

Supporting Information

**Identifying a Non-Chiral Ligand for the Efficient Chirality  
Transfer in Carbonylation of Propargylic Mesylates**

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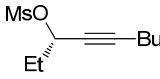
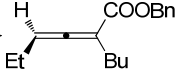
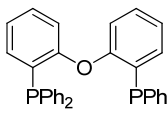
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**General Information.** NMR spectra were taken with Bruker-300 spectrometer (300 MHz for  $^1\text{H}$  NMR, 75.4 MHz for  $^{13}\text{C}$  NMR) and Bruker-400 spectrometer (400 MHz for  $^1\text{H}$  NMR, 100 MHz for  $^{13}\text{C}$  NMR) in  $\text{CDCl}_3$ . Chemical shifts were recorded in ppm relative to the residue of  $\text{CHCl}_3$  in  $\text{CDCl}_3$  and coupling constants were reported in Hz. All reactions were carried out in oven-dried tubes.  $(\text{NH}_4)_2\text{HPO}_4$  was purchased from Sinopharm Chemical Reagent Co., Ltd; DPEphos was purchased from Acros Chemicals. MTBE was dried over sodium wire with benzophenone as indicator and distilled freshly before use. The enantioenriched propargylic mesylates were synthesized from the corresponding enantiopure propargylic alcohols, and used without further purification. Purification by column chromatography was performed using Haiyang (Shandong, China) silica gel (10-40  $\mu$ ).

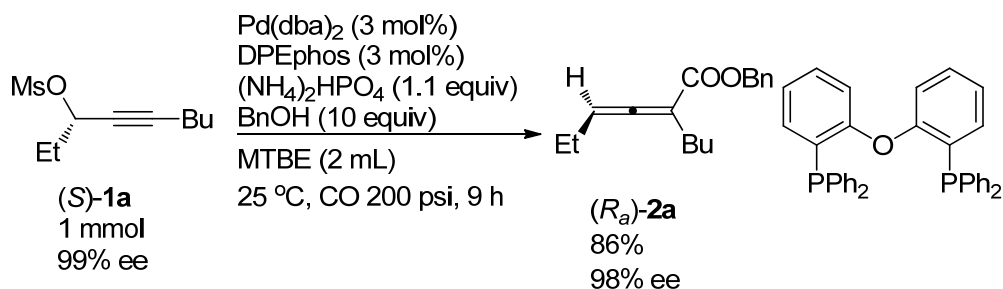
**Table S1 Pressure effect in DPEphos-assisted carboxylation of enantioenriched (S)-1a**

<div style="display: flex; align-items: center; justify-content: space-around;"> <div style="text-align: center;">  <p>(S)-<b>1a</b> ≥99% ee 0.25 mmol</p> </div> <div style="text-align: center;"> <p> <math>\text{Pd(dba)}_2</math> (3 mol%)  DPEphos (3 mol%)  <math>(\text{NH}_4)_2\text{HPO}_4</math> (1.1 equiv)  BnOH (10 equiv)  MTBE (2 mL)  25 °C, CO (X psi), 9 h </p> </div> <div style="text-align: center;">  <p>(R)-<b>2a</b></p> </div> <div style="text-align: center;">  <p>DPEphos</p> </div> </div>			
Entry	X (psi)	Yield% of (R <sub>a</sub> )- <b>2a</b> <sup>a)</sup>	Ee% of (R <sub>a</sub> )- <b>2a</b> <sup>b)</sup>
1	15	75	87
2	40	74	86
3	60	79	95
4	80	76	96
5	100	74	97
6	200	82	97

<sup>a)</sup> Isolated yield. <sup>b)</sup> ee value of the product as determined by HPLC.

## Experimental details and analytical data

### (1) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-2,3-hexadienoate ((*R<sub>a</sub>*)-2a) (zwl-1-92)



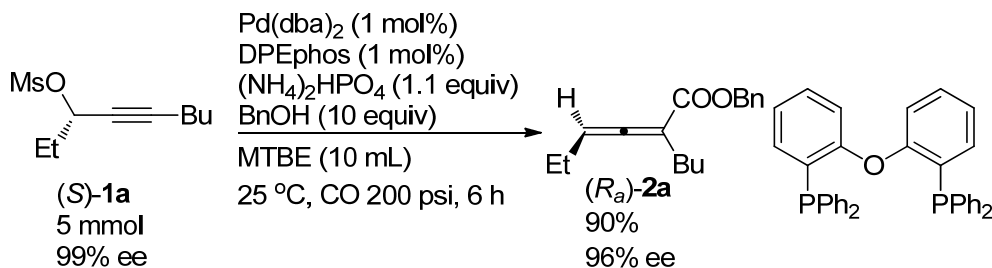
**Typical procedure 1:** To a flame-dried Schlenk tube were added Pd(dba)<sub>2</sub> (17.5 mg, 0.03 mmol) and DPEphos (16.6 mg, 0.03 mmol). Then the tube was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Freshly distilled MTBE (1 mL) was subsequently added under argon. The resulting Pd(0) mixture was stirred for 1 hour at room temperature.

To a flame-dried tube filled with argon were added (*S*)-**1a** (218.2 mg, 1.0 mmol) / MTBE (0.5 mL), BnOH (1.0815 g, 10 mmol) / MTBE (0.5 mL) and (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.8 mg, 1.1 mmol). After the Pd(0) mixture was transferred by syringe to the tube, it was placed into the Parr reactor. The air in the reactor was replaced by CO gas three times sequentially, the Parr reactor was charged to 200 psi with CO gas. The mixture was stirred at room temperature till the reaction ended, then the gas was vented in the hood safely. The resulting mixture was diluted with Et<sub>2</sub>O (10 mL), washed with brine (10 mL), and dried over Na<sub>2</sub>SO<sub>4</sub>. After filtration and concentration under reduced pressure, the crude product was purified by flash chromatography on silica gel to afford (*R<sub>a</sub>*)-**2a** (223.3 mg, 86%) as an oil [eluent: petroleum ether (b.p. 60-90 °C)/ethyl ether = 250/1]: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 0.5 mL/min, λ = 214 nm, *t<sub>R</sub>* (minor) = 16.1 min, *t<sub>R</sub>* (major) = 18.1 min); [α]<sub>D</sub><sup>25</sup> = -38.5 (c = 1.17, CHCl<sub>3</sub>) [96% ee, [α]<sub>D</sub><sup>24</sup> = -38.0 (c = 1.17, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.40-7.23 (m, 5 H, Ar-H), 5.63-5.54 (m, 1 H, =CH), 5.22 (d, *J* = 12.6 Hz, 1 H, one proton from Bn), 5.14 (d, *J* = 12.9 Hz, 1 H, one proton from Bn), 2.32-2.18 (m, 2 H, CH<sub>2</sub>), 2.18-2.05 (m, 2 H, CH<sub>2</sub>), 1.50-1.27 (m, 4 H, 2 x CH<sub>2</sub>), 1.04 (t, *J* = 7.2 Hz, 3 H, CH<sub>3</sub>), 0.90 (t, *J* = 7.1 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR

(75 MHz, CDCl<sub>3</sub>):  $\delta$  = 209.8, 167.6, 136.4, 128.3, 127.8, 127.5, 101.1, 96.7, 66.1, 30.3, 28.1, 22.2, 21.3, 13.9, 13.2.

The following compounds ((*R<sub>a</sub>*)-**2b**-(*R<sub>a</sub>*)-**2l** and (*S<sub>a</sub>*)-**2m**) in Table 4 were prepared according to this **Typical Procedure 1**.

**Reaction on gram scale:**

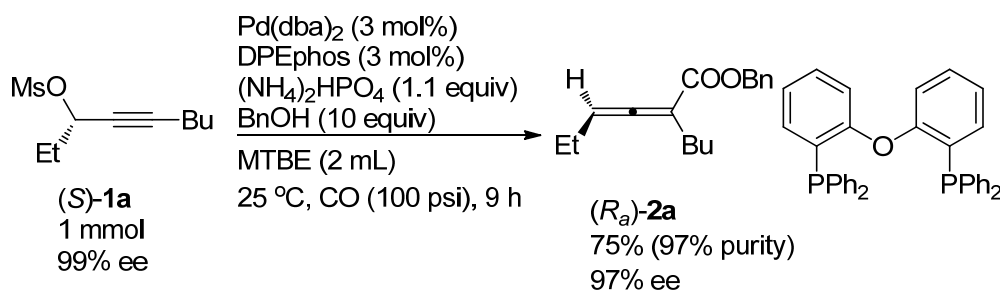


To a flame-dried Schlenk tube were added Pd(dba)<sub>2</sub> (28.8 mg, 0.05 mmol) and DPEphos (27.9 mg, 0.05 mmol). Then the tube was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Freshly distilled MTBE (2 mL) was subsequently added under argon. The resulting Pd(0) mixture was stirred for 1 hour at room temperature.

To a flame-dried tube filled with argon were added (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (0.7260 g, 5.5 mmol), (*S*)-**1a** (1.1021 g, 5 mmol) / MTBE (4 mL), and BnOH (5.4088 g, 50 mmol) / MTBE (4 mL). After the Pd(0) mixture was transferred by syringe to the tube, it was placed into the Parr reactor. The air in the reactor was replaced by CO gas three times sequentially, the Parr reactor was charged to 200 psi with CO gas. The mixture was stirred at room temperature till the reaction ended, then the gas was vented in the hood safely. The resulting mixture was diluted with Et<sub>2</sub>O (20 mL), washed with brine (20 mL), and dried over Na<sub>2</sub>SO<sub>4</sub>. After filtration and concentration under reduced pressure, the crude product was purified by flash chromatography on silica gel to afford (*R<sub>a</sub>*)-**2a** (1.1750 g, 90%) as an oil [eluent: petroleum ether (b.p. 60-90 °C)/ethyl ether = 250/1]: 96% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 11.3 min,  $t_R$  (major) = 13.0 min);  $[\alpha]_D^{21}$  = -37.8 ( $c$  = 1.21, CHCl<sub>3</sub>) [96% ee,  $[\alpha]_D^{24}$  = -38.0 ( $c$  = 1.17, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.39-7.23 (m, 5 H, Ar-H), 5.63-5.54 (m, 1 H, =CH), 5.22 (d,  $J$  = 12.6 Hz, 1 H, one proton from Bn), 5.14 (d,  $J$  = 12.6 Hz, 1 H,

one proton from Bn), 2.32-2.18 (m, 2 H, CH<sub>2</sub>), 2.18-2.06 (m, 2 H, CH<sub>2</sub>), 1.49-1.23 (m, 4 H, 2 x CH<sub>2</sub>), 1.04 (t, *J* = 7.4 Hz, 3 H, CH<sub>3</sub>), 0.90 (t, *J* = 6.9 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 209.8, 167.6, 136.5, 128.4, 127.8, 127.5, 101.1, 96.7, 66.1, 30.3, 28.1, 22.2, 21.4, 13.9, 13.2.

## (2) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-2,3-hexadienoate ((*R<sub>a</sub>*)-2a) (wyl-17-64)



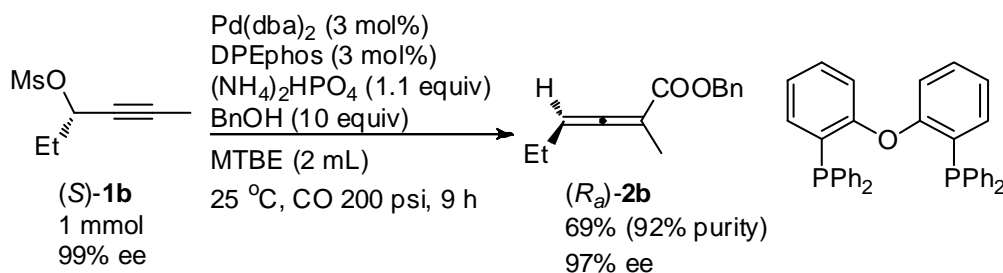
**Typical procedure 2:** To a flame-dried Schlenk tube were added Pd(dba)<sub>2</sub> (17.3 mg, 0.03 mmol) and DPEphos (16.8 mg, 0.03 mmol). Then the tube was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Freshly distilled MTBE (1 mL) was subsequently added under argon. The resulting Pd(0) mixture was stirred for 1 hour at room temperature.

To a flame-dried tube filled with argon were added (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (144.9 mg, 1.1 mmol), (*S*)-**1a** (218.9 mg, 1.0 mmol) / MTBE (0.5 mL), and BnOH (1.0808 g, 10 mmol) / MTBE (0.5 mL). After the Pd(0) mixture was transferred by syringe to the tube, it was placed into the Parr reactor. The air in the reactor was replaced by CO gas three times sequentially, the Parr reactor was charged to 100 psi with CO gas. The mixture was stirred at room temperature till the reaction ended, then the gas was vented in the hood safely. The resulting mixture was diluted with Et<sub>2</sub>O (10 mL), washed with brine (10 mL), and dried over Na<sub>2</sub>SO<sub>4</sub>. After filtration and concentration under reduced pressure, the crude product was purified by flash chromatography on silica gel to afford (*R<sub>a</sub>*)-**2a** (201.3 mg, 75%, 97% purity) as an oil [eluent: petroleum ether (b.p. 60-90 °C)/ethyl ether = 250/1]: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, *t<sub>R</sub>* (minor) = 10.8 min, *t<sub>R</sub>* (major) = 12.5 min); [α]<sub>D</sub><sup>19</sup> = -38.2 (*c* = 1.25, CHCl<sub>3</sub>) [96% ee, [α]<sub>D</sub><sup>24</sup> = -38.0 (*c* = 1.17, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.40-7.25 (m, 5 H, Ar-H),

5.63-5.55 (m, 1 H, =CH), 5.22 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 5.15 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 2.32-2.18 (m, 2 H, CH<sub>2</sub>), 2.18-2.06 (m, 2 H, CH<sub>2</sub>), 1.48-1.28 (m, 4 H, 2 x CH<sub>2</sub>), 1.04 (t,  $J = 7.4$  Hz, 3 H, CH<sub>3</sub>), 0.90 (t,  $J = 7.2$  Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 209.8, 167.5, 136.4, 128.3, 127.8, 127.5, 101.1, 96.7, 66.1, 30.2, 28.1, 22.2, 21.3, 13.8, 13.2$ .

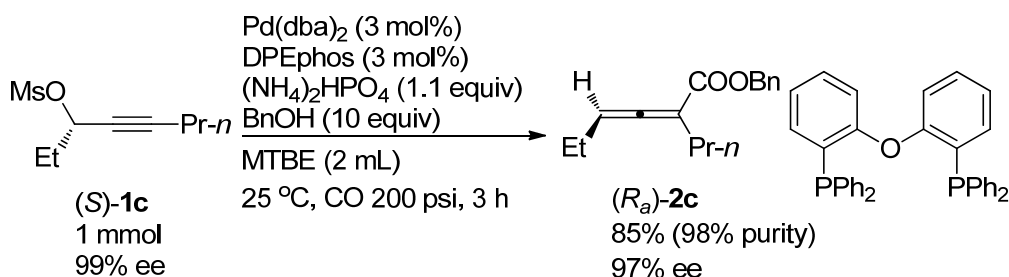
The following compounds ((*R<sub>a</sub>*)-**2j**-(*R<sub>a</sub>*)-**2l** and (*S<sub>a</sub>*)-**2n**) in Table 4 were prepared according to this **Typical Procedure 2**.

### (3) Preparation of (*R<sub>a</sub>*)-benzyl 2-methyl-2,3-hexadienoate ((*R<sub>a</sub>*)-**2b**) (zwl-2-111)



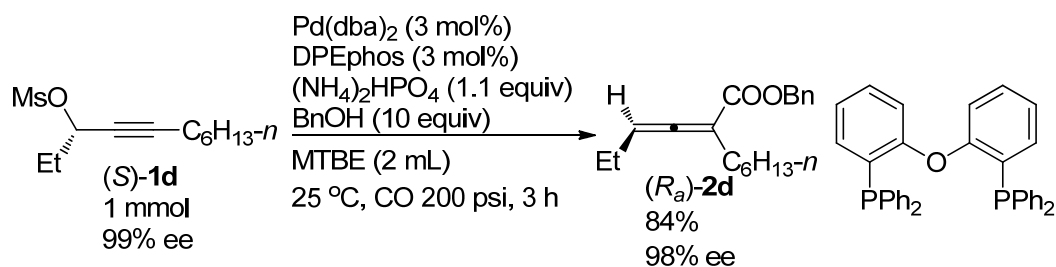
Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.4 mg, 0.03 mmol), DPEphos (16.9 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.5 mg, 1.1 mmol), (*S*)-**1b** (176.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0811 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2b** (162.3 mg, 69%, 92% purity) as an oil: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda = 214$  nm,  $t_R$  (minor) = 10.6 min,  $t_R$  (major) = 12.3 min);  $[\alpha]_D^{22} = -62.4$  ( $c = 1.40$ , CHCl<sub>3</sub>) [90% ee,  $[\alpha]_D^{20} = -65.6$  ( $c = 1.17$ , CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta = 7.41$ - $7.22$  (m, 5 H, Ar-H), 5.57-5.48 (m, 1 H, =CH), 5.22 (d,  $J = 12.3$  Hz, 1 H, one proton from Bn), 5.14 (d,  $J = 12.6$  Hz, 1 H, one proton from Bn), 2.18-2.05 (m, 2 H, CH<sub>2</sub> from Et), 1.89 (d,  $J = 3.0$  Hz, 3 H, CH<sub>3</sub> from Me), 1.03 (t,  $J = 7.5$  Hz, 3 H, CH<sub>3</sub> from Et); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 210.1, 167.8, 136.4, 128.4, 127.8, 127.5, 96.1, 95.6, 66.2, 21.2, 15.2, 13.2$ .

### (4) Preparation of (*R<sub>a</sub>*)-benzyl 2-propyl-2,3-hexadienoate ((*R<sub>a</sub>*)-**2c**) (zwl-2-90)



Following **Typical procedure 1**. The reaction of  $\text{Pd(dba)}_2$  (17.3 mg, 0.03 mmol), DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.0 mg, 1.1 mmol), **(S)-1c** (205.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0800 g, 10 mmol) / MTBE (0.5 mL) afforded **(R<sub>a</sub>)-2c** (213.3 mg, 85%, 98% purity) as an oil: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 11.8 min,  $t_R$  (major) = 13.5 min);  $[\alpha]_D^{24} = -44.3$  ( $c$  = 1.22,  $\text{CHCl}_3$ ) [97% ee,  $[\alpha]_D^{22} = -44.2$  ( $c$  = 1.08,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.40-7.22 (m, 5 H, Ar-H), 5.63-5.53 (m, 1 H, =CH), 5.21 (d,  $J$  = 12.3 Hz, 1 H, one proton from Bn), 5.14 (d,  $J$  = 12.3 Hz, 1 H, one proton from Bn), 2.32-2.17 (m, 2 H,  $\text{CH}_2$ ), 2.17-2.05 (m, 2 H,  $\text{CH}_2$ ), 1.55-1.40 (m, 2 H,  $\text{CH}_2$ ), 1.04 (t,  $J$  = 7.2 Hz, 3 H,  $\text{CH}_3$ ), 0.93 (t,  $J$  = 7.4 Hz, 3 H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 209.9, 167.6, 136.4, 128.4, 127.8, 127.5, 101.0, 96.7, 66.1, 30.5, 21.3, 13.6, 13.2.

##### (5) Preparation of **(R<sub>a</sub>)-benzyl 2-*n*-hexyl-2,3-hexadienoate ((R<sub>a</sub>)-2d) (zwl-2-47)**

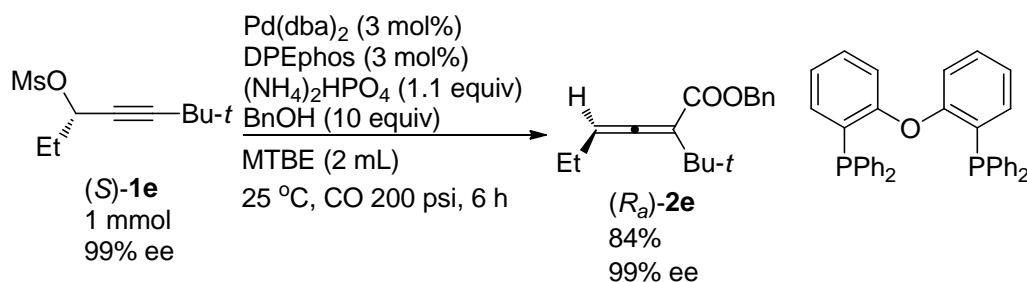


Following **Typical procedure 1**. The reaction of  $\text{Pd(dba)}_2$  (17.4 mg, 0.03 mmol), DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.3 mg, 1.1 mmol), **(S)-1d** (246.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0814 g, 10 mmol) / MTBE (0.5 mL) afforded **(R<sub>a</sub>)-2d** (240.1 mg, 84%) as an oil: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 0.5 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 12.1 min,  $t_R$  (major) = 13.8 min);  $[\alpha]_D^{30} = -33.6$  ( $c$  = 1.48,  $\text{CHCl}_3$ ) [95% ee,  $[\alpha]_D^{24} = -33.3$  ( $c$  = 1.21,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.40-7.24 (m, 5



H, Ar-H), 5.63-5.54 (m, 1 H, =CH), 5.22 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 5.15 (d,  $J = 12.6$  Hz, 1 H, one proton from Bn), 2.32-2.18 (m, 2 H, CH<sub>2</sub>), 2.18-2.06 (m, 2 H, CH<sub>2</sub>), 1.50-1.20 (m, 8 H, 4 x CH<sub>2</sub>), 1.04 (t,  $J = 7.4$  Hz, 3 H, CH<sub>3</sub>), 0.88 (t,  $J = 6.9$  Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 209.8, 167.6, 136.5, 128.4, 127.8, 127.5, 101.2, 96.7, 66.1, 31.6, 28.8, 28.4, 28.1, 22.6, 21.4, 14.0, 13.3$ .

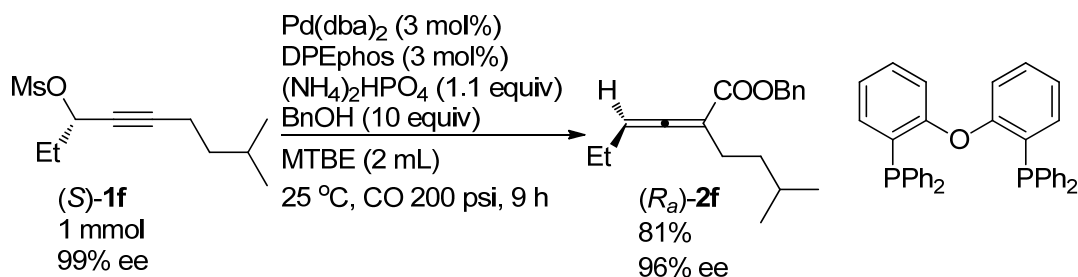
**(6) Preparation of (*R<sub>a</sub>*)-benzyl 2-*t*-butyl-2,3-hexadienoate ((*R<sub>a</sub>*)-2e) (zwl-1-176)**



Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.2 mg, 0.03 mmol), DPEphos (16.6 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.4 mg, 1.1 mmol), (*S*)-**1e** (218.5 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0807 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2e** (216.8 mg, 84%) as an oil: 99% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200:1, 0.3 mL/min,  $\lambda = 214$  nm,  $t_R$  (minor) = 20.4 min,  $t_R$  (major) = 22.2 min);  $[\alpha]_D^{22} = -43.1$  ( $c = 1.70$ , CHCl<sub>3</sub>) [97% ee,  $[\alpha]_D^{23} = -38.4$  ( $c = 1.01$ , CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta = 7.37$ -7.22 (m, 5 H, Ar-H), 5.55 (t,  $J = 6.2$  Hz, 1 H, =CH), 5.19 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 5.12 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 2.16-2.05 (m, 2 H, CH<sub>2</sub>), 1.20 (s, 9 H, 3 x CH<sub>3</sub> from *t*-Bu), 1.03 (t,  $J = 7.5$  Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 208.6, 166.5, 136.5, 128.3, 127.7, 127.3, 110.0, 96.5, 65.6, 33.0, 29.5, 21.3, 13.0$ .

**(7) Preparation of (*R<sub>a</sub>*)-benzyl 2-(3-methylbutyl)-2,3-hexadienoate ((*R<sub>a</sub>*)-2f)**

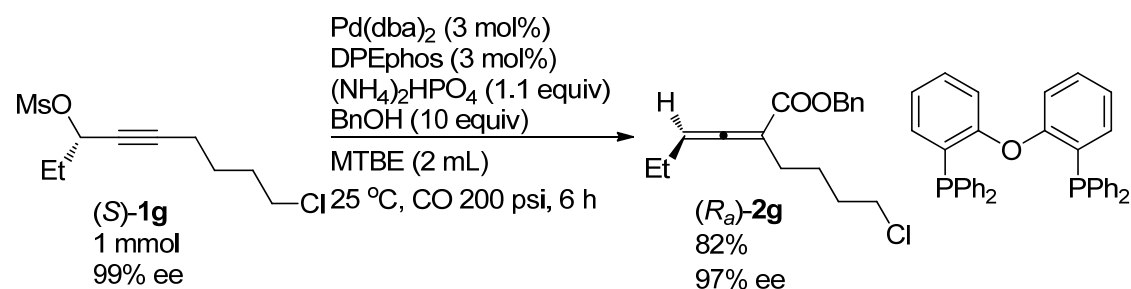
**(wyl-16-113)**



Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.3 mg, 0.03 mmol),

DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (144.9 mg, 1.1 mmol), (*S*)-**1f** (232.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0807 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2f** (220.4 mg, 81%) as an oil: 96% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 300/1, 0.7 mL/min, λ = 214 nm, *t<sub>R</sub>* (minor) = 19.1 min, *t<sub>R</sub>* (major) = 22.1 min); [α]<sup>24</sup><sub>D</sub> = -34.9 (*c* = 1.24, CHCl<sub>3</sub>) [94% ee, [α]<sup>22</sup><sub>D</sub> = -33.0 (*c* = 1.01, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.40-7.23 (m, 5 H, Ar-H), 5.63-5.55 (m, 1 H, =CH), 5.22 (d, *J* = 12.6 Hz, 1 H, one proton from Bn), 5.15 (d, *J* = 12.9 Hz, 1 H, one proton from Bn), 2.29-2.18 (m, 2 H, CH<sub>2</sub>), 2.18-2.06 (m, 2 H, CH<sub>2</sub>), 1.66-1.51 (m, 1 H, CH), 1.37-1.24 (m, 2 H, CH<sub>2</sub>), 1.04 (t, *J* = 7.4 Hz, 3 H, CH<sub>3</sub>), 0.89 (d, *J* = 6.6 Hz, 6 H, 2 x CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 209.7, 167.6, 136.4, 128.3, 127.8, 127.5, 101.3, 96.8, 66.1, 37.2, 27.5, 26.4, 22.5, 21.3, 13.2.

