Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

A Concise Construction of Carbohydrate-tethered Axially Chiral Allenes via Copper Catalysis

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Supporting Information

Figure S1. The apparatus used in this study

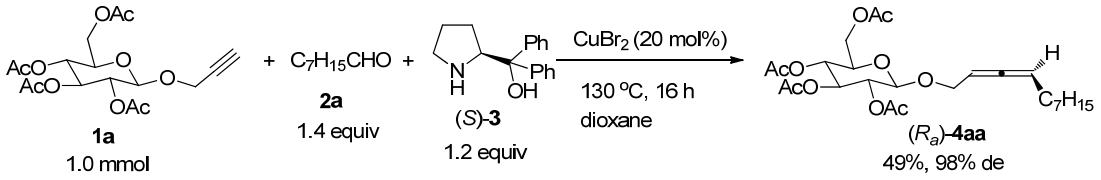
General Experimental Methods

^{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. Copper(II) bromide and copper(I) bromide were purchased from J&K. Copper(I) iodide was purchased from Shanghai Darui Fine Chemicals. $ZnBr_2$ was purchased from Alfa Aesar and kept in a glove box. (*S*)-α,α-Diphenylprolinol and (*R*)-α,α-diphenylprolinol were purchased from Shanghai Darui Fine Chemicals. Aldehydes were distilled right before use. Dioxane and toluene were dried over sodium wire with benzophenone as the indicator and distilled freshly before use. All the temperatures are referred to the oil baths used.

The following examples (R_a)-**4aa**~(R_a)-**4ie** were prepared according to the Typical Procedure I except (R_a)-**4ob**. Their corresponding racemic diastereoisomers were prepared by following the same procedure using racemic diphenylprolinol ***rac*-3.**

1. Preparation of (R_a)-4aa and (S_a)-4aa.

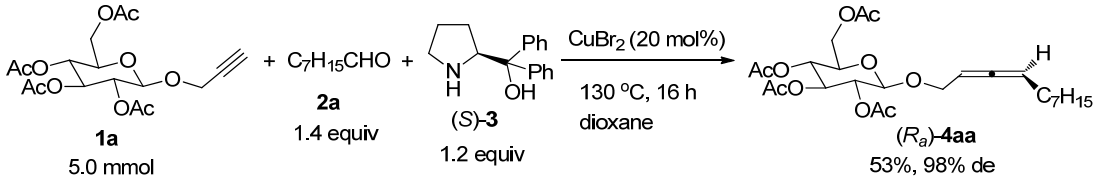
(1) Preparation of (R_a)-4aa on 1.0 mmol scale. hx-10-7



Typical Procedure I: To a flame-dried Schlenk tube were added $CuBr_2$ (44.9 mg, 0.2 mmol), **1a** (387.0 mg, 1.0 mmol), (*S*)-**3** (304.5 mg, 1.2 mmol), and **2a** (180.1 mg, 1.4 mmol)/dioxane (3.0 mL) sequentially under nitrogen atmosphere. The Schlenk tube was then equipped with a condenser and the outlet connected to the vacuum line with a nitrogen flow was closed (For an apparatus, see Fig.1 in SI). The reaction was complete after being stirred at 130 ^oC for 16 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 3/1). Then the resulting mixture was diluted with ethyl acetate (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 ethyl acetate (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 = 3/1) on silica gel to afford (R_a)-**4aa** (246.5 mg, 49%) as a liquid: 98% de (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 95/5, 0.3 mL/min, λ = 214 nm, t_R(major) = 24.9 min, t_R(minor) = 26.8 min); $[\alpha]_D^{20}$ = - 32.2 (*c* = 1.07, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$)

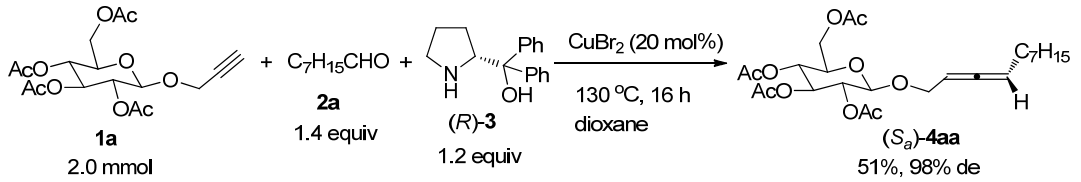
δ 5.28-4.95 (m, 5 H), 4.64 (d, *J* = 7.8 Hz, 1 H), 4.35-4.24 (m, 2 H), 4.18-4.07 (m, 2 H), 3.74-3.63 (m, 1 H), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.12-1.94 (m, 2 H, CH_2), 1.48-1.20 (m, 10 H, CH_2 × 5), 0.89 (t, *J* = 6.9 Hz, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.4, 170.5, 170.2, 169.3, 169.2, 98.8, 92.1, 87.2, 72.8, 71.6, 71.0, 68.2, 67.8, 61.7, 31.7, 28.98, 28.95, 28.9, 28.3, 22.5, 20.6, 20.52, 20.45, 20.4, 13.9; IR (neat) ν (cm^{-1}) 2928, 2856, 1963, 1757, 1435, 1370, 1226, 1165, 1041; MS (ESI, m/z) 521 ($M+Na^+$), 516 ($M+NH_4^+$); Anal. Calcd. for $C_{25}H_{38}O_{10}$ (%): C 60.23, H 7.68; Found: C 60.21, H 7.37.

(2) Preparation of (R_a)-4aa on 5.0 mmol scale. hx-10-97



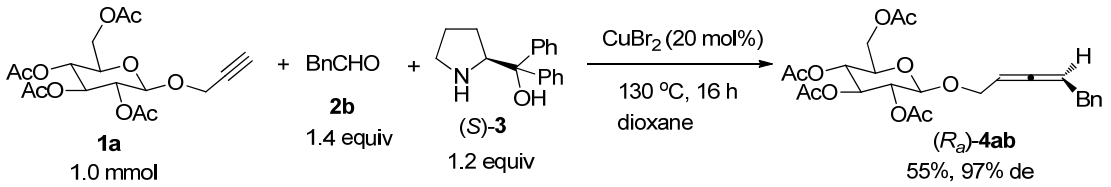
The reaction of $CuBr_2$ (0.2242 g, 1.0 mmol), **1a** (1.9282 g, 5.0 mmol), (*S*)-**3** (1.5181 g, 6.0 mmol), and **2a** (0.8974 g, 7.0 mmol) in dioxane (15.0 mL) afforded (R_a)-**4aa** (1.3199 g, 53%) (eluent: petroleum ether/ethyl acetate = 3/1) as a liquid: 98% de (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 95/5, 0.4 mL/min, λ = 214 nm, t_R(major) = 35.2 min, t_R(minor) = 37.2 min); $[\alpha]_D^{20}$ = -29.8 (*c* = 1.16, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.27-4.97 (m, 5 H), 4.64 (d, *J* = 7.8 Hz, 1 H), 4.34-4.25 (m, 2 H), 4.17-4.07 (m, 2 H), 3.73-3.64 (m, 1 H), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.10-1.96 (m, 2 H, CH_2), 1.48-1.22 (m, 10 H, CH_2 × 5), 0.89 (t, *J* = 6.8 Hz, 3 H, Me).

(3) Preparation of (S_a)-4aa on 2.0 mmol scale. hx-10-98



The reaction of $CuBr_2$ (89.3 mg, 0.4 mmol), **1a** (772.5 mg, 2.0 mmol), (*R*)-**3** (607.6 mg, 2.4 mmol), and **2a** (360.6 mg, 2.8 mmol) in dioxane (6.0 mL) afforded (S_a)-**4aa** (508.2 mg, 51%) (eluent: petroleum ether/ethyl acetate = 3/1) as a liquid: 98% de (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 95/5, 0.4 mL/min, λ = 214 nm, t_R(minor) = 35.5 min, t_R(major) = 36.5 min); $[\alpha]_D^{20}$ = +36.8 (*c* = 0.975, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.28-4.95 (m, 5 H), 4.64 (d, *J* = 7.8 Hz, 1 H), 4.34-4.25 (m, 2 H), 4.18-4.07 (m, 2 H), 3.75-3.66 (m, 1 H), 2.08 (s, 3 H, Me), 2.04 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.00 (s, 3 H, Me), 2.12-1.95 (m, 2 H, CH_2), 1.48-1.18 (m, 10 H, CH_2 × 5), 0.88 (t, *J* = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.1, 170.3, 169.9, 169.1, 169.0, 98.7, 92.0, 87.1, 72.6, 71.5, 70.9, 68.1, 67.5, 61.6, 31.5, 28.8, 28.7, 28.1, 22.3, 20.39, 20.35, 20.3, 13.8; IR (neat) ν (cm^{-1}) 2929, 2857, 1962, 1759, 1435, 1367, 1227, 1166, 1040; MS (ESI, m/z) 516 ($M+NH_4^+$); Anal. Calcd. for $C_{25}H_{38}O_{10}$ (%): C 60.23, H 7.68; Found: C 60.61, H 7.71.

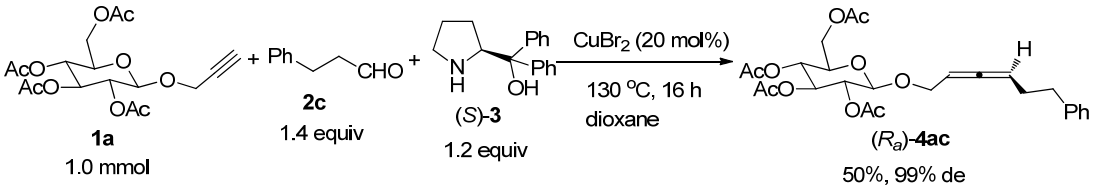
2. Preparation of (R_a)-4ab. hx-10-33



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1a** (388.1 mg, 1.0 mmol), (*S*)-**3** (303.3 mg, 1.2 mmol), and **2b** (168.8 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ab** (273.0 mg, 55%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a liquid:

97% de (HPLC conditions: Chiralcel IA-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 16.4 min, t_R(minor) = 23.0 min); $[\alpha]_D^{20}$ = -30.4 (*c* = 1.405, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39-7.19 (m, 5 H, ArH), 5.50-5.37 (m, 1 H), 5.22-5.11 (m, 2 H), 5.06 (t, *J* = 9.6 Hz, 1 H), 4.95 (t, *J* = 8.9 Hz, 1 H), 4.35 (d, *J* = 7.8 Hz, 1 H), 4.32-4.19 (m, 2 H), 4.13-4.02 (m, 2 H), 3.55-3.29 (m, 3 H), 2.07 (s, 3 H, Me), 2.029 (s, 3 H, Me), 2.026 (s, 3 H, Me), 2.00 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.7, 170.4, 170.0, 169.15, 169.06, 139.2, 128.34, 128.28, 126.3, 98.2, 91.5, 88.0, 72.5, 71.3, 70.8, 68.0, 67.0, 61.5, 34.7, 20.5, 20.42, 20.35; IR (neat) ν (cm^{-1}) 3063, 3028, 2945, 2884, 1964, 1756, 1602, 1495, 1450, 1433, 1370, 1226, 1165, 1041; MS (ESI, m/z) 529 ($M+K^+$), 513 ($M+Na^+$), 508 ($M+NH_4^+$); Anal. Calcd. for $C_{25}H_{30}O_{10}$ (%): C 61.22, H 6.16; Found: C 61.32, H 6.03.

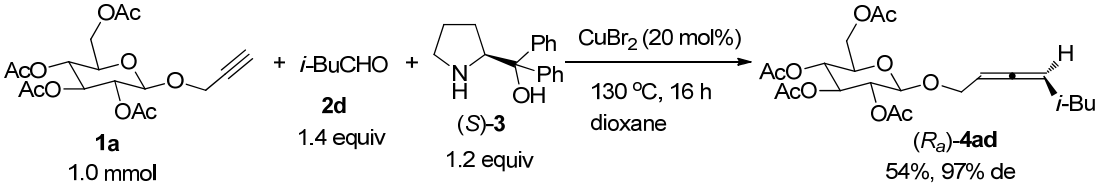
3. Preparation of (R_a)-4ac. xc-12-18



The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1a** (386.1 mg, 1.0 mmol), (*S*)-**3** (303.5 mg, 1.2 mmol), and **2c** (187.6 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ac** (253.4 mg, 50%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a liquid: 99% de (HPLC conditions: Chiralcel OD-H column, hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 214 nm, t_R(major) = 13.1 min, t_R(minor) = 19.3 min); $[\alpha]_D^{20}$ = -37.7 (*c* = 1.32, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34-7.15 (m, 5 H), 5.31-4.95 (m, 5 H, ArH), 4.57 (d, *J* = 8.1 Hz, 1 H), 4.32-4.18 (m, 2 H), 4.16-4.01 (m, 2 H), 3.70-3.61 (m, 1 H), 2.74 (t, *J* = 7.7 Hz, 2 H), 2.40-2.28 (m, 2 H), 2.07 (s, 3 H, Me), 2.03 (s, 3 H,

Me), 2.02 (s, 3 H, Me), 2.00 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.3, 170.5, 170.2, 169.3, 169.2, 141.2, 128.4, 128.2, 125.9, 99.2, 91.5, 87.9, 72.8, 71.7, 71.2, 68.3, 67.7, 61.8, 35.1, 30.0, 20.59, 20.56, 20.49, 20.47; IR (neat) ν (cm^{-1}) 3063, 3024, 2942, 2861, 1964, 1757, 1603, 1496, 1453, 1432, 1369, 1225, 1165, 1041; MS (ESI, m/z) 527 ($M+Na^+$), 522 ($M+NH_4^+$); Anal. Calcd. for $C_{26}H_{32}O_{10}$ (%): C 61.90, H 6.39; Found: C 61.41, H 6.25. HRMS calcd. for $C_{26}H_{36}NO_{10}$ ($M+NH_4^+$): 522.2334; Found: 522.2322.

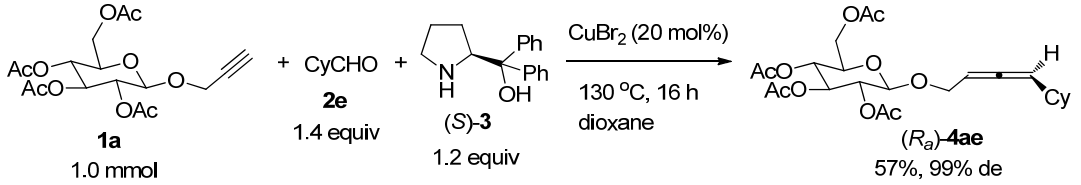
4. Preparation of (R_a)-4ad. hx-10-30



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1a** (385.4 mg, 1.0 mmol), (*S*)-**3** (304.1 mg, 1.2 mmol), and **2d** (121.2 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ad** (245.2 mg, 54%) (eluent: petroleum ether/ethyl acetate = 3/1) as a solid: 97% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(minor) = 17.6 min, t_R(major) = 18.9 min); $[\alpha]_D^{20}$ = -26.7 (*c* = 1.00, $CHCl_3$); m.p. 68-69 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.28-4.95 (m, 5 H), 4.63 (d, *J* = 8.1 Hz, 1 H), 4.36-4.21 (m, 2 H), 4.20-4.04 (m, 2 H), 3.73-3.61 (m, 1 H), 2.09 (s, 3 H, Me), 2.06 (s, 3 H, Me), 2.04 (s, 3 H, Me), 2.02 (s, 3 H, Me), 2.17-1.87 (m, 2 H, CH_2), 1.76-1.59 (m, 1 H, CH), 0.94 (d, *J* = 6.6 Hz, 6 H, Me × 2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.9, 170.5, 170.2, 169.23, 169.16, 98.8, 90.5, 86.5, 72.7, 71.6, 71.0, 68.1, 67.8, 61.7, 37.8, 28.1, 22.0, 21.9, 20.54, 20.51, 20.44, 20.42; IR (KBr) ν (cm^{-1}) 2957, 2871, 1964, 1757, 1434, 1369, 1226, 1165, 1041; MS

(ESI, m/z) 479 ($M+Na^+$), 474 ($M+NH_4^+$); Anal. Calcd. for $C_{22}H_{32}O_{10}$ (%): C 57.88, H 7.07; Found: C 57.89, H 7.08.

5. Preparation of (R_a)-4ae. hx-10-34



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1a** (388.5 mg, 1.0 mmol), (*S*)-**3** (305.5 mg, 1.2 mmol), and **2e** (157.5 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ae** (275.1 mg, 57%) (eluent: petroleum ether/ethyl acetate = 3/1) as a solid: 99% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(minor) = 16.4 min, t_R(major) = 19.1 min); $[\alpha]_D^{20}$ = -37.5 (*c* = 0.92, $CHCl_3$); m.p. 102-103 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.27-4.96 (m, 5 H), 4.66 (d, *J* = 8.1 Hz, 1 H), 4.36-4.22 (m, 2 H), 4.19-4.05 (m, 2 H), 3.72-3.63 (m, 1 H), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.22-1.89 (m, 1 H, CH), 1.81-1.60 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.40-1.00 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.3, 170.4, 170.0, 169.2, 169.1, 98.7, 98.0, 88.1, 72.7, 71.5, 71.0, 68.1, 67.8, 61.6, 36.6, 32.8, 32.6, 25.8, 25.6, 20.5, 20.42, 20.36; IR (KBr) ν (cm^{-1}) 2925, 2851, 1965, 1741, 1447, 1412, 1380, 1287, 1260, 1227, 1171, 1115, 1094, 1058, 1036; MS (ESI, m/z) 505 ($M+Na^+$), 500 ($M+NH_4^+$); Anal. Calcd. for $C_{24}H_{34}O_{10}$ (%): C 59.74, H 7.10; Found: C 59.80, H 7.04.

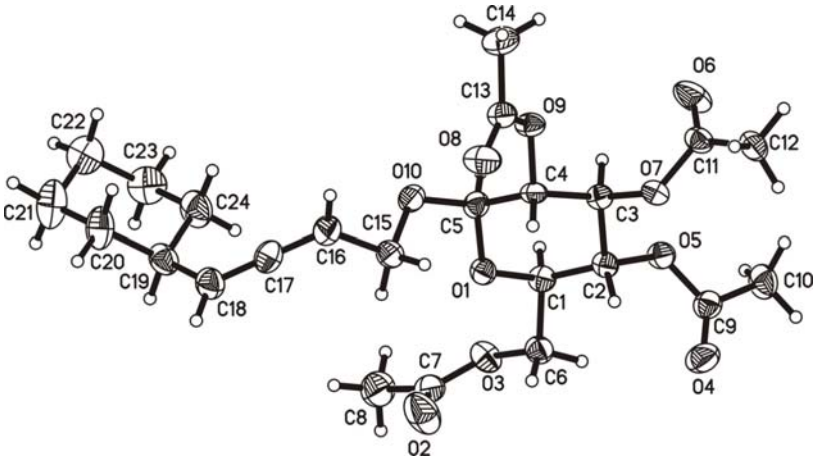
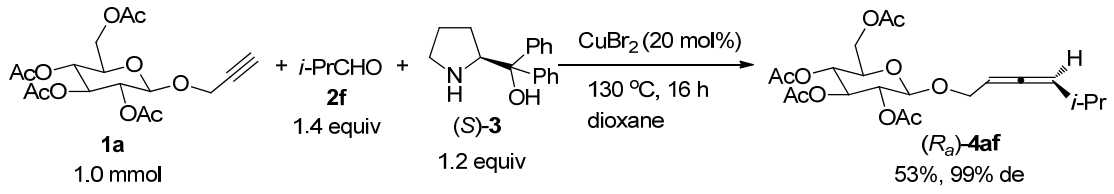


Figure S2. ORTEP representation of (R_a)-4ae.

Crystal data for compound (R_a)-**4ae**: $C_{24}H_{34}O_{10}$, MW = 482.51, orthorhombic, space group *P 21 21 21*, final *R* indices [I > 2σ(I)], *R1* = 0.0800, *wR2* = 0.2036, *R* indices (all data) *R1* = 0.1020, *wR2* = 0.2267, a = 5.8507(10) Å, b = 14.177(2) Å, c = 30.834(4) Å, α = 90.00°, β = 90.00°, γ = 90.00°, V = 2557.5(7) Å^3, T = 293(2) K, Z = 4, reflections collected/unique 15536 / 2706 (Rint = 0.1325), number of observations [> 2σ(I)] 2010, parameters: 313. CCDC 1013803 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

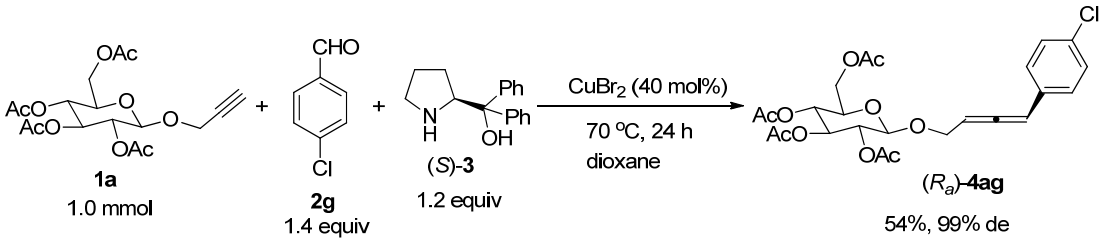
6. Preparation of (R_a)-4af. hx-10-40



The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1a** (384.2 mg, 1.0 mmol), (*S*)-**3** (305.5 mg, 1.2 mmol), and **2f** (101.4 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4af** (233.9 mg, 53%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a liquid:

99% de (HPLC conditions: Chiralcel IA-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(minor) = 15.1 min, t_R(major) = 16.1 min); $[\alpha]_D^{20}$ = -23.1 (*c* = 1.08, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.30-5.14 (m, 3 H), 5.10 (t, *J* = 9.6 Hz, 1 H), 5.01 (dd, J_1 = 9.6 Hz, J_2 = 8.1 Hz, 1 H), 4.66 (d, *J* = 7.8 Hz, 1 H), 4.36-4.24 (m, 2 H), 4.17-4.07 (m, 2 H), 3.72-3.64 (m, 1 H), 2.40-2.24 (m, 1 H, CH), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 1.03 (d, *J* = 6.6 Hz, 6 H, Me × 2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.0, 170.5, 170.2, 169.3, 169.2, 99.5, 98.7, 88.5, 72.8, 71.6, 71.1, 68.2, 67.9, 61.8, 27.5, 22.3, 22.2, 20.6, 20.51, 20.45, 20.4; IR (neat) ν (cm^{-1}) 2962, 2871, 1961, 1755, 1434, 1367, 1227, 1165, 1040; MS (ESI, m/z) 460 ($M+NH_4^+$); Anal. Calcd. for $C_{21}H_{30}O_{10}$ (%): C 57.01, H 6.83; Found: C 57.05, H 6.72.

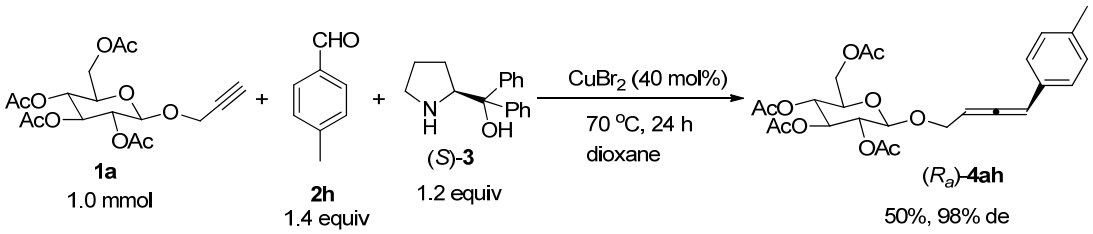
7. Preparation of (R_a)-4ag. hx-11-91, hx-10-53



The reaction of $CuBr_2$ (89.7 mg, 0.4 mmol), **1a** (386.8 mg, 1.0 mmol), (*S*)-**3** (304.6 mg, 1.2 mmol), and **2g** (196.8 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ag** (277.3 mg, 54%) (eluent: petroleum ether/ethyl acetate = 2/1) as a liquid: 99% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 0.6 mL/min, λ = 214 nm, t_R(major) = 57.1 min, t_R(minor) = 62.3 min); $[\alpha]_D^{20}$ = -111.8 (*c* = 1.04, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34-7.27 (m, 2 H, ArH), 7.25-7.19 (m, 2 H, ArH), 6.23 (dt, J_1 = 6.2 Hz, J_2 = 2.3 Hz, 1 H, one proton of CH=C=CH), 5.65 (dd, J_1 = 13.8 Hz, J_2 = 6.3 Hz, 1 H, one proton of CH=C=CH), 5.22 (t, *J* = 9.5 Hz, 1 H),

5.14-4.97 (m, 2 H), 4.65 (d, J = 7.8 Hz, 1 H), 4.47-4.37 (m, 1 H), 4.29-4.18 (m, 2 H), 4.10 (dd, J_1 = 12.3 Hz, J_2 = 2.4 Hz, 1 H), 3.66-3.58 (m, 1 H), 2.03 (s, 3 H, Me), 2.02 (s, 3 H, Me), 2.00 (s, 3 H, Me), 1.99 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 206.4, 170.3, 169.9, 169.1, 169.0, 132.8, 132.0, 128.7, 127.9, 99.2, 94.9, 91.8, 72.7, 71.7, 71.1, 68.2, 66.6, 61.7, 20.4, 20.3; IR (neat) ν (cm^{-1}) 2956, 2925, 2869, 2849, 1953, 1755, 1492, 1456, 1429, 1376, 1224, 1039; MS (ESI, m/z) 535 (M(^{37}Cl)+Na^+), 533 (M(^{35}Cl)+Na^+), 530 (M(^{37}Cl)+NH_4^+), 528 (M(^{35}Cl)+NH_4^+); HRMS calcd. for $C_{24}H_{31}{}^{35}ClNO_{10}$ (M(^{35}Cl)+NH_4^+): 528.1631; Found: 528.1614.

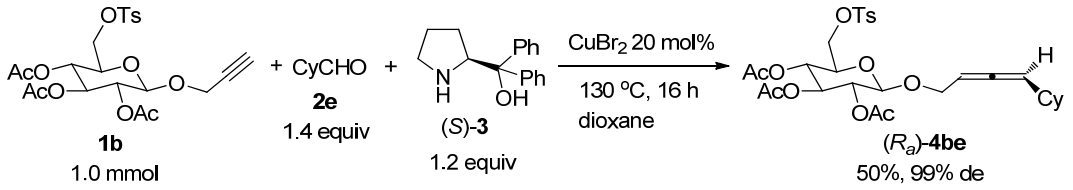
8. Preparation of (R_a)-4ah. hx-12-174, hx-10-62



The reaction of $CuBr_2$ (89.6 mg, 0.4 mmol), **1a** (385.8 mg, 1.0 mmol), (*S*)-**3** (304.2 mg, 1.2 mmol), and **2h** (169.1 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ah** (244.0 mg, 50%) (eluent: petroleum ether/ethyl acetate = 2/1) as a liquid: 98% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 94/6, 0.6 mL/min, λ = 214 nm, t_R(major) = 91.9 min, t_R(minor) = 99.9 min); $[\alpha]_D^{20}$ = -90.4 (c = 0.81, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25-7.09 (m, 4 H, ArH), 6.28-6.20 (m, 1 H, one proton of CH=C=CH), 5.64-5.54 (m, 1 H, one proton of CH=C=CH), 5.22 (t, J = 9.5 Hz, 1 H), 5.14-4.97 (m, 2 H), 4.66 (d, J = 7.8 Hz, 1 H), 4.41 (ddd, J_1 = 12.0 Hz, J_2 = 6.0 Hz, J_3 = 2.4 Hz, 1 H), 4.29-4.17 (m, 2 H), 4.05 (dd, J_1 = 12.3 Hz, J_2 = 2.1 Hz, 1 H), 3.55 (ddd, J_1 = 9.9 Hz, J_2 = 4.5 Hz, J_3 = 2.4 Hz, 1 H), 2.34 (s, 3 H, Me), 2.02 (s,

6 H, Me × 2), 2.01 (s, 3 H, Me), 2.00 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 206.4, 170.3, 169.9, 169.1, 169.0, 136.9, 130.2, 129.2, 126.5, 98.7, 95.3, 90.9, 72.6, 71.5, 70.9, 68.0, 66.8, 61.4, 20.9, 20.33, 20.30, 20.26; IR (neat) ν (cm^{-1}) 2944, 2869, 1950, 1755, 1513, 1432, 1367, 1227, 1166, 1040; MS (ESI, m/z) 529 ($M+K^+$), 513 ($M+Na^+$), 508 ($M+NH_4^+$); HRMS calcd. for $C_{25}H_{34}NO_{10}$ ($M+NH_4^+$): 508.2177; Found: 508.2164.

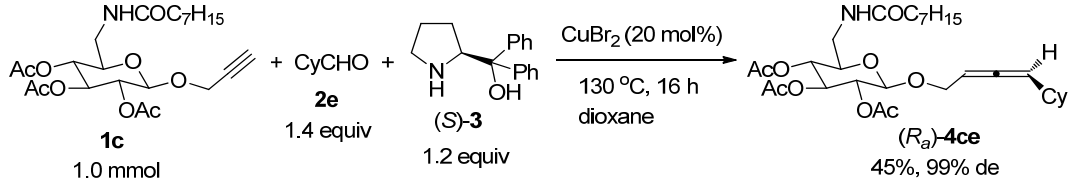
9. Preparation of (R_a)-4be. hx-10-57



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1b** (498.9 mg, 1.0 mmol), (*S*)-**3** (304.1 mg, 1.2 mmol), and **2e** (156.9 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (*R*)-**4be** (297.4 mg, 50%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a solid: 99% de (HPLC conditions: Chiralcel IA-H column, hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 214 nm, t_R(minor) = 9.5 min, t_R(major) = 10.9 min); $[\alpha]_D^{20}$ = -21.6 (*c* = 0.97, $CHCl_3$); m.p. 117-118 °C (EtOAc/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 2 H, ArH), 7.35 (d, *J* = 8.1 Hz, 2 H, ArH), 5.27-5.08 (m, 3 H), 4.97-4.86 (m, 2 H), 4.58 (d, *J* = 8.1 Hz, 1 H), 4.27-4.17 (m, 1 H), 4.16-3.97 (m, 3 H), 3.77-3.68 (m, 1 H), 2.45 (s, 3 H), 2.03 (s, 3 H, Me), 2.00 (s, 3 H, Me), 1.99 (s, 3 H, Me), 2.10-1.95 (m, 1 H, CH), 1.82-1.59 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.40-0.98 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.2, 170.1, 169.3, 169.1, 145.0, 132.2, 129.8, 127.9, 98.7, 98.2, 88.2, 72.4, 71.3, 70.8, 68.4, 67.9, 67.5, 36.6, 32.8, 32.7, 25.9, 25.7, 21.5, 20.47, 20.42, 20.39; IR (KBr)

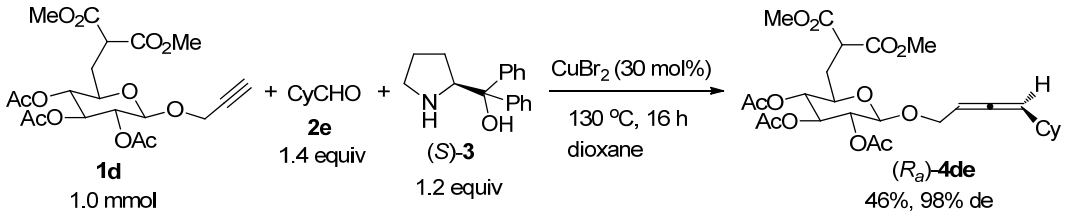
ν (cm^{-1}) 2926, 2852, 1962, 1758, 1598, 1449, 1369, 1245, 1218, 1178, 1040; MS (ESI, m/z) 612 ($M+NH_4^+$); Anal. Calcd. for $C_{29}H_{38}O_{11}S$ (%): C 58.57, H 6.44; Found: C 58.81, H 6.38.

10. Preparation of (R_a)-4ce. hx-10-56



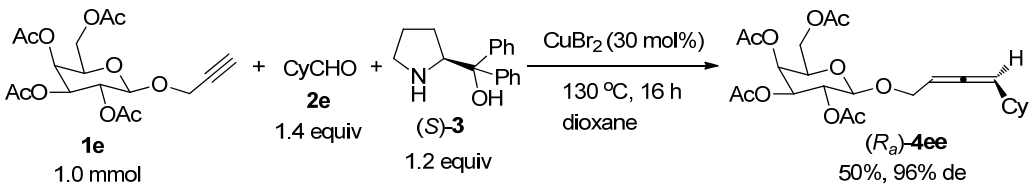
The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1c** (472.5 mg, 1.0 mmol), (*S*)-**3** (303.2 mg, 1.2 mmol), and **2e** (158.1 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ce** (256.2 mg, 45%) (eluent: petroleum ether/ethyl acetate = 1.5/1) as a liquid: 99% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(minor) = 27.3 min, t_R(major) = 29.6 min); $[\alpha]_D^{20}$ = -38.9 (*c* = 1.35, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.89 (t, *J* = 5.4 Hz, 1 H), 5.27-5.13 (m, 3 H), 5.02-4.85 (m, 2 H), 4.62 (d, *J* = 8.1 Hz, 1 H), 4.34-4.24 (m, 1 H), 4.18-4.07 (m, 1 H), 3.61-3.40 (m, 3 H), 2.23-2.11 (m, 2 H), 2.06 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.00 (s, 3 H, Me), 2.10-1.95 (m, 1 H, CH), 1.82-1.54 (m, 7 H, CH_2 × 3 and one proton of CH_2), 1.39-1.00 (m, 13 H, CH_2 × 6 and one proton of CH_2), 0.88 (t, *J* = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.3, 173.1, 170.1, 169.5, 169.2, 99.2, 98.3, 88.3, 72.7, 72.4, 71.1, 68.7, 68.4, 38.8, 36.7, 36.5, 32.9, 32.7, 31.5, 29.1, 28.9, 25.9, 25.7, 25.4, 22.5, 20.52, 20.47, 13.9; IR (neat) ν (cm^{-1}) 3312, 2926, 2853, 1961, 1760, 1651, 1538, 1447, 1373, 1248, 1220, 1165, 1050; MS (ESI, m/z) 604 ($M+K^+$), 588 ($M+Na^+$), 566 ($M+H^+$); Anal. Calcd. for $C_{30}H_{47}NO_9$ (%): C 63.70, H 8.37, N 2.48; Found: C 63.60, H 8.39, N 2.29.

11. Preparation of (R_a)-4de. hx-10-76



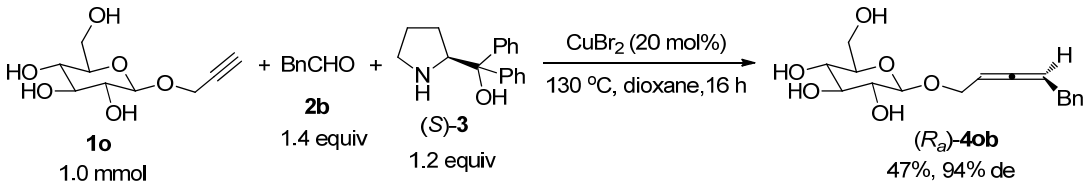
The reaction of $CuBr_2$ (67.0 mg, 0.3 mmol), **1d** (457.3 mg, 1.0 mmol), (*S*)-**3** (304.0 mg, 1.2 mmol), and **2e** (157.3 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4de** (255.0 mg, 46%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a liquid: 98% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 80/20, 0.5 mL/min, λ = 214 nm, t_R(minor) = 12.2 min, t_R(major) = 14.3 min); $[\alpha]_D^{20}$ = -30.0 (*c* = 1.375, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.26-5.12 (m, 3 H), 4.96 (dd, J_1 = 9.8 Hz, J_2 = 8.0 Hz, 1 H), 4.88 (t, *J* = 9.5 Hz, 1 H), 4.52 (d, *J* = 8.1 Hz, 1 H), 4.25 (ddd, J_1 = 11.4 Hz, J_2 = 6.3 Hz, J_3 = 2.7 Hz, 1 H), 4.06 (ddd, J_1 = 11.7 Hz, J_2 = 7.5 Hz, J_3 = 2.4 Hz, 1 H), 3.76 (s, 3 H, Me), 3.74 (s, 3 H, Me), 3.67 (dd, J_1 = 9.6 Hz, J_2 = 5.1 Hz, 1 H), 3.52 (td, J_1 = 9.6 Hz, J_2 = 2.9 Hz, 1 H, CH), 2.30-2.19 (m, 1 H), 2.13-1.94 (m, 11 H), 1.81-1.60 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.40-1.00 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.1, 170.1, 169.5, 169.2, 169.1, 169.0, 99.2, 98.1, 88.3, 72.6, 71.6, 71.1, 71.0, 68.1, 52.6, 52.5, 47.4, 36.6, 32.8, 32.7, 30.2, 25.9, 25.7, 20.52, 20.49, 20.4; IR (neat) ν (cm^{-1}) 2927, 2852, 1961, 1755, 1436, 1367, 1245, 1218, 1159, 1046; MS (ESI, m/z) 577 ($M+Na^+$), 572 ($M+NH_4^+$); Anal. Calcd. for $C_{27}H_{38}O_{12}$ (%): C 58.47, H 6.91; Found: C 58.04, H 6.68. HRMS calcd. for $C_{27}H_{42}NO_{12}$ ($M+NH_4^+$): 572.2702; Found: 572.2688.

12. Preparation of (R_a)-4ee. hx-10-75



The reaction of $CuBr_2$ (67.3 mg, 0.3 mmol), **1e** (388.0 mg, 1.0 mmol), (*S*)-**3** (303.7 mg, 1.2 mmol), and **2e** (158.0 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ee** (240.3 mg, 50%) (eluent: petroleum ether/ethyl acetate = 3/1) as a liquid: 96% de (HPLC conditions: Chiralcel AD-H column, hexane/*i*-PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R(major) = 20.0 min, t_R(minor) = 23.5 min); $[\alpha]_D^{20}$ = -26.7 (*c* = 1.24, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.40 (d, *J* = 3.0 Hz, 1 H), 5.27-5.13 (m, 3 H), 5.02 (dd, J_1 = 10.4 Hz, J_2 = 3.2 Hz, 1 H), 4.62 (d, *J* = 8.1 Hz, 1 H), 4.37-4.27 (m, 1 H), 4.23-4.07 (m, 3 H), 3.89 (t, *J* = 6.6 Hz, 1 H), 2.16 (s, 3 H, Me), 2.09 (s, 3 H, Me), 2.06 (s, 3 H, Me), 1.99 (s, 3 H, Me), 2.20-1.93 (m, 1 H, CH), 1.83-1.60 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.40-0.99 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.3, 170.12, 170.09, 170.0, 169.2, 99.2, 98.0, 88.2, 70.8, 70.4, 68.6, 67.8, 66.8, 61.1, 36.6, 32.8, 32.6, 25.8, 25.7, 20.5, 20.43, 20.36; IR (neat) ν (cm^{-1}) 2926, 2852, 1961, 1754, 1449, 1370, 1223, 1170, 1132, 1075, 1057; MS (ESI, m/z) 505 ($M+Na^+$), 500 ($M+NH_4^+$); Anal. Calcd. for $C_{24}H_{34}O_{10}$ (%): C 59.74 H 7.10; Found: C 59.77, H 6.97.

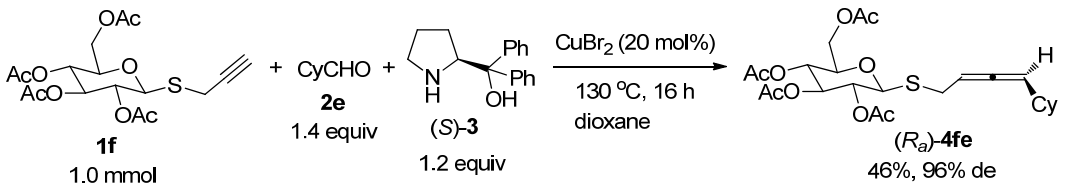
13. Preparation of (R_a)-4lb. hx-12-190



To a flame-dried Schlenk tube were added $CuBr_2$ (44.8 mg, 0.2 mmol), **1o** (221.3

mg, 1.0 mmol), (*S*)-**3** (304.2 mg, 1.2 mmol), and **2b** (168.7 mg, 1.4 mmol)/dioxane (3.0 mL) sequentially under nitrogen atmosphere. The Schlenk tube was then equipped with a condenser and the outlet connected to the vacuum line with a nitrogen flow was closed (For an apparatus, see Fig.1 in SI). The reaction was complete after being stirred at 130 ^{o}C for 16 h as monitored by TLC (eluent: EtOAc/MeOH = 6/1). After cooling to room temperature, the crude reaction mixture was filtrated through a short pad of silica gel eluted with MeOH (30 mL). After evaporation, the residue was purified by chromatography on silica gel to afford (R_a)-**4ob** (152.0 mg, 47%) (eluent: EtOAc/MeOH = 8/1) as a syrup: 94% de (HPLC conditions: Chiralcel OJ column, hexane/*i*-PrOH = 80/20, 1.0 mL/min, λ = 214 nm, t_R(major) = 9.1 min, t_R(minor) = 19.0 min); $[\alpha]_D^{20}$ = -36.2 (*c* = 0.79, MeOH); ^{1}H NMR (300 MHz, d_6-DMSO) δ 7.44-7.13 (m, 5 H, ArH), 5.53-5.42 (m, 1 H), 5.42-5.31 (m, 1 H), 5.13-4.92 (m, 3 H), 4.60 (t, *J* = 5.6 Hz, 1 H), 4.35-4.19 (m, 2 H), 4.17-4.04 (m, 1 H), 3.71 (dd, J_1 = 11.3 Hz, J_2 = 5.6 Hz, 1 H), 3.56-3.31 (m, 3 H), 3.27-2.95 (m, 4 H); ^{13}C NMR (75 Hz, d_6-DMSO) δ 205.6, 140.8, 129.38, 129.35, 127.2, 102.6, 92.3, 89.9, 77.9, 77.7, 74.4, 71.0, 67.2, 62.0, 35.6; IR (neat) ν (cm^{-1}) 3389, 3027, 2971, 2920, 2881, 1965, 1602, 1494, 1453, 1417, 1354, 1273, 1156, 1077, 1046, 1025; MS (ESI, m/z) 367 (M+COOH$^-$); HRMS calcd. for $C_{17}H_{22}O_6{}^{35}Cl$ (M+(^{35}Cl)$^-$): 357.1110; Found: 357.1109.

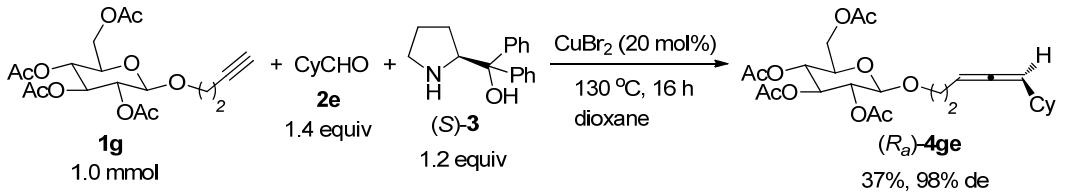
14. Preparation of (R_a)-4fe. hx-10-55



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1f** (400.8 mg, 1.0 mmol), (*S*)-**3** (304.6 mg, 1.2 mmol), and **2e** (157.2 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4fe** (230.3 mg, 46%) (eluent: petroleum ether/ethyl acetate = 3/1) as a solid: 96% de (HPLC conditions: Chiralcel AS-H column, hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 214 nm, t_R(minor) = 23.0 min, t_R(major) = 24.5 min); $[\alpha]_D^{20}$ = -47.9 (*c* = 1.24, $CHCl_3$); m.p. 103-104 °C (EtOAc/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.27-5.14 (m, 3 H), 5.14-5.01 (m, 2 H), 4.58 (d, J = 9.9 Hz, 1 H), 4.25 (dd, J_1 = 12.5 Hz, J_2 = 5.0 Hz, 1 H), 4.13 (dd, J_1 = 12.2 Hz, J_2 = 2.0 Hz, 1 H), 3.71-3.62 (m, 1 H), 3.35 (ddd, J_1 = 13.8 Hz, J_2 = 7.5 Hz, J_3 = 2.3 Hz, 1 H), 3.24 (ddd, J_1 = 13.8 Hz, J_2 = 6.5 Hz, J_3 = 3.0 Hz, 1 H), 2.08 (s, 3 H, Me), 2.07 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.02 (s, 3 H, Me), 2.12-1.94 (m, 1 H, CH), 1.82-1.60 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.40-1.00 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 203.7, 170.5, 170.0, 169.2, 98.5, 88.8, 82.7, 75.7, 73.8, 69.7, 68.1, 61.9, 37.2, 33.0, 32.9, 30.1, 25.9, 25.7, 20.6, 20.5, 20.4; IR (KBr) ν (cm^{-1}) 2926, 2852, 1953, 1756, 1448, 1371, 1225, 1040; MS (ESI, m/z) 516 ($M+NH_4^+$); Anal. Calcd. for $C_{24}H_{34}O_9S$ (%): C 57.81, H 6.87; Found: C 58.03, H 6.82.

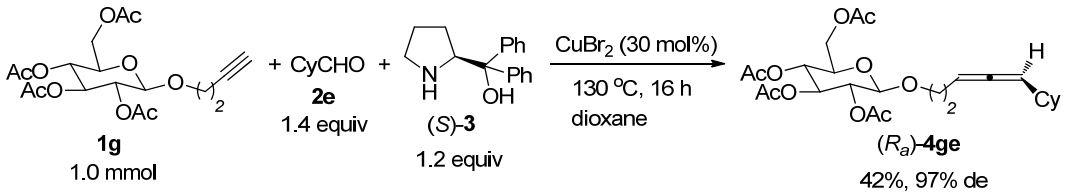
15. Preparation of (R_a)-4ge.

