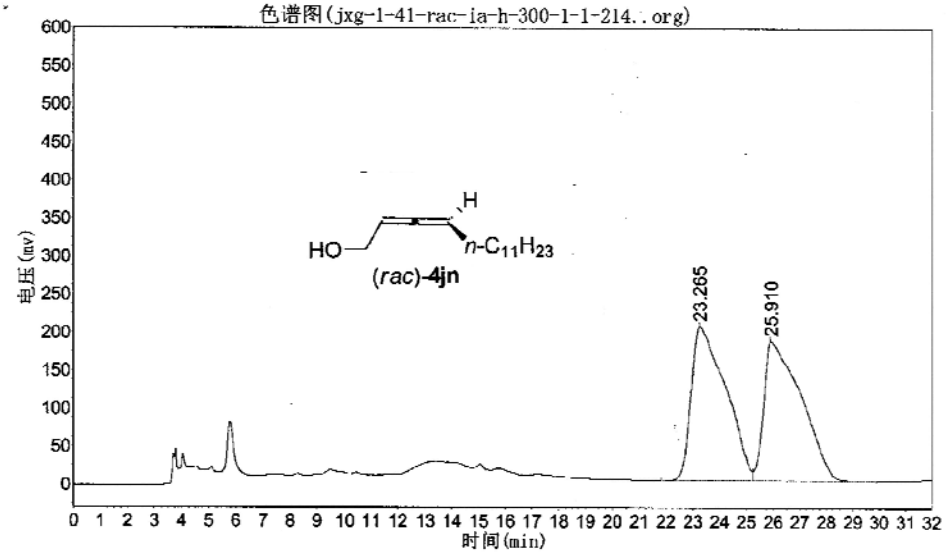
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		24.428	151431.531	9186750.000	96.6876
2		27.723	5941.284	314729.219	3.3124
总计			157372.815	9501479.219	100.0000

jxg-1-41-rac-ia-300-1-1-214

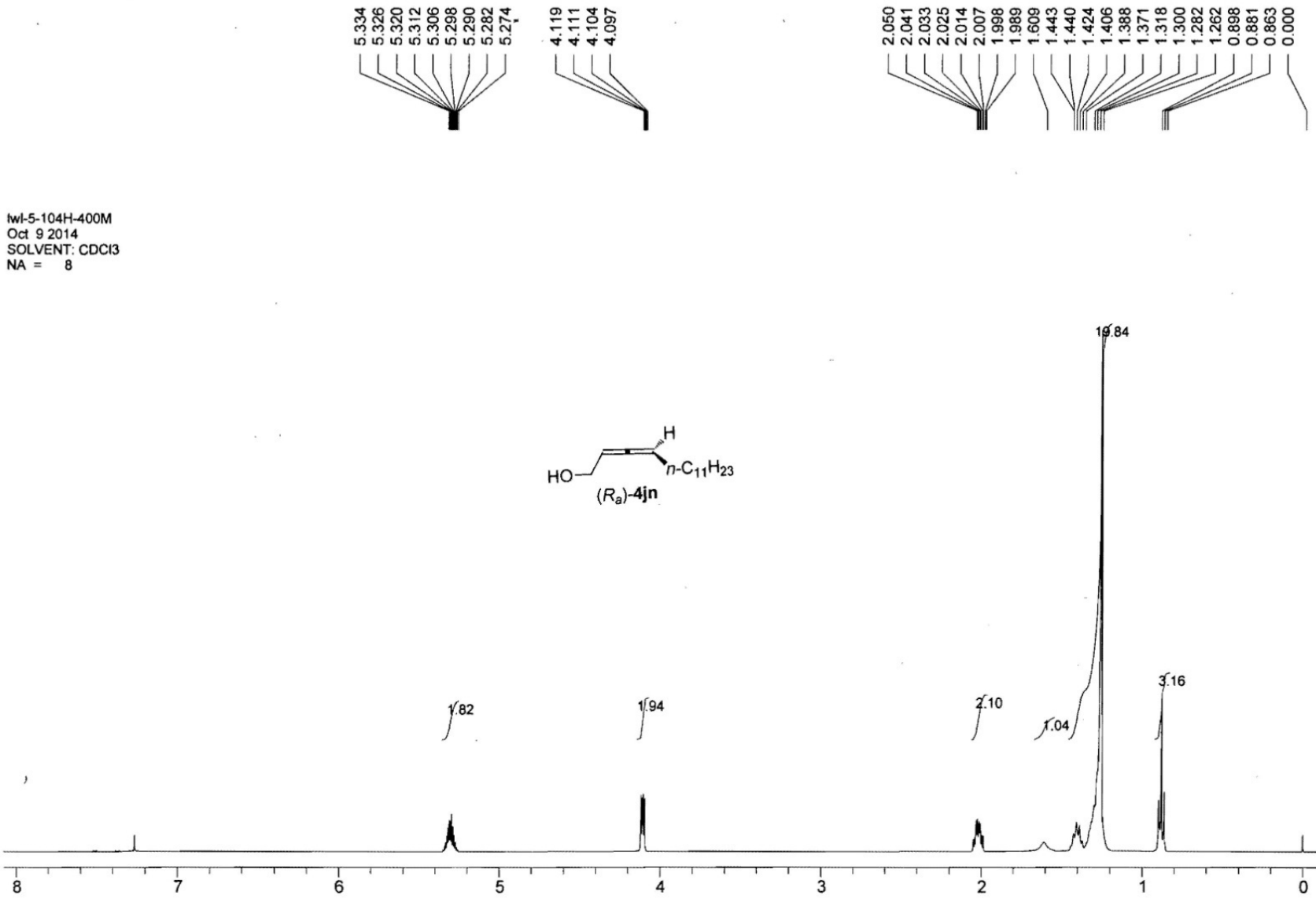
实验时间：2014-08-11,17:09:48　　报告时间：2014-08-11,19:12:18
谱图文件:D:\zhuguangjiong\jxg\20140811\jxg-1-41-rac-ia-h-300-1-1-214..org

实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		23.265	201993.031	18010684.000	50.2584
2		25.910	183076.875	17825486.000	49.7416
总计			385069.906	35836170.000	100.0000

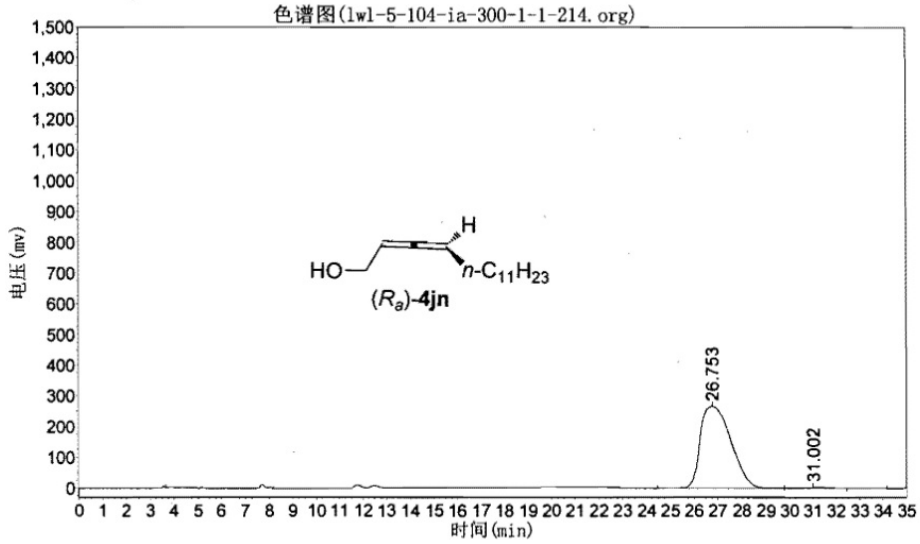

5.334
5.326
5.320
5.312
5.306
5.298
5.290
5.282
5.274
4.119
4.111
4.104
4.097
2.050
2.041
2.033
2.025
2.014
2.007
1.998
1.989
1.609
1.443
1.440
1.424
1.406
1.388
1.371
1.318
1.300
1.282
1.262
0.898
0.881
0.863
0.000
lwl-5-104H-400M
Oct 9 2014
SOLVENT: CDCl3
NA = 8
HO
H
n-C11H23
(Ra)-4jn
19.84
1.82
1.94
2.10
1.04
3.16
8
7
6
5
4
3
2
1
0
PPM

PPM
0
50
100
150
200
14.079
22.656
28.631
29.056
29.101
29.321
29.412
29.595
29.625
31.887
60.763
76.681
77.000
77.319
91.696
94.019
202.941
28.631
29.056
29.101
29.321
29.412
29.595
29.625
28.8
29.0
29.2
29.4
29.6
kwl-5-104C-400M
Oct 9 2014
SOLVENT: cdcl3
NA = 80
H
n-C11H23
HO
(Ra)-4jn

lwl-5-104-ia-300-1-1-214

实验时间：2014-10-09,16:43:30　　报告时间：2014-10-09,17:58:48
谱图文件:D:\zhuguangjiong\jxg\20141009\lwl-5-104-ia-300-1-1-214.org

实验内容简介：



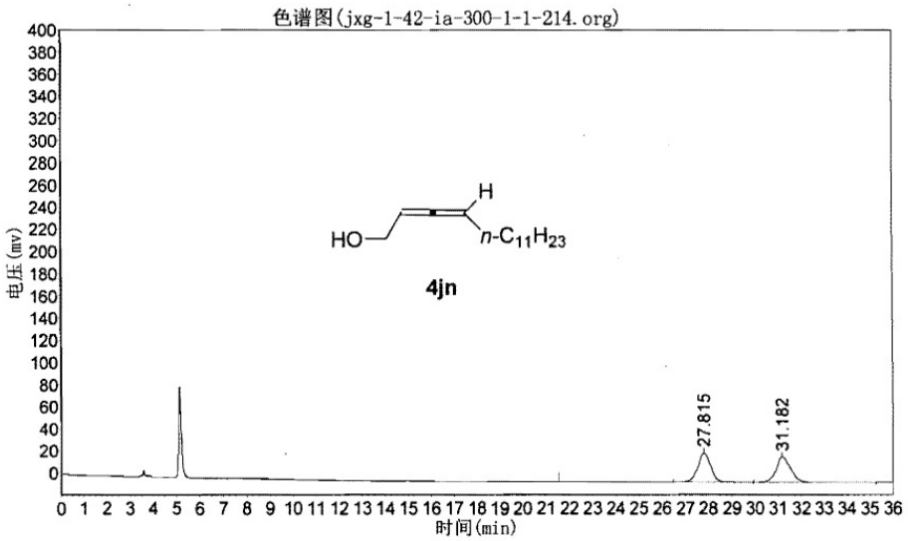
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		26.753	266640.063	23868370.000	99.0159
2		31.002	3173.606	237218.750	0.9841
总计			269813.668	24105588.750	100.0000

jxg-1-42-ia-300-1-1-214

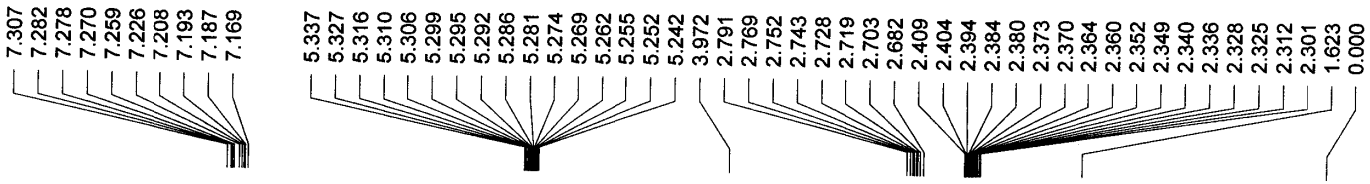
实验时间：2014-10-09,16:04:01　　报告时间：2014-10-09,17:56:11
谱图文件:D:\zhuguangjiong\jxg\20141009\jxg-1-42-ia-300-1-1-214.org

实验内容简介：



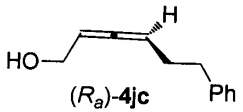
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.815	26575.361	1021568.563	50.1091
2		31.182	23382.260	1017120.250	49.8909
总计			49957.621	2038688.813	100.0000

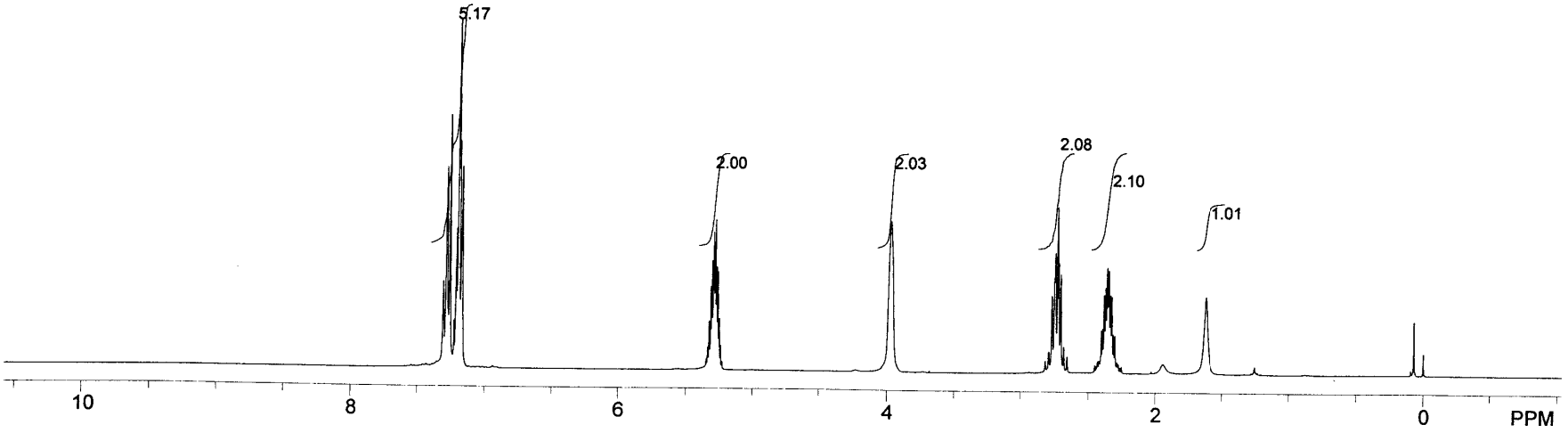


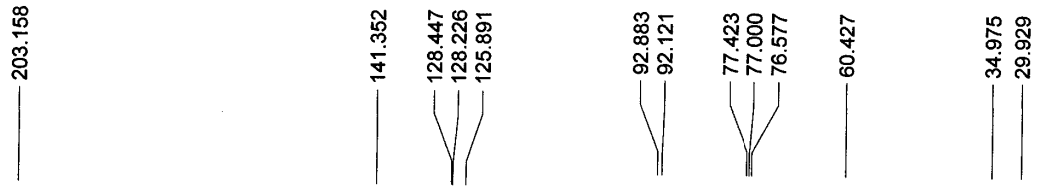
hx-10-166

2014-04-25 09:48:22.750
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 9



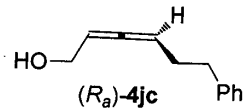
(R_a)-**4jc**



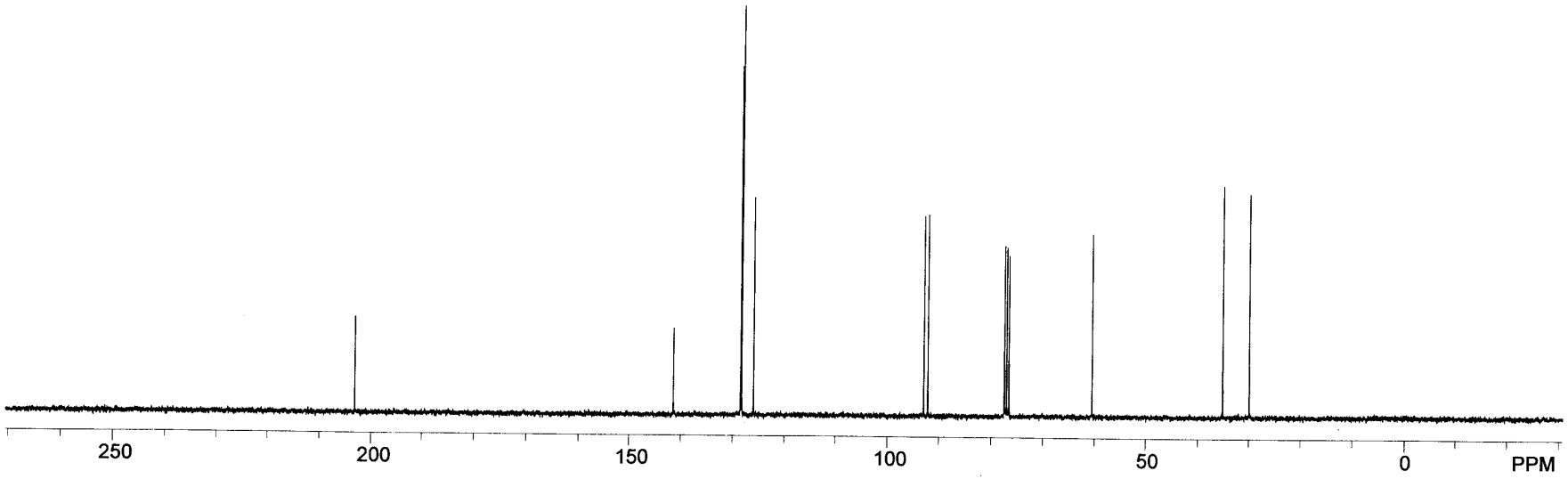


hx-10-167

2014-04-25 10:02:07.171
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 104



(R_a)-**4jc**

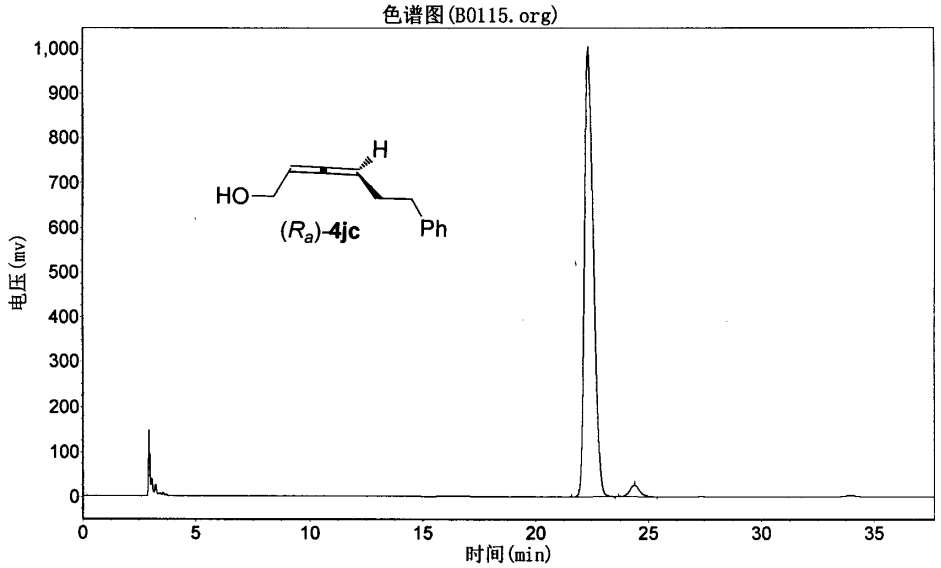


hx-10-166

实验时间：2014-04-26,10:36:14
谱图文件:D:\浙大智达\N2000\样品\B0115.org

实验者：hx
报告时间：2014-04-26,11:16:57
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 100/1, 214 nm, 1.0 ml/min



分析结果表

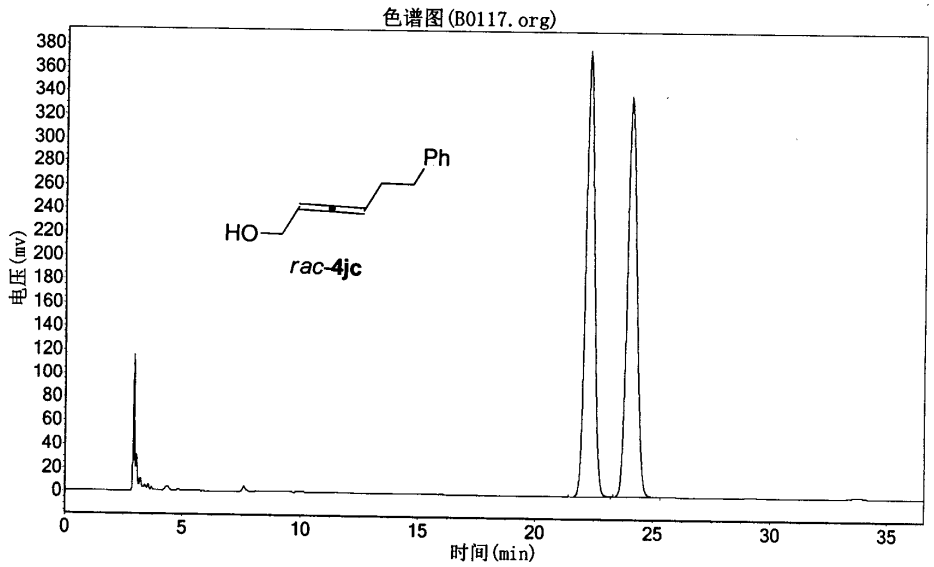
峰号	峰名	保留时间	峰高	峰面积	含量
1		22.353	993602.188	27298644.000	97.2946
2		24.387	25298.291	759071.750	2.7054
总计			1018900.479	28057715.750	100.0000

hx-10-168

实验时间：2014-04-26,11:54:21
谱图文件:D:\浙大智达\N2000\样品\B0117.org

实验者：hx
报告时间：2014-04-26,12:32:07
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 100/1, 214 nm, 1.0 ml/min

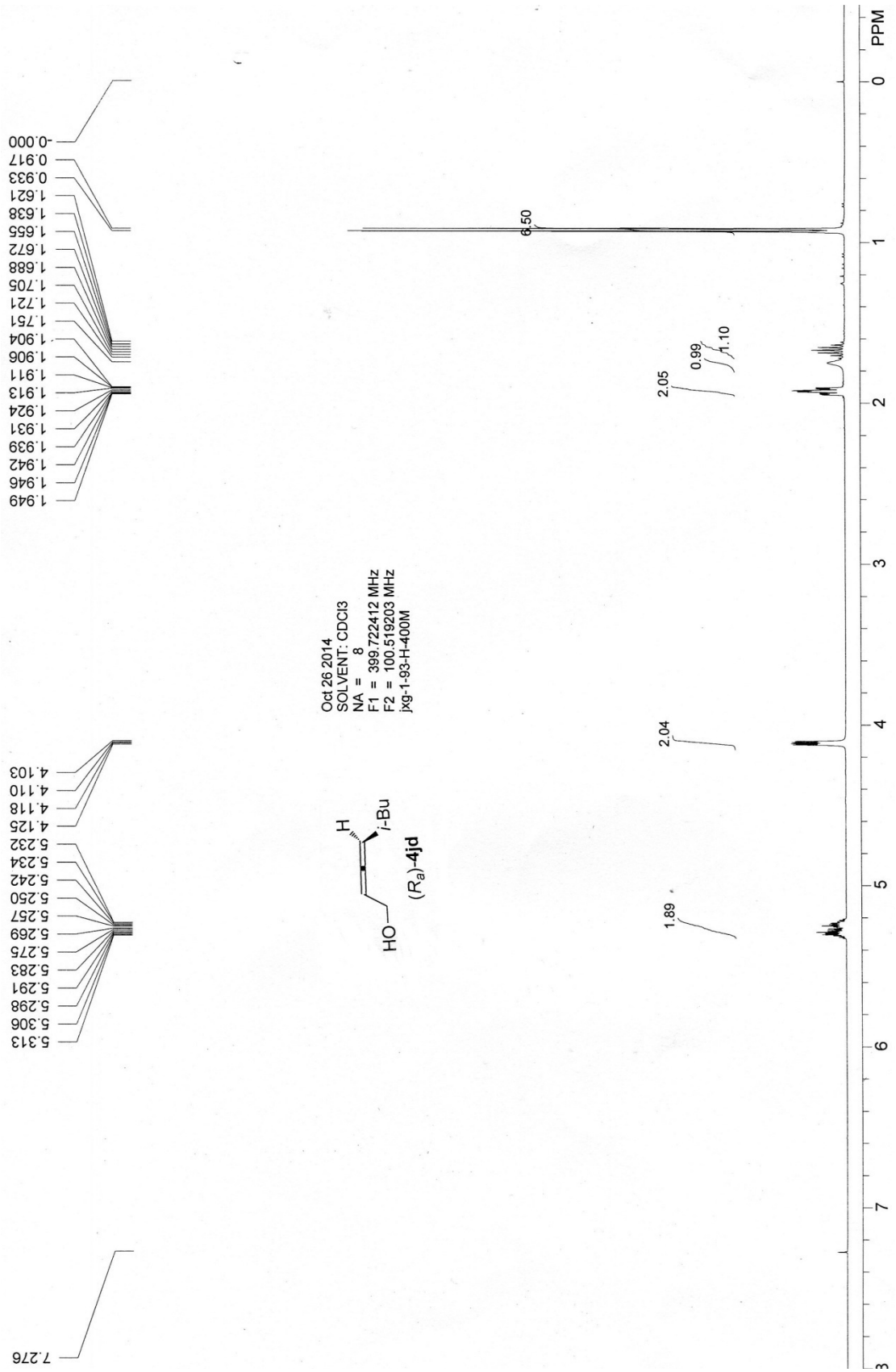


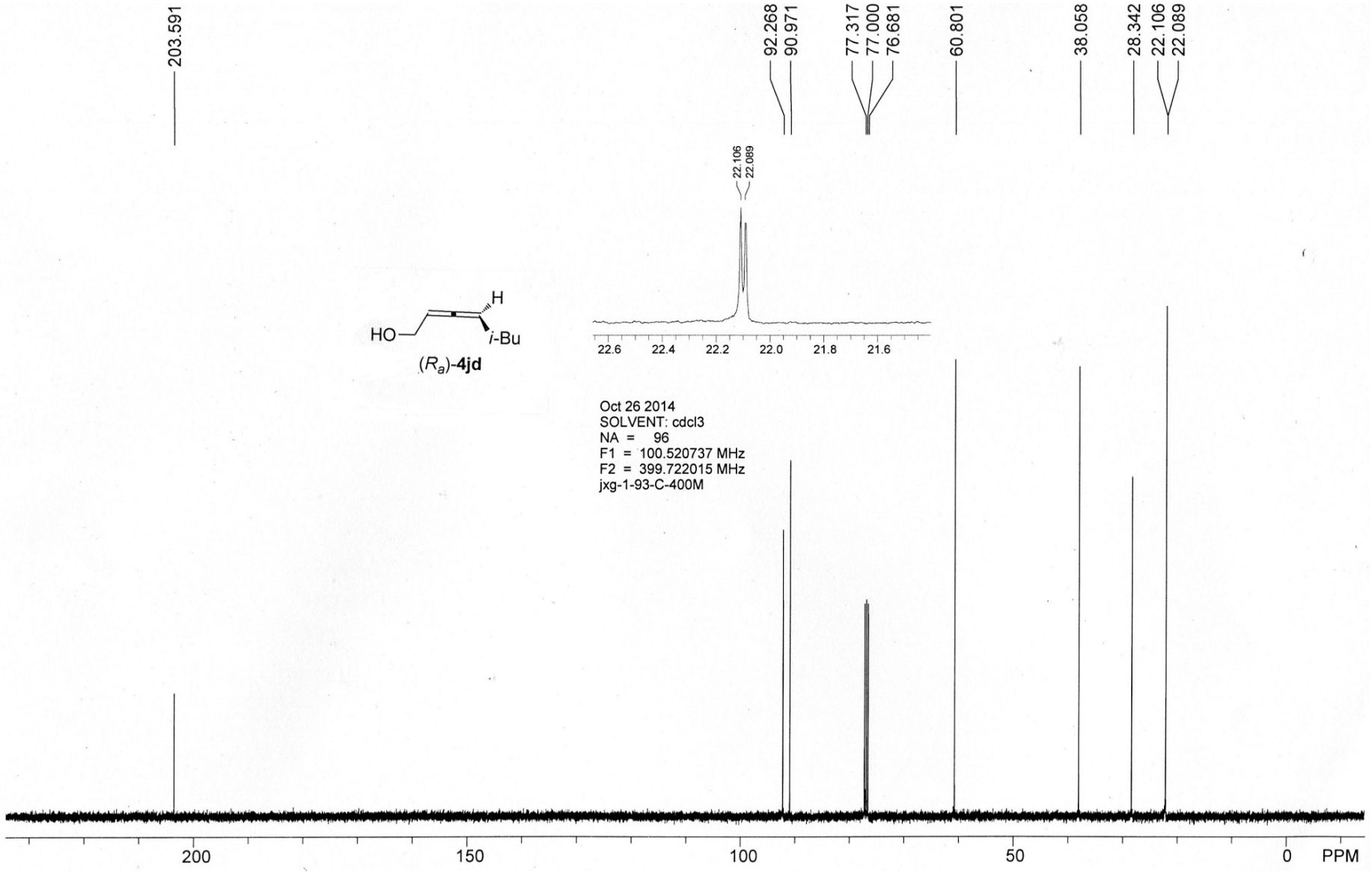
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		22.265	374109.656	9649850.000	50.1776
2		24.055	335473.156	9581534.000	49.8224
总计			709582.813	19231384.000	100.0000

2014-04-26

浙江大学智能信息研究所


Oct 26 2014
SOLVENT: CDCl3
NA = 8
F1 = 399.722412 MHz
F2 = 100.519203 MHz
jxg-1-93-H-400M
H
i-Bu
HO
(Ra)-4jd
6.50
2.05
0.99
1.10
2.04
1.89
PPM
0
1
2
3
4
5
6
7
-0.000
0.917
0.933
1.621
1.638
1.655
1.672
1.688
1.705
1.721
1.751
1.904
1.906
1.911
1.913
1.924
1.931
1.939
1.942
1.946
1.949
4.103
4.110
4.118
4.125
5.232
5.234
5.242
5.250
5.257
5.269
5.275
5.283
5.291
5.298
5.306
5.313
7.276


203.591
92.268
90.971
77.317
77.000
76.681
60.801
38.058
28.342
22.106
22.089
22.106
22.089
22.6
22.4
22.2
22.0
21.8
21.6
HO
H
i-Bu
(Ra)-4jd
Oct 26 2014
SOLVENT: cdcl3
NA = 96
F1 = 100.520737 MHz
F2 = 399.722015 MHz
jxg-1-93-C-400M
200
150
100
50
0
PPM

中国科学院上海有机化学研究所

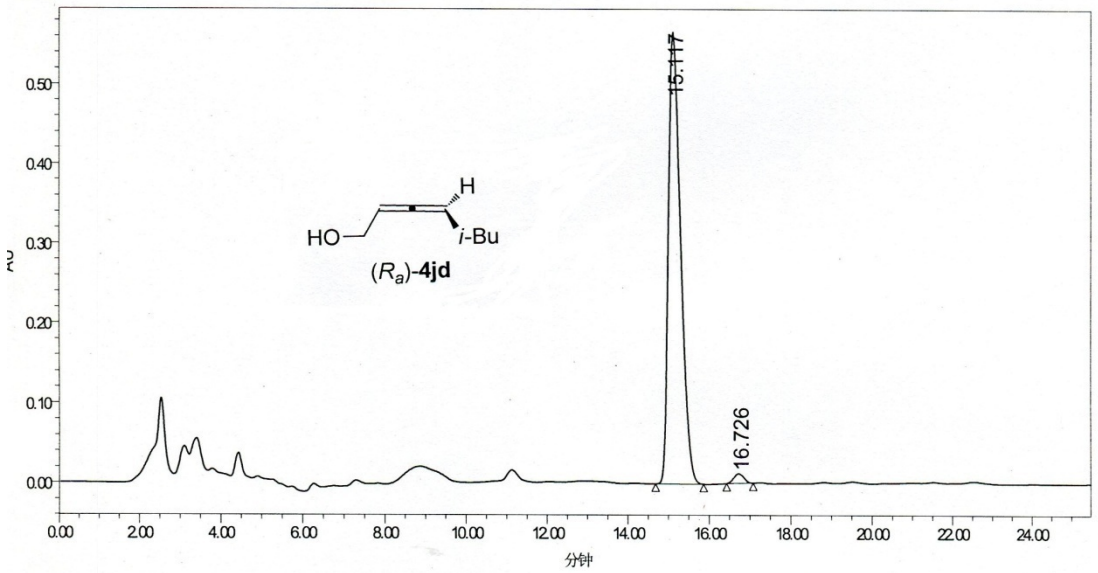
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	jxg-1-93-ad-h-100-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/10/28 12:15:46 CST
Vial:	1	Acq. Method:	zgj1001
Injection #:	12	Date Processed:	2014/10/28 15:20:08 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	35.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (礦sec)	%Area	Height (礦)	% Height
1	15.117	11770253	98.27	567162	97.91
2	16.726	207543	1.73	12116	2.09