**(8) Preparation of (*R<sub>a</sub>*)-benzyl 2-(4-chlorobutyl)-2,3-hexadienoate ((*R<sub>a</sub>*)-**2g**) (wyl-16-116)**

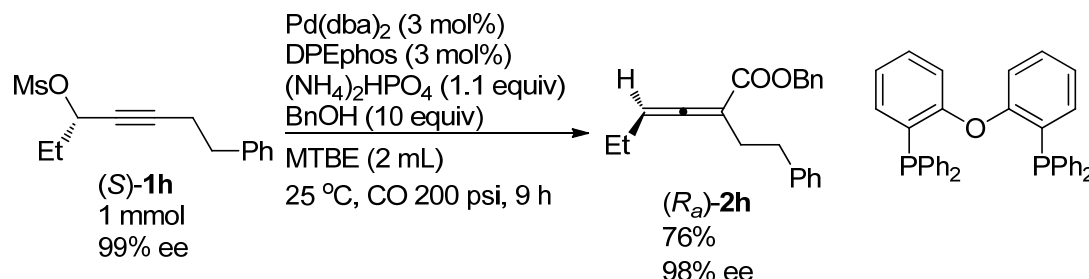


Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.4 mg, 0.03 mmol), DPEphos (16.7 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.0 mg, 1.1 mmol), (*S*)-**1g** (253.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0807 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2g** (240.2 mg, 82%) as an oil: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min, λ = 214 nm, *t<sub>R</sub>* (minor) = 16.8 min, *t<sub>R</sub>* (major) = 18.8 min); [α]<sup>27</sup><sub>D</sub> = -29.7 (*c* = 1.10, CHCl<sub>3</sub>) [95% ee, [α]<sup>22</sup><sub>D</sub> = -29.3 (*c* = 1.17, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.40-7.23 (m, 5 H, Ar-H), 5.66-5.58 (m, 1 H, =CH), 5.22 (d, *J* = 12.6 Hz, 1 H, one proton from Bn), 5.15 (d, *J* = 12.6 Hz, 1 H, one proton from Bn), 3.53 (t, *J* = 6.8 Hz, 2 H, CH<sub>2</sub>), 2.36-2.19 (m, 2 H, CH<sub>2</sub>), 2.19-2.07 (m, 2 H, CH<sub>2</sub>), 1.90-1.74 (m, 2 H, CH<sub>2</sub>), 1.68-1.53 (m, 2 H, CH<sub>2</sub>), 1.05 (t, *J* = 7.4 Hz, 3 H, CH<sub>3</sub> from Et); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ

= 209.7, 167.4, 136.3, 128.4, 127.9, 127.5, 100.5, 97.2, 66.2, 44.7, 31.9, 27.6, 25.3, 21.3, 13.2.

### (9) Preparation of (*R<sub>a</sub>*)-benzyl 2-(2-phenylethyl)-2,3-hexadienoate ((*R<sub>a</sub>*)-2h)

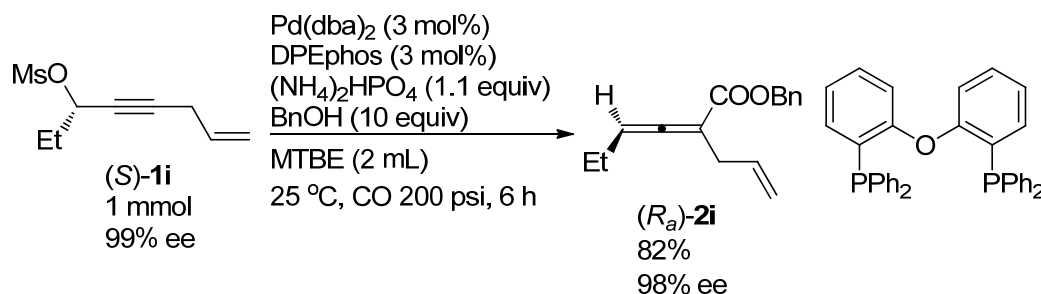
(zwl-1-114)



Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.3 mg, 0.03 mmol), DPEphos (16.6 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.5 mg, 1.1 mmol), (*S*)-1h (266.4 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0813 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-2h (232.2 mg, 76%) as an oil: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 300/1, 1.0 mL/min, λ = 214 nm, *t<sub>R</sub>* (minor) = 26.0 min, *t<sub>R</sub>* (major) = 29.0 min); [α]<sub>D</sub><sup>26</sup> = -14.4 (*c* = 1.50, CHCl<sub>3</sub>) [92% ee, [α]<sub>D</sub><sup>24</sup> = -13.8 (*c* = 1.48, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.45-7.10 (m, 10 H, Ar-H), 5.59-5.50 (m, 1 H, =CH), 5.22 (d, *J* = 12.9 Hz, 1 H, one proton from Bn), 5.15 (d, *J* = 12.6 Hz, 1 H, one proton from Bn), 2.83-2.68 (m, 2 H, CH<sub>2</sub>), 2.68-2.47 (m, 2 H, CH<sub>2</sub>), 2.10-1.96 (m, 2 H, CH<sub>2</sub>), 0.96 (t, *J* = 7.4 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 209.9, 167.2, 141.4, 136.3, 128.4, 128.3, 128.2, 127.8, 127.5, 125.8, 100.3, 97.0, 66.1, 34.3, 30.2, 21.2, 13.1.

### (10) Preparation of (*R<sub>a</sub>*)-benzyl 2-(2-propylenyl)-2,3-hexadienoate ((*R<sub>a</sub>*)-2i)

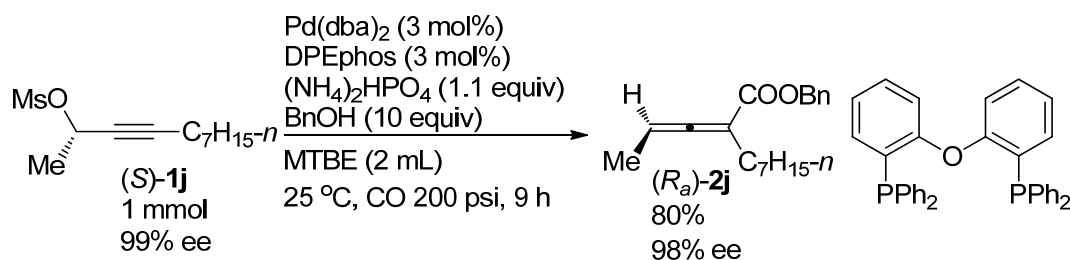
(zwl-2-142)



Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.3 mg, 0.03 mmol), DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.0 mg, 1.1 mmol),

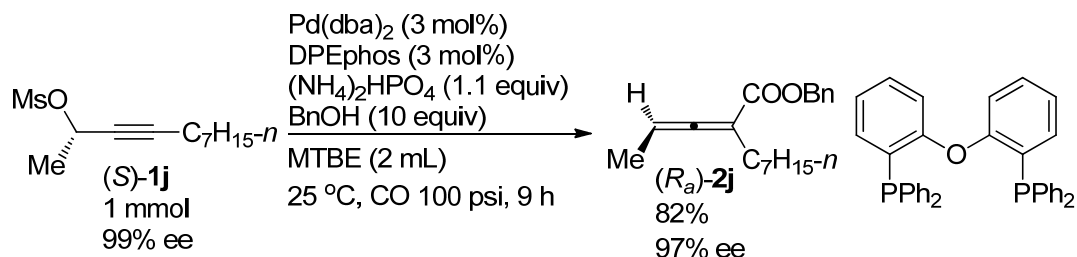
(*S*)-**1i** (202.0 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0809 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2i** (198.7 mg, 82%) as an oil: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 12.9 min,  $t_R$  (major) = 15.0 min);  $[\alpha]_D^{21}$  = -41.7 ( $c$  = 1.57, CHCl<sub>3</sub>) [91% ee,  $[\alpha]_D^{26}$  = -38.0 ( $c$  = 1.21, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.40-7.25 (m, 5 H, Ar-H), 5.91-5.75 (m, 1 H, CH=), 5.67-5.59 (m, 1 H, =CH), 5.22 (d,  $J$  = 12.9 Hz, 1 H, one proton from Bn), 5.15 (d,  $J$  = 12.9 Hz, 1 H, one proton from Bn), 5.13-4.99 (m, 2 H, =CH<sub>2</sub>), 3.09-2.93 (m, 2 H, CH<sub>2</sub>), 2.18-2.06 (m, 2 H, CH<sub>2</sub>), 1.03 (t,  $J$  = 7.4 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 210.1, 167.0, 136.3, 135.2, 128.3, 127.8, 127.5, 116.0, 99.6, 97.1, 66.2, 33.1, 21.2, 13.1.

**(11) Preparation of (*R<sub>a</sub>*)-benzyl 2-*n*-heptyl-2,3-pentadienoate ((*R<sub>a</sub>*)-**2j**) (zwl-1-129)**



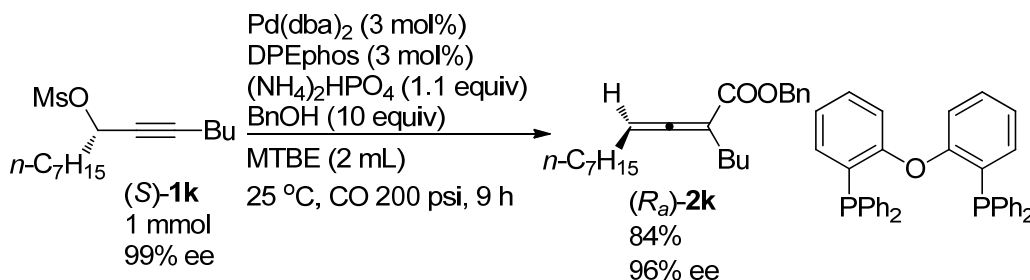
Following **Typical procedure 1**. The reaction of Pd(dba)<sub>2</sub> (17.3 mg, 0.03 mmol), DPEphos (17.0 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.5 mg, 1.1 mmol), (*S*)-**1j** (246.6 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0820 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2j** (229.3 mg, 80%) as an oil: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 0.5 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 14.2 min,  $t_R$  (major) = 17.0 min);  $[\alpha]_D^{26}$  = -29.8 ( $c$  = 1.24, CHCl<sub>3</sub>) [93% ee,  $[\alpha]_D^{23}$  = -27.0 ( $c$  = 1.15, CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.43-7.23 (m, 5 H, Ar-H), 5.56-5.44 (m, 1 H, =CH), 5.18 (s, 2 H, CH<sub>2</sub> from Bn), 2.32-2.13 (m, 2 H, CH<sub>2</sub>), 1.76 (d,  $J$  = 7.2 Hz, 3 H, CH<sub>3</sub>), 1.52-1.16 (m, 10 H, 5 x CH<sub>2</sub>), 0.88 (t,  $J$  = 6.3 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 210.7, 167.5, 136.4, 128.4, 127.8, 127.6, 100.0, 89.7, 66.1, 31.8, 29.1, 29.0, 28.4, 28.0, 22.6, 14.1, 13.3.

**(12) Preparation of (*R<sub>a</sub>*)-benzyl 2-*n*-heptyl-2,3-pentadienoate ((*R<sub>a</sub>*)-**2j**) (wyl-17-62)**



Following **Typical procedure 2**. The reaction of  $\text{Pd(dba)}_2$  (17.3 mg, 0.03 mmol), DPEphos (16.7 mg, 0.03 mmol) / MTBE (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.5 mg, 1.1 mmol),  $(S)\text{-1j}$  (246.9 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0807 g, 10 mmol) / MTBE (0.5 mL) afforded  $(R_a)\text{-2j}$  (234.8 mg, 82%) as an oil: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 10.2 min,  $t_R$  (major) = 12.2 min);  $[\alpha]_D^{19} = -29.5$  ( $c$  = 0.97,  $\text{CHCl}_3$ ) [93% ee,  $[\alpha]_D^{23} = -27.0$  ( $c$  = 1.15,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.40-7.25 (m, 5 H, Ar-H), 5.55-5.45 (m, 1 H, =CH), 5.18 (s, 2 H,  $\text{CH}_2$  from Bn), 2.32-2.14 (m, 2 H,  $\text{CH}_2$ ), 1.76 (d,  $J$  = 6.9 Hz, 3 H,  $\text{CH}_3$ ), 1.50-1.15 (m, 10 H, 5 x  $\text{CH}_2$ ), 0.88 (t,  $J$  = 6.8 Hz, 3 H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 210.7, 167.6, 136.4, 128.4, 127.8, 127.6, 99.9, 89.7, 66.1, 31.8, 29.1, 29.0, 28.4, 28.0, 22.6, 14.1, 13.3.

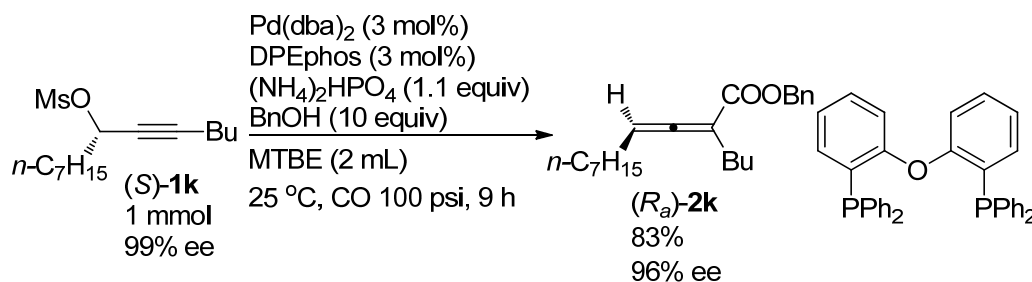
### (13) Preparation of $(R_a)$ -benzyl 2-butyl-2,3-undecadienoate ( $(R_a)\text{-2k}$ ) (zwl-2-95)



Following **Typical procedure 1**. The reaction of  $\text{Pd(dba)}_2$  (17.3 mg, 0.03 mmol), DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.0 mg, 1.1 mmol),  $(S)\text{-1k}$  (288.6 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0809 g, 10 mmol) / MTBE (0.5 mL) afforded  $(R_a)\text{-2k}$  (276.1 mg, 84%) as an oil: 96% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 10.3 min,  $t_R$  (major) = 11.9 min);  $[\alpha]_D^{21} = -43.3$  ( $c$  = 1.06,  $\text{CHCl}_3$ ) [94% ee,  $[\alpha]_D^{24} = -42.6$  ( $c$  = 1.32,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.40-7.21 (m, 5 H, Ar-H), 5.58-5.47 (m, 1 H, =CH), 5.21 (d,  $J$  = 12.9 Hz, 1 H, one proton from Bn),

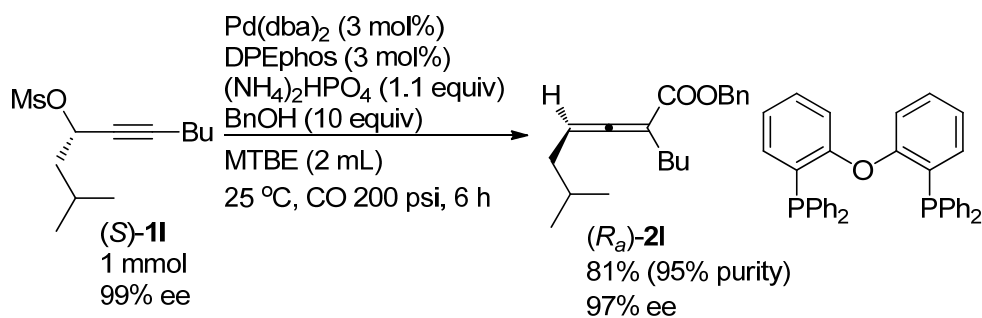
5.15 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 2.33-2.15 (m, 2 H, CH<sub>2</sub>), 2.10 (q,  $J = 7.1$  Hz, 2 H, CH<sub>2</sub>), 1.51-1.12 (m, 14 H, 7 x CH<sub>2</sub>), 0.90 (t,  $J = 7.4$  Hz, 3 H, CH<sub>3</sub>), 0.88 (t,  $J = 7.4$  Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 210.0, 167.6, 136.4, 128.4, 127.8, 127.6, 100.4, 95.1, 66.1, 31.7, 30.3, 29.05, 28.97, 28.2, 28.1, 22.6, 22.2, 14.1, 13.9$ .

**(14) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-2,3-undecadienoate ((*R<sub>a</sub>*)-2k) (wyl-17-61)**



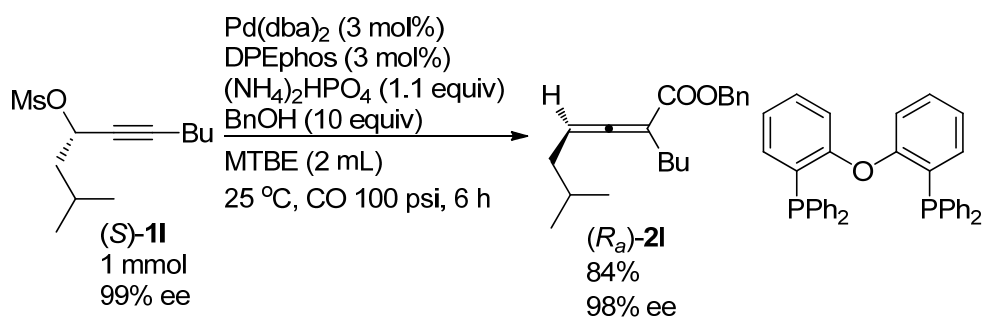
Following **Typical procedure 2**. The reaction of Pd(dba)<sub>2</sub> (17.4 mg, 0.03 mmol), DPEphos (16.6 mg, 0.03 mmol) / MTBE (1 mL), (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> (145.0 mg, 1.1 mmol), (*S*)-**1k** (288.9 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0811 g, 10 mmol) / MTBE (0.5 mL) afforded (*R<sub>a</sub>*)-**2k** (272.5 mg, 83%) as an oil: 96% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda = 214$  nm,  $t_R$  (minor) = 9.3 min,  $t_R$  (major) = 11.1 min);  $[\alpha]_D^{21} = -43.3$  ( $c = 1.03$ , CHCl<sub>3</sub>) [94% ee,  $[\alpha]_D^{24} = -42.6$  ( $c = 1.32$ , CHCl<sub>3</sub>)]<sup>[1]</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta = 7.40$ -7.24 (m, 5 H, Ar-H), 5.56-5.47 (m, 1 H, =CH), 5.21 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 5.15 (d,  $J = 13.2$  Hz, 1 H, one proton from Bn), 2.30-2.15 (m, 2 H, CH<sub>2</sub>), 2.10 (q,  $J = 7.2$  Hz, 2 H, CH<sub>2</sub>), 1.49-1.17 (m, 14 H, 7 x CH<sub>2</sub>), 0.90 (t,  $J = 7.2$  Hz, 3 H, CH<sub>3</sub>), 0.88 (t,  $J = 7.4$  Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta = 210.0, 167.6, 136.4, 128.3, 127.8, 127.6, 100.4, 95.1, 66.1, 31.7, 30.3, 29.04, 28.96, 28.2, 28.1, 22.6, 22.2, 14.1, 13.9$ .

**(15) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-6-methyl-2,3-heptadienoate ((*R<sub>a</sub>*)-2l) (wyl-16-154)**



Following **Typical procedure 1**. The reaction of  $\text{Pd(dba)}_2$  (17.4 mg, 0.03 mmol),  $\text{DPEphos}$  (16.7 mg, 0.03 mmol) /  $\text{MTBE}$  (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.6 mg, 1.1 mmol), **(S)-1I** (246.6 mg, 1 mmol) /  $\text{MTBE}$  (0.5 mL),  $\text{BnOH}$  (1.0817 g, 10 mmol) /  $\text{MTBE}$  (0.5 mL) afforded **(R<sub>a</sub>)-2I** (243.7 mg, 81%, 95% purity) as an oil: 97% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 400/1, 0.5 mL/min,  $\lambda$  = 214 nm,  $t_R$  (minor) = 14.9 min,  $t_R$  (major) = 16.9 min);  $[\alpha]_D^{22} = -42.2$  ( $c$  = 1.75,  $\text{CHCl}_3$ ) [97% ee,  $[\alpha]_D^{24} = -42.5$  ( $c$  = 1.13,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.41-7.21 (m, 5 H, Ar-H), 5.51-5.41 (m, 1 H, =CH), 5.19 (d,  $J$  = 12.3 Hz, 1 H, one proton from Bn), 5.13 (d,  $J$  = 12.6 Hz, 1 H, one proton from Bn), 2.30-2.18 (m, 2 H,  $\text{CH}_2$ ), 1.99 (t,  $J$  = 7.2 Hz, 2 H,  $\text{CH}_2$ ), 1.78-1.60 (m, 1 H, CH), 1.50-1.25 (m, 4 H, 2 x  $\text{CH}_2$ ), 0.97-0.77 (m, 9 H, 3 x  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 210.3, 167.6, 136.3, 128.3, 127.8, 99.7, 93.5, 66.2, 37.4, 30.2, 28.4, 28.2, 22.14, 22.09, 13.8.

**(16) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-6-methyl-2,3-heptadienoate ((*R<sub>a</sub>*)-2I)**  
**(wyl-17-60)**

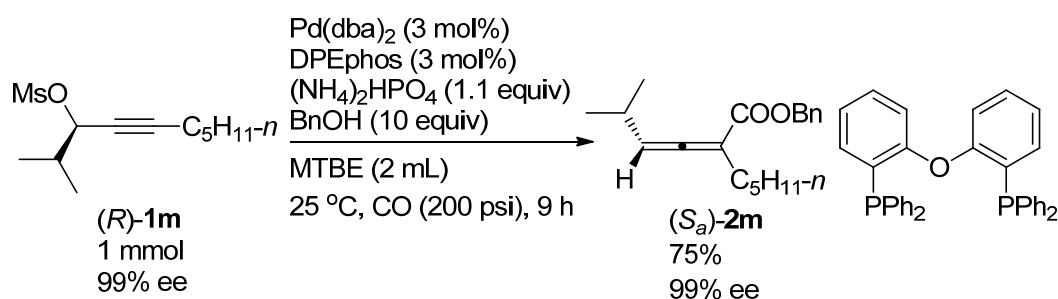


Following **Typical procedure 2**. The reaction of  $\text{Pd(dba)}_2$  (17.2 mg, 0.03 mmol),  $\text{DPEphos}$  (16.6 mg, 0.03 mmol) /  $\text{MTBE}$  (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (146.0 mg, 1.1 mmol), **(S)-1I** (247.0 mg, 1 mmol) /  $\text{MTBE}$  (0.5 mL),  $\text{BnOH}$  (1.0820 g, 10 mmol) /  $\text{MTBE}$  (0.5 mL) afforded **(R<sub>a</sub>)-2I** (240.3 mg, 84%) as an oil: 98% ee (HPLC conditions: Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 400/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$

(minor) = 9.4 min,  $t_R$  (major) = 10.7 min);  $[\alpha]^{19}_D = -42.2$  ( $c = 1.32$ ,  $\text{CHCl}_3$ ) [97% ee,  $[\alpha]^{24}_D = -42.5$  ( $c = 1.13$ ,  $\text{CHCl}_3$ )]<sup>[1]</sup>;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.40$ -7.24 (m, 5 H, Ar-H), 5.51-5.41 (m, 1 H, =CH), 5.20 (d,  $J = 12.3$  Hz, 1 H, one proton from Bn), 5.14 (d,  $J = 12.6$  Hz, 1 H, one proton from Bn), 2.30-2.15 (m, 2 H,  $\text{CH}_2$ ), 2.00 (t,  $J = 7.2$  Hz, 2 H,  $\text{CH}_2$ ), 1.77-1.61 (m, 1 H, CH), 1.50-1.27 (m, 4 H, 2 x  $\text{CH}_2$ ), 0.97-0.82 (m, 9 H, 3 x  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 210.3$ , 167.6, 136.3, 128.3, 127.8, 99.7, 93.5, 66.2, 37.4, 30.2, 28.4, 28.2, 22.2, 22.1, 13.9.