(1) Preparation of (R_a)-4ge with $CuBr_2$ 20 mol%. hx-10-77



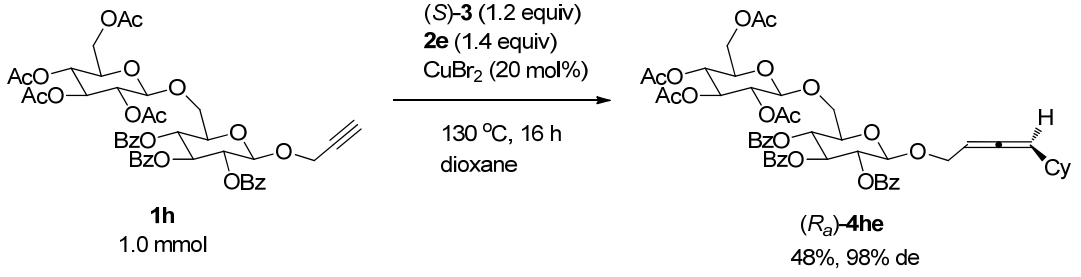
The reaction of $CuBr_2$ (44.7 mg, 0.2 mmol), **1g** (402.0 mg, 1.0 mmol), (*S*)-**3** (306.1 mg, 1.2 mmol), and **2e** (157.2 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ge** (183.2 mg, 37%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a solid: 98% de (HPLC conditions: Chiralcel IC column, hexane/*i*-PrOH = 96/4, 0.4 mL/min, λ = 214 nm, t_R(minor) = 76.6 min, t_R(major) = 78.3 min); $[\alpha]_D^{20}$ = -47.8 (*c* = 1.38, $CHCl_3$); m.p. 86-87 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.21 (t, *J* = 9.5 Hz, 1 H), 5.14-4.94 (m, 4 H), 4.53 (d, *J* = 8.1 Hz, 1 H), 4.28 (dd, J_1 = 12.3 Hz, J_2 = 4.8 Hz, 1 H), 4.13 (dd, J_1 = 12.3 Hz, J_2 = 2.4 Hz, 1 H), 3.92 (dt, J_1 = 9.6 Hz, J_2 = 6.9 Hz, 1 H), 3.71 (ddd, J_1 = 10.1 Hz, J_2 = 4.8 Hz, J_1 = 2.4 Hz, 1 H), 3.55 (dt, J_1 = 9.6 Hz, J_2 = 7.2 Hz, 1 H), 2.32-2.20 (m, 2 H, CH_2), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.13-1.88 (m, 1 H, CH), 1.80-1.58 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.37-0.97 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 203.2, 170.6, 170.2, 169.3, 169.2, 100.7, 97.4, 87.4, 72.7, 71.6, 71.1, 69.7, 68.2, 61.8, 36.9, 32.9, 32.8, 29.3, 26.0, 25.8, 20.6, 20.5, 20.4; IR (KBr) ν (cm^{-1}) 2926, 2852, 1959, 1757, 1448, 1369, 1225, 1170, 1040; MS (ESI, m/z) 514 ($M+NH_4^+$); Anal. Calcd. for $C_{25}H_{36}O_{10}$ (%): C 60.47, H 7.31; Found: C 60.54, H 7.25.

(2) Preparation of (R_a)-4ge with $CuBr_2$ 30 mol%. hx-10-78



The reaction of $CuBr_2$ (67.4 mg, 0.3 mmol), **1g** (400.6 mg, 1.0 mmol), (*S*)-**3** (305.2 mg, 1.2 mmol), and **2e** (157.1 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ge** (210.4 mg, 42%) (eluent: petroleum ether/ethyl acetate = 2.5/1) as a solid: 97% de (HPLC conditions: Chiralcel IC column, hexane/*i*-PrOH = 96/4, 0.4 mL/min, λ = 214 nm, t_R(minor) = 75.4 min, t_R(major) = 77.0 min); $[\alpha]_D^{20}$ = -47.2 (*c* = 1.153, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.21 (t, *J* = 9.5 Hz, 1 H), 5.14-4.94 (m, 4 H), 4.53 (d, *J* = 7.8 Hz, 1 H), 4.28 (dd, J_1 = 12.3 Hz, J_2 = 4.5 Hz, 1 H), 4.13 (dd, J_1 = 12.3 Hz, J_2 = 2.4 Hz, 1 H), 3.92 (dt, J_1 = 9.6 Hz, J_2 = 6.9 Hz, 1 H), 3.71 (ddd, J_1 = 9.5 Hz, J_2 = 4.7 Hz, J_1 = 2.4 Hz, 1 H), 3.55 (dt, J_1 = 9.6 Hz, J_2 = 7.4 Hz, 1 H), 2.32-2.20 (m, 2 H, CH_2), 2.09 (s, 3 H, Me), 2.05 (s, 3 H, Me), 2.03 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.13-1.87 (m, 1 H, CH), 1.80-1.58 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.37-0.97 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 203.2, 170.6, 170.2, 169.3, 169.2, 100.7, 97.4, 87.4, 72.7, 71.6, 71.1, 69.7, 68.3, 61.8, 36.9, 32.93, 32.85, 29.3, 26.0, 25.8, 20.6, 20.54, 20.49, 20.47.

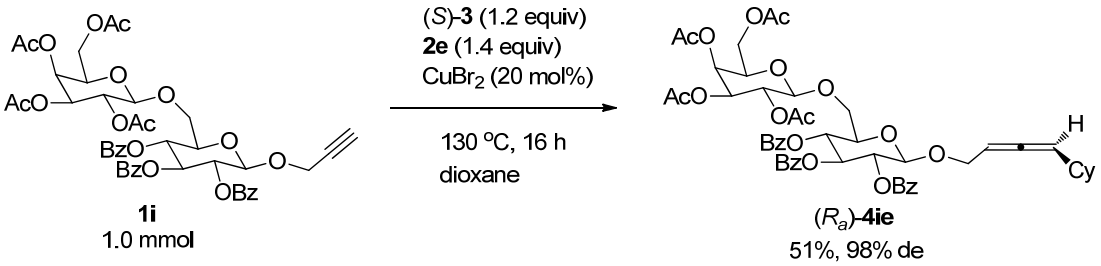
16. Preparation of (R_a)-4he. hx-10-58, hx-8-81



The reaction of $CuBr_2$ (44.9 mg, 0.2 mmol), **1h** (862.1 mg, 1.0 mmol), (*S*)-**3**

(304.4 mg, 1.2 mmol), and **2e** (157.5 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (*R*)-**4he** (456.7 mg, 48%) (eluent: petroleum ether/ethyl acetate = 1.5/1) as a syrup: 98% de (HPLC conditions: (Supercritical Fluid Chromatography) Chiralcel IA column, CO_2/*i*-PrOH = 80/20, 1.5 mL/min, λ = 214 nm, t_R(minor) = 8.3 min, t_R(major) = 14.2 min); $[\alpha]_D^{20}$ = -13.8 (*c* = 1.21, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98-7.88 (m, 4 H, ArH), 7.84-7.78 (m, 2 H, ArH), 7.55-7.45 (m, 2 H, ArH), 7.43-7.32 (m, 5 H, ArH), 7.28-7.20 (m, 2 H, ArH), 5.88 (t, *J* = 9.6 Hz, 1 H), 5.51 (dd, J_1 = 9.6 Hz, J_2 = 8.1 Hz, 1 H), 5.41 (t, *J* = 9.8 Hz, 1 H), 5.26-5.12 (m, 3 H), 5.11-4.98 (m, 2 H), 4.94 (d, *J* = 7.8 Hz, 1 H), 4.65 (d, *J* = 7.8 Hz, 1 H), 4.43-4.33 (m, 1 H), 4.29-4.12 (m, 2 H), 4.10-3.97 (m, 3 H), 3.78 (dd, *J* = 18.8 Hz, *J* = 11.3 Hz, 1 H), 3.74-3.65 (m, 1 H), 2.09 (s, 3 H, Me), 2.01 (s, 3 H, Me), 2.00 (s, 3 H, Me), 1.99 (s, 3 H, Me), 2.12-1.85 (m, 1 H, CH), 1.79-1.57 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.34-0.95 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.1, 170.3, 170.0, 169.2, 169.1, 165.5, 165.1, 164.8, 133.4. 133.02, 132.99, 129.6, 129.54, 129.47, 129.1, 128.5, 128.4, 128.3, 128.11, 128.05, 100.6, 99.2, 98.2, 88.2, 73.5, 72.8, 72.6, 71.6, 71.5, 70.9, 69.5, 68.2, 68.0, 67.7, 61.6, 36.5, 32.8, 32.5, 25.8, 25.7, 25.6, 20.5, 20.4, 20.3; IR (neat) ν (cm^{-1}) 3063, 2927, 2852, 1959, 1754, 1739, 1602, 1452, 1369, 1284, 1251, 1224, 1176, 1094, 1069, 1037; MS (MALDI, m/z) 995 ($M+K^+$) 979 ($M+Na^+$); Anal. Calcd. for $C_{51}H_{56}O_{18}$ (%): C 64.01, H 5.90; Found: C 64.00, H 5.80.

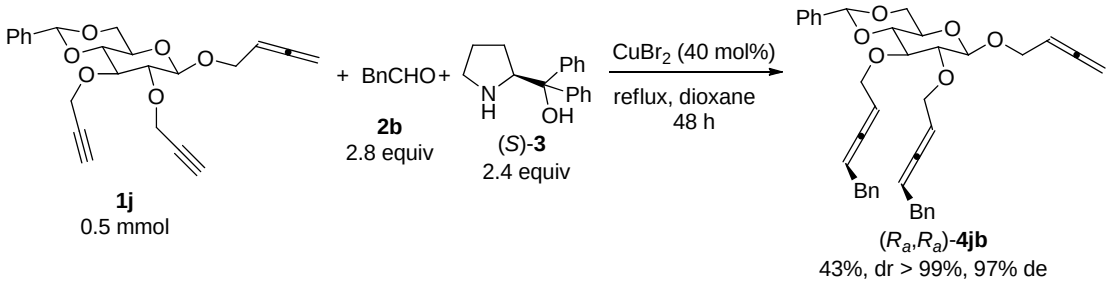
17. Preparation of (R_a)-4ie. hx-10-70, hx-8-98



The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1i** (861.1 mg, 1.0 mmol), (*S*)-**3** (304.2 mg, 1.2 mmol), and **2e** (157.2 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ie** (492.2 mg, 51%) (eluent: petroleum ether/ethyl acetate = 1.5/1) as a syrup: 98% de (HPLC conditions: (Supercritical Fluid Chromatography) Chiralcel IA column, CO_2/*i*-PrOH = 70/30, 1.5 mL/min, λ = 214 nm, t_R(minor) = 4.5 min, t_R(major) = 6.1 min); $[\alpha]_D^{20}$ = -13.5 (*c* = 1.12, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80-7.88 (m, 4 H, ArH), 7.85-7.78 (m, 2 H, ArH), 7.55-7.20 (m, 9 H, ArH), 5.88 (t, *J* = 9.8 Hz, 1 H), 5.51 (dd, J_1 = 9.8 Hz, J_2 = 8.0 Hz, 1 H), 5.46-5.36 (m, 2 H), 5.26 (dd, J_1 = 10.4 Hz, J_2 = 8.0 Hz, 1 H), 5.20-5.12 (m, 2 H), 5.03 (dd, J_1 = 10.4 Hz, J_2 = 3.5 Hz, 1 H), 4.95 (d, *J* = 7.8 Hz, 1 H), 4.62 (d, *J* = 7.8 Hz, 1 H), 4.44-4.34 (m, 1 H), 4.25-3.97 (m, 5 H), 3.92 (t, *J* = 6.5 Hz, 1 H), 3.79 (dd, J_1 = 10.8 Hz, J_2 = 7.5 Hz, 1 H), 2.110 (s, 3 H, Me), 2.107 (s, 3 H, Me), 2.01 (s, 3 H, Me), 1.98 (s, 3 H, Me), 2.18-1.85 (m, 1 H, CH), 1.79-1.57 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.34-0.95 (m, 5 H, CH_2 × 2 and one proton of CH_2); ^{13}C NMR (75 Hz, $CDCl_3$) δ 204.1, 170.1, 170.0, 169.9, 169.2, 165.5, 165.1, 164.8, 133.4, 133.02, 132.98, 129.5, 129.4, 129.0, 128.5, 128.4, 128.3, 128.1, 128.0, 101.0, 99.2, 98.2, 88.2, 73.5, 72.8, 71.5, 70.7, 70.4, 69.5, 68.4, 68.2, 67.7, 66.8, 61.0, 36.4, 32.8, 32.5, 25.8, 25.7, 25.6, 20.6, 20.4, 20.33, 20.28; IR (neat) ν (cm^{-1}) 3066, 2927, 2852, 1959, 1740, 1602, 1451, 1370, 1281, 1255, 1218, 1177, 1090, 1069; MS (MALDI, m/z) 979 ($M+Na^+$); Anal. Calcd. for $C_{51}H_{56}O_{18}$ (%): C

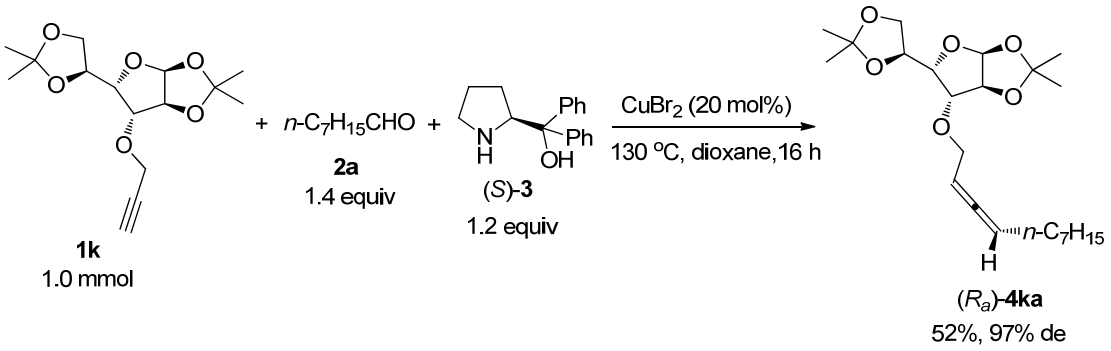
64.01, H 5.90; Found: C 64.04, H 5.88.

18. Preparation of (R_a,R_a)-4jb. xc-13-61



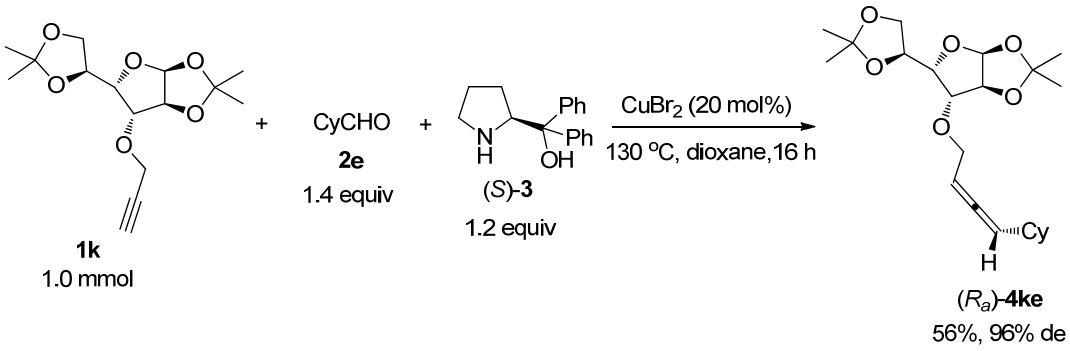
The reaction of $CuBr_2$ (45.1 mg, 0.2 mmol), **1j** (198.1 mg, 0.5 mmol), (*S*)-**3** (303.9 mg, 1.2 mmol), and **2b** (168.0 mg, 1.4 mmol) in dioxane (1.5 mL) afforded (R_a,R_a)-**4jb** (131.2 mg, 43%) (eluent: petroleum ether/ethyl acetate = 10/1) as an oil: dr > 99%, 97% de (HPLC conditions: (Supercritical Fluid Chromatography) Chiralcel OJ column, CO_2/*i*-PrOH = 80/20, 1.3 mL/min, λ = 214 nm, t_R(minor) = 24.7 min, t_R(major) = 27.1 min); $[\alpha]_D^{20}$ = -69.8 (*c* = 0.97, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.55-7.42 (m, 2 H, ArH), 7.41-7.08 (m, 13 H, ArH), 5.53 (s, 1 H), 5.43-5.18 (m, 5 H), 4.89-4.72 (m, 2 H), 4.50 (d, *J* = 7.8 Hz, 1 H, CH), 4.44-4.11 (m, 7 H), 3.76 (t, *J* = 10.2 Hz, 1 H), 3.66-3.50 (m, 2 H), 3.43-3.24 (m, 6 H); ^{13}C NMR (75 Hz, $CDCl_3$) δ 209.4, 205.2, 205.1, 140.03, 140.01, 137.2, 128.9, 128.4, 128.2, 126.2, 126.1, 126.0, 102.7, 101.0, 91.2, 89.4, 89.3, 87.2, 81.3, 80.8, 80.1, 76.1, 71.3, 71.2, 68.7, 67.6, 66.0, 35.33, 35.28; IR (neat) ν (cm^{-1}) 3062, 3027, 2982, 2871, 1959, 1602, 1494, 1453, 1379, 1363, 1275, 1261, 1174, 1085, 1043, 1000; MS (ESI, m/z) 643 ($M+K^+$), 627 ($M+Na^+$); HRMS calcd for $C_{39}H_{40}O_6Na$ ($M+Na^+$): 627.2717, found: 627.2717.

19. Preparation of (R_a)-4ka. hx-12-139



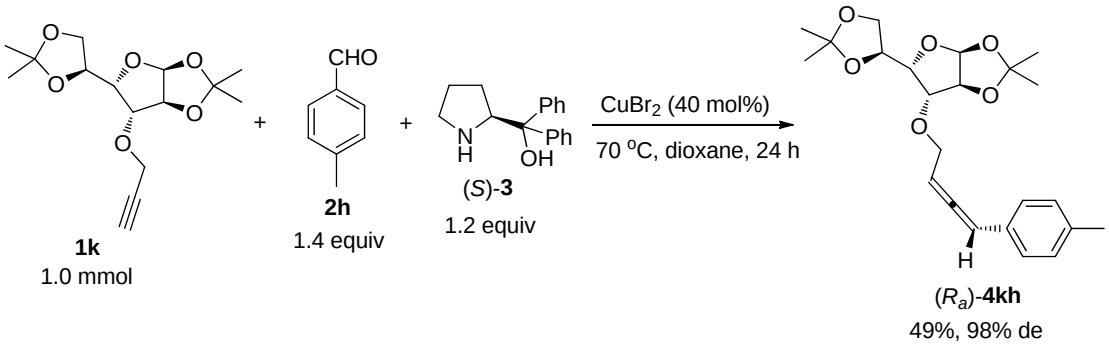
The reaction of $CuBr_2$ (45.0 mg, 0.2 mmol), **1k** (301.2 mg, 1.0 mmol), (*S*)-**3** (304.7 mg, 1.2 mmol), and **2a** (179.8 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ka** (214.5 mg, 52%) (eluent: petroleum ether/ethyl acetate = 20/1) as a liquid: 97% de (HPLC conditions: Chiralcel OZ-H column, hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, t_R(minor) = 12.4 min, t_R(major) = 14.3 min); $[\alpha]_D^{20}$ = -43.2 (*c* = 1.09, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.88 (d, *J* = 3.9 Hz, 1 H), 5.29-5.12 (m, 2 H), 4.55 (d, *J* = 3.6 Hz, 1 H), 4.34-4.25 (m, 1 H), 4.19-4.03 (m, 4 H), 4.03-3.95 (m, 2 H), 2.02 (ddd, J_1 = 14.1 Hz, J_2 = 6.9 Hz, J_3 = 3.0 Hz, 2 H, CH_2), 1.49 (s, 3 H, Me), 1.43 (s, 3 H, Me), 1.35 (s, 3 H, Me), 1.31 (s, 3 H, Me), 1.47-1.20 (m, 10 H, CH_2 × 5), 0.88 (t, *J* = 6.6 Hz, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.0, 111.6, 108.7, 105.1, 92.2, 88.0, 82.7, 81.0, 80.8, 72.5, 68.9, 67.0, 31.7, 29.0, 28.9, 28.4, 26.73, 26.68, 26.1, 25.3, 22.5, 14.0; IR (neat) ν (cm^{-1}) 2986, 2929, 2856, 1963, 1456, 1381, 1372, 1346, 1253, 1216, 1166, 1119, 1078, 1023; MS(EI): *m/z* (%) 410 (M^+, 3.2), 101 (100), 43 (100); HRMS calcd for $C_{23}H_{38}O_6$ [M^+]: 410.2668, found: 410.2667.

20. Preparation of (R_a)-4ke. hx-12-171



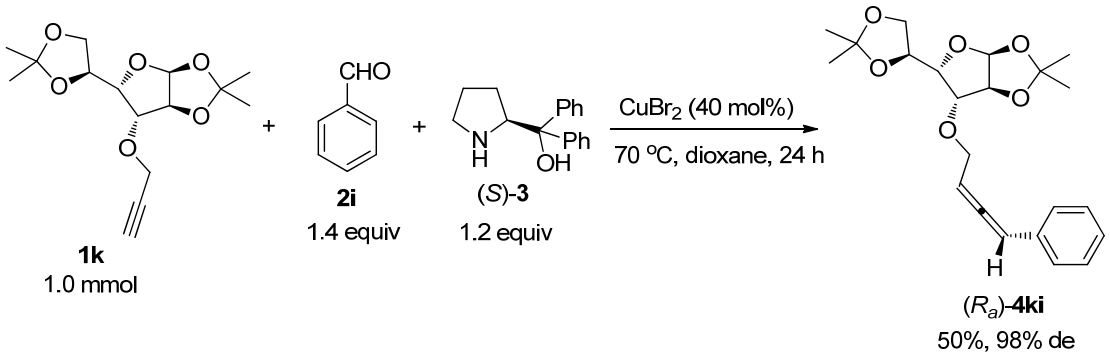
The reaction of $CuBr_2$ (44.8 mg, 0.2 mmol), **1k** (298.6 mg, 1.0 mmol), (*S*)-**3** (303.8 mg, 1.2 mmol), and **2e** (157.1 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ke** (220.6 mg, 56%) (eluent: petroleum ether/ethyl acetate = 20/1 to 10/1) as a liquid: 96% de (HPLC conditions: Chiralcel OZ-H column, hexane/*i*-PrOH = 100/1, 0.3 mL/min, λ = 214 nm, t_R(minor) = 26.4 min, t_R(major) = 28.2 min); $[\alpha]_D^{20}$ = -63.8 (*c* = 0.94, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.88 (d, *J* = 3.6 Hz, 1 H), 5.29-5.17 (m, 2 H), 4.56 (d, *J* = 3.6 Hz, 1 H), 4.35-4.25 (m, 1 H), 4.20-3.95 (m, 6 H), 2.10-1.92 (m, 1 H, CH), 1.84-1.57 (m, 5 H, five protons from *Cy*), 1.50 (s, 3 H, Me), 1.43 (s, 3 H, Me), 1.35 (s, 3 H, Me), 1.31 (s, 3 H, Me), 1.39-1.00 (m, 5 H, five protons from *Cy*); ^{13}C NMR (75 Hz, $CDCl_3$) δ 203.9, 111.5, 108.6, 105.1, 98.1, 88.8, 82.6, 80.9, 80.8, 72.4, 69.0, 67.0, 36.7, 32.8, 26.7, 26.6, 26.1, 25.9, 25.8, 25.2; IR (neat) ν (cm^{-1}) 2986, 2927, 2852, 1962, 1450, 1382, 1372, 1347, 1255, 1216, 1166, 1117, 1077, 1022; MS (ESI, m/z) 417 ($M+Na^+$), 395 ($M+H^+$); HRMS calcd. for $C_{22}H_{34}NaO_6$ ($M+Na^+$): 417.2248; Found: 417.2231.

21. Preparation of (R_a)-4kh. hx-12-186



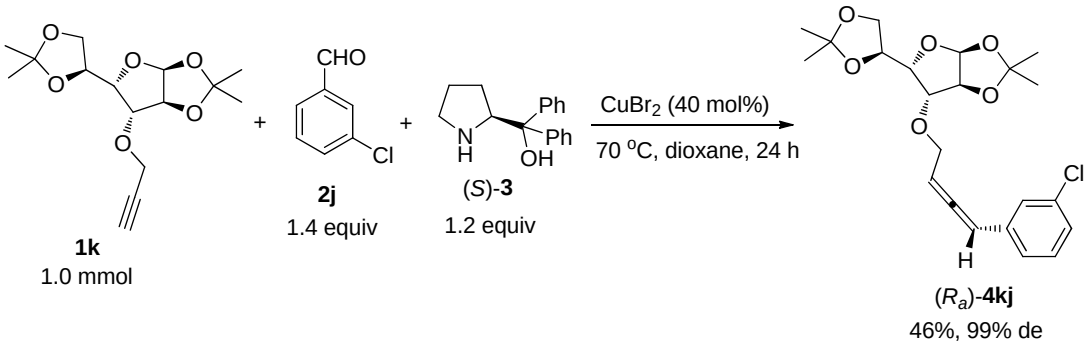
The reaction of $CuBr_2$ (89.4 mg, 0.4 mmol), **1k** (299.4 mg, 1.0 mmol), (*S*)-**3** (303.8 mg, 1.2 mmol), and **2h** (168.6 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4kh** (196.6 mg, 49%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 98% de (HPLC conditions: Chiralcel IA column, hexane/*i*-PrOH = 100/1, 0.8 mL/min, λ = 214 nm, t_R(minor) = 13.8 min, t_R(major) = 14.8 min); $[\alpha]_D^{20}$ = -150.3 (*c* = 1.08, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 (d, *J* = 7.8 Hz, 2 H, ArH), 7.10 (d, *J* = 7.8 Hz, 2 H, ArH), 6.25 (dt, J_1 = 6.0 Hz, J_2 = 2.3 Hz, 1 H, one proton of CH=C=CH), 5.86 (d, *J* = 3.9 Hz, 1 H), 5.64 (dd, J_1 = 13.1 Hz, J_2 = 6.5 Hz, 1 H, one proton of CH=C=CH), 4.54 (d, *J* = 3.6 Hz, 1 H), 4.39-3.94 (m, 7 H), 2.32 (s, 3 H, Me), 1.48 (s, 3 H, Me), 1.43 (s, 3 H, Me), 1.30 (s, 3 H, Me), 1.26 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.7, 136.9, 130.5, 129.3, 126.7, 111.6, 108.8, 105.1, 95.7, 92.2, 82.7, 81.4, 81.0, 72.4, 68.3, 67.1, 26.7, 26.0, 25.2, 21.1; IR (neat) ν (cm^{-1}) 2986, 2935, 1950, 1513, 1455, 1382, 1372, 1346, 1255, 1215, 1165, 1117, 1078, 1021; MS (ESI, m/z) 425 ($M+Na^+$), 403 ($M+H^+$); HRMS calcd. for $C_{23}H_{30}NaO_6$ ($M+Na^+$): 425.1935; Found: 425.1934.

22. Preparation of (R_a)-4ki. xc-13-50

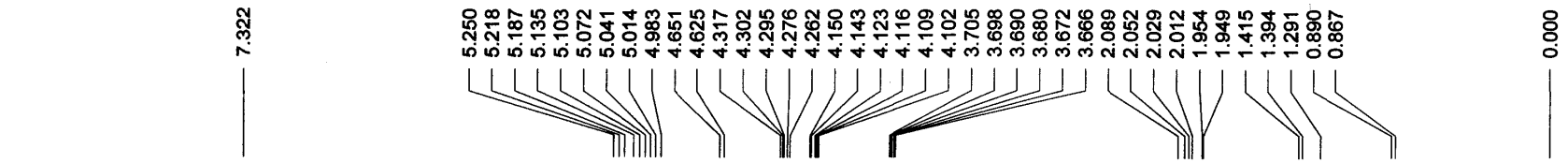


The reaction of $CuBr_2$ (90.1 mg, 0.4 mmol), **1k** (298.5 mg, 1.0 mmol), (*S*)-**3** (304.3 mg, 1.2 mmol), and **2i** (149.0 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4ki** (193.3 mg, 50%) (eluent: petroleum ether/ethyl acetate = 10/1) as an oil: 98% de (HPLC conditions: Chiralcel IB column, hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 8.6 min, t_R(minor) = 11.4 min); $[\alpha]_D^{20}$ = -154.2 (*c* = 1.08, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.17 (m, 5 H, ArH), 6.28 (dt, J_1 = 6.3 Hz, J_2 = 2.4 Hz, 1 H, one proton of CH=C=CH), 5.86 (d, *J* = 3.6 Hz, 1 H), 5.67 (dd, J_1 = 12.9 Hz, J_2 = 6.6 Hz, 1 H, one proton of CH=C=CH), 4.55 (d, *J* = 3.9 Hz, 1 H), 4.39-4.18 (m, 3 H), 4.16-3.96 (m, 4 H), 1.49 (s, 3 H, Me), 1.43 (s, 3 H, Me), 1.31 (s, 3 H, Me), 1.27 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 205.9, 133.6, 128.6, 127.2, 126.8, 111.7, 108.9, 105.2, 95.9, 92.4, 82.8, 81.5, 81.1, 72.4, 68.3, 67.2, 26.8, 26.1, 25.3; IR (neat) ν (cm^{-1}) 2986, 2935, 2890, 1951, 1496, 1459, 1382, 1372, 1255, 1216, 1165, 1117, 1077, 1021; MS (ESI, m/z) 411 ($M+Na^+$); HRMS calcd. for $C_{22}H_{28}NaO_6$ ($M+Na^+$): 411.1778; Found: 411.1798.

23. Preparation of (R_a)-4kj. hx-12-179



The reaction of $CuBr_2$ (89.5 mg, 0.4 mmol), **1k** (297.7 mg, 1.0 mmol), (*S*)-**3** (303.8 mg, 1.2 mmol), and **2j** (197.2 mg, 1.4 mmol) in dioxane (3.0 mL) afforded (R_a)-**4kj** (195.5 mg, 46%) (eluent: petroleum ether/ethyl acetate = 10/1) as a liquid: 99% de (HPLC conditions: Chiralcel IA column, hexane/*i*-PrOH = 200/1, 0.8 mL/min, λ = 214 nm, t_R(minor) = 27.4 min, t_R(major) = 29.6 min); $[\alpha]_D^{20}$ = -151.6 (*c* = 1.23, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.13 (m, 4 H, ArH), 6.22 (dt, J_1 = 6.3 Hz, J_2 = 2.4 Hz, 1 H, one proton of CH=C=CH), 5.87 (d, *J* = 3.6 Hz, 1 H), 5.64 (dd, J_1 = 12.9 Hz, J_2 = 6.6 Hz, 1 H, one proton of CH=C=CH), 4.55 (d, *J* = 3.6 Hz, 1 H), 4.37-4.20 (m, 3 H), 4.17-4.05 (m, 2 H), 4.04-3.96 (m, 2 H), 1.49 (s, 3 H, Me), 1.43 (s, 3 H, Me), 1.31 (s, 3 H, Me), 1.27 (s, 3 H, Me); ^{13}C NMR (75 Hz, $CDCl_3$) δ 206.0, 135.6, 134.5, 129.7, 127.1, 126.6, 124.9, 111.6, 108.8, 105.1, 95.0, 92.8, 82.7, 81.5, 81.0, 72.3, 67.9, 67.1, 26.69, 26.66, 26.0, 25.1; IR (neat) ν (cm^{-1}) 2987, 2935, 2890, 1953, 1594, 1571, 1478, 1454, 1382, 1372, 1347, 1254, 1216, 1165, 1078, 1021; MS (ESI, m/z) 447 (M(^{37}Cl)+Na^+), 445 (M(^{35}Cl)+Na^+); HRMS calcd. for $C_{22}H_{27}O_6{}^{35}ClNa$ (M(^{35}Cl)+Na^+): 445.1388; Found: 445.1386.



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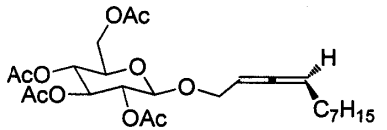
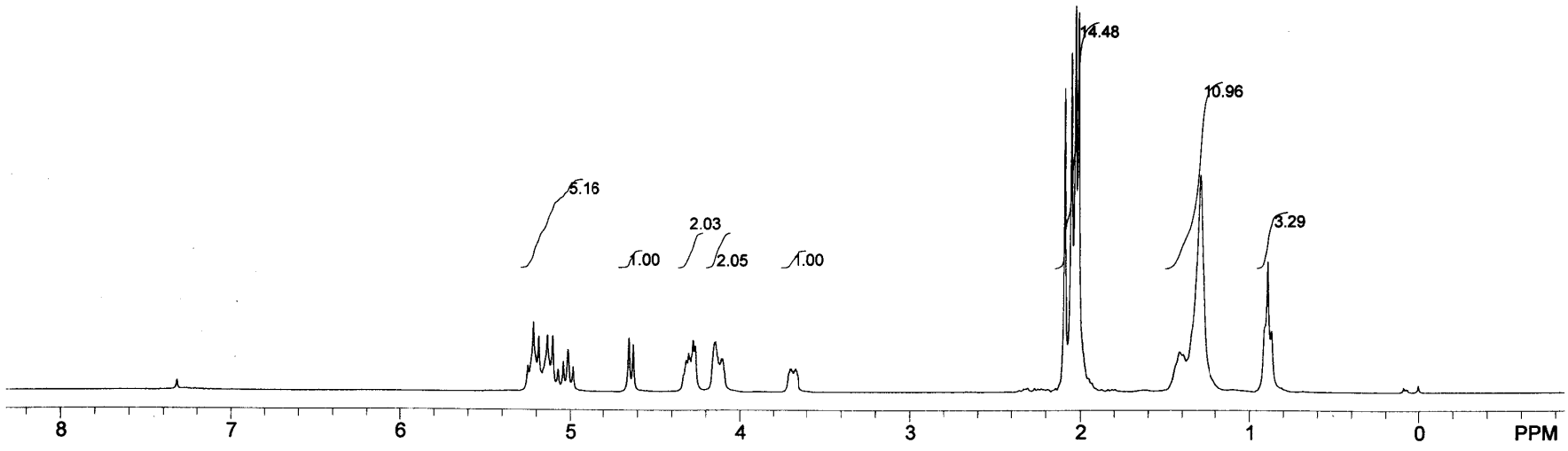


(R_a)-**4aa**

1.0 mmol scale



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71.044
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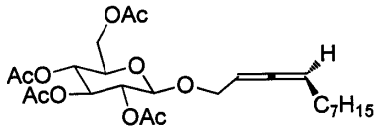

(R_a)-**4aa**

1.0 mmol scale

hx-10-7

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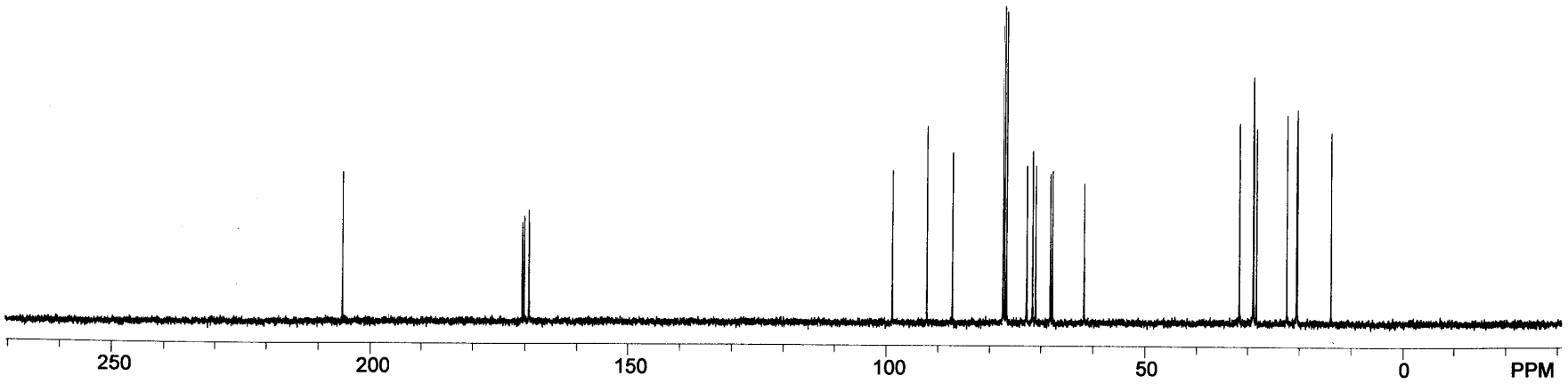

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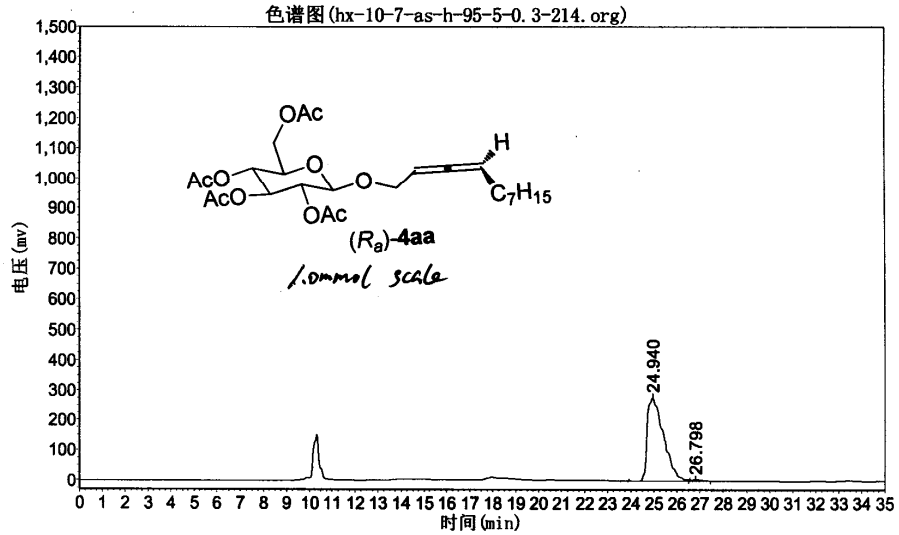


分析结果表

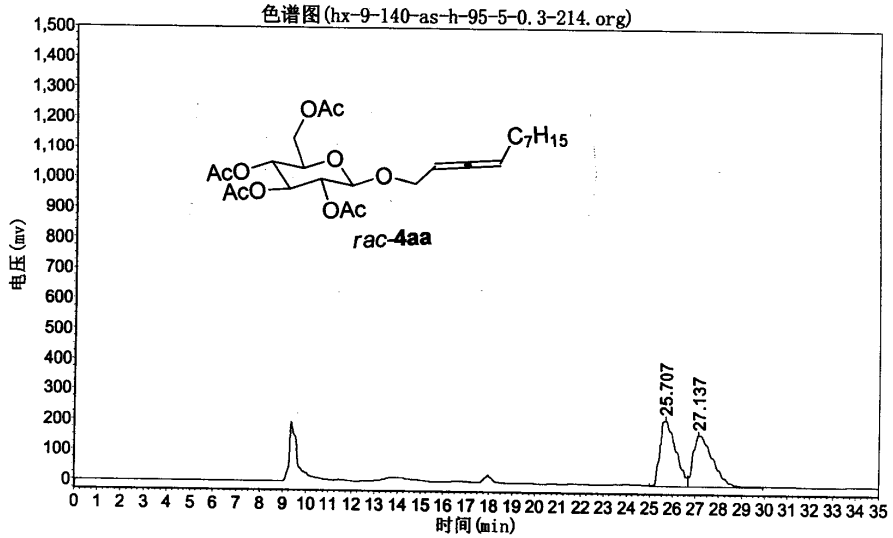
峰号	峰名	保留时间	峰高	峰面积	含量
1		24.940	278361.063	13509224.000	98.9368
2		26.798	4297.991	145171.766	1.0632
总计			282659.053	13654395.766	100.0000

hx-9-140-as-h-95-5-0.3-214

实验时间：2013-12-24,8:49:22
谱图文件:D:\zhuguangjiong\hx\20131224\hx-9-140-as-h-95-5-0.3-214.org

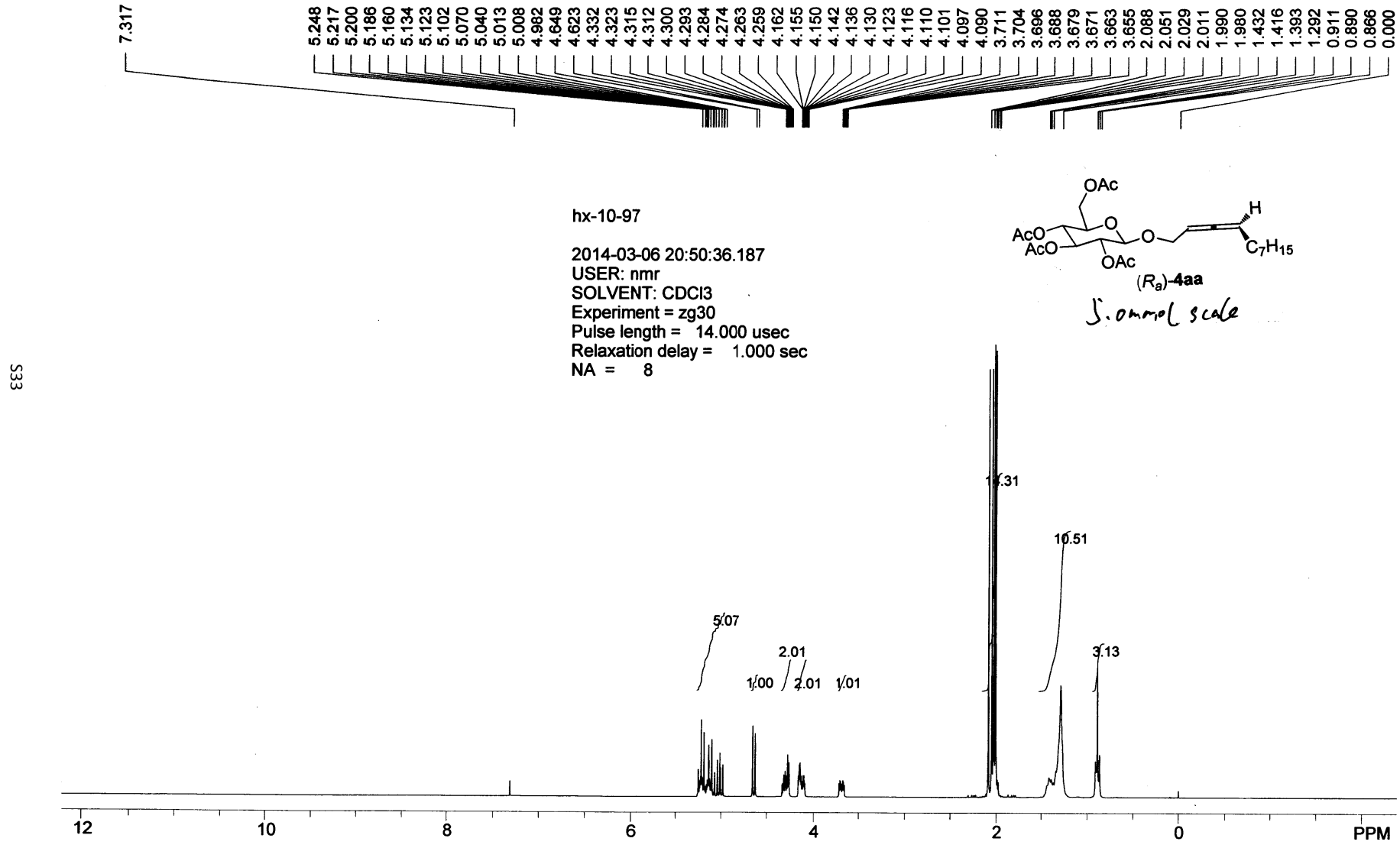
报告时间：2013-12-25,17:09:36

实验内容简介：



分析结果表

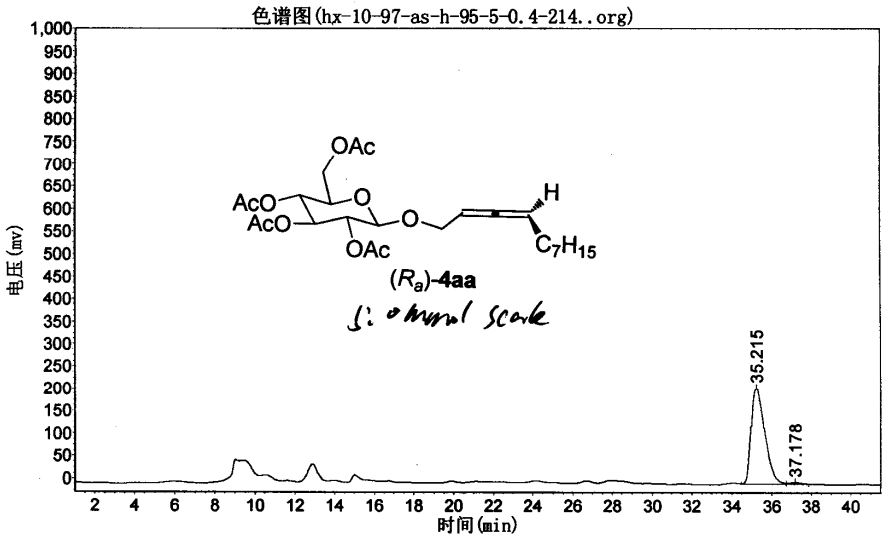
峰号	峰名	保留时间	峰高	峰面积	含量
1		25.707	215984.016	10384250.000	50.5456
2		27.137	167907.828	10160053.000	49.4544
总计			383891.844	20544303.000	100.0000


7.317
5.248
5.217
5.200
5.186
5.160
5.134
5.123
5.102
5.070
5.040
5.013
5.008
4.982
4.649
4.623
4.332
4.323
4.315
4.312
4.300
4.293
4.284
4.274
4.263
4.259
4.162
4.155
4.150
4.142
4.136
4.130
4.123
4.116
4.110
4.101
4.097
4.090
3.711
3.704
3.696
3.688
3.679
3.671
3.663
3.655
2.088
2.051
2.029
2.011
1.990
1.980
1.432
1.416
1.393
1.292
0.911
0.890
0.866
0.000
hx-10-97
2014-03-06 20:50:36.187
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
AcO
OAc
O
O
H
C7H15
(Ra)-4aa
5.0mmol scale
5.07
2.01
1.00
2.01
1.01
10.51
3.13
12
10
8
6
4
2
0
PPM

hx-10-97-as-h-95-5-0.4-214

实验时间：2014-03-11,15:53:03　　报告时间：2014-03-12,15:37:31
谱图文件:D:\zhuguangjiong\hx\20140311\hx-10-97-as-h-95-5-0.4-214..org