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2014/10/28 15:21:24 PRC

中国科学院上海有机化学研究所

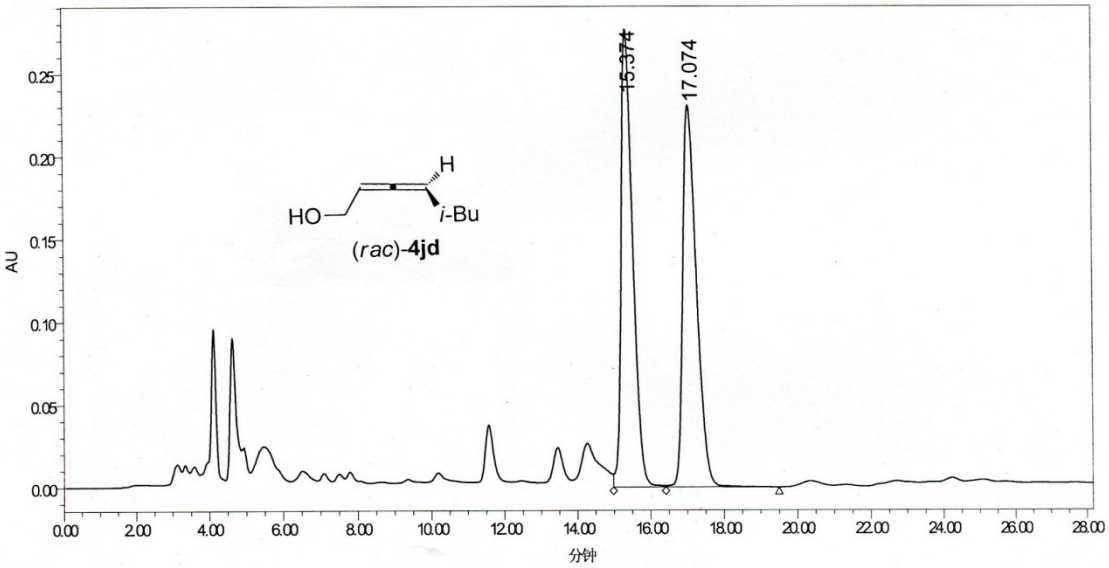
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-12-15-ad-h-100-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/10/28 10:11:58 CST
Vial:	1	Acq. Method:	zgj1001
Injection #:	10	Date Processed:	2014/10/28 15:20:27 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	35.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	

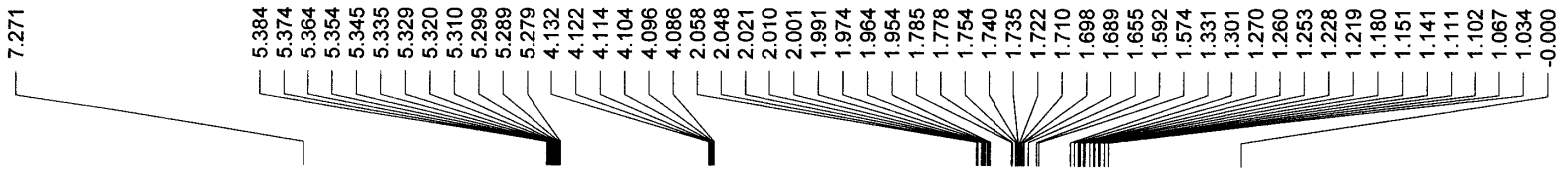


	RT (min)	Area (碘sec)	%Area	Height (碘)	% Height
1	15.374	6097452	50.30	277476	54.54
2	17.074	6025788	49.70	231242	45.46

Report Method: Individual Report ASC

Page: 1 (共计 1)

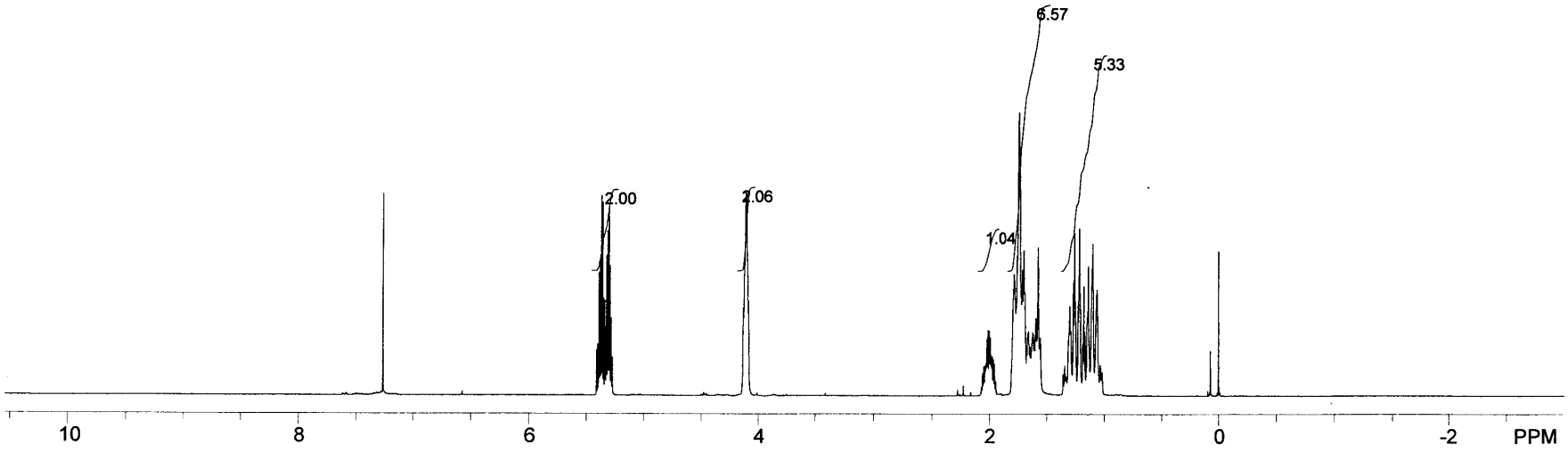
Printed: 2014/10/28 15:20:54 PRC

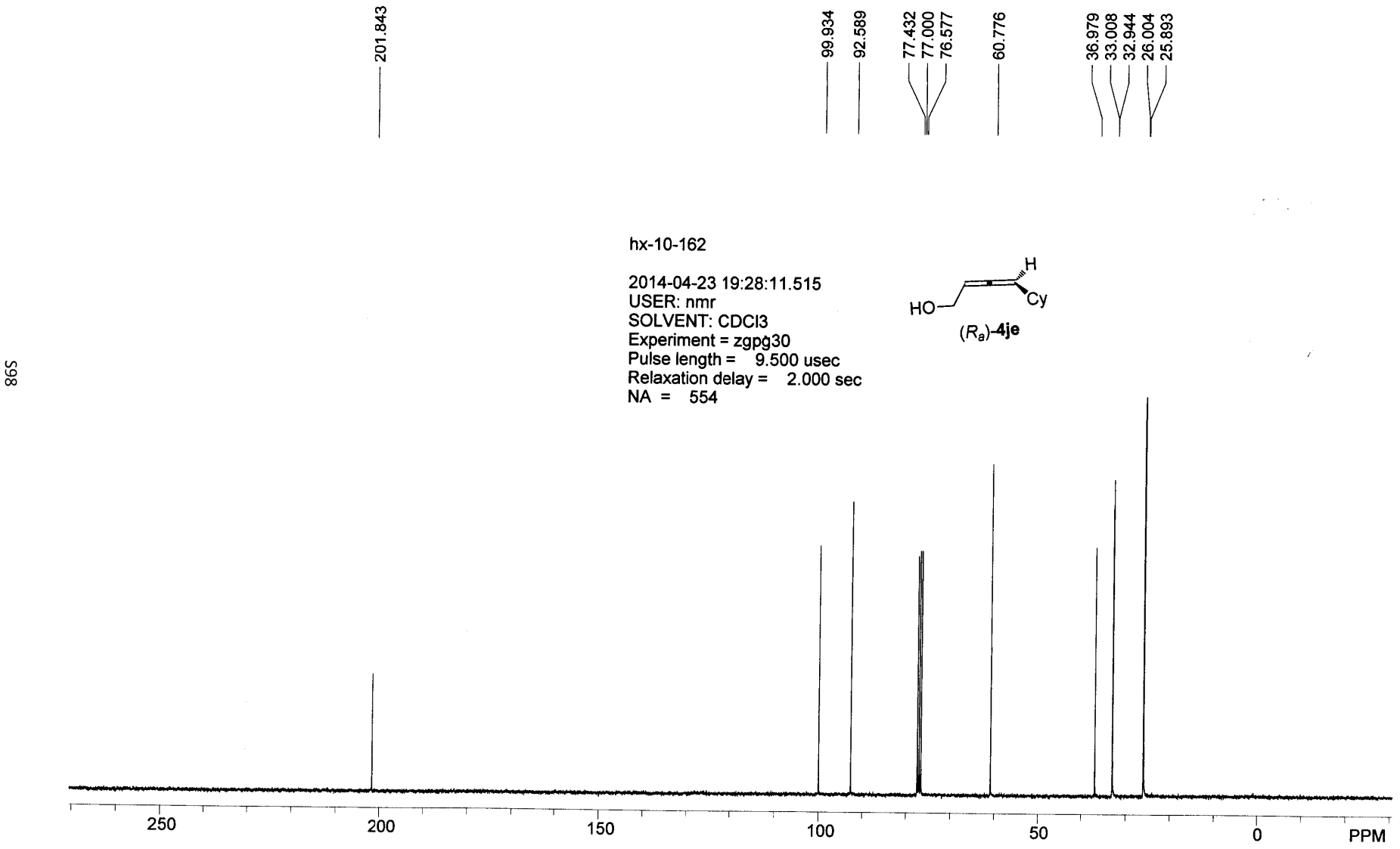


hx-10-161

2014-04-23 18:33:18.265
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 12

(R_a)-**4je**




201.843
99.934
92.589
77.432
77.000
76.577
60.776
36.979
33.008
32.944
26.004
25.893
hx-10-162
2014-04-23 19:28:11.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 554
HO
H
Cy
(Ra)-4je
250
200
150
100
50
0
PPM

N2000 数据工作站

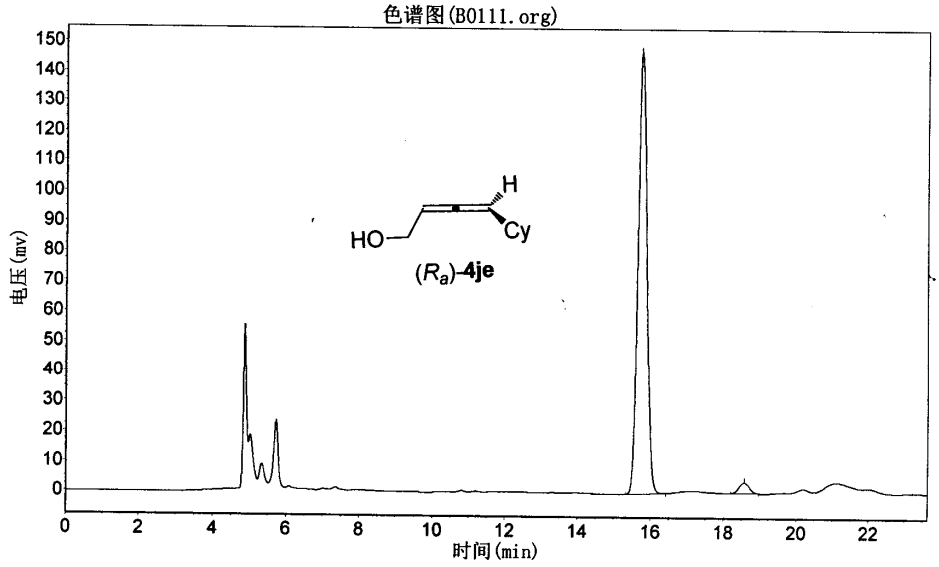
hx-10-161

实验时间：2014-04-23,21:20:00
谱图文件:D:\浙大智达\N2000\样品\B0111.org

实验者：hx
报告时间：2014-04-23,21:45:05
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 98/2, 214 nm, 0.6 ml/min

色谱图(B0111.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.748	147142.844	2490712.750	97.2282
2		18.572	3610.368	71005.398	2.7718
总计			150753.212	2561718.148	100.0000

2014-04-23

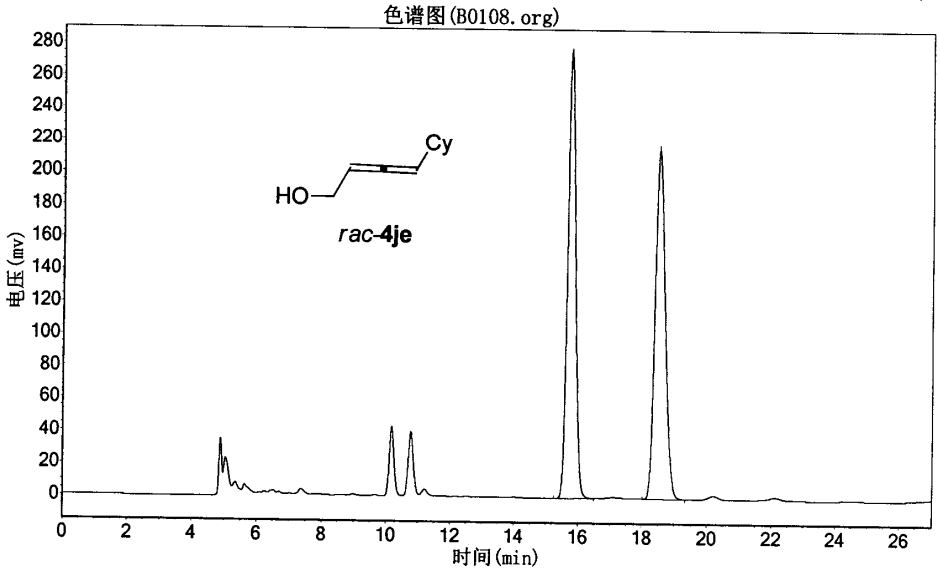
浙江大学智能信息研究所

hx-10-160

实验时间：2014-04-23,19:45:06
谱图文件:D:\浙大智达\N2000\样品\B0108.org

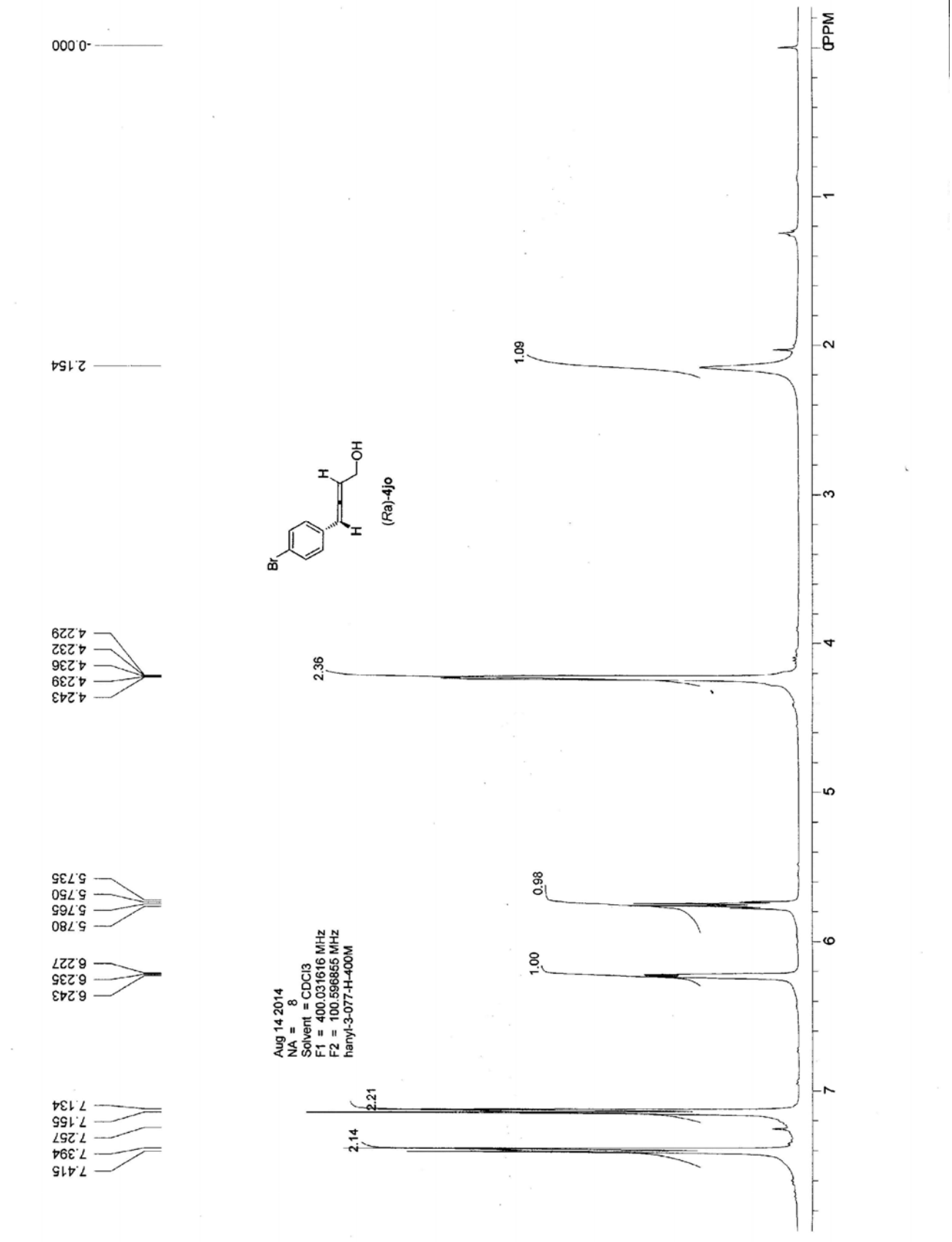
实验者：hx
报告时间：2014-04-23,20:14:45
积分方法:面积归一法

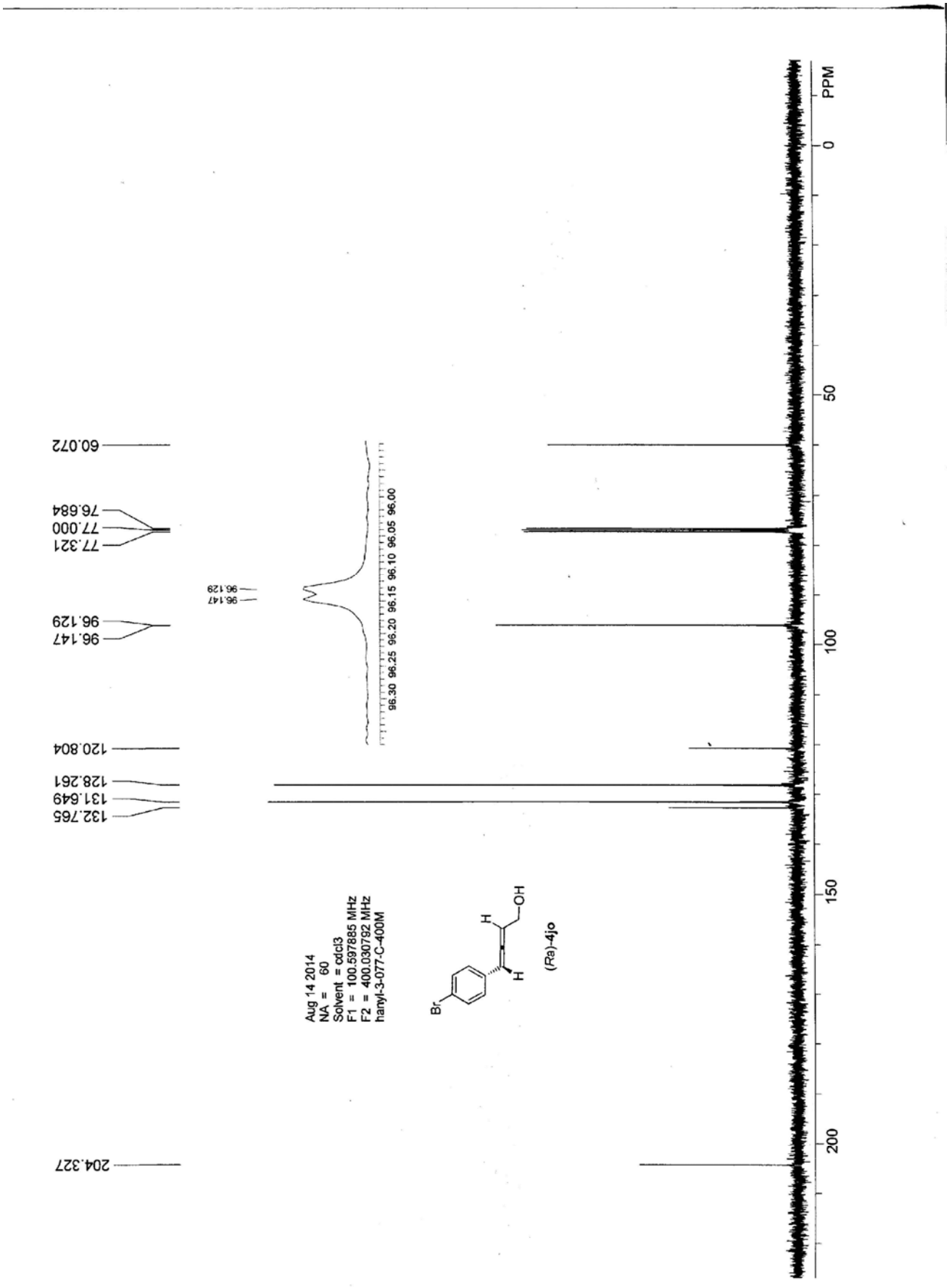
实验内容简介：
AS-H column, n-hexane/iPrOH = 98/2, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.740	275755.500	4757725.000	50.2984
2		18.495	214834.594	4701277.500	49.7016
总计			490590.094	9459002.500	100.0000

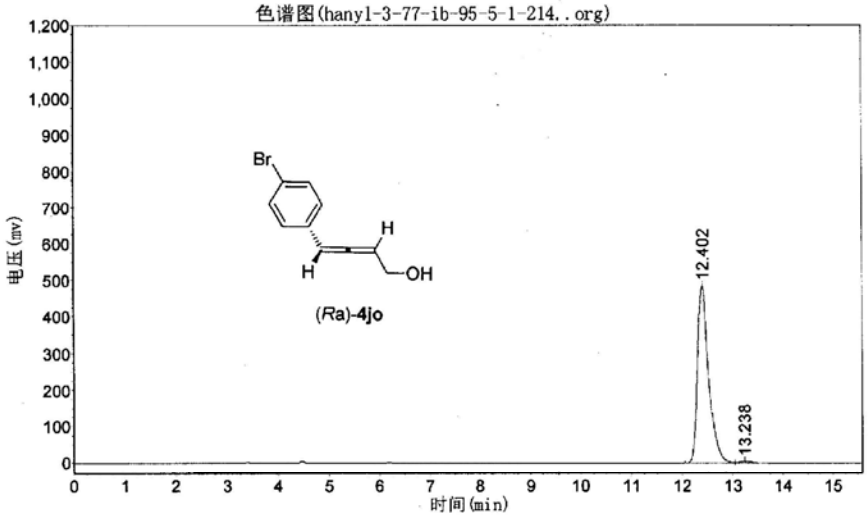

Aug 14 2014
NA = 8
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
hanyl-3-077-H-400M
(Ra)-4jo
OH
H
H
Br
PPM
0
1
2
3
4
5
6
7
1.09
2.36
0.98
1.00
2.21
2.14
-0.000
2.154
4.229
4.232
4.236
4.239
4.243
5.735
5.750
5.765
5.780
6.227
6.235
6.243
7.134
7.155
7.257
7.394
7.415


PPM
0
50
100
150
200
204.327
132.765
131.649
128.261
120.804
96.147
96.129
77.321
77.000
76.684
60.072
96.30 96.25 96.20 96.15 96.10 96.05 96.00
96.147
96.129
Aug 14 2014
NA = 60
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
hanyl-3-077-C-400M
Br
OH
H
H
(Ra)-4jo

hanyl-3-77-ib-95-5-1-214

实验时间：2014-08-14,17:33:33　　报告时间：2014-08-14,17:53:28
谱图文件:D:\zhuguangjiong\hyl\20140814\hanyl-3-77-ib-95-5-1-214..org

实验内容简介：



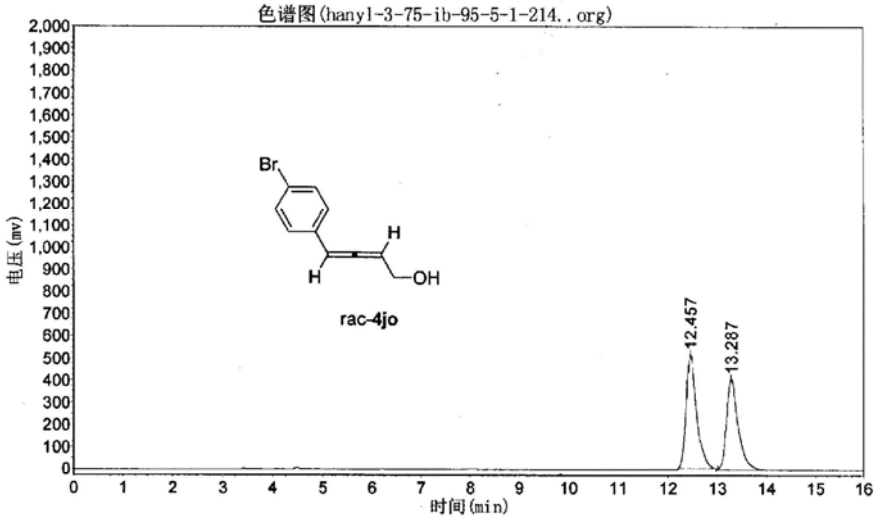
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.402	485290.969	7368597.000	98.7697
2		13.238	5448.435	91784.086	1.2303
总计			490739.404	7460381.086	100.0000

hanyl-3-75-rac-ib-95-5-1-214

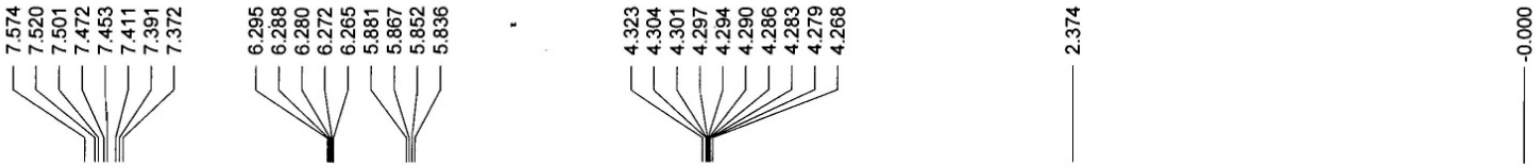
实验时间：2014-08-14,17:00:49　　报告时间：2014-08-14,17:24:32
谱图文件:D:\zhuguangjiong\hyl\20140814\hanyl-3-75-ib-95-5-1-214..org

实验内容简介：

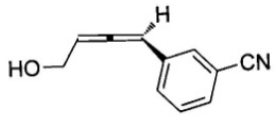


分析结果表

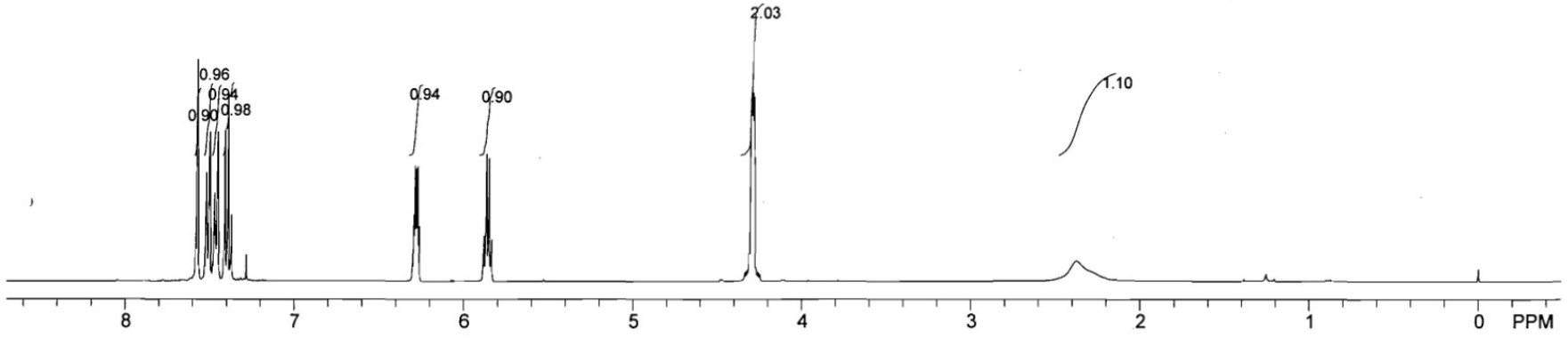
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.457	515468.625	7674716.000	53.3991
2		13.287	411075.063	6697640.500	46.6009
总计			926543.688	14372356.500	100.0000