### (17) Preparation of (*S<sub>a</sub>*)-benzyl 2-pentyl-5-methyl-2,3-hexadienoate ((*S<sub>a</sub>*)-2m)

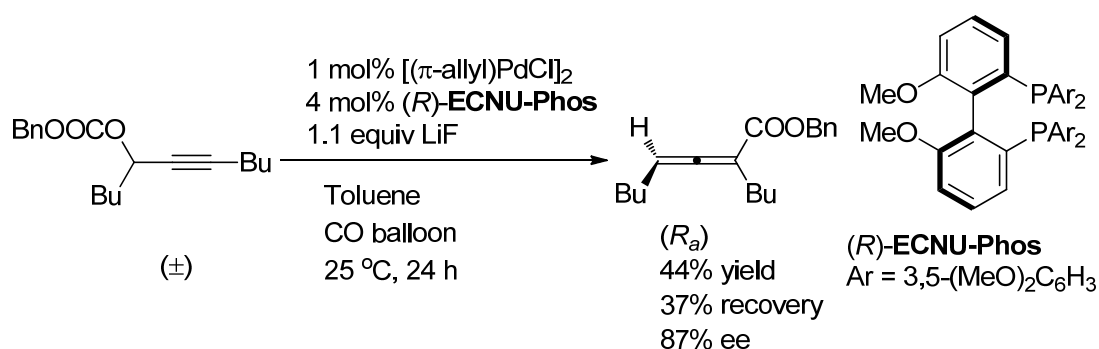
(zwl-1-158)



Following **Typical procedure 1**. The reaction of  $\text{Pd}(\text{dba})_2$  (17.3 mg, 0.03 mmol), DPEphos (16.8 mg, 0.03 mmol) / MTBE (1 mL),  $(\text{NH}_4)_2\text{HPO}_4$  (145.5 mg, 1.1 mmol), (*R*)-**1m** (246.5 mg, 1 mmol) / MTBE (0.5 mL), BnOH (1.0808 g, 10 mmol) / MTBE (0.5 mL) afforded (*S<sub>a</sub>*)-**2m** (214.9 mg, 75%) as an oil: 99% ee (HPLC conditions: Regis (*S, S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 0.5 mL/min,  $\lambda = 214$  nm,  $t_R$  (minor) = 19.9 min,  $t_R$  (major) = 21.9 min);  $[\alpha]^{21}_D = -39.3$  ( $c = 1.22$ ,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.40$ -7.22 (m, 5 H, Ar-H), 5.58-5.50 (m, 1 H, =CH), 5.23 (d,  $J = 12.9$  Hz, 1 H, one proton from Bn), 5.12 (d,  $J = 13.2$  Hz, 1 H, one proton from Bn), 2.52-2.34 (m, 1 H, CH), 2.33-2.15 (m, 2 H,  $\text{CH}_2$ ), 1.52-1.23 (m, 6 H, 3 x  $\text{CH}_2$ ), 1.05 (d,  $J = 6.9$  Hz, 6 H, 2 x  $\text{CH}_3$ ), 0.88 (t,  $J = 6.9$  Hz, 3 H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta = 208.8$ , 167.5, 136.4, 128.3, 127.8, 127.5, 102.2, 101.6, 66.1, 31.3, 28.3, 28.1, 27.8, 22.4, 22.34, 22.31, 14.0; MS (ESI  $m/z$ ): 341 ( $\text{M}+\text{Na}+\text{MeOH}$ )<sup>+</sup>, 325 ( $\text{M}+\text{K}$ )<sup>+</sup>, 309 ( $\text{M}+\text{Na}$ )<sup>+</sup>, 287 ( $\text{M}+\text{H}$ )<sup>+</sup>; IR (neat):  $\nu = 2967$ , 1956, 1711, 1253, 1193, 1121, 1057  $\text{cm}^{-1}$ ; HRMS calcd. for  $\text{C}_{19}\text{H}_{26}\text{O}_2$  [ $\text{M}^+$ ]: 286.1933, found: 286.1935.

### (18) Preparation of (*R<sub>a</sub>*)-benzyl 2-butyl-2,3-octadienoate (wyl-16-65)



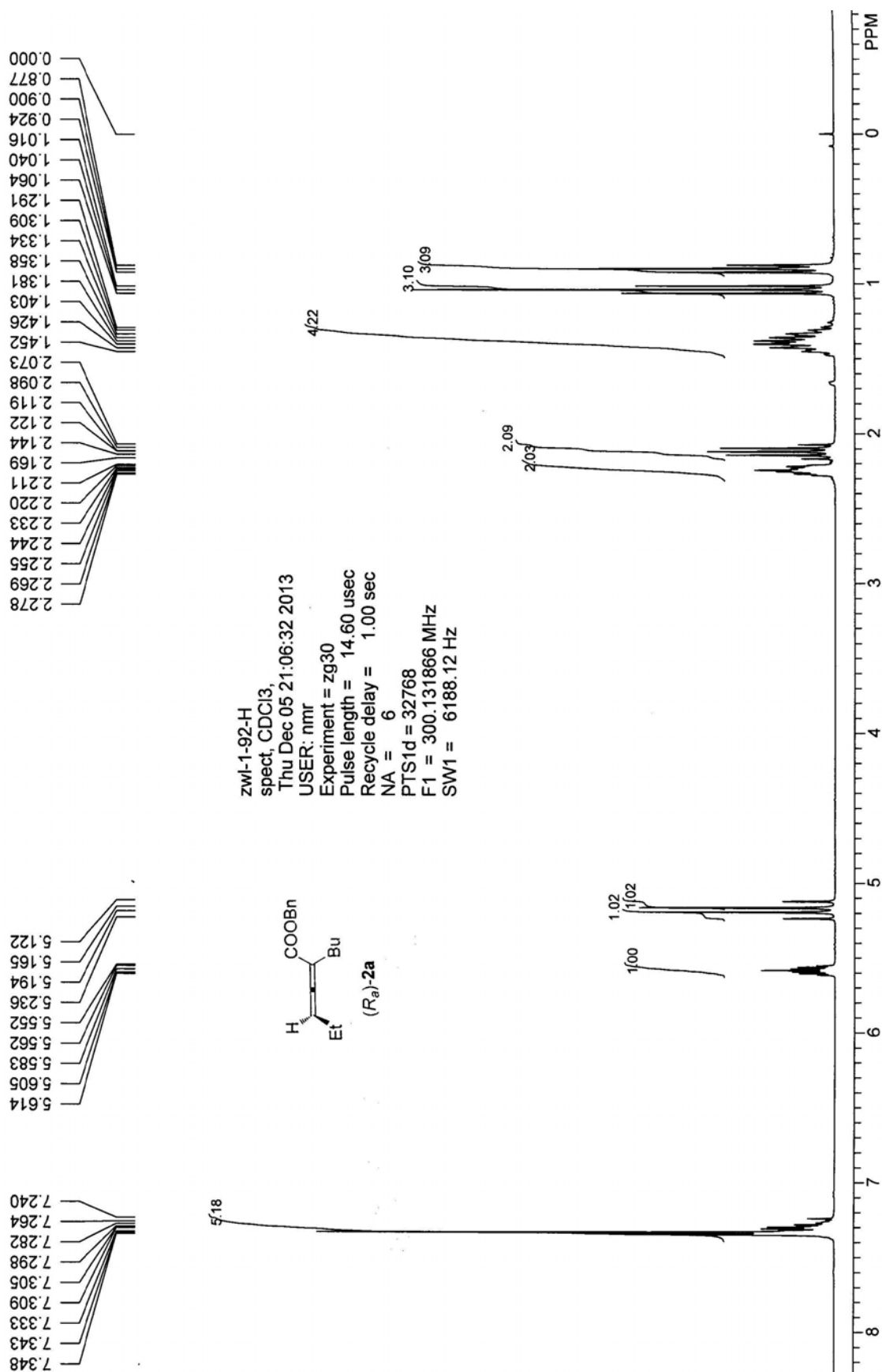


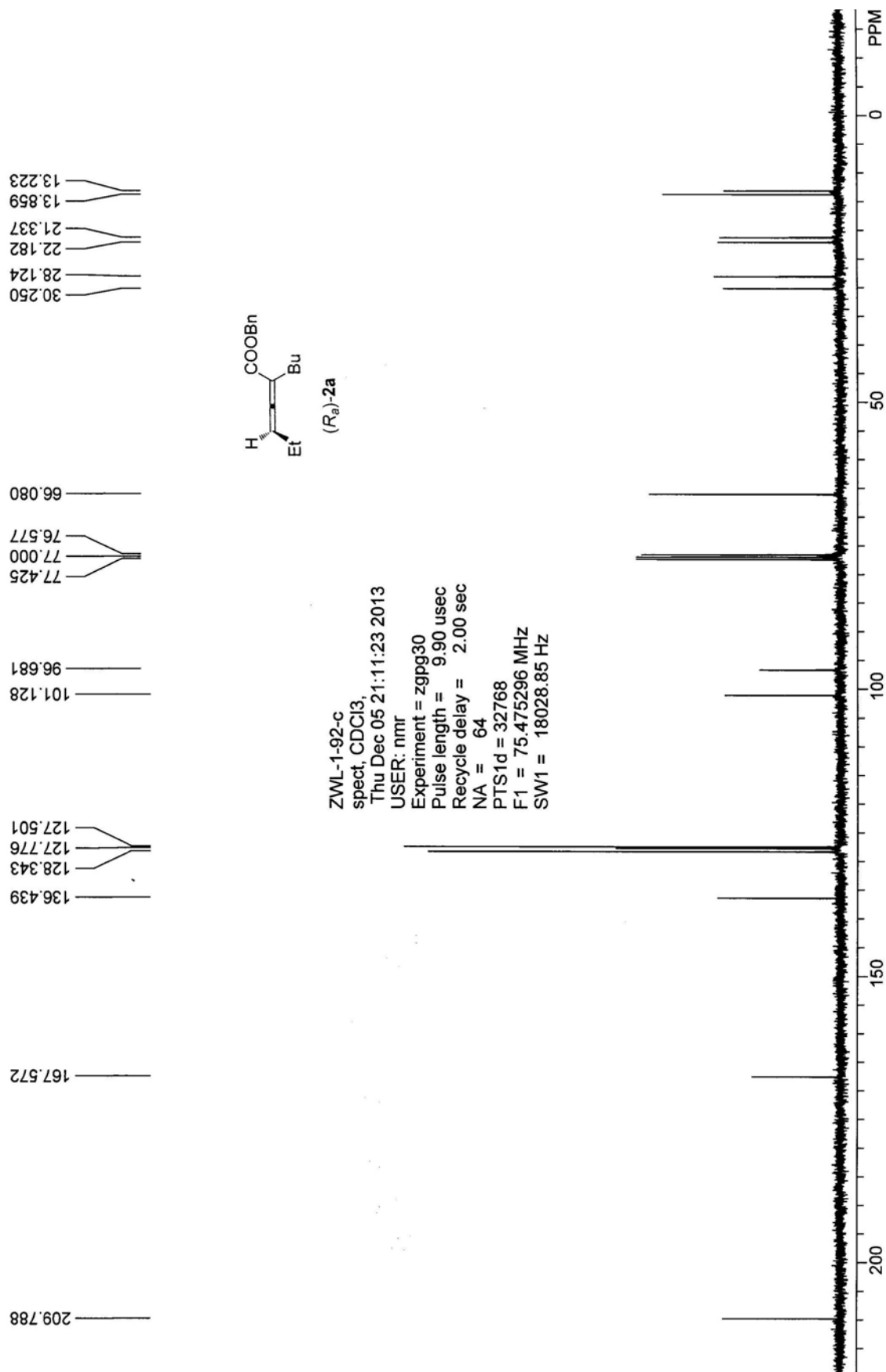
To a flame-dried Schlenk bottle (50 mL) were added  $[(\pi\text{-allyl})\text{PdCl}]_2$  (3.8 mg, 0.01 mmol) and **(*R*)-ECNU-Phos** (33.3 mg, 0.04 mmol). After addition of each of chemical, the bottle was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then freshly distilled toluene (2 mL) was added under argon. The resulting mixture was stirred for 1 hour at room temperature, which was followed by the addition of anhydrous LiF (28.5 mg, 1.1 mmol) (kept in a glove box) and ( $\pm$ )-**1a** (302.6 mg, 1.0 mmol)/toluene (3 mL) sequentially. The mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO (about 1 L) for three times. Then the resulting mixture was stirred at 25 °C for 24 h. After that, the resulting mixture was diluted with 20 mL of Et<sub>2</sub>O, washed with brine (20 mL), and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The resulting mixture was filtered through a short pad of silica gel (1.5 cm) eluted with Et<sub>2</sub>O (20 mL), and concentrated. The residue was purified by chromatography on silica gel to afford (*R<sub>a</sub>*)-benzyl 2-butyl-2,3-octadienoate (126.8 mg, 44%; 37% recovery determined by <sup>1</sup>H NMR spectrum using 1,3,5-trimethylbenzene as internal standard) as an oil [eluent: petroleum ether (b.p. 60-90 °C)/ethyl ether = 250/1]: 87% ee (Regis (*S,S*) Whelk-O column, hexane/*i*-PrOH = 200/1, 1.0 mL/min,  $\lambda$  = 214 nm,  $t_R$ (minor) = 12.5 min,  $t_R$ (major) = 14.6 min);  $[\alpha]_D^{30}$  = -21.4 ( $c$  = 1.10, CHCl<sub>3</sub>); <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.42-7.23 (m, 5 H, Ar-H), 5.56-5.46 (m, 1 H, =CH), 5.21 (d,  $J$  = 12.6 Hz, 1 H, one proton from Bn), 5.14 (d,  $J$  = 12.6 Hz, 1 H, one proton from Bn), 2.34-2.15 (m, 2 H, CH<sub>2</sub>), 2.10 (q,  $J$  = 7.0 Hz, 2 H, CH<sub>2</sub>), 1.50-1.24 (m, 8 H, 4 x CH<sub>2</sub>), 0.90 (t,  $J$  = 7.1 Hz, 3 H, CH<sub>3</sub>), 0.87 (t,  $J$  = 7.2 Hz, 3 H, CH<sub>3</sub>); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>):  $\delta$  = 210.0, 167.6, 136.4, 128.3, 127.8, 127.6, 100.4, 95.0, 66.1, 31.0, 30.2, 28.1, 27.7, 22.2, 22.0, 13.9, 13.8; MS (ESI  $m/z$ ): 341

$(M+Na+MeOH)^+$ , 309  $(M+Na)^+$ ; IR (neat):  $\nu = 2970, 2925, 2903, 1957, 1711, 1262, 1054\text{ cm}^{-1}$ ; HRMS calcd. for  $C_{19}H_{26}O_2$   $[M^+]$ : 286.1933, found: 286.1932.

Reference:

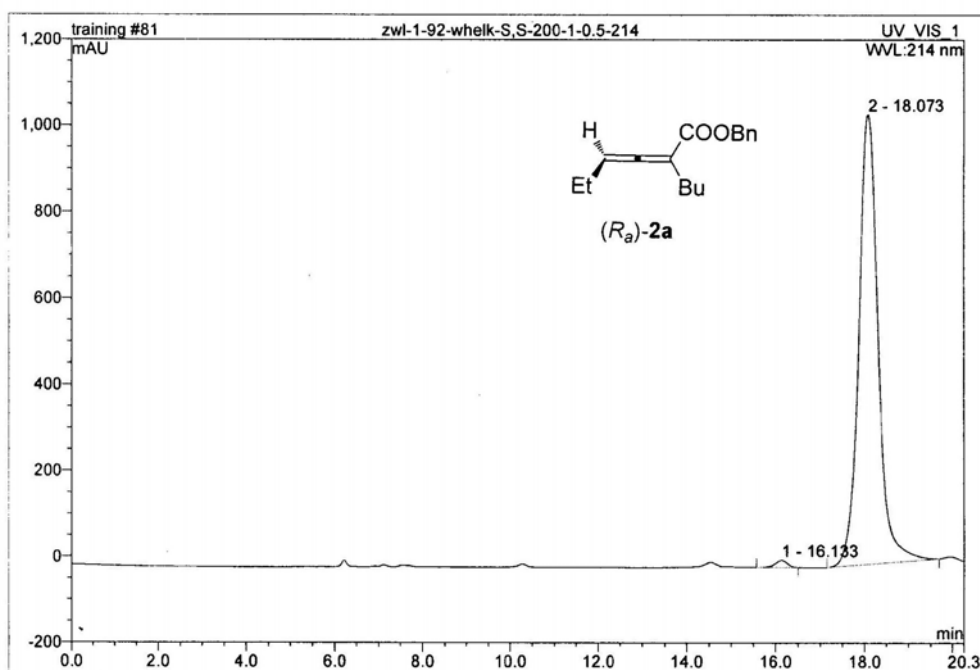
1. Wang, Y.; Ma, S. *Adv. Synth. Catal.* 2013, 355, 741.





**81 zwl-1-92-wheelk-S,S-200-1-0.5-214**

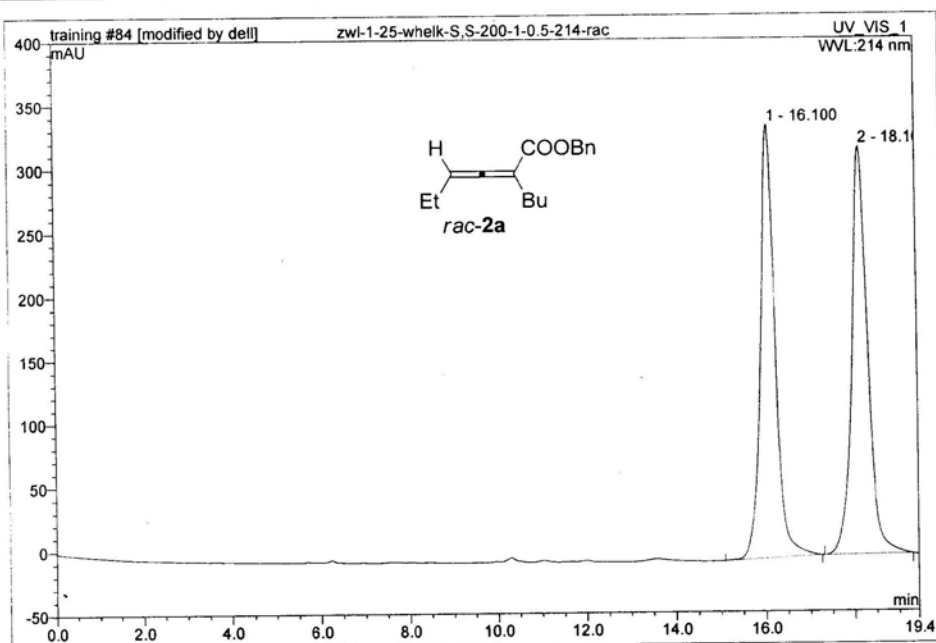
Sample Name:	zw1-1-92-wheelk-S,S-200-1-0.5-214	Injection Volume:	20.0
Vial Number:	81	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	zw1-allenoate	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2013-3-16 14:56	Sample Weight:	1.0000
Run Time (min):	20.27	Sample Amount:	1.0000



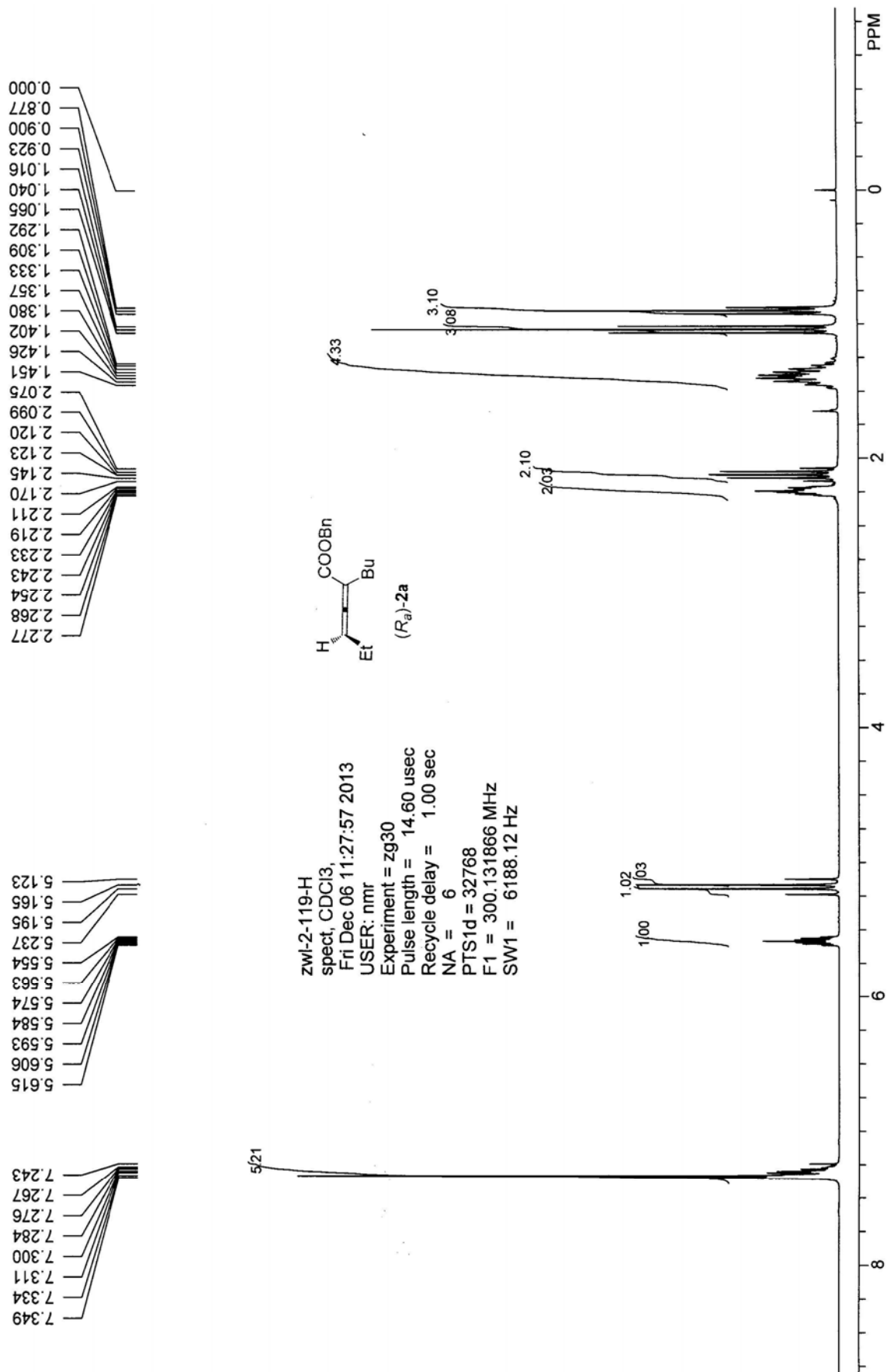
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	16.13	n.a.	16.606	5.302	1.00	n.a.	BMB
2	18.07	n.a.	1042.303	526.108	99.00	n.a.	BMB
Total:			1058.909	531.410	100.00	0.000	

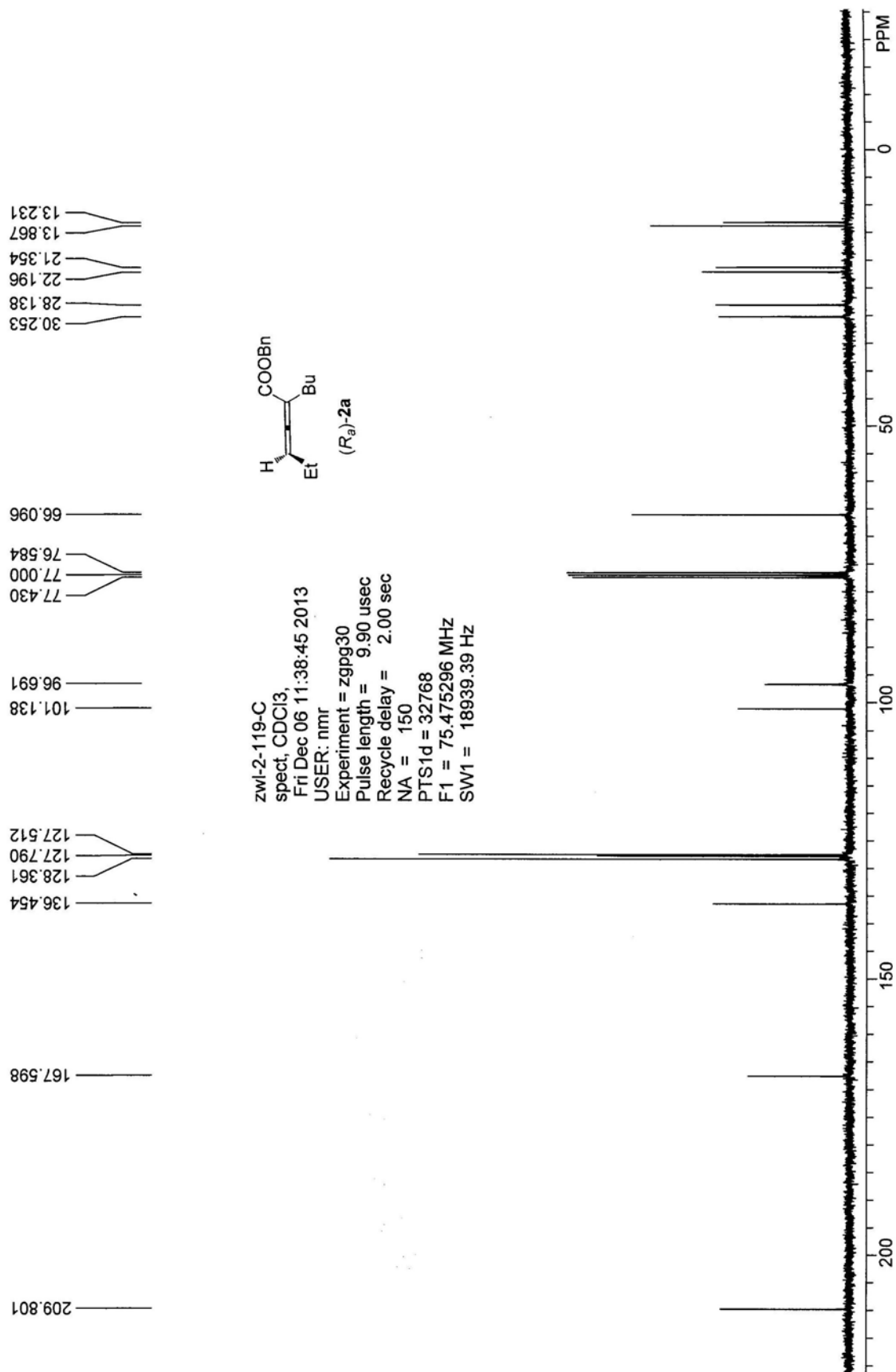
**84 zwl-1-25-whelk-S,S-200-1-0.5-214-rac**