实验内容简介：



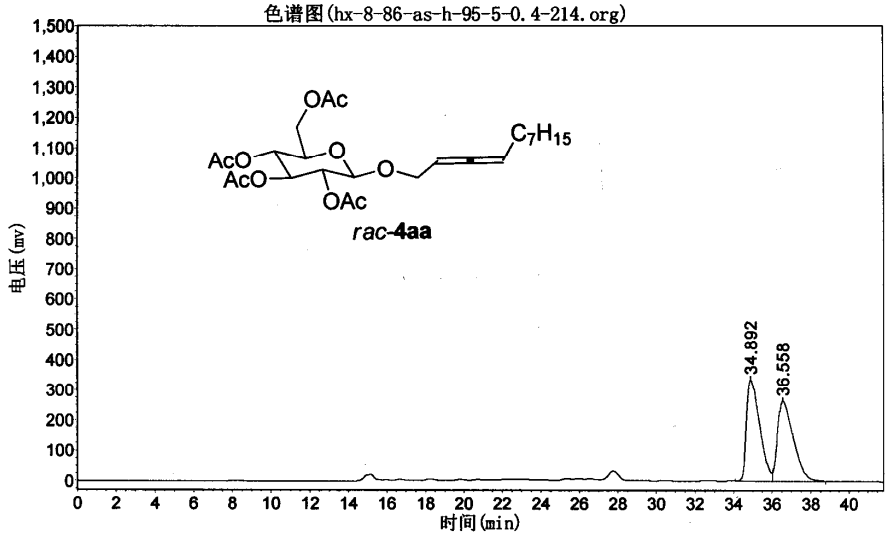
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		35.215	212151.531	10118550.000	98.7541
2		37.178	3154.016	127653.867	1.2459
总计			215305.548	10246203.867	100.0000

hx-8-86-as-h-95-5-0.4-214

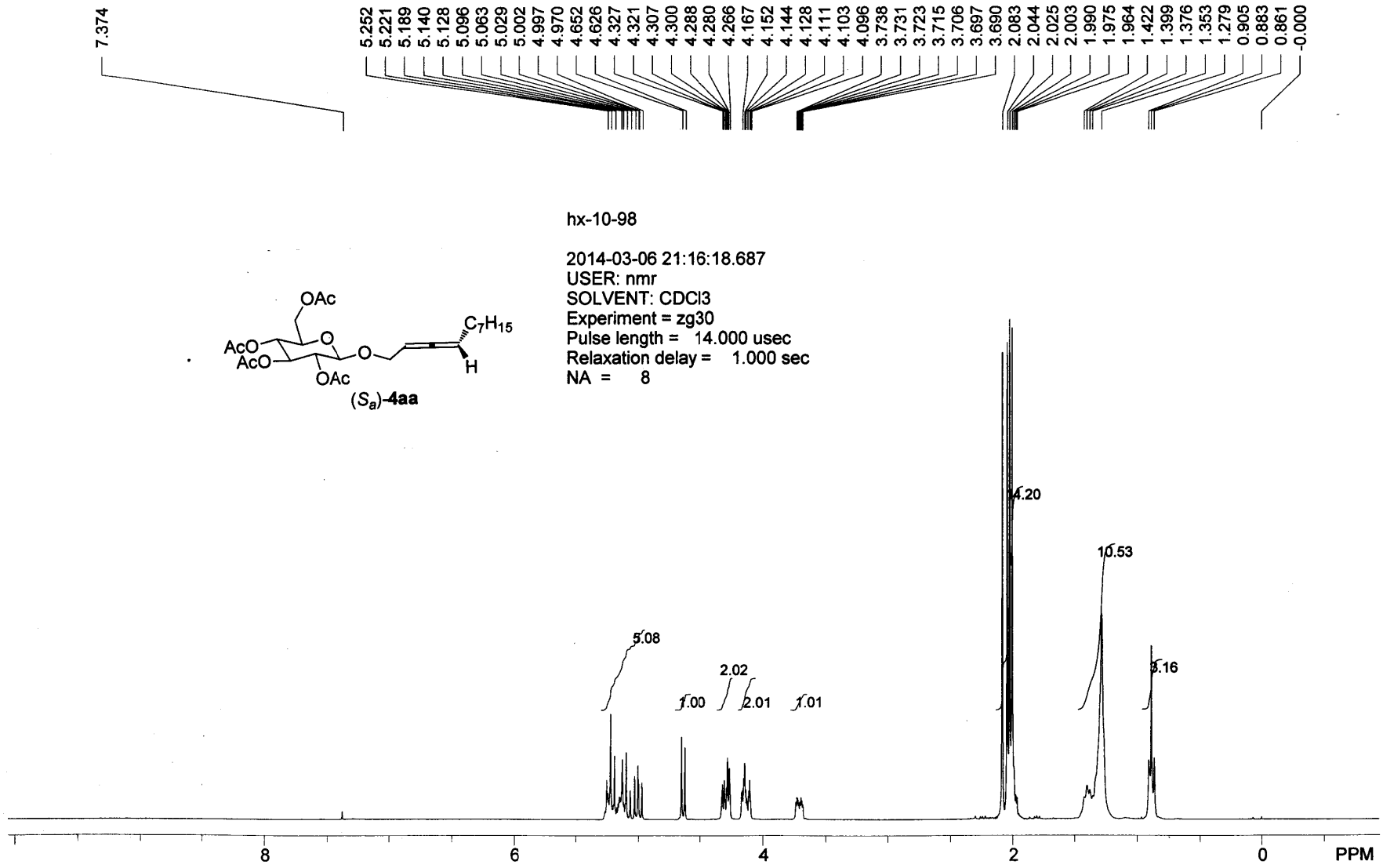
实验时间：2014-03-11,14:12:21　　报告时间：2014-03-11,14:59:09
谱图文件:D:\zhuguangjiong\hx\20140311\hx-8-86-as-h-95-5-0.4-214.org

实验内容简介：



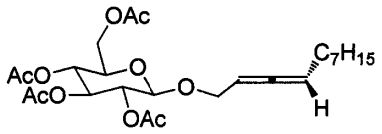
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		34.892	334952.438	16051996.000	51.4118
2		36.558	264060.406	15170393.000	48.5882
总计			599012.844	31222389.000	100.0000

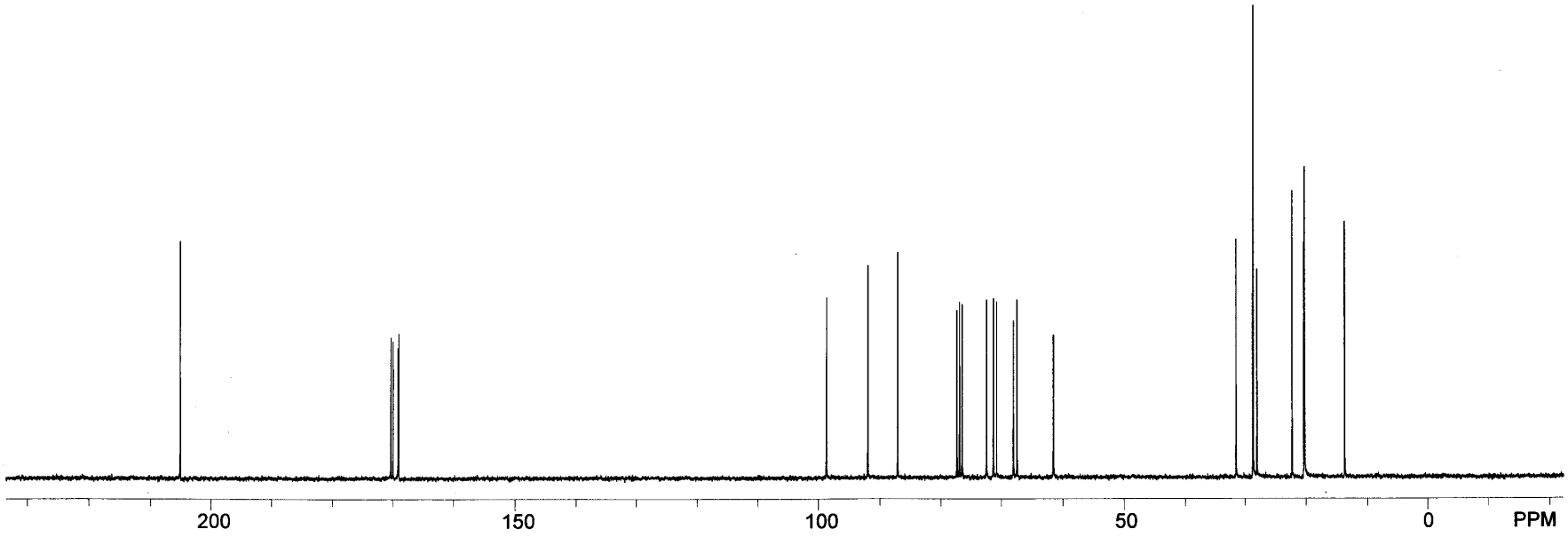

7.374
5.252
5.221
5.189
5.140
5.128
5.096
5.063
5.029
5.002
4.997
4.970
4.652
4.626
4.327
4.321
4.307
4.300
4.288
4.280
4.266
4.167
4.152
4.144
4.128
4.111
4.103
4.096
3.738
3.731
3.723
3.715
3.706
3.697
3.690
2.083
2.044
2.025
2.003
1.990
1.975
1.964
1.422
1.399
1.376
1.353
1.279
0.905
0.883
0.861
-0.000
hx-10-98
2014-03-06 21:16:18.687
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
AcO
OAc
O
O
C_7H_{15}
H
(S_a)-4aa
5.08
1.00
2.02
2.01
1.01
4.20
10.53
3.16
8
6
4
2
0
PPM

hx-10-98

2014-03-06 21:25:39.656
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 139



(S_a)-**4aa**

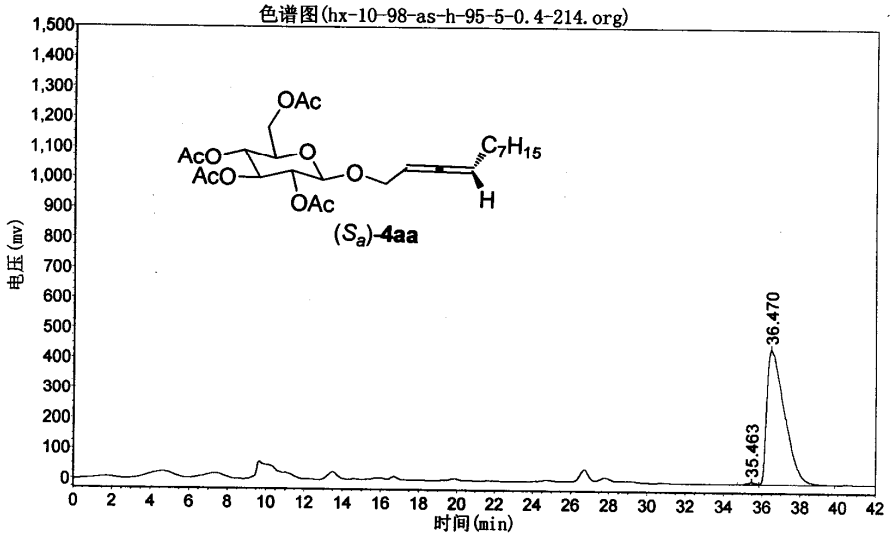


hx-10-98-as-h-95-5-0.4-214

实验时间：2014-03-11,17:29:11
谱图文件:D:\zhuguangjiong\hx\20140311\hx-10-98-as-h-95-5-0.4-214.org

报告时间：2014-03-12,15:39:19

实验内容简介：



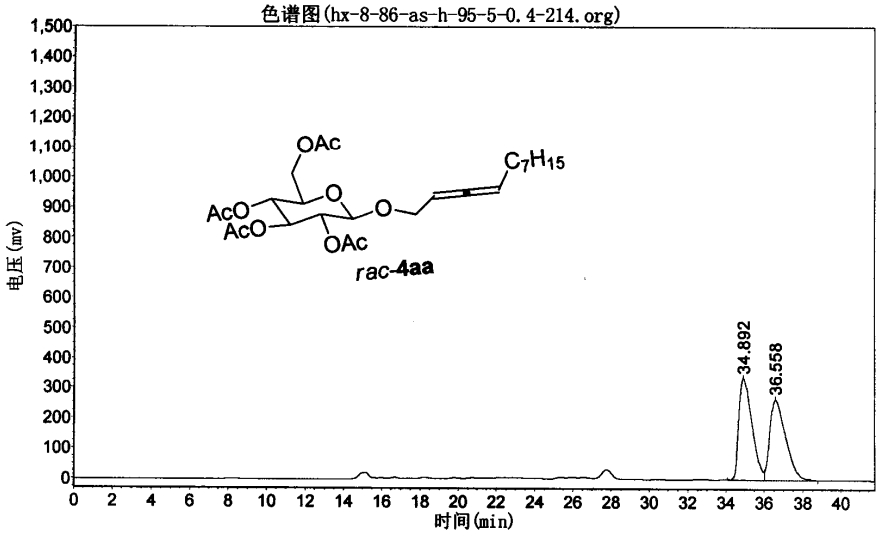
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		35.463	7538.812	254559.391	0.8754
2		36.470	445148.000	28826192.000	99.1246
总计			452686.812	29080751.391	100.0000

hx-8-86-as-h-95-5-0.4-214

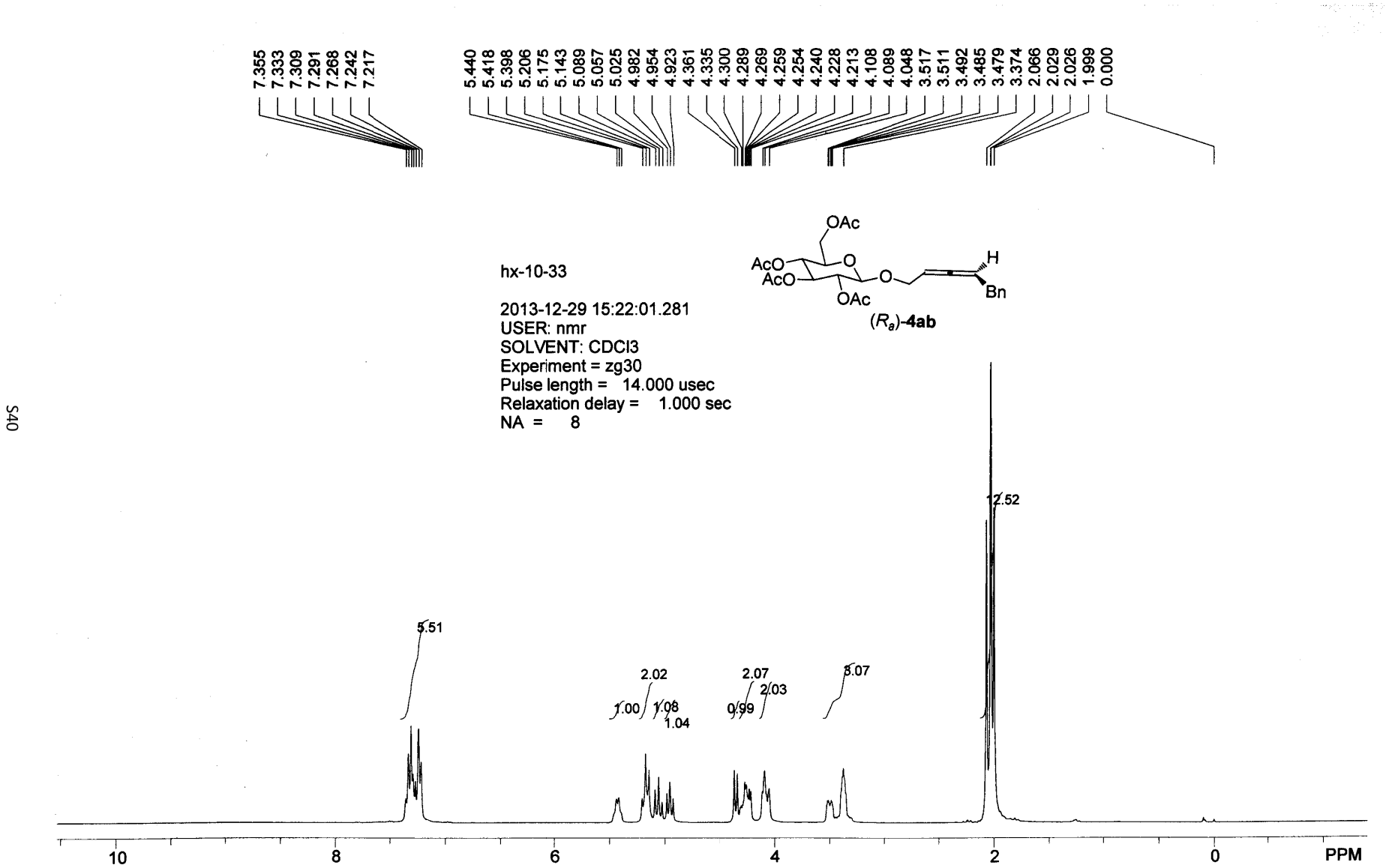
实验时间：2014-03-11,14:12:21　　报告时间：2014-03-11,14:59:09
谱图文件:D:\zhuguangjiong\hx\20140311\hx-8-86-as-h-95-5-0.4-214.org

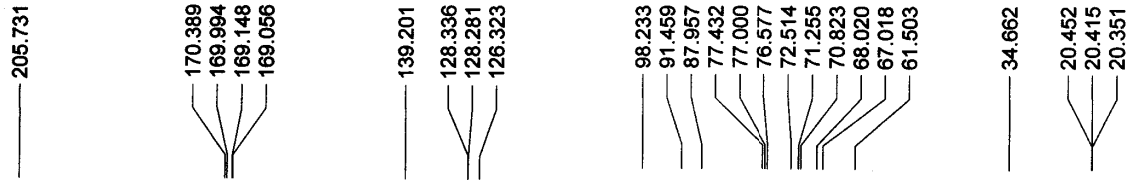
实验内容简介：



分析结果表

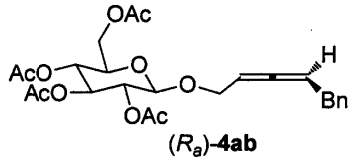
峰号	峰名	保留时间	峰高	峰面积	含量
1		34.892	334952.438	16051996.000	51.4118
2		36.558	264060.406	15170393.000	48.5882
总计			599012.844	31222389.000	100.0000


7.355
7.333
7.309
7.291
7.268
7.242
7.217
5.440
5.418
5.398
5.206
5.175
5.143
5.089
5.057
5.025
4.982
4.954
4.923
4.361
4.335
4.300
4.289
4.269
4.259
4.254
4.240
4.228
4.213
4.108
4.089
4.048
3.517
3.511
3.492
3.485
3.479
3.374
2.066
2.029
2.026
1.999
0.000
OAc
AcO
AcO
O
O
OAc
H
Bn
(R_a)-4ab
hx-10-33
2013-12-29 15:22:01.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
5.51
2.02
1.00
1.08
1.04
2.07
0.99
2.03
3.07
12.52
10
8
6
4
2
0
PPM

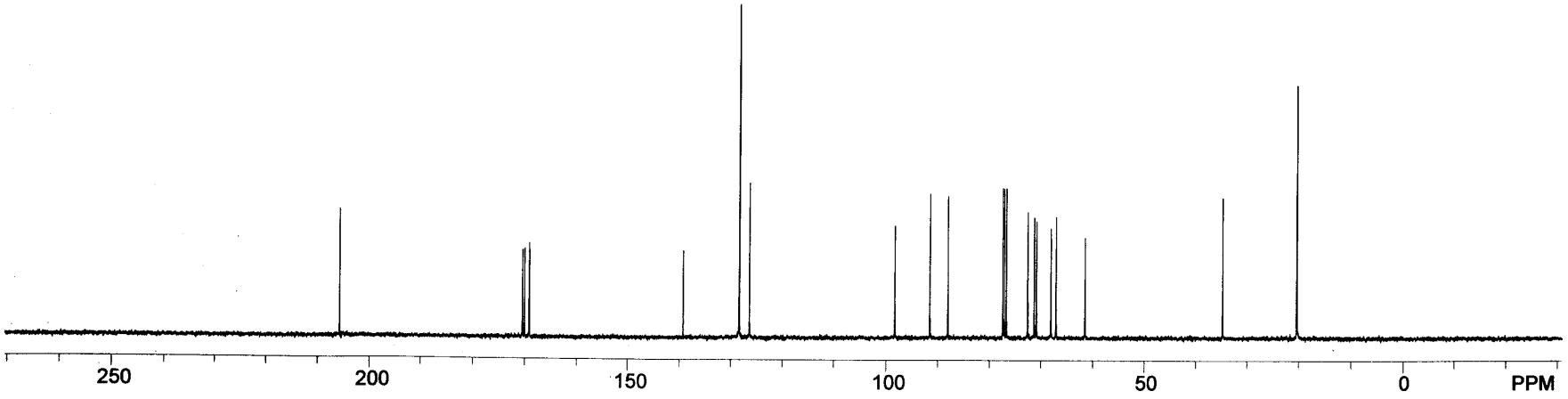


hx-10-33

2013-12-29 15:34:13.468
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 111



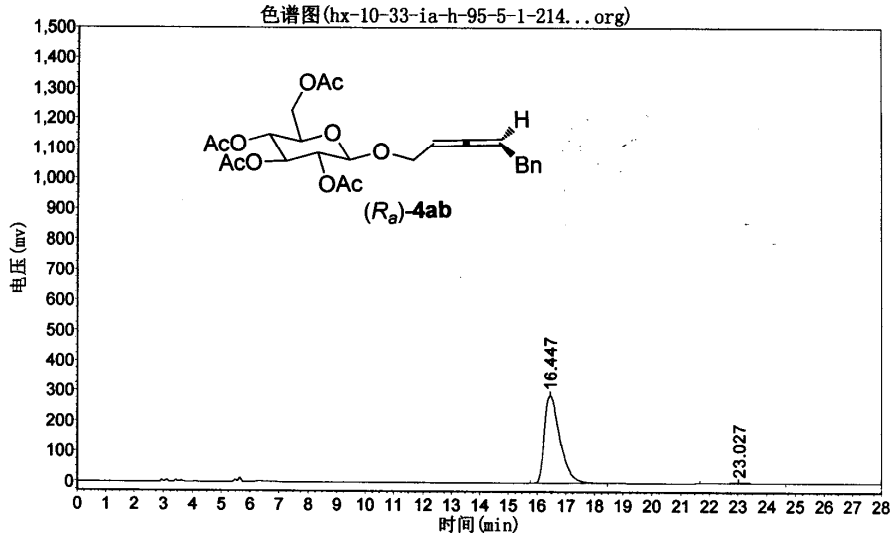
(R_a)-**4ab**



hx-10-33-ia-h-95-5-1-214

实验时间：2014-01-10,10:45:54　　报告时间：2014-01-10,11:59:29
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-33-ia-h-95-5-1-214...org

实验内容简介：



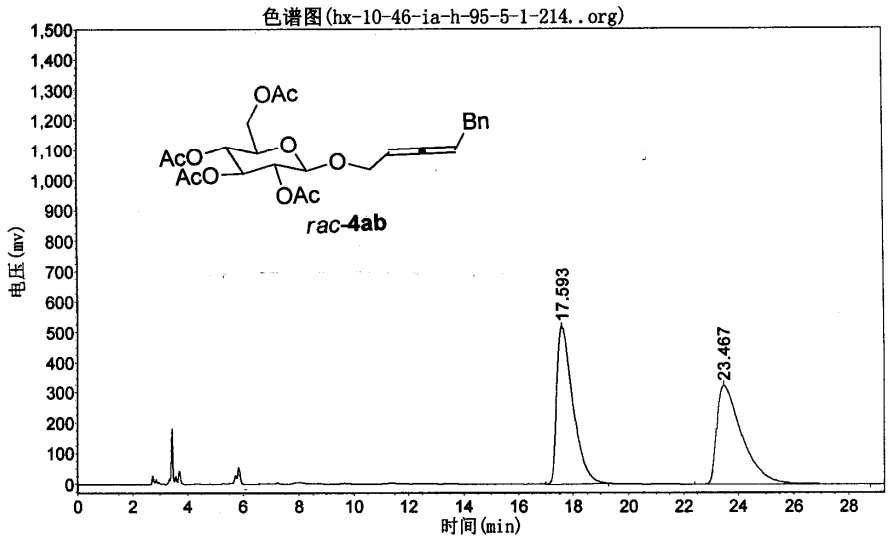
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.447	289720.625	10930804.000	98.6397
2		23.027	2218.483	150741.797	1.3603
总计			291939.108	11081545.797	100.0000

hx-10-46-ia-h-95-5-1-214

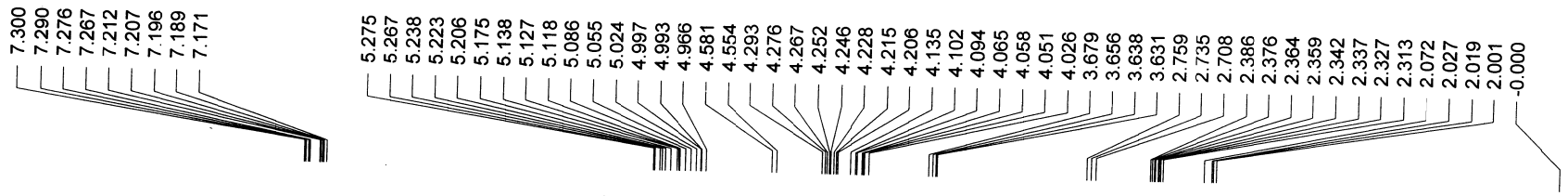
实验时间：2014-01-10,9:48:51　　报告时间：2014-01-10,11:57:13
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-46-ia-h-95-5-1-214..org

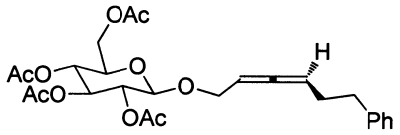
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.593	518016.875	21159832.000	50.7949
2		23.467	323006.969	20497596.000	49.2051
总计			841023.844	41657428.000	100.0000

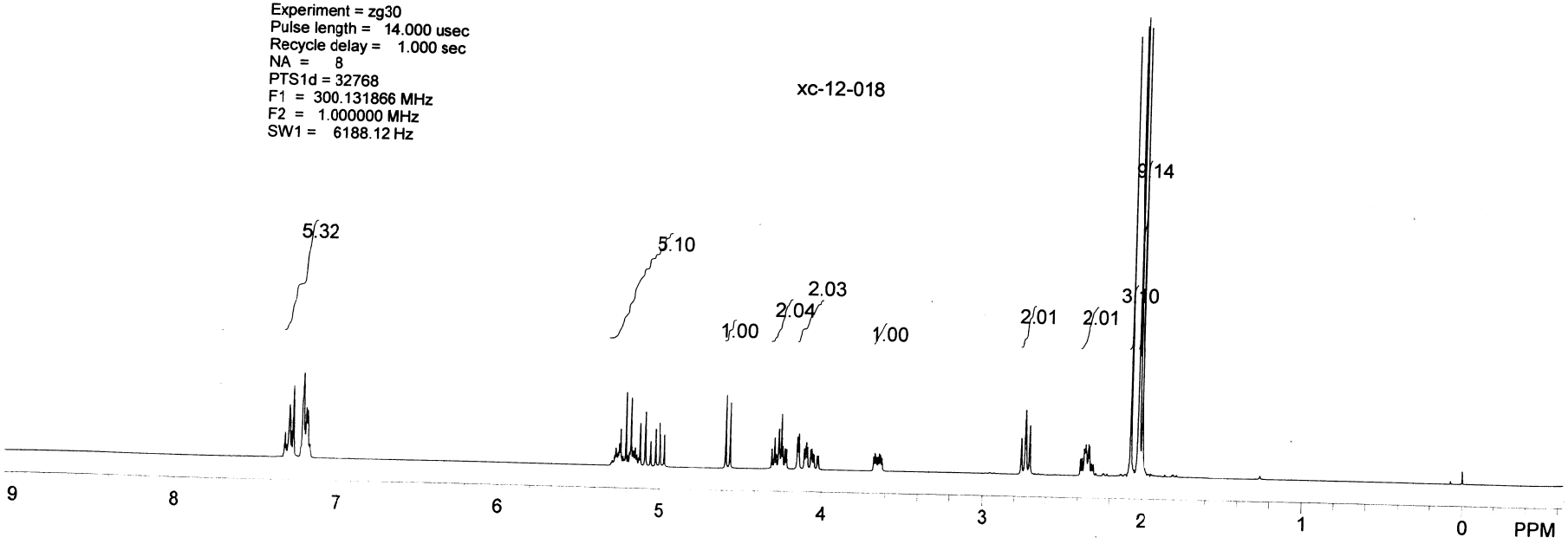


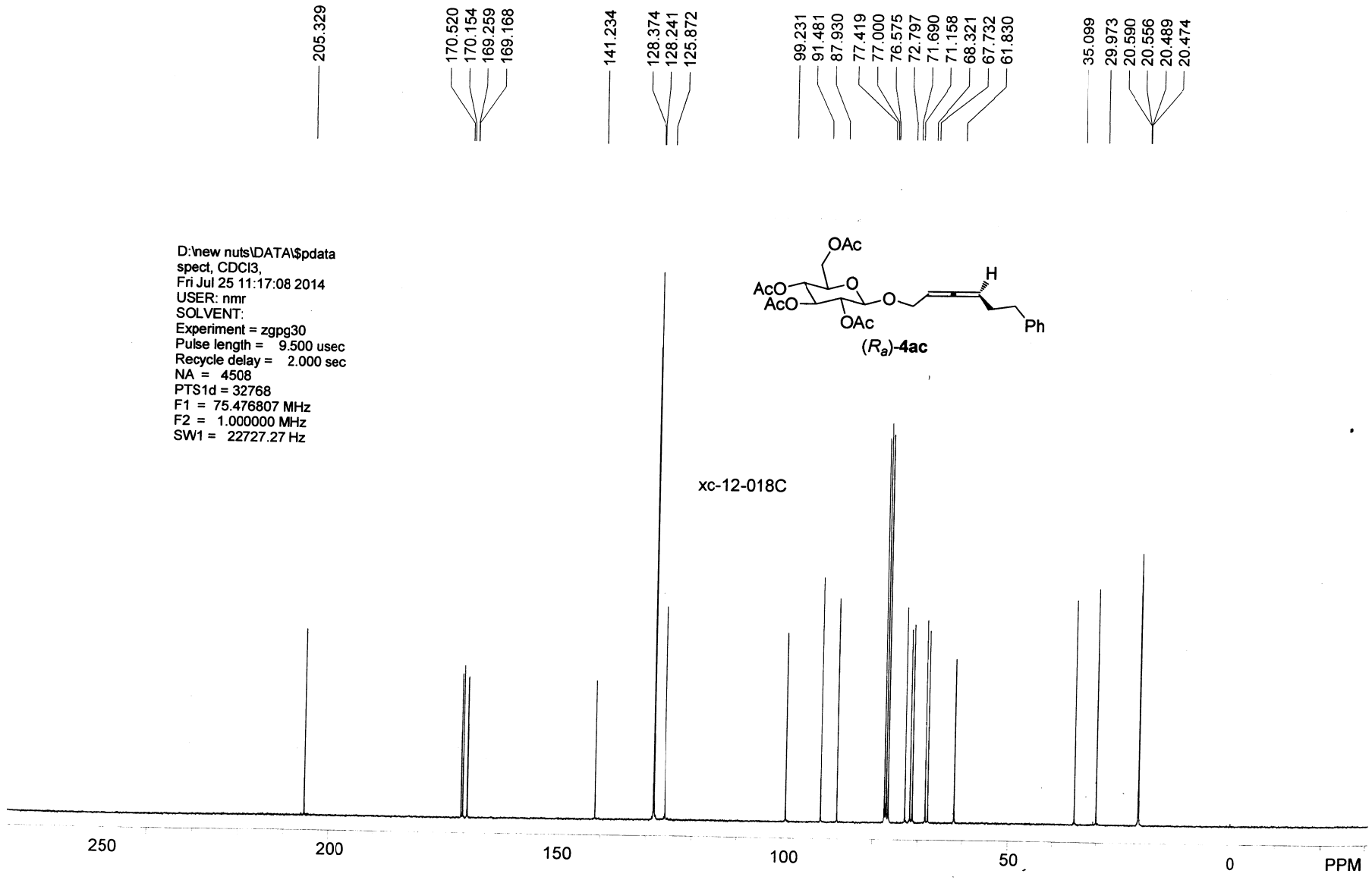


(R_a)-**4ac**

D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 25 06:53:27 2014
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

xc-12-018



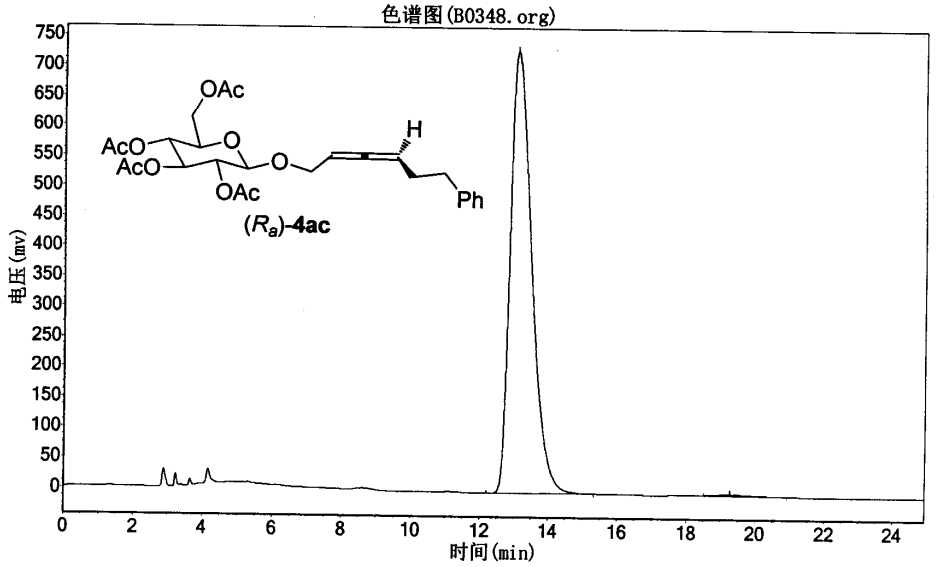

205.329
170.520
170.154
169.259
169.168
141.234
128.374
128.241
125.872
99.231
91.481
87.930
77.419
77.000
76.575
72.797
71.690
71.158
68.321
67.732
61.830
35.099
29.973
20.590
20.556
20.489
20.474
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jul 25 11:17:08 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 4508
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OAc
AcO
AcO
O
O
OAc
H
Ph
(Ra)-4ac
xc-12-018C
250
200
150
100
50
0
PPM

xc-12-18

实验单位：zju
实验时间：2014-07-23,19:30:25
谱图文件:D:\浙大智达\N2000\样品\B0348.org

报告时间：2014-07-23,20:05:15
积分方法:面积归一法

实验内容简介：
OD-H column, n-hexane/iPrOH = 90/10, 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		13.078	730860.813	31448738.000	99.5941
2		19.270	1979.851	128167.211	0.4059
总计			732840.663	31576905.211	100.0000

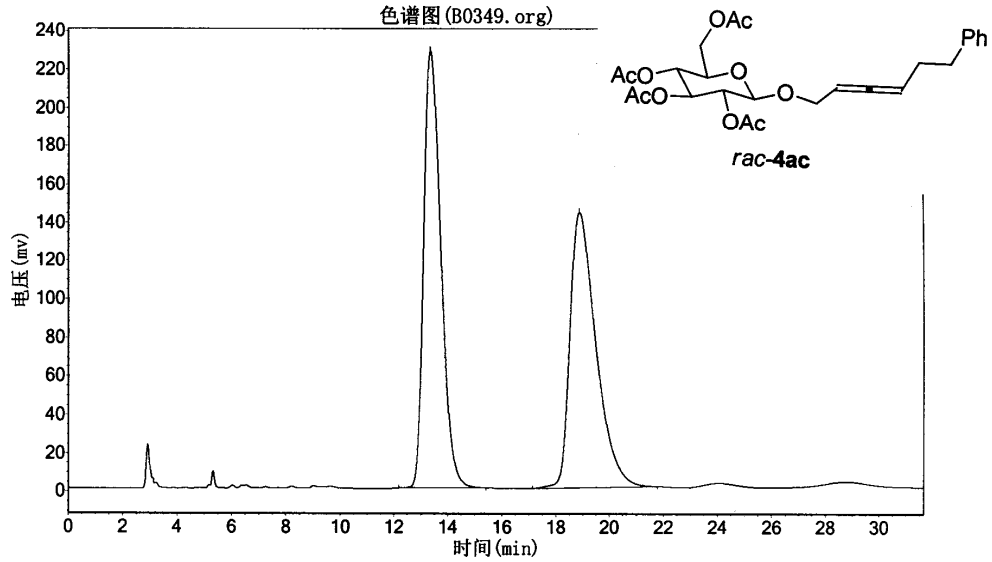
2014-07-23 浙江大学智能信息研究所

hx-8-108

实验单位：zju
实验时间：2014-07-23,19:58:04
谱图文件:D:\浙大智达\N2000\样品\B0349.org

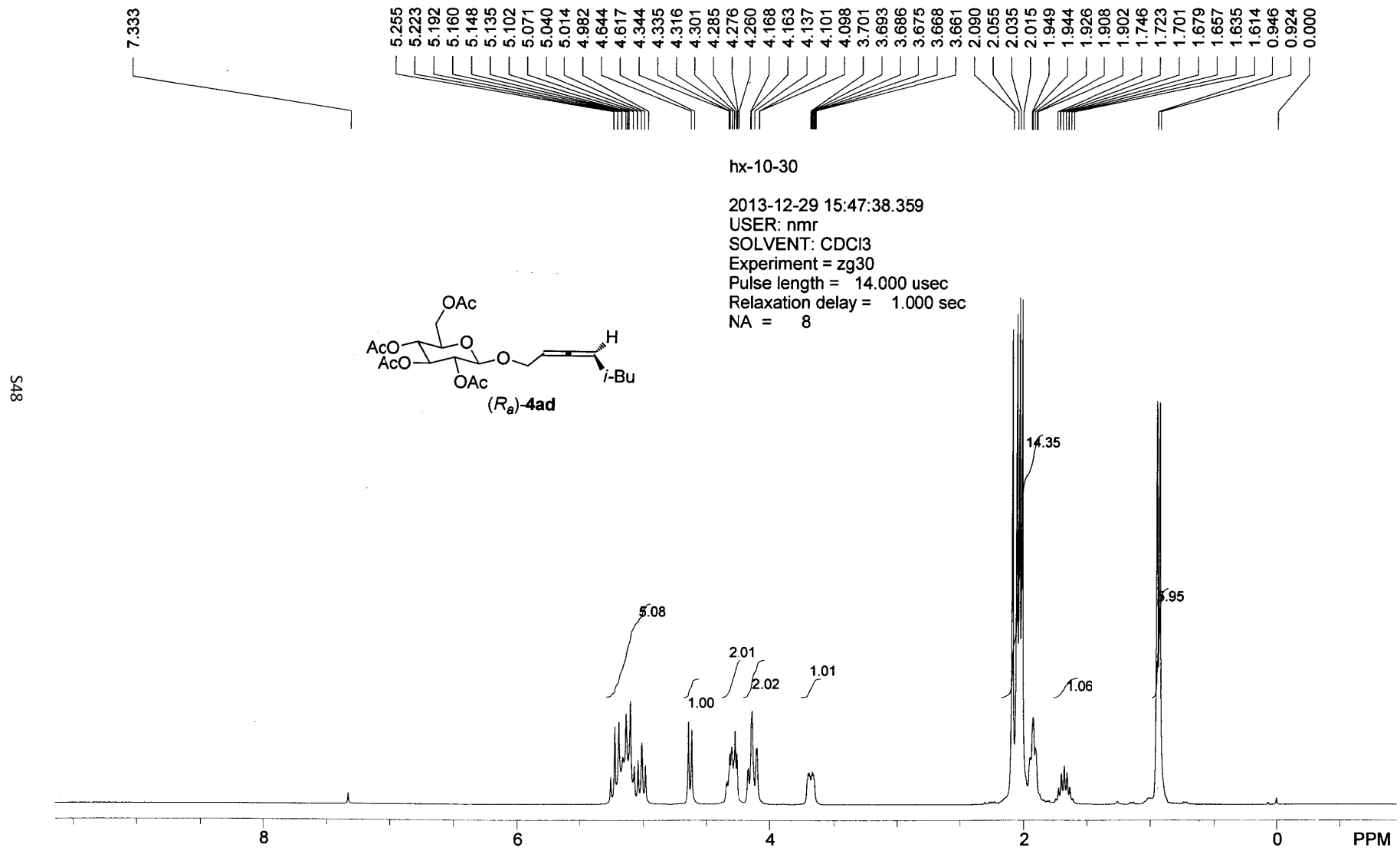
实验者：hx
报告时间：2014-07-23,20:39:07
积分方法:面积归一法

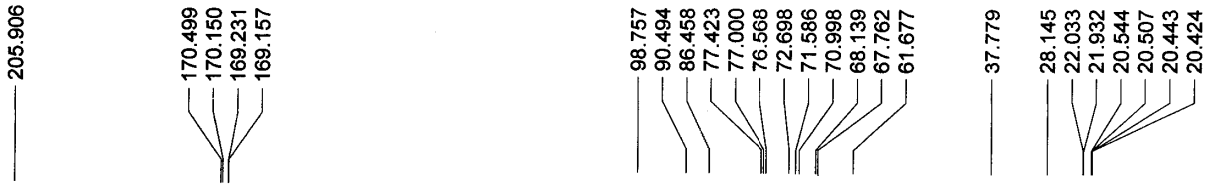
实验内容简介：
OD-H column, n-hexane/iPrOH = 90/10, 214 nm, 1.0 ml/min

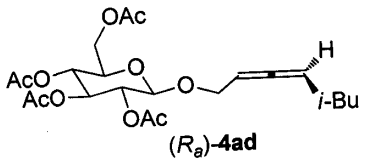


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.357	227702.375	10106964.000	51.2781
2		18.887	143049.047	9603139.000	48.7219
总计			370751.422	19710103.000	100.0000


7.333
5.255
5.223
5.192
5.160
5.148
5.135
5.102
5.071
5.040
5.014
4.982
4.644
4.617
4.344
4.335
4.316
4.301
4.285
4.276
4.260
4.168
4.163
4.137
4.101
4.098
3.701
3.693
3.686
3.675
3.668
3.661
2.090
2.055
2.035
2.015
1.949
1.944
1.926
1.908
1.902
1.746
1.723
1.701
1.679
1.657
1.635
1.614
0.946
0.924
0.000
hx-10-30
2013-12-29 15:47:38.359
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
AcO
O
O
OAc
H
i-Bu
(Ra)-4ad
5.08
1.00
2.01
2.02
1.01
14.35
1.06
5.95
8
6
4
2
0
PPM

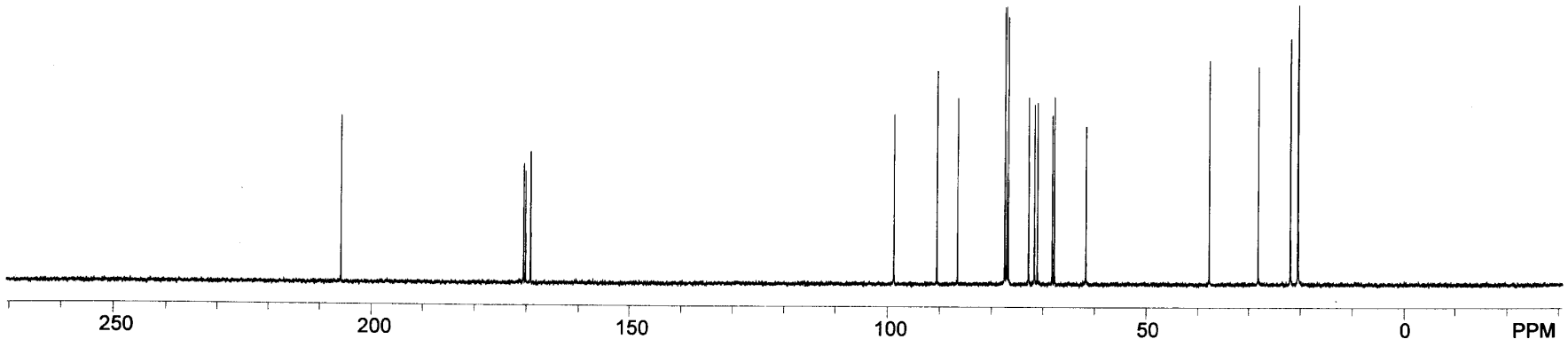




(R_a)-**4ad**

hx-10-30

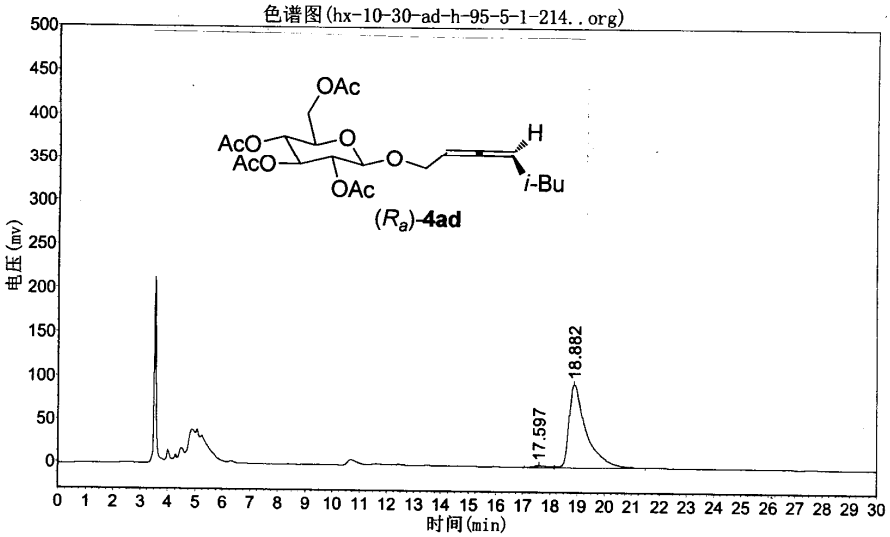
2013-12-29 16:12:07.046
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 395



hx-10-30-ad-h-95-5-1-214

实验时间：2014-01-07,16:03:50　　报告时间：2014-01-09,14:39:08
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-30-ad-h-95-5-1-214..org