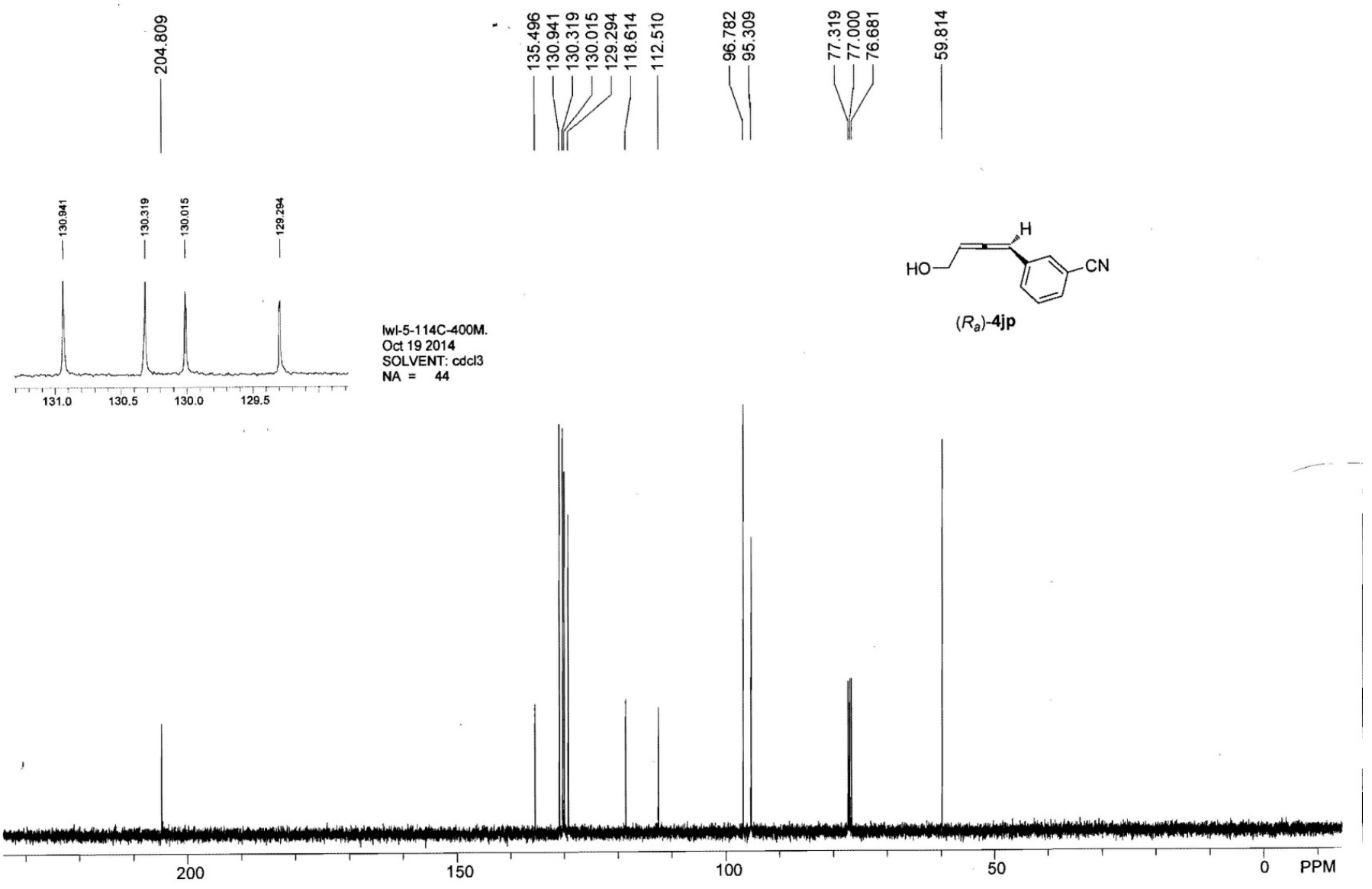


lwl-5-114H-400M
Oct 19 2014
SOLVENT: CDCl3
NA = 8



(R_a)-**4jp**

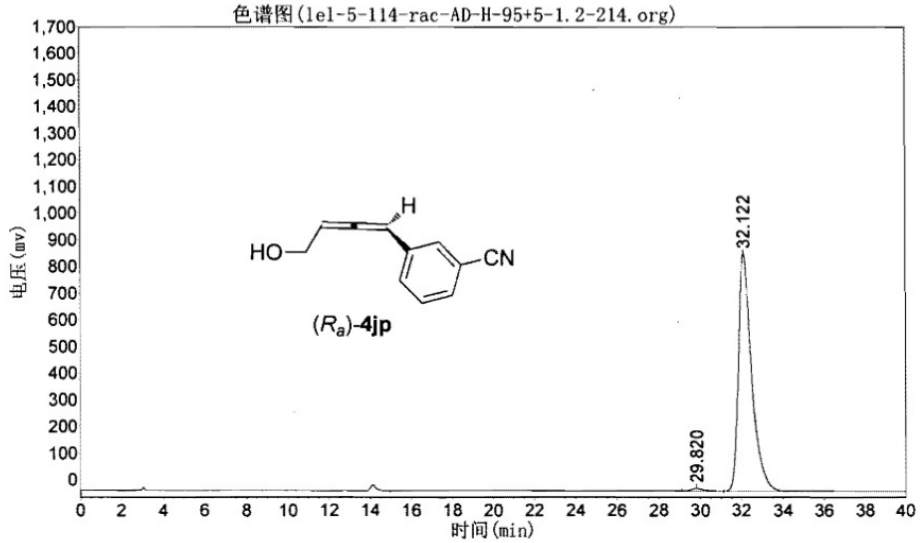



204.809
135.496
130.941
130.319
130.015
129.294
118.614
112.510
96.782
95.309
77.319
77.000
76.681
59.814
130.941
130.319
130.015
129.294
131.0
130.5
130.0
129.5
lwl-5-114C-400M.
Oct 19 2014
SOLVENT: cdcl3
NA = 44
HO
H
CN
(Ra)-4jp
200
150
100
50
0
PPM

lwl-5-114

实验时间：2014-10-20,13:50:31　　报告时间：2014-10-20,13:52:10
谱图文件:F:\slf\linweilong\2013-10-20\lwl-5-114\lel-5-114-rac-AD-H-95+5-1.2-214.org

实验内容简介：
AD-H　　95:5
214nm　　1.2ml/min



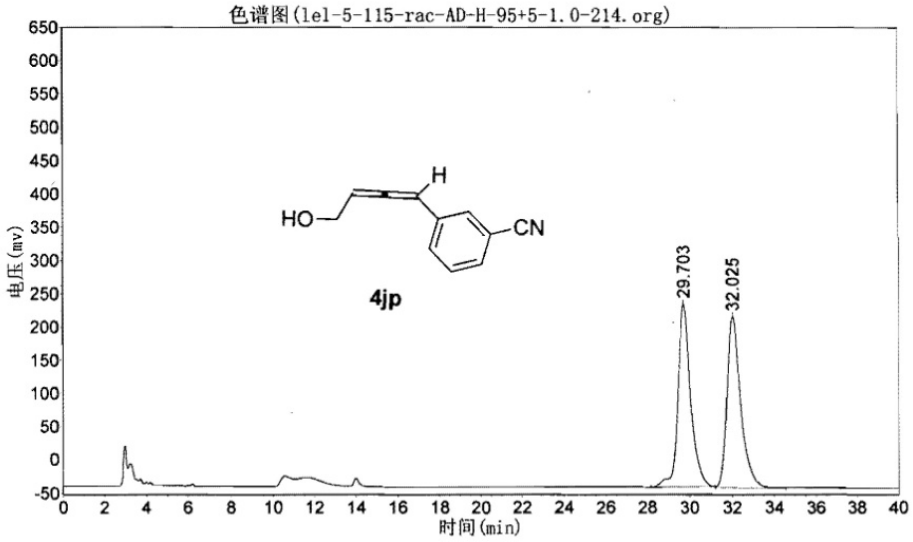
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.820	9710.256	396260.688	0.9875
2		32.122	889913.688	39729392.000	99.0125
总计			899623.943	40125652.688	100.0000

lwl-5-115-arc

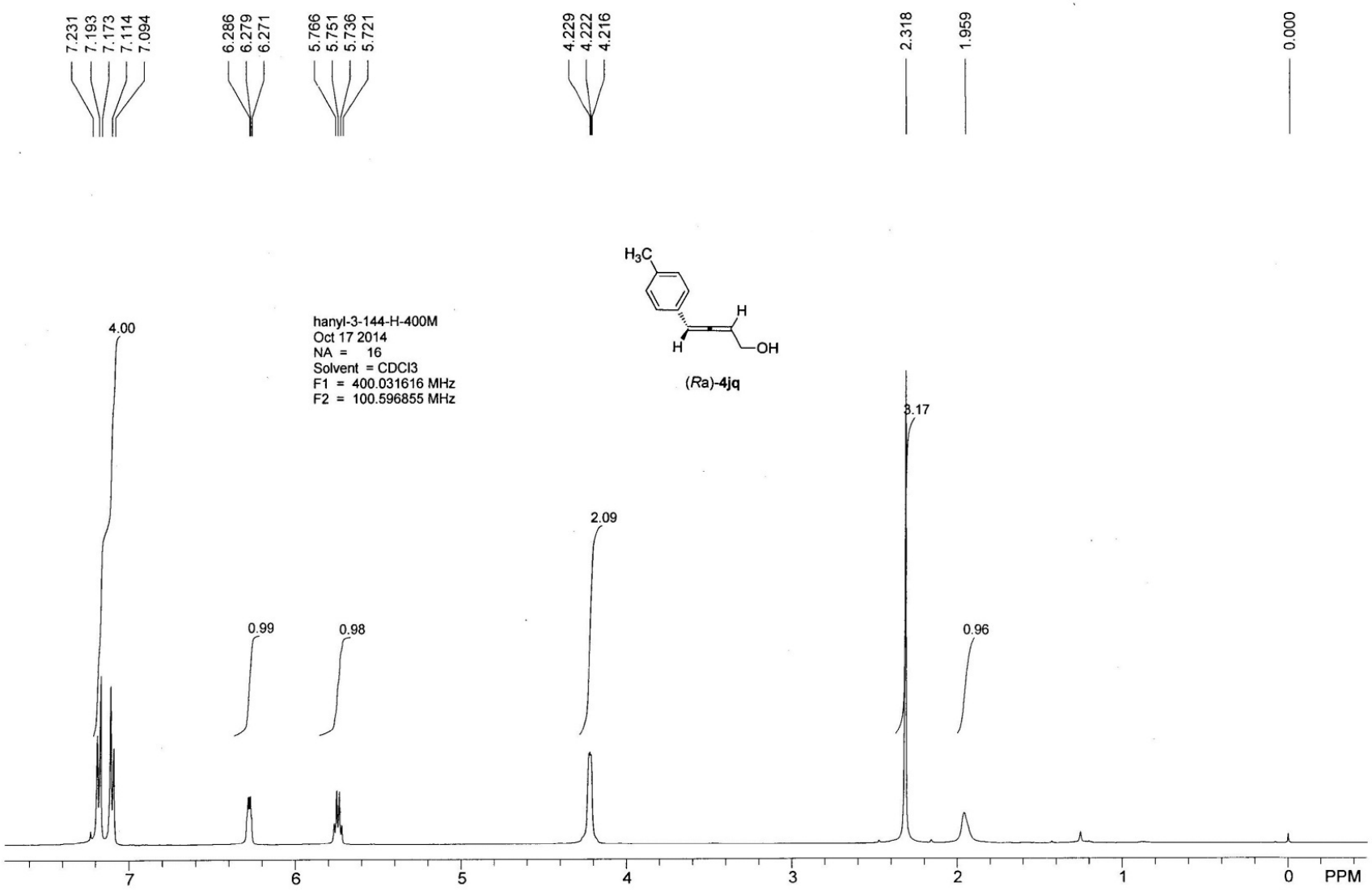
实验时间：2014-10-20,13:09:14　　报告时间：2014-10-20,13:15:10
谱图文件:F:\slf\linweilong\2013-10-20\lwl-5-115-rac\新建文件夹 (3)\lel-5-115-rac-AD-H-95+5-1.0-214.org

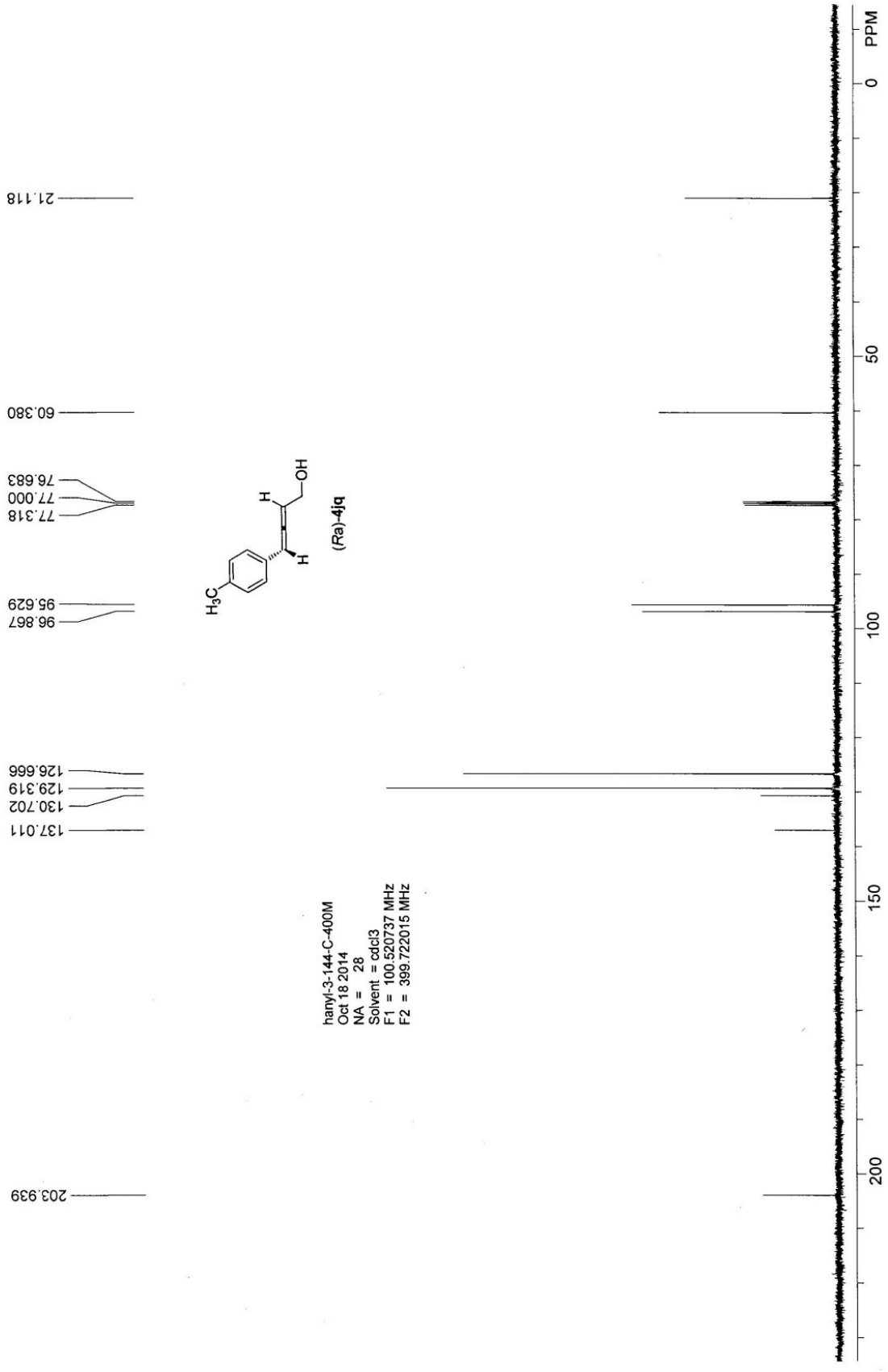
实验内容简介：
AD-H　　95:5
214nm　　1.2ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.703	275233.313	11437088.000	50.0253
2		32.025	258050.297	11425505.000	49.9747
总计			533283.609	22862593.000	100.0000


7.231
7.193
7.173
7.114
7.094
6.286
6.279
6.271
5.766
5.751
5.736
5.721
4.229
4.222
4.216
2.318
1.959
0.000
hanyl-3-144-H-400M
Oct 17 2014
NA = 16
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
H3C
H
H
OH
(Ra)-4jq
4.00
0.99
0.98
2.09
3.17
0.96
7
6
5
4
3
2
1
0
PPM


21.118
60.380
76.683
77.000
77.318
95.629
96.867
126.666
129.319
130.702
137.011
203.939
hanyl-3-144-C-400M
Oct 18 2014
NA = 28
Solvent = cdcl3
F1 = 100.520737 MHz
F2 = 399.722015 MHz
H3C
H
H
OH
(Ra)-4jq
PPM
0
50
100
150
200

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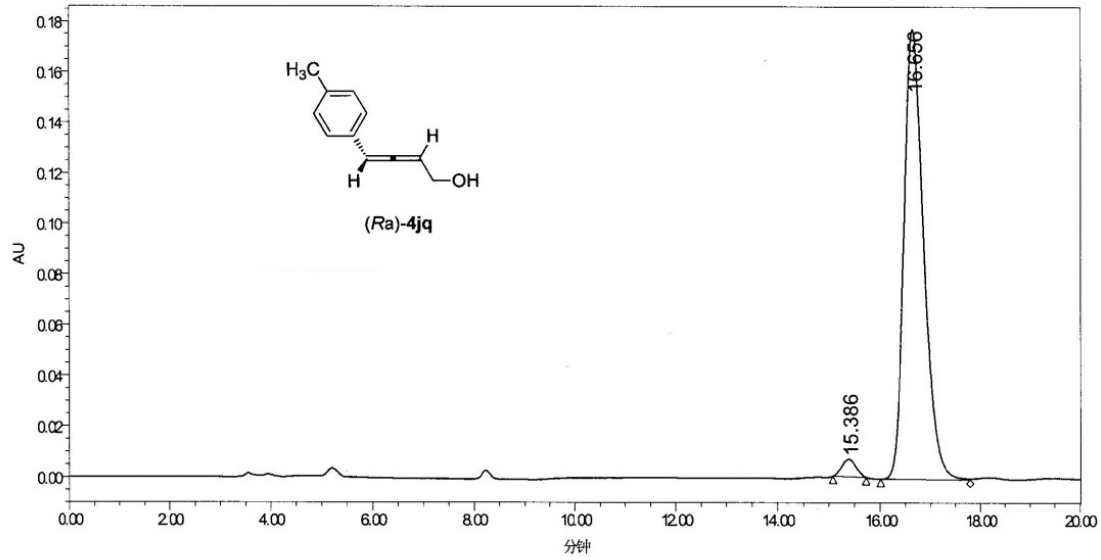
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hanyl-3-144-ay-h-95-5-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/10/18 17:26:35 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	13	Date Processed:	2014/10/18 18:08:01 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	60.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (碘sec)	%Area	Height (碘)	% Height
1	15.386	133335	2.70	6873	3.72
2	16.656	4809531	97.30	177947	96.28

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2014/10/18 18:09:06 PRC

中国科学院上海有机化学研究所

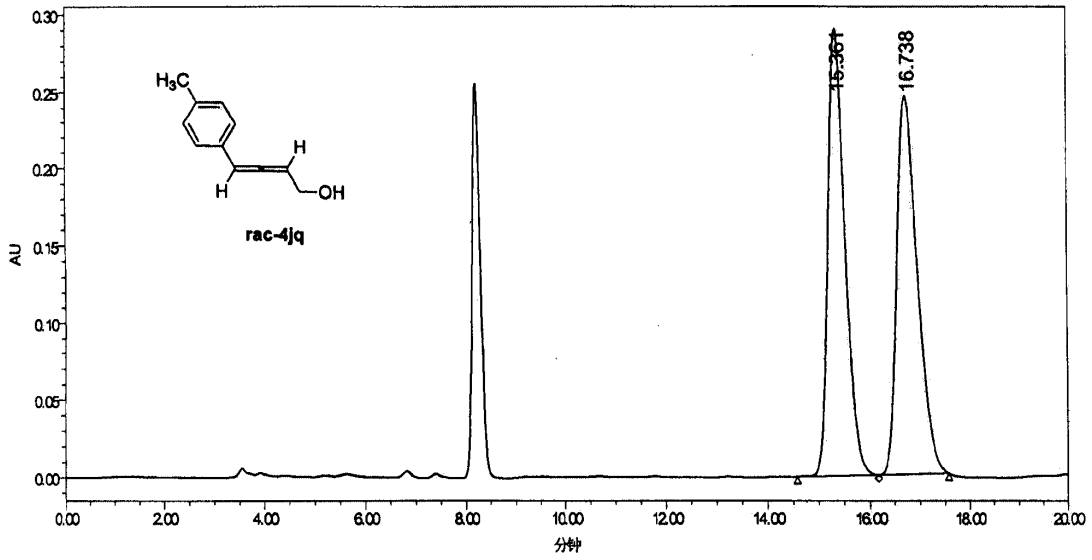
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hanyl-3-78-ay-h-95-5-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/10/18 17:46:58 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	14	Date Processed:	2014/10/18 18:07:37 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	20.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	

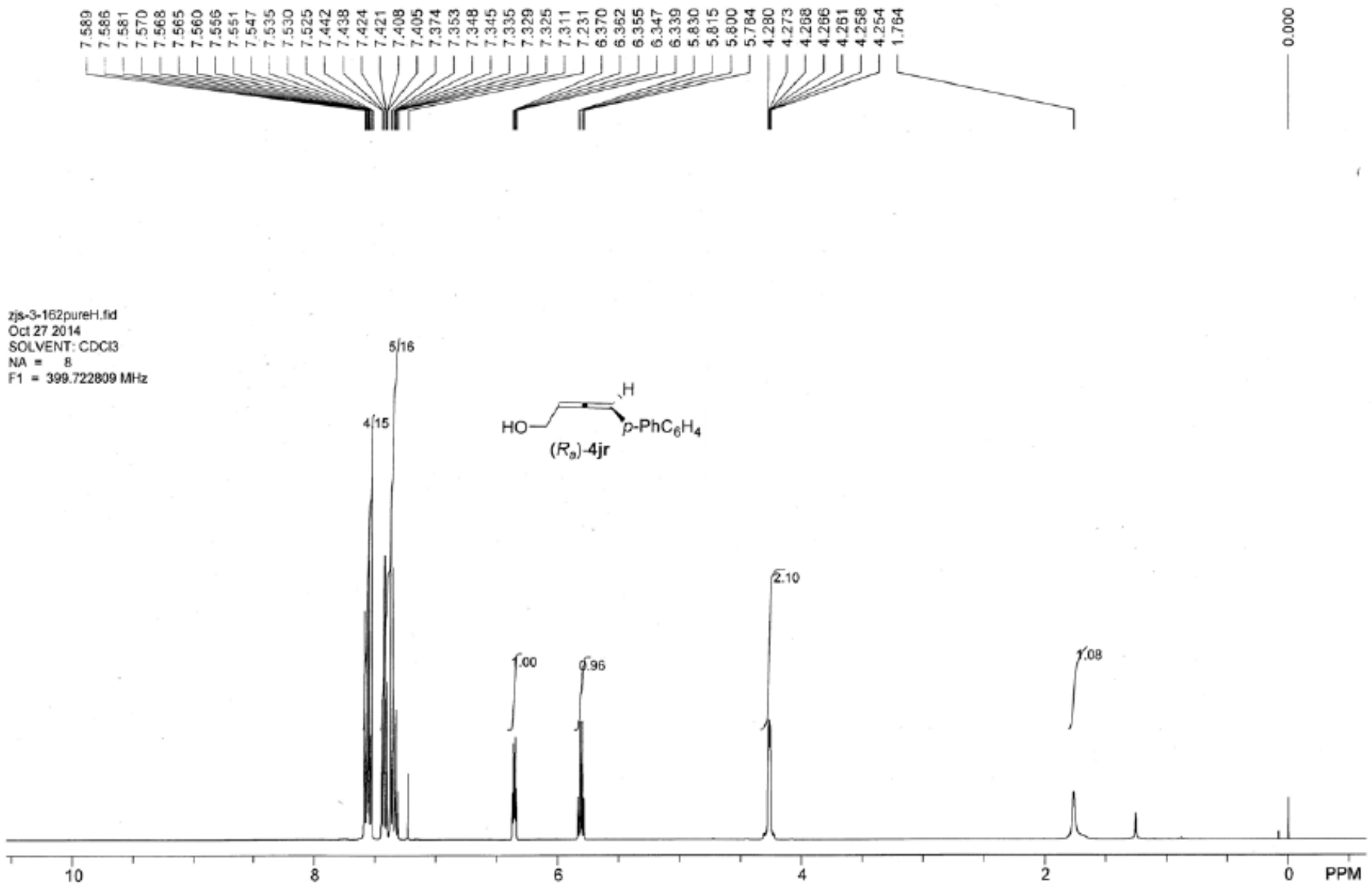


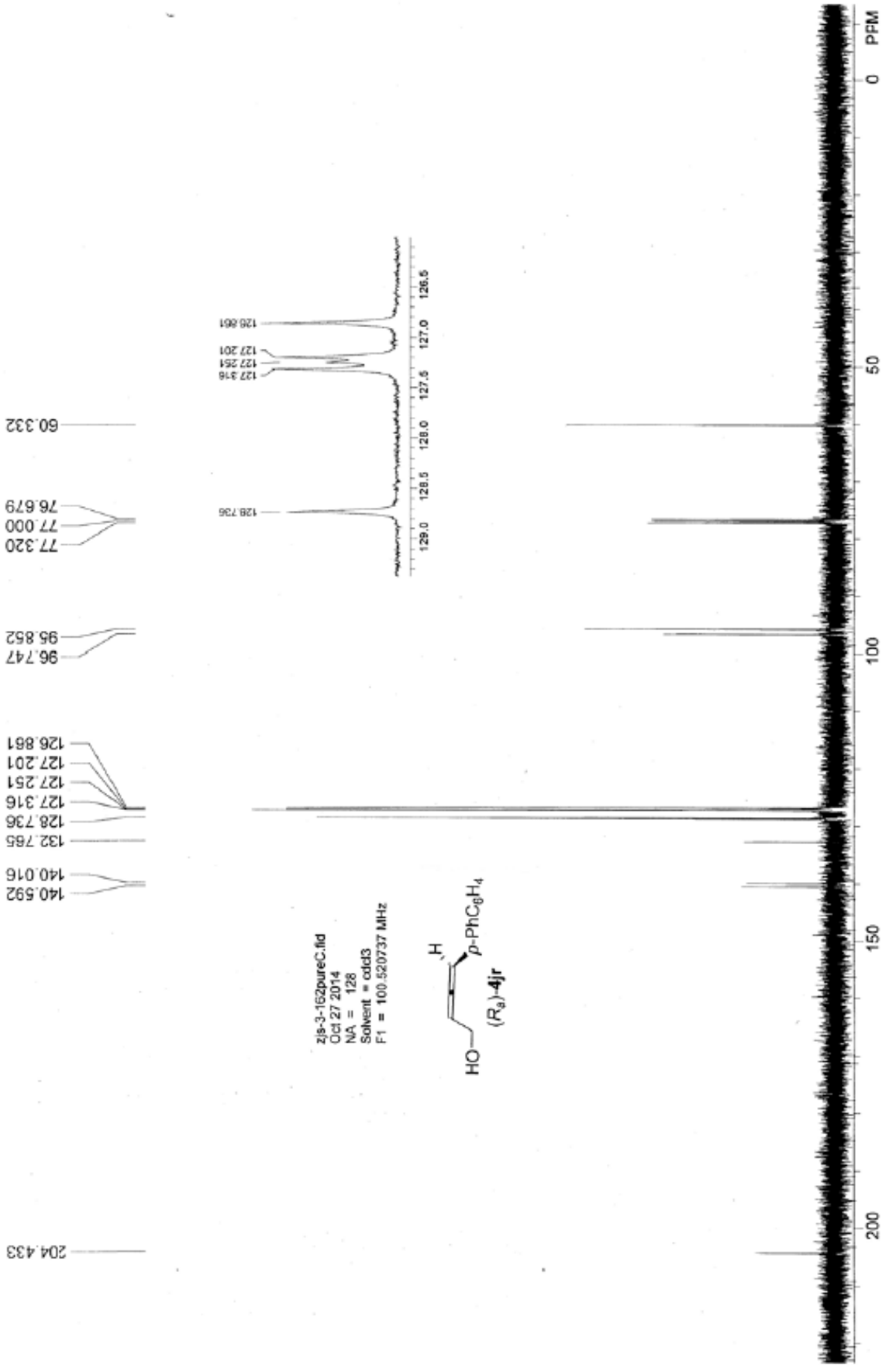
	RT (min)	Area (微伏*sec)	%Area	Height (微伏)	% Height
1	15.361	675791[illegible]	49.38	290180	54.13
2	16.738	692800[illegible]	50.62	245941	45.87

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2014/10/18 18:08:23 PRC

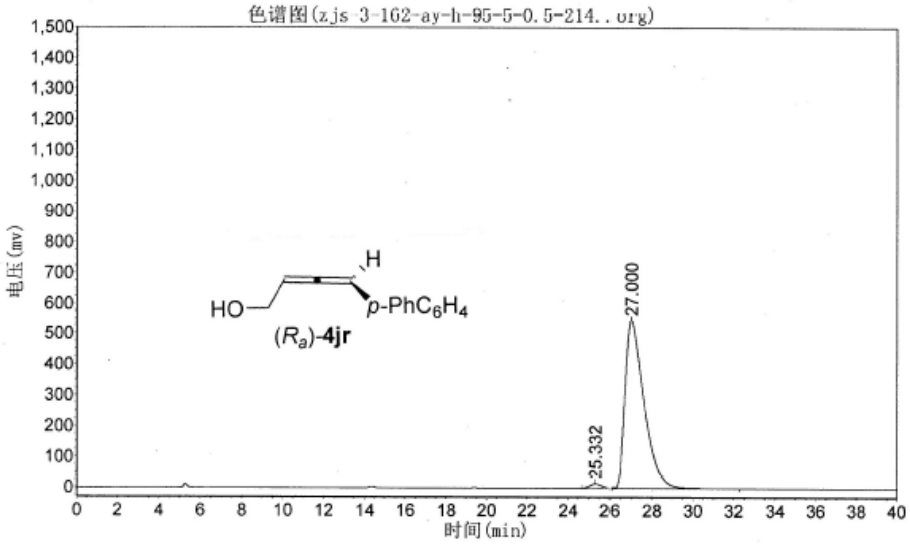

7.589
7.586
7.581
7.570
7.568
7.565
7.560
7.556
7.551
7.547
7.535
7.530
7.525
7.442
7.438
7.424
7.421
7.408
7.405
7.374
7.353
7.348
7.345
7.335
7.329
7.325
7.311
7.231
6.370
6.362
6.355
6.347
6.339
5.830
5.815
5.800
5.784
4.280
4.273
4.268
4.266
4.261
4.258
4.254
1.764
0.000
zjs-3-162pureH.fid
Oct 27 2014
SOLVENT: CDCl3
NA = 8
F1 = 399.722809 MHz
5.16
4.15
HO
H
p-PhC6H4
(Ra)-4jr
2.10
1.00
0.96
1.08
10
8
6
4
2
0
PPM


zjs-3-162pureC.fid
Oct 27 2014
NA = 128
Solvent = cdcl3
F1 = 100.520737 MHz
HO
H
p-PhC6H4
(Ra)-4jr
204.433
140.592
140.016
132.765
128.736
127.316
127.251
127.201
126.861
96.747
95.852
77.320
77.000
76.679
60.332
PPM

zjs-3-162-ay-h-95-5-0.5-214

实验时间：2014-10-29,17:31:08　　报告时间：2014-10-29,18:21:08
谱图文件:D:\zhuguangjiong\zjs\20141029\zjs-3-162-ay-h-95-5-0.5-214..org