Sample Name:	zwl-1-25-whelk-S,S-200-1-0.5-214-rac	Injection Volume:	20.0
Vial Number:	84	Channel:	UV_VIS_1
Sample Type:	unknown	Wavelength:	214
Control Program:	zwl-allenoate	Bandwidth:	n.a.
Quantif. Method:	test	Dilution Factor:	1.0000
Recording Time:	2013-3-16 15:59	Sample Weight:	1.0000
Run Time (min):	19.44	Sample Amount:	1.0000



No.	Ret. Time min	Peak Name	Height mAU	Area mAU*min	Rel. Area %	Amount	Type
1	16.10	n.a.	340.382	129.324	49.69	n.a.	BMB*
2	18.16	n.a.	320.346	130.924	50.31	n.a.	BMB*
Total:			660.728	260.248	100.00	0.000	



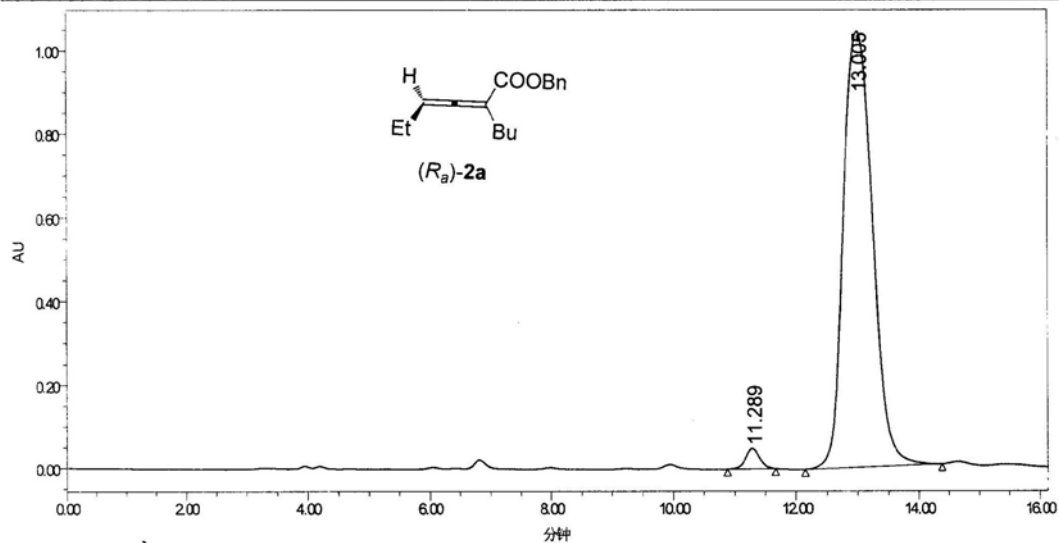




## SAMPLE INFORMATION

Sample Name: zw-2-119-whale-200-1-1-214  
Sample Type: 未知  
Vial: 1  
Injection #: 17  
Injection Volume: 10.00  $\mu$ l  
Run Time: 20.00 Minutes

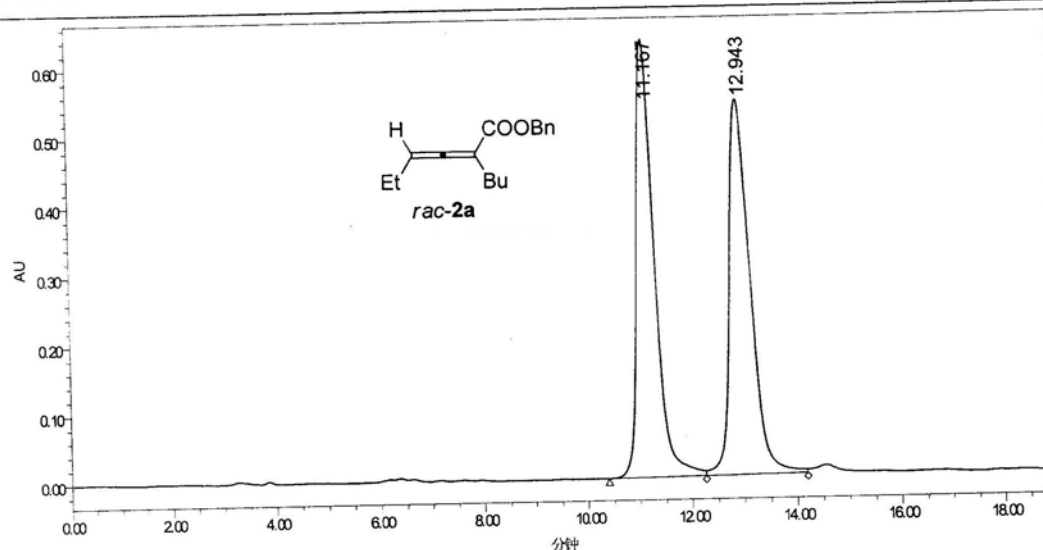
Acquired By: Breeze  
Date Acquired: 2013/10/23 13:05:06 CST  
Acq. Method: zg100  
Date Processed: 2013/10/23 13:40:29 CST  
Channel Name: V2489 CHA  
Sample Set Name:



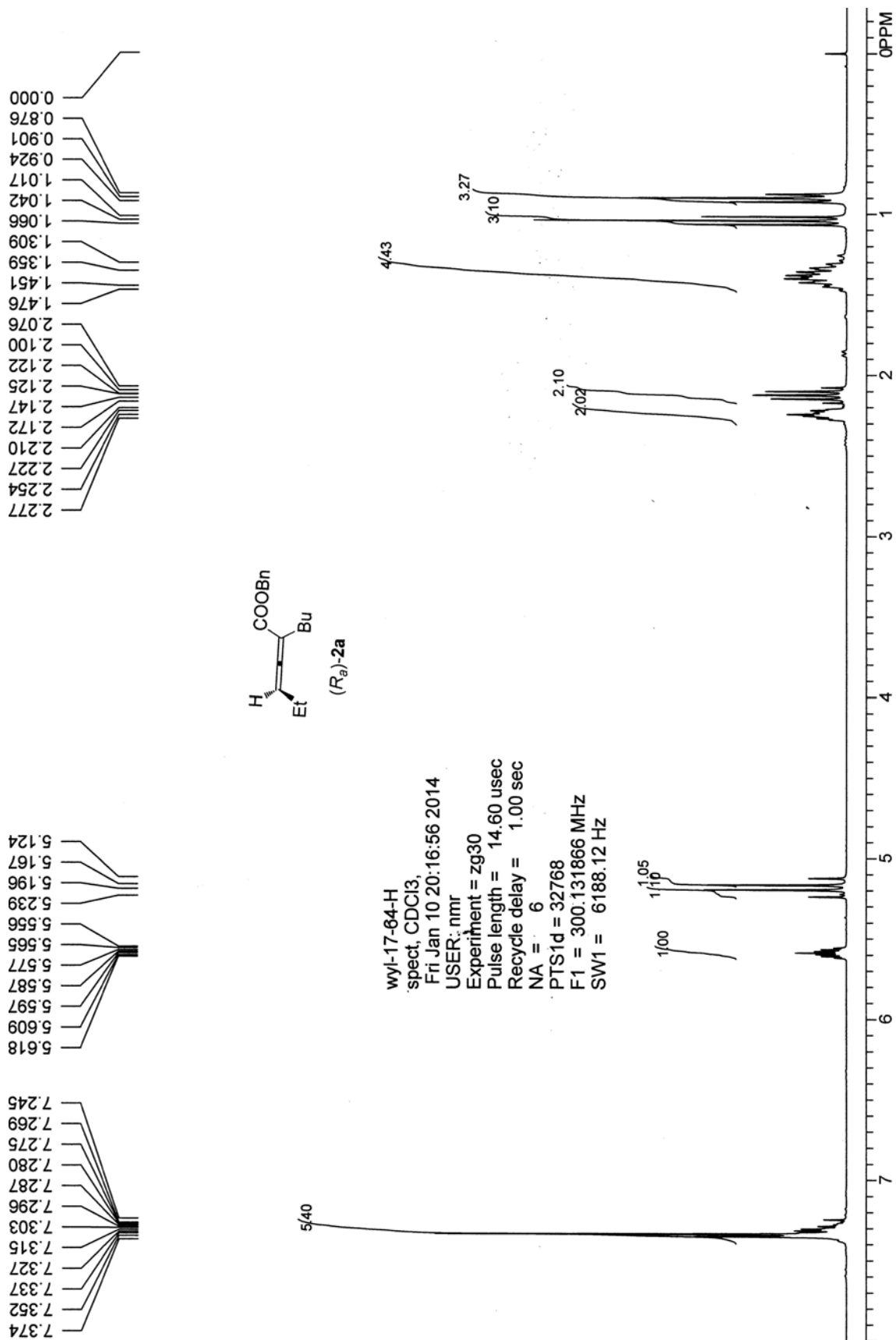
	RT (min)	Area (AUsec)	%Area	Height (AU)	% Height
1	11.289	783852	2.21	49414	4.52
2	13.008	34655380	97.79	1044699	95.48

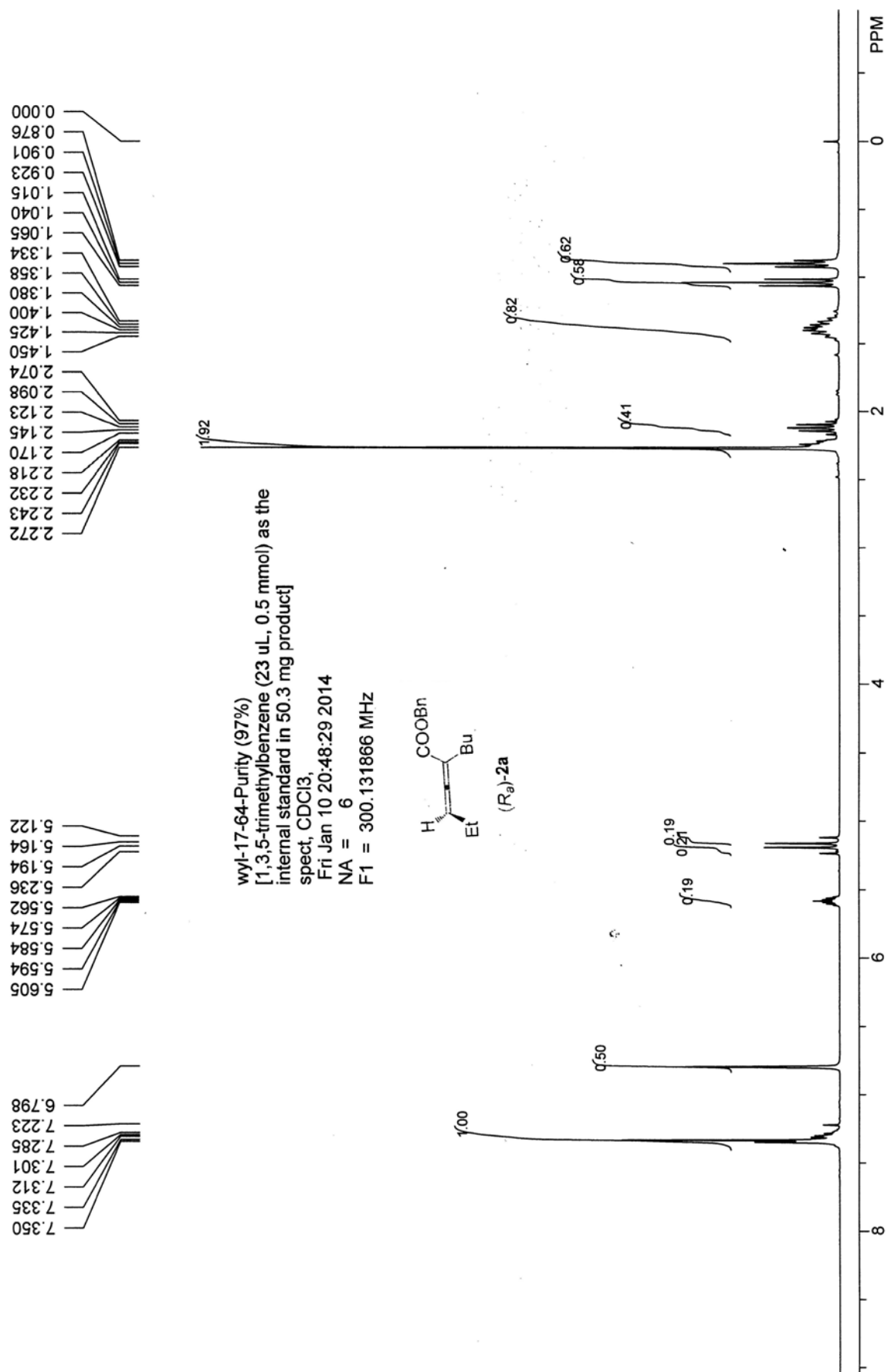
# SAMPLE INFORMATION

Sample Name:	zw-1-25-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/10/23 12:06:48 CST
Vial:	1	Acq. Method:	zgj100
Injection #:	14	Date Processed:	2013/10/23 13:39:27 CST
Injection Volume:	10.00 uL	Channel Name:	V2489 ChA
Run Time:	160.00 Minutes	Sample Set Name:	

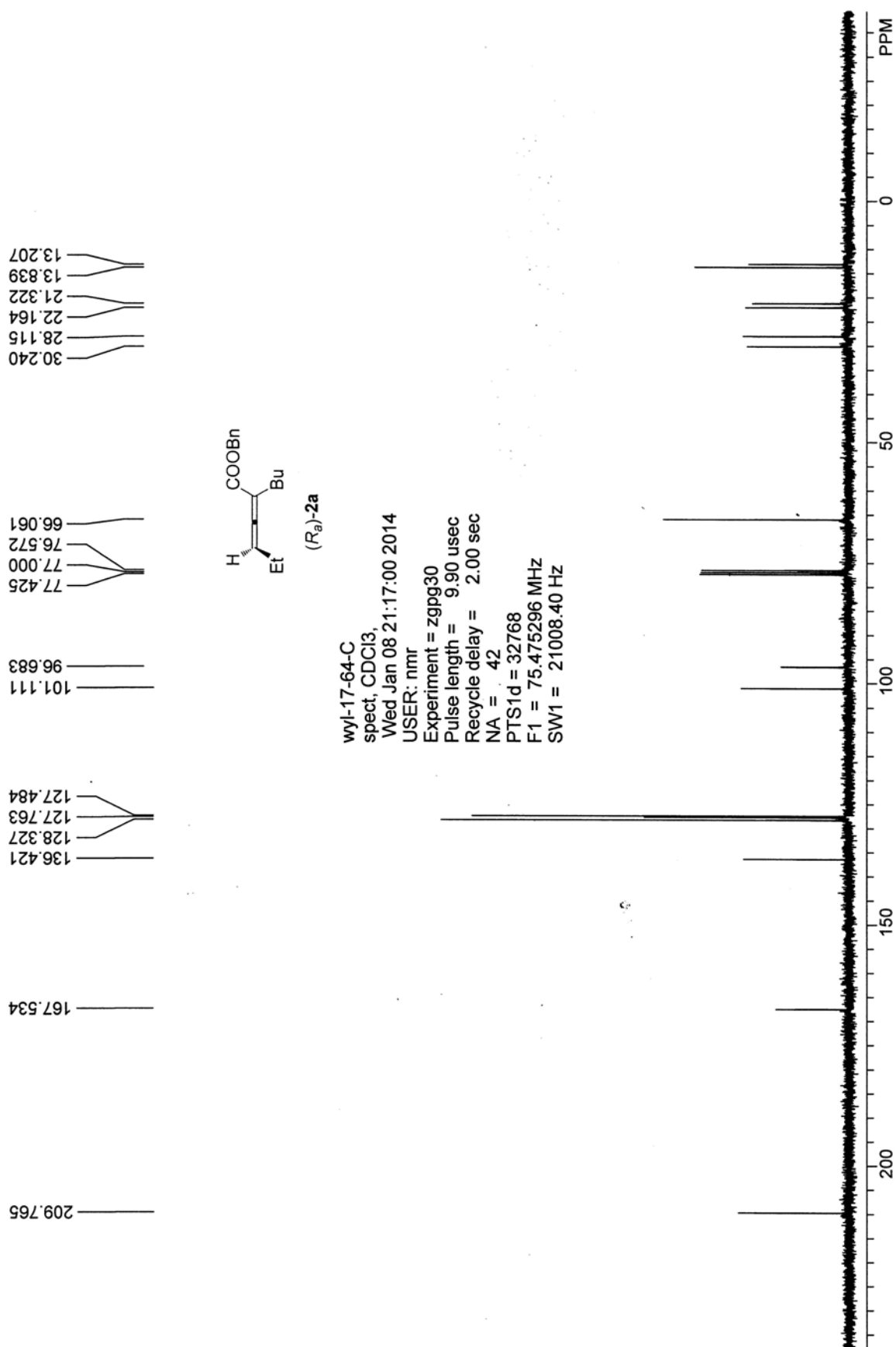


	RT (min)	Area (AUsec)	%Area	Height (AU)	% Height
1	11.167	15160646	50.03	635354	53.88
2	12.943	15142193	49.97	543942	46.12



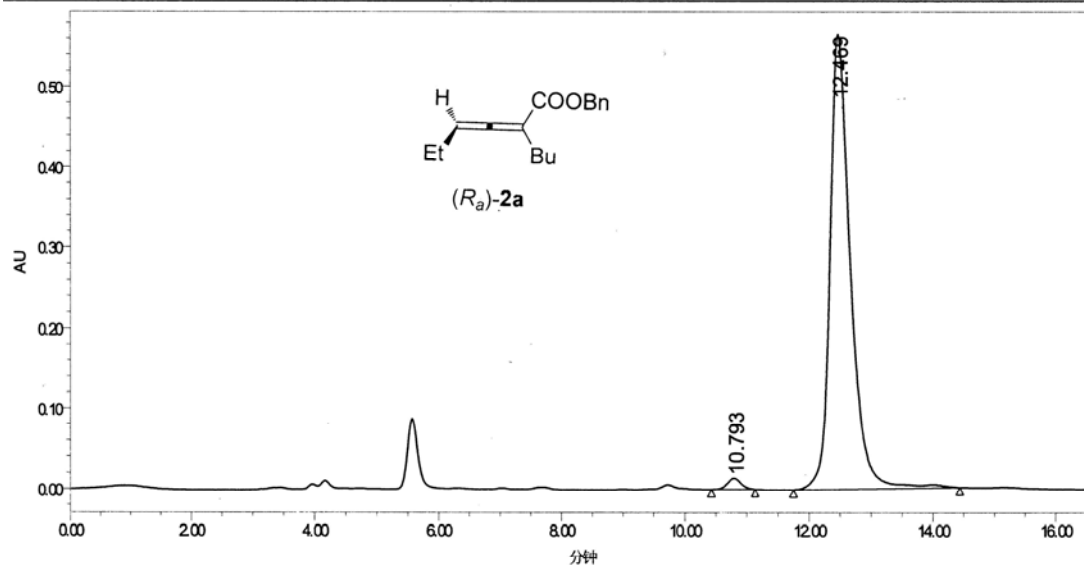


wyl-17-64-Purity (97%)  
 [1,3,5-trimethylbenzene (23  $\mu\text{L}$ , 0.5 mmol) as the  
 internal standard in 50.3 mg product]  
 spect,  $\text{CDCl}_3$ ,  
 Fri Jan 10 20:48:29 2014  
 NA = 6  
 F1 = 300.131866 MHz



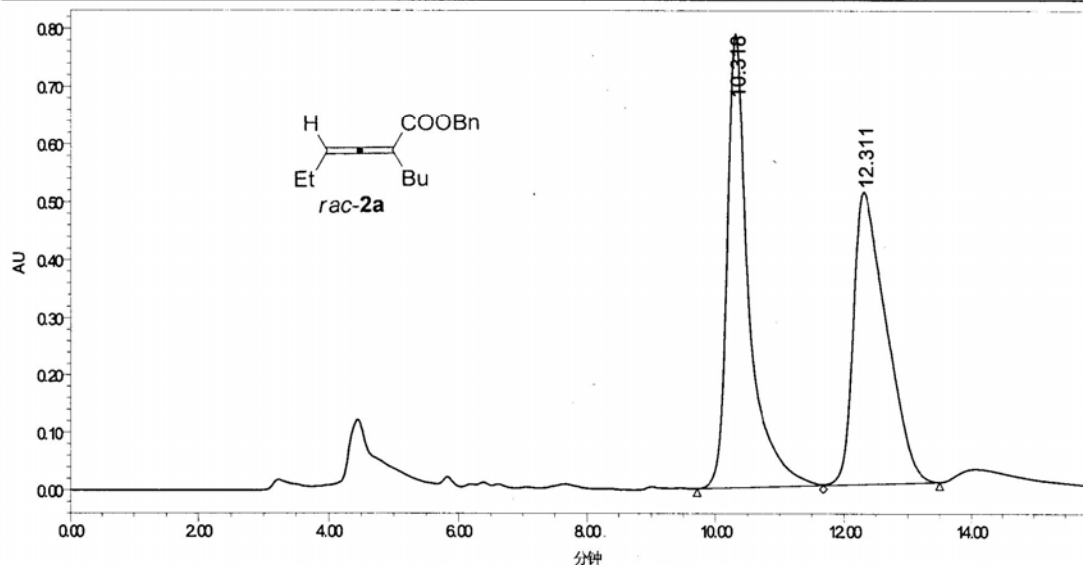
## SAMPLE INFORMATION

Sample Name:	wyl-17-64-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2014/1/3 14:57:26 CST
Vial:	1	Acq. Method:	zg100
Injection #:	22	Date Processed:	2014/1/3 16:48:20 CST
Injection Volume:	10.00 ul	Channel Name:	V2489 ChA
Run Time:	100.00 Minutes	Sample Set Name:	

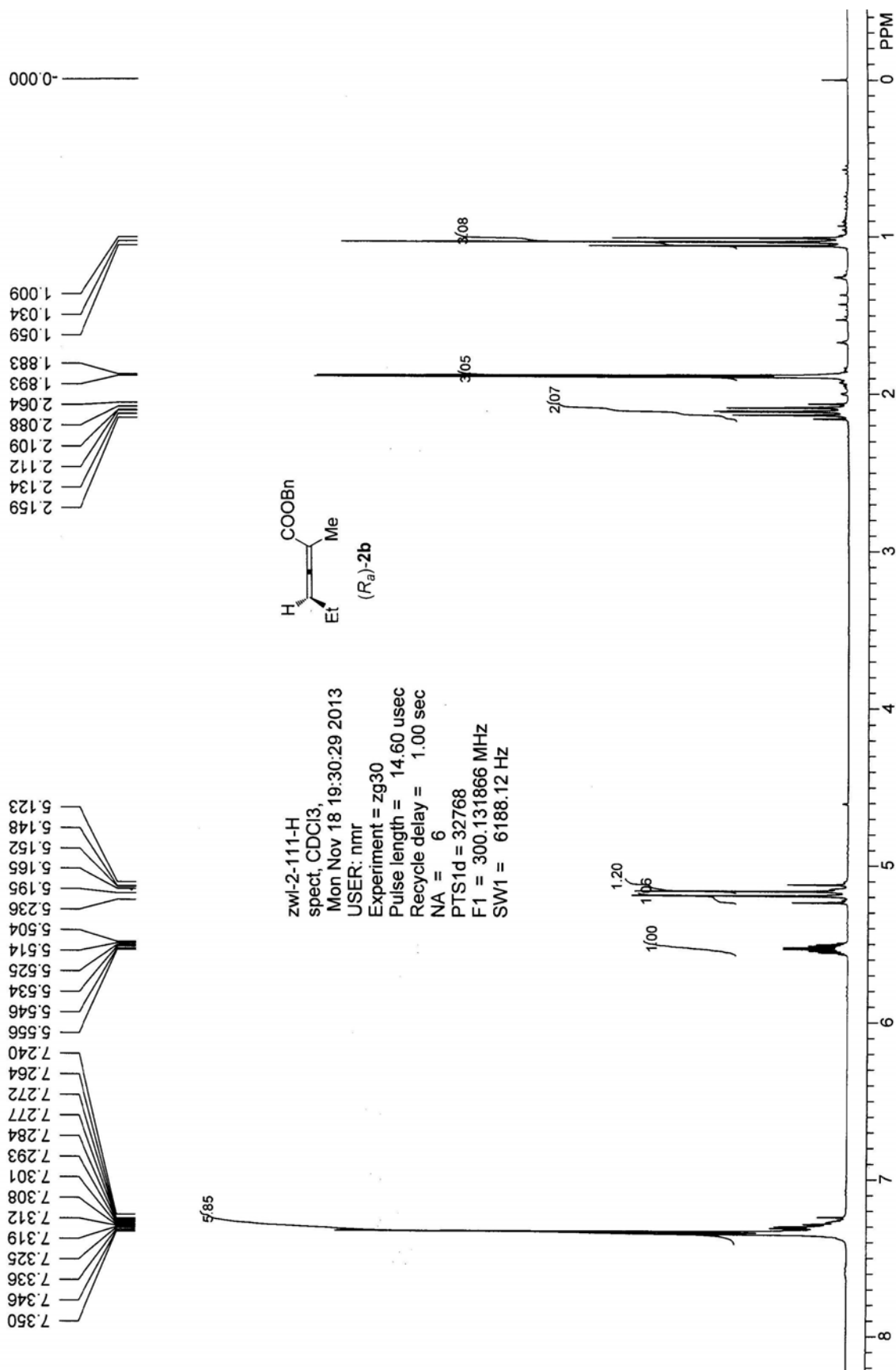


	RT (min)	Area (AUsec)	%Area	Height (AU)	% Height
1	10.793	217533	1.66	14194	2.45
2	12.466	12962557	98.35	566829	97.55