实验内容简介：



分析结果表

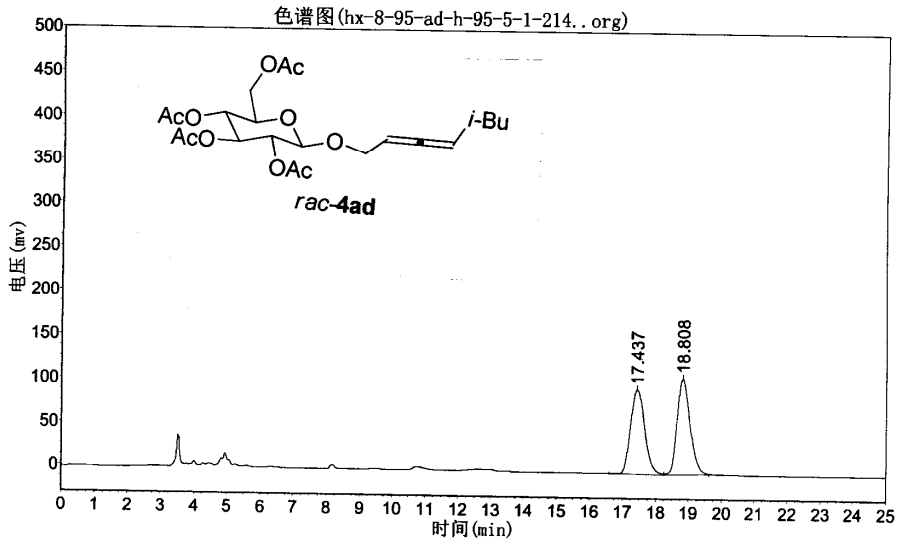
峰号	峰名	保留时间	峰高	峰面积	含量
1		17.597	1936.501	72566.539	1.7016
2		18.882	95601.313	4191978.500	98.2984
总计			97537.813	4264545.039	100.0000

hx-8-95-ad-h-95-5-1-214

实验时间：2014-01-07,15:35:45
谱图文件:D:\zhuguangjiong\hx\20140107\hx-8-95-ad-h-95-5-1-214..org

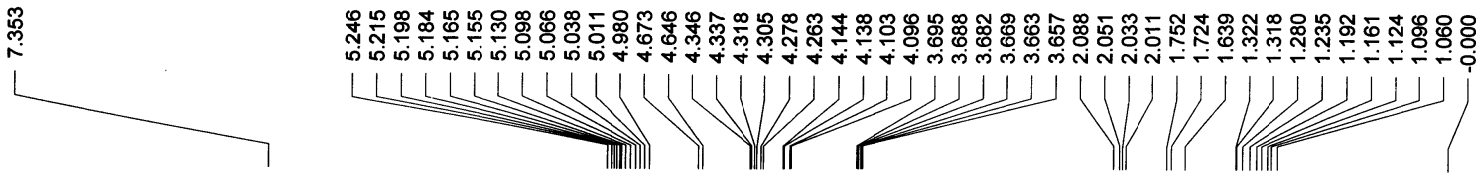
报告时间：2014-01-09,14:27:08

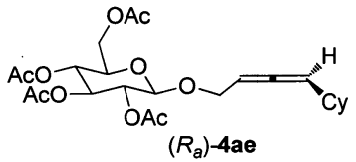
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.437	95504.164	2853676.250	49.2189
2		18.808	108893.492	2944248.500	50.7811
总计			204397.656	5797924.750	100.0000

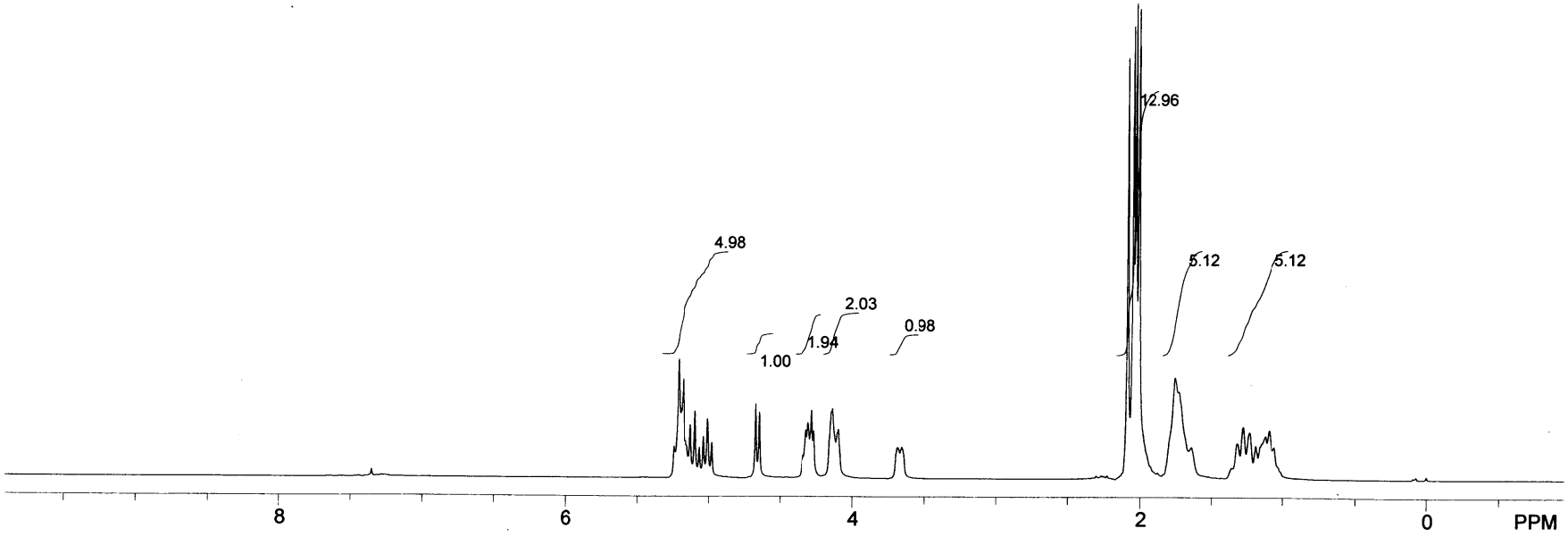


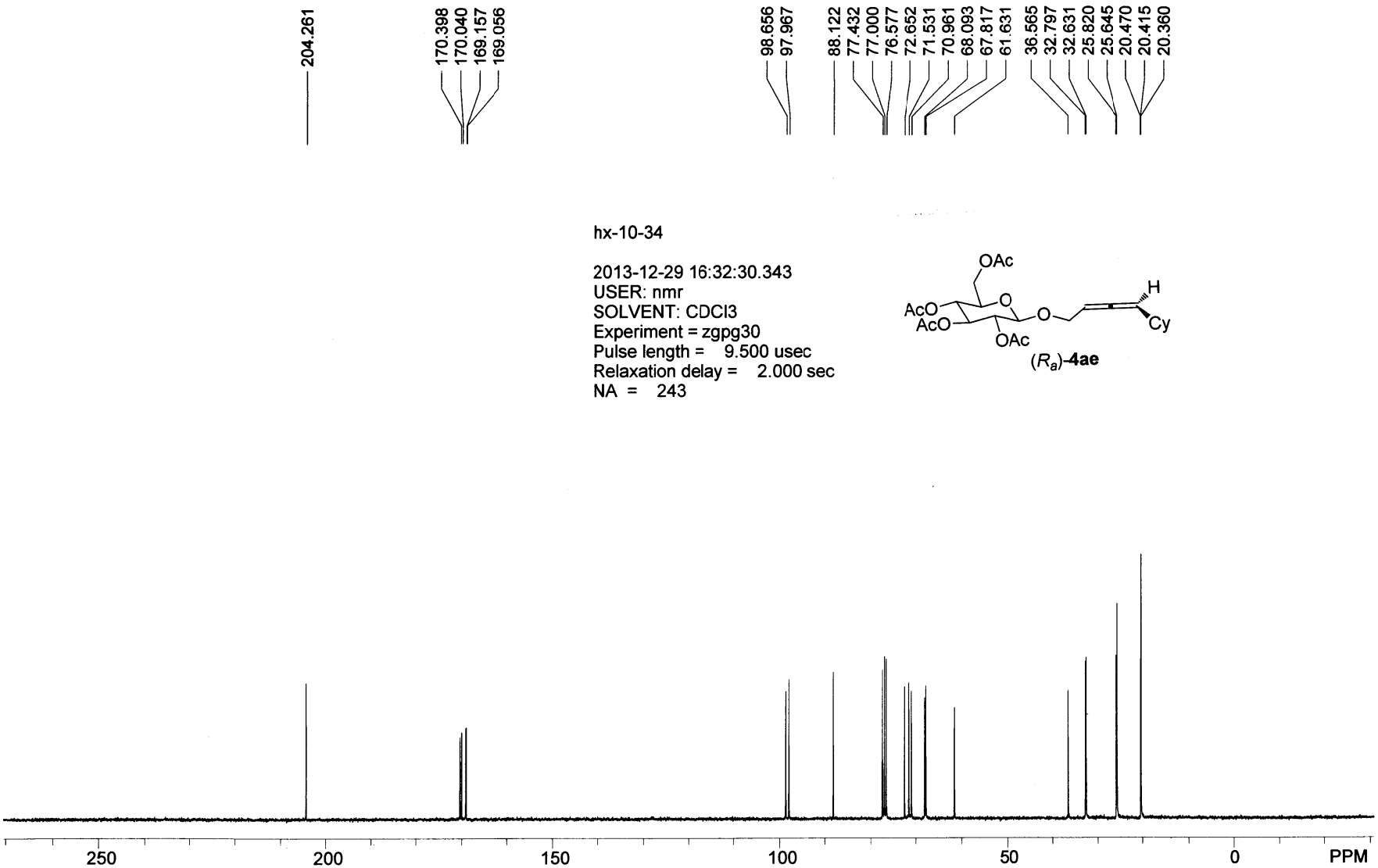


(R_a)-**4ae**

hx-10-34

2013-12-29 16:16:54.921
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8

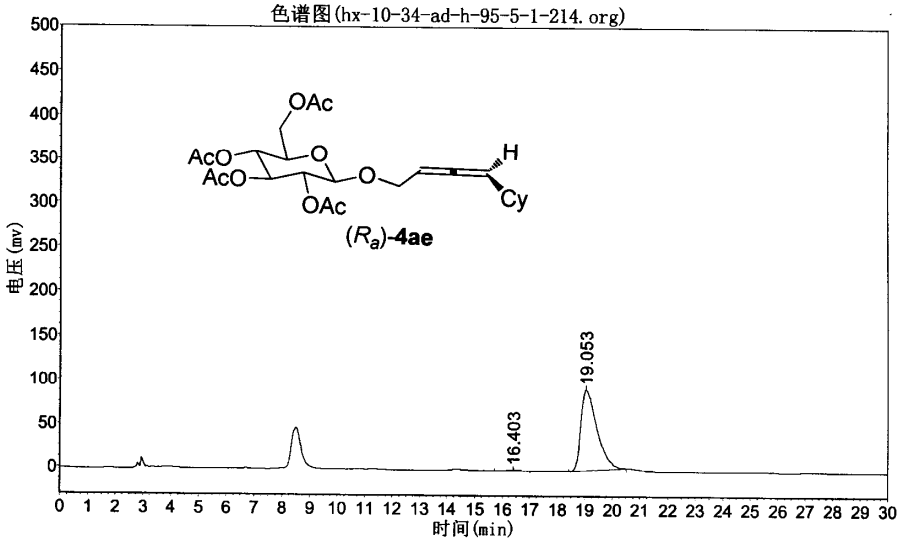



204.261
170.398
170.040
169.157
169.056
98.656
97.967
88.122
77.432
77.000
76.577
72.652
71.531
70.961
68.093
67.817
61.631
36.565
32.797
32.631
25.820
25.645
20.470
20.415
20.360
hx-10-34
2013-12-29 16:32:30.343
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 243
OAc
AcO
AcO
OAc
O
O
H
Cy
(Ra)-4ae
250
200
150
100
50
0
PPM

hx-10-34-ad-h-95-5-1-214

实验时间：2014-01-09,9:40:47　　报告时间：2014-01-09,14:25:43
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-34-ad-h-95-5-1-214.org

实验内容简介：



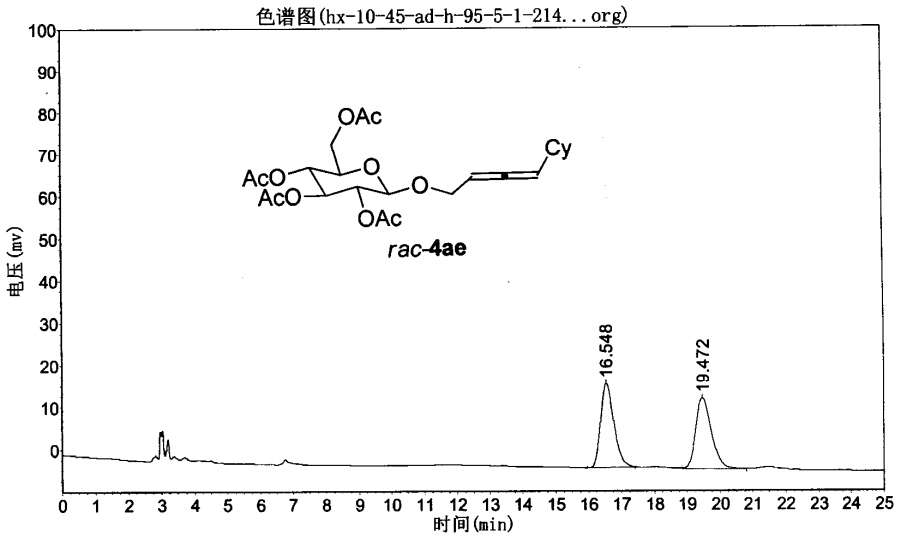
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.403	819.081	25692.760	0.6863
2		19.053	91244.938	3717946.750	99.3137
总计			92064.018	3743639.510	100.0000

hx-10-45-ad-h-95-5-1-214

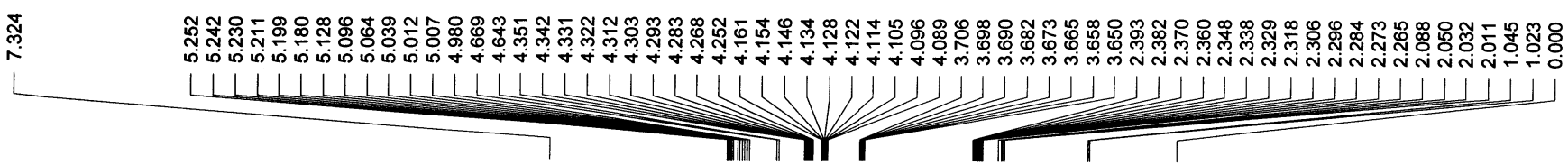
实验时间：2014-01-09,8:50:28　　报告时间：2014-01-09,14:23:55
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-45-ad-h-95-5-1-214...org

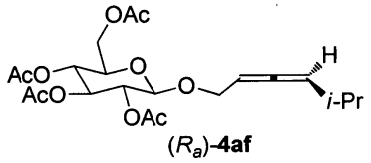
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.548	19824.600	556005.563	49.9148
2		19.472	16627.025	557902.688	50.0852
总计			36451.625	1113908.250	100.0000

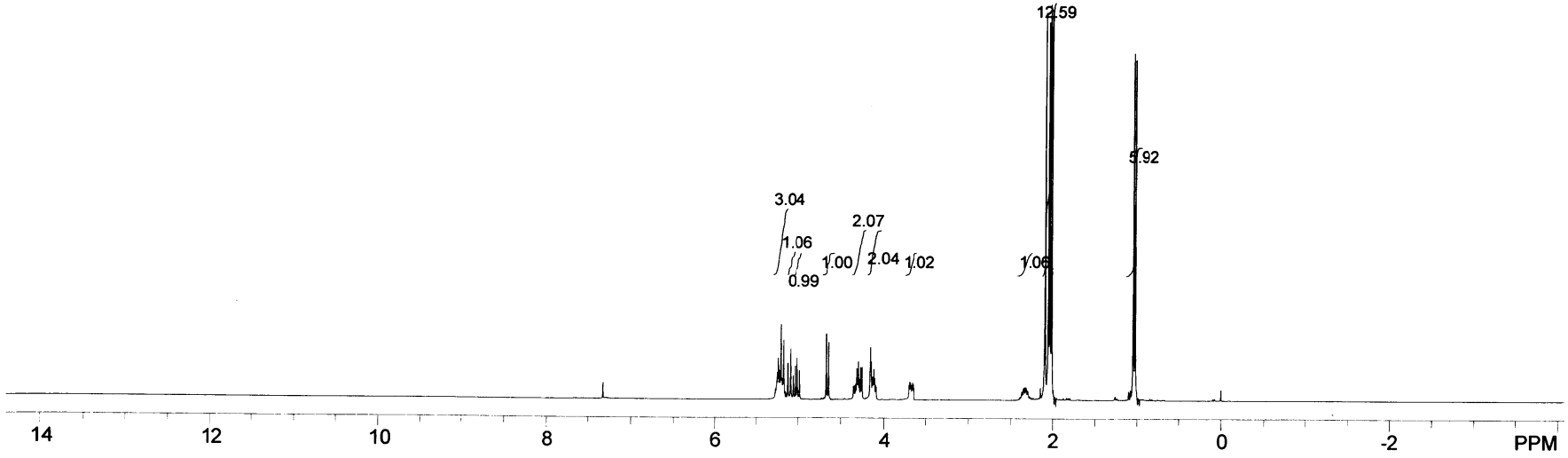




(R_a)-**4af**

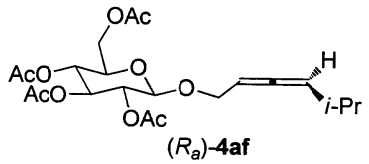
hx-10-40

2014-01-08 19:14:37.500
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 11

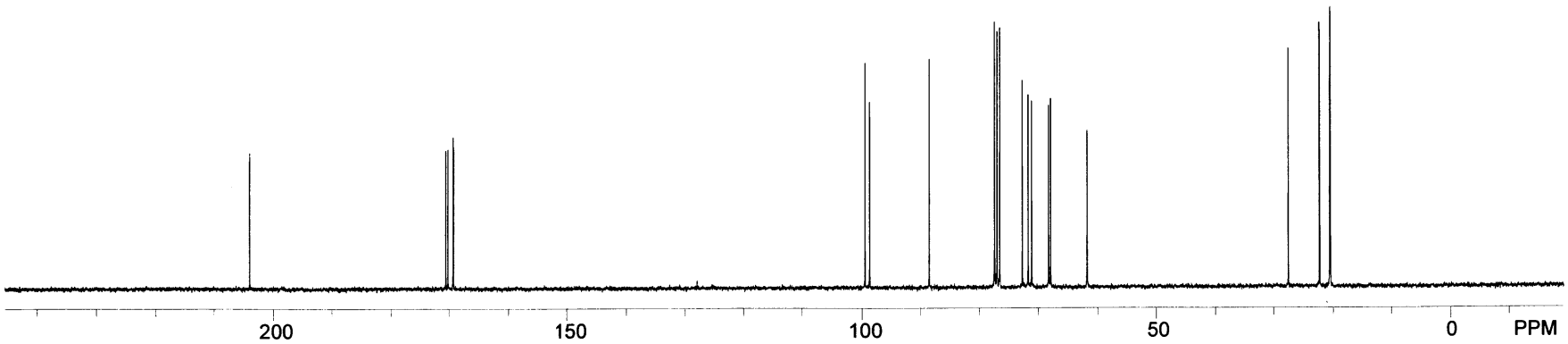


hx-10-40

2014-01-08 19:38:58.562
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 400



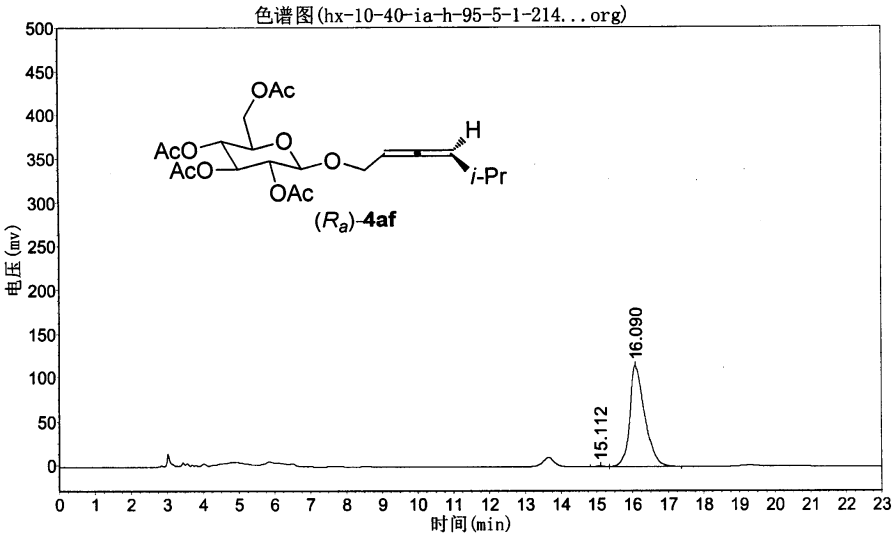
(R_a)-**4af**



hx-10-40-ia-h-95-5-1-214

实验时间：2014-01-08,9:59:07　　报告时间：2014-01-09,14:41:52
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-40-ia-h-95-5-1-214...org

实验内容简介：



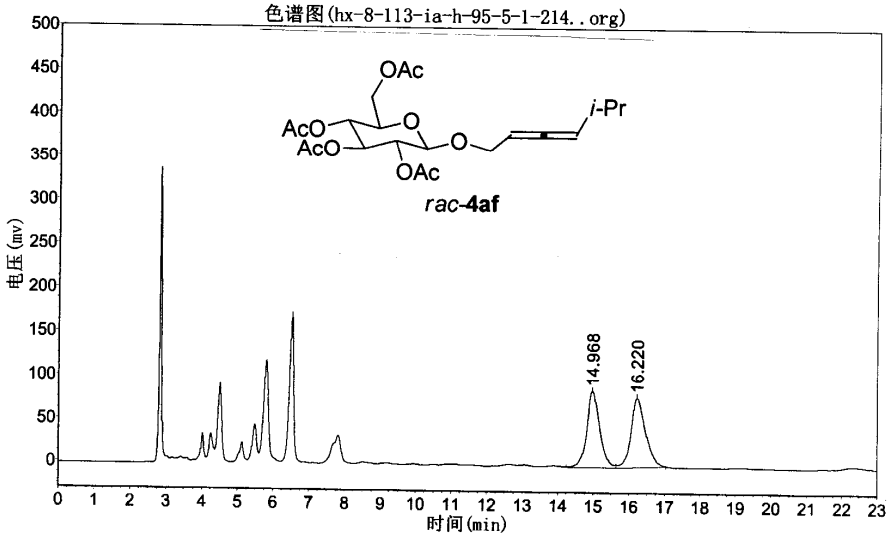
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.112	661.322	13353.986	0.3981
2		16.090	113967.414	3341092.000	99.6019
总计			114628.736	3354445.986	100.0000

hx-8-113-ia-h-95-5-1-214

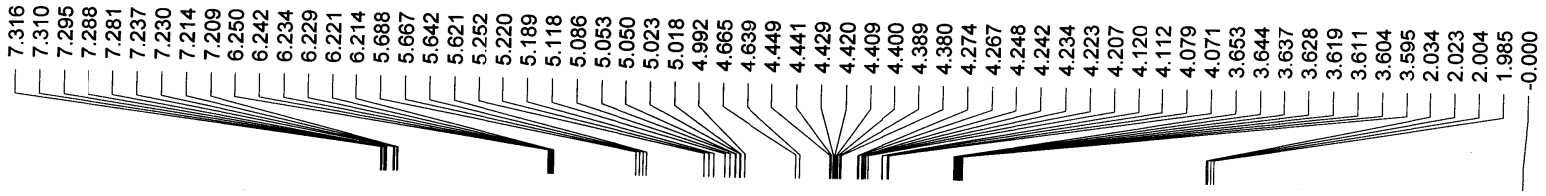
实验时间：2014-01-08,9:04:28　　报告时间：2014-01-09,14:40:49
谱图文件:D:\zhuguangjiong\hx\20140107\hx-8-113-ia-h-95-5-1-214..org

实验内容简介：



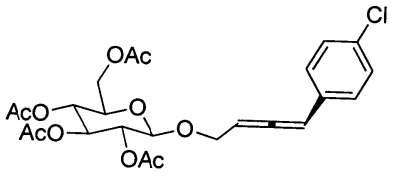
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.968	87313.367	2211914.500	49.7424
2		16.220	79058.297	2234822.250	50.2576
总计			166371.664	4446736.750	100.0000

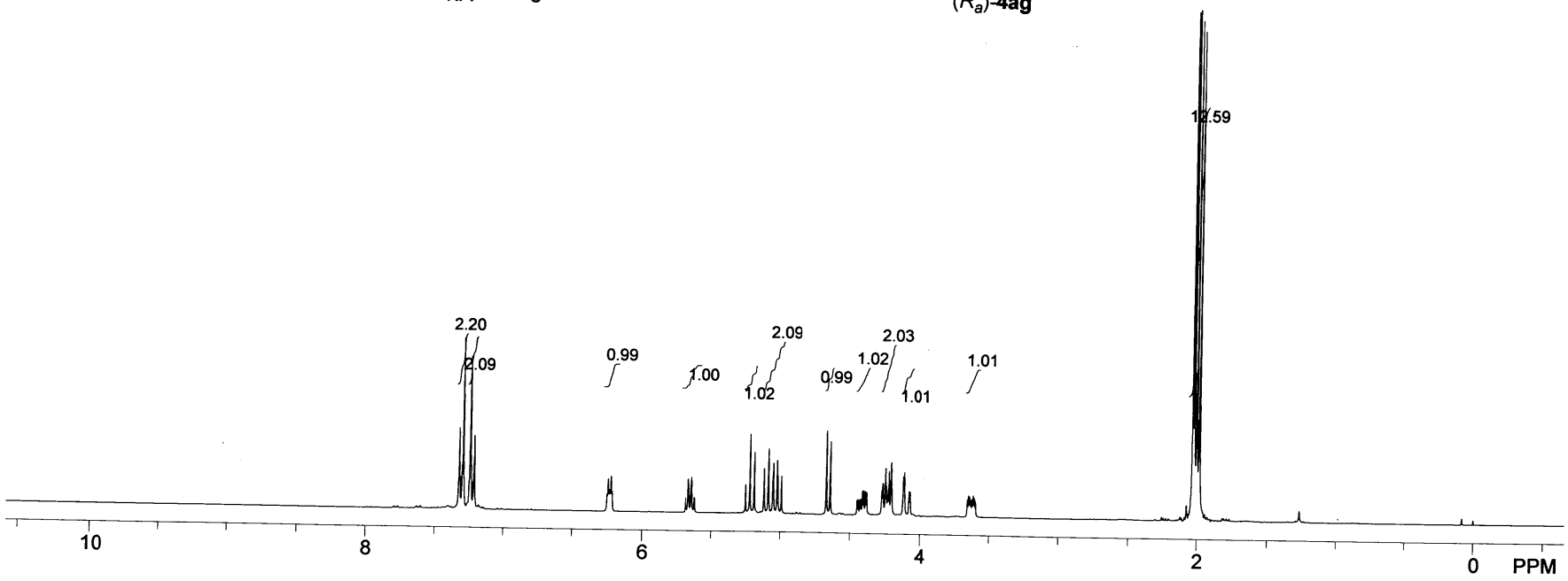


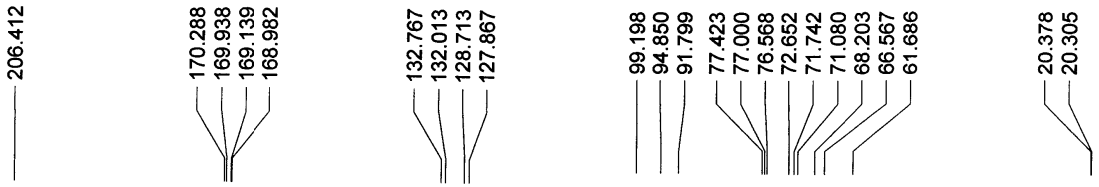
hx-11-91

2014-07-12 20:37:01.906
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



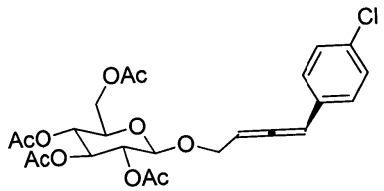
(R_a)-**4ag**



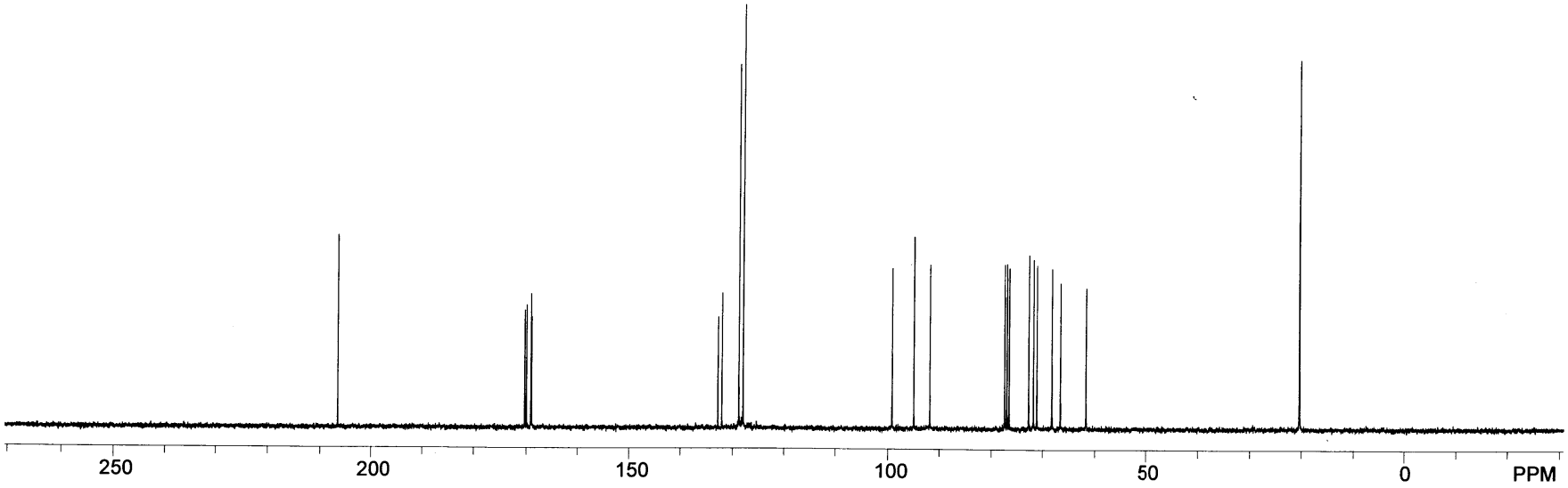


hx-11-91

2014-07-12 20:47:19.062
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 149



(R_a)-**4ag**



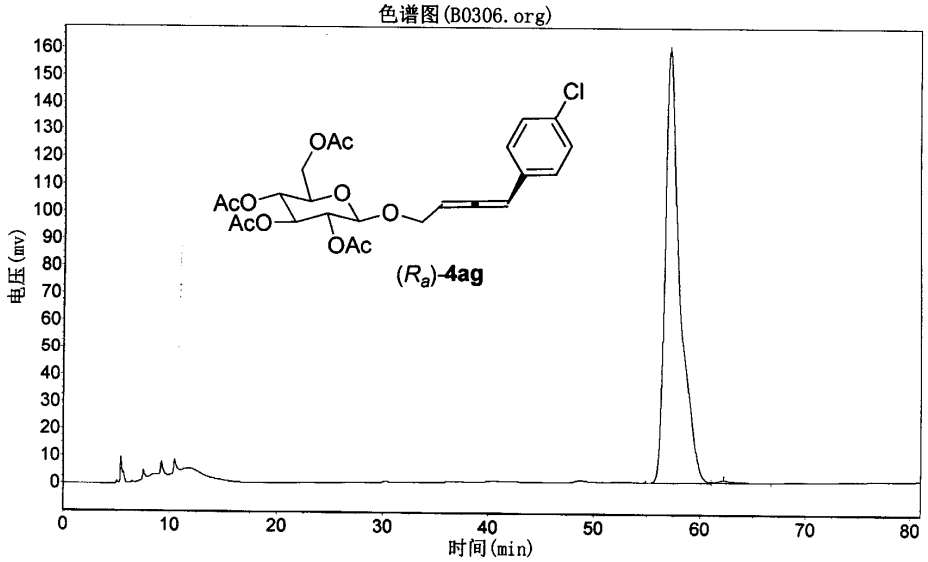
hx-11-91

实验单位：zju
实验时间：2014-07-13,12:15:35
谱图文件:D:\浙大智达\N2000\样品\B0306.org

实验者：hx
报告时间：2014-07-13,13:38:35
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

色谱图(B0306.org)



分析结果表

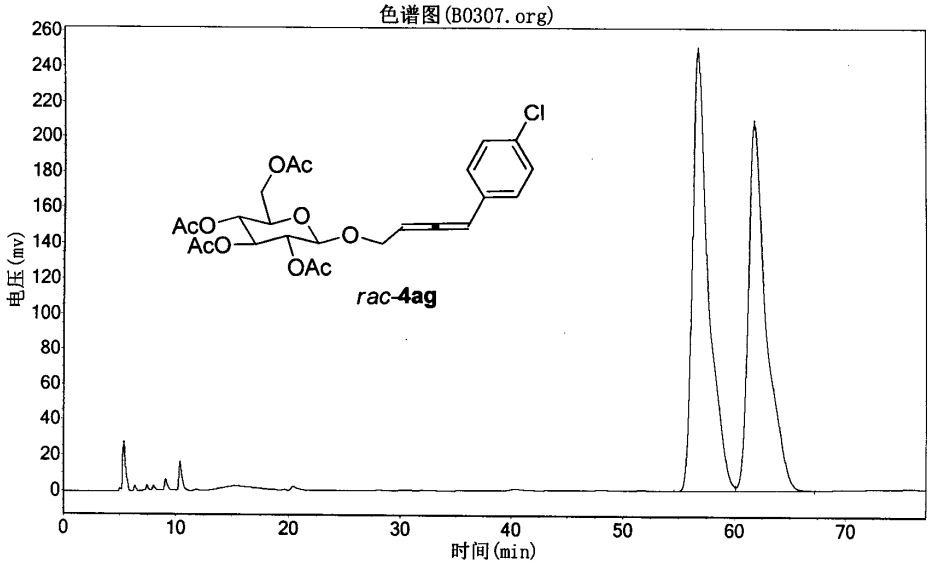
峰号	峰名	保留时间	峰高	峰面积	含量
1		57.147	158217.703	15314433.000	99.4761
2		62.250	812.018	80649.086	0.5239
总计			159029.722	15395082.086	100.0000

hx-11-88

实验单位：zju
实验时间：2014-07-13,13:39:59
谱图文件:D:\浙大智达\N2000\样品\B0307.org

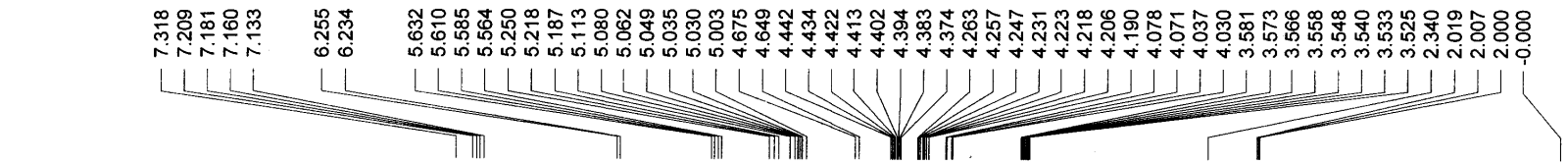
实验者：hx
报告时间：2014-07-13,14:59:12
积分方法:面积归一法

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



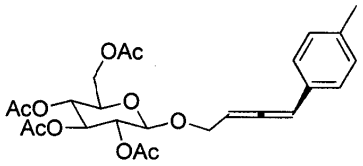
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		56.707	248123.828	24632214.000	51.8366
2		61.752	206067.656	22886768.000	48.1634
总计			454191.484	47518982.000	100.0000

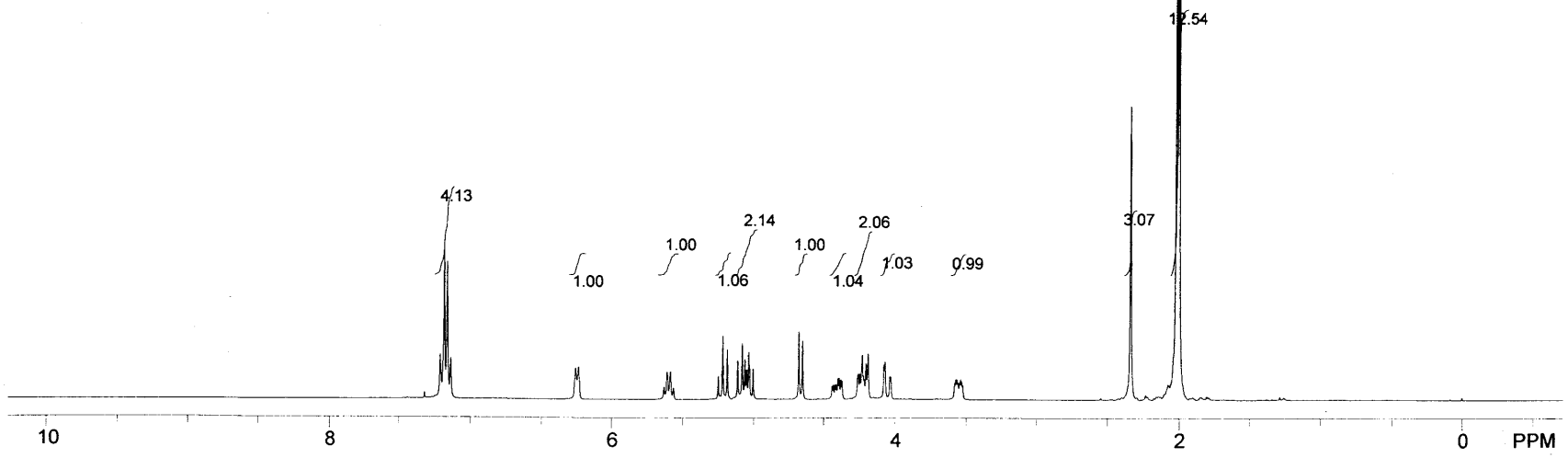


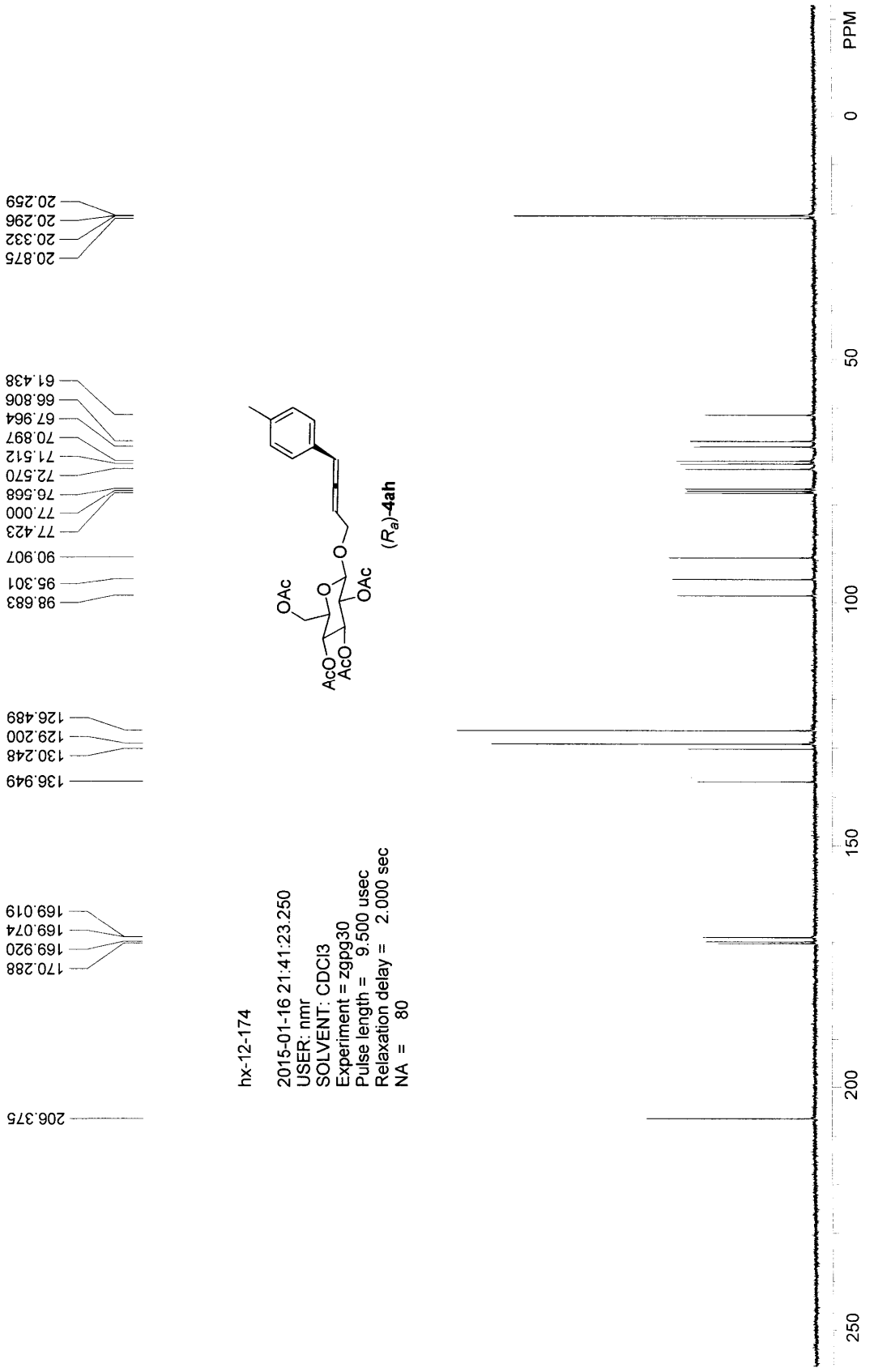
hx-12-174

2015-01-16 21:34:52.578
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ah**

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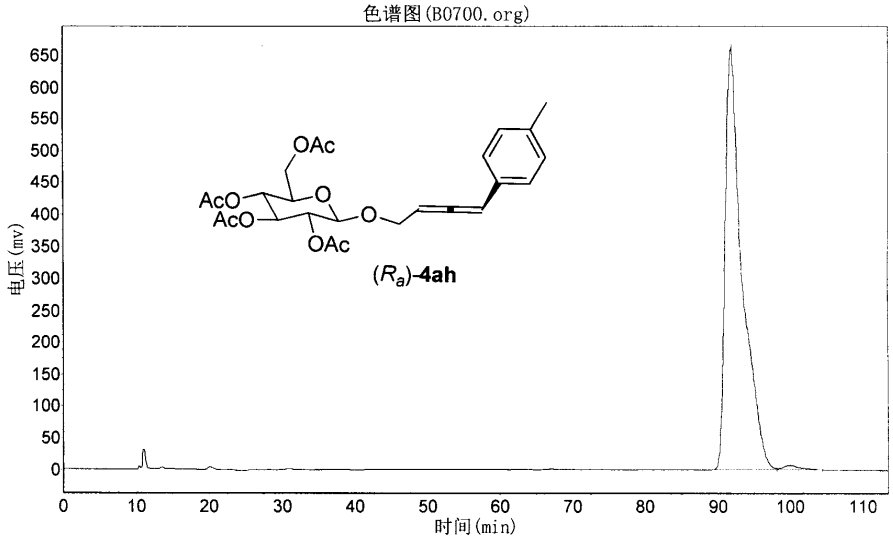
20.296
20.332
20.875
61.438
66.806
67.964
70.897
71.512
72.570
76.568
77.000
77.423
90.907
95.301
98.683
126.489
129.200
130.248
136.949
169.019
169.074
169.920
170.288
206.375
hx-12-174
2015-01-16 21:41:23.250
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 80
OAc
AcO
AcO
OAc
O
O
O
(Ra)-4ah
250
200
150
100
50
0
PPM

hx-12-174

实验单位：zju
实验时间：2015-01-18,11:47:09
谱图文件:D:\浙大智达\N2000\样品\B0700.org

实验者：hx
报告时间：2015-01-19,10:17:03
积分方法:面积归一法

实验内容简介：
AD-H, n-hexane1/i-PrOH = 94/6, 214 nm, 0.6 ml/min



分析结果表

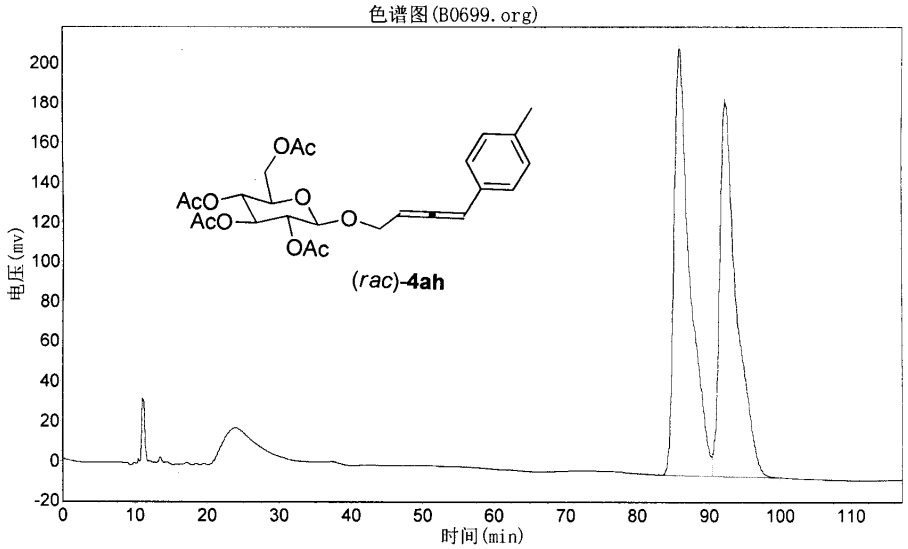
峰号	峰名	保留时间	峰高	峰面积	含量
1		91.860	662101.313	107778720.000	98.8298
2		99.942	7587.738	1276131.375	1.1702
总计			669689.051	109054851.375	100.0000

hx-12-175

实验单位：zju
实验时间：2015-01-18,9:42:47
谱图文件:D:\浙大智达\N2000\样品\B0699.org

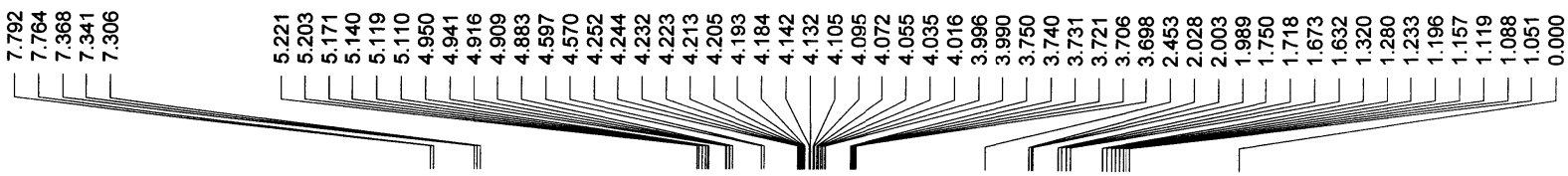
实验者：hx
报告时间：2015-01-19,10:15:16
积分方法:面积归一法