实验内容简介：



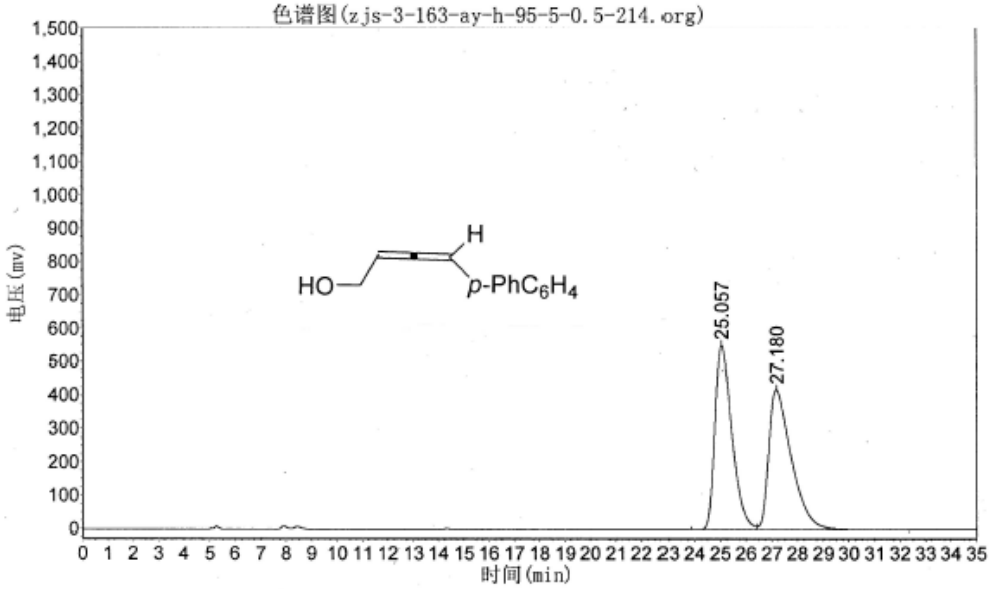
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		25.332	13727.231	531709.375	1.5029
2		27.000	544979.000	34847708.000	98.4971
总计			558706.231	35379417.375	100.0000

zjs-3-163-ay-h-95-5-0.5-214

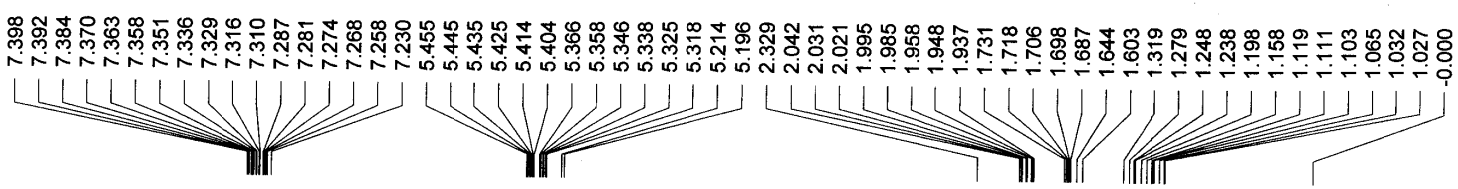
实验时间：2014-10-29,16:17:33　　报告时间：2014-10-29,17:18:58
谱图文件:D:\zhuguangjiong\zjs\20141029\zjs-3-163-ay-h-95-5-0.5-214.org

实验内容简介：



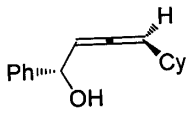
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		25.057	552455.313	25345842.000	49.6819
2		27.180	418491.469	25670442.000	50.3181
总计			970946.781	51016284.000	100.0000

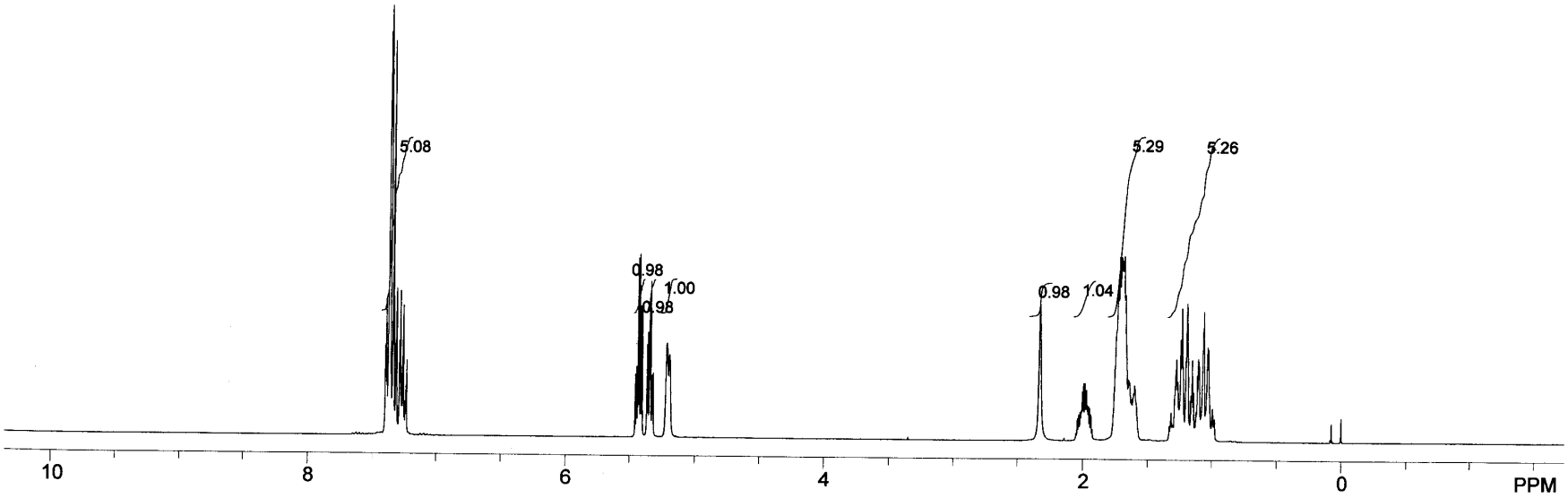


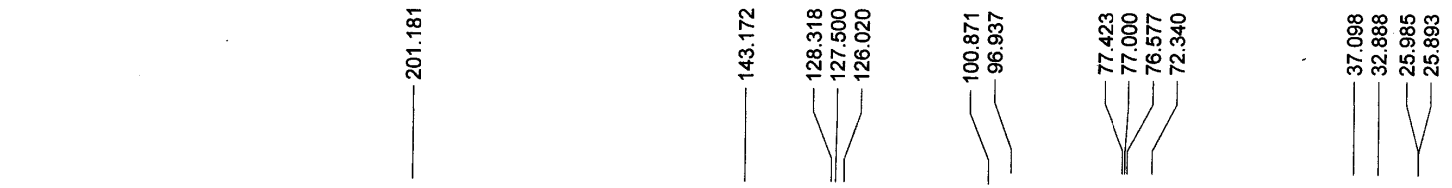
hx-10-194

2014-05-13 20:06:45.593
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



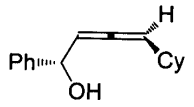
(S,R_a)-**4ke**



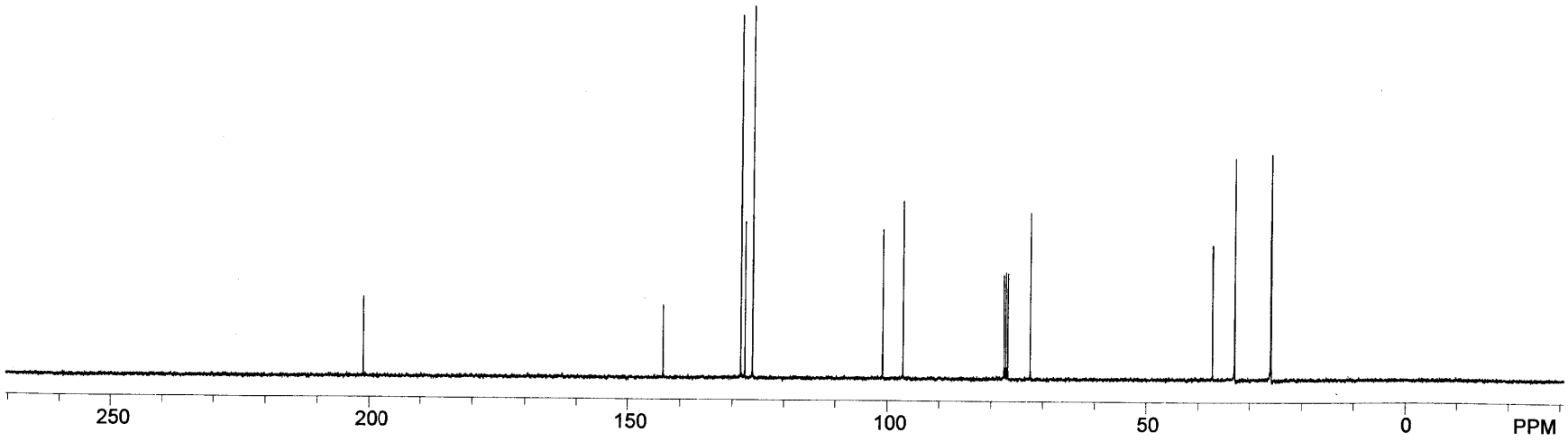


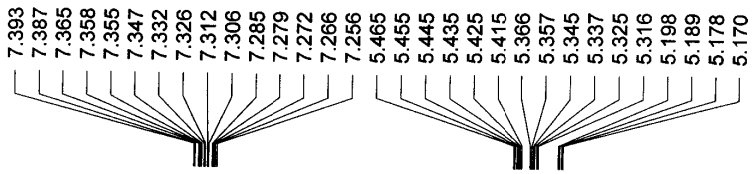
hx-10-194

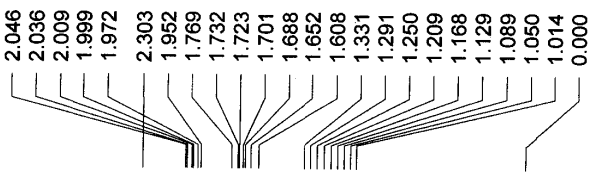
2014-05-13 20:16:01.203
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 134



(S,R_a)-**4ke**

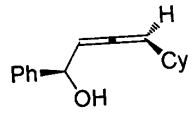




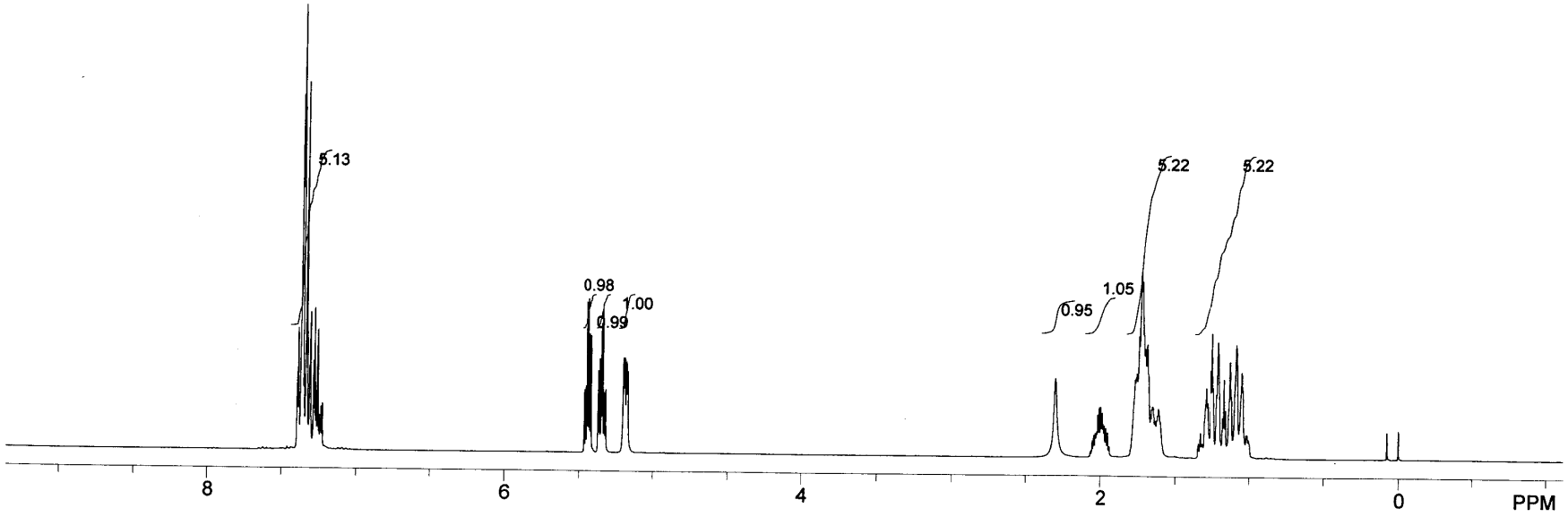


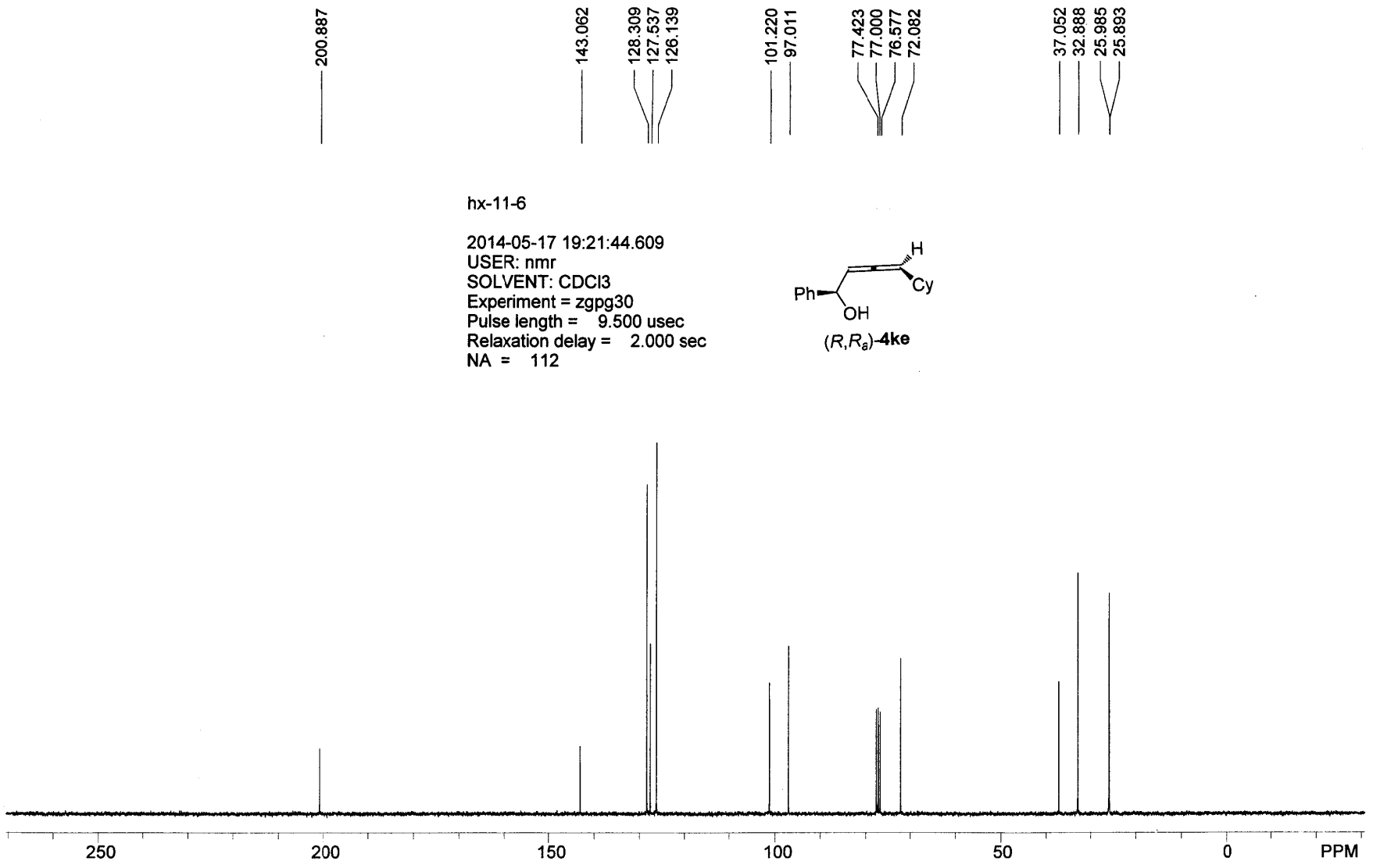
hx-11-6

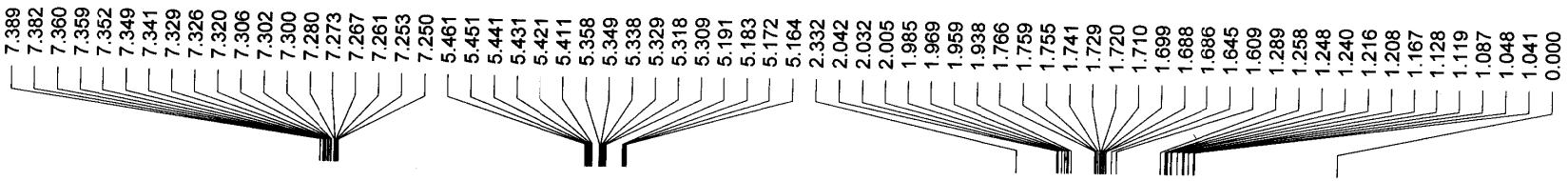
2014-05-17 19:13:46.234
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(*R*,*R*$_a$)-**4ke**

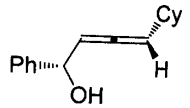



200.887
143.062
128.309
127.537
126.139
101.220
97.011
77.423
77.000
76.577
72.082
37.052
32.888
25.985
25.893
hx-11-6
2014-05-17 19:21:44.609
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 112
H
Cy
Ph
OH
(R,Ra)-4ke
250
200
150
100
50
0
PPM

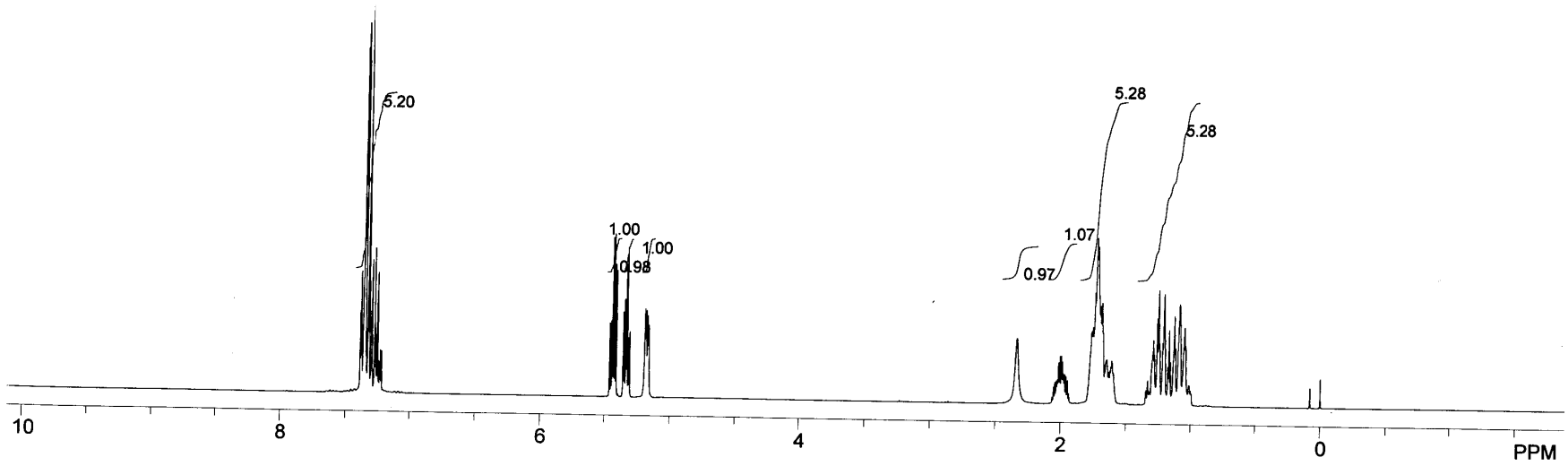


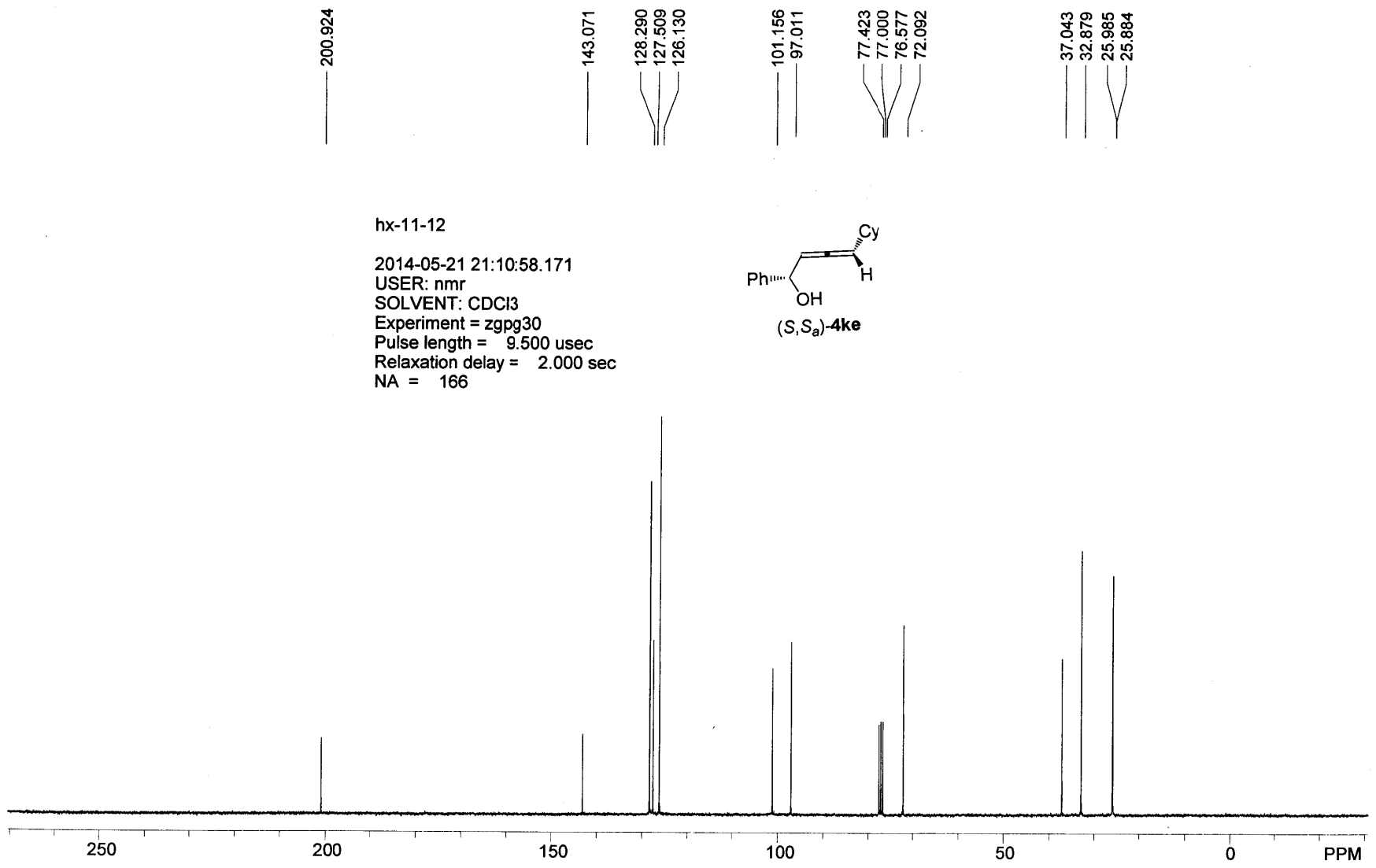
hx-11-12

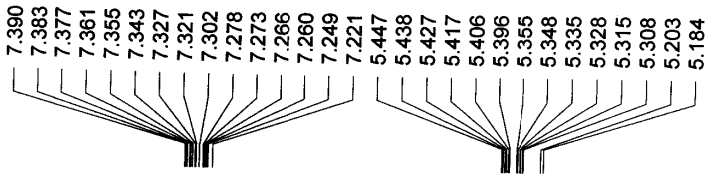
2014-05-21 20:56:28.890
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8

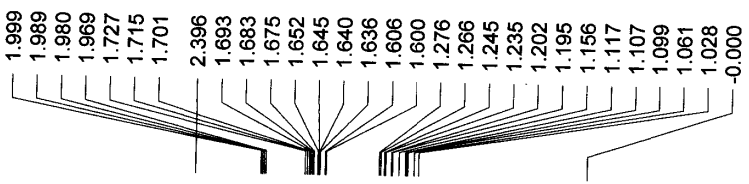


(S,S_a)-**4ke**



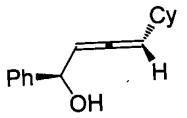

200.924
143.071
128.290
127.509
126.130
101.156
97.011
77.423
77.000
76.577
72.092
37.043
32.879
25.985
25.884
hx-11-12
2014-05-21 21:10:58.171
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 166
Cy
H
Ph
OH
(S,Sa)-4ke
250
200
150
100
50
0
PPM



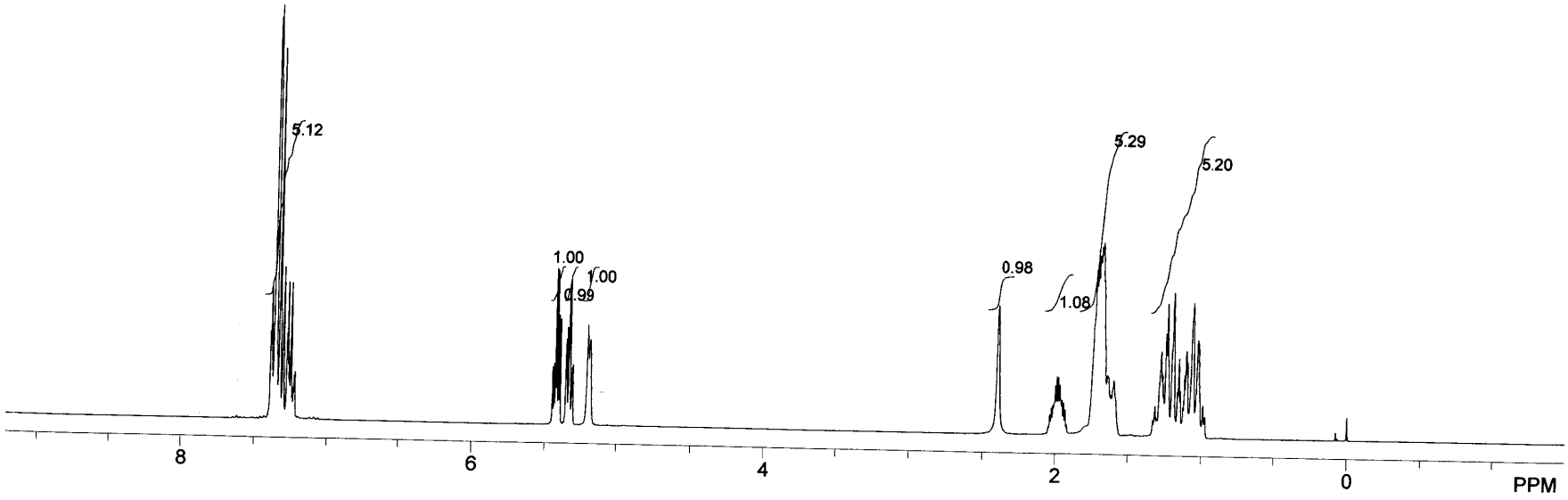


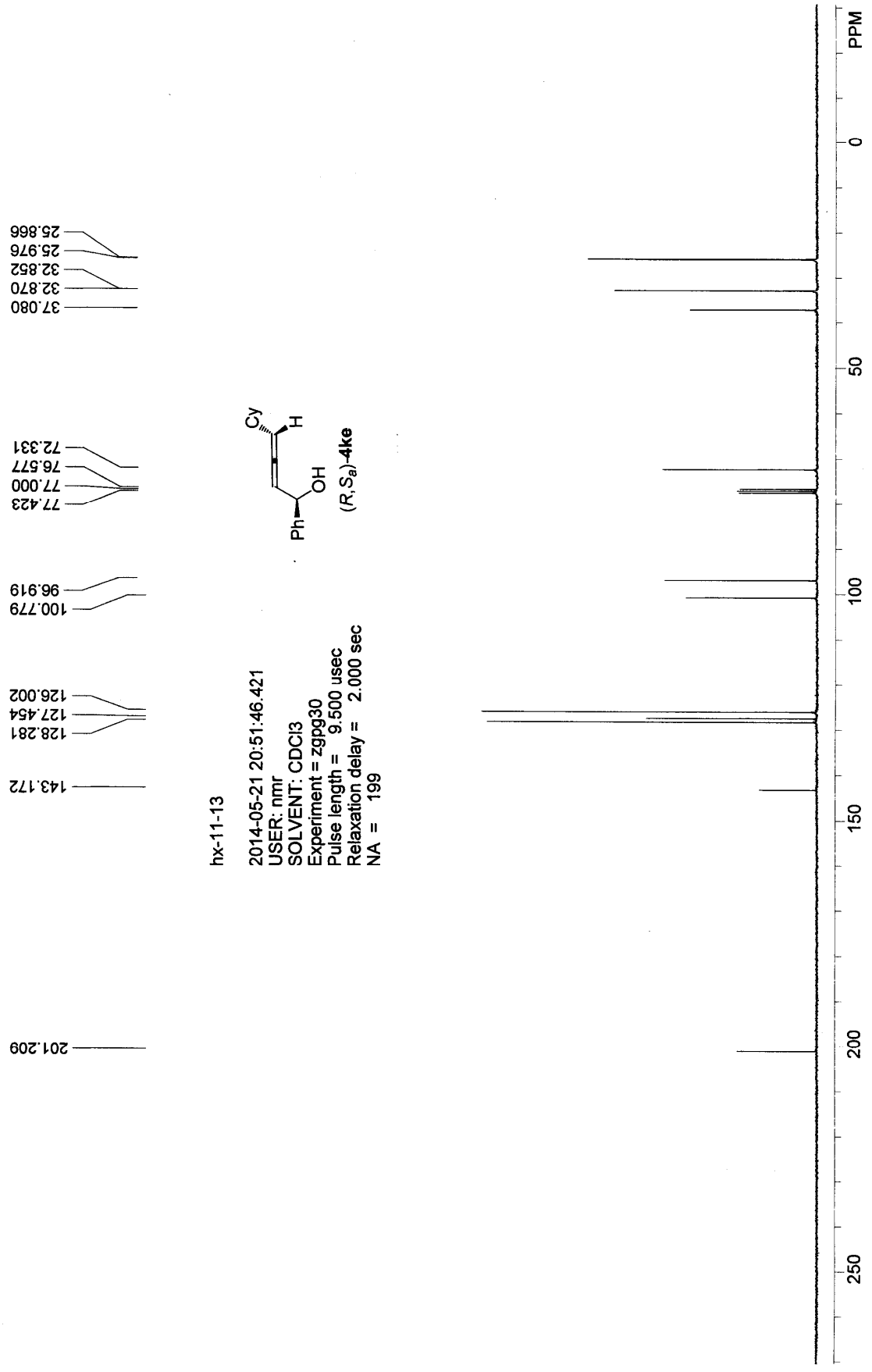
hx-11-13

2014-05-21 20:37:23.875
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R,S_a)-**4ke**




hx-11-13
2014-05-21 20:51:46.421
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 199
Cy
H
OH
Ph
(R,Sa)-4ke
201.209
143.172
128.281
127.454
126.002
100.779
96.919
77.423
77.000
76.577
72.331
37.080
32.870
32.852
25.976
25.866
PPM
250
200
150
100
50
0

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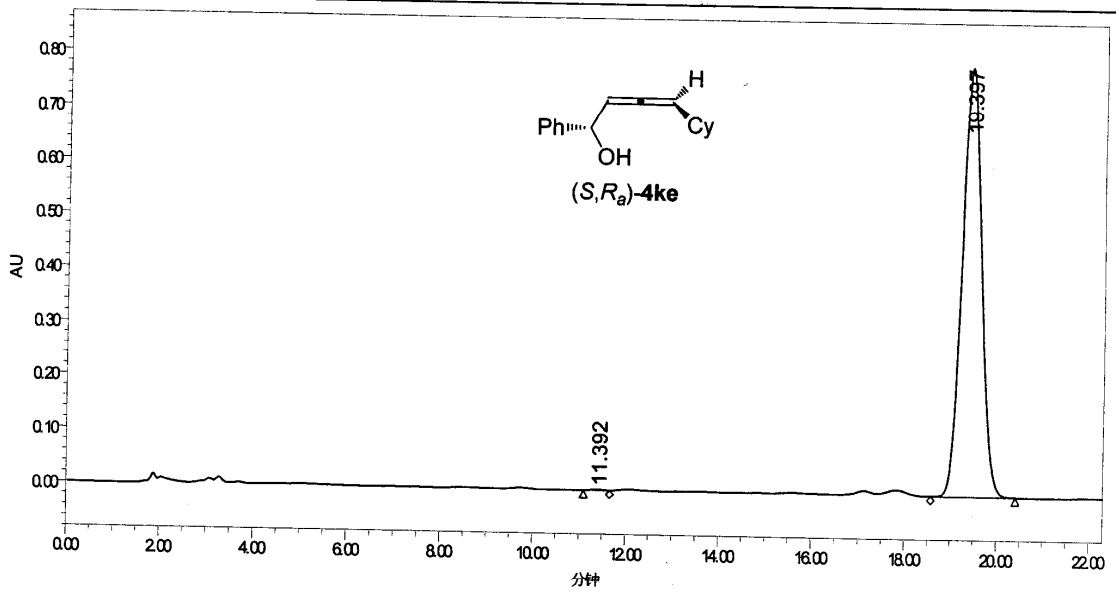
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-10-194-cd-98-2-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/5/20 18:49:45 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	21	Date Processed:	2014/5/20 19:17:16 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	25.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (磷sec)	%Area	Height (磷	% Height
1	11.392	35457	0.15	2126	0.27
2	19.397	23076393	99.85	792170	99.73