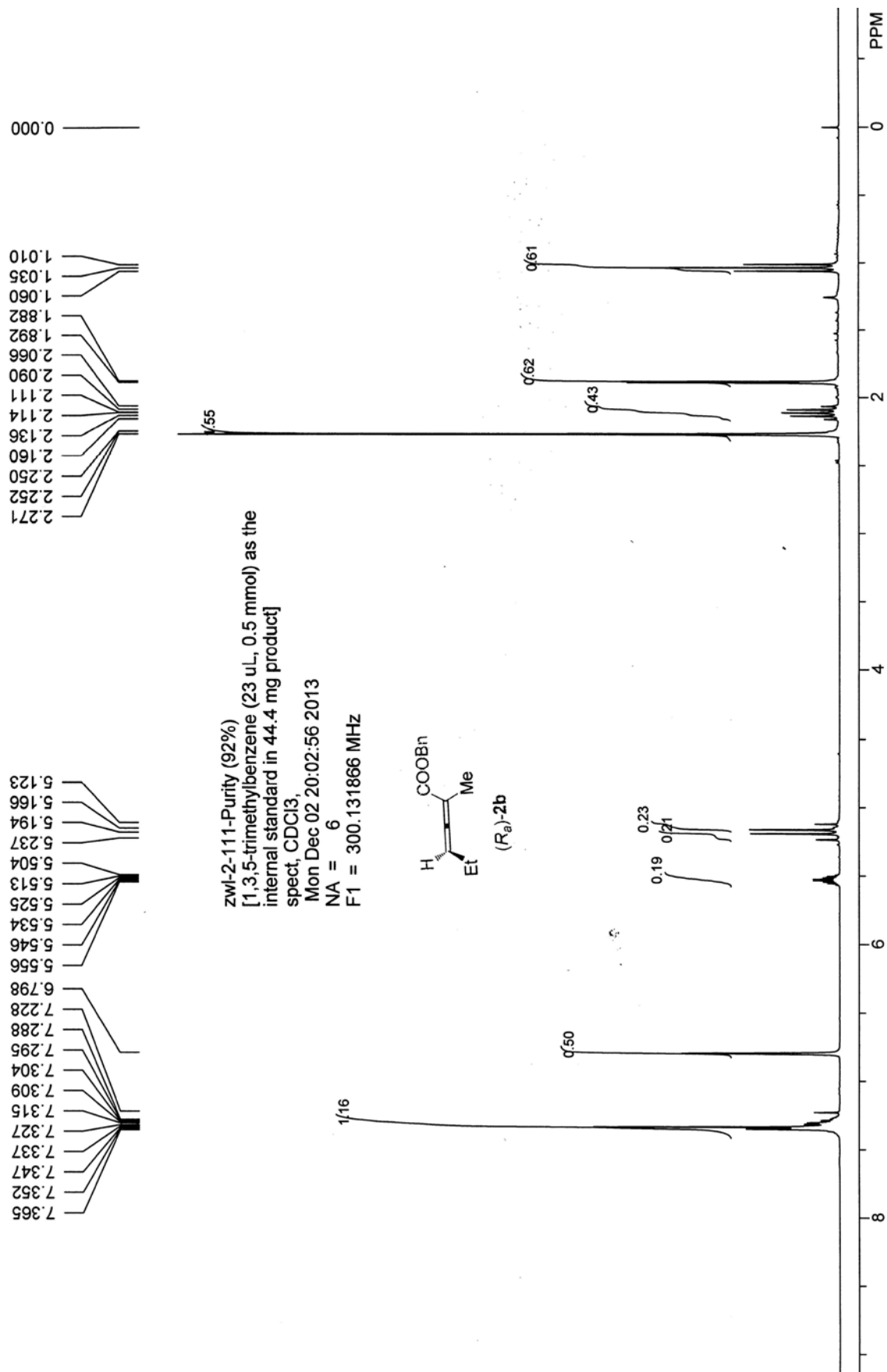
## SAMPLE INFORMATION

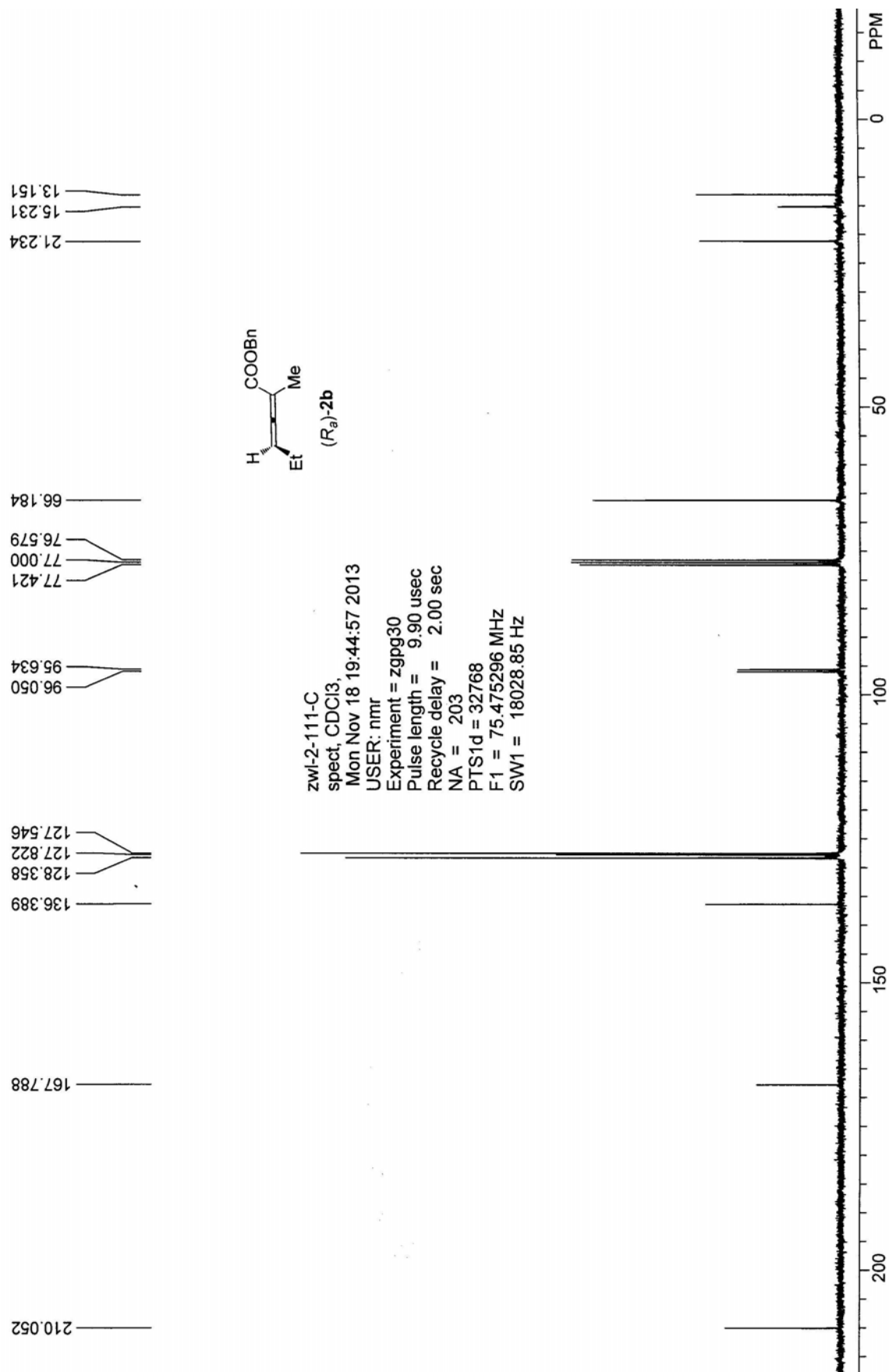
Sample Name: zwf-1-25-whale-200-1-1-214  
Sample Type: 未知  
Vial: 1  
Injection #: 24  
Injection Volume: 10.00  $\mu$ l  
Run Time: 100.00 MinutesAcquired By: Breeze  
Date Acquired: 2014/1/3 15:36:15 CST  
Acq. Method: zg100  
Date Processed: 2014/1/3 16:46:51 CST  
Channel Name: V0489 CHA  
Sample Set Name:

	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	10.311	18390438	50.21	786657	60.82
2	12.311	18237239	49.79	506832	39.18



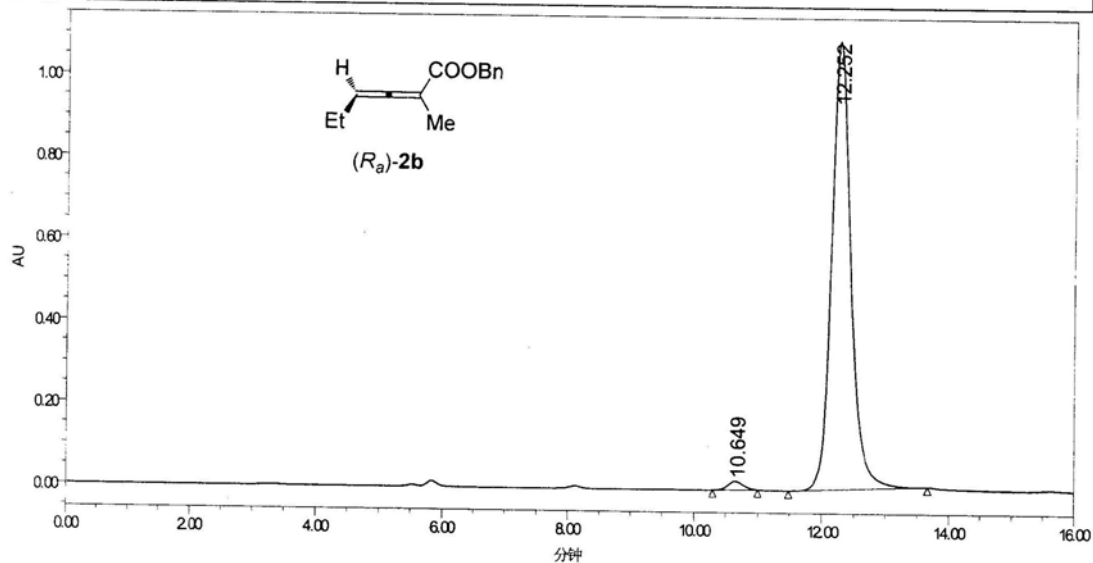






# SAMPLE INFORMATION

Sample Name:	zwf-2-111-whalk-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/10/17 11:02:23 CST
Vial:	1	Acq. Method:	zg100
Injection #:	54	Date Processed:	2013/10/17 17:18:59 CST
Injection Volume:	10.00 $\mu$ l	Channel Name:	V0489 ChA
Run Time:	16.00 Minutes	Sample Set Name:	



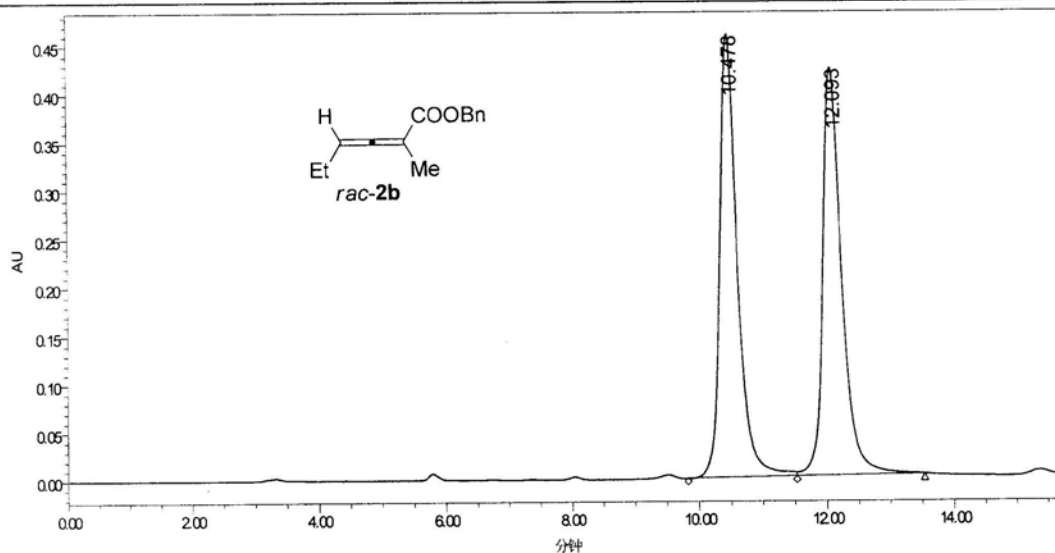
	RT (min)	Area (sec)	%Area	Height (mm)	% Height
1	10.649	361224	1.56	21320	1.92
2	12.252	22866192	98.44	1091875	98.08

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

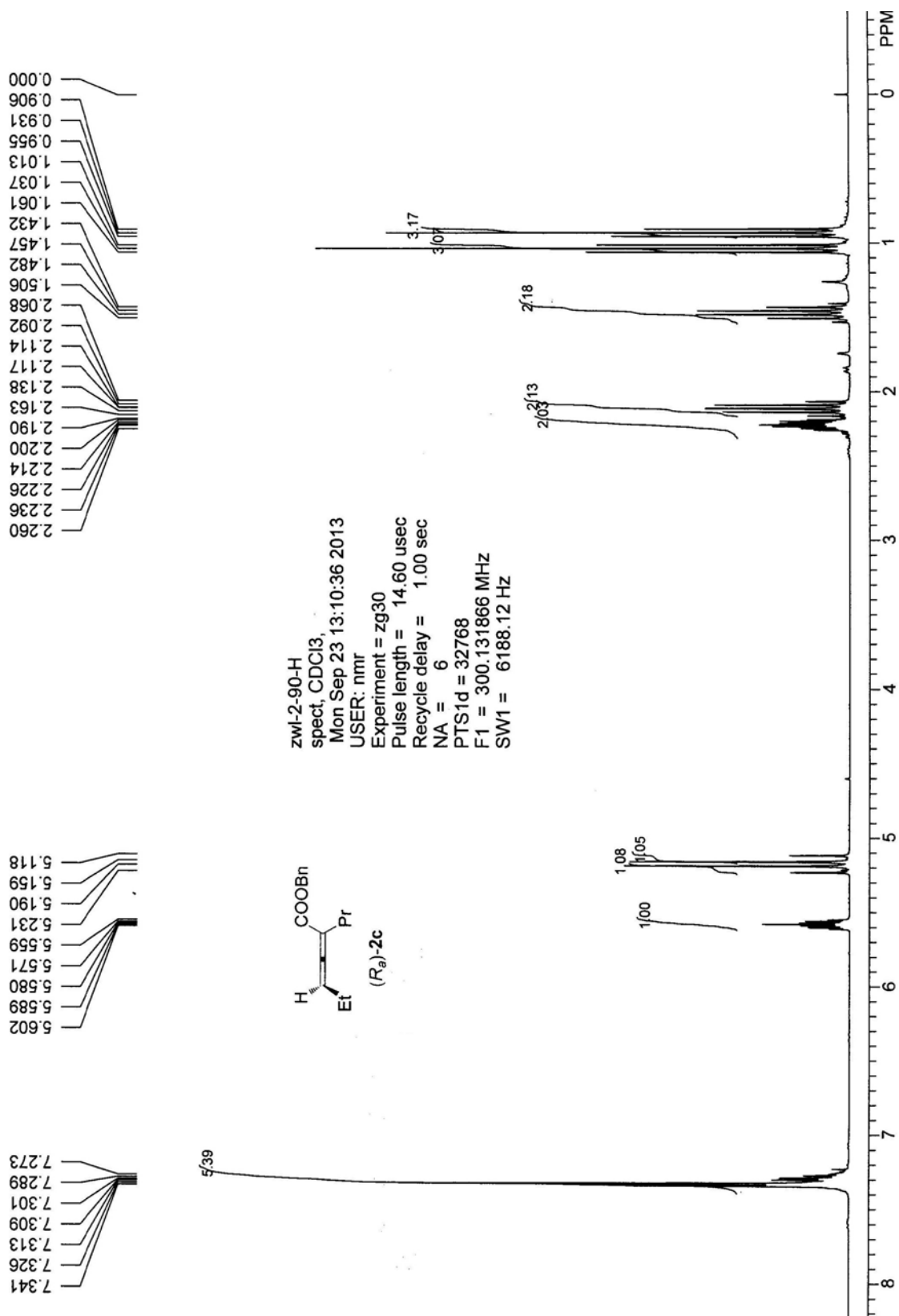


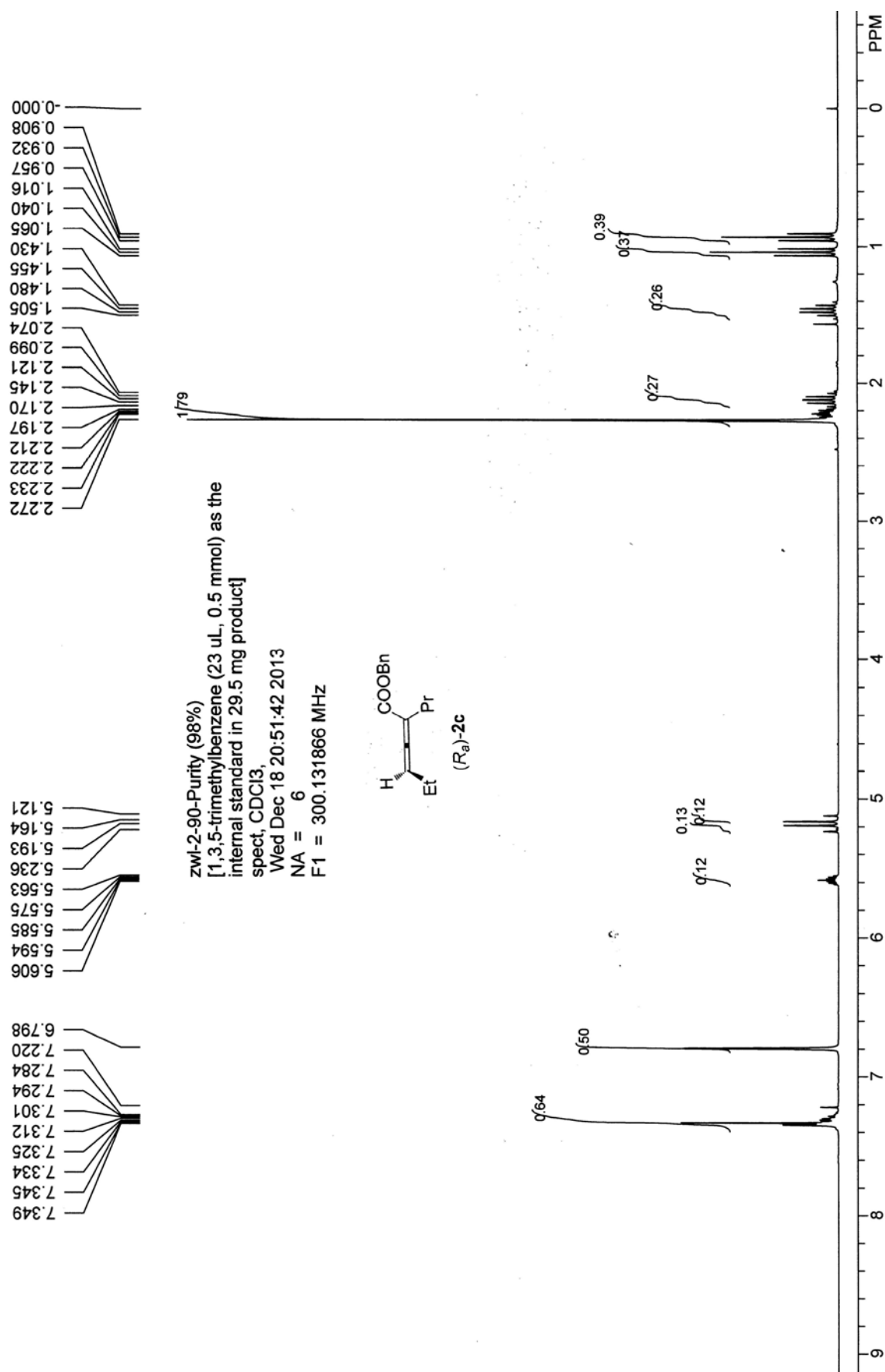
# SAMPLE INFORMATION

Sample Name:	zwf-2-110-whisk-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/10/17 10:44:03 CST
Vial:	1	Acq. Method:	zgj100
Injection #:	52	Date Processed:	2013/10/17 17:18:23 CST
Injection Volume:	10.00 uL	Channel Name:	V2489 CHA
Run Time:	35.00 Minutes	Sample Set Name:	



	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	10.478	8747774	50.18	459668	52.08
2	12.093	8686717	49.82	422981	47.92





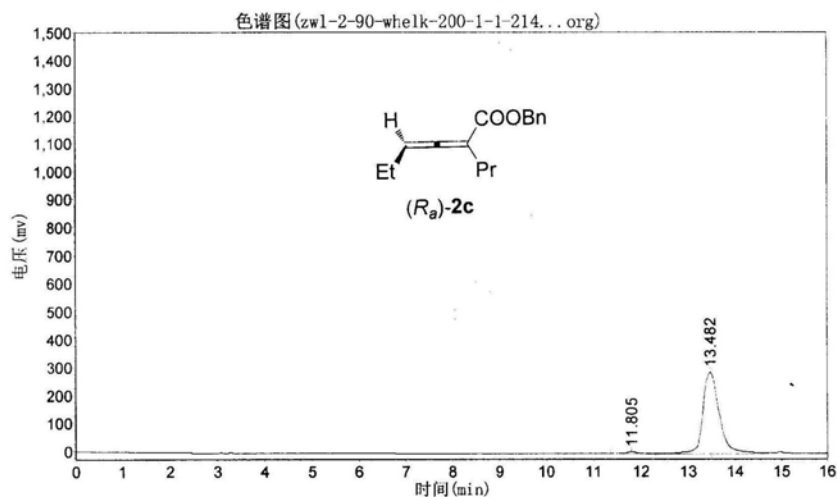
# zw1-2-90-whelk-200-1-1-214

实验时间: 2013/9/24, 10:59:21

报告时间: 2013/9/24, 11:25:35

谱图文件:D:\zhuguangjiong\zw1\20130924\zw1-2-90-whelk-200-1-1-214...org

实验内容简介:



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.805	5360.698	89576.852	1.2682
2		13.482	291471.531	6973487.500	98.7318
总计			296832.229	7063064.352	100.0000