实验内容简介：
AD-H, n-hexane1/i-PrOH = 94/6, 214 nm, 0.6 ml/min



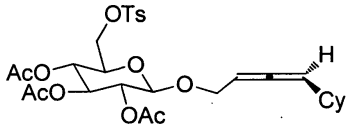
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		85.940	214032.500	33073264.000	51.9456
2		92.350	187784.531	30595828.000	48.0544
总计			401817.031	63669092.000	100.0000

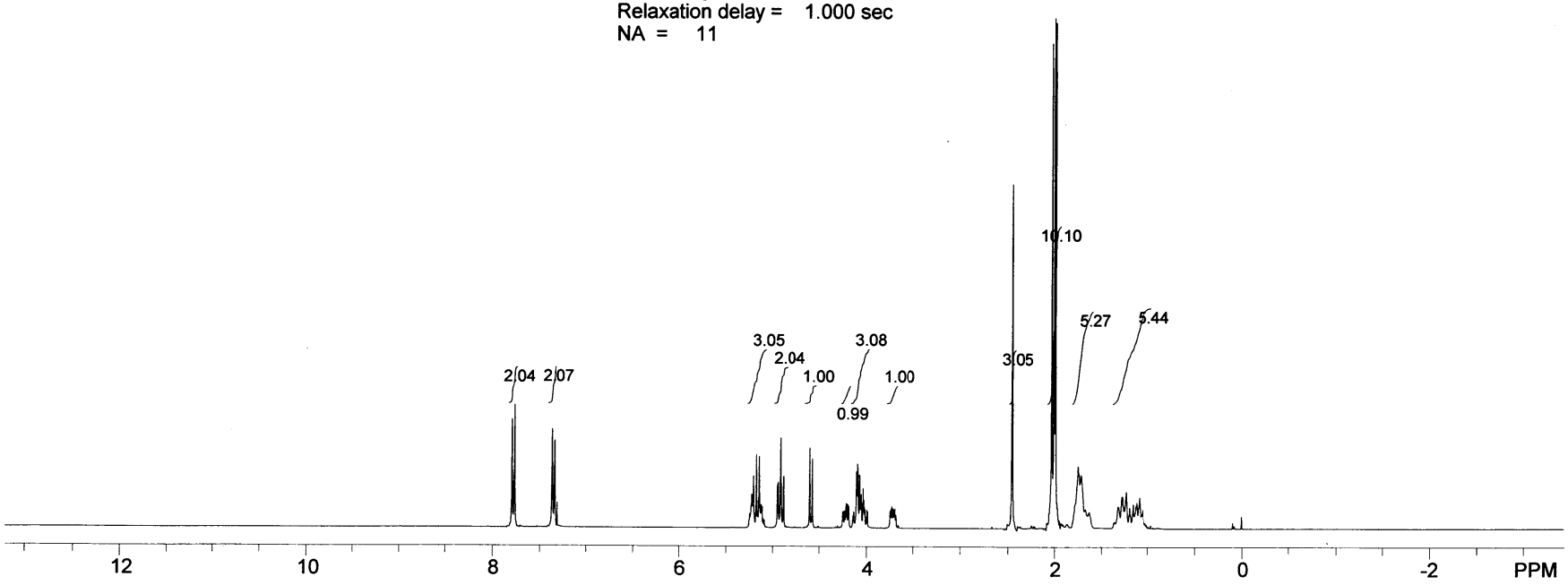


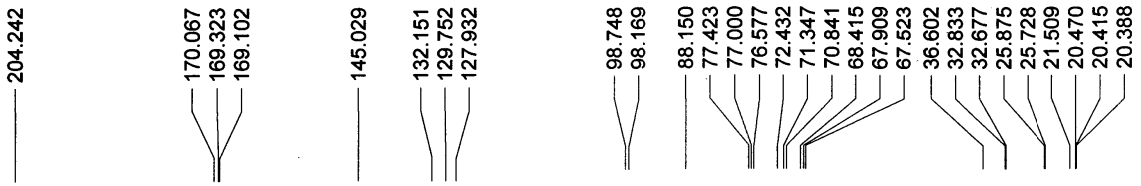
hx-10-57

2014-01-13 19:27:05.484
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 11



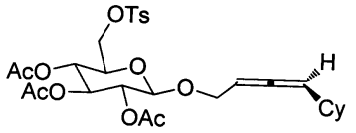
(R_a)-**4be**



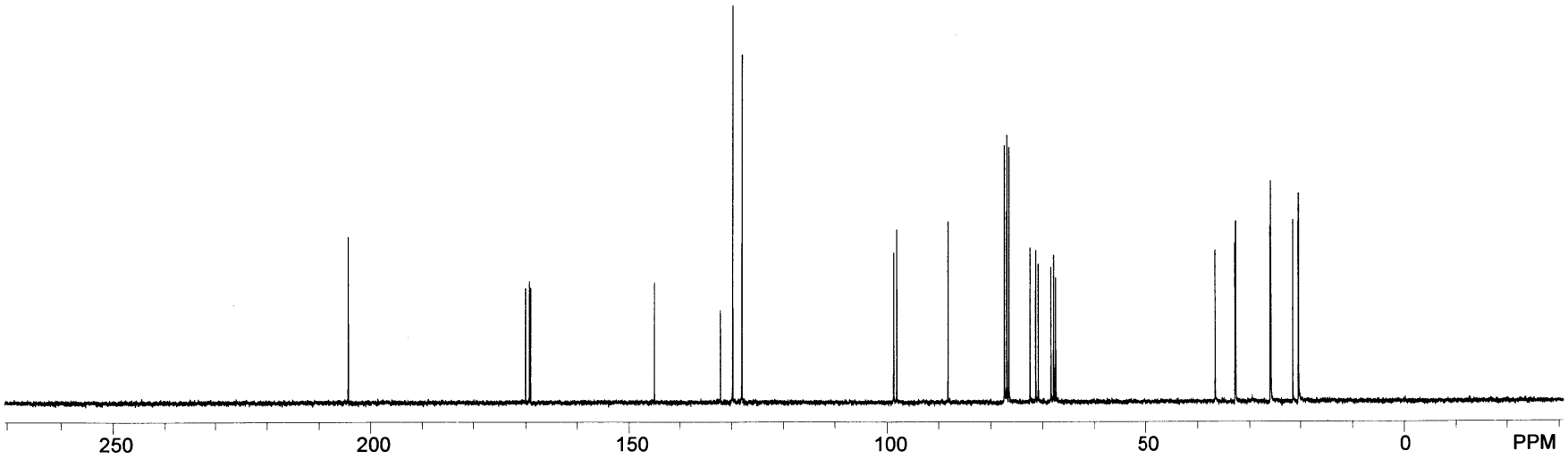


hx-10-57

2014-01-13 19:43:21.953
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 260



(R_a)-**4be**

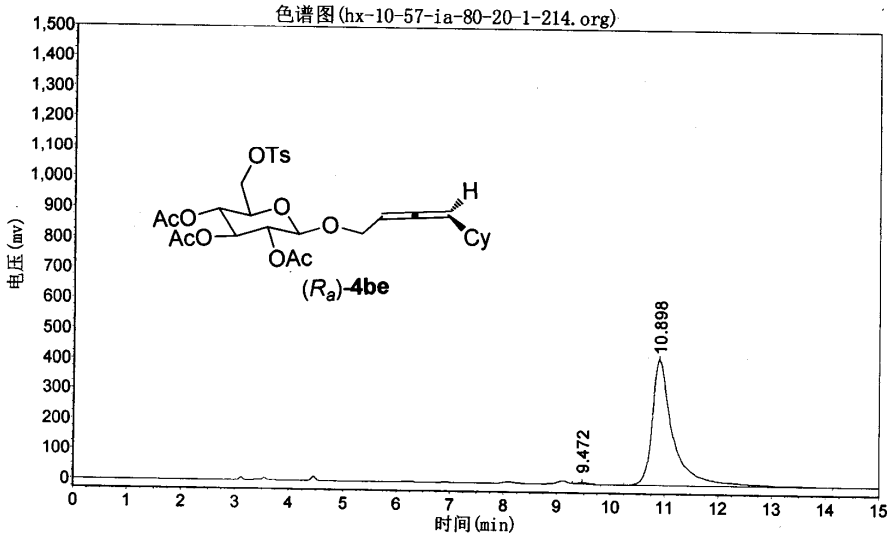


hx-10-57-ia-80-20-1-214

实验时间：2014-01-15,12:33:45
谱图文件:D:\zhuguangjiong\hx\20140115\hx-10-57-ia-80-20-1-214.org

报告时间：2014-01-17,15:25:24

实验内容简介：



分析结果表

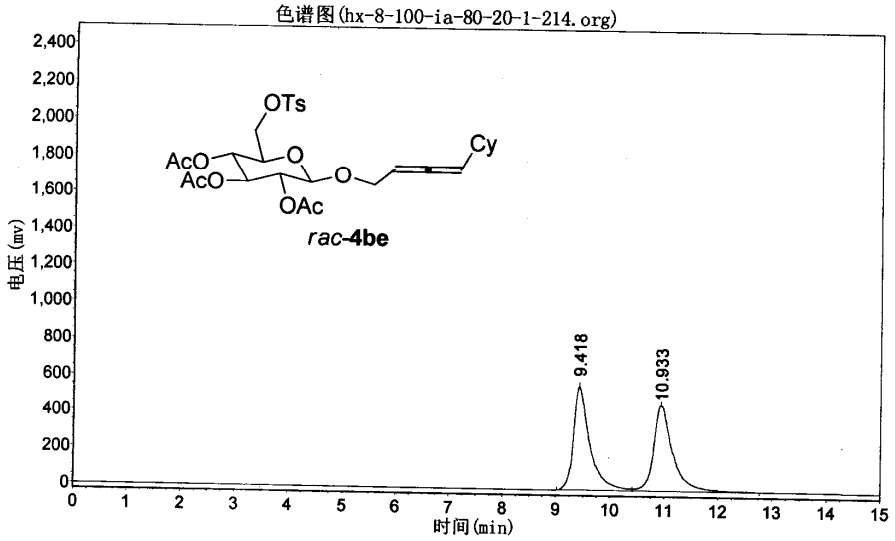
峰号	峰名	保留时间	峰高	峰面积	含量
1		9.472	4041.587	49915.180	0.4298
2		10.898	417859.031	11564718.000	99.5702
总计			421900.619	11614633.180	100.0000

hx-8-100-ia-80-20-1-214

实验时间：2014-01-15,11:39:33
谱图文件:D:\zhuguangjiong\hx\20140115\hx-8-100-ia-80-20-1-214.org

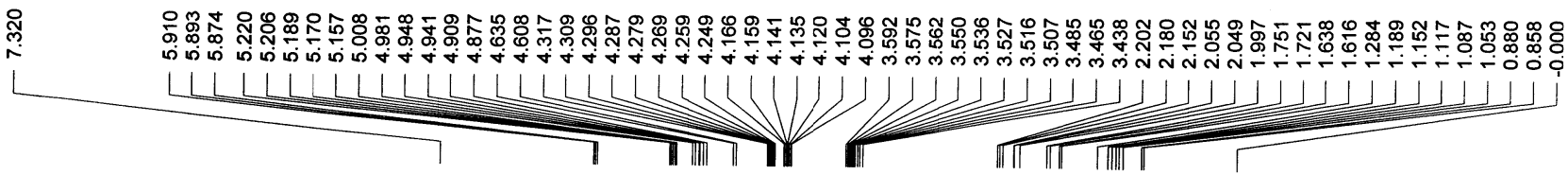
报告时间：2014-01-17,15:24:12

实验内容简介：



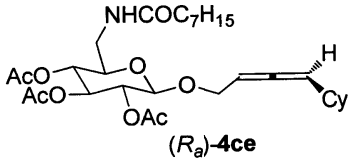
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.418	566933.063	11880676.000	50.9917
2		10.933	464119.688	11418548.000	49.0083
总计			1031052.750	23299224.000	100.0000

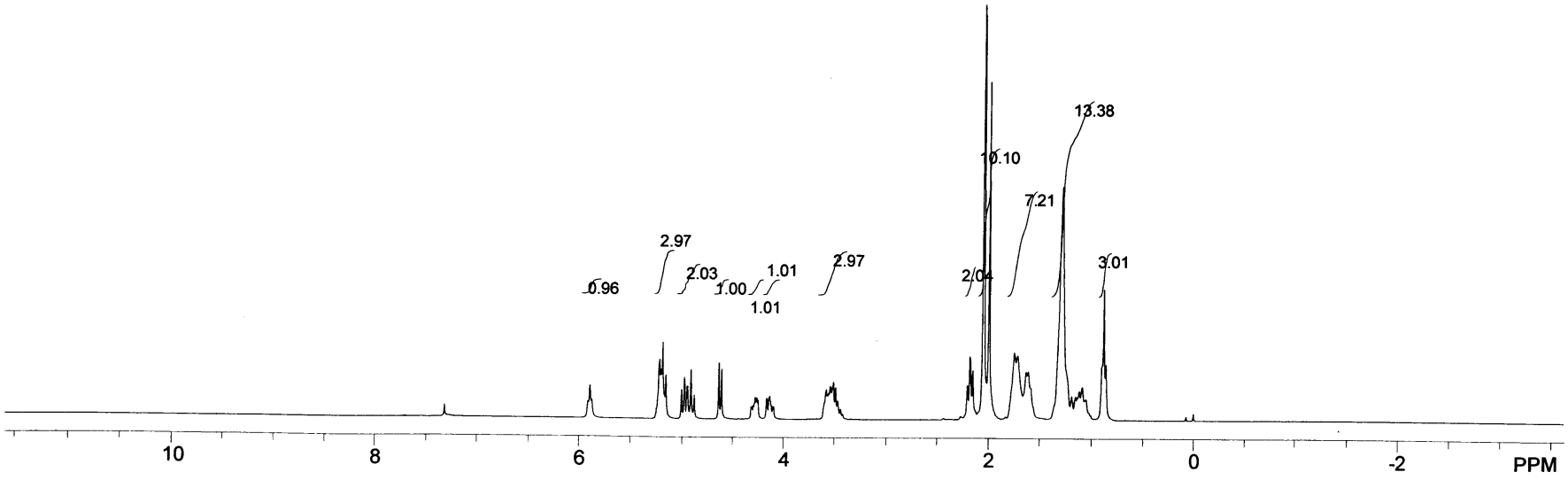


hx-10-56

2014-01-07 14:22:19.406
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8

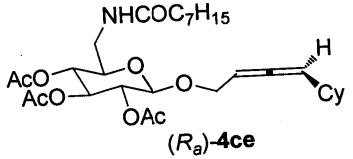


(R_a)-**4ce**

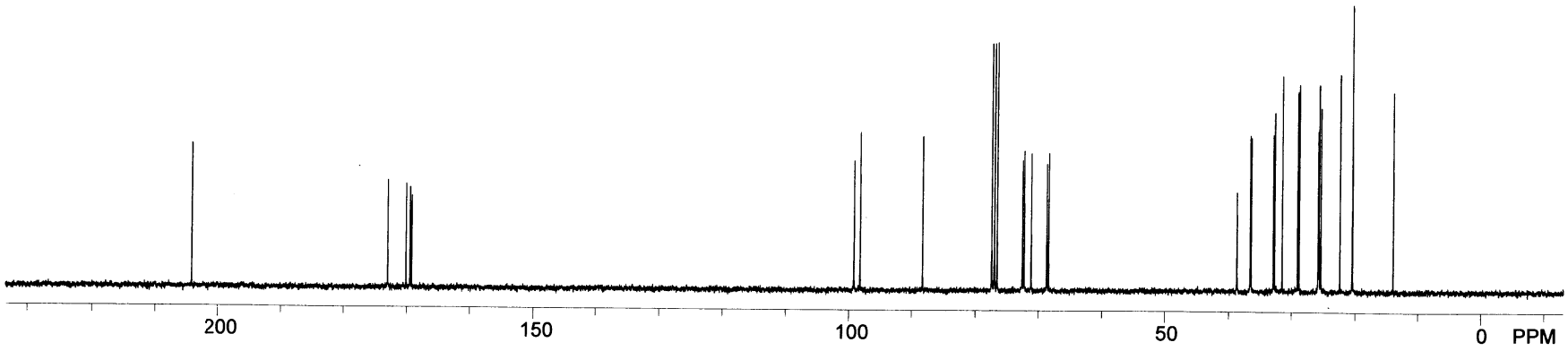


hx-10-56

2014-01-08 10:42:18.671
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 202



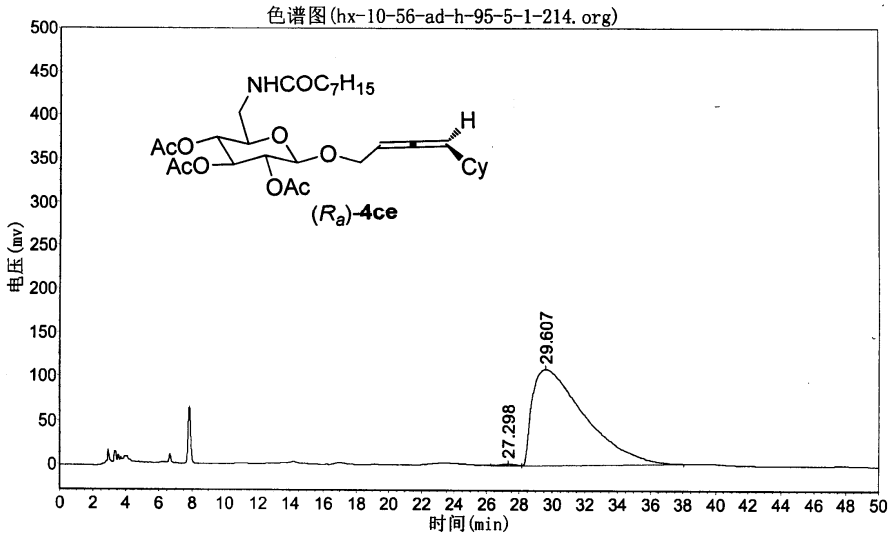
(R_a)-**4ce**



hx-10-56-ad-h-95-5-1-214

实验时间：2014-01-09,10:13:49　　报告时间：2014-01-09,14:20:32
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-56-ad-h-95-5-1-214.org

实验内容简介：



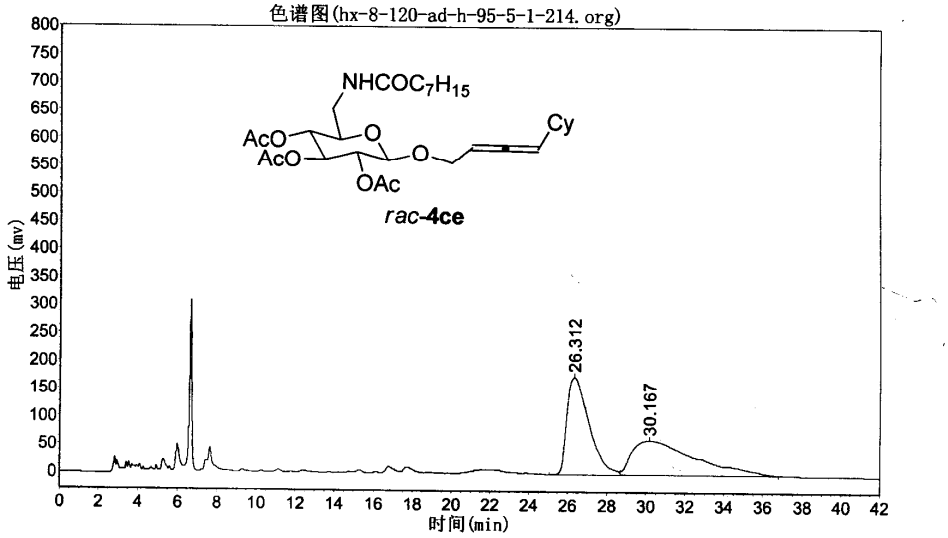
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.298	1821.676	111795.945	0.4631
2		29.607	107752.578	24028170.000	99.5369
总计			109574.254	24139965.945	100.0000

hx-8-120-ad-h-95-5-1-214

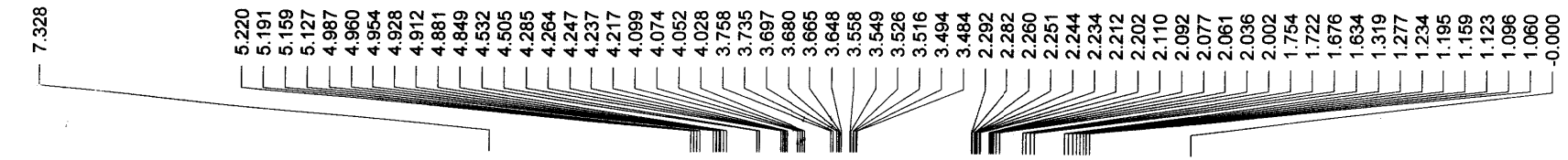
实验时间：2014-01-09,11:23:14　　报告时间：2014-01-09,14:22:20
谱图文件:D:\zhuguangjiong\hx\20140107\hx-8-120-ad-h-95-5-1-214.org

实验内容简介：



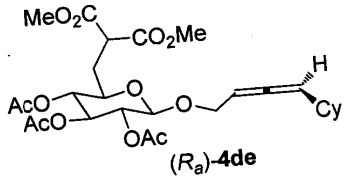
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		26.312	173837.453	13615594.000	50.1499
2		30.167	60579.004	13534182.000	49.8501
总计			234416.457	27149776.000	100.0000

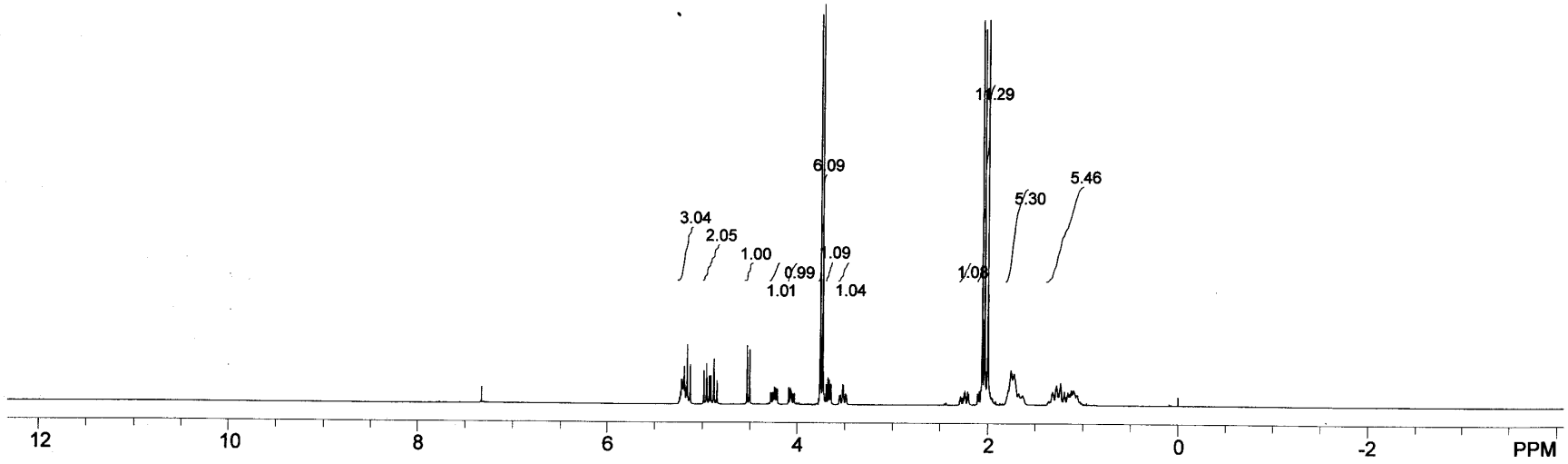


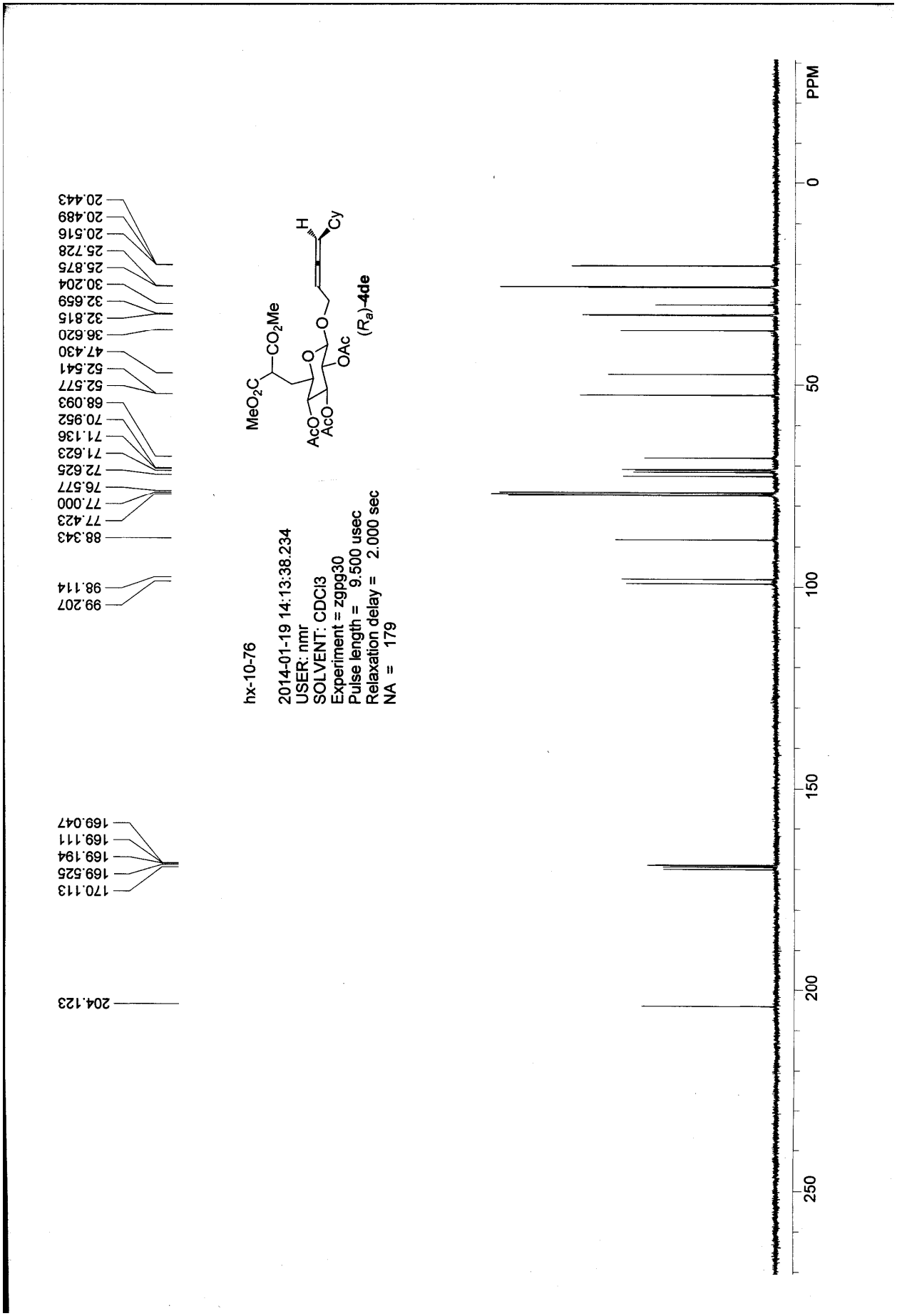
hx-10-76

2014-01-19 14:01:31.718
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4de**

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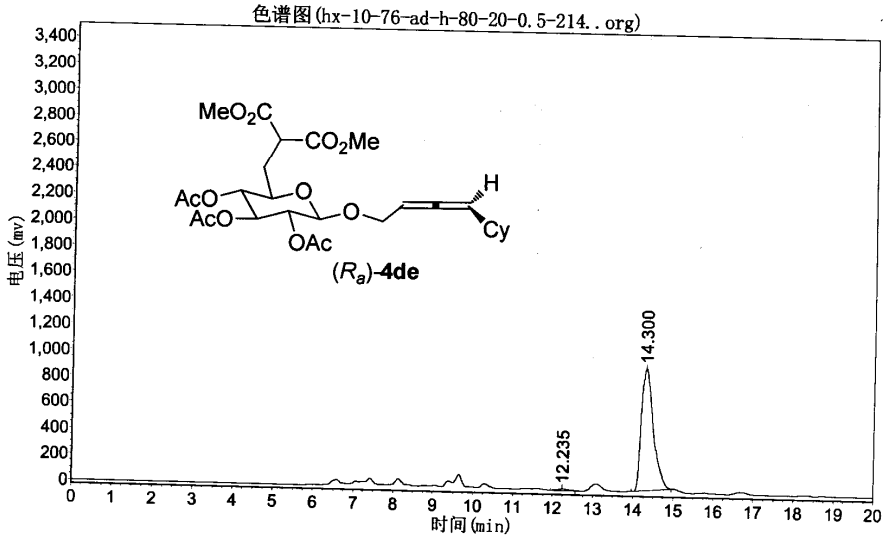
20.489
20.516
25.728
25.875
30.204
32.659
32.815
36.620
47.430
52.541
52.577
68.093
70.952
71.136
71.623
72.625
76.577
77.000
77.423
88.343
98.114
99.207
170.113
169.525
169.194
169.111
169.047
204.123
hx-10-76
2014-01-19 14:13:38.234
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 179
MeO2C
CO2Me
AcO
AcO
OAc
H
Cy
(Ra)-4de
PPM
250
200
150
100
50
0

hx-10-76-ad-h-80-20-0.5-214

实验时间：2014-01-22,14:38:14
谱图文件:D:\zhuguangjiong\hx\20140122\hx-10-76-ad-h-80-20-0.5-214..org

报告时间：2014-01-22,15:43:11

实验内容简介：



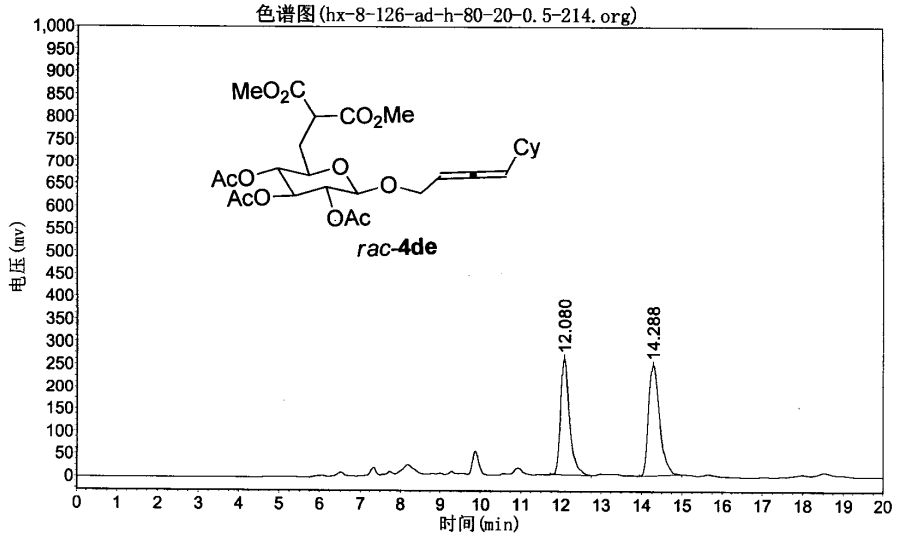
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.235	12098.110	184974.281	0.9560
2		14.300	931759.688	19162808.000	99.0440
总计			943857.798	19347782.281	100.0000

hx-8-126-ad-h-80-20-0.5-214

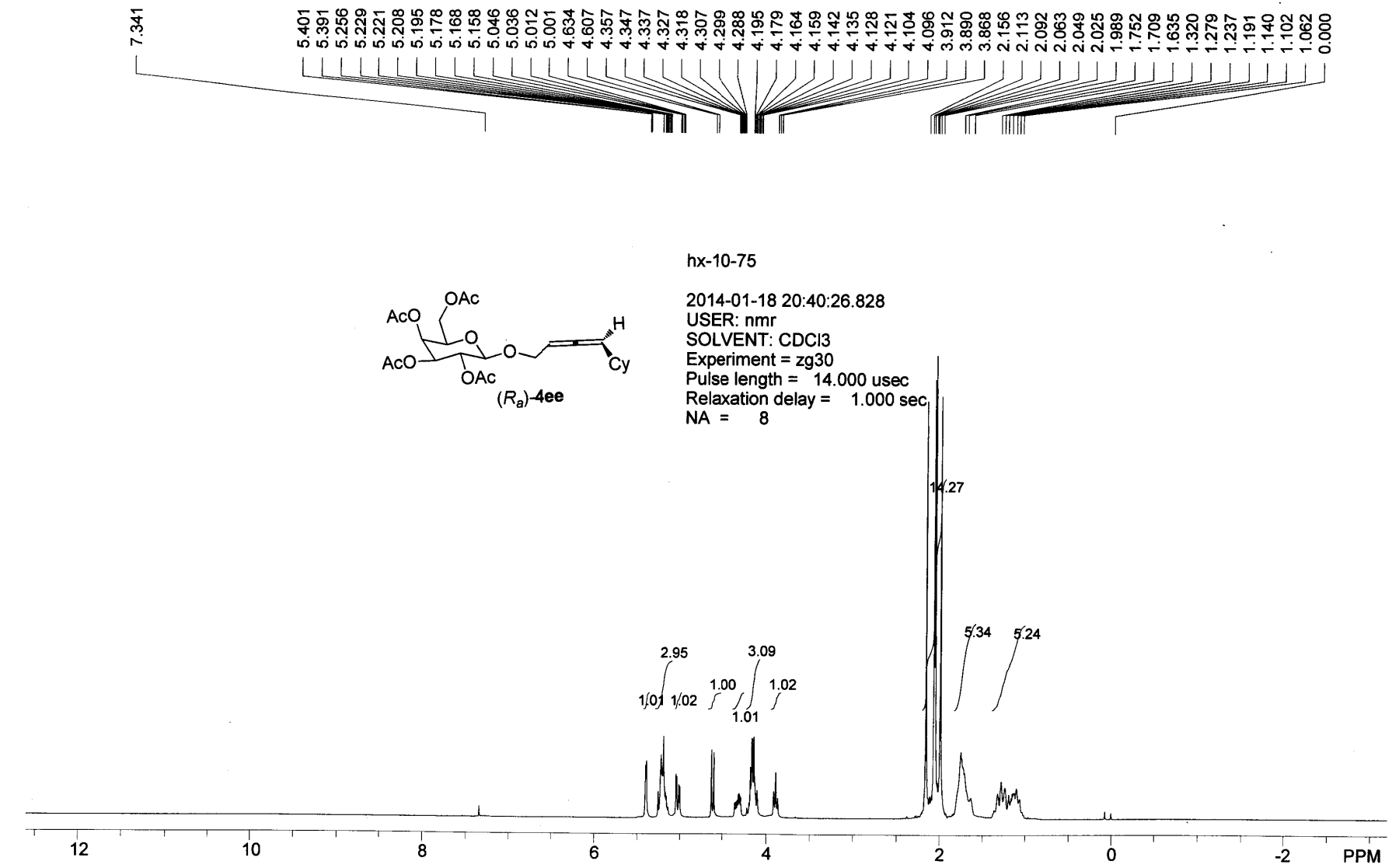
实验时间：2014-01-22,13:34:04　　报告时间：2014-01-22,15:40:07
谱图文件:D:\zhuguangjiong\hx\20140122\hx-8-126-ad-h-80-20-0.5-214.org

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		12.080	258087.094	3988516.750	45.8252
2		14.288	245347.188	4715249.000	54.1748
总计			503434.281	8703765.750	100.0000


7.341
5.401
5.391
5.256
5.229
5.221
5.208
5.195
5.178
5.168
5.158
5.046
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5.012
5.001
4.634
4.607
4.357
4.347
4.337
4.327
4.318
4.307
4.299
4.288
4.195
4.179
4.164
4.159
4.142
4.135
4.128
4.121
4.104
4.096
3.912
3.890
3.868
2.156
2.113
2.092
2.063
2.049
2.025
1.989
1.752
1.709
1.635
1.320
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1.191
1.140
1.102
1.062
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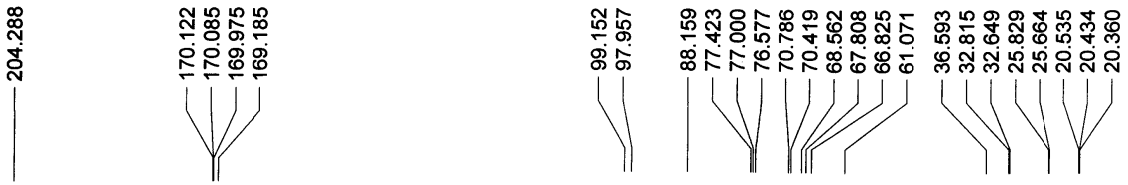
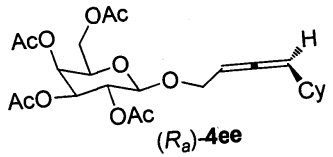
hx-10-75
2014-01-18 20:40:26.828
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
O
H
AcO
O
Cy
OAc
(Ra)-4ee
14.27
5.34
5.24
2.95
3.09
1.00
1.02
1.01
1.01
1.02
12
10
8
6
4
2
0
-2
PPM

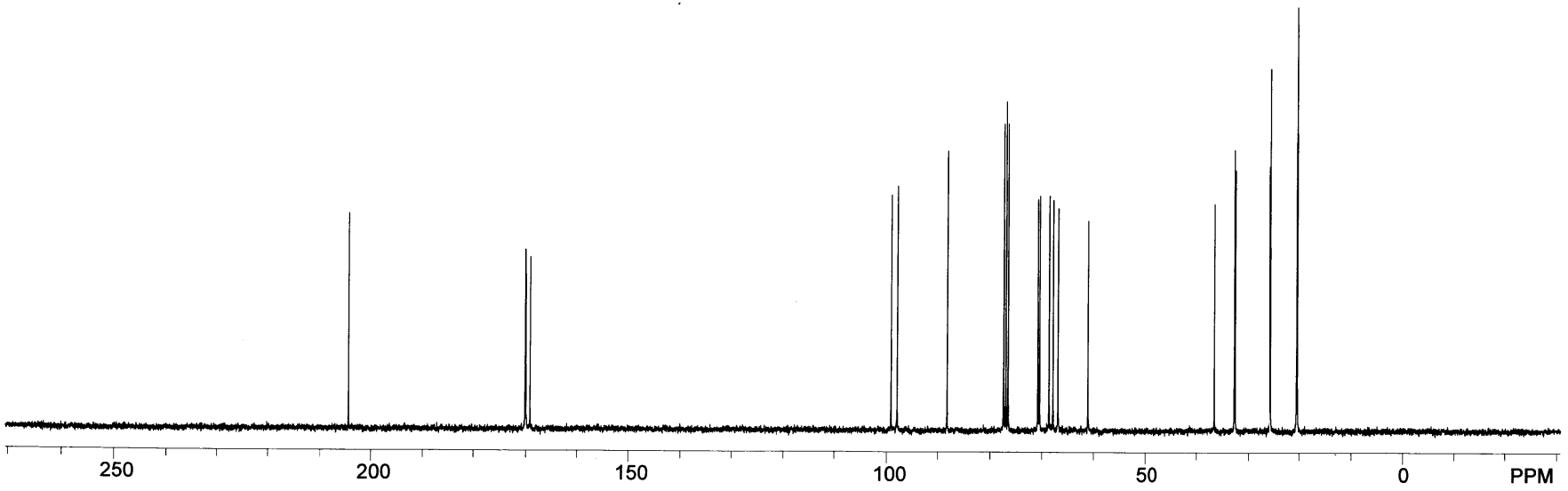


hx-10-75

2014-01-18 20:56:47.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 253



(R_a)-**4ee**



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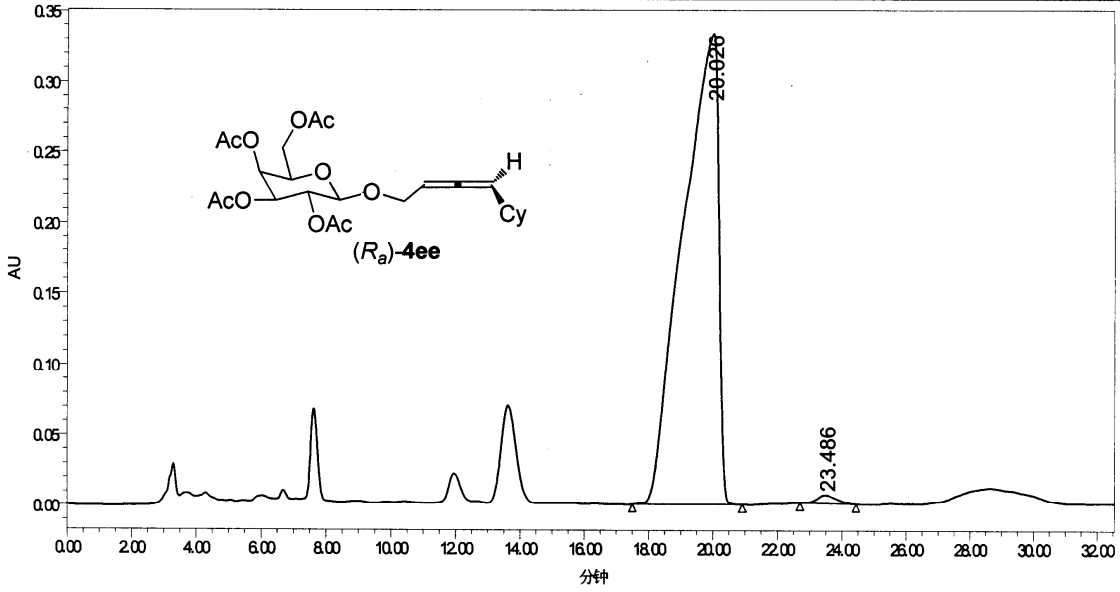
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-10-75-ad-h-95-5-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/1/23 15:00:00 CST
Vial:	1	Acq. Method:	zgj95
Injection #:	34	Date Processed:	2014/1/23 16:40:29 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	300.00 Minutes	Sample Set Name:	



	RT (min)	Area (碛sec)	%Area	Height (碛)	% Height
1	20.02€	2560039€	99.0C	333042	98.19
2	23.48€	25860€	1.0C	613€	1.81

Report Method: 无标题

Page: 1 (共计 1)

Printed: 2014/1/23 17:17:30 PRC

中国科学院上海有机化学研究所

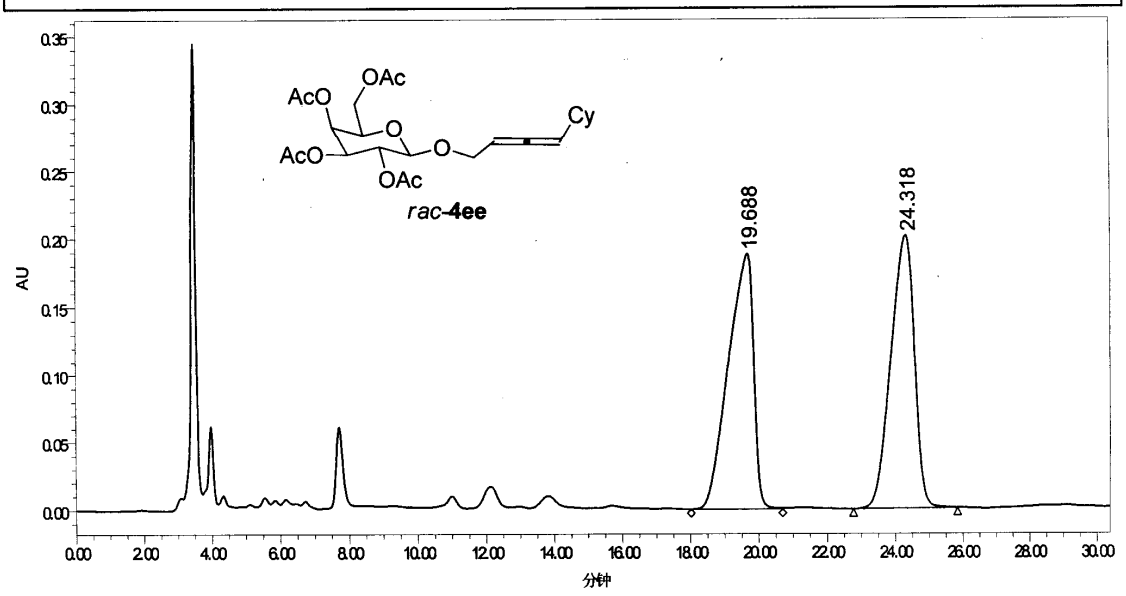
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-10-83-ad-h-95-5-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/1/23 16:09:02 CST
Vial:	1	Acq. Method:	zgj95
Injection #:	36	Date Processed:	2014/1/23 16:39:51 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	300.00 Minutes	Sample Set Name:	