Report Method: Individual Report HP

Page: 1 (共计 1)

Printed: 2014/5/22 8:58:51 PRC

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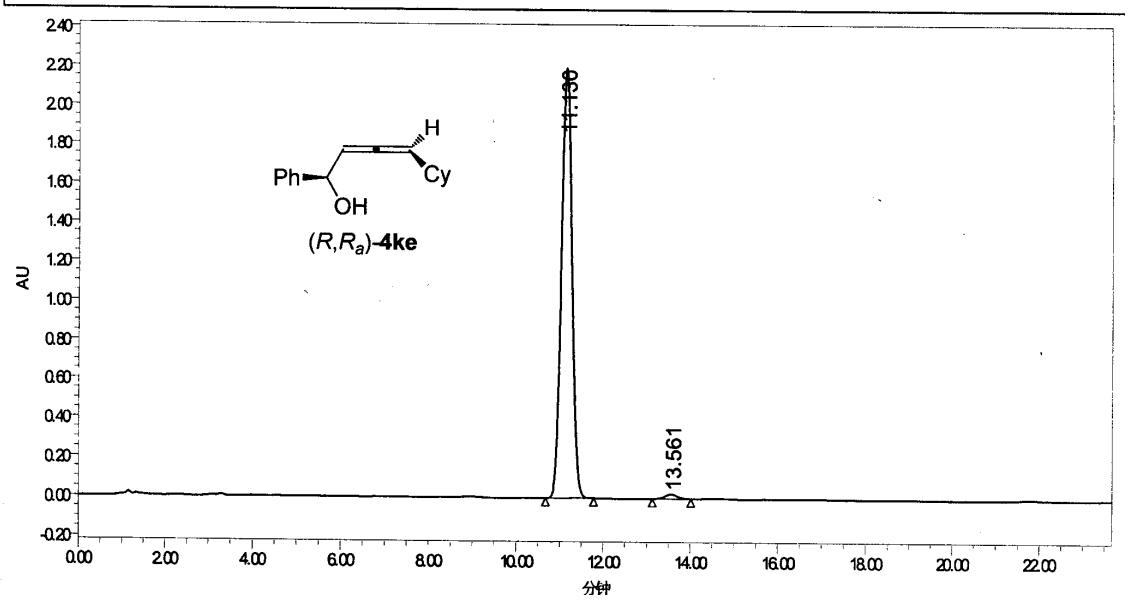
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-11-6-cd-98-2-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/5/20 17:58:43 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	19	Date Processed:	2014/5/20 19:16:41 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	25.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (磷sec)	%Area	Height (磷)	% Height
1	11.130	37093367	98.83	2198088	99.01
2	13.561	437847	1.17	22029	0.99

Report Method: Individual Report HP

Page: 1 (共计 1)

Printed: 2014/5/22 9:00:29 PRC

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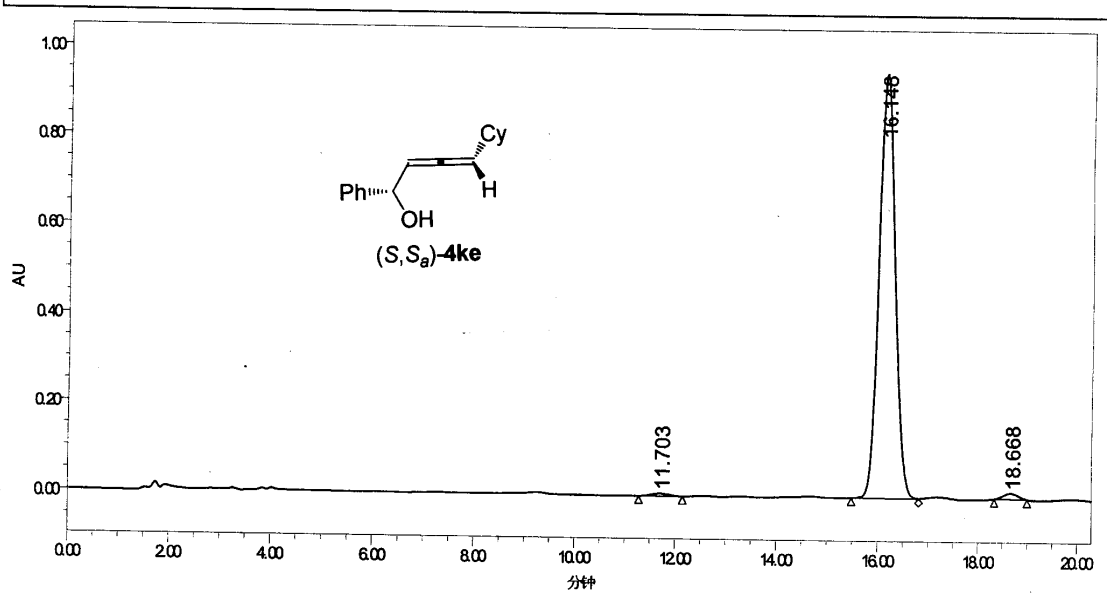
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-11-12-cd-98-2-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/5/20 16:48:57 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	14	Date Processed:	2014/5/20 19:15:23 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (微sec)	%Area	Height (微)	% Height
1	11.703	137431	0.64	5895	0.61
2	16.148	21043705	98.07	951738	98.04
3	18.668	277025	1.29	13107	1.35

Report Method: Individual Report HP

Page: 1 (共计 1)

Printed: 2014/5/22 9:01:03 PRC

中国科学院上海有机化学研究所

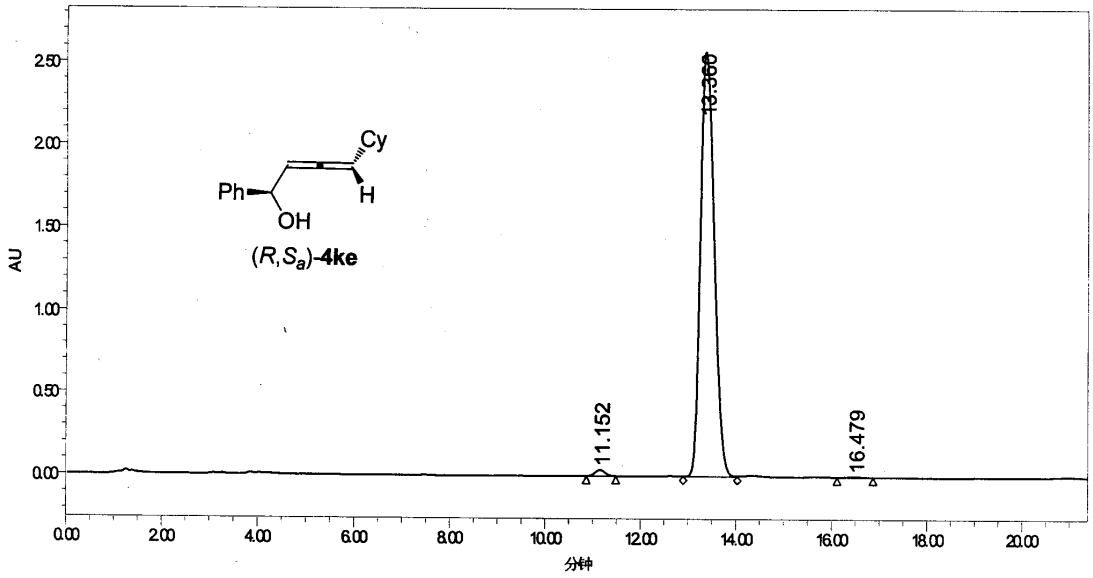
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-11-13-cd-98-2-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/5/20 17:12:38 CST
Vial:	1	Acq. Method:	zg98
Injection #:	16	Date Processed:	2014/5/20 19:15:01 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (微sec)	%Area	Height (微)	% Height
1	11.152	517227	1.00	35169	1.35
2	13.360	51412050	98.91	2572866	98.56
3	16.479	48326	0.09	2299	0.09

Report Method: Individual Report HP

Page: 1 (共计 1)

Printed: 2014/5/22 9:01:25 PRC

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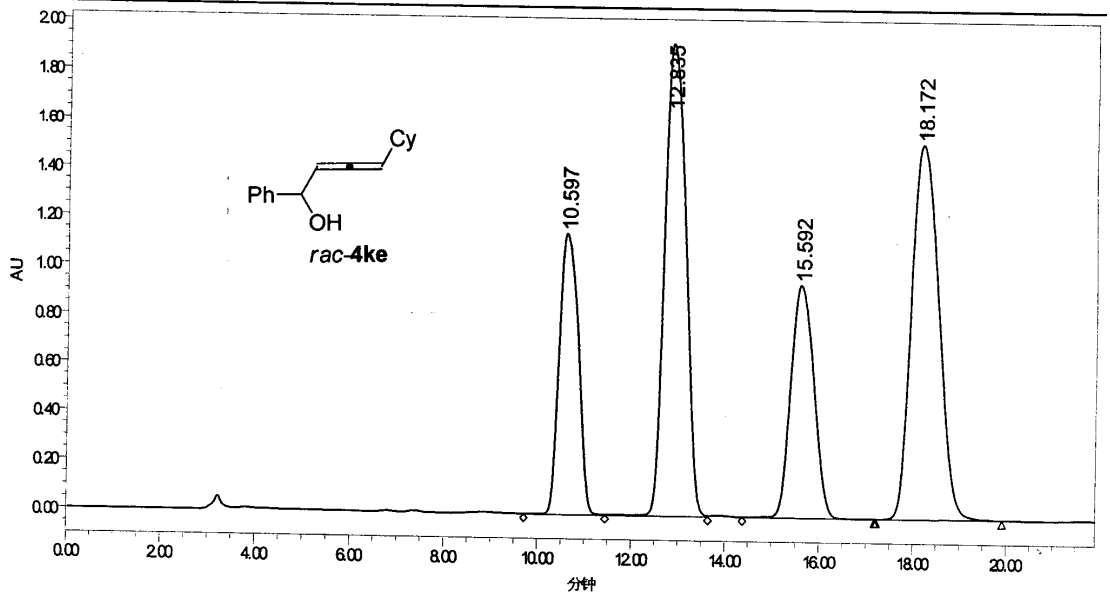
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-10-187-cd-98-2-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/5/20 16:23:28 CST
Vial:	1	Acq. Method:	zgj98
Injection #:	11	Date Processed:	2014/5/20 19:36:30 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	

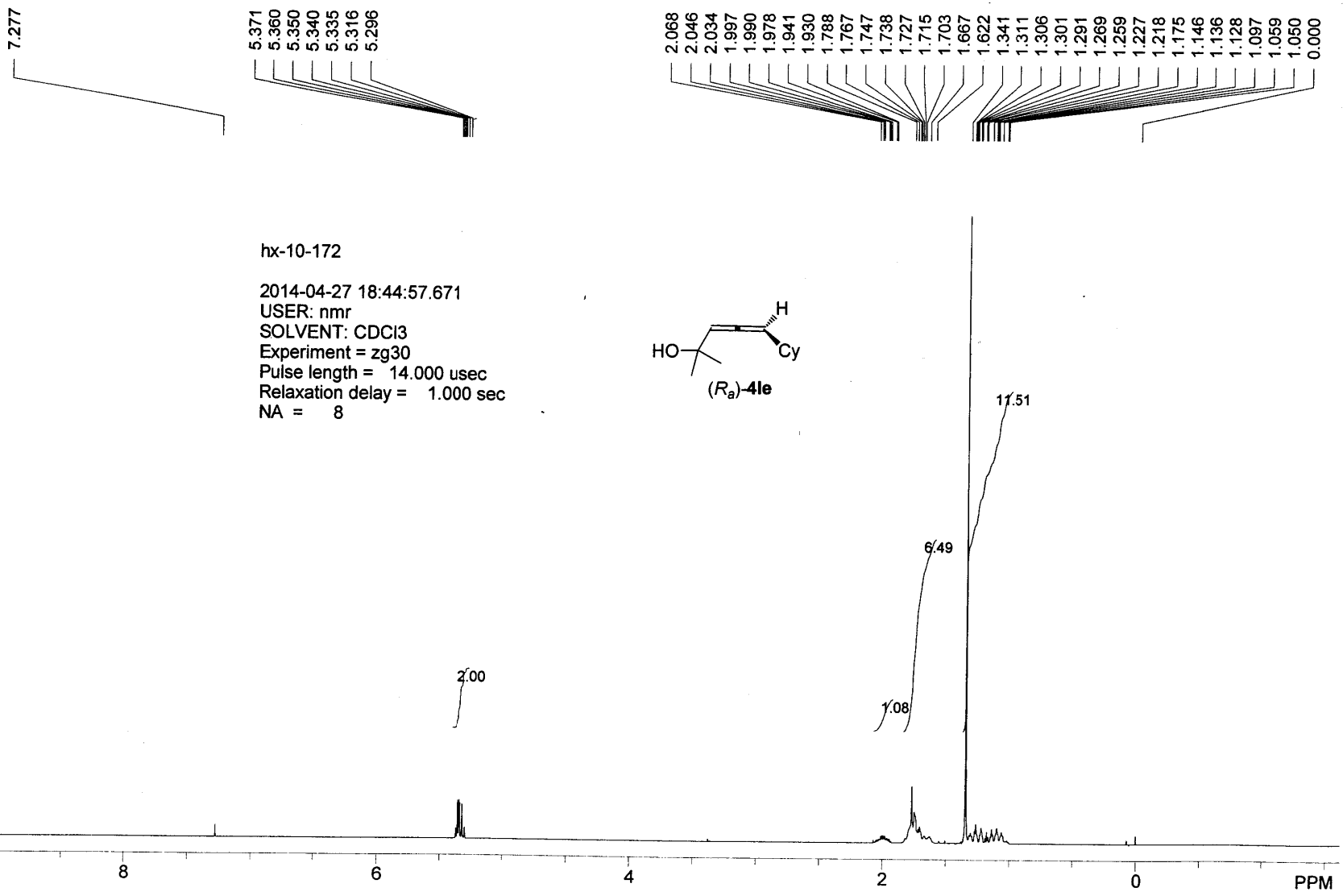


	RT (min)	Area (碘sec)	%Area	Height (碘)	%Height
1	10.597	32444451	17.31	1146374	20.64
2	12.836	60487130	32.28	1929820	34.74
3	15.592	32407686	17.29	950448	17.11
4	18.172	62086600	33.12	1528009	27.51

Report Method: Gradient Overlay Report ASC

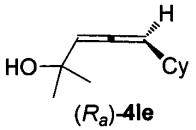
Page: 1 (共计 1)

Printed: 2014/5/20 19:37:17 PRC

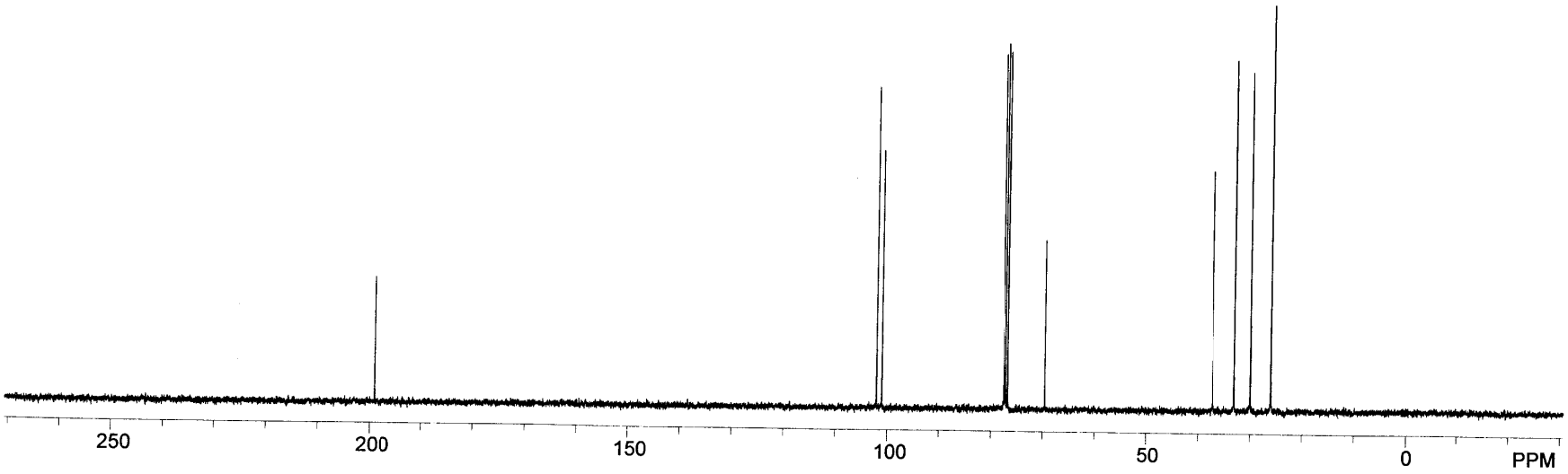

7.277
5.371
5.360
5.350
5.340
5.335
5.316
5.296
2.068
2.046
2.034
1.997
1.990
1.978
1.941
1.930
1.788
1.767
1.747
1.738
1.727
1.715
1.703
1.667
1.622
1.341
1.311
1.306
1.301
1.291
1.269
1.259
1.227
1.218
1.175
1.146
1.136
1.128
1.097
1.059
1.050
0.000
hx-10-172
2014-04-27 18:44:57.671
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
HO
H
Cy
(Ra)-4le
11.51
6.49
2.00
1.08
8
6
4
2
0
PPM

hx-10-172

2014-04-27 19:02:53.000
USER: nmr-
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 288



(R_a)-**4le**

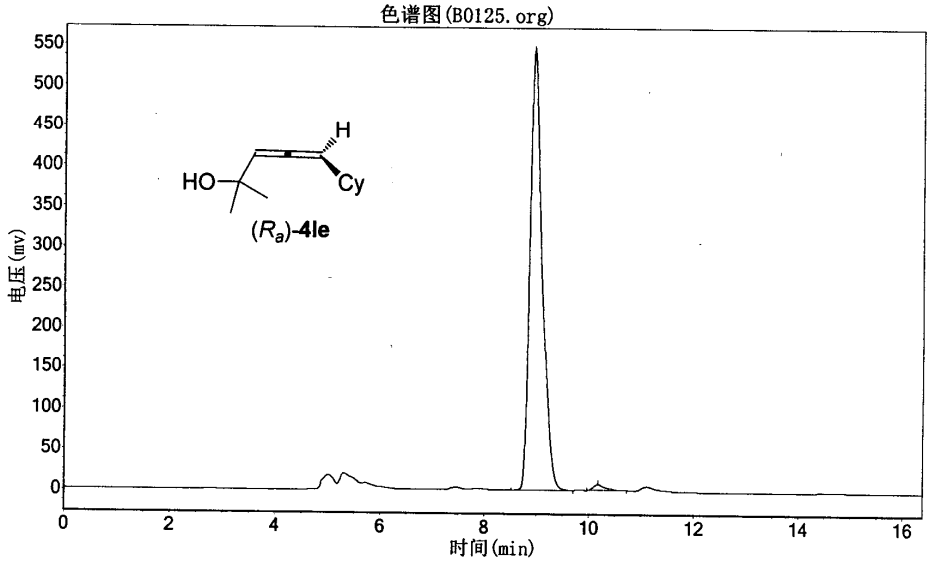


hx-10-172

实验时间：2014-04-27,18:49:12
谱图文件:D:\浙大智达\N2000\样品\B0125.org

实验者：hx
报告时间：2014-04-27,19:07:34
积分方法:面积归一法

实验内容简介：
AD-H column, n-hexane/iPrOH = 95/5, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		8.940	543544.625	8867722.000	98.6956
2		10.177	7370.146	117196.359	1.3044
总计			550914.771	8984918.359	100.0000

2014-04-27

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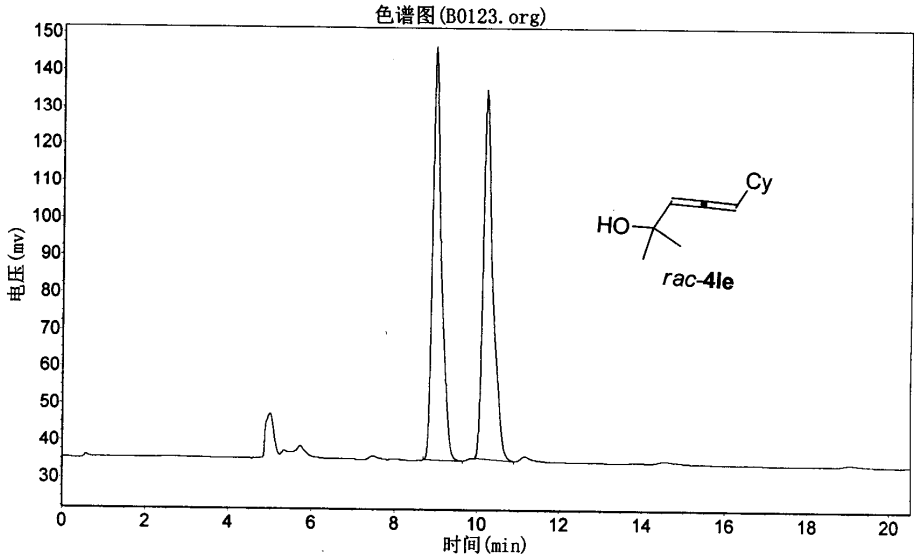
hx-10-174

实验时间：2014-04-27,18:21:09
谱图文件:D:\浙大智达\N2000\样品\B0123.org

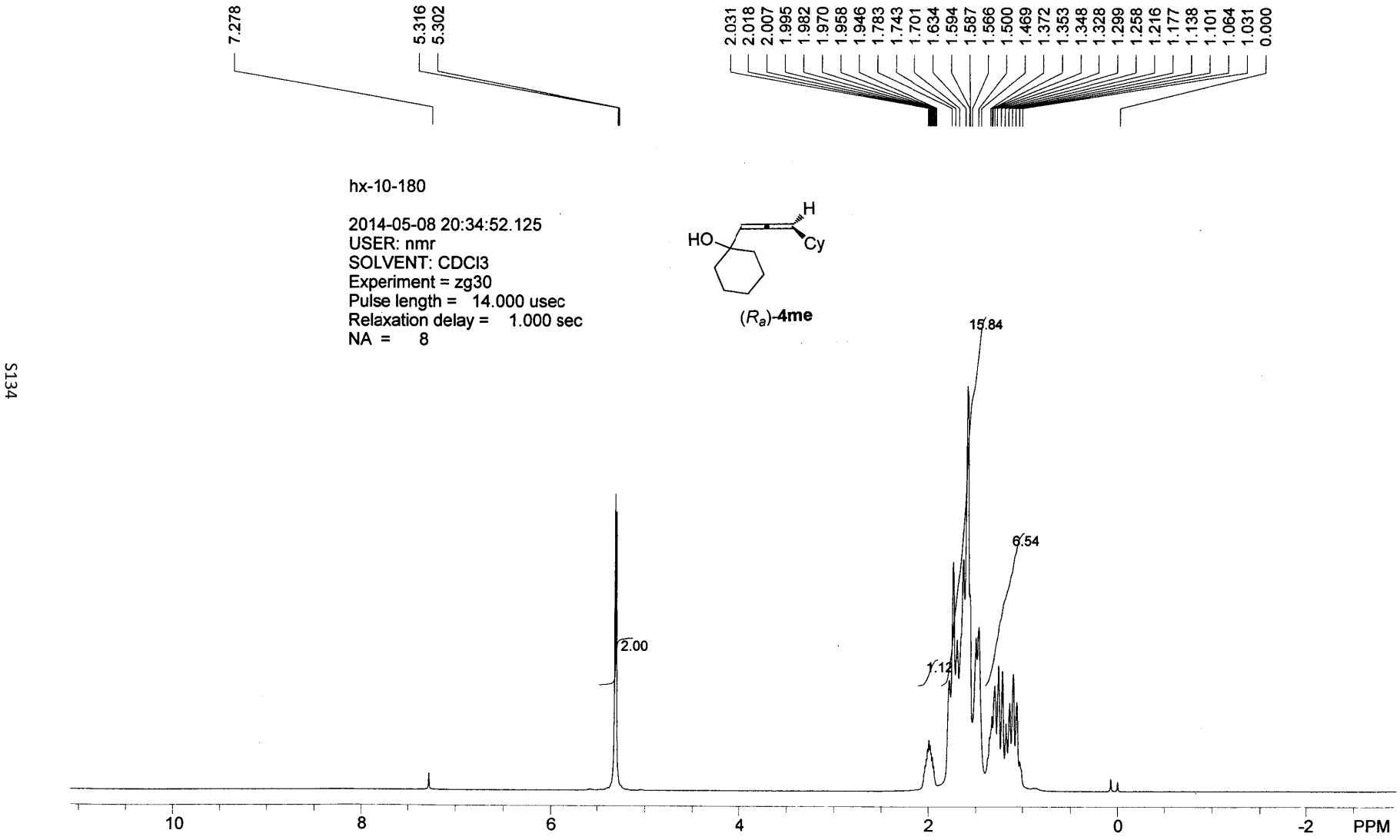
实验者：hx
报告时间：2014-04-27,18:43:15
积分方法:面积归一法

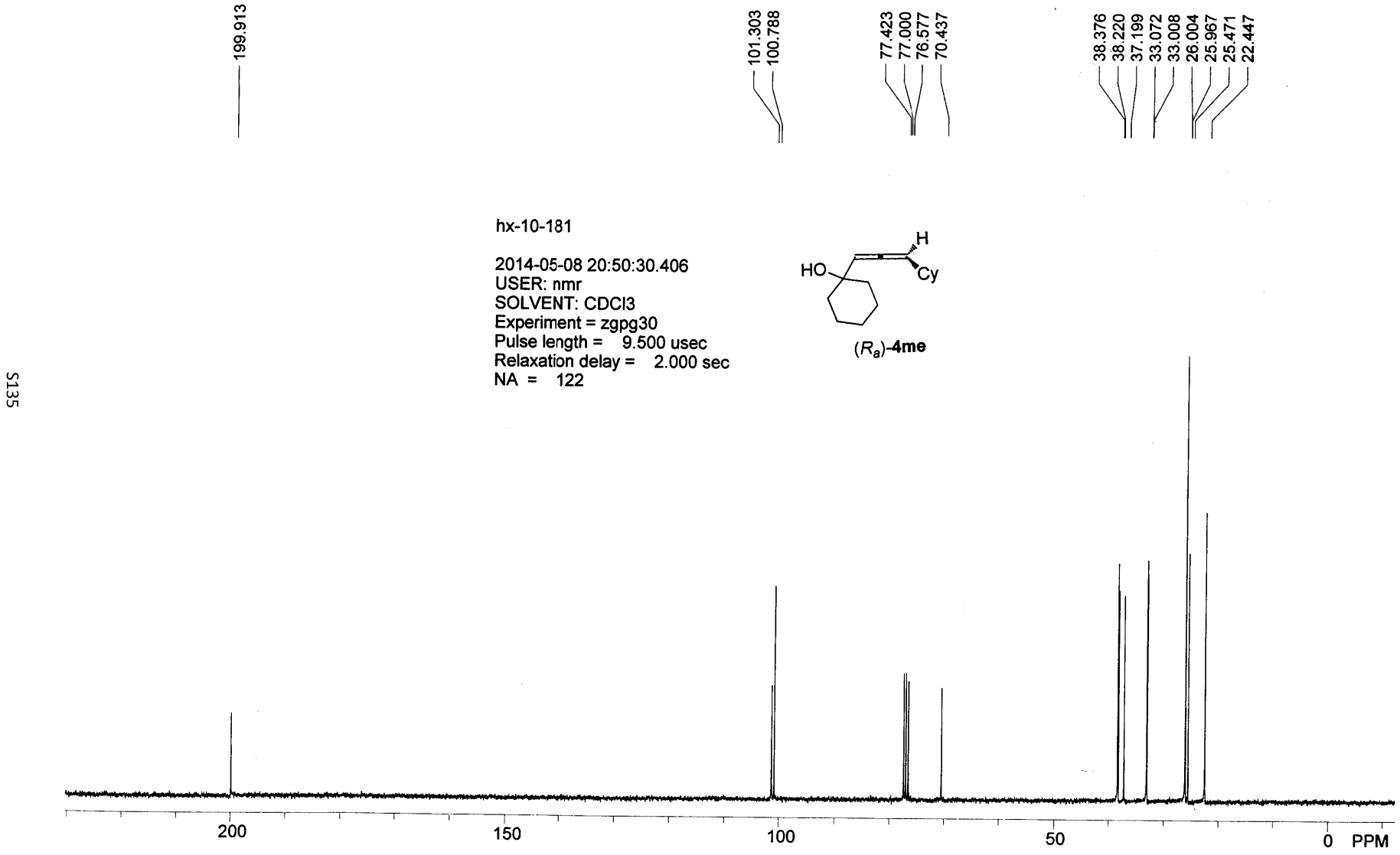
实验内容简介：
AD-H column, n-hexane/iPrOH = 95/5, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		8.975	110516.063	1642626.000	50.3349
2		10.213	98516.391	1620771.125	49.6651
总计			209032.453	3263397.125	100.0000


7.278
5.316
5.302
2.031
2.018
2.007
1.995
1.982
1.970
1.958
1.946
1.783
1.743
1.701
1.634
1.594
1.587
1.566
1.500
1.469
1.372
1.353
1.348
1.328
1.299
1.258
1.216
1.177
1.138
1.101
1.064
1.031
0.000
hx-10-180
2014-05-08 20:34:52.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
HO
H
Cy
(Ra)-4me
15.84
6.54
2.00
1.12
10
8
6
4
2
0
-2
PPM