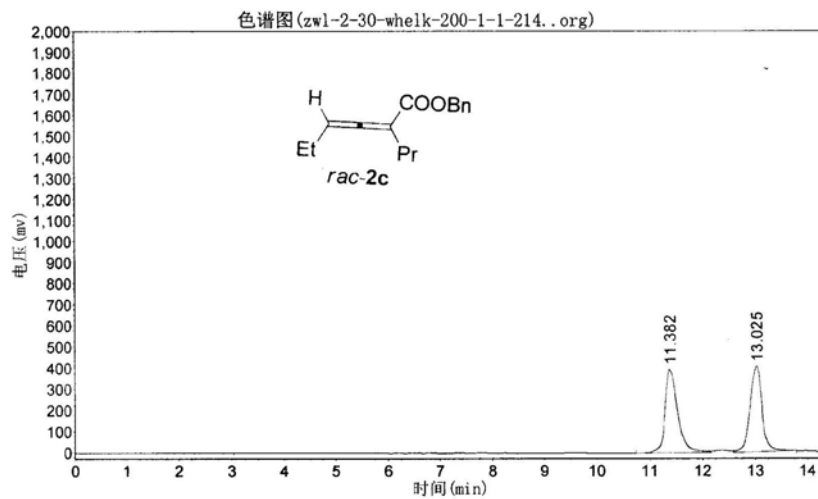
# zw1-2-30-wheelk-200-1-1-214

实验时间: 2013/9/24, 10:26:45

报告时间: 2013/9/24, 11:26:41

谱图文件:D:\zhuguangjiong\zw1\20130924\zw1-2-30-wheelk-200-1-1-214..org

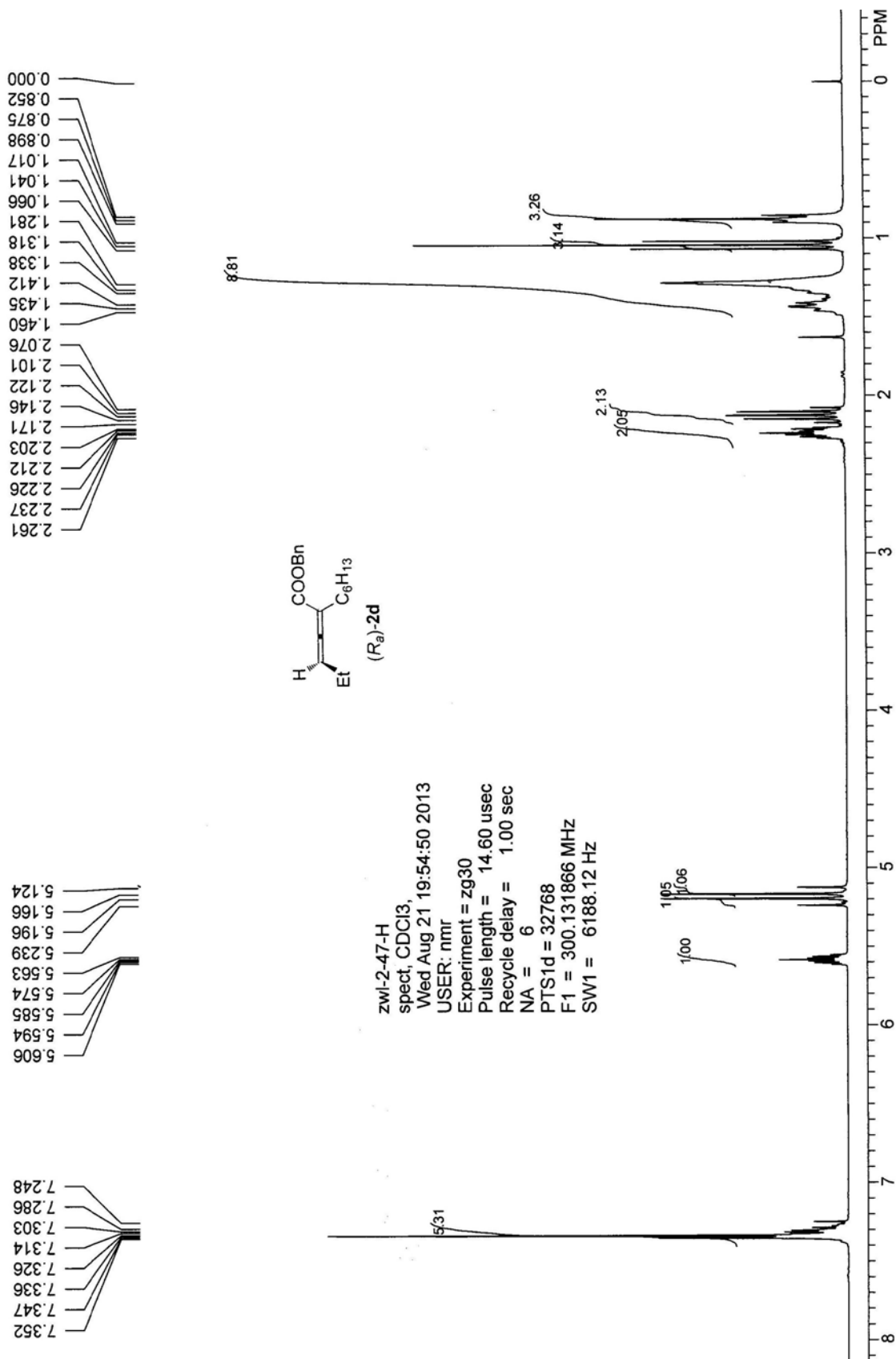
实验内容简介:

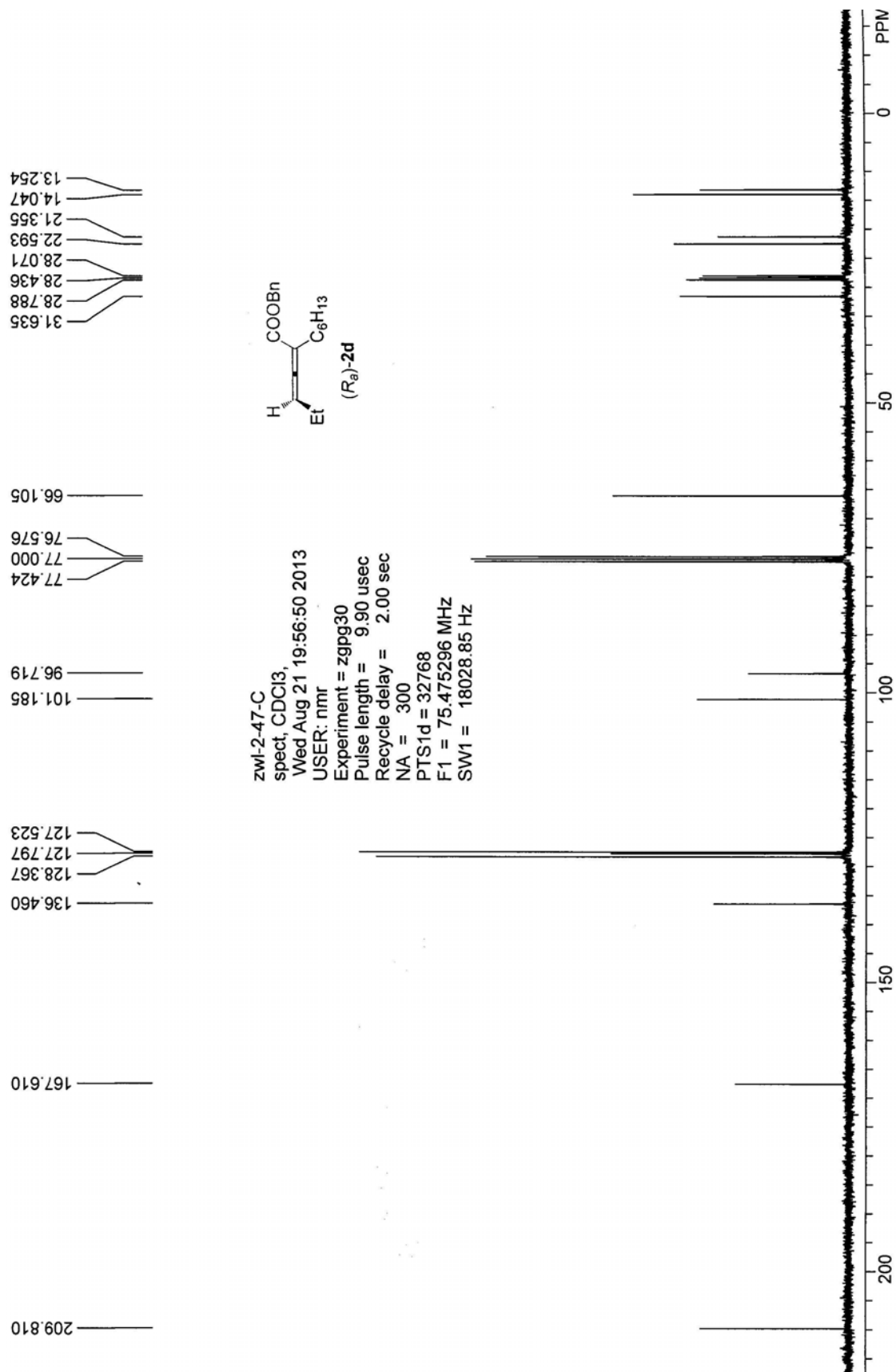


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.382	391553.875	6489409.500	50.2369
2		13.025	403771.500	6428203.500	49.7631
总计			795325.375	12917613.000	100.0000

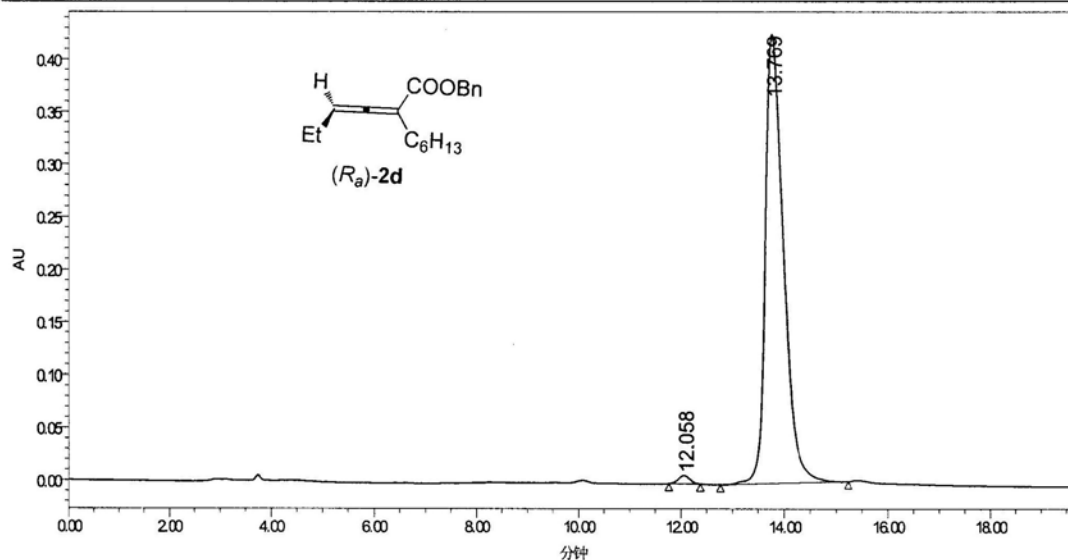






# SAMPLE INFORMATION

Sample Name:	zw-2-47-whale-s,s-200-1-0.5-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/9/3 15:12:23 CST
Val:	1	Acq Method:	zg100
Injection #:	19	Date Processed:	2013/9/3 15:51:22 CST
Injection Volume:	10.00 ul	Channel Name:	V2489 ChA
Run Time:	35.00 Minutes	Sample Set Name:	

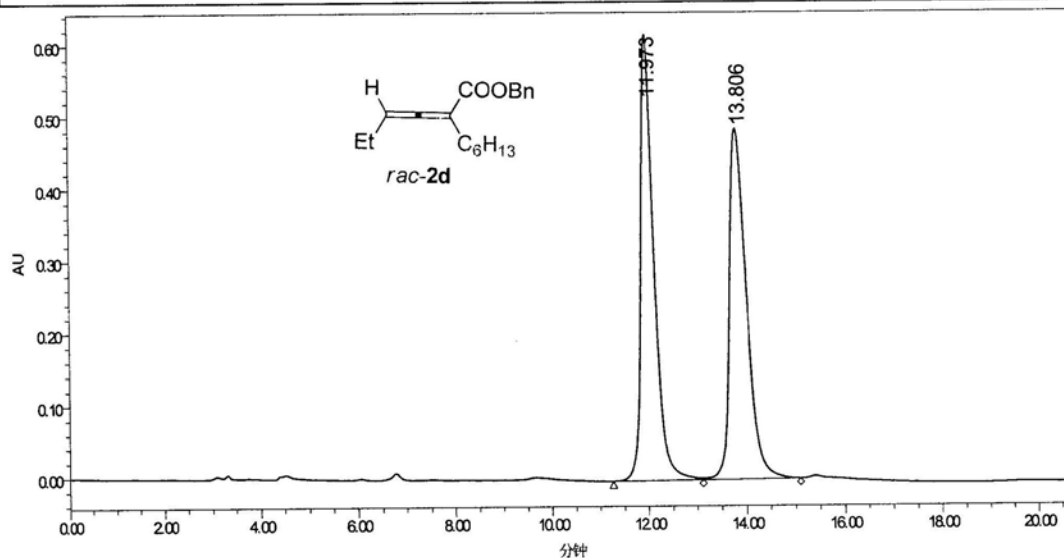


	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	12.058	125130	1.20	8004	1.84
2	13.760	10323491	98.80	426883	98.16

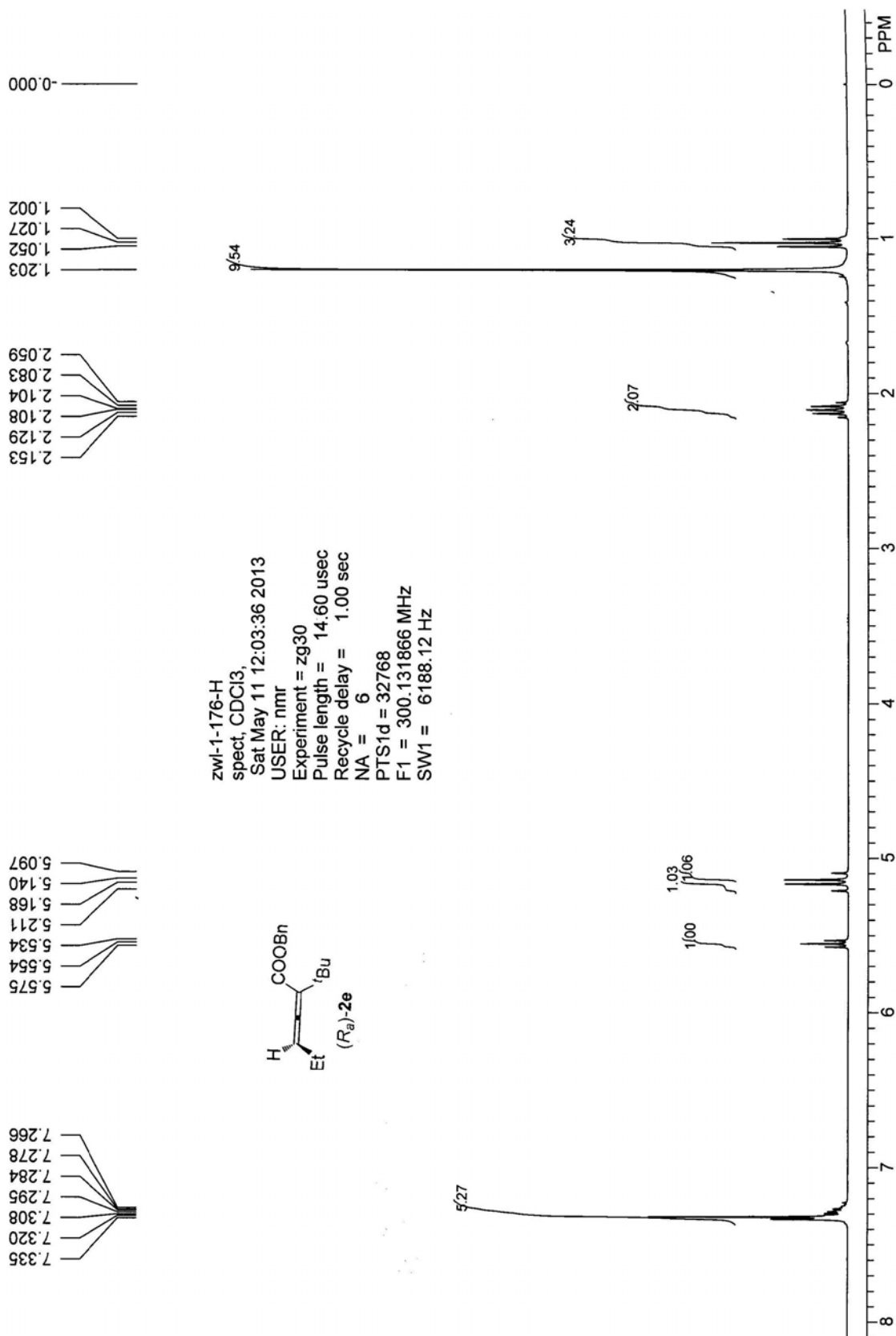
## SAMPLE INFORMATION

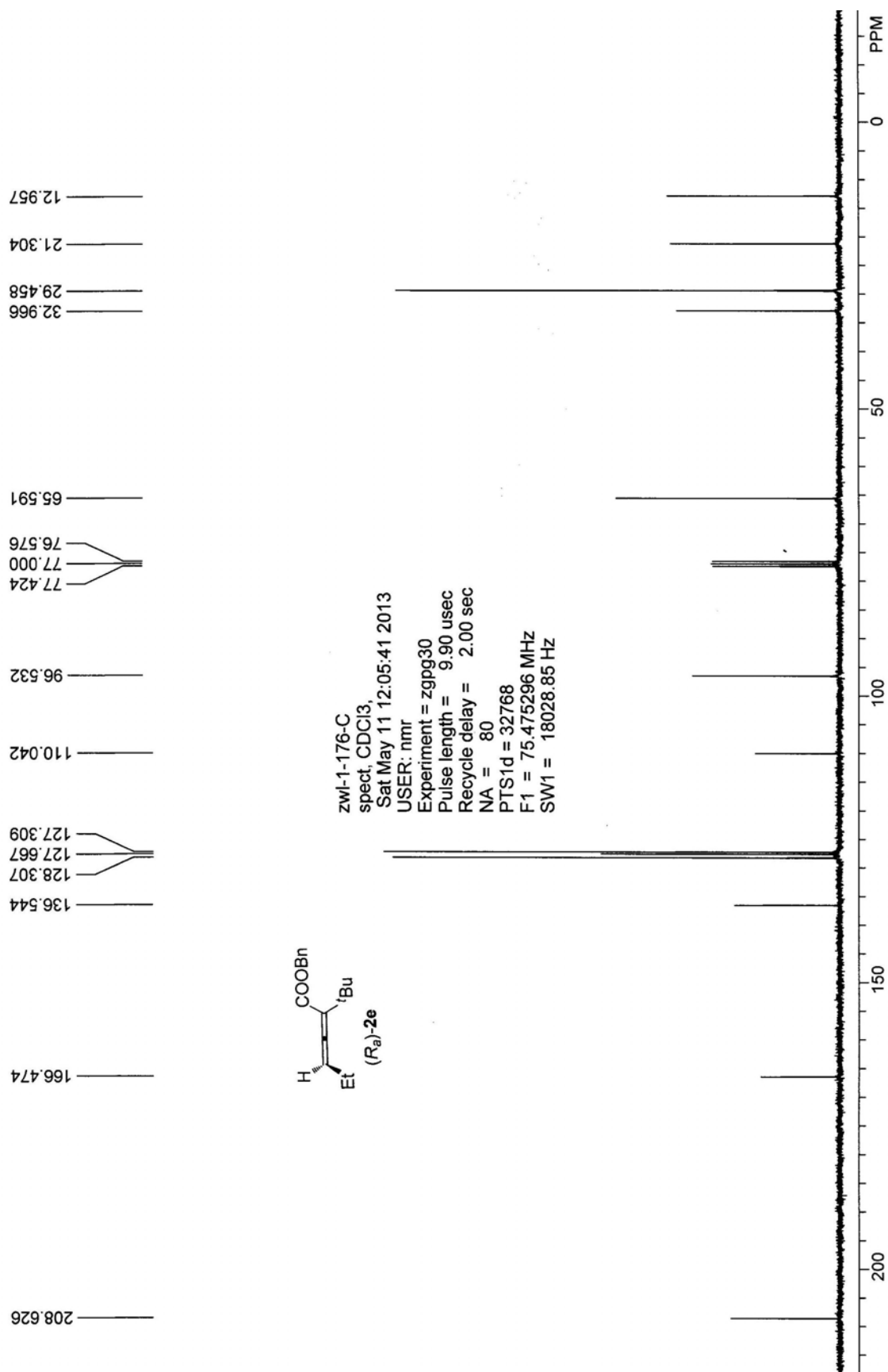
Sample Name: zwf-2-5-whetk-s-200-1-0.5-214  
Sample Type: 未知  
Val: 1  
Injection #: 16  
Injection Volume: 10.00  $\mu$ l  
Run Time: 35.00 Minutes

Acquired By: Breeze  
Date Acquired: 2013/9/3 14:27:39 CST  
Acq. Method: zig100  
Date Processed: 2013/9/3 15:52:21 CST  
Channel Name: V2489 ChA  
Sample Set Name:



	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	11.973	1195267	50.31	620860	56.04
2	13.806	1180306	49.69	486963	43.96





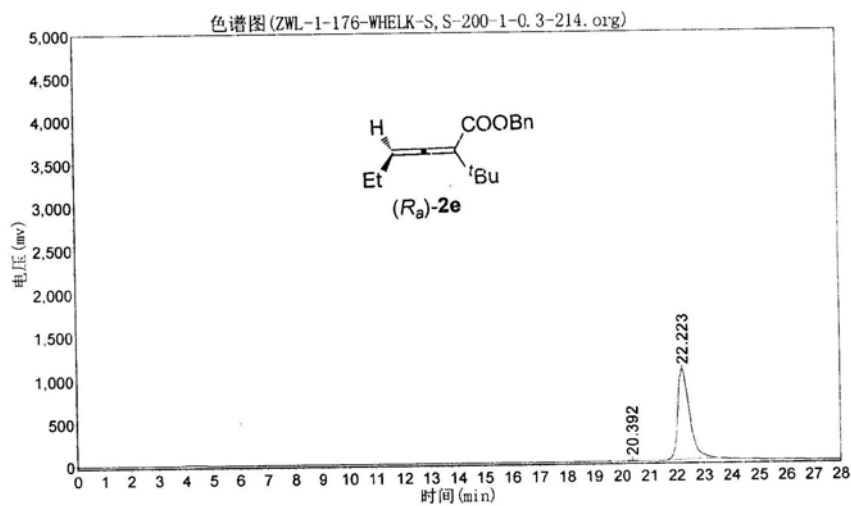
# zwl-1-176-whelk-s, s-200-1-0.3-214

实验时间: 2013-05-15, 9:40:01

报告时间: 2013-05-15, 16:31:07

谱图文件: D:\zhuguangjiong\zwl\20130515\ZWL-1-176-WHELK-S, S-200-1-0.3-214.org

实验内容简介:  
whelk-s, s 200+1  
0.3ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.392	8943.304	230134.391	0.7233
2		22.223	1052257.500	31587076.000	99.2767
总计			1061200.804	31817210.391	100.0000

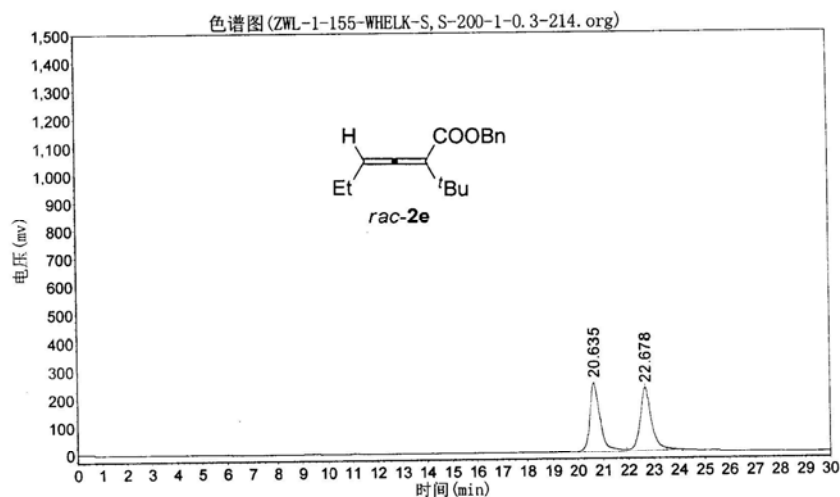
# zwl-1-155-whelk-s, s-200-1-0.3-214

实验时间: 2013-05-15, 9:02:14

报告时间: 2013-05-15, 16:29:23

谱图文件: D:\zhuguangjiong\zwl\20130515\ZWL-1-155-WHELK-S, S-200-1-0.3-214.org

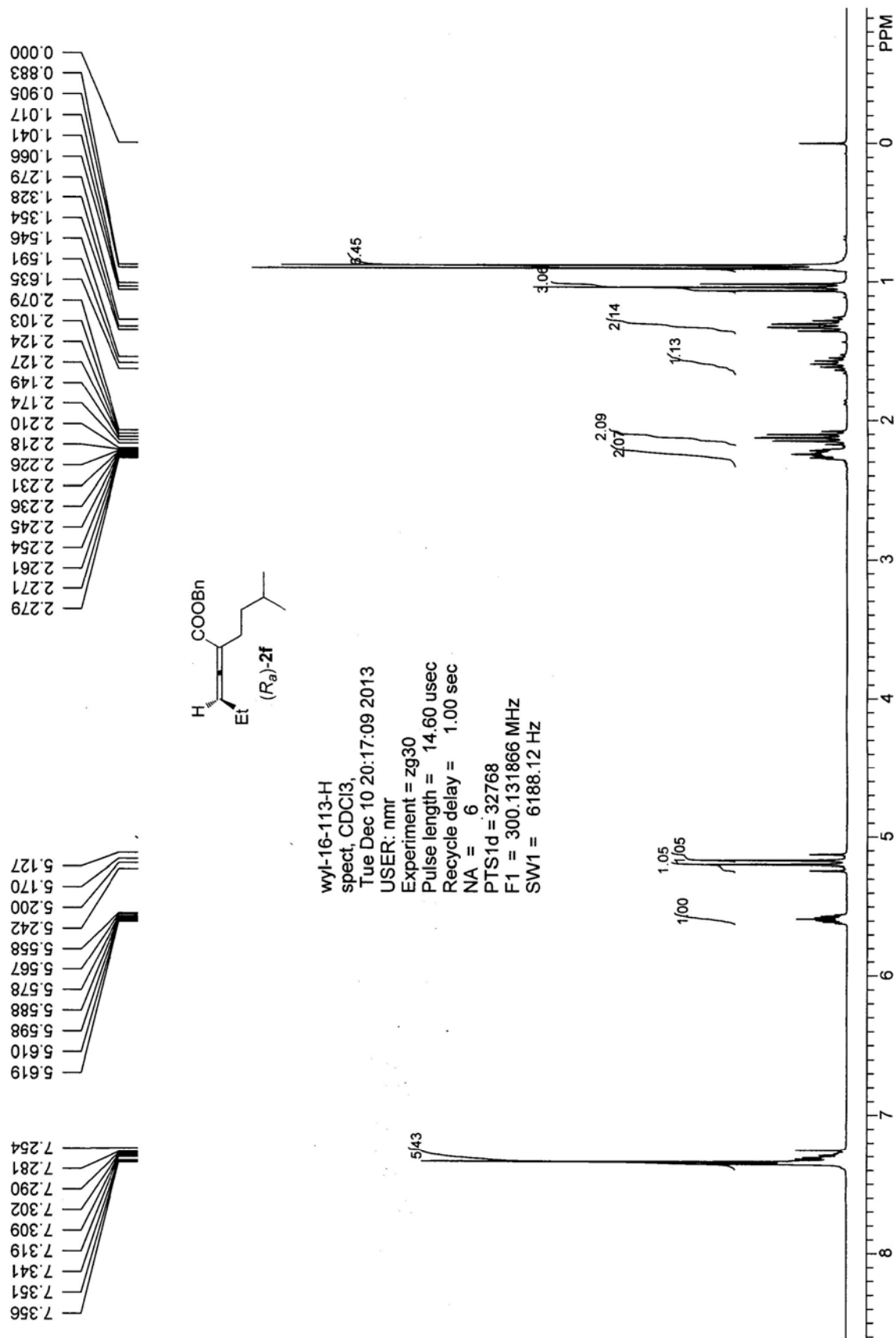
实验内容简介:  
whelk-s, s 200+1  
0.3ml/min 214nm