	RT (min)	Area (微sec)	%Area	Height (微	% Height
1	19.688	9693001	50.11	188182	48.34
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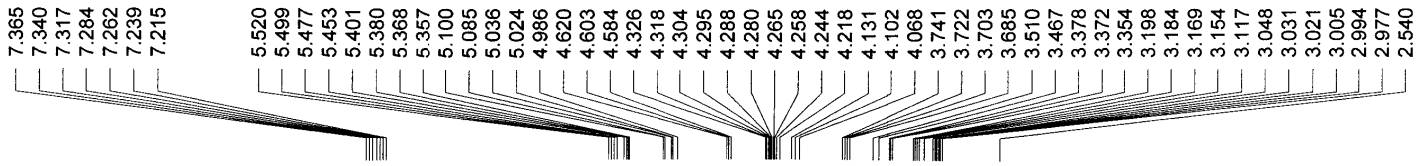
Report Method: 无标题

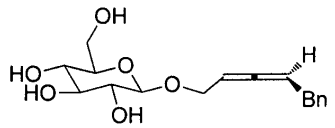
Page: 1 (共计 1)

Printed: 2014/1/23 16:40:43 PRC

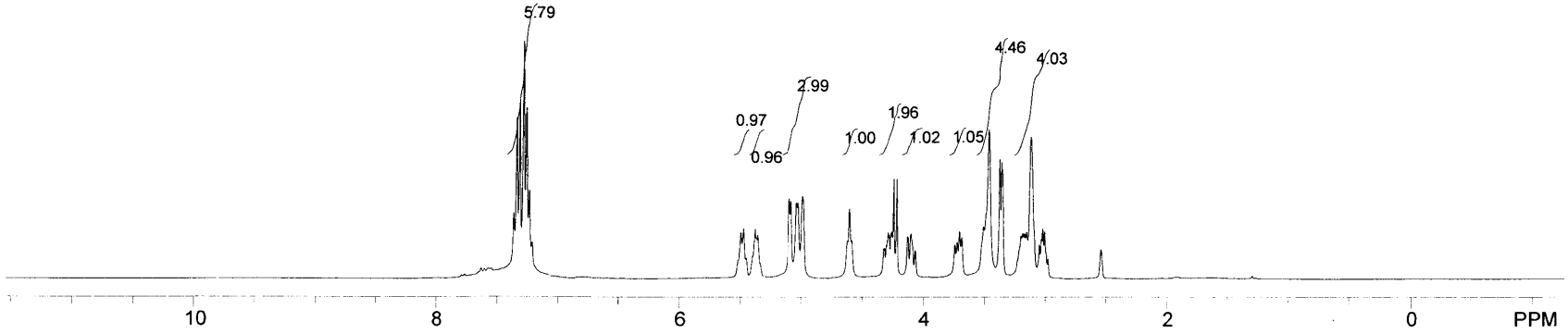


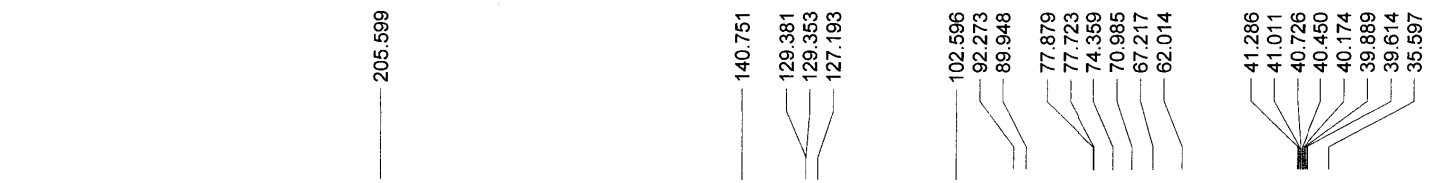
hx-12-190-DMSO

2015-02-06 22:44:00.578
USER: nmr
SOLVENT: DMSO
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 13



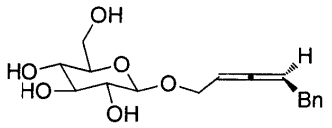
(R_a)-**4ob**



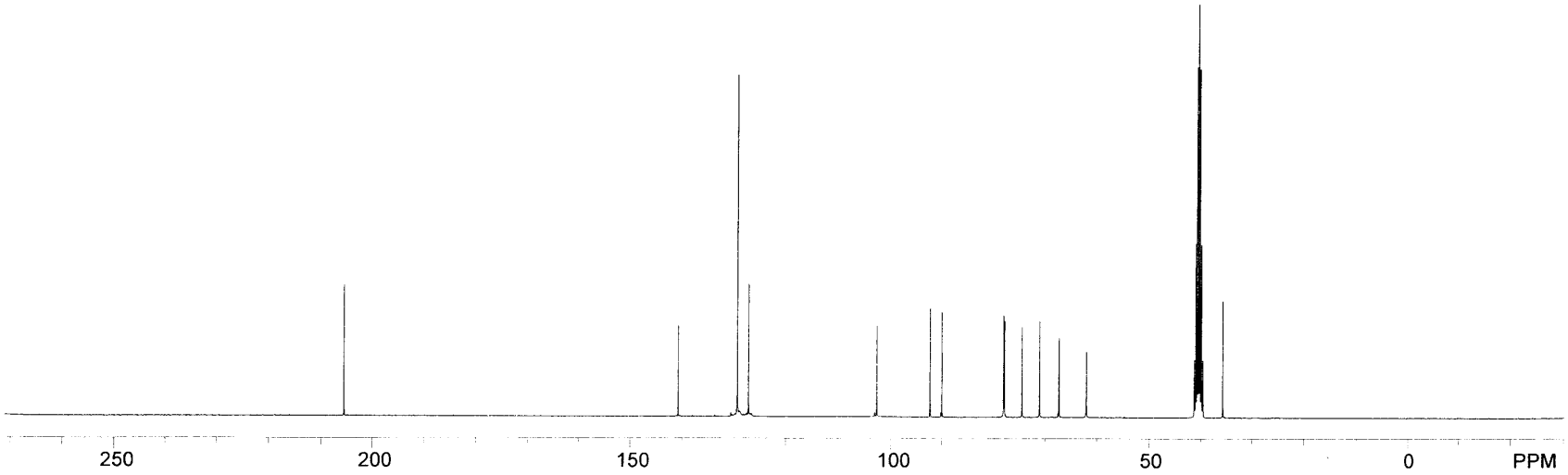


hx-12-190-DMSO

2015-02-07 09:01:33.609
USER: nmr
SOLVENT: DMSO
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 10533



(R_a)-**4ob**



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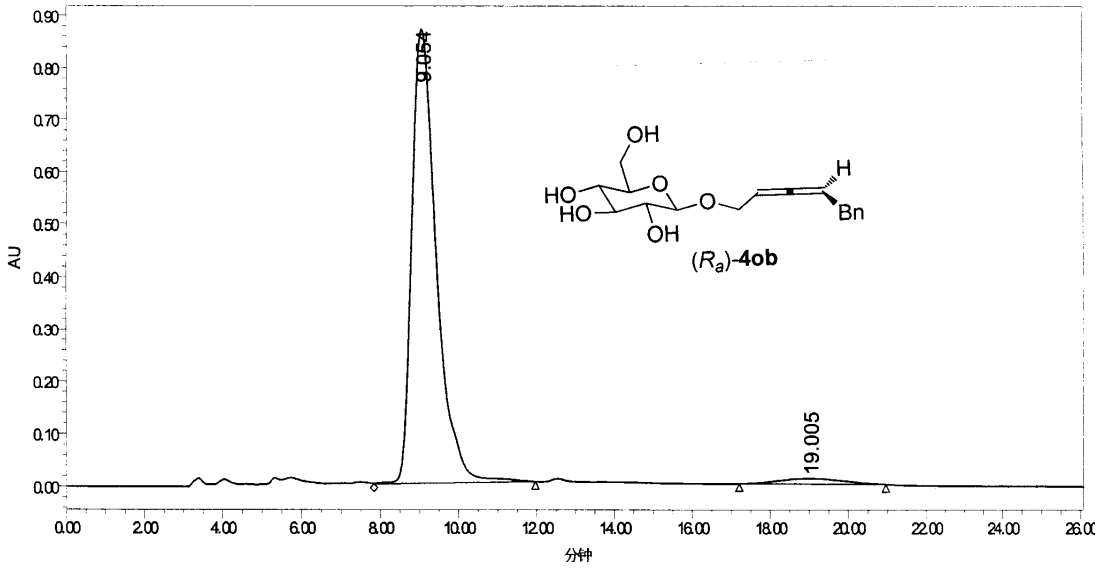
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-12-190-cj-80-20-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2015/2/9 18:16:30 CST
Vial:	1	Acq. Method:	zgj80
Injection #:	11	Date Processed:	2015/2/9 19:34:59 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	30.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (毫sec)	%Area	Height (毫	% Height
1	9.054	3775327(	96.93	867868	98.79
2	19.005	119652(	3.07	10698	1.21

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2015/2/10 10:18:03 PRC

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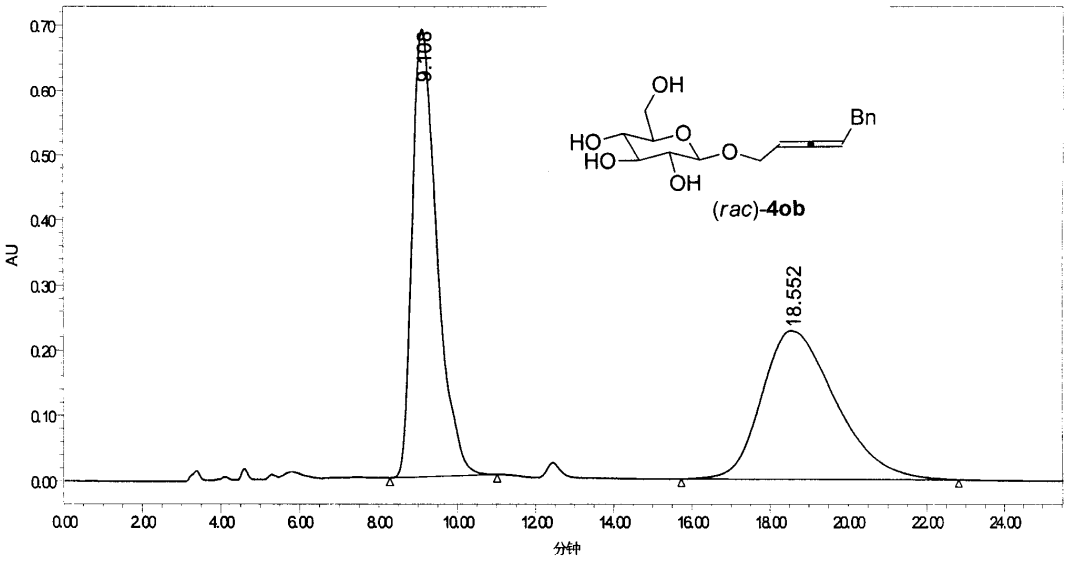
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-12-200-cj-80-20-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2015/2/9 17:49:47 CST
Vial:	1	Acq. Method:	zcj80
Injection #:	10	Date Processed:	2015/2/9 18:18:10 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	

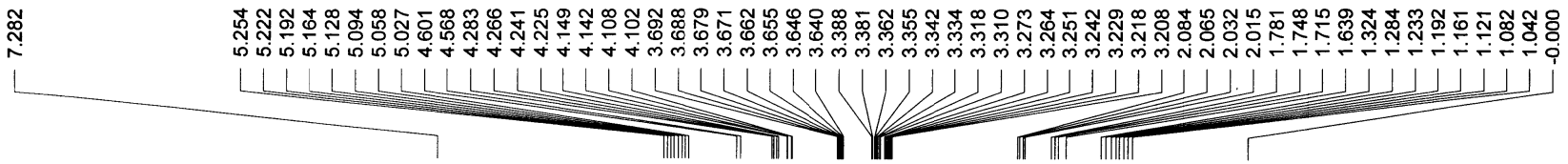


	RT (min)	Area (μV*sec)	%Area	Height (μV)	% Height
1	9.108	30007091	50.31	686946	75.25
2	18.552	29643113	49.69	225891	24.75

Report Method: Individual Report ASC

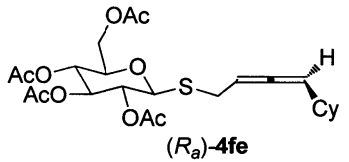
Page: 1 (共计 1)

Printed: 2015/2/9 18:36:34 PRC

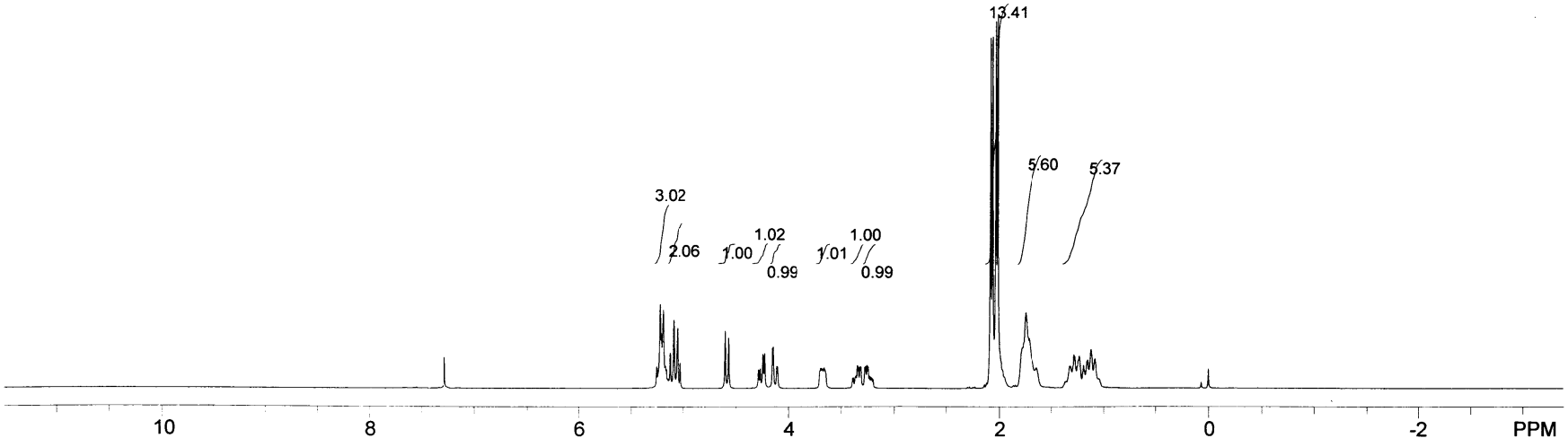


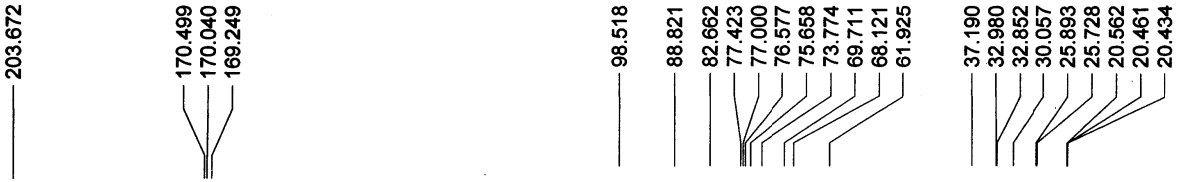
hx-10-55

2014-01-07 10:50:34.515
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



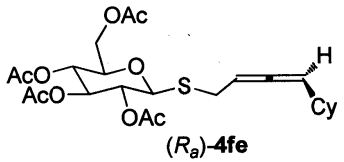
(R_a)-**4fe**



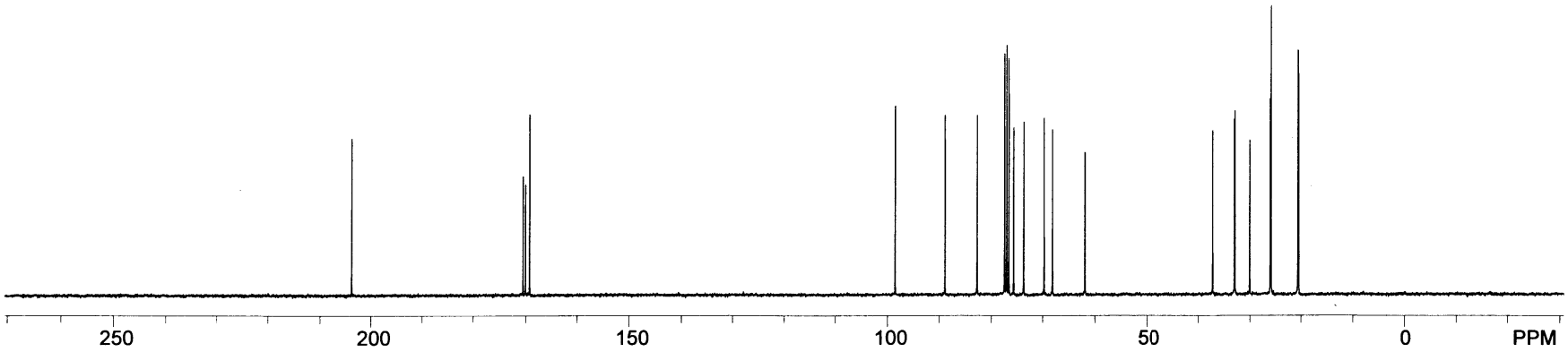


hx-10-55

2014-01-08 11:22:19.546
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 563



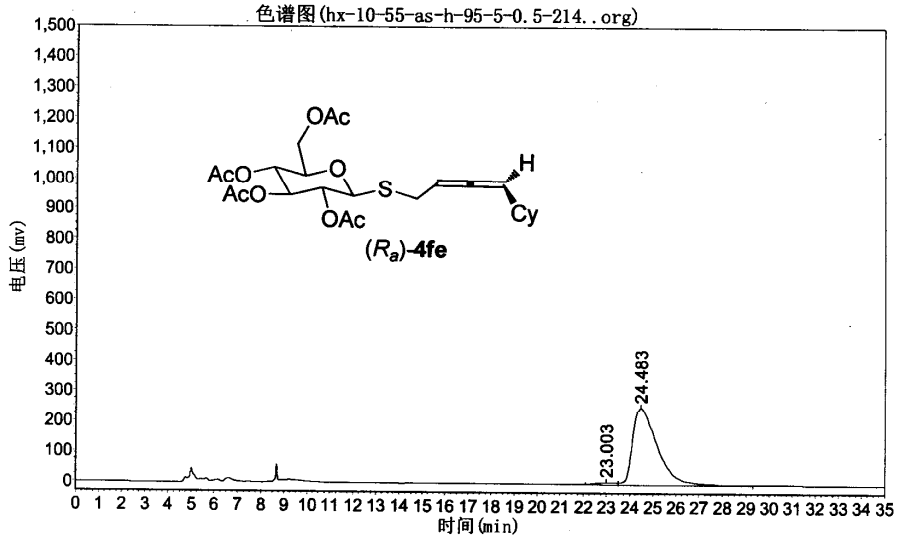
(R_a)-**4fe**



hx-10-55-as-h-95-5-0.5-214

实验时间：2014-01-09,16:51:33　　报告时间：2014-01-10,12:01:37
谱图文件:D:\zhuguangjiong\hx\20140107\hx-10-55-as-h-95-5-0.5-214..org

实验内容简介：



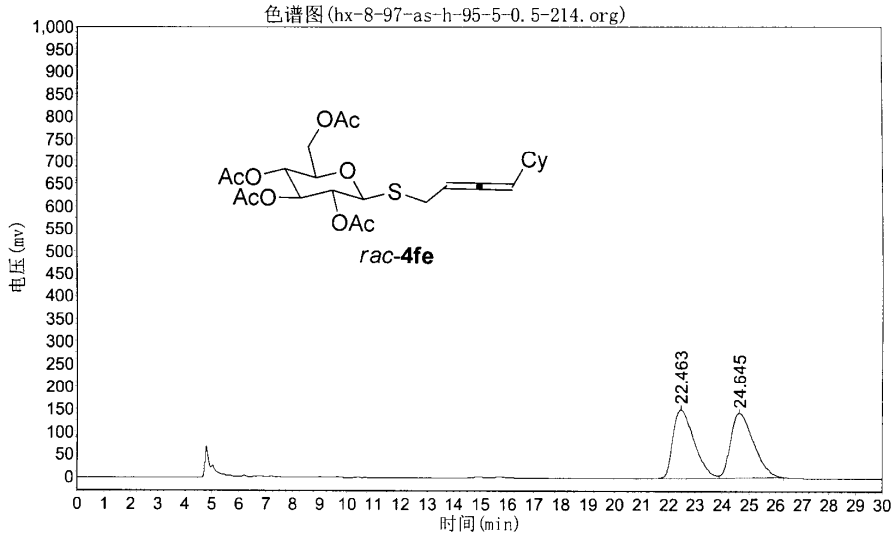
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		23.003	6496.188	385279.594	1.9777
2		24.483	253253.203	19095954.000	98.0223
总计			259749.391	19481233.594	100.0000

hx-8-97-as-h-95-5-0.5-214

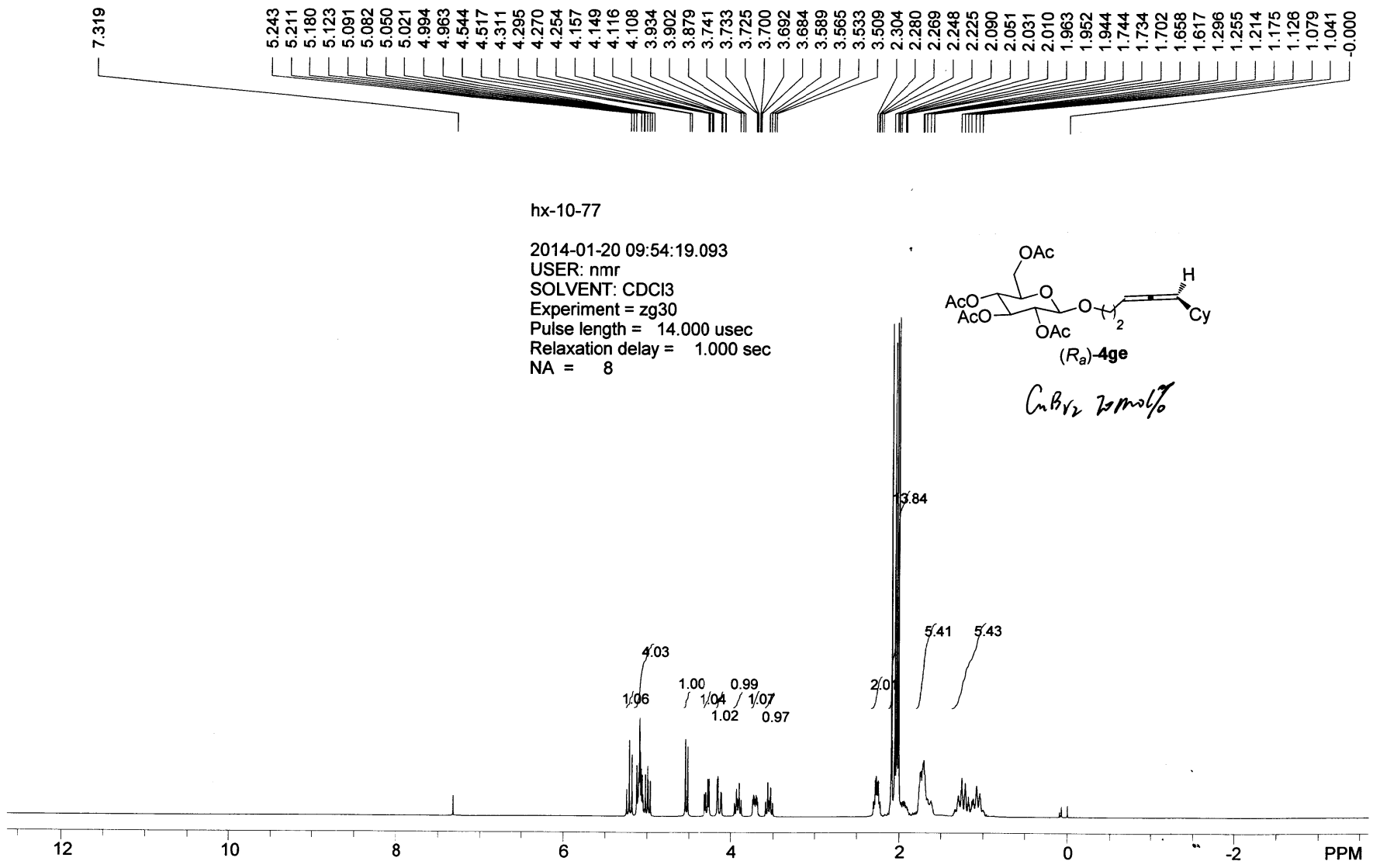
实验时间：2014-01-09,15:47:28　　报告时间：2014-01-10,12:02:41
谱图文件:D:\zhuguangjiong\hx\20140107\hx-8-97-as-h-95-5-0.5-214.org

实验内容简介：



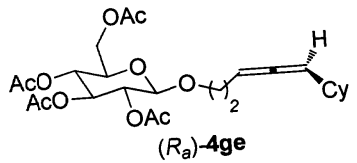
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		22.463	150183.109	8207924.500	49.4230
2		24.645	140891.609	8399574.000	50.5770
总计			291074.719	16607498.500	100.0000


7.319
5.243
5.211
5.180
5.123
5.091
5.082
5.050
5.021
4.994
4.963
4.544
4.517
4.311
4.295
4.270
4.254
4.157
4.149
4.116
4.108
3.934
3.902
3.879
3.741
3.733
3.725
3.700
3.692
3.684
3.589
3.565
3.533
3.509
2.304
2.280
2.269
2.248
2.225
2.090
2.051
2.031
2.010
1.963
1.952
1.944
1.744
1.734
1.702
1.658
1.617
1.296
1.255
1.214
1.175
1.126
1.079
1.041
-0.000
hx-10-77
2014-01-20 09:54:19.093
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
AcO
OAc
O
O
2
H
Cy
(Ra)-4ge
CuBr2 2mol%
3.84
5.41
5.43
4.03
1.00
0.99
1.06
1.04
1.07
1.02
0.97
2.01
12
10
8
6
4
2
0
-2
PPM

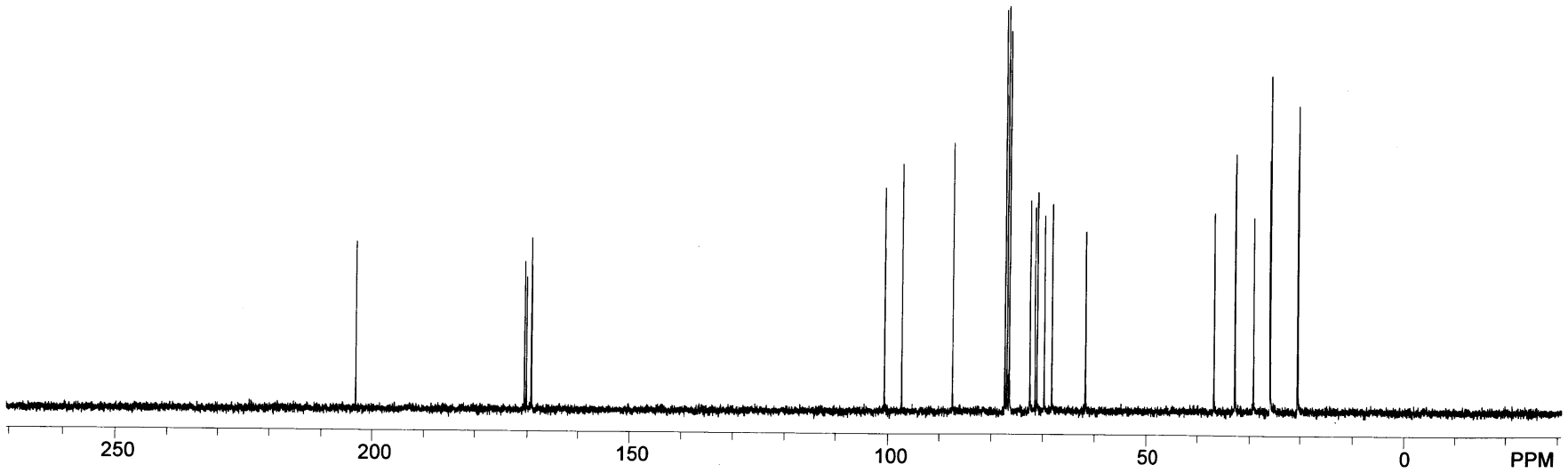
hx-10-77

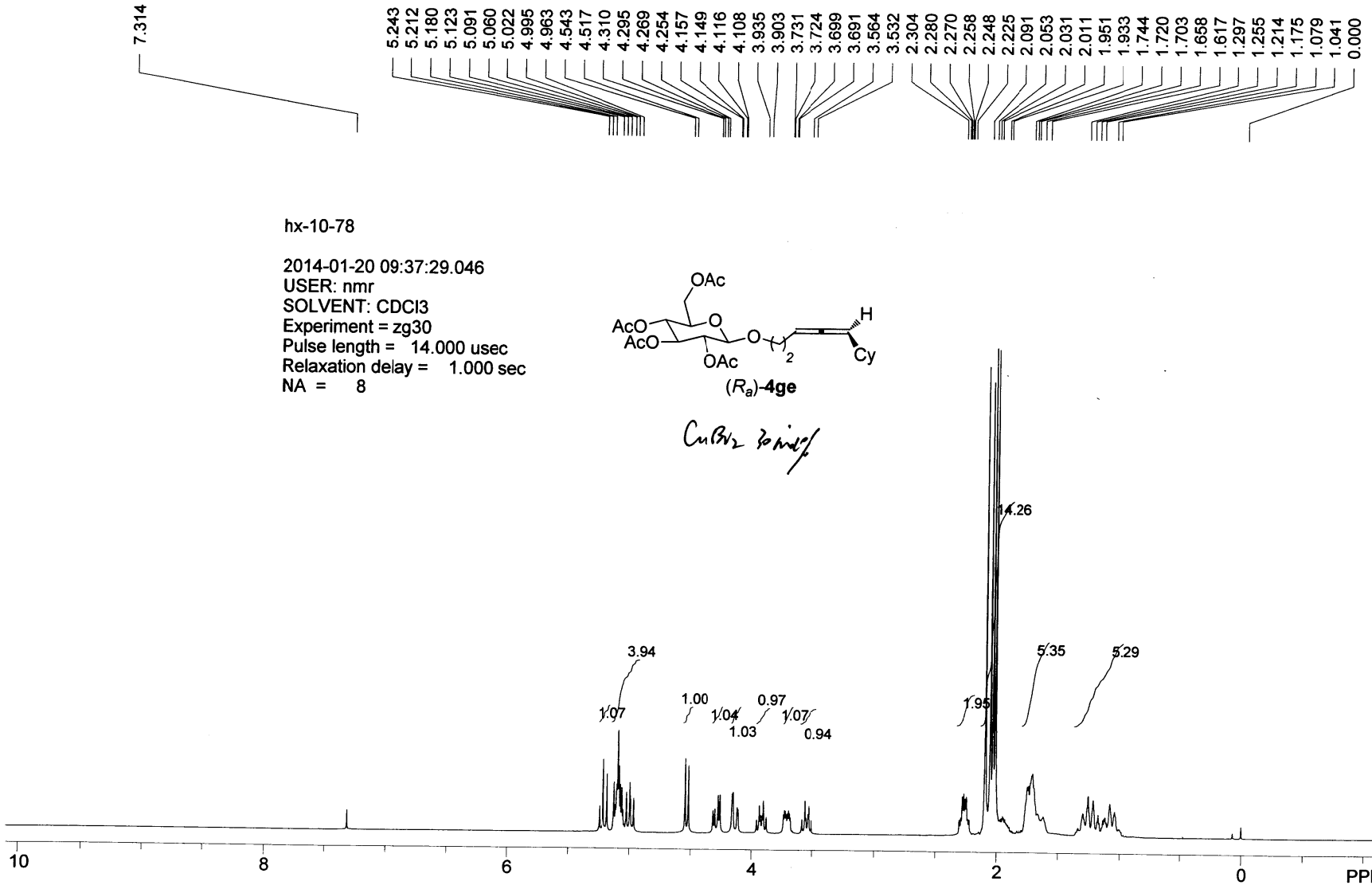
2014-01-20 10:05:37.546
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 170



(R_a)-**4ge**

$CuBr_2$ 20 mol%



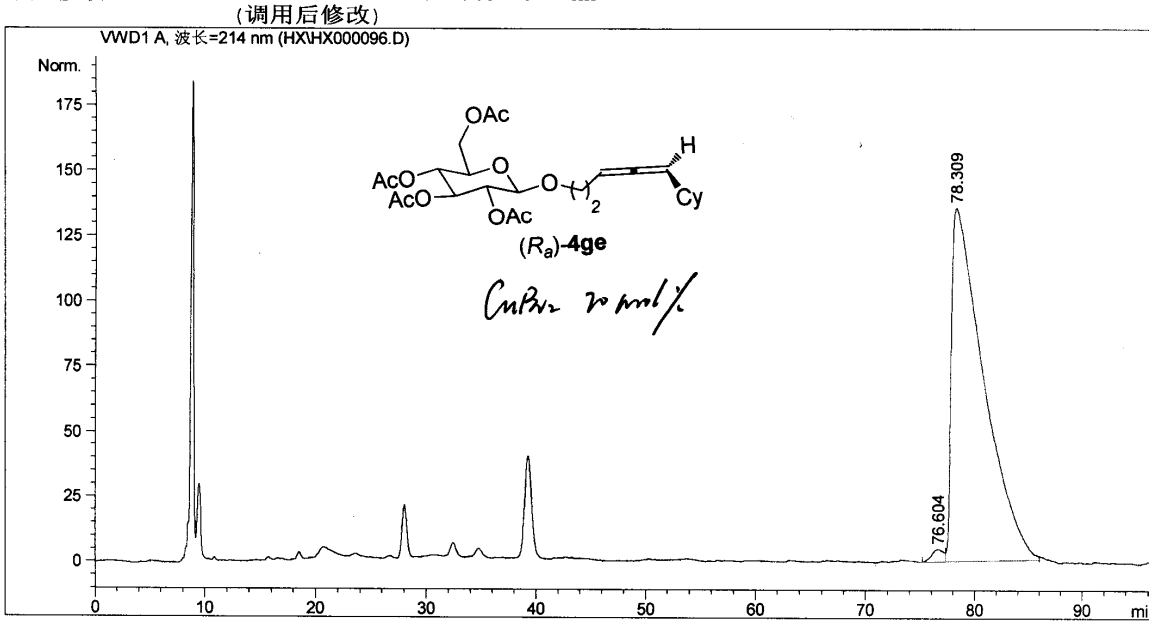

7.314
5.243
5.212
5.180
5.123
5.091
5.060
5.022
4.995
4.963
4.543
4.517
4.310
4.295
4.269
4.254
4.157
4.149
4.116
4.108
3.935
3.903
3.731
3.724
3.699
3.691
3.564
3.532
2.304
2.280
2.270
2.258
2.248
2.225
2.091
2.053
2.031
2.011
1.951
1.933
1.744
1.720
1.703
1.658
1.617
1.297
1.255
1.214
1.175
1.079
1.041
0.000
hx-10-78
2014-01-20 09:37:29.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
OAc
AcO
AcO
OAc
O
O
2
H
Cy
(Ra)-4ge
CuBr2 30 mol%
14.26
3.94
1.07
1.00
1.04
0.97
1.03
1.07
0.94
1.95
5.35
5.29
10
8
6
4
2
0
PPM

数据文件　　D:\CHEM32\1\DATA\HX\HX000096.D
样品名: hx-10-77

IC, n-hexane/i-PrOH =96/4, 0.4ml/min; 214nm

==========

进样日期	: 2004-1-1 11:34:19	
样品名称	: hx-10-77	位置 : -
操作者	: hx	
仪器	: 仪器 1	
采集方法	: D:\CHEM32\1\METHODS\ZYY_LC.M	
最后修改	: 2004-1-1 9:45:34 : hx	
	(调用后修改)	
分析方法	: D:\CHEM32\1\METHODS\ZYY_LC.M	
最后修改	: 2004-1-1 14:42:48 : hx	
	(调用后修改)	



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面积百分比报告

==========

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子

信号 1: VWD1 A, 波长=214 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	76.604	BV	0.9009	346.61212	4.79888	1.2314
2	78.309	VB	2.6037	2.78005e4	135.47583	98.7686

总量 : 2.81471e4 140.27471

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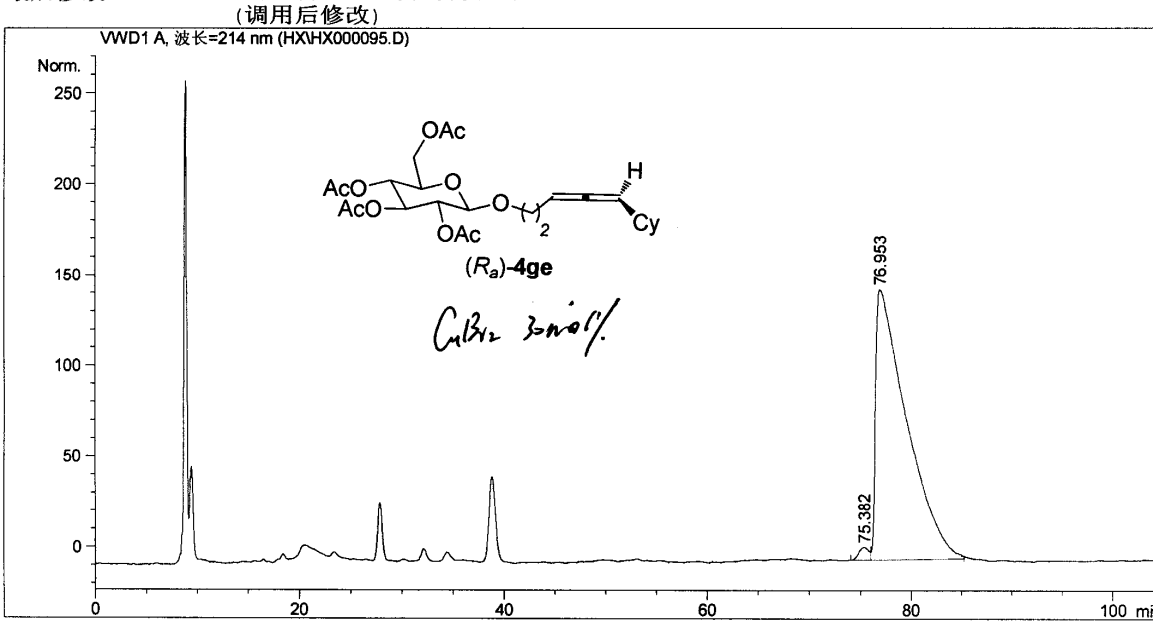
*** 报告结束 ***

数据文件　　D:\CHEM32\1\DATA\HX\HX000095.D
样品名: hx-10-78

IC, n-hexane/i-PrOH =96/4, 0.4ml/min; 214nm

==========

进样日期　　: 2004-1-1 9:46:49
样品名称　　: hx-10-78　　位置　: -
操作者　　: hx
仪器　　: 仪器 1
方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2004-1-1 9:45:34　: hx
(调用后修改)



==========

面积百分比报告

==========

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

信号 1: VWD1 A, 波长=214 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	75.382	BV	0.8557	478.08835	7.04543	1.4899
2	76.953	VB	2.7431	3.16103e4	148.29825	98.5101

总量 :　　3.20884e4　　155.34368

==========

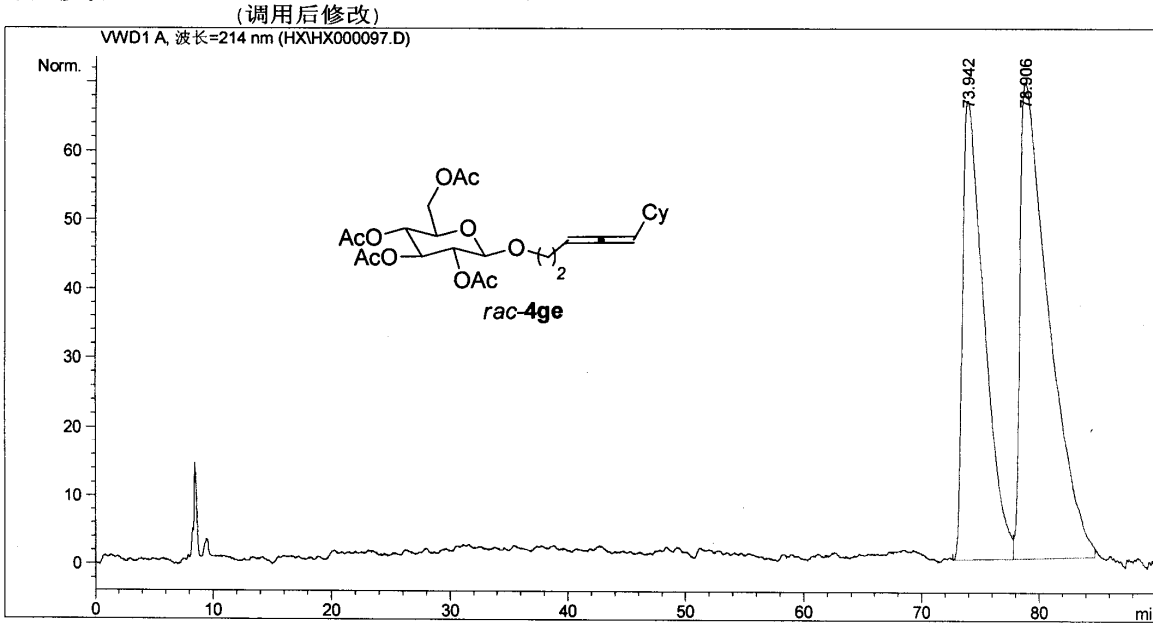
*** 报告结束 ***

数据文件　　D:\Chem32\1\DATA\HX\HX000097.D
样品名: hx-8-80

IC, n-hexane/i-PrOH =96/4, 0.4ml/min; 214nm

==========

进样日期　　: 2004-1-1 13:11:59
样品名称　　: hx-8-80　　位置　: -
操作者　　: hx
仪器　　: 仪器 1
采集方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2004-1-1 14:38:08　: hx
(调用后修改)
分析方法　　: D:\CHEM32\1\METHODS\ZYY_LC.M
最后修改　　: 2004-1-1 14:42:48　: hx
(调用后修改)



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面积百分比报告

==========

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

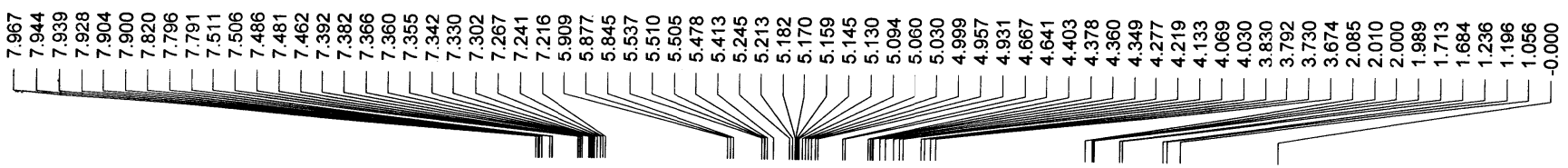
信号 1: VWD1 A, 波长=214 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	73.942	BV	1.6905	8520.73438	66.34748	41.8911
2	78.906	VB	2.2042	1.18195e4	68.93280	58.1089

总量 :　　2.03402e4　　135.28028

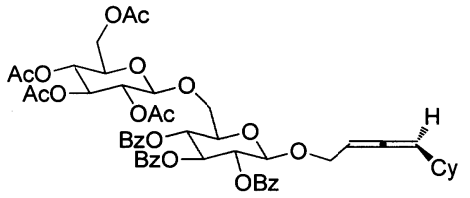
==========

*** 报告结束 ***

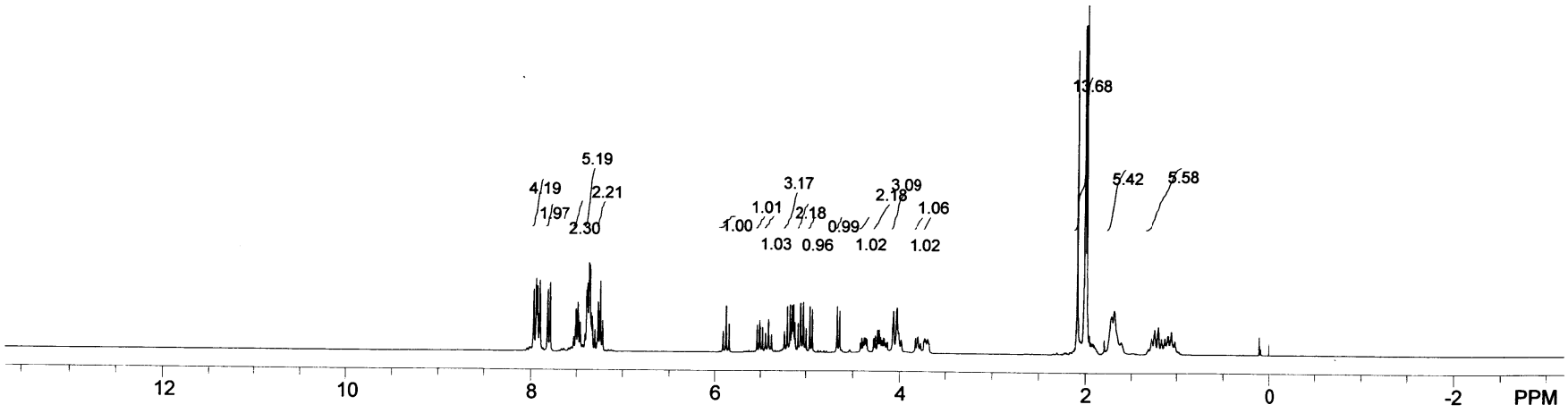


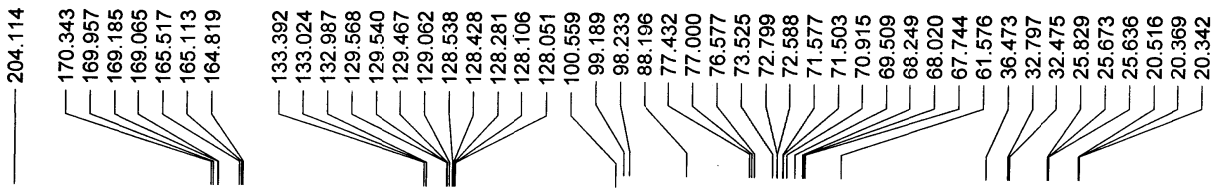
hx-10-58

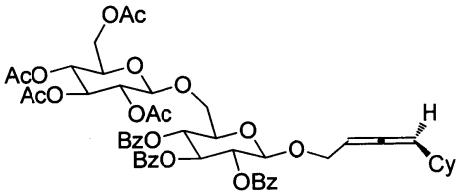
2014-01-11 14:53:12.937
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4he**