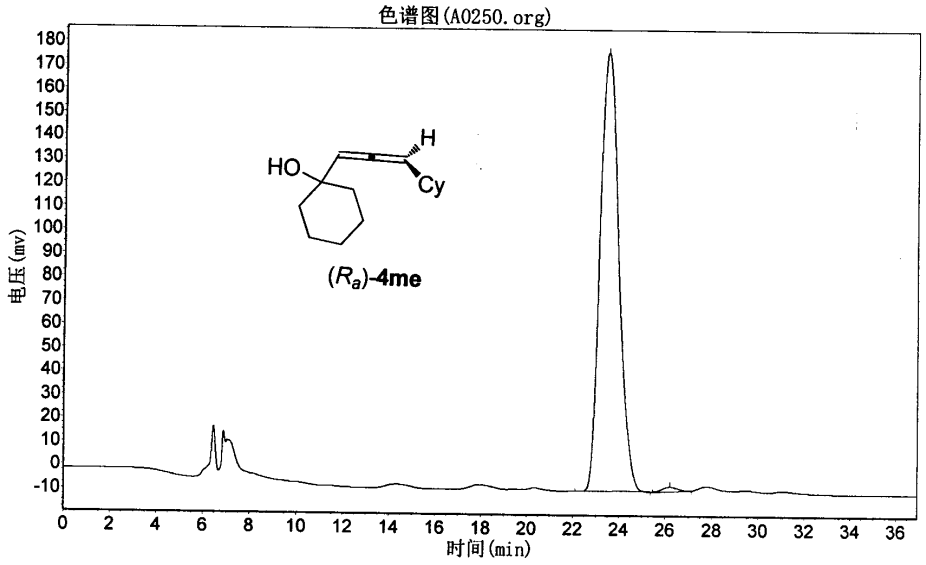

199.913
101.303
100.788
77.423
77.000
76.577
70.437
38.376
38.220
37.199
33.072
33.008
26.004
25.967
25.471
22.447
hx-10-181
2014-05-08 20:50:30.406
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 122
HO
H
Cy
(Ra)-4me
200
150
100
50
0 PPM

hx-10-180

实验单位：zju
实验时间：2014-05-10,12:39:41
谱图文件:D:\浙大智达\N2000\样品\A0250.org

实验者：hx
报告时间：2014-05-10,13:23:12
积分方法:面积归一法

实验内容简介：
AD-H column, n-hexane/iPrOH = 100/1, 214 nm, 0.5 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		23.473	186187.734	10534962.000	99.1492
2		26.205	1956.608	90402.984	0.8508
总计			188144.343	10625364.984	100.0000

2014-05-10

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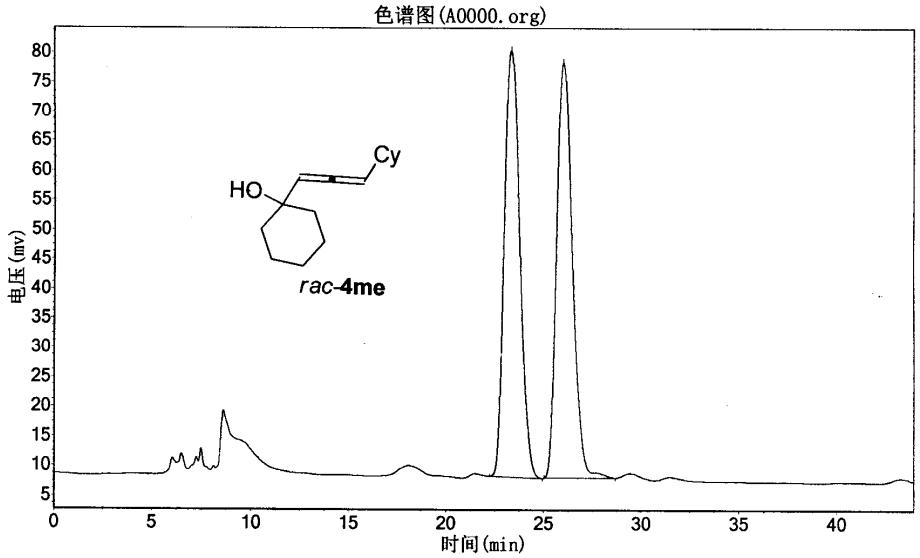
hx-10-179

实验单位：zju
实验时间：2014-05-10,13:25:11
谱图文件:D:\浙大智达\N2000\A0000.org

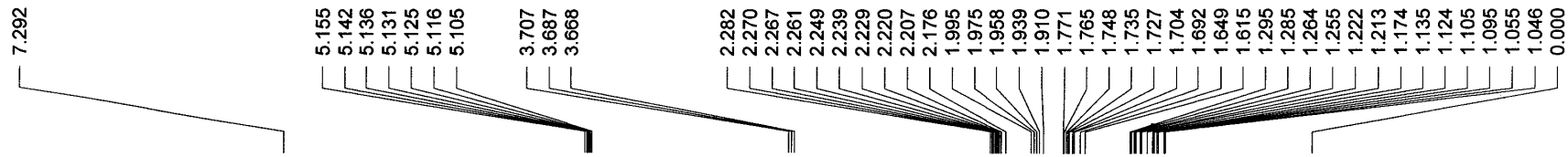
实验者：hx
报告时间：2014-05-10,14:13:54
积分方法:面积归一法

实验内容简介：
AD-H column, n-hexane/iPrOH = 100/1, 214 nm, 0.5 ml/min



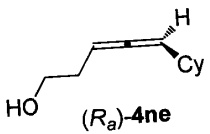
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		23.338	72246.195	4016893.750	50.3305
2		26.017	70282.305	3964131.750	49.6695
总计			142528.500	7981025.500	100.0000

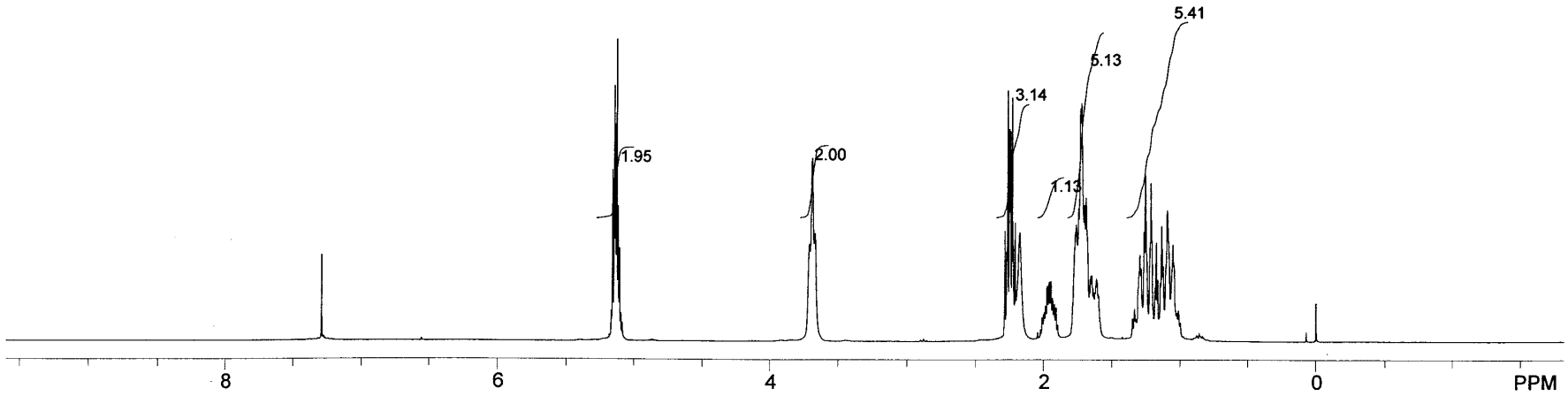


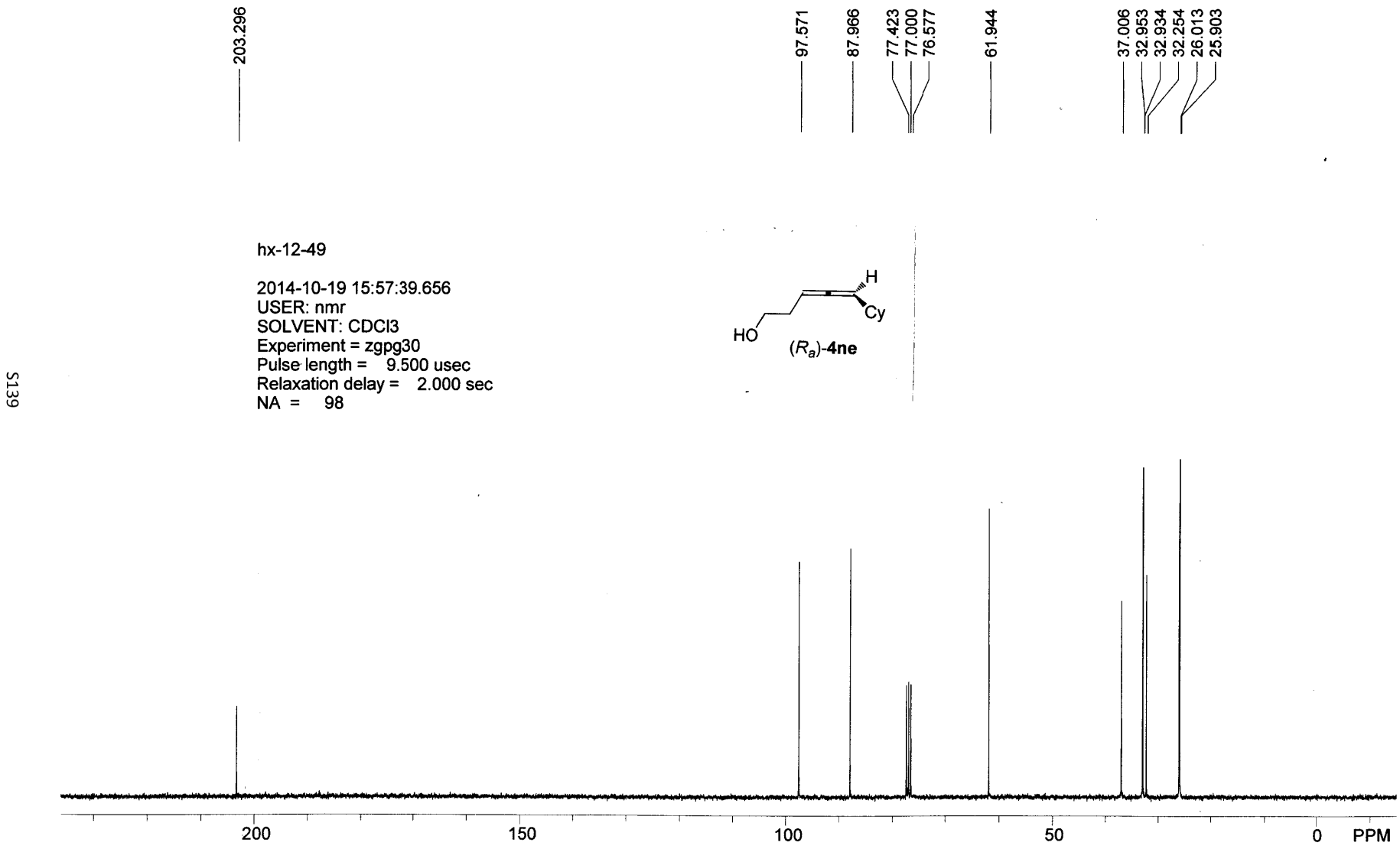
hx-12-49

2014-10-19 15:49:38.640
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ne**



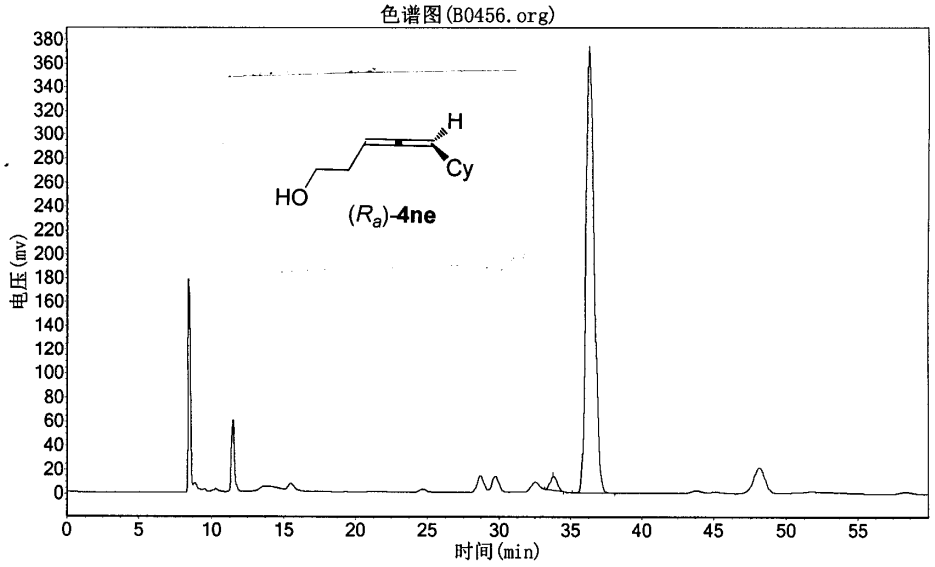

203.296
97.571
87.966
77.423
77.000
76.577
61.944
37.006
32.953
32.934
32.254
26.013
25.903
hx-12-49
2014-10-19 15:57:39.656
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 98
H
Cy
HO
(Ra)-4ne
200
150
100
50
0
PPM

hx-12-49

实验单位：zju
实验时间：2014-10-19,20:20:21
谱图文件:D:\浙大智达\N2000\样品\B0456.org

实验者：hx
报告时间：2014-10-19,22:02:50
积分方法:面积归一法

实验内容简介：
IC, hexane/i-PrOH = 100/1, 0.6 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		33.800	11637.059	396643.156	2.6225
2		36.355	370347.781	14727859.000	97.3775
总计			381984.840	15124502.156	100.0000

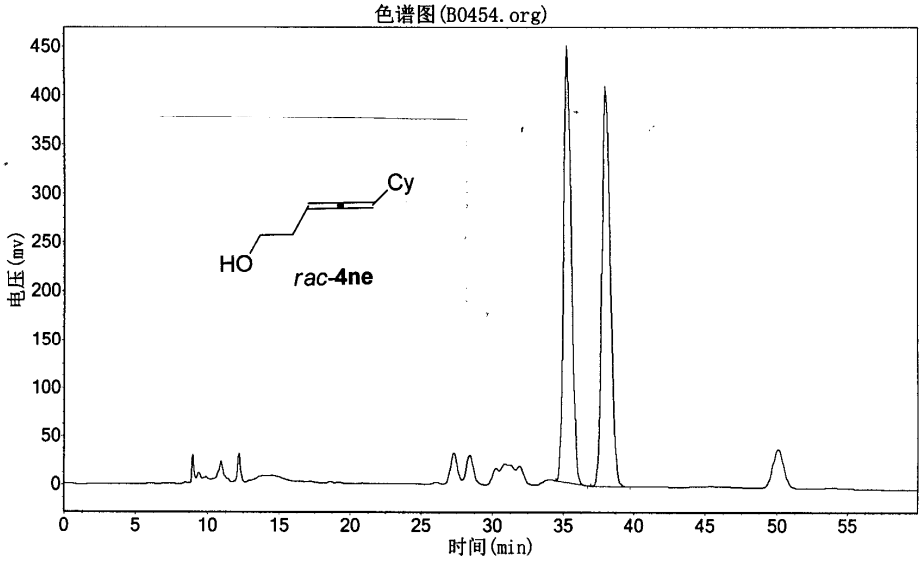
hx-12-16

实验单位：zju
实验时间：2014-10-19,17:16:24
谱图文件:D:\浙大智达\N2000\样品\B0454.org

实验者：hx
报告时间：2014-10-19,18:27:26
积分方法:面积归一法

实验内容简介：
IC, hexane/i-PrOH = 100/1, 0.6 ml/min, 214 nm

色谱图(B0454.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		35.303	444911.438	16779034.000	49.8608
2		37.992	406877.219	16872708.000	50.1392
总计			851788.656	33651742.000	100.0000

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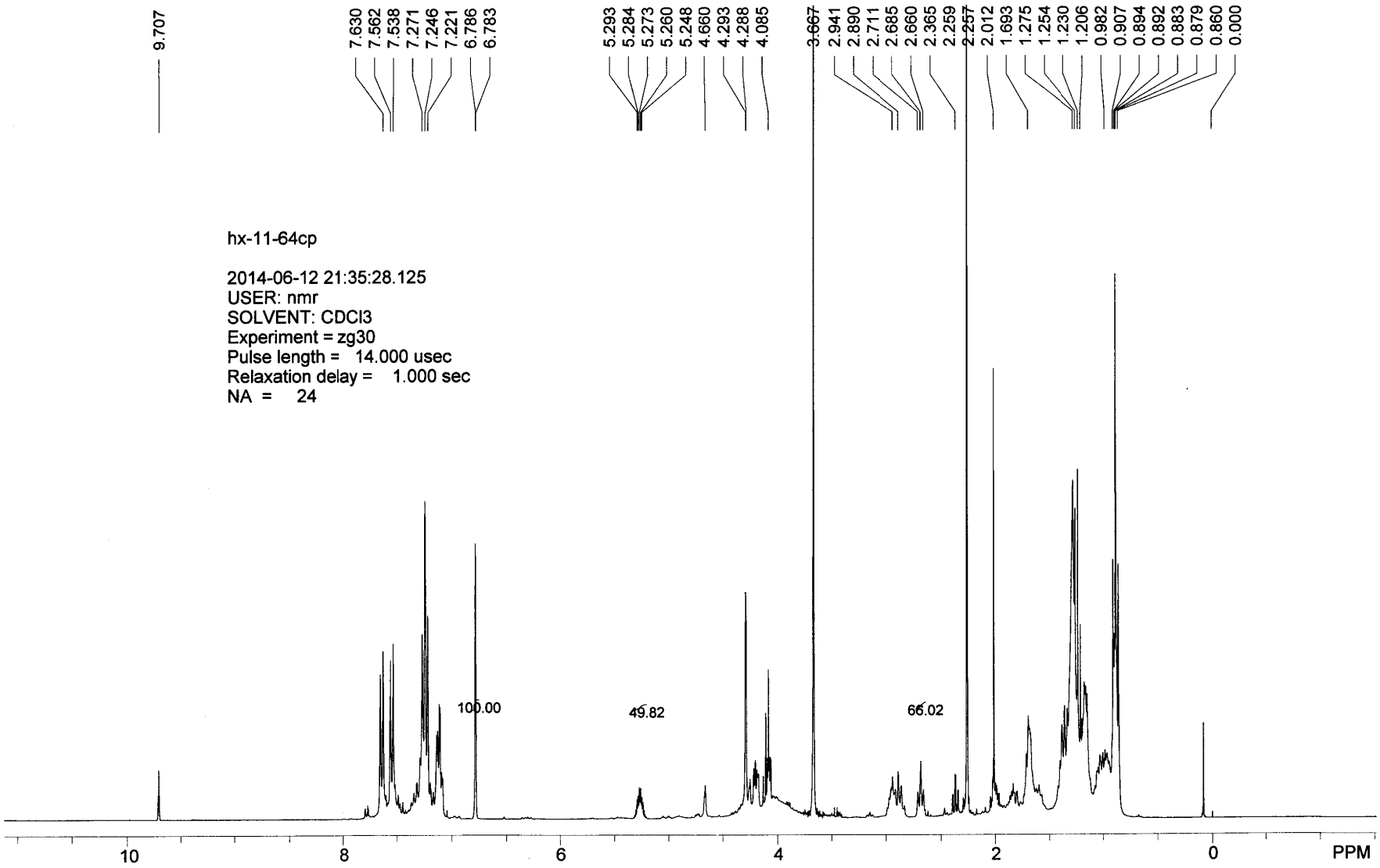
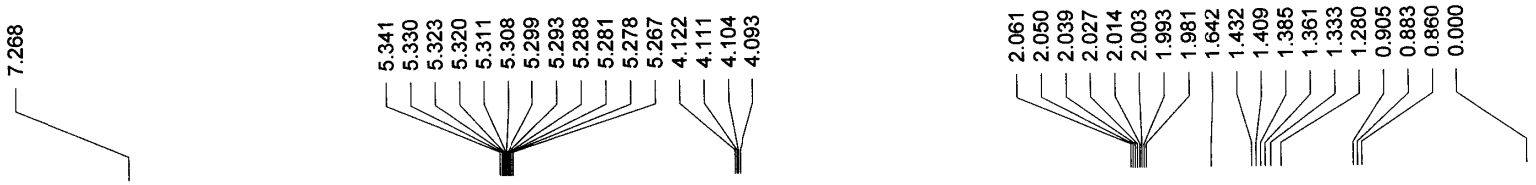
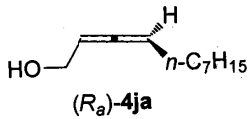

9.707
7.630
7.562
7.538
7.271
7.246
7.221
6.786
6.783
5.293
5.284
5.273
5.260
5.248
4.660
4.293
4.288
4.085
3.667
2.941
2.890
2.711
2.685
2.660
2.365
2.259
2.257
2.012
1.693
1.275
1.254
1.230
1.206
0.982
0.907
0.894
0.892
0.883
0.879
0.860
0.000
hx-11-64cp
2014-06-12 21:35:28.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 24
100.00
49.82
66.02
10
8
6
4
2
0
PPM

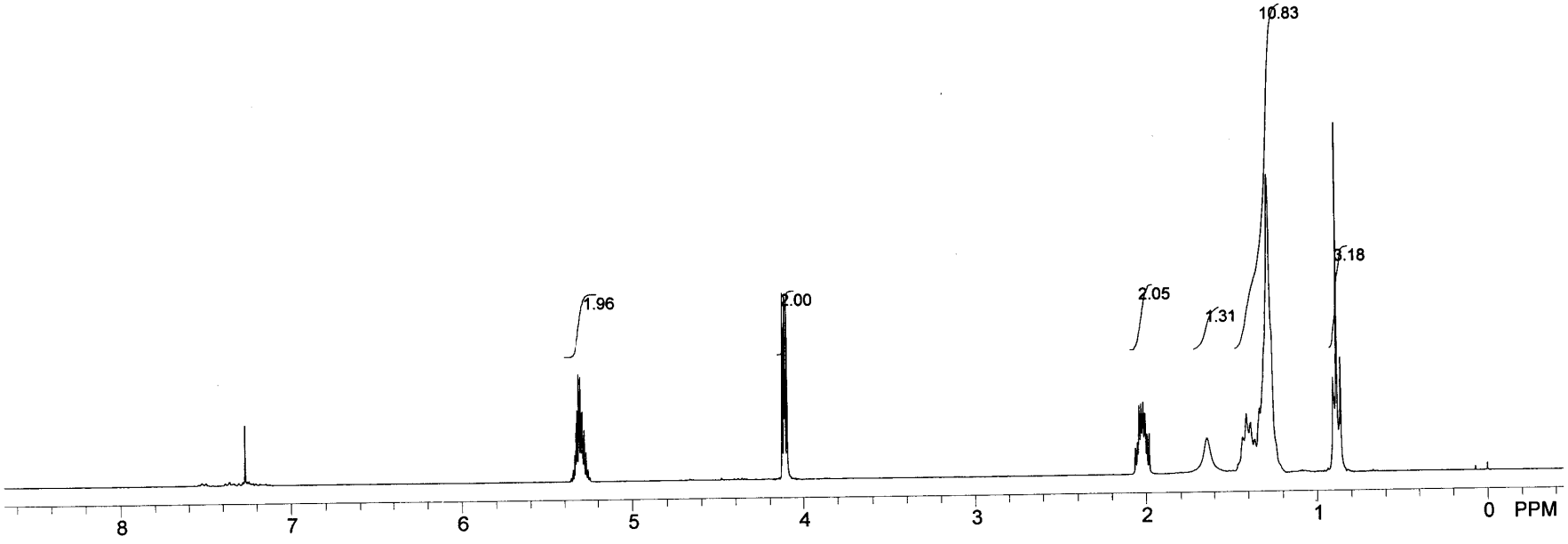


hx-11-64-1

2014-06-23 16:24:35.500
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ja**



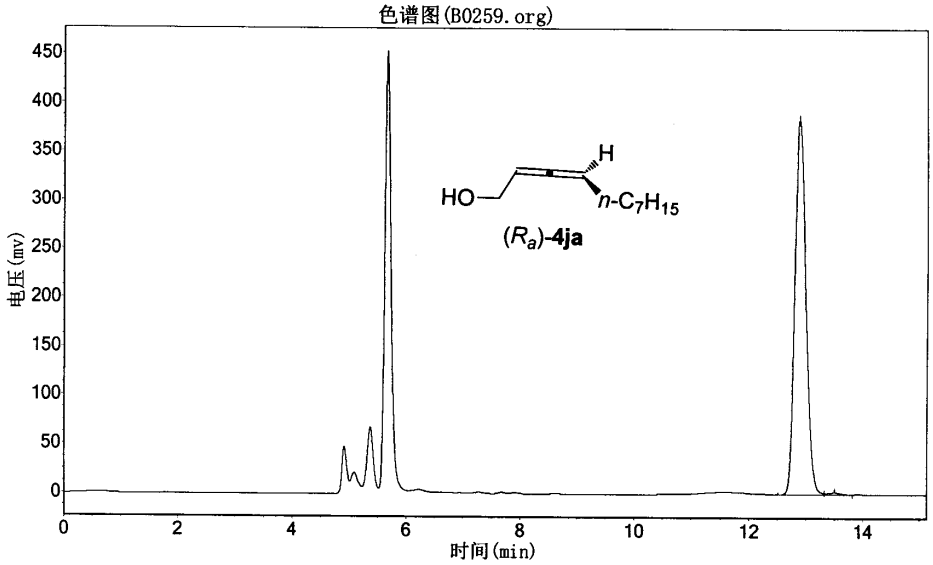
hx-11-64-1

单位：zju
时间：2014-06-18,16:24:33
文件:D:\浙大智达\N2000\样品\B0259.org

实验者：hx
报告时间：2014-06-18,16:40:39
积分方法:面积归一法

内容简介：
column, n-hexane/iPrOH = 100/1, 214 nm, 0.6 ml/min

色谱图(B0259.org)



分析结果表

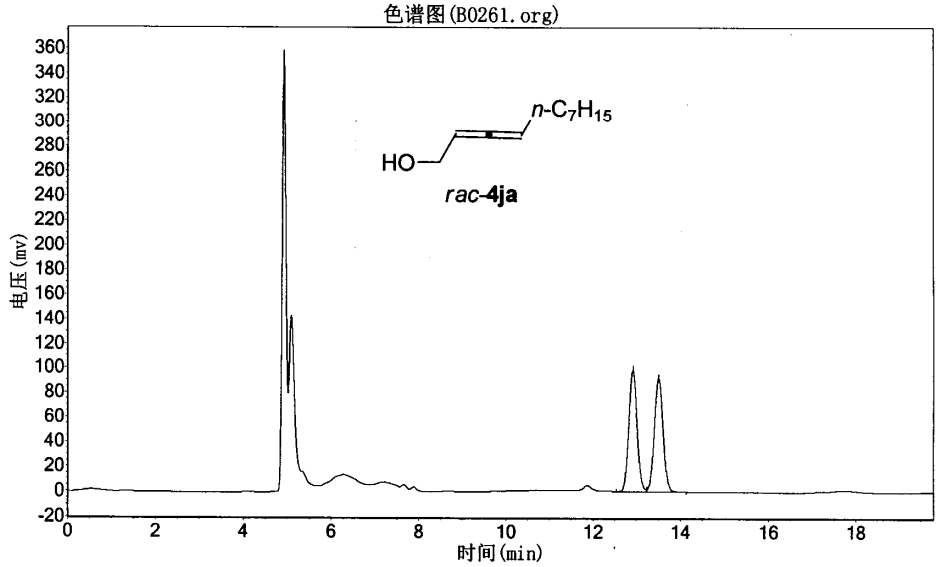
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.890	384044.188	5131786.000	99.4223
2		13.500	1977.669	29820.711	0.5777
计			386021.857	5161606.711	100.0000

hx-10-159

实验单位：zju
实验时间：2014-06-18,17:01:10
谱图文件:D:\浙大智达\N2000\样品\B0261.org

实验者：hx
报告时间：2014-06-20,13:35:57
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 100/1, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.903	97951.031	1289484.000	50.2282
2		13.492	91542.875	1277764.750	49.7718
总计			189493.906	2567248.750	100.0000

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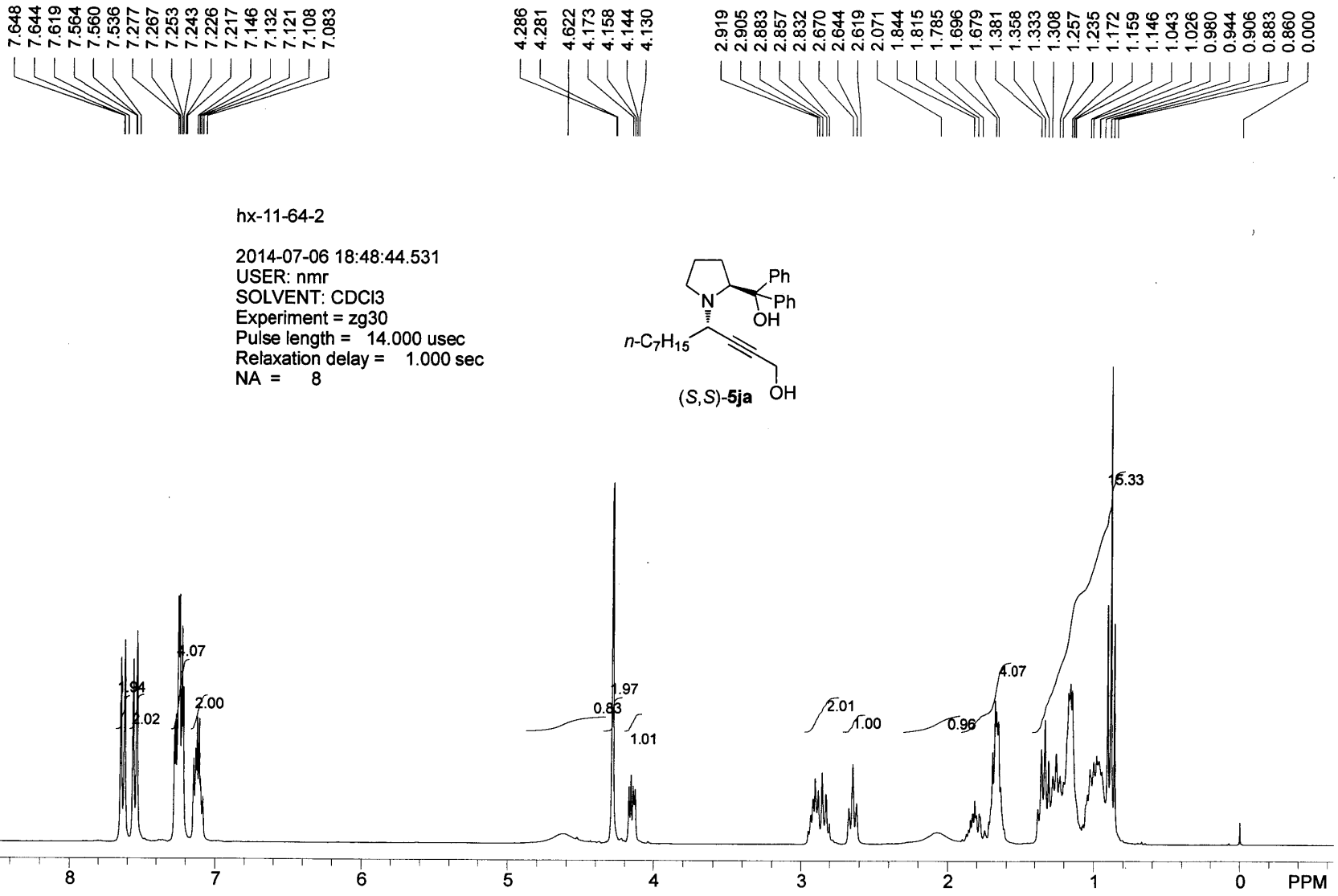
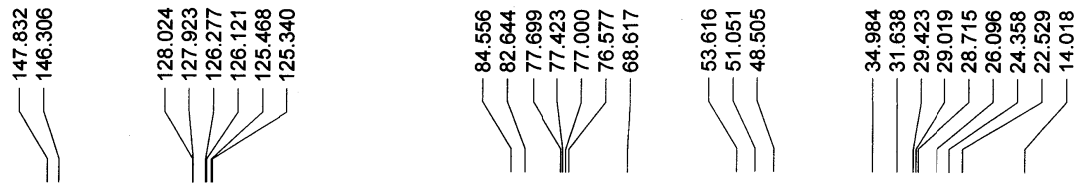
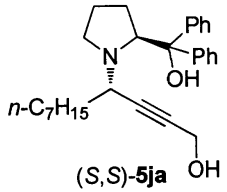