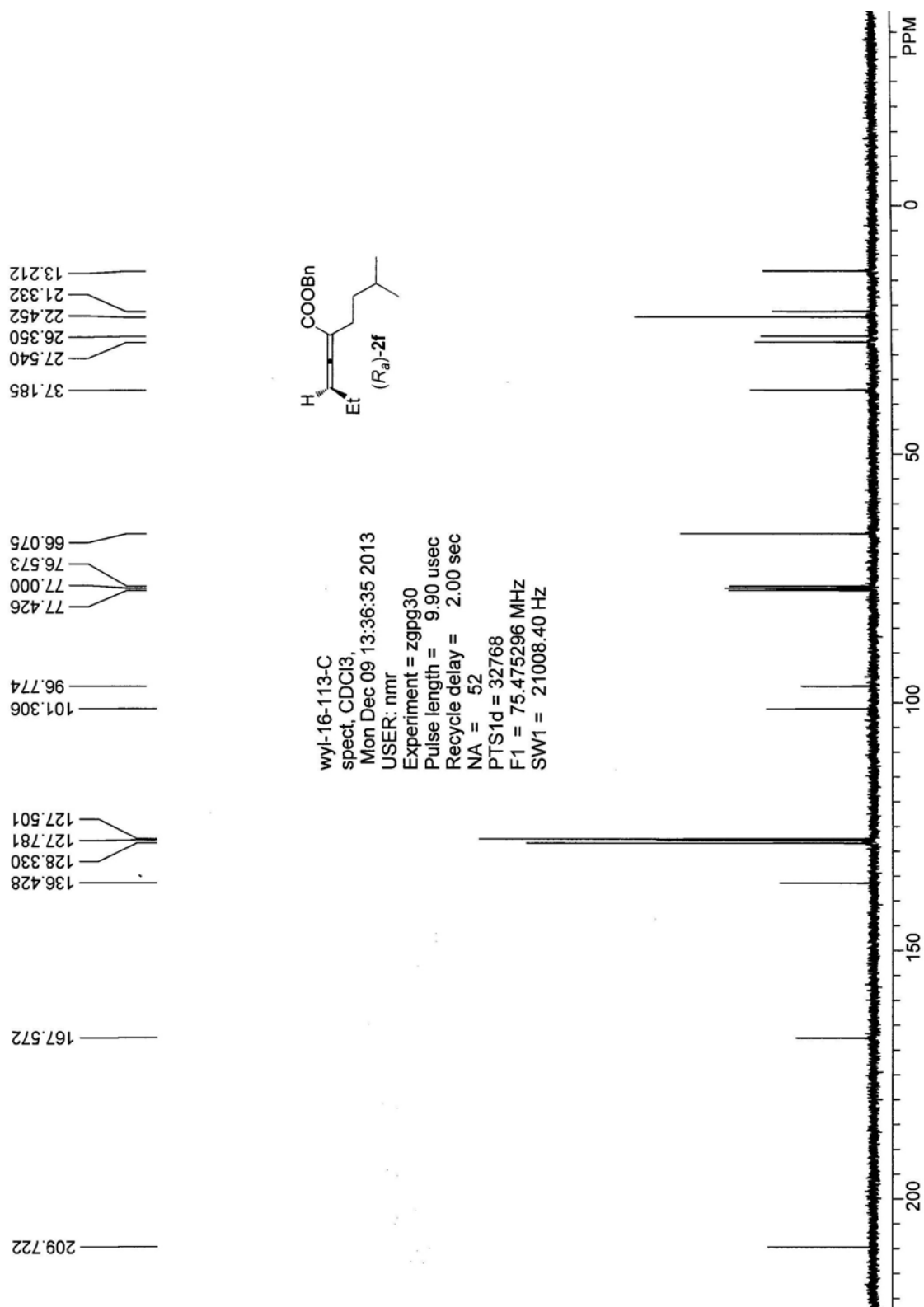


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.635	247871.281	7089518.000	50.0137
2		22.678	227785.813	7085625.000	49.9863
总计			475657.094	14175143.000	100.0000







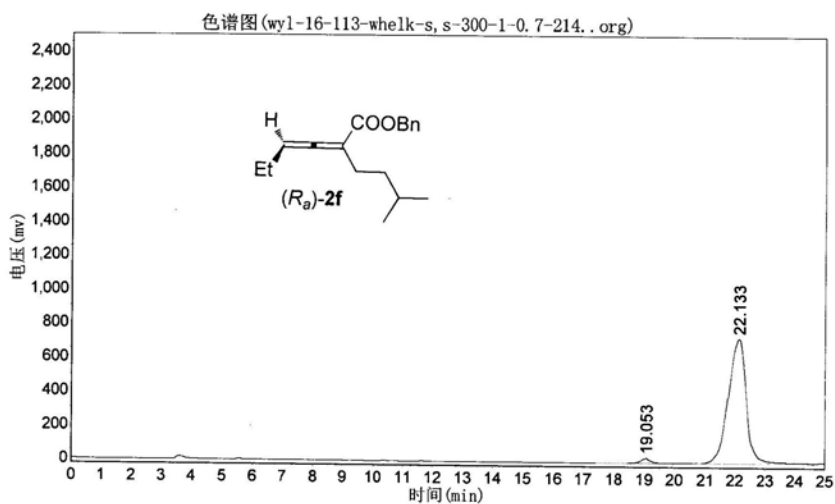
# wyl-16-113-wheelk-s, s-300-1-0.7-214

实验时间: 2013-06-13, 15:48:06

报告时间: 2013-06-13, 16:47:35

谱图文件: D:\zhuguangjiong\wyl\20130613\wyl-16-113-wheelk-s, s-300-1-0.7-214..org

实验内容简介:  
wheelk-s, s 300+1  
0.7ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		19.053	24829.750	618617.000	1.9914
2		22.133	729771.188	30445266.000	98.0086
总计			754600.938	31063883.000	100.0000

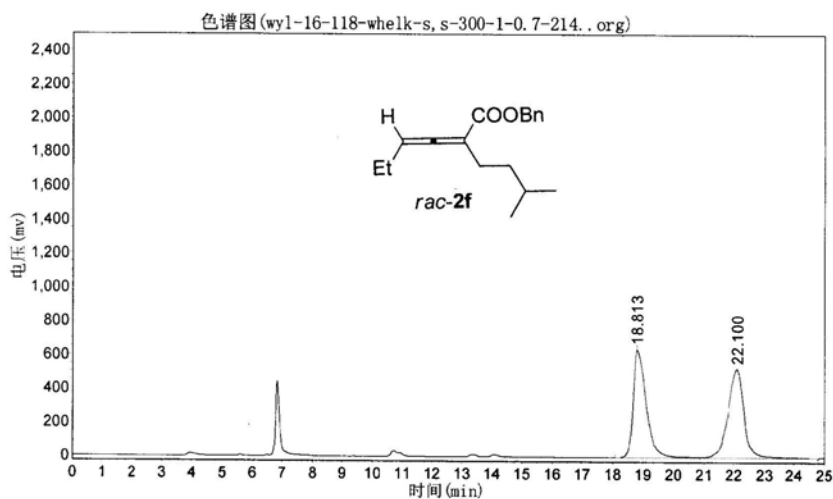
# wyl-16-118-whelk-s, s-300-1-0.7-214

实验时间: 2013-06-13, 15:20:35

报告时间: 2013-06-13, 16:45:34

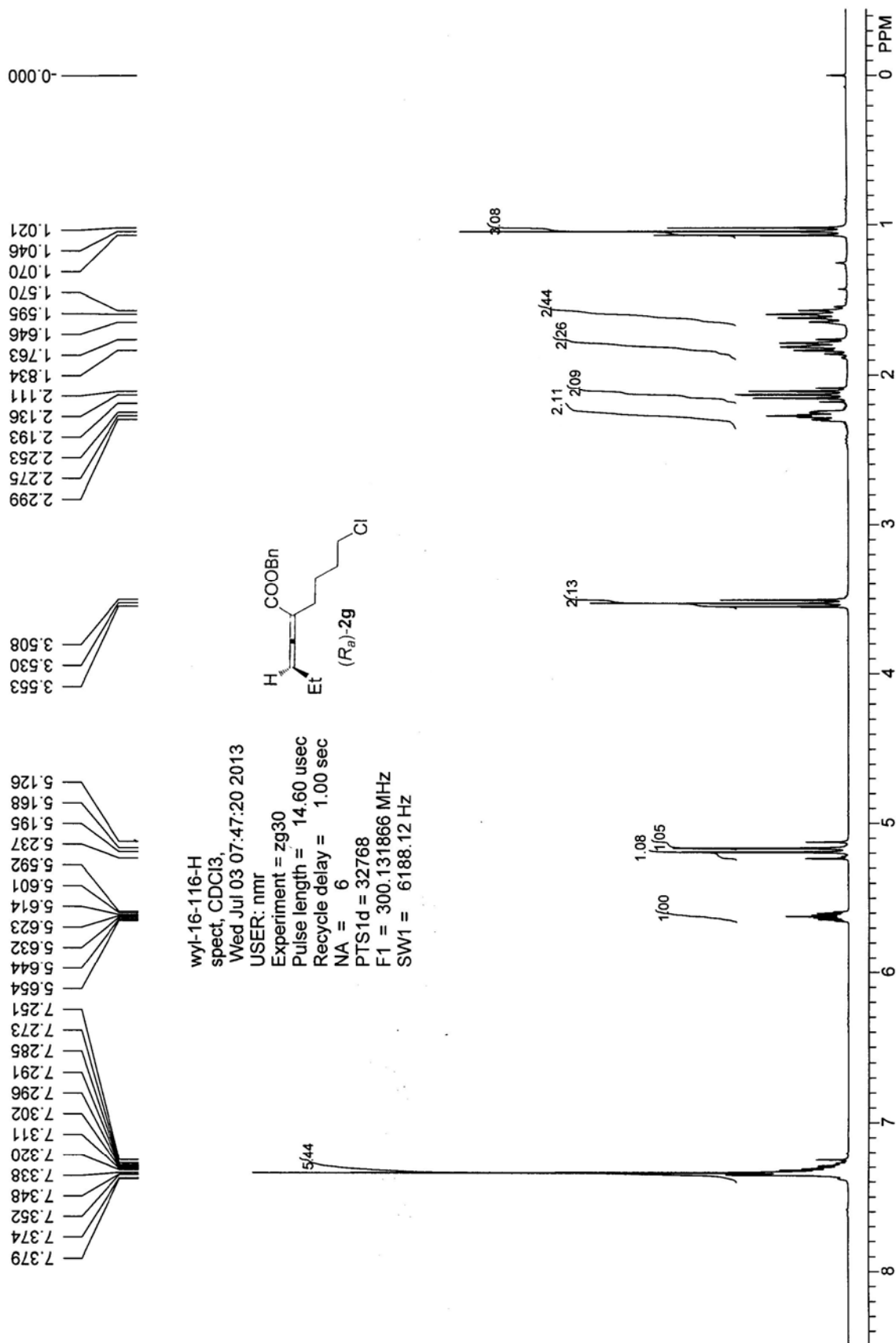
谱图文件: D:\zhuguangjiong\wyl\20130613\wyl-16-118-whelk-s, s-300-1-0.7-214. .org

实验内容简介:  
whelk-s, s 300+1  
0.7ml/min 214nm

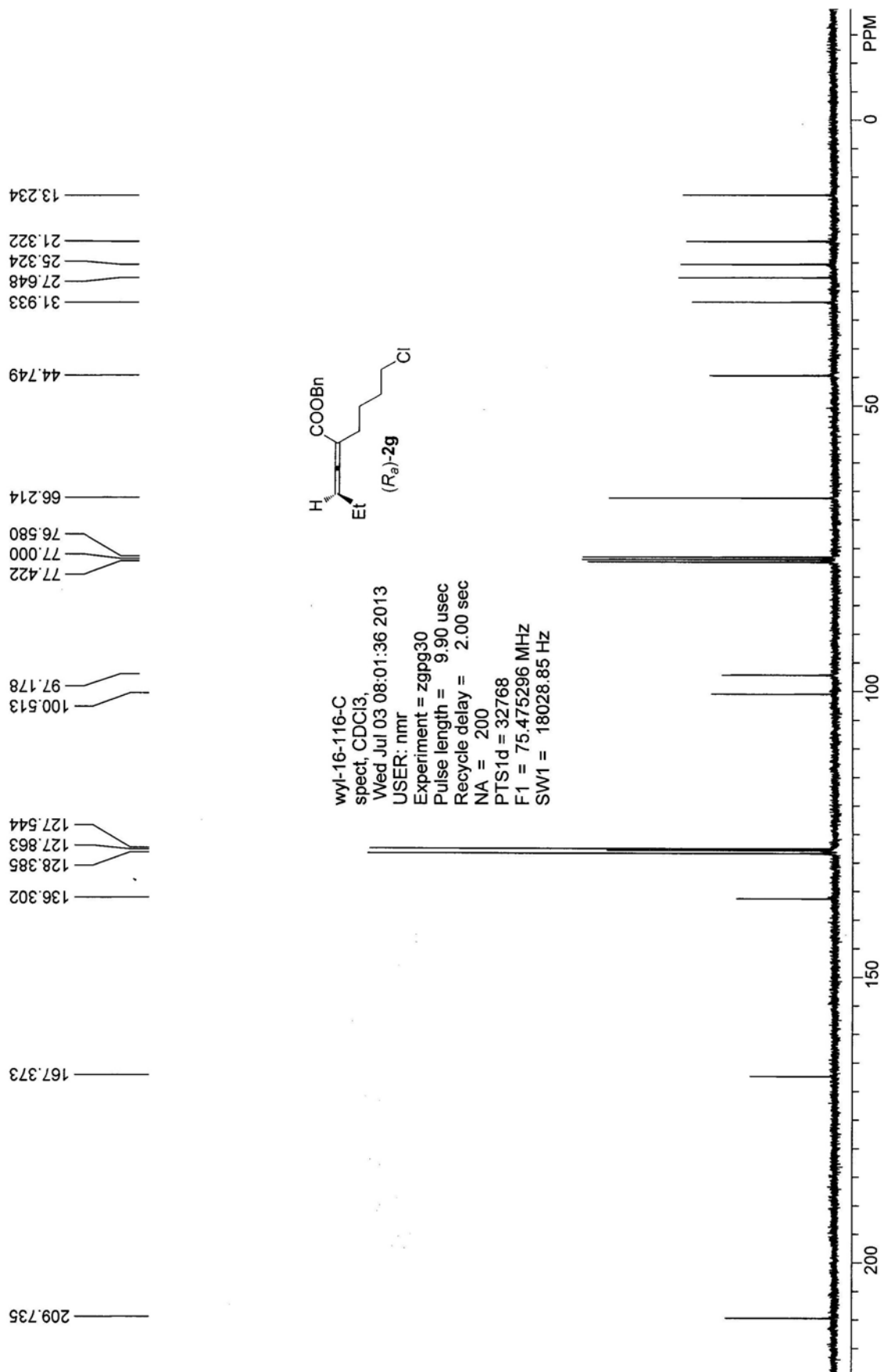


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		18.813	641433.000	19134010.000	49.5780
2		22.100	524517.750	19459710.000	50.4220
总计			1165950.750	38593720.000	100.0000

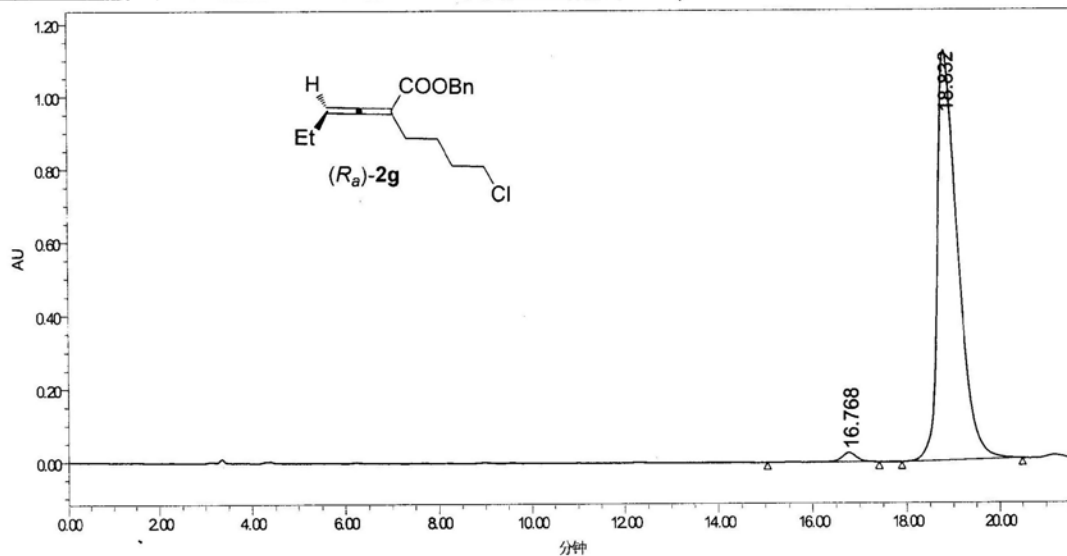


wyl-16-116-H  
spect, CDCl<sub>3</sub>,  
Wed Jul 03 07:47:20 2013  
USER: nmr  
Experiment = zg30  
Pulse length = 14.60 usec  
Recycle delay = 1.00 sec  
NA = 6  
PTS1d = 32768  
F1 = 300.131866 MHz  
SW1 = 6188.12 Hz



## SAMPLE INFORMATION

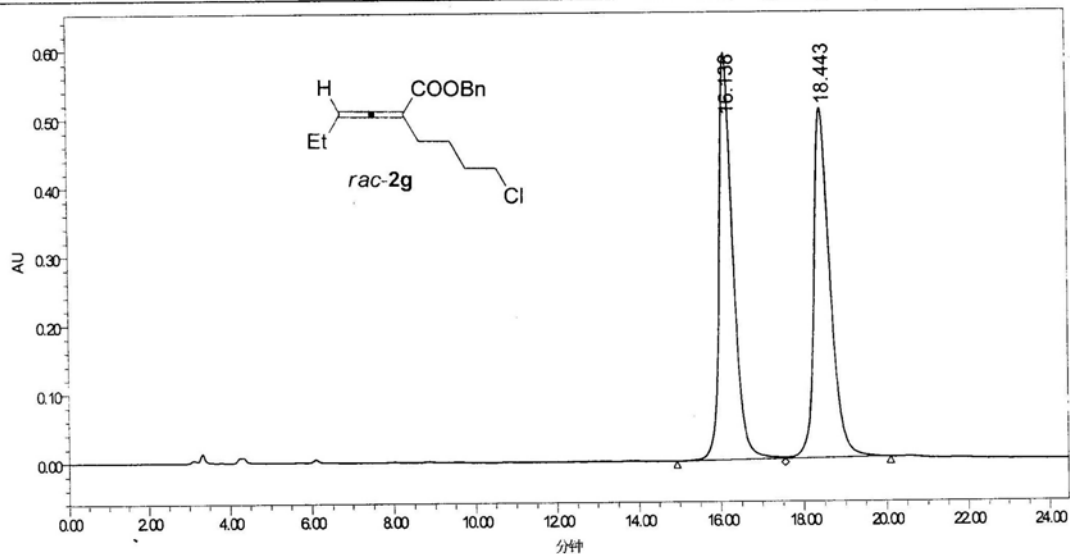
Sample Name:	wyl-16-116-whalek.s.s-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/6/14 13:57:40 CST
Vial:	1	Acq. Method:	zgj100
Injection #:	19	Date Processed:	2013/6/14 17:18:39 CST
Injection Volume:	10.00 ul	Channel Name:	V2489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	V2489 ChA.214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	16.768	599057	1.74	25980	2.26
2	18.832	33922444	98.26	1123185	97.74

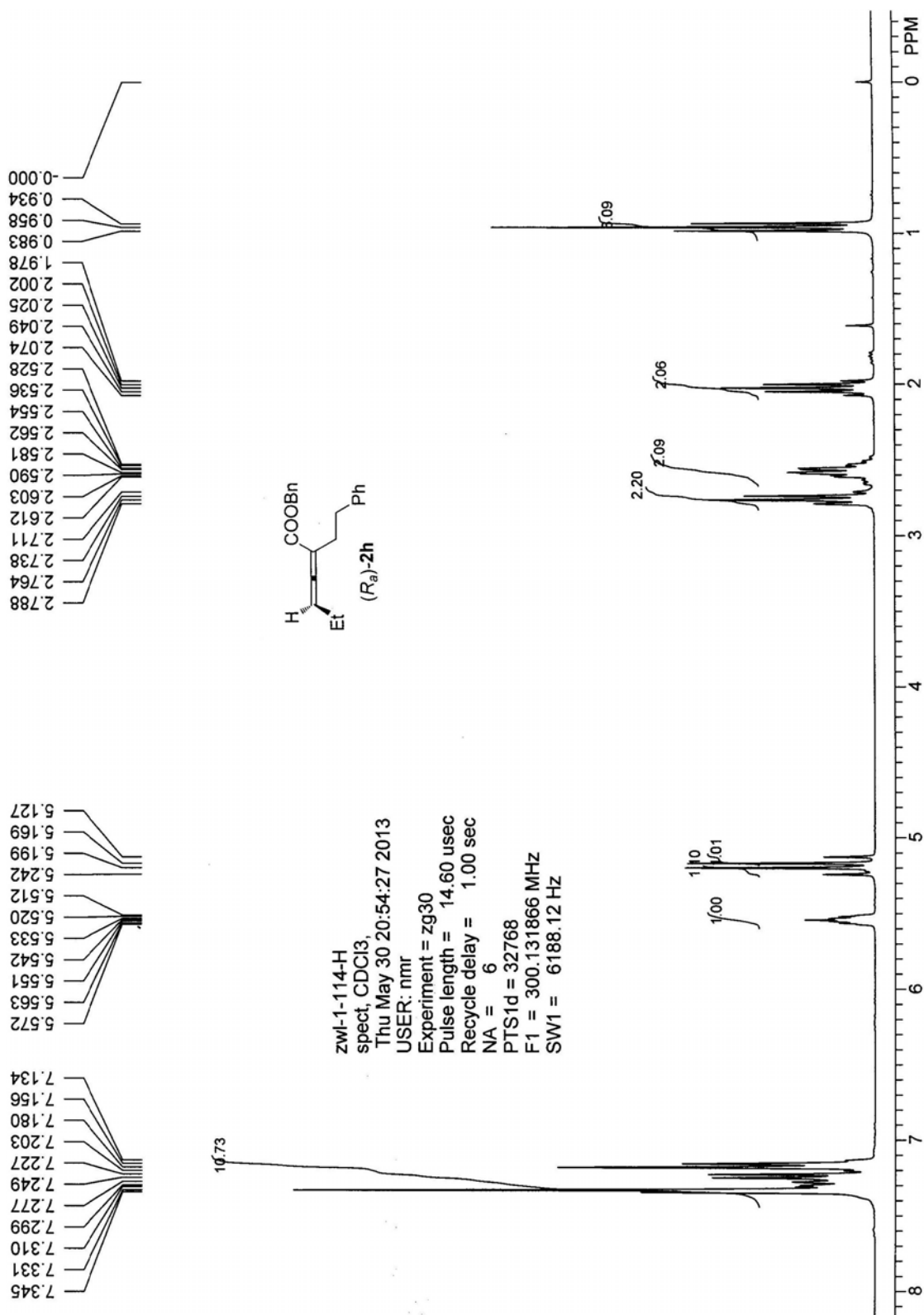
## SAMPLE INFORMATION

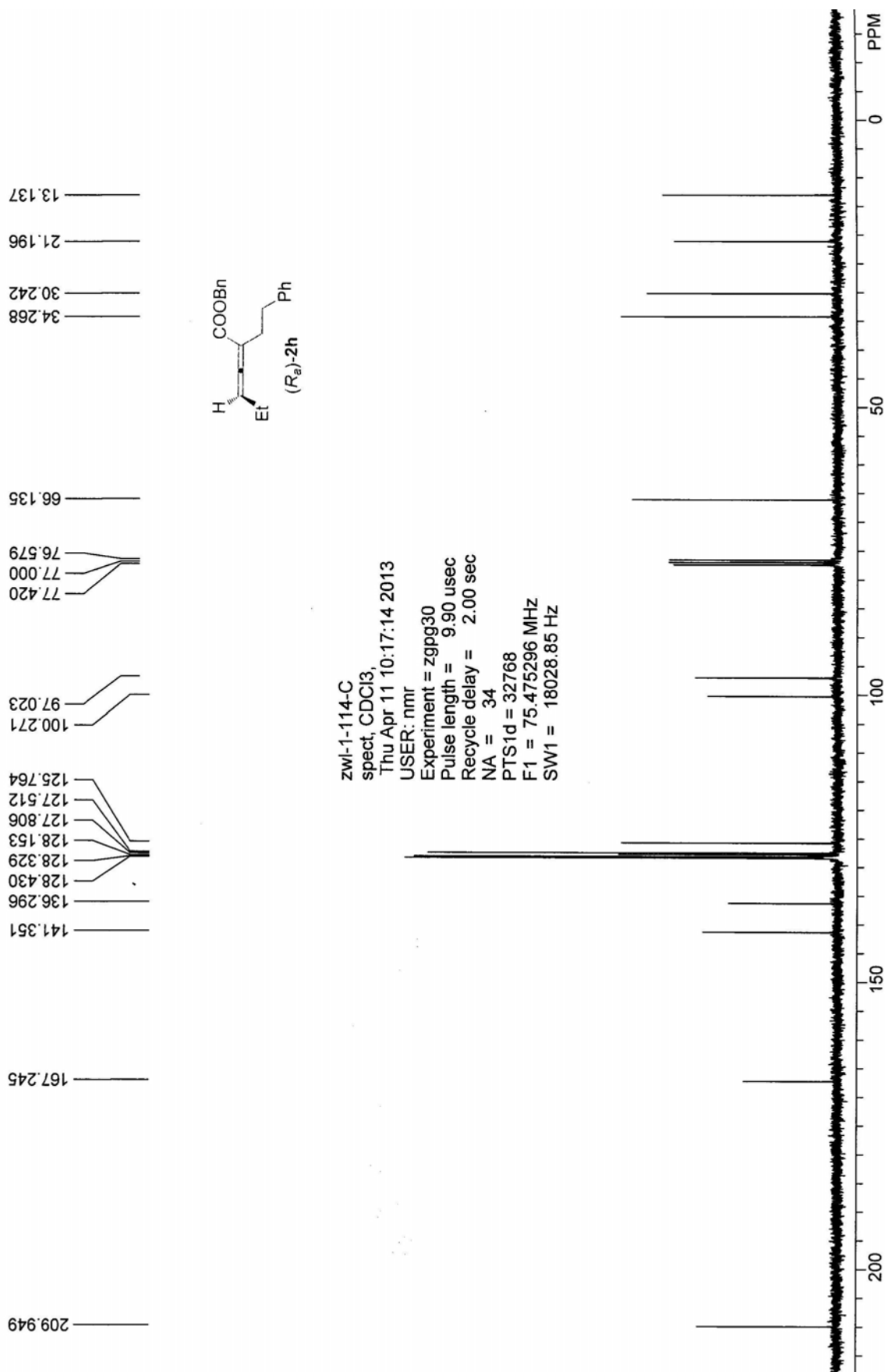
Sample Name:	zwf-2-11-whale-s-s-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/6/14 10:26:52 CST
Vial:	1	Acq. Method:	zg100
Injection #:	15	Date Processed:	2013/6/14 17:18:20 CST
Injection Volume:	10.00 uL	Channel Name:	V0489 ChA
Run Time:	200.00 Minutes	Channel Desc.:	V0489 ChA.214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (msec)	%Area	Height (msec)	% Height
1	16.136	13694479	50.01	593010	53.79
2	18.443	13689914	49.99	509347	46.21