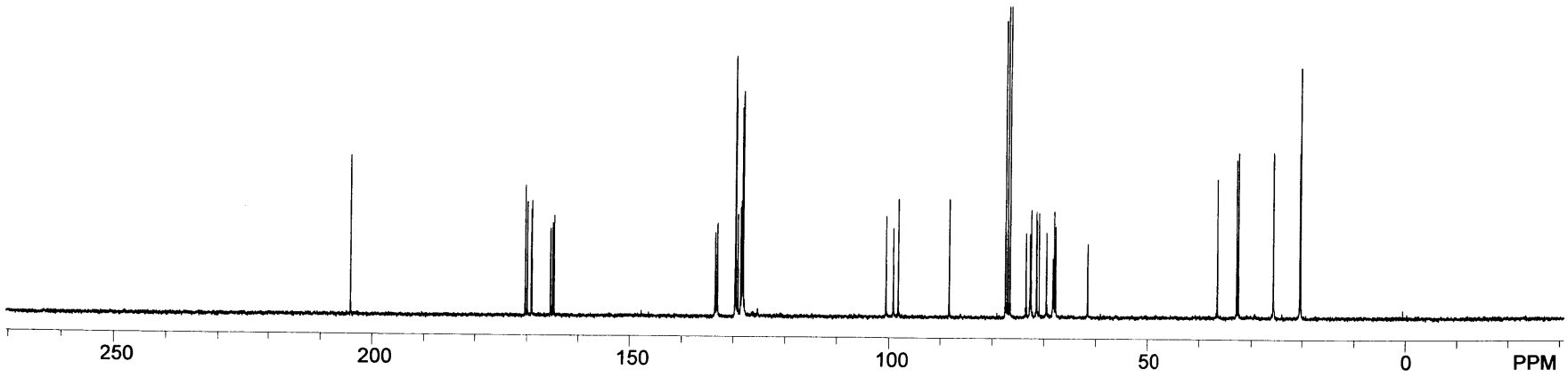




(R_a)-**4he**

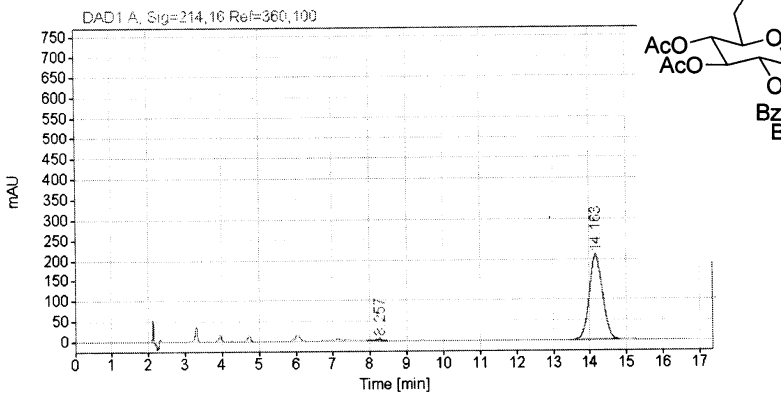
hx-10-58

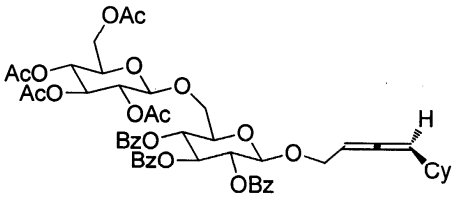
2014-01-11 15:30:44.687
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 543



Agilent Technologies

Data file:	C:\CHEM32\1\DATA\2014-4621.D		
Sample name:	hx-10-58		
Instrument:	SFC	Sample type:	Sample
Injection date:	5/5/2014 3:29:06 PM	Injection:	1 of 1





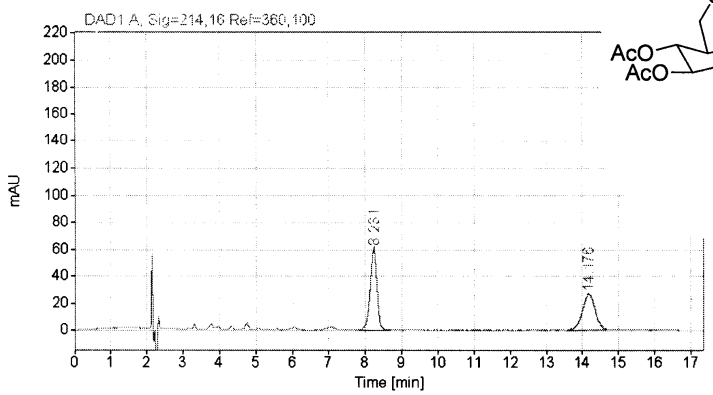
(R_a)-**4he**

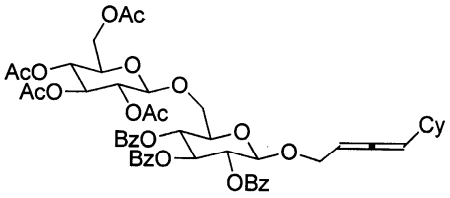
Signal: DAD1 A, Sig=214,16 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%
8.257	VB	0.1838	48.2732	3.2780	0.9177
14.163	BB	0.3806	5212.0527	207.8039	99.0823
		Sum	5260.3259		

Agilent Technologies

Data file:	C:\CHEM32\1\DATA\2014-4622.D		
Sample name:	hx-8-82-rac-IA-8-2-1.5		
Instrument:	SFC	Sample type:	Sample
Injection date:	5/5/2014 3:58:12 PM	Injection:	1 of 1

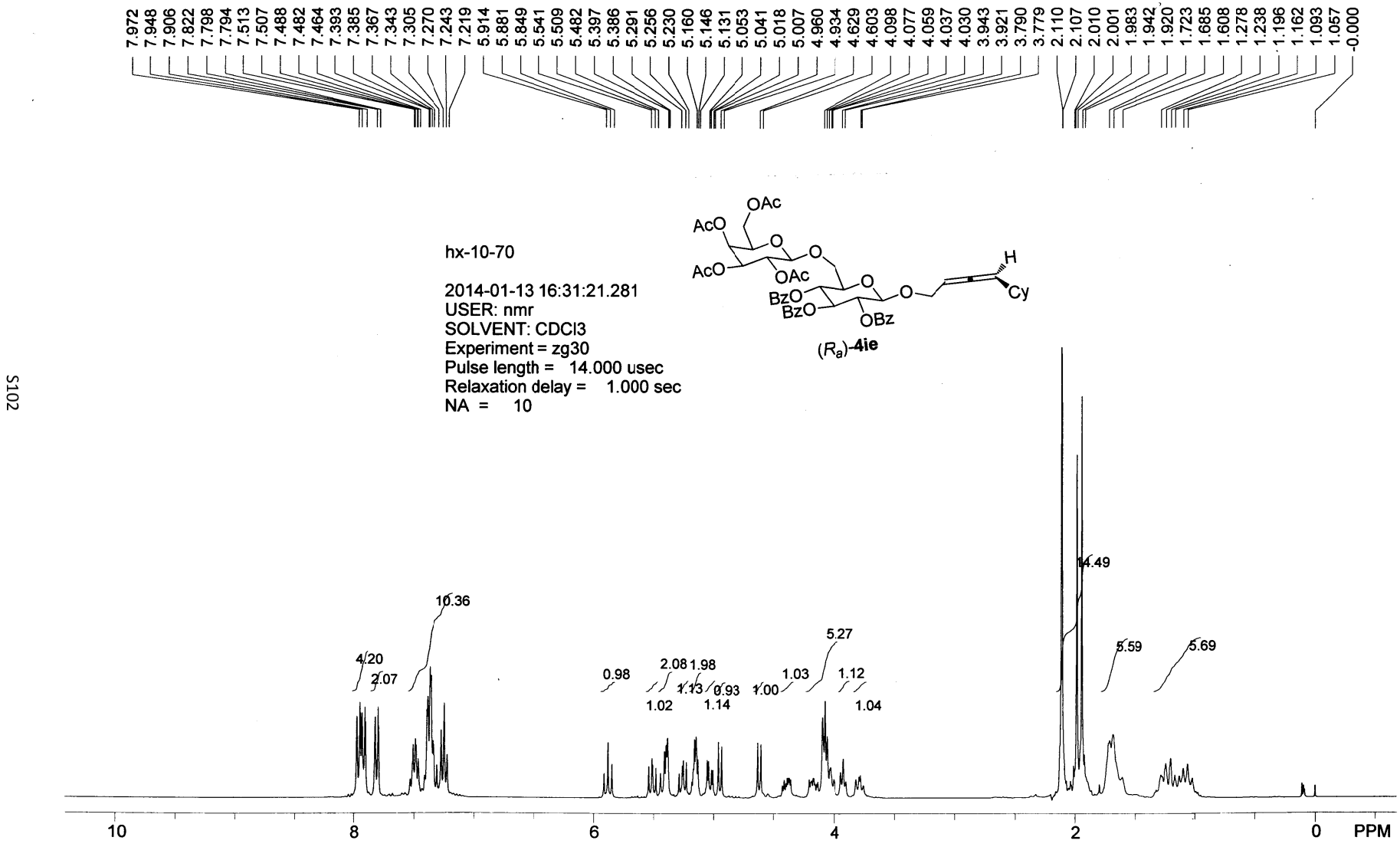


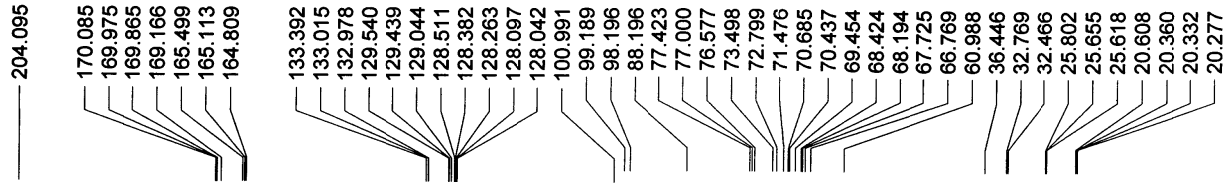


rac-**4he**

Signal: DAD1 A, Sig=214,16 Ref=360,100

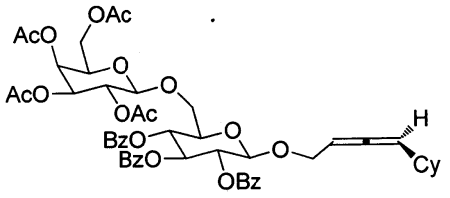
RT [min]	Type	Width [min]	Area	Height	Area%
8.231	BB	0.1976	763.8271	59.9527	54.4052
14.176	VV	0.2991	640.1336	26.1039	45.5948
		Sum	1403.9607		


7.972 7.948 7.906 7.822 7.798 7.794 7.513 7.507 7.488 7.482 7.464 7.393 7.385 7.367 7.343 7.305 7.270 7.243 7.219 5.914 5.881 5.849 5.541 5.509 5.482 5.397 5.386 5.291 5.256 5.230 5.160 5.146 5.131 5.053 5.041 5.018 5.007 4.960 4.934 4.629 4.603 4.098 4.077 4.059 4.037 4.030 3.943 3.921 3.790 3.779 2.110 2.107 2.010 2.001 1.983 1.942 1.920 1.723 1.685 1.608 1.278 1.238 1.196 1.162 1.093 1.057 -0.000
hx-10-70
2014-01-13 16:31:21.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 10
OAc
AcO
AcO
OAc
BzO
BzO
OBz
H
Cy
(R_a)-4ie
10.36
4.20
2.07
0.98
2.08
1.98
1.02
1.13
0.93
1.14
1.00
1.03
5.27
1.12
1.04
14.49
5.59
5.69
10
8
6
4
2
0
PPM

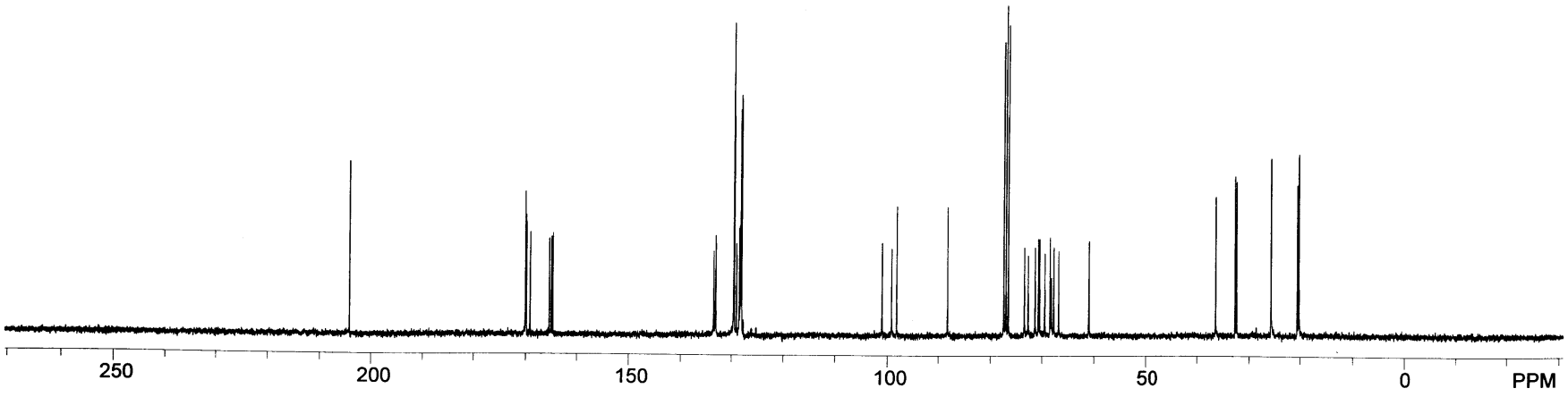


hx-10-70

2014-01-13 19:23:34.203
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 240

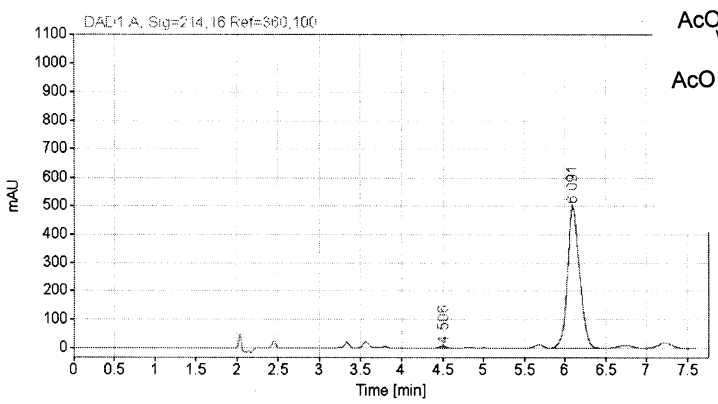


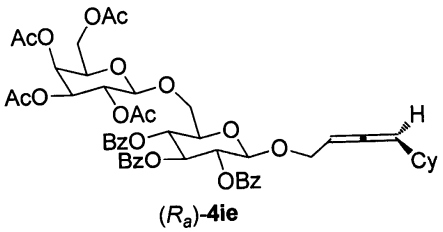
(R_a)-**4ie**



Agilent Technologies

Data file:	C:\CHEM32\1\DATA\2014-4609.D		
Sample name:	HX-10-70		
Instrument:	SFC	Sample type:	Sample
Injection date:	4/29/2014 4:11:15 PM	Injection:	1 of 1





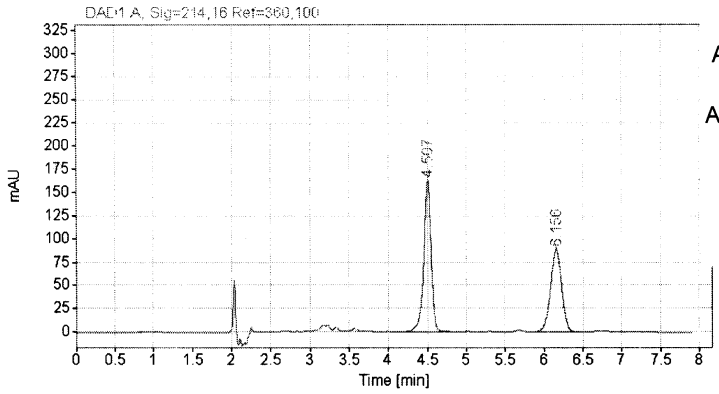
(R_a)-**4ie**

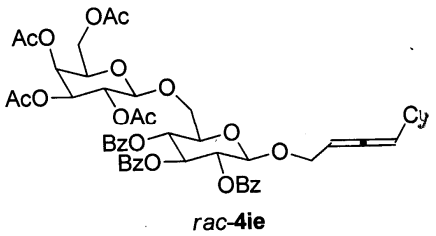
Signal: DAD1 A, Sig=214,16 Ref=360,100

RT [min]	Type	Width [min]	Area	Height	Area%
4.506	BB	0.1045	51.9958	7.2691	0.9978
6.091	VV	0.1572	5158.8315	496.7322	99.0022
		Sum	5210.8274		

Agilent Technologies

Data file:	C:\CHEM32\1\DATA\2014-4610.D		
Sample name:	HX-8-99-rac-ia-7-3-1.5-214		
Instrument:	SFC	Sample type:	Sample
Injection date:	4/29/2014 4:22:54 PM	Injection:	1 of 1

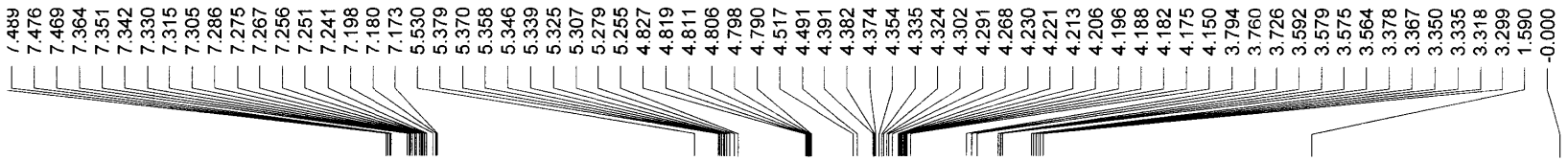




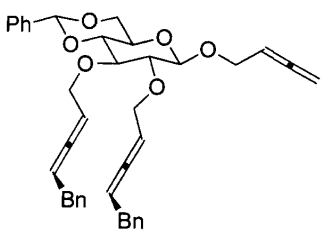
rac-**4ie**

Signal: DAD1 A, Sig=214,16 Ref=360,100

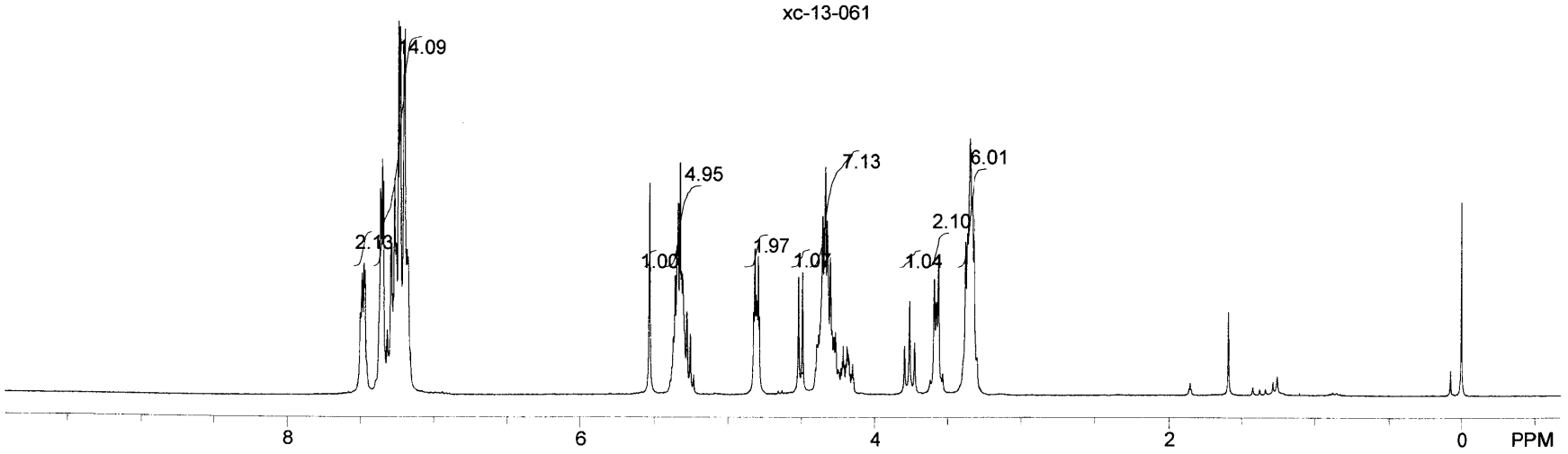
RT [min]	Type	Width [min]	Area	Height	Area%
4.507	VB	0.1003	1076.8519	162.5107	54.8004
6.156	BV	0.1525	888.1928	88.9660	45.1996
		Sum	1965.0447		

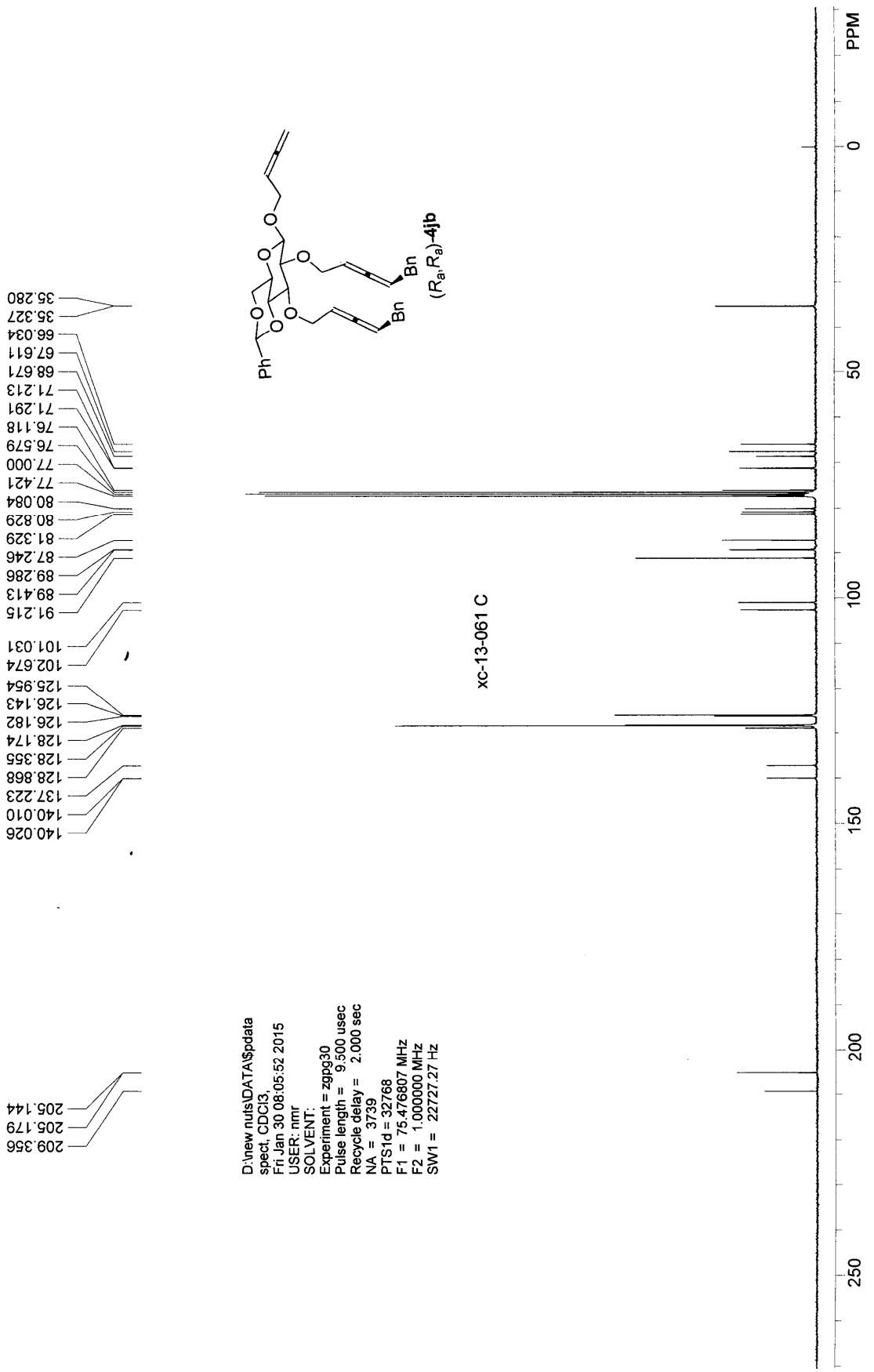


D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 30 08:03:11 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



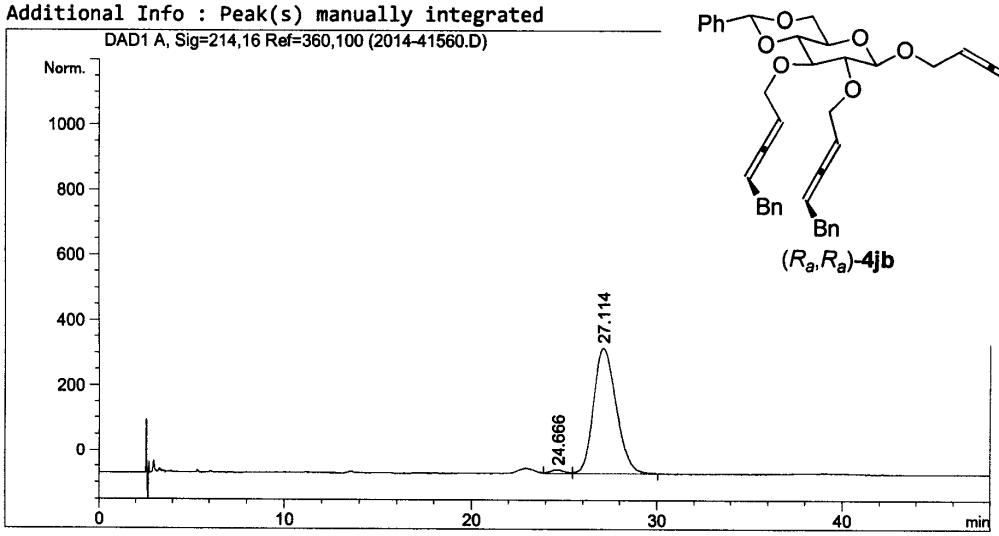
(R_a,R_a)-**4jb**




35.280
35.327
66.034
67.611
68.671
71.213
71.291
76.118
76.579
77.000
77.421
80.084
80.829
81.329
87.246
89.286
89.413
91.215
101.031
102.674
125.954
126.143
126.182
128.174
128.355
128.868
137.223
140.010
140.026
205.144
205.179
209.356
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 30 08:05:52 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 3739
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
xc-13-061 C
Ph
Bn
Bn
(Ra,Ra)-4jb
PPM
0
50
100
150
200
250

Data File C:\CHEM32\1\DATA\2014-41560.D
Sample Name: xc-13-061

```
==========================================================================
Acq. Operator   : 系统
Sample Operator : 系统
Acq. Instrument : SFC                              Location : Vial 32
Injection Date  : 2/10/2015 2:57:56 PM
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\AGILENT_SFC6.M
Last changed    : 2/10/2015 2:43:51 PM by 系统
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AGILENT_SFC6.M
Last changed    : 2/11/2015 9:56:08 AM by 系统
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

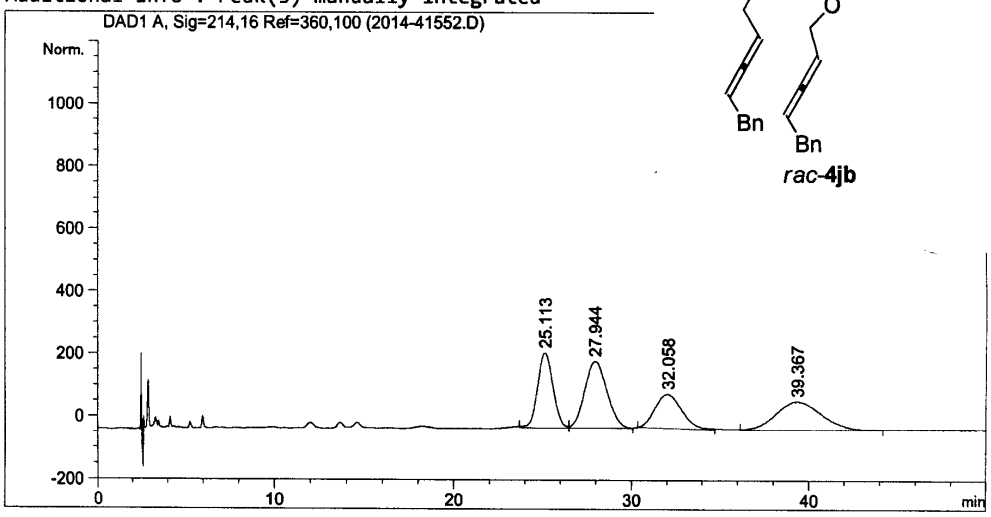


(R_a,R_a)-**4jb**

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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=214,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.666 MF R  0.8034  268.68335    5.34289   1.5081
   2  27.114 FM R  1.4091 1.75478e4   205.48734  98.4919

Totals :                  1.78164e4   210.83022

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

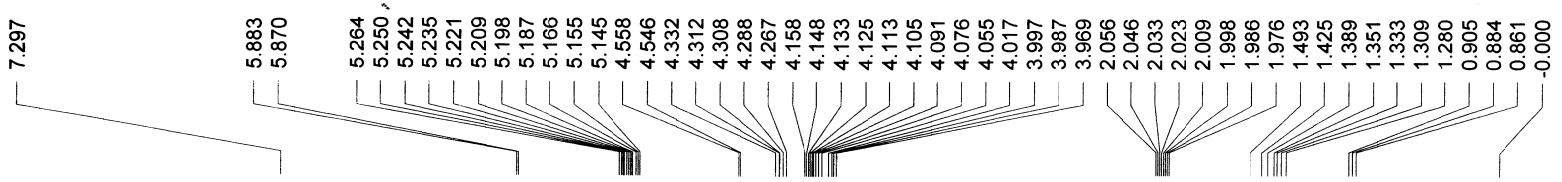
Data File C:\CHEM32\1\DATA\2014-41552.D
Sample Name: xc-13-55-1-rac-oj-8-2-1.3-214

```
==========================================================================
Acq. Operator   : 系统
Sample Operator : 系统
Acq. Instrument : SFC                              Location : Vial 31
Injection Date  : 2/9/2015 2:26:37 PM
                                                   Inj Volume : 5.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\AGILENT_SFC6.M
Last changed    : 2/9/2015 1:52:27 PM by 系统
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\AGILENT_SFC6.M
Last changed    : 2/11/2015 9:49:25 AM by 系统
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



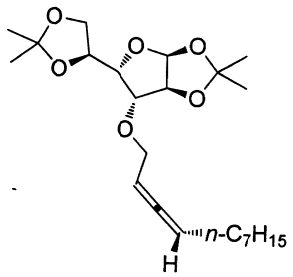
rac-**4jb**

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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=214,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.113 MF R  0.9331 4871.58350   81.16325  23.6625
   2  27.944 FM R  1.2599 6243.45752   72.01936  30.3261
   3  32.058 MM R  1.7414 3840.86035   36.76054  18.6560
   4  39.367 MM R  2.8739 5631.86719   30.75925  27.3554

Totals :                  2.05878e4   220.70240

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

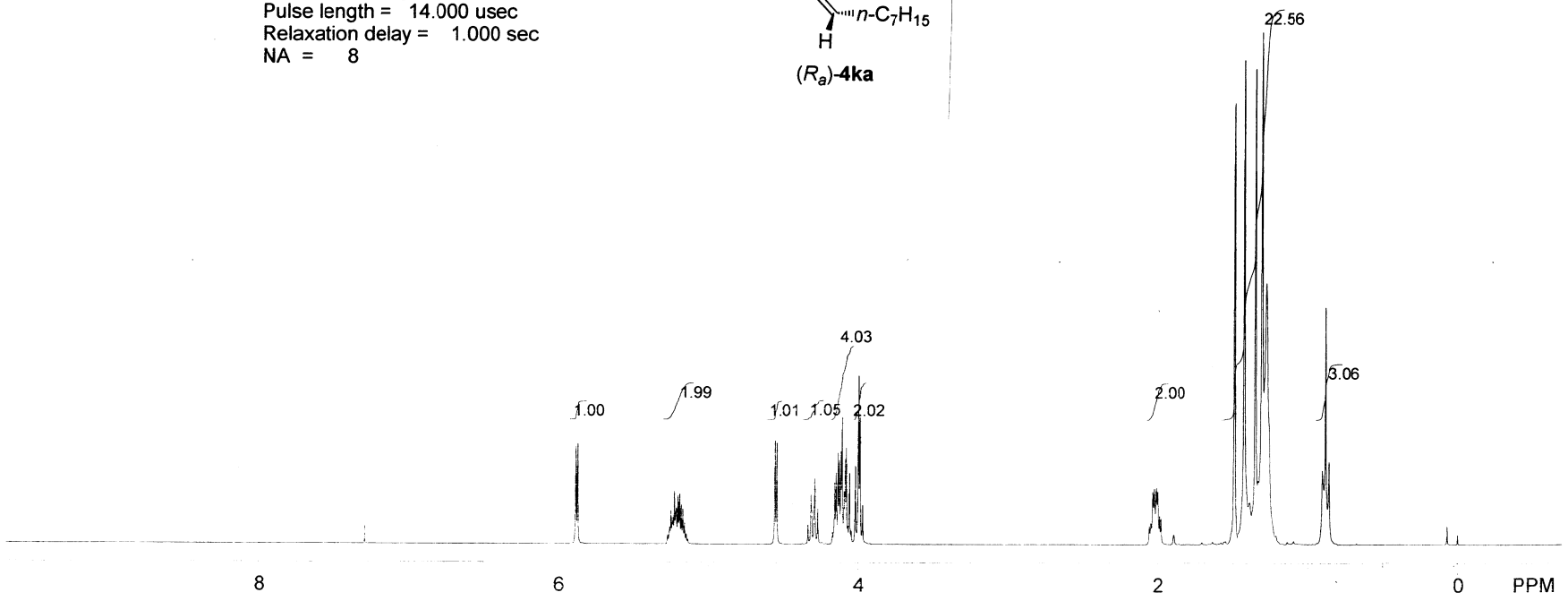


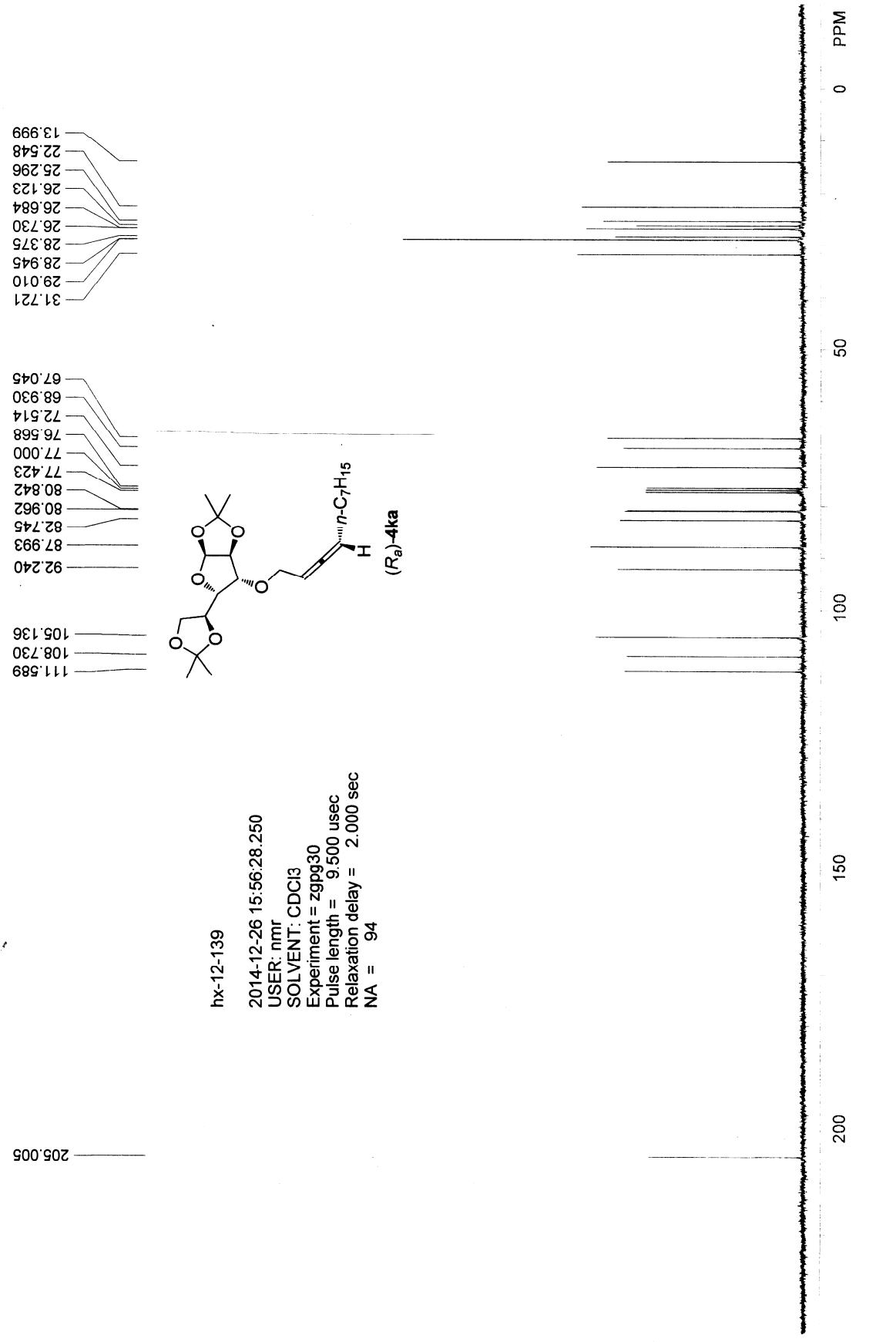
hx-12-139

2014-12-26 15:49:48.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8



(R_a)-**4ka**

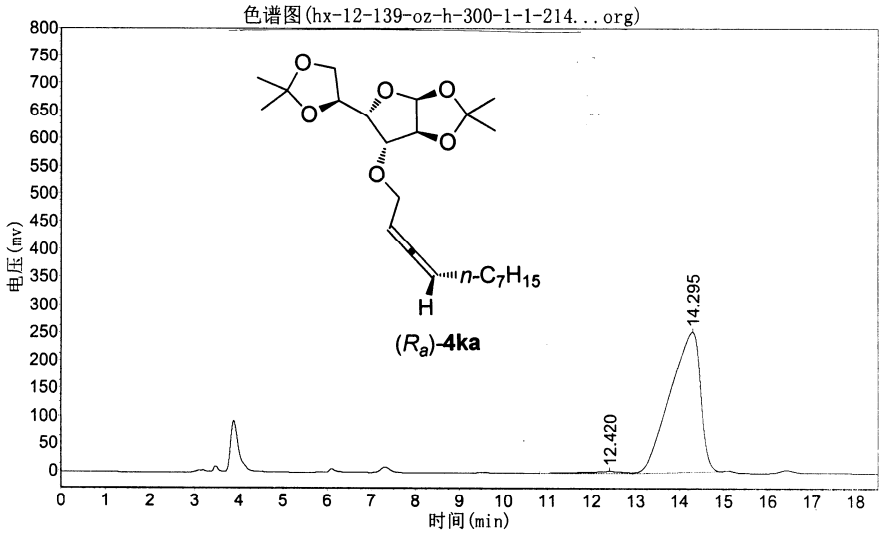



13.999
22.548
25.296
26.123
26.684
26.730
28.375
28.945
29.010
31.721
67.045
68.930
72.514
76.568
77.000
77.423
80.842
80.962
82.745
87.993
92.240
105.136
108.730
111.589
205.005
200
150
100
50
0
PPM
hx-12-139
2014-12-26 15:56:28.250
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 94
n-C7H15
H
(Ra)-4ka

hx-12-139-oz-h-300-1-1-214

实验时间：2015-01-07,12:18:20　　报告时间：2015-01-07,18:13:11
谱图文件:D:\zhuguangjiong\hx\20150107\hx-12-139-oz-h-300-1-1-214...org

实验内容简介：



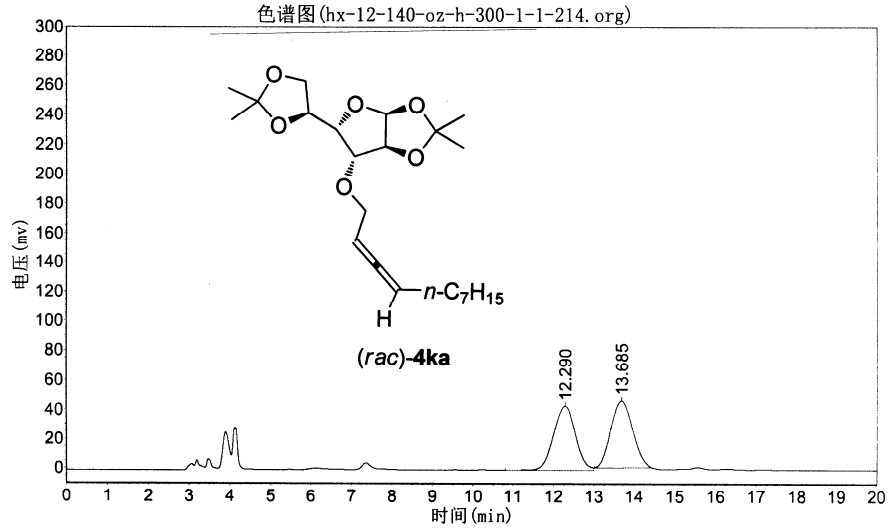
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.420	3300.135	192895.328	1.5743
2		14.295	252215.625	12059923.000	98.4257
总计			255515.760	12252818.328	100.0000

hx-12-140-oz-h-300-1-1-214

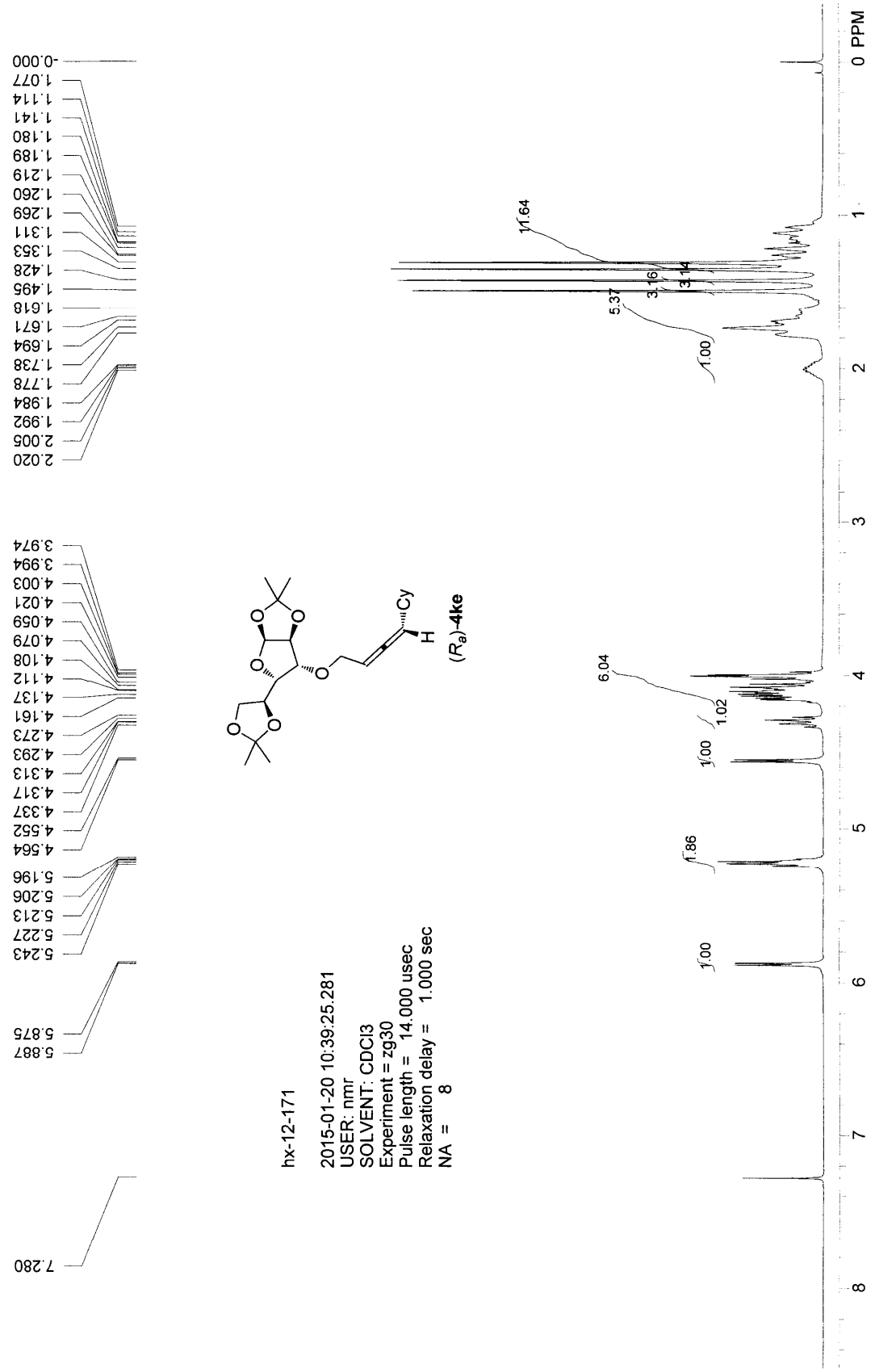
实验时间：2015-01-07,11:02:22　　报告时间：2015-01-08,10:49:50
谱图文件:d:\zhuguangjiong\hx\20150107\hx-12-140-oz-h-300-1-1-214.org