7.648
7.644
7.619
7.564
7.560
7.536
7.277
7.267
7.253
7.243
7.226
7.217
7.146
7.132
7.121
7.108
7.083
4.286
4.281
4.622
4.173
4.158
4.144
4.130
2.919
2.905
2.883
2.857
2.832
2.670
2.644
2.619
2.071
1.844
1.815
1.785
1.696
1.679
1.381
1.358
1.333
1.308
1.257
1.235
1.172
1.159
1.146
1.043
1.026
0.980
0.944
0.906
0.883
0.860
0.000
hx-11-64-2
2014-07-06 18:48:44.531
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Ph
Ph
N
OH
n-C7H15
OH
(S,S)-5ja
15.33
4.07
2.01
1.00
0.96
4.07
1.97
0.83
1.01
2.00
1.94
2.02
8
7
6
5
4
3
2
1
0
PPM

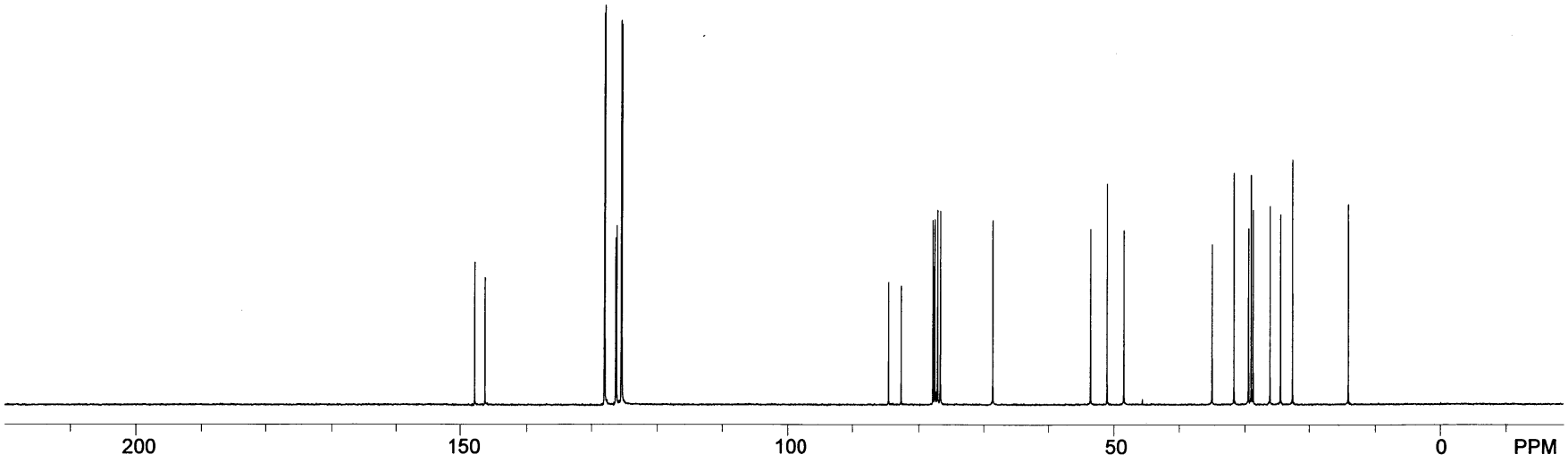


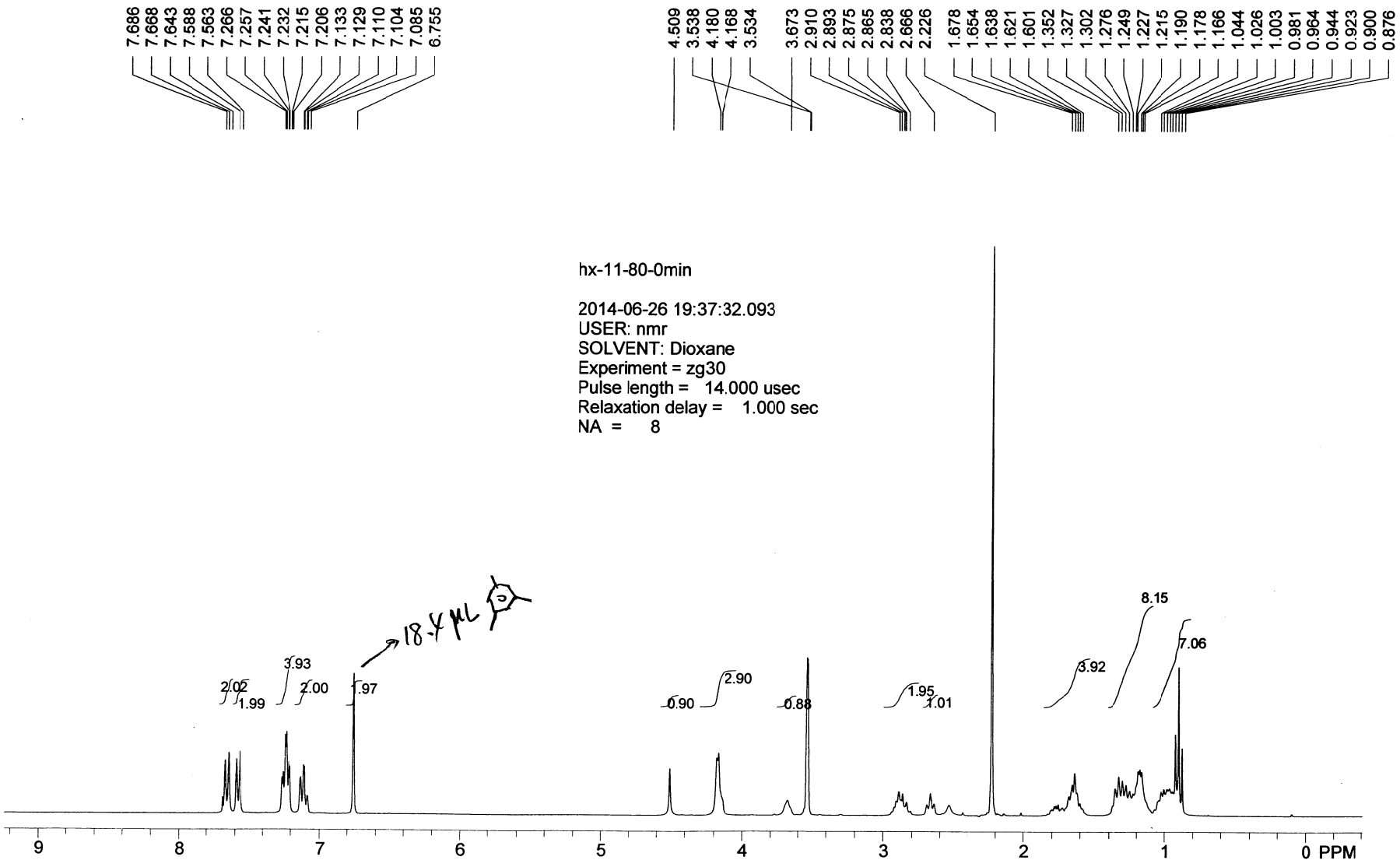
hx-11-64-2

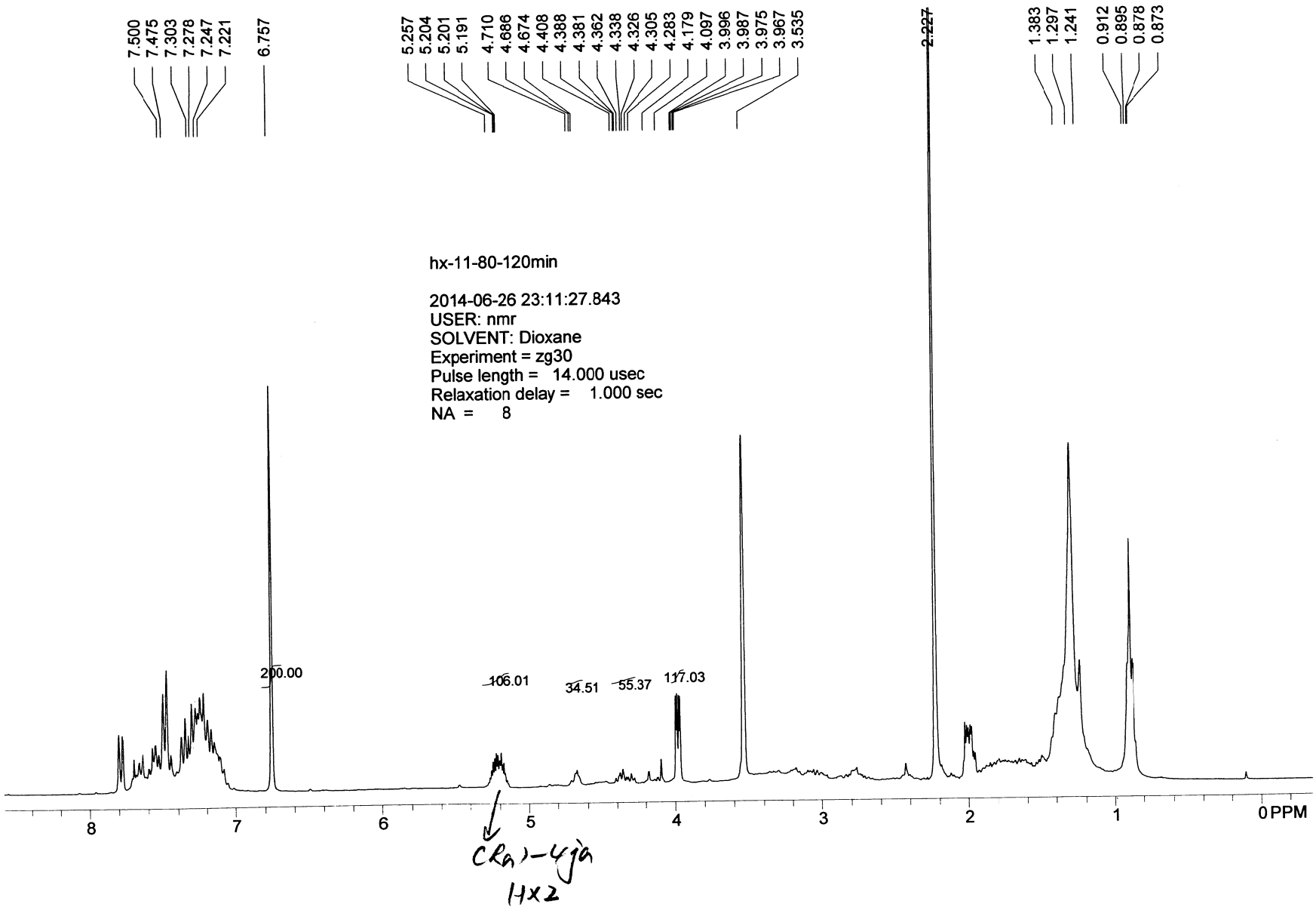
2014-07-06 20:46:58.343
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1999

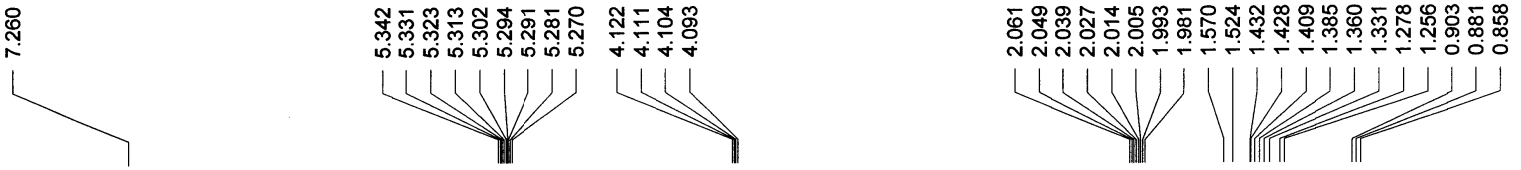


(*S*,*S*)-**5ja**



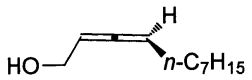

7.686
7.668
7.643
7.588
7.563
7.266
7.257
7.241
7.232
7.215
7.206
7.133
7.129
7.110
7.104
7.085
6.755
4.509
3.538
4.180
4.168
3.534
3.673
2.910
2.893
2.875
2.865
2.838
2.666
2.226
1.678
1.654
1.638
1.621
1.601
1.352
1.327
1.302
1.276
1.249
1.227
1.215
1.190
1.178
1.166
1.044
1.026
1.003
0.981
0.964
0.944
0.923
0.900
0.876
hx-11-80-0min
2014-06-26 19:37:32.093
USER: nmr
SOLVENT: Dioxane
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
18.4 mL
2.02
1.99
3.93
2.00
1.97
0.90
2.90
0.88
1.95
1.01
3.92
8.15
7.06
9
8
7
6
5
4
3
2
1
0 PPM


7.500
7.475
7.303
7.278
7.247
7.221
6.757
5.257
5.204
5.201
5.191
4.710
4.686
4.674
4.408
4.388
4.381
4.362
4.338
4.326
4.305
4.283
4.179
4.097
3.996
3.987
3.975
3.967
3.535
2.227
1.383
1.297
1.241
0.912
0.895
0.878
0.873
hx-11-80-120min
2014-06-26 23:11:27.843
USER: nmr
SOLVENT: Dioxane
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
200.00
106.01
34.51
55.37
117.03
8
7
6
5
4
3
2
1
0 PPM
CRa1-4ja
1Hx2

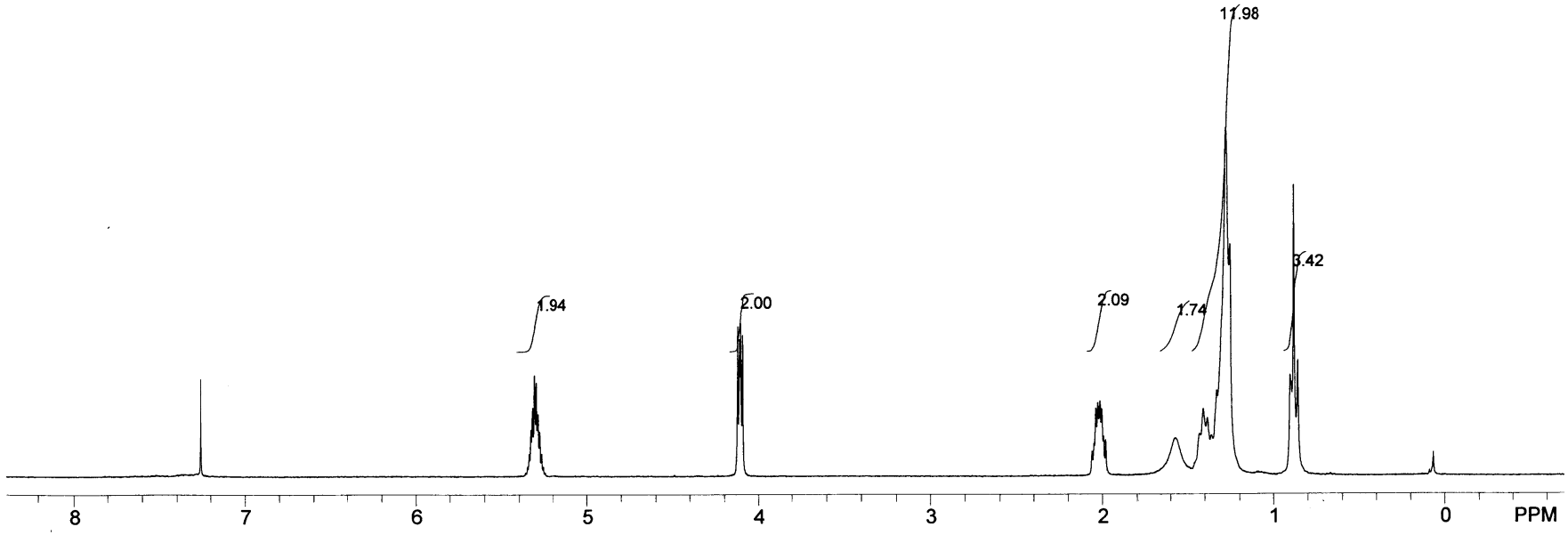


hx-11-80

2014-07-09 17:42:34.984
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ja**



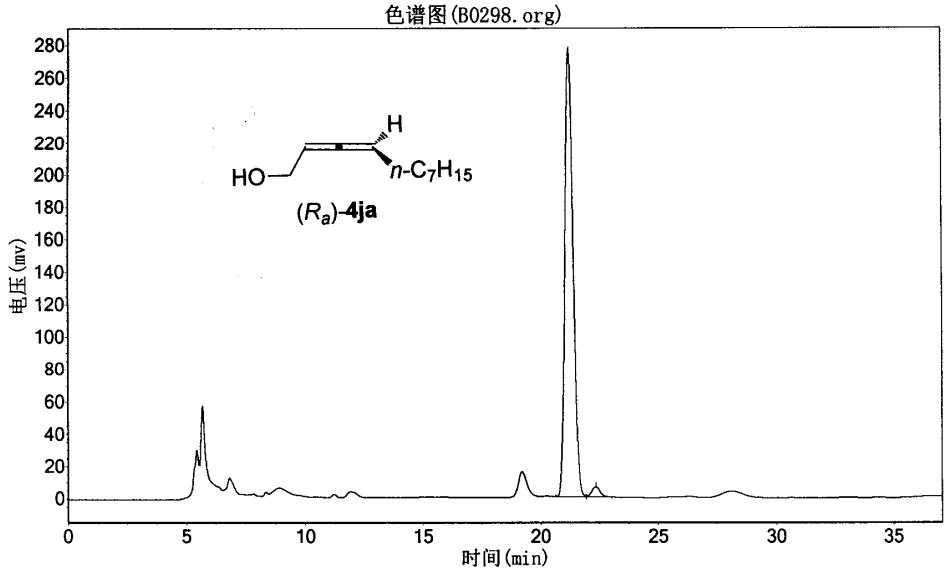
hx-11-80

实验单位：zju
实验时间：2014-07-09,19:50:50
谱图文件:D:\浙大智达\N2000\样品\B0298.org

实验者：hx
报告时间：2014-07-09,20:42:45
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 200/1, 214 nm, 0.6 ml/min

色谱图(B0298.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		21.223	275229.281	6707292.000	97.8280
2		22.343	6212.212	148917.688	2.1720
总计			281441.493	6856209.688	100.0000

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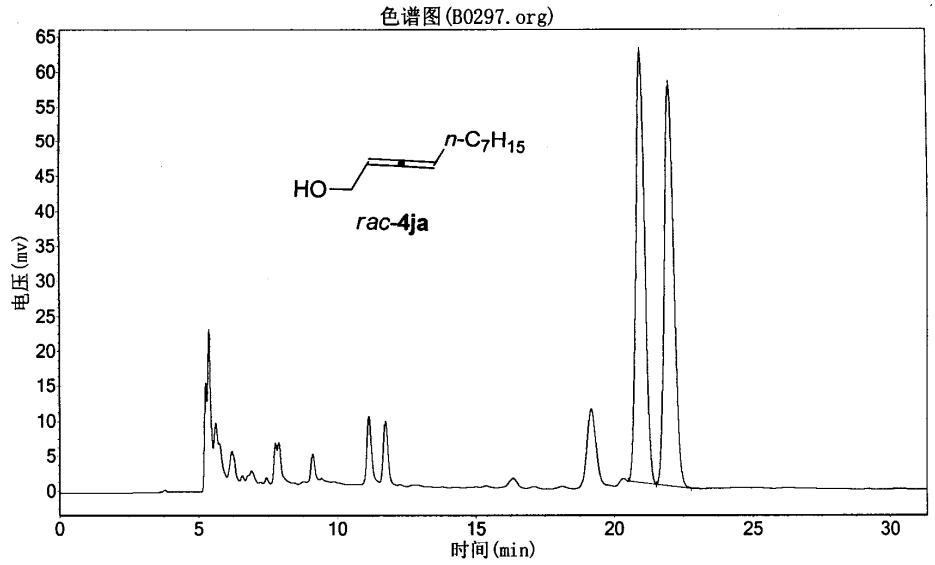
hx-10-159

实验单位：zju
实验时间：2014-07-09,19:18:23
谱图文件:D:\浙大智达\N2000\样品\B0297.org

实验者：hx
报告时间：2014-07-09,19:53:57
积分方法:面积归一法

实验内容简介：
AS-H column, n-hexane/iPrOH = 200/1, 214 nm, 0.6 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.962	61642.344	1357803.000	50.1331
2		21.988	57351.395	1350592.875	49.8669
总计			118993.738	2708395.875	100.0000

2014-07-09

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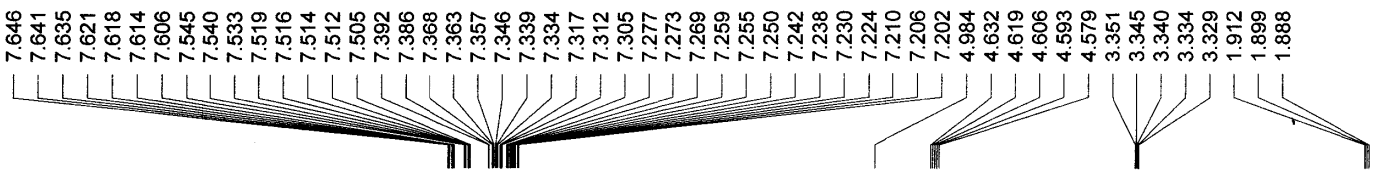
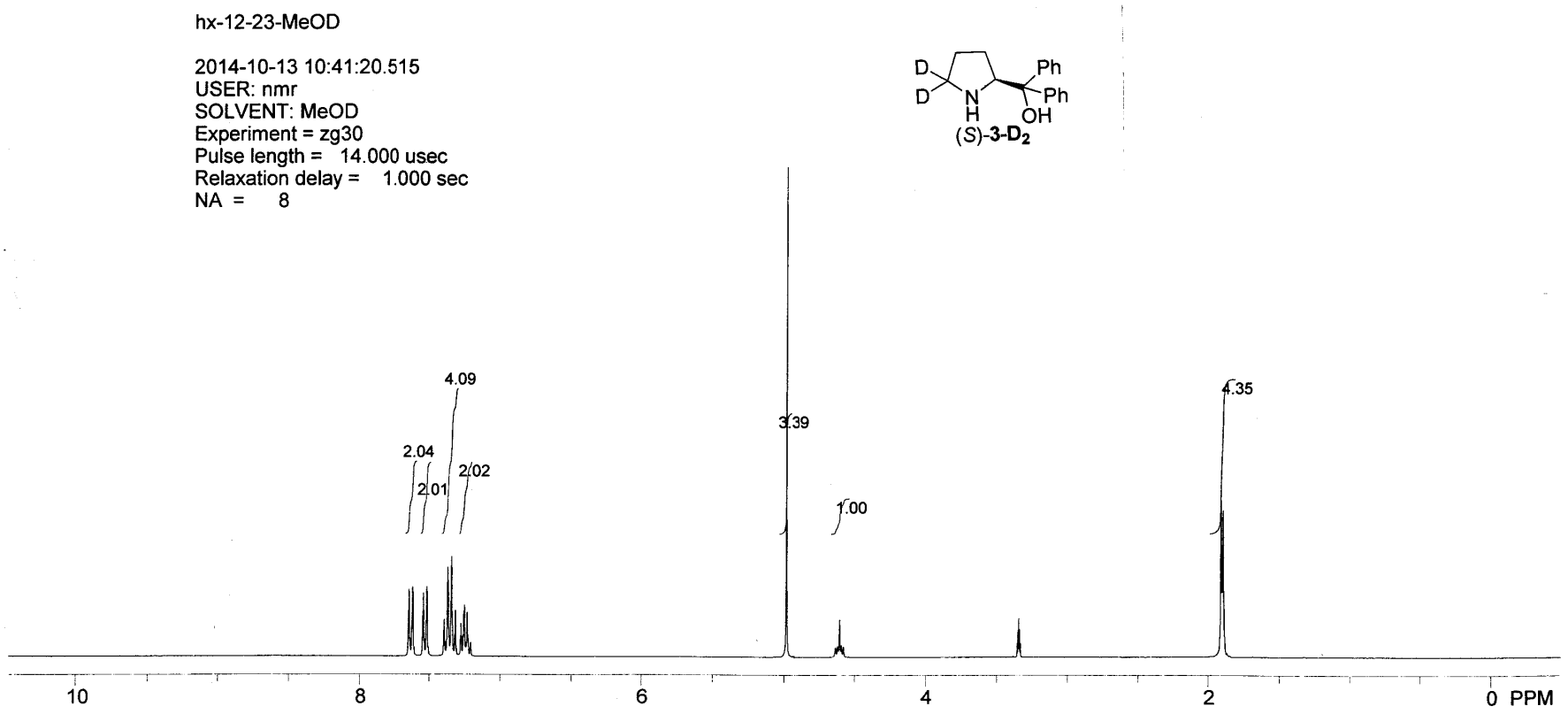
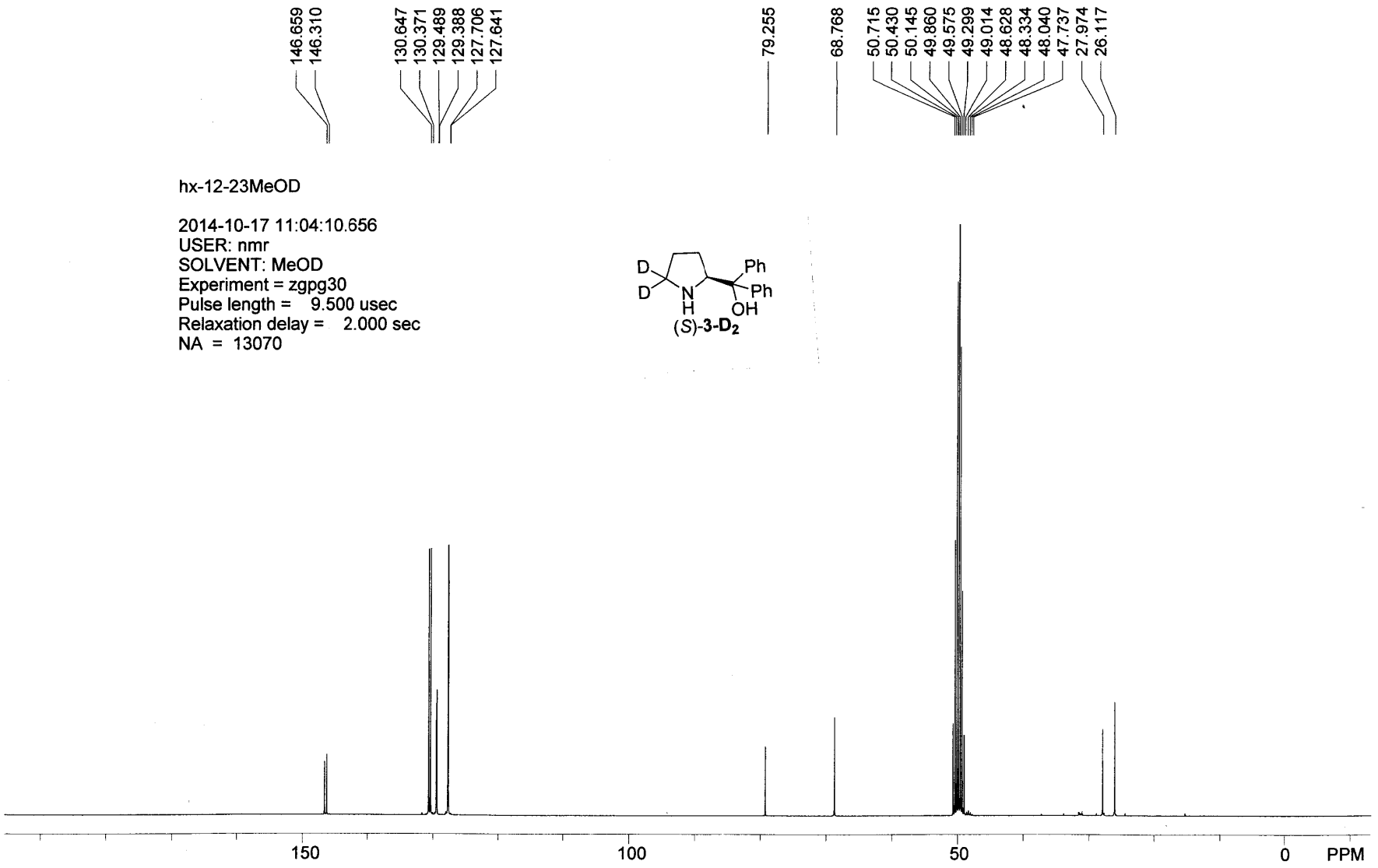
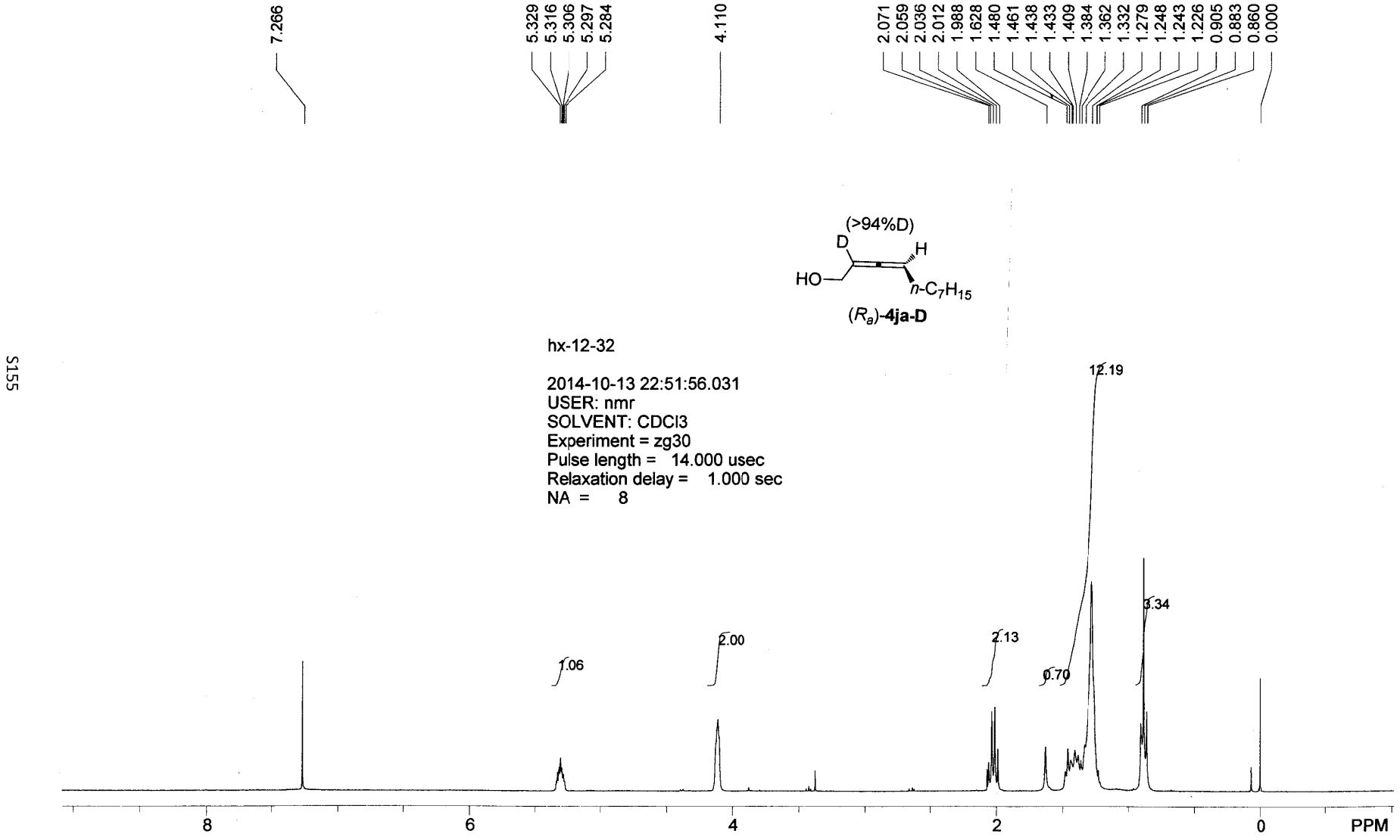
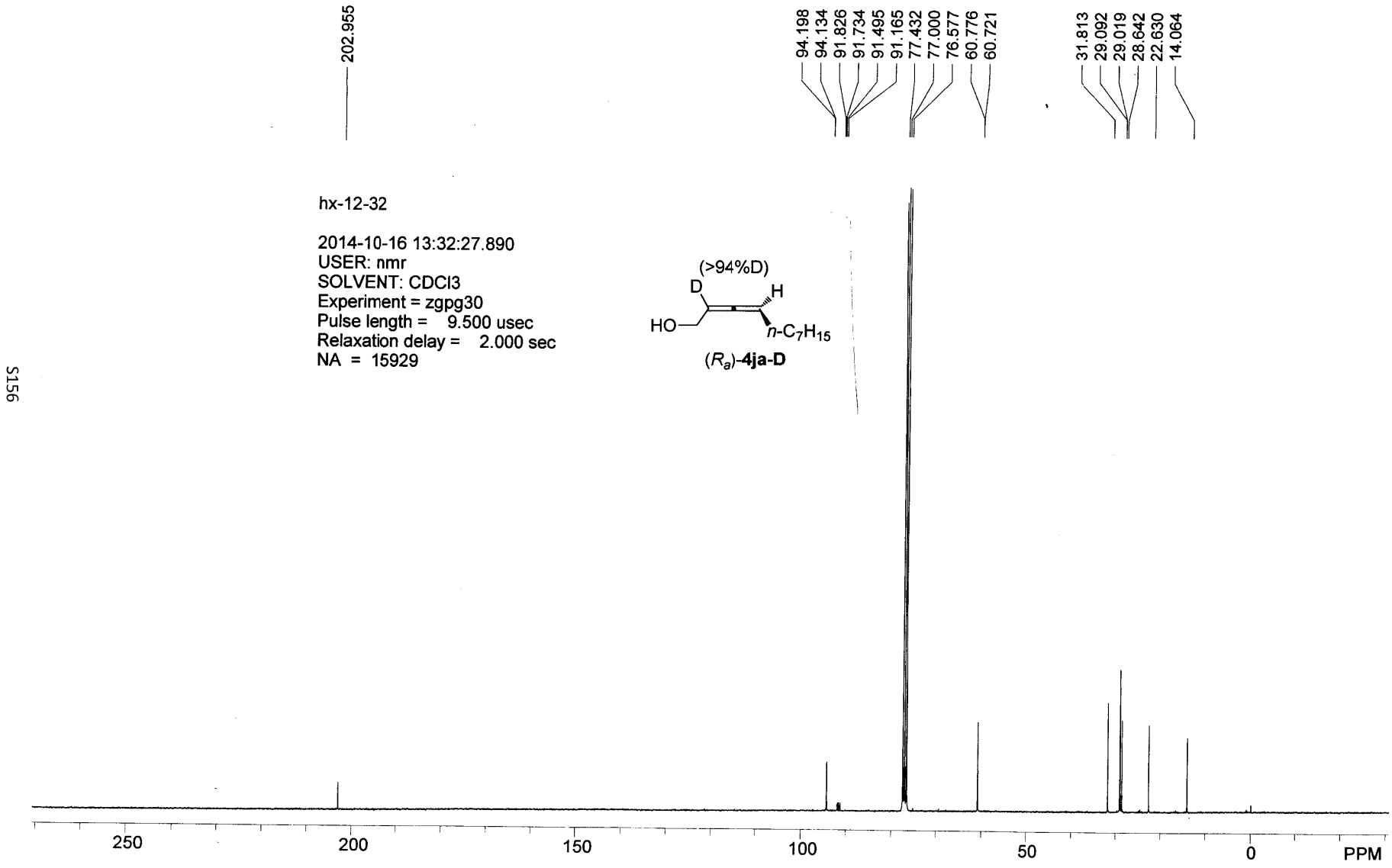