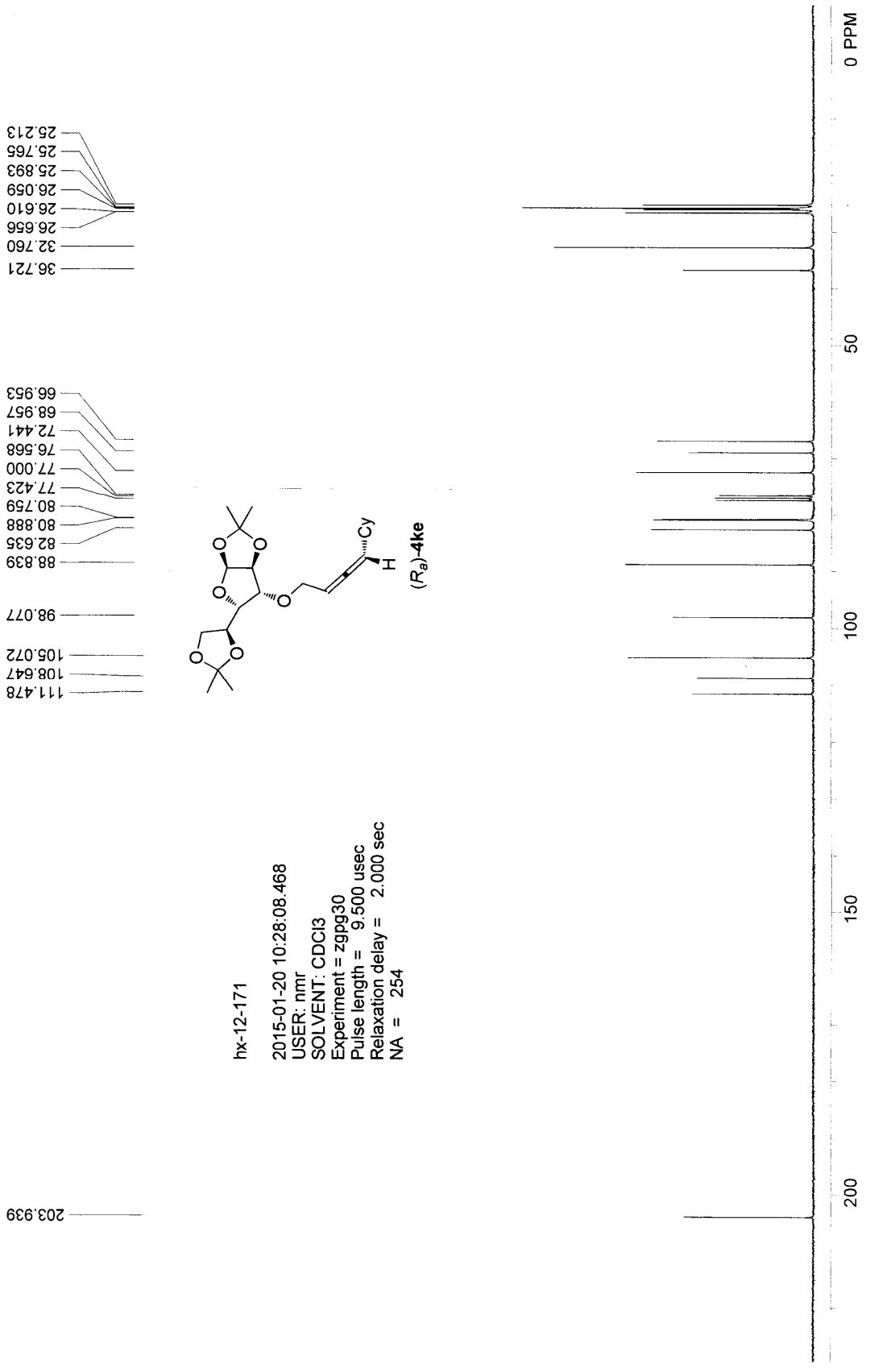
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.290	43782.520	1654257.625	49.3741
2		13.685	45445.836	1696201.375	50.6259
总计			89228.355	3350459.000	100.0000

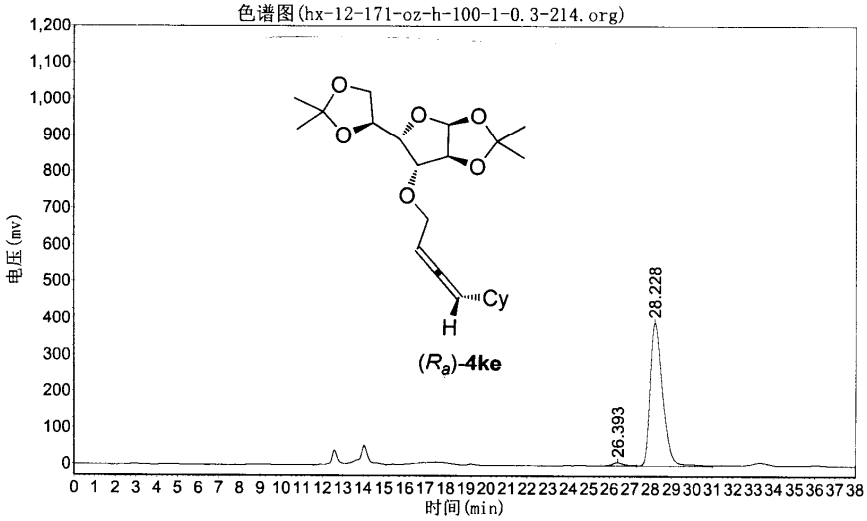

hx-12-171
2015-01-20 10:39:25.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
(Ra)-4ke
Cy
H
O
O
O
O
O
O
11.64
6.04
5.37
3.16
3.14
1.86
1.02
1.00
1.00
1.00
0 PPM
7.280
5.887
5.875
5.243
5.227
5.213
5.206
5.196
4.564
4.552
4.337
4.317
4.313
4.293
4.273
4.161
4.137
4.112
4.108
4.079
4.059
4.021
4.003
3.994
3.974
2.020
2.005
1.992
1.984
1.778
1.738
1.694
1.671
1.618
1.495
1.428
1.353
1.311
1.269
1.260
1.219
1.189
1.180
1.141
1.114
1.077
-0.000


203.939
111.478
108.647
105.072
98.077
88.839
82.635
80.888
80.759
77.423
77.000
76.568
72.441
68.957
66.953
36.721
32.760
26.656
26.610
26.059
25.893
25.765
25.213
0 PPM
50
100
150
200
hx-12-171
2015-01-20 10:28:08.468
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 254
Cy
H
(Ra)-4ke

hx-12-171-oz-h-100-1-0.3-214

实验时间：2015-01-21,17:12:51　　报告时间：2015-01-21,17:52:07
谱图文件:D:\zhuguangjiong\hx\20150121\hx-12-171-oz-h-100-1-0.3-214.org

实验内容简介：



分析结果表

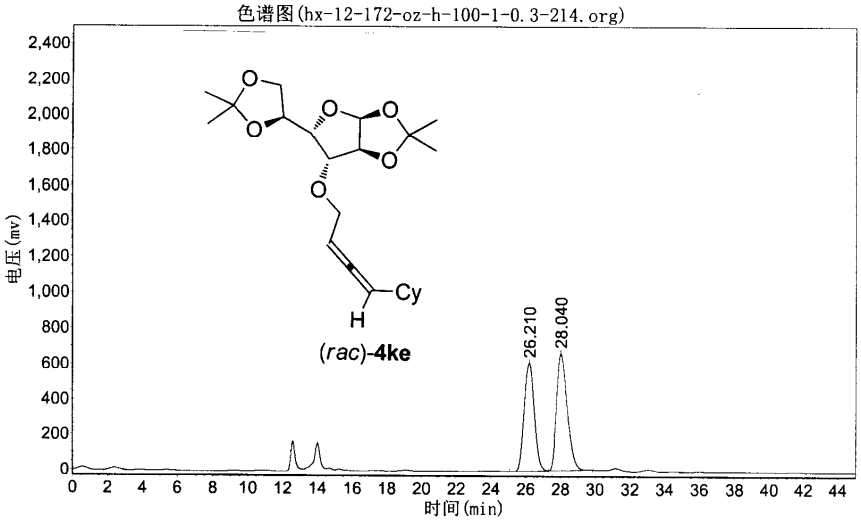
峰号	峰名	保留时间	峰高	峰面积	含量
1		26.393	8439.984	335459.781	1.9956
2		28.228	391919.188	16474806.000	98.0044
总计			400359.172	16810265.781	100.0000

hx-12-172-oz-h-100-1-0.3-214

实验时间：2015-01-21,15:44:52　　报告时间：2015-01-21,17:46:20
谱图文件:D:\zhuguangjiong\hx\20150121\hx-12-172-oz-h-100-1-0.3-214.org

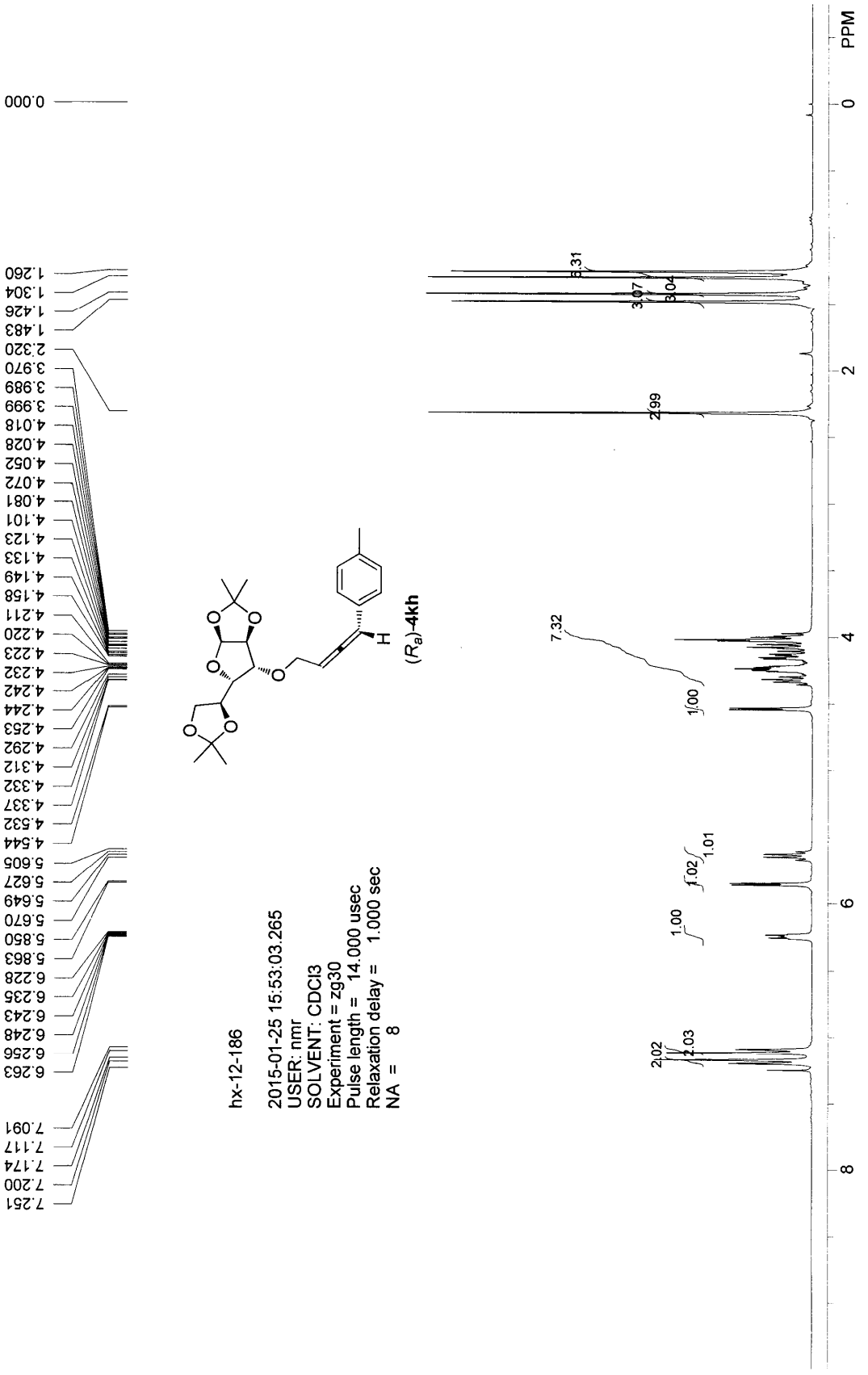
实验内容简介：

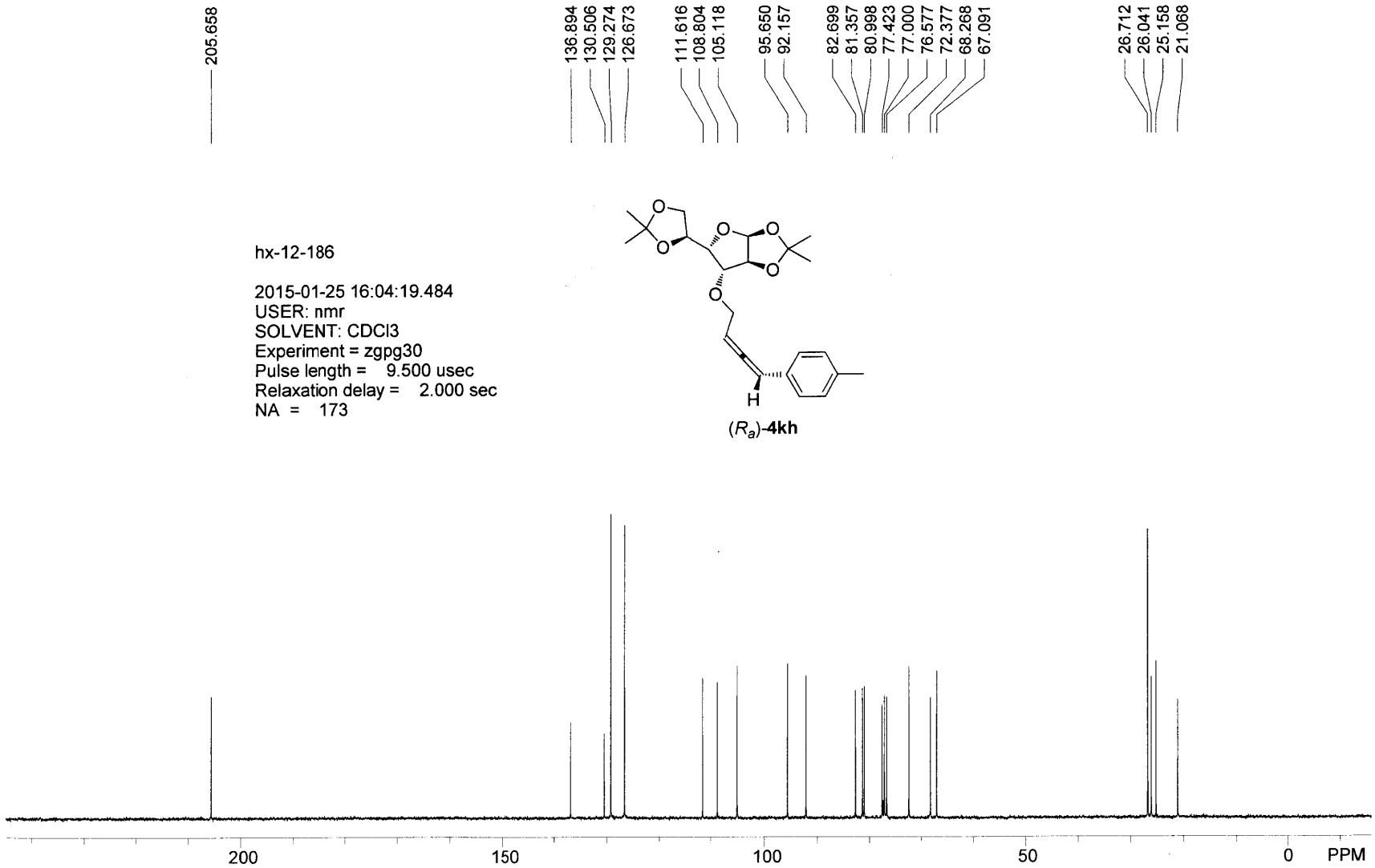
色谱图(hx-12-172-oz-h-100-1-0.3-214.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		26.210	613467.375	24778642.000	46.5889
2		28.040	659881.563	28407096.000	53.4111
总计			1273348.938	53185738.000	100.0000


hx-12-186
2015-01-25 15:53:03.265
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
(Ra)-4kh
PPM

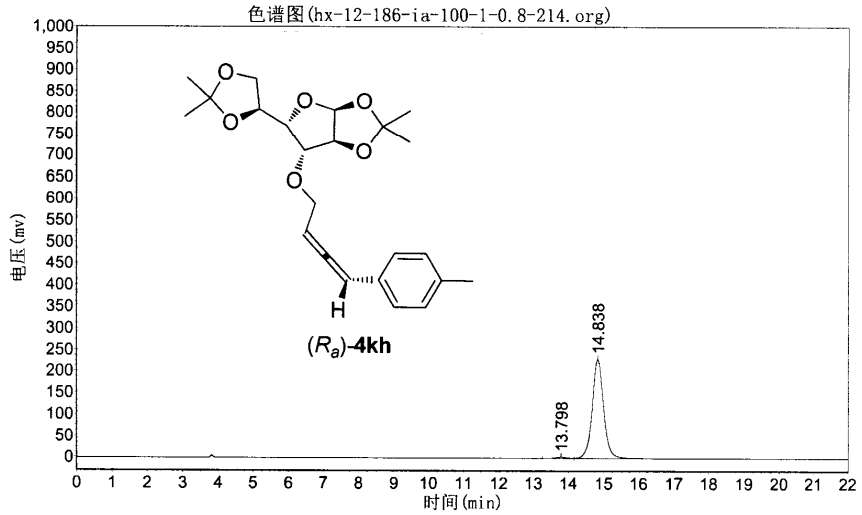

205.658
136.894
130.506
129.274
126.673
111.616
108.804
105.118
95.650
92.157
82.699
81.357
80.998
77.423
77.000
76.577
72.377
68.268
67.091
26.712
26.041
25.158
21.068
hx-12-186
2015-01-25 16:04:19.484
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 173
O
O
O
O
O
O
H
200
150
100
50
0
PPM

(R_a)-**4kh**

hx-12-186-ia-100-1-0.8-214

实验时间：2015/2/5,14:32:25　　报告时间：2015/2/5,17:05:31
谱图文件:D:\zhuguangjiong\hx\20150205\hx-12-186-ia-100-1-0.8-214.org

实验内容简介：



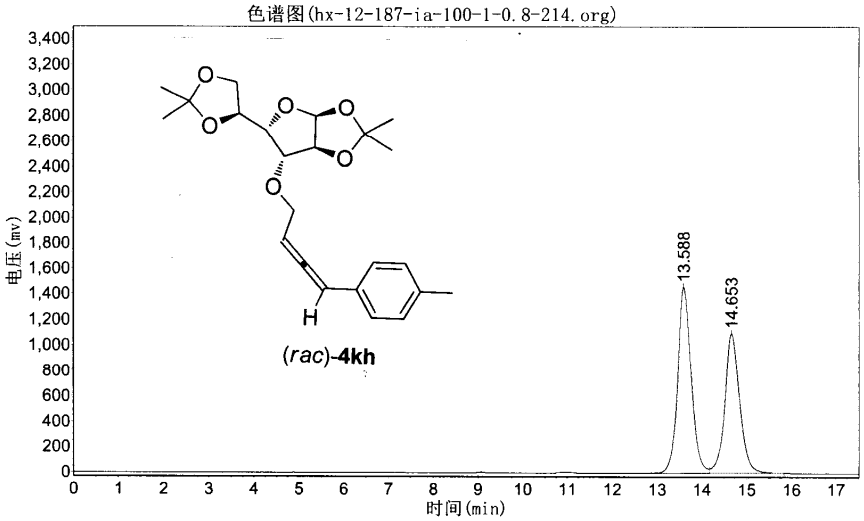
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.798	2998.733	59516.047	1.1298
2		14.838	231547.188	5208389.000	98.8702
总计			234545.921	5267905.047	100.0000

hx-12-187-ia-100-1-0.8-214

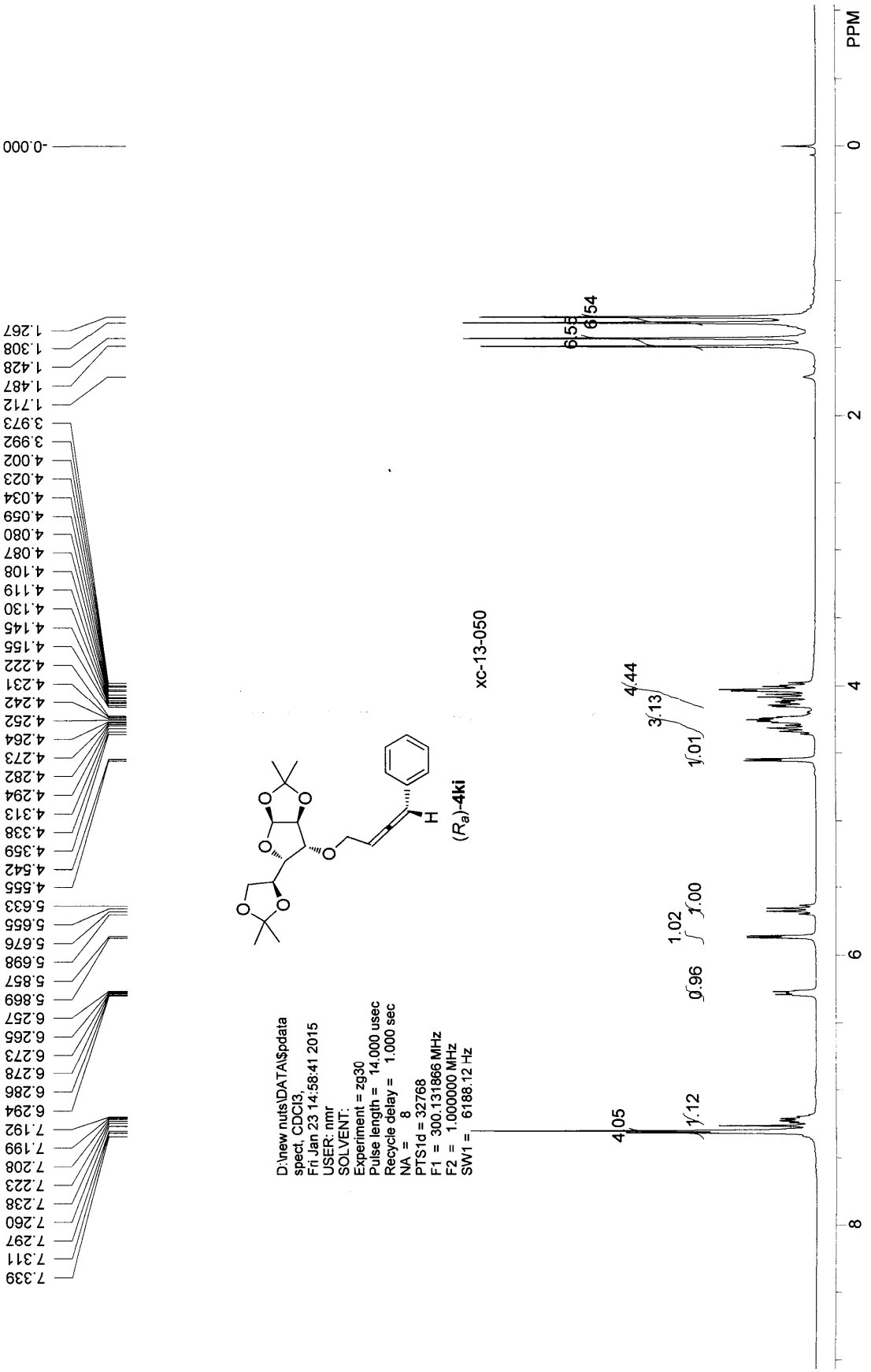
实验时间：2015/2/5,13:54:57　　报告时间：2015/2/5,17:04:30
谱图文件:D:\zhuguangjiong\hx\20150205\hx-12-187-ia-100-1-0.8-214.org

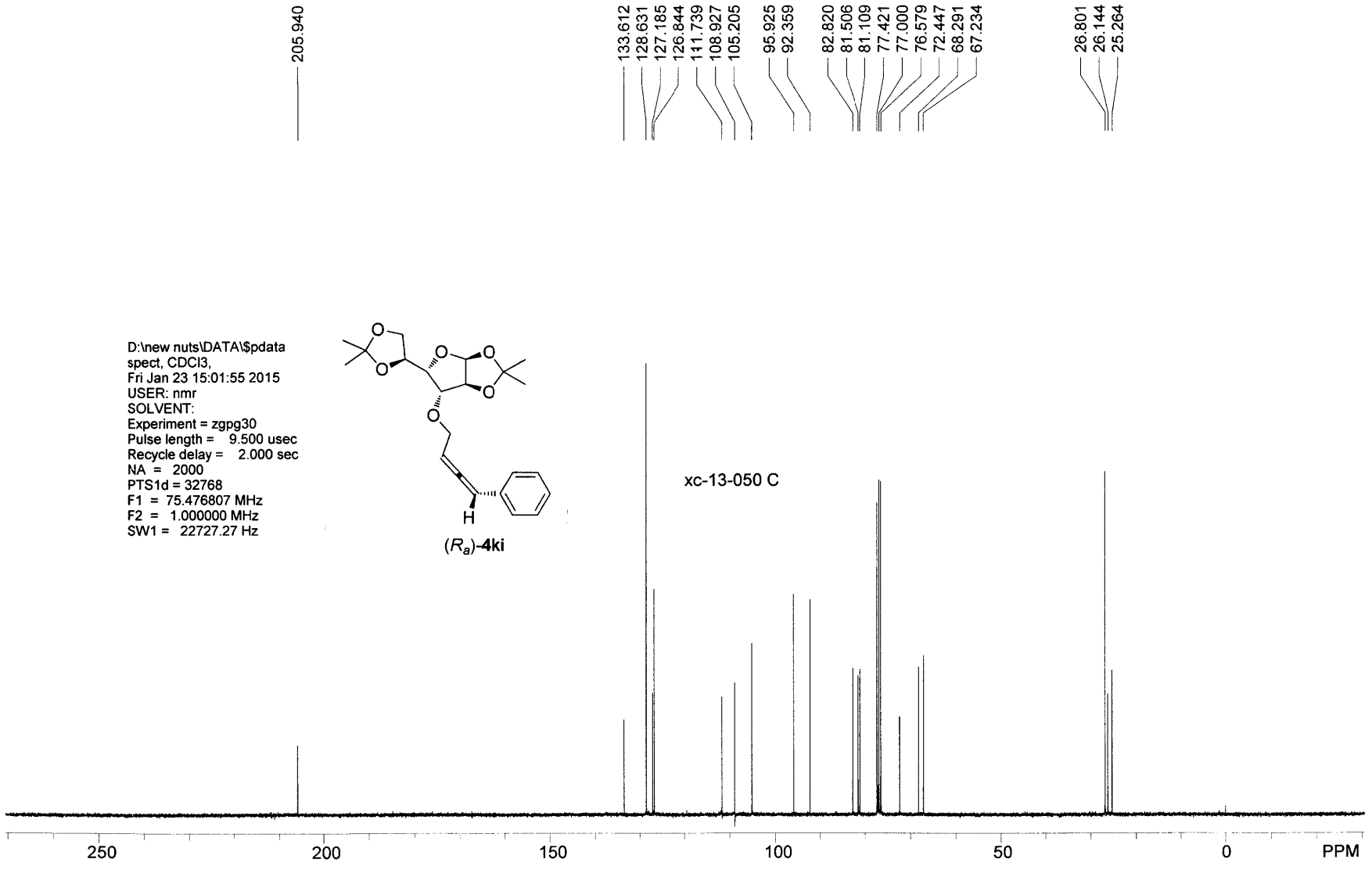
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.588	1456278.625	30375580.000	54.7016
2		14.653	1096109.250	25153990.000	45.2984
总计			2552387.875	55529570.000	100.0000


xc-13-050
(Ra)-4ki
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 23 14:58:41 2015
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
4.05
1.12
0.96
1.02
1.00
1.01
3.13
4.44
6.56
6.54
PPM
0
2
4
6
8
-0.000
1.267
1.308
1.428
1.487
1.712
3.973
3.992
4.002
4.023
4.034
4.059
4.080
4.087
4.108
4.119
4.130
4.145
4.155
4.222
4.231
4.242
4.262
4.264
4.273
4.282
4.294
4.313
4.338
4.359
4.542
4.555
5.633
5.655
5.676
5.698
5.857
5.869
6.257
6.265
6.273
6.278
6.286
6.294
7.192
7.199
7.208
7.223
7.238
7.260
7.297
7.311
7.339


205.940
133.612
128.631
127.185
126.844
111.739
108.927
105.205
95.925
92.359
82.820
81.506
81.109
77.421
77.000
76.579
72.447
68.291
67.234
26.801
26.144
25.264
D:\new nuts\DATA\$pdata
spect, CDCl3,
Fri Jan 23 15:01:55 2015
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2000
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
xc-13-050 C
(Ra)-4ki
250
200
150
100
50
0
PPM

中国科学院上海有机化学研究所

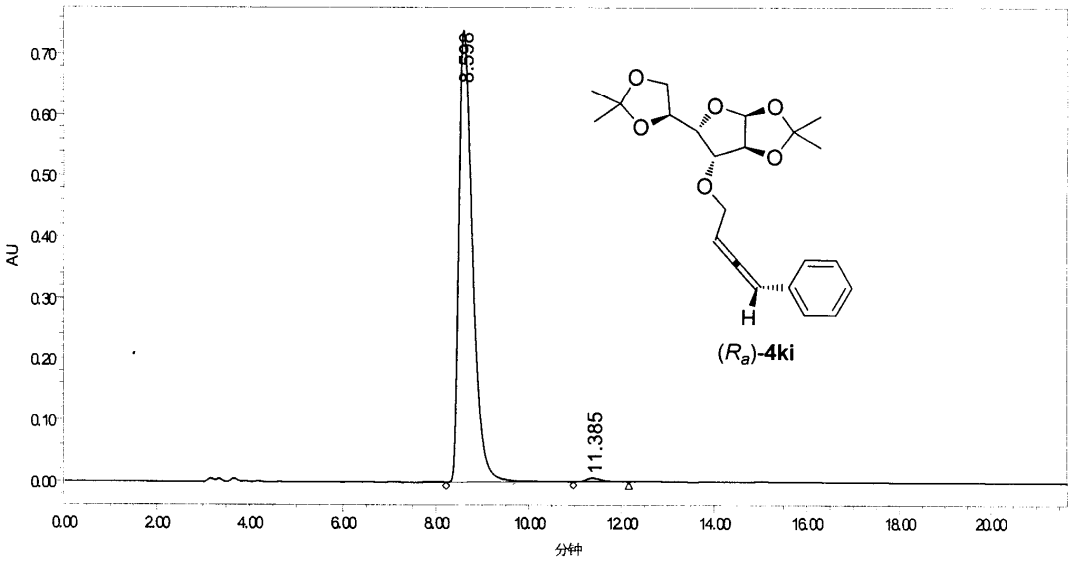
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	xc-13-50-ib-100-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2015/2/6 10:28:08 CST
Vial:	1	Acq. Method:	zgj1001
Injection #:	27	Date Processed:	2015/2/6 17:25:41 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	40.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



(R_a)-**4ki**

	RT (min)	Area (微伏*sec)	%Area	Height (微伏)	% Height
1	8.598	1523825	99.15	739535	99.21
2	11.385	131097	0.85	5890	0.79

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2015/2/6 17:27:05 PRC

中国科学院上海有机化学研究所

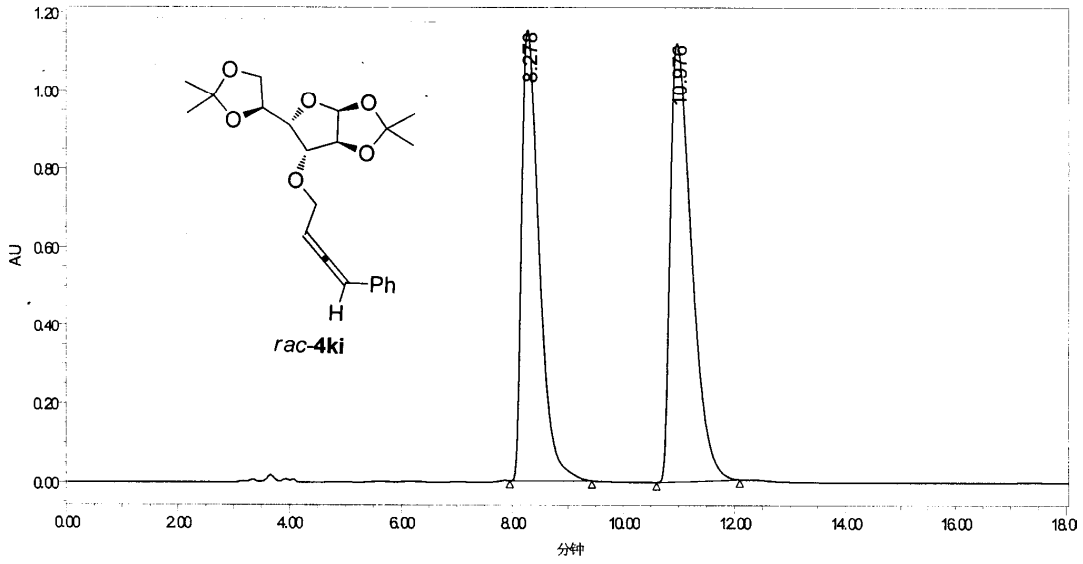
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	xc-13-52-ib-100-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2015/2/6 9:51:46 CST
Vial:	1	Acq. Method:	zgj1001
Injection #:	25	Date Processed:	2015/2/6 17:25:09 CST
Injection Volume:	25.00 ul	Channel Name:	W2489 ChA
Run Time:	40.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



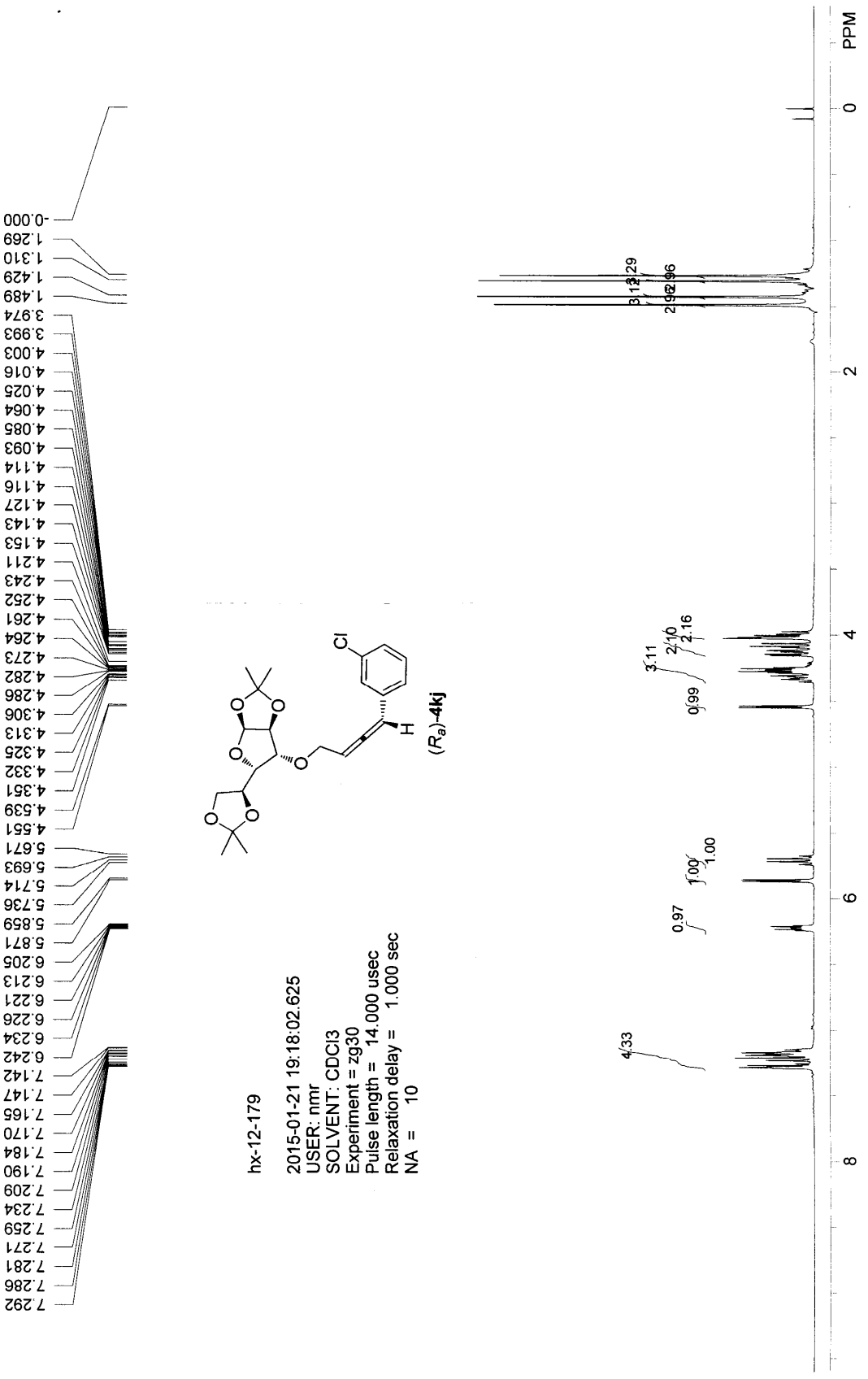
rac-**4ki**

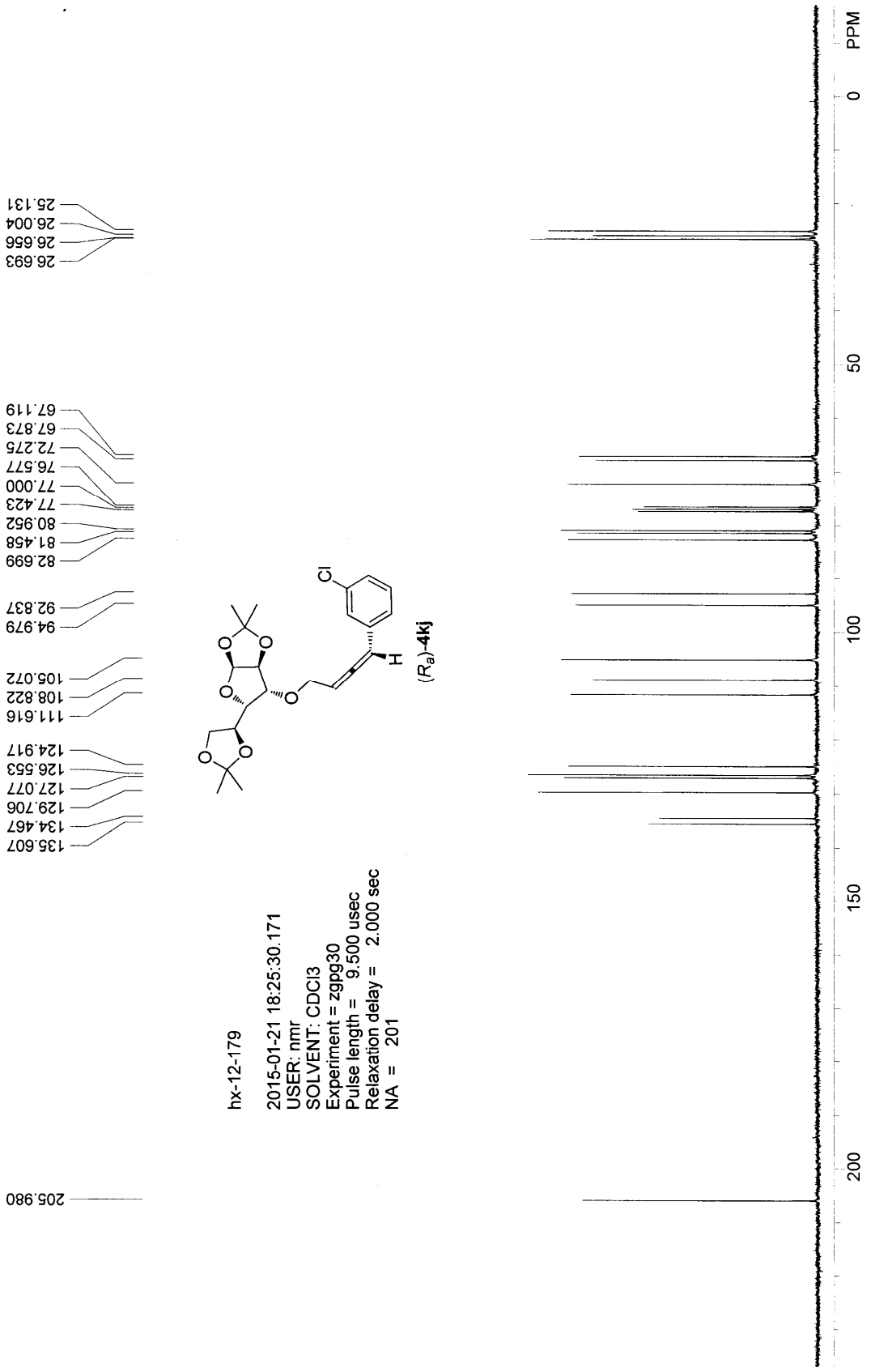
	RT (min)	Area (碘sec)	%Area	Height (碘)	% Height
1	8.278	25298880	45.88	1151925	50.69
2	10.976	29837412	54.12	1120655	49.31

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2015/2/6 17:26:28 PRC

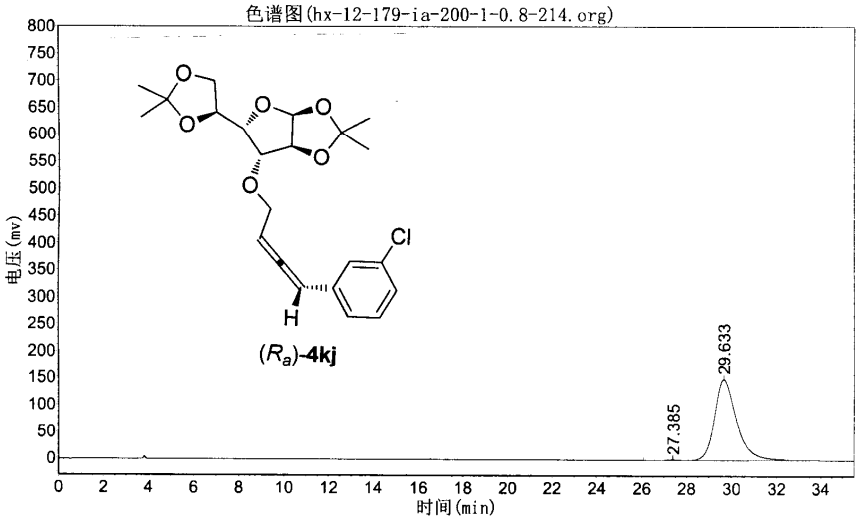

hx-12-179
2015-01-21 19:18:02.625
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 10
(Ra)-4kj
Cl
H
PPM
0
2
4
6
8
4.33
0.97
1.00
1.00
0.99
3.11
2.10
2.16
3.12
3.29
2.96
2.96
-0.000
1.269
1.310
1.429
1.489
3.974
3.993
4.003
4.016
4.025
4.064
4.085
4.093
4.114
4.116
4.127
4.143
4.153
4.211
4.243
4.252
4.261
4.264
4.273
4.282
4.286
4.306
4.313
4.325
4.332
4.351
4.539
4.551
5.671
5.693
5.714
5.736
5.859
5.871
6.205
6.213
6.221
6.226
6.234
6.242
7.142
7.147
7.165
7.170
7.184
7.190
7.209
7.234
7.259
7.271
7.281
7.286
7.292


25.131
26.004
26.656
26.693
67.119
67.873
72.275
76.577
77.000
77.423
80.952
81.458
82.699
92.837
94.979
105.072
108.822
111.616
124.917
126.553
127.077
129.706
134.467
135.607
205.980
hx-12-179
2015-01-21 18:25:30.171
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 201
Cl
H
(Ra)-4kj
PPM
0
50
100
150
200

hx-12-179-ia-200-1-0.8-214

实验时间：2015/2/5,17:12:44　　报告时间：2015/2/5,17:50:47
谱图文件:D:\zhuguangjiong\hx\20150205\hx-12-179-ia-200-1-0.8-214.org

实验内容简介：



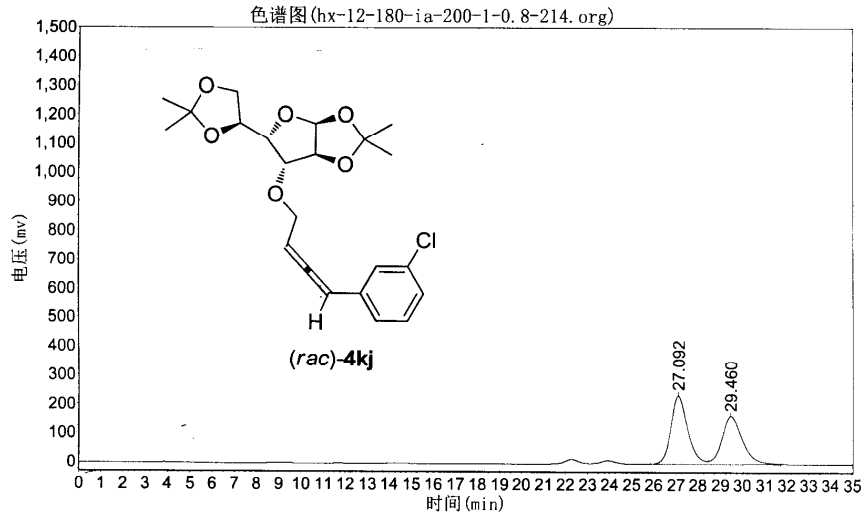
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.385	943.689	48243.895	0.4687
2		29.633	149705.563	10245188.000	99.5313
总计			150649.252	10293431.895	100.0000

hx-12-180-ia-200-1-0.8-214

实验时间：2015/2/5,16:33:13　　报告时间：2015/2/5,17:49:31
谱图文件:D:\zhuguangjiong\hx\20150205\hx-12-180-ia-200-1-0.8-214.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		27.092	235268.906	12366506.000	54.2830
2		29.460	163780.641	10415041.000	45.7170
总计			399049.547	22781547.000	100.0000