7.646
7.641
7.635
7.621
7.618
7.614
7.606
7.545
7.540
7.533
7.519
7.516
7.514
7.512
7.505
7.392
7.386
7.368
7.363
7.357
7.346
7.339
7.334
7.317
7.312
7.305
7.277
7.273
7.269
7.259
7.255
7.250
7.242
7.238
7.230
7.224
7.210
7.206
7.202
4.984
4.632
4.619
4.606
4.593
4.579
3.351
3.345
3.340
3.334
3.329
1.912
1.899
1.888


hx-12-23-MeOD
2014-10-13 10:41:20.515
USER: nmr
SOLVENT: MeOD
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
D
D
Ph
Ph
N
H
OH
(S)-3-D2
4.09
2.04
2.01
2.02
3.39
1.00
4.35
10
8
6
4
2
0 PPM


146.659
146.310
130.647
130.371
129.489
129.388
127.706
127.641
79.255
68.768
50.715
50.430
50.145
49.860
49.575
49.299
49.014
48.628
48.334
48.040
47.737
27.974
26.117
hx-12-23MeOD
2014-10-17 11:04:10.656
USER: nmr
SOLVENT: MeOD
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 13070
D
D
N
H
Ph
Ph
OH
(S)-3-D2
150
100
50
0
PPM


7.266
5.329
5.316
5.306
5.297
5.284
4.110
2.071
2.059
2.036
2.012
1.988
1.628
1.480
1.461
1.438
1.433
1.409
1.384
1.362
1.332
1.279
1.248
1.243
1.226
0.905
0.883
0.860
0.000
(>94%D)
D
H
HO
n-C7H15
(Ra)-4ja-D
hx-12-32
2014-10-13 22:51:56.031
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
1.06
2.00
2.13
0.70
12.19
3.34
8
6
4
2
0
PPM


202.955
94.198
94.134
91.826
91.734
91.495
91.165
77.432
77.000
76.577
60.776
60.721
31.813
29.092
29.019
28.642
22.630
14.064
hx-12-32
2014-10-16 13:32:27.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 15929
(>94%D)
D
H
HO
n-C7H15
(Ra)-4ja-D
250
200
150
100
50
0
PPM

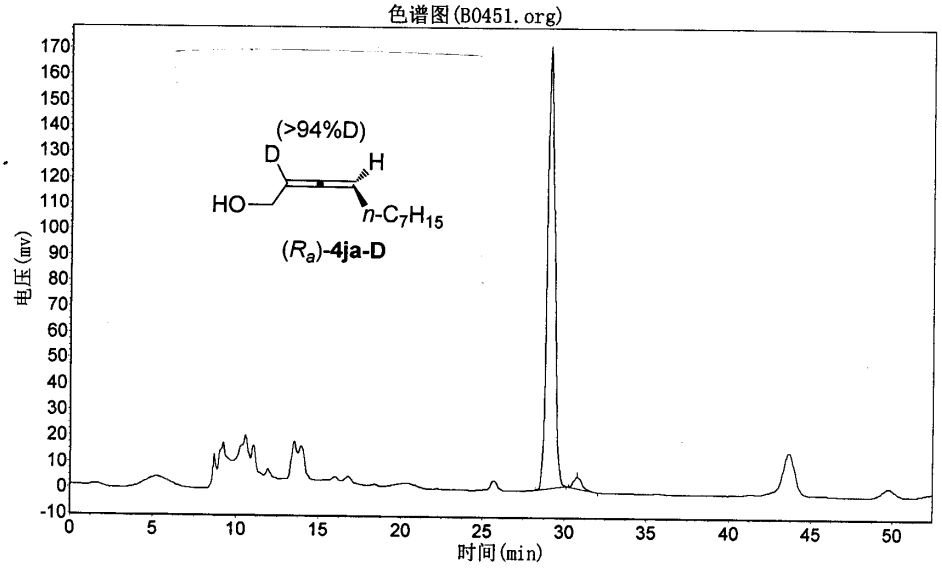
hx-12-32

实验单位：zju
实验时间：2014-10-18,15:30:39
谱图文件:D:\浙大智达\N2000\样品\B0451.org

实验者：hx
报告时间：2014-10-18,16:24:48
积分方法:面积归一法

实验内容简介：
AS-H, hexane/i-PrOH = 200/1, 0.6 ml/min, 214 nm

色谱图(B0451.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.107	168817.938	5334799.000	97.1399
2		30.797	4587.960	157072.906	2.8601
总计			173405.897	5491871.906	100.0000

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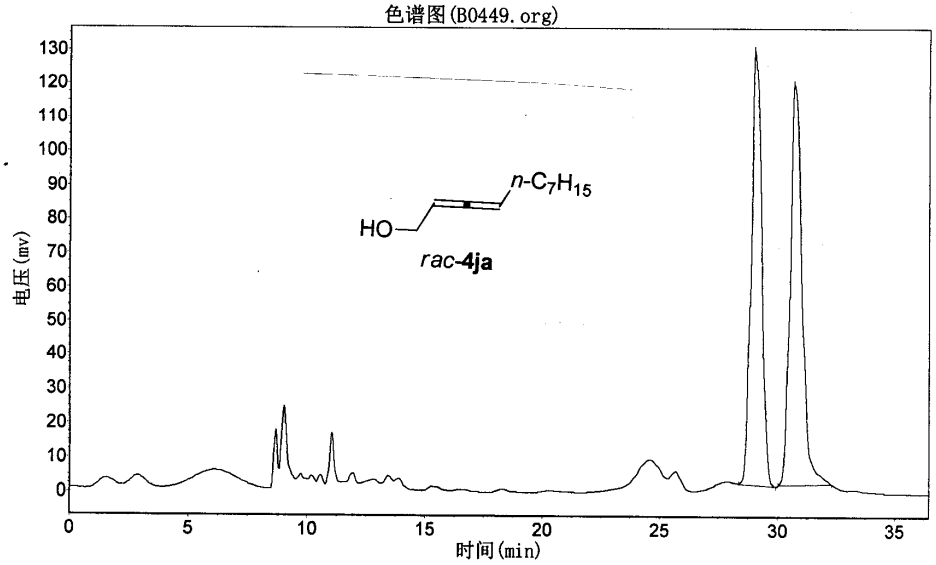
hx-11-185

实验单位：zju
实验时间：2014-10-18,14:00:00
谱图文件:D:\浙大智达\N2000\样品\B0449.org

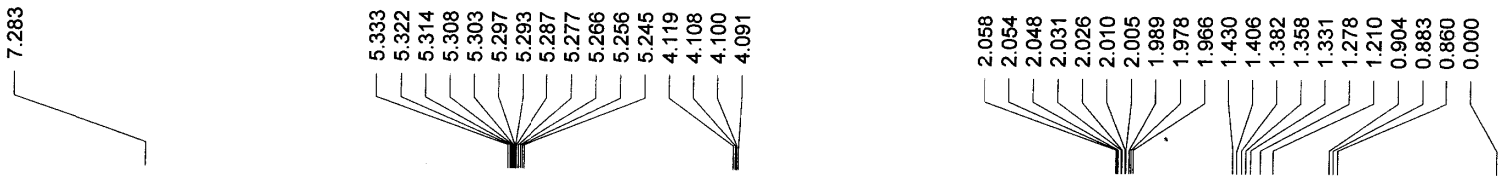
实验者：hx
报告时间：2014-10-18,14:40:47
积分方法:面积归一法

实验内容简介：
AS-H, hexane/i-PrOH = 200/1, 0.6 ml/min, 214 nm



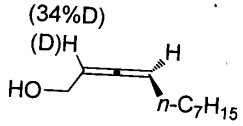
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.040	127655.711	3902991.500	48.5773
2		30.700	117624.805	4131603.500	51.4227
总计			245280.516	8034595.000	100.0000

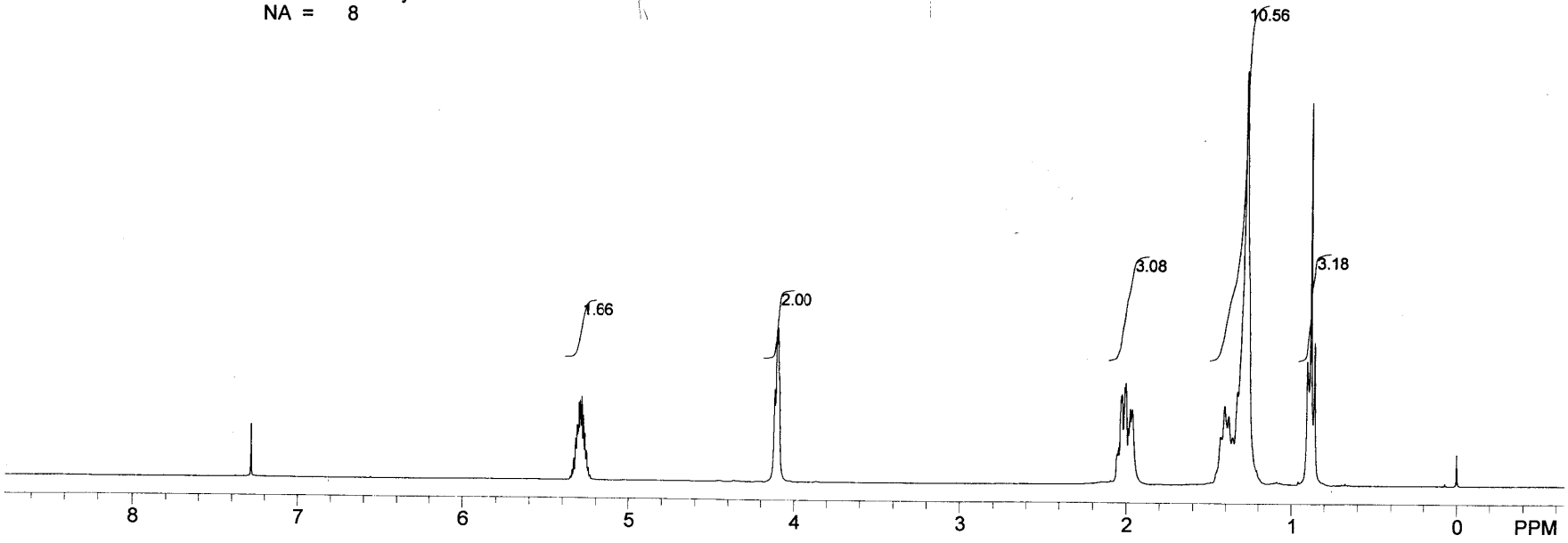


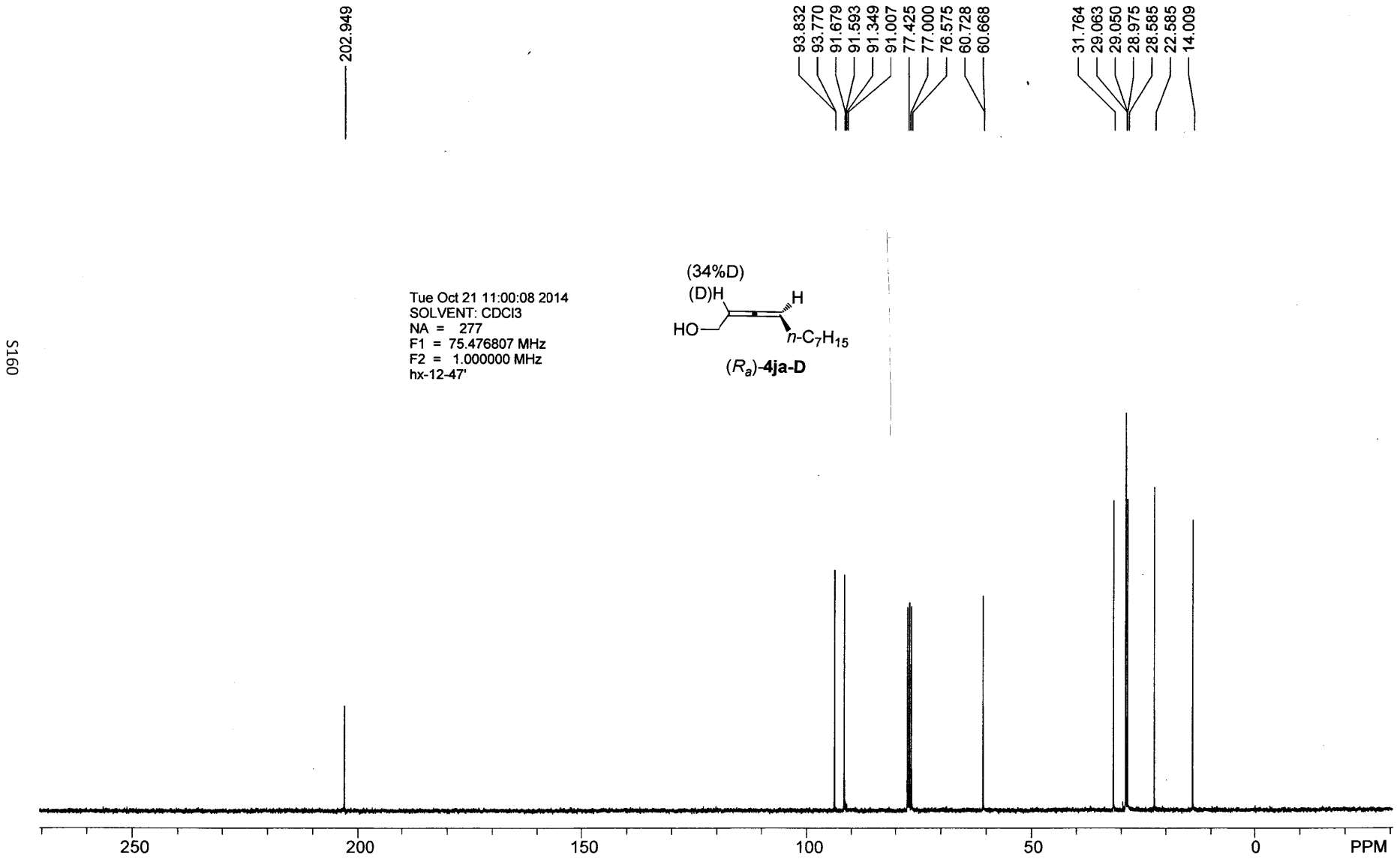
hx-12-47'

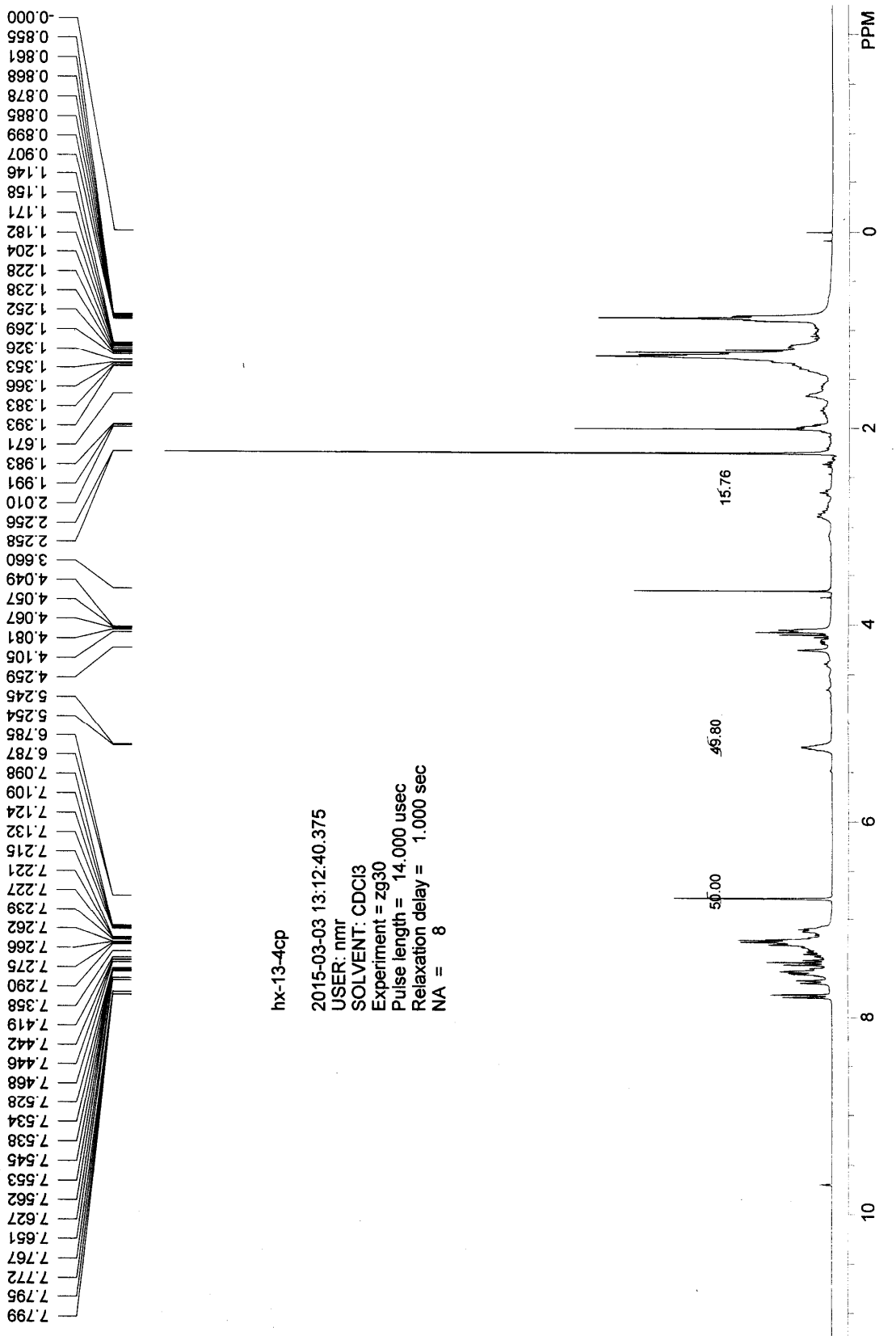
2014-10-21 18:42:08.890
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8

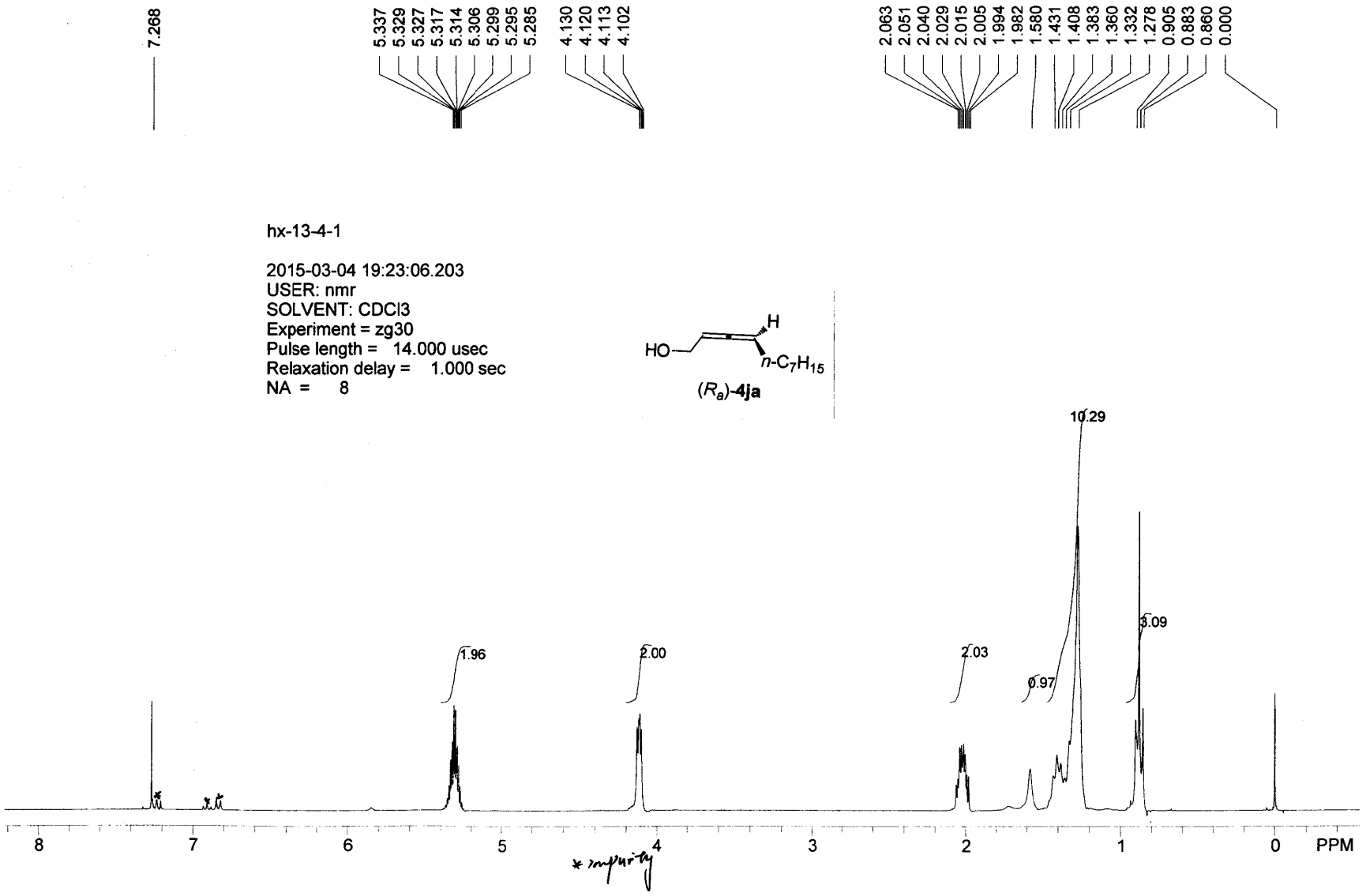


(R_a)-**4ja-D**




202.949
93.832
93.770
91.679
91.593
91.349
91.007
77.425
77.000
76.575
60.728
60.668
31.764
29.063
29.050
28.975
28.585
22.585
14.009
Tue Oct 21 11:00:08 2014
SOLVENT: CDCl3
NA = 277
F1 = 75.476807 MHz
F2 = 1.000000 MHz
hx-12-47'
(34%D)
(D)H
H
HO
n-C7H15
(Ra)-4ja-D
250
200
150
100
50
0
PPM


hx-13-4cp
2015-03-03 13:12:40.375
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
50.00
49.80
15.76
PPM
0
2
4
6
8
10
-0.000
0.855
0.861
0.868
0.878
0.885
0.899
0.907
1.146
1.158
1.171
1.182
1.204
1.228
1.238
1.262
1.269
1.326
1.353
1.366
1.383
1.393
1.671
1.983
1.991
2.010
2.256
2.258
3.660
4.049
4.057
4.067
4.081
4.105
4.259
5.245
5.254
6.785
6.787
7.098
7.109
7.124
7.132
7.215
7.221
7.227
7.239
7.262
7.266
7.275
7.290
7.358
7.419
7.442
7.446
7.468
7.528
7.534
7.538
7.545
7.553
7.562
7.627
7.651
7.767
7.772
7.795
7.799


7.268
5.337
5.329
5.327
5.317
5.314
5.306
5.299
5.295
5.285
4.130
4.120
4.113
4.102
2.063
2.051
2.040
2.029
2.015
2.005
1.994
1.982
1.580
1.431
1.408
1.383
1.360
1.332
1.278
0.905
0.883
0.860
0.000
hx-13-4-1
2015-03-04 19:23:06.203
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
HO
H
n-C7H15
(Ra)-4ja
10.29
3.09
2.03
2.00
1.96
0.97
8
7
6
5
4
3
2
1
0
PPM
* impurity

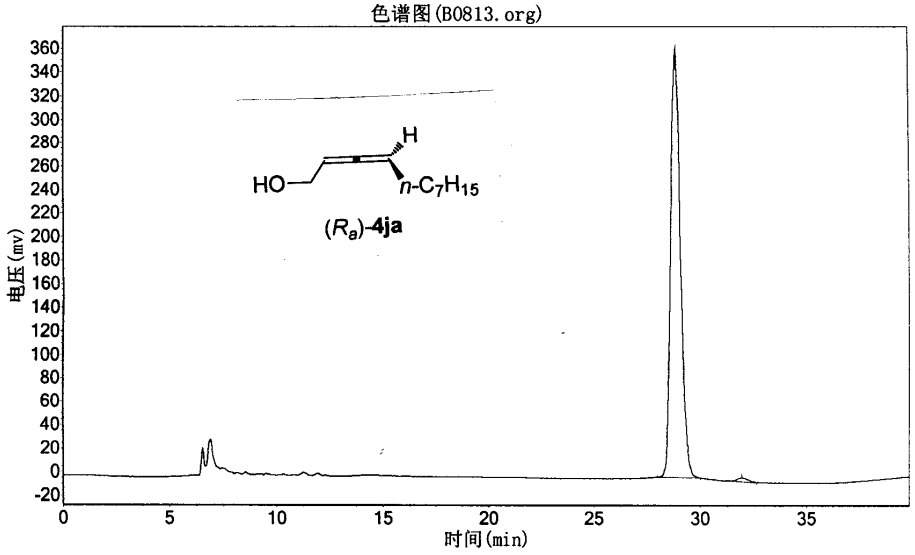
hx-13-4-1

实验单位：zju
实验时间：2015-03-04,12:28:01
谱图文件:D:\浙大智达\N2000\样品\B0813.org

实验者：hx
报告时间：2015-03-04,13:09:43
积分方法:面积归一法

实验内容简介：
AS-H, n-hexane/i-PrOH = 200/1, 214 nm, 1.0 ml/min

色谱图(B0813.org)



分析结果表

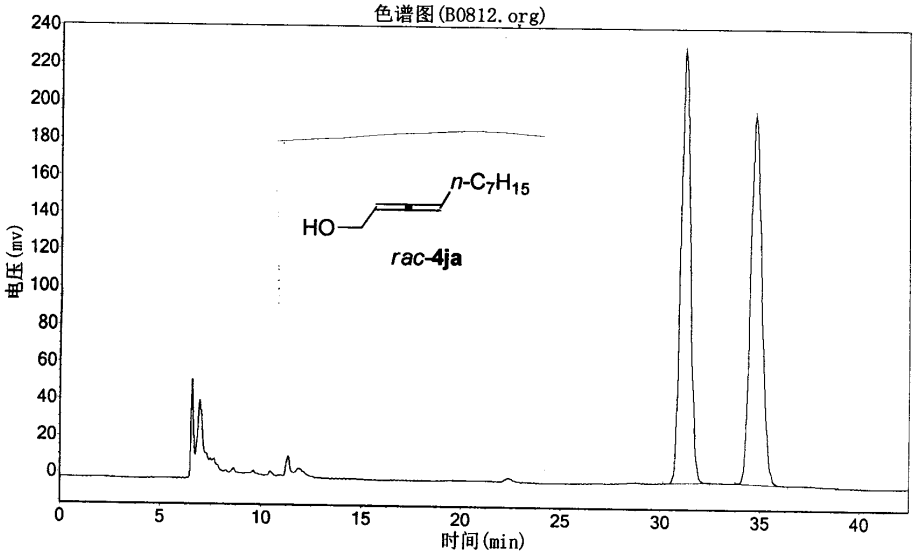
峰号	峰名	保留时间	峰高	峰面积	含量
1		28.818	363976.094	11593127.000	99.0375
2		31.950	3337.797	112667.016	0.9625
总计			367313.891	11705794.016	100.0000

hx-13-6

实验单位：zju
实验时间：2015-03-04,11:44:36
谱图文件:D:\浙大智达\N2000\样品\B0812.org

实验者：hx
报告时间：2015-03-04,12:29:34
积分方法:面积归一法

实验内容简介：
AS-H, n-hexane/i-PrOH = 200/1, 214 nm, 1.0 ml/min



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		31.153	231147.047	7677437.000	49.8305
2		34.673	197123.719	7729661.500	50.1695
总计			428270.766	15407098.500	100.0000

2015-03-04

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