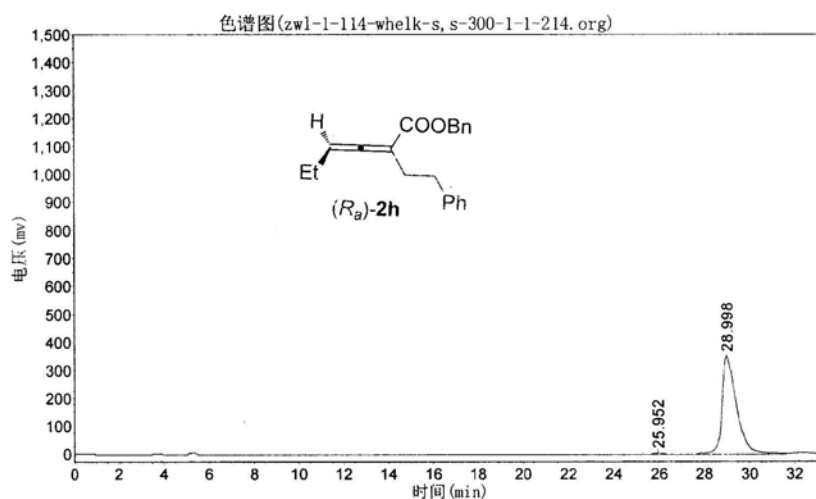
# zwl-1-114-whelk-s, s-300-1-1-214

实验时间: 2013-05-10, 12:12:00

报告时间: 2013-05-10, 12:47:58

谱图文件:D:\zhuguangjiong\zwl\20130510\zwl-1-114-whelk-s, s-300-1-1-214. org

实验内容简介:  
whelk-s, s 300+1  
1ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		25.952	4517.988	149698.406	0.9257
2		28.998	351228.844	16021067.000	99.0743
总计			355746.832	16170765.406	100.0000

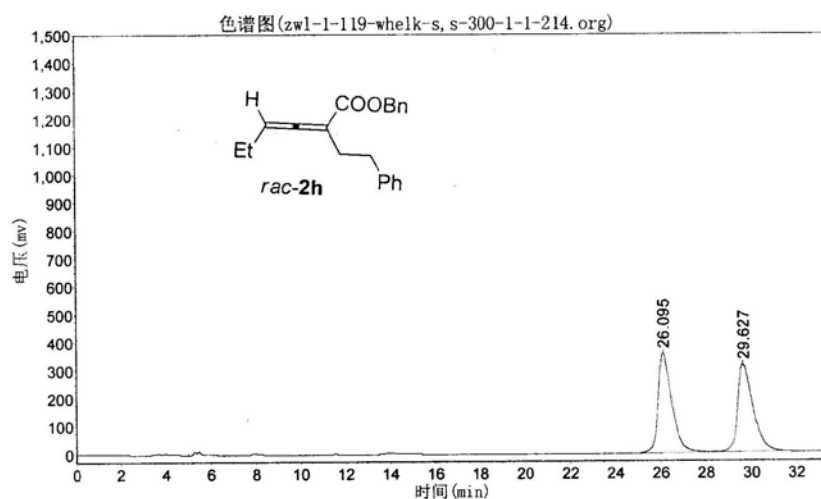
# zwl-1-119-whelk-s, s-300-1-1-214

实验时间: 2013-05-10, 11:32:44

报告时间: 2013-05-10, 12:46:52

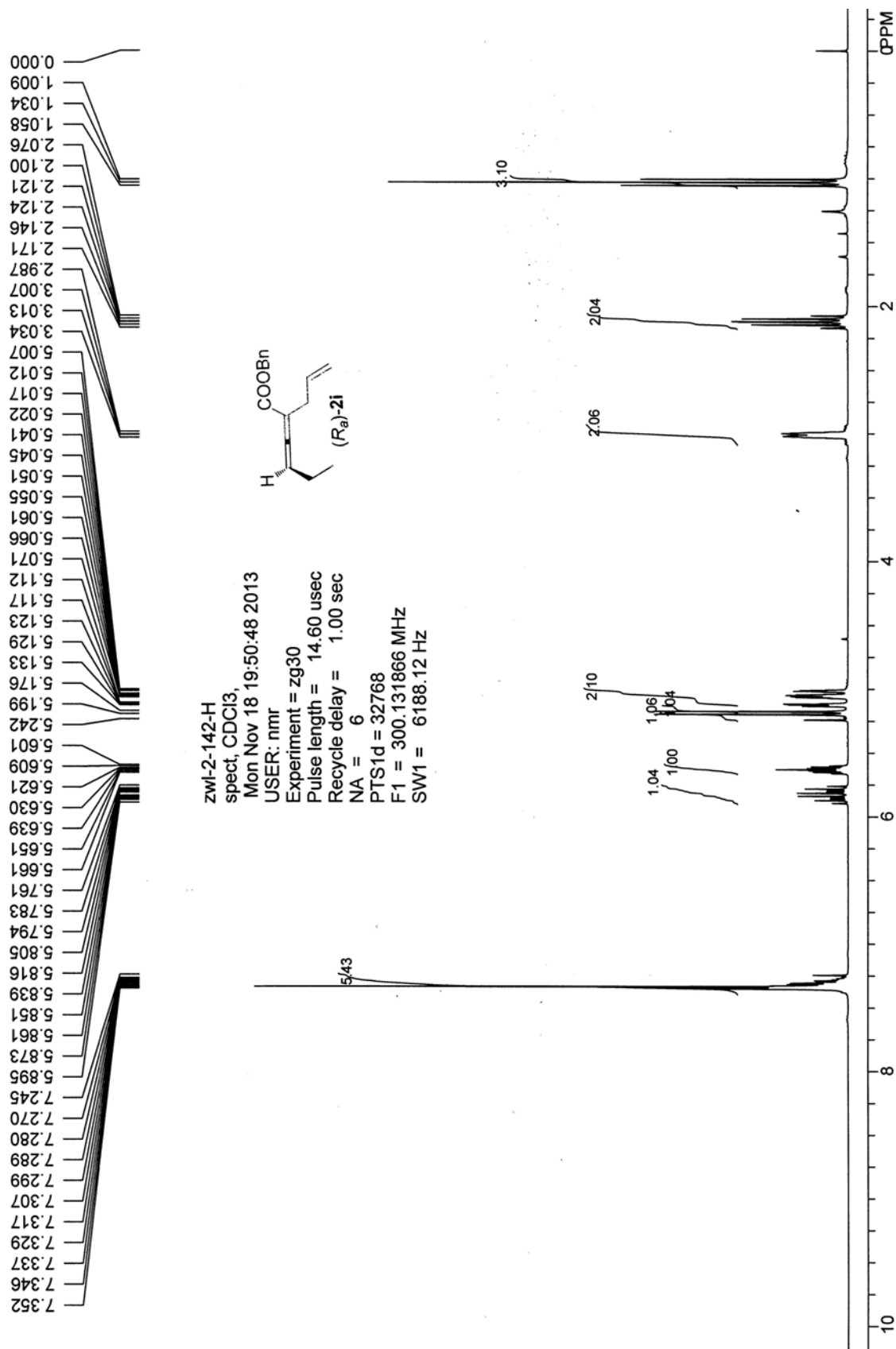
谱图文件: D:\zhuguangjiong\zwl\20130510\zwl-1-119-whelk-s, s-300-1-1-214.org

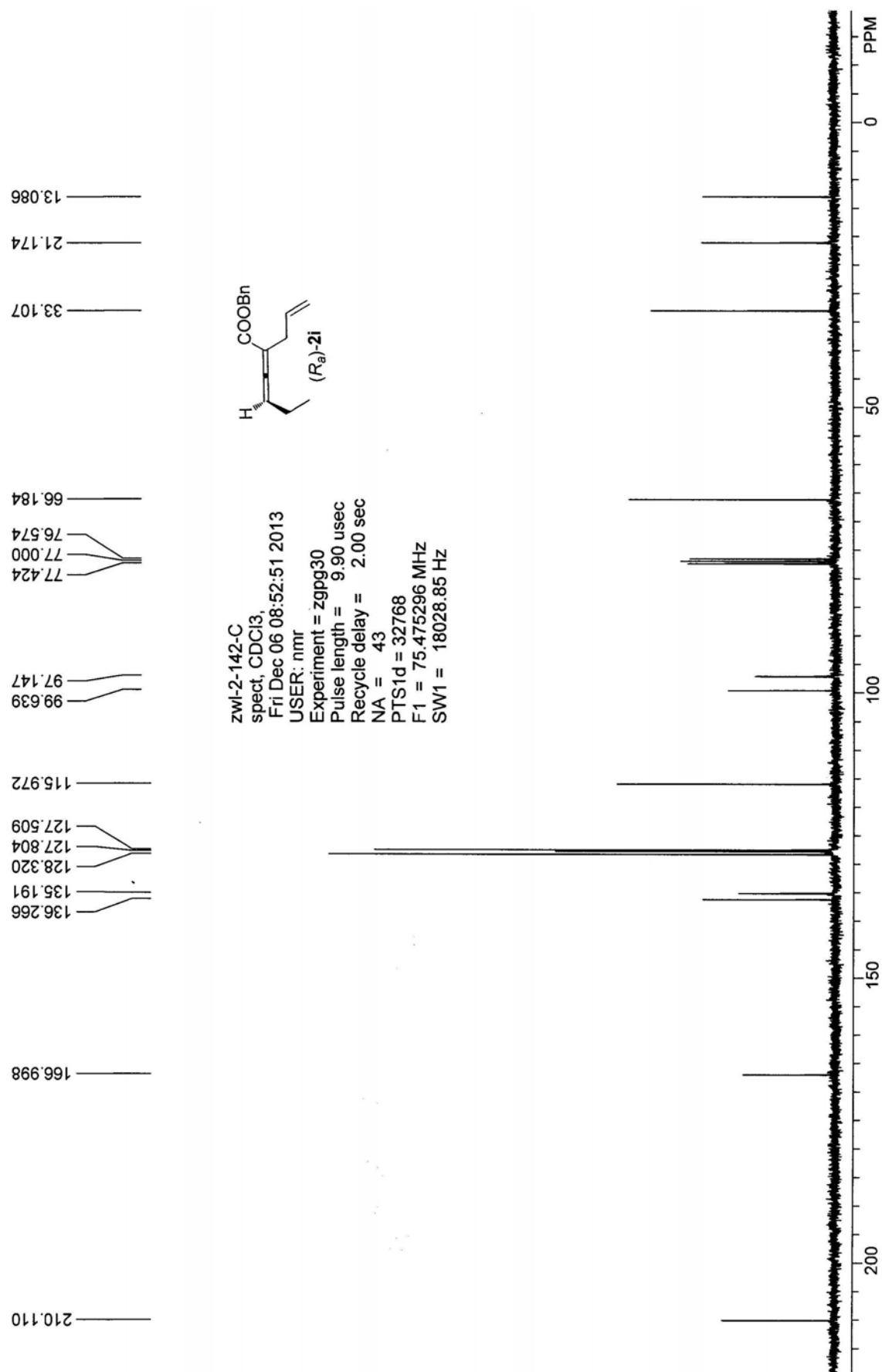
实验内容简介:  
whelk-s, s 300+1  
1ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		26.095	357798.969	15080448.000	50.2131
2		29.627	315047.281	14952478.000	49.7869
总计			672846.250	30032926.000	100.0000





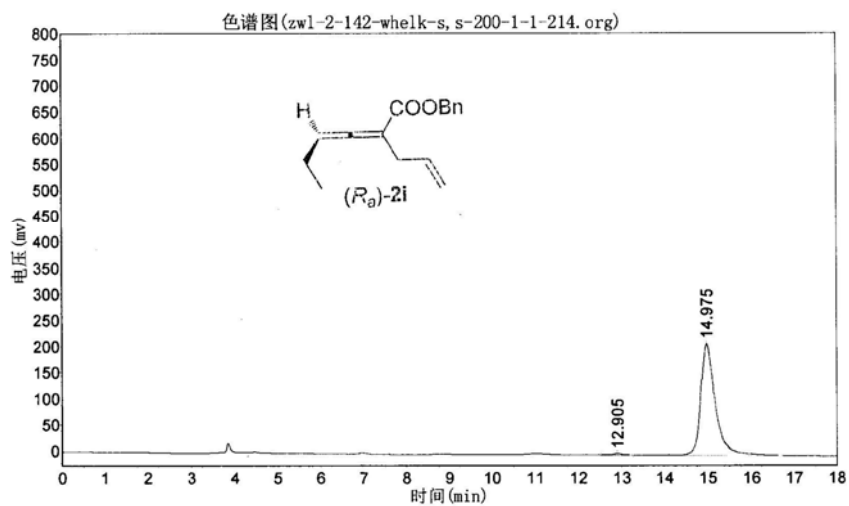
# zw1-2-142-wheelk-s, s-200-1-1-214

实验时间: 2013-11-14, 14:43:41

报告时间: 2013-11-14, 15:47:01

谱图文件: D:\zhuguangjiong\zw1\20131114\zw1-2-142-wheelk-s, s-200-1-1-214.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		12.905	3443.599	58046.414	1.1443
2		14.975	213389.750	5014817.500	98.8557
总计			216833.349	5072863.914	100.0000

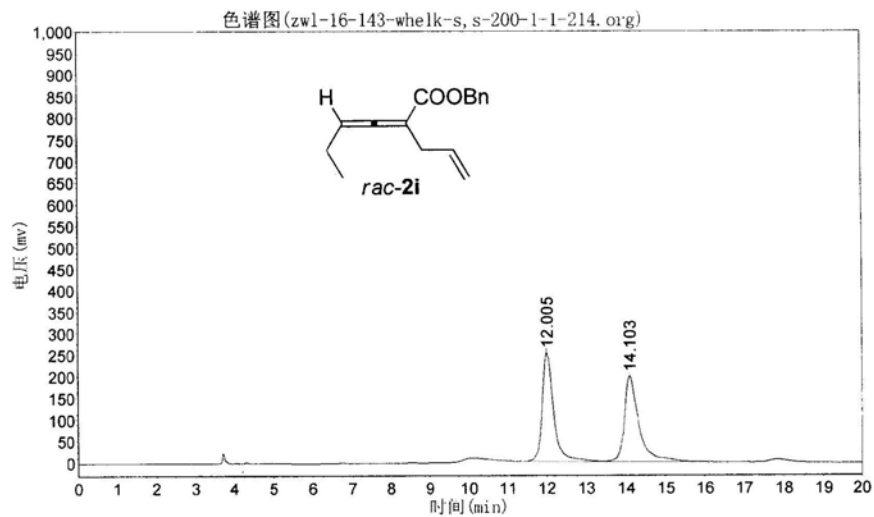
# zw1-16-143-whelk-s, s-200-1-1-214

实验时间: 2013-11-14, 13:59:37

报告时间: 2013-11-14, 15:45:39

谱图文件:D:\zhuguangjiong\zw1\20131114\zw1-16-143-whelk-s, s-200-1-1-214.org

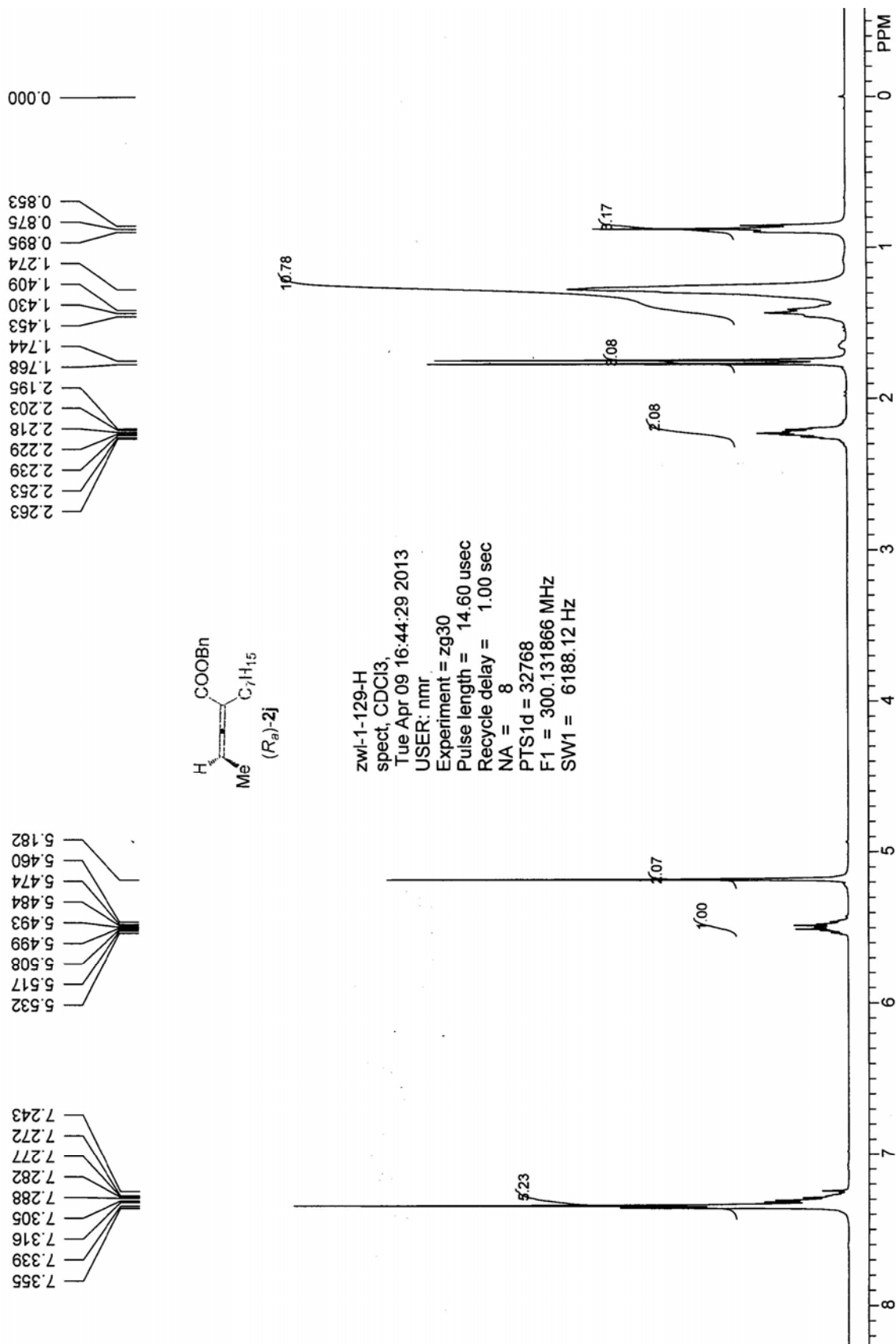
实验内容简介:

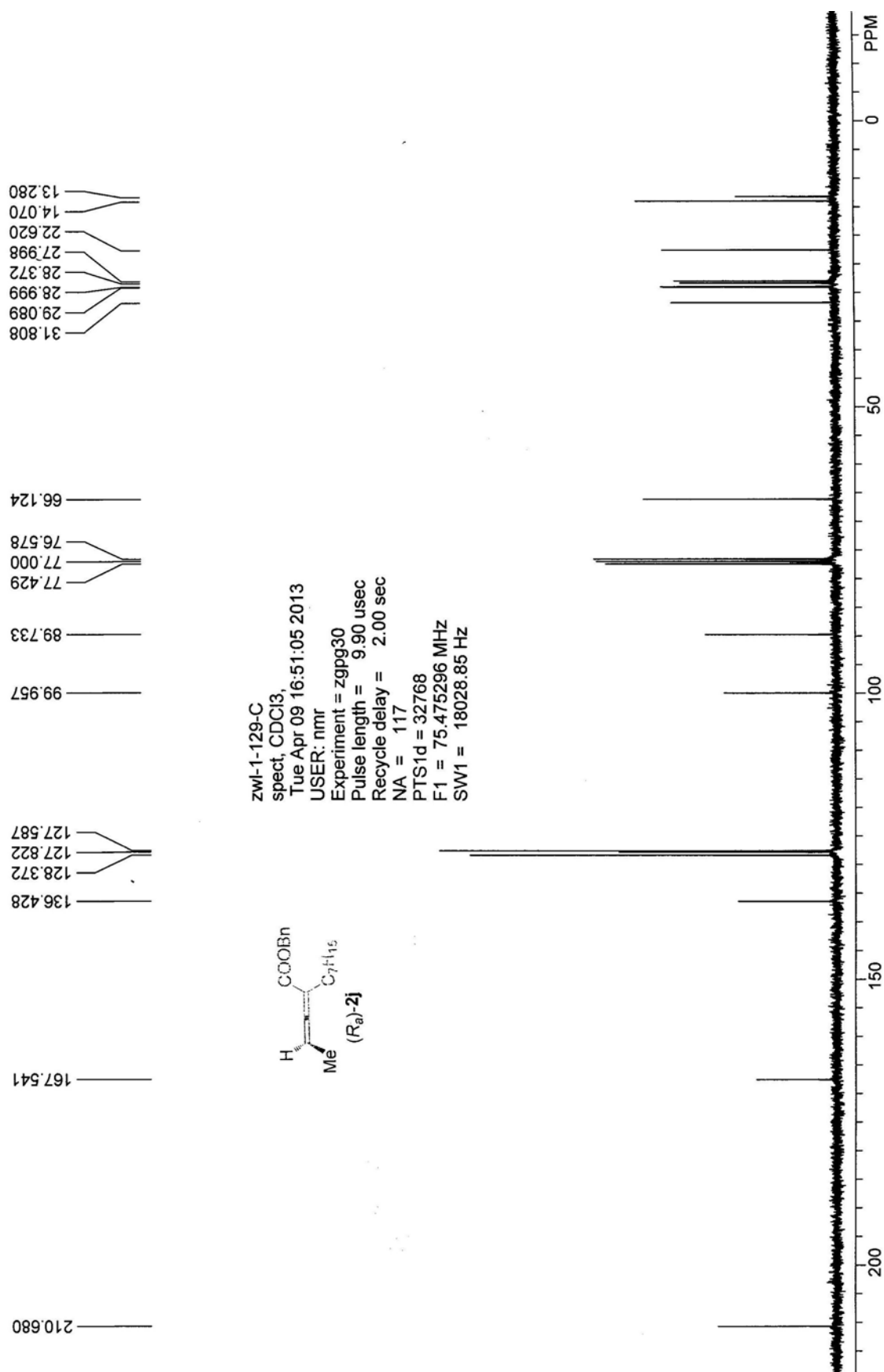


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.005	251432.641	5090092.000	49.7997
2		14.103	197256.656	5131036.500	50.2003
总计			448689.297	10221128.500	100.0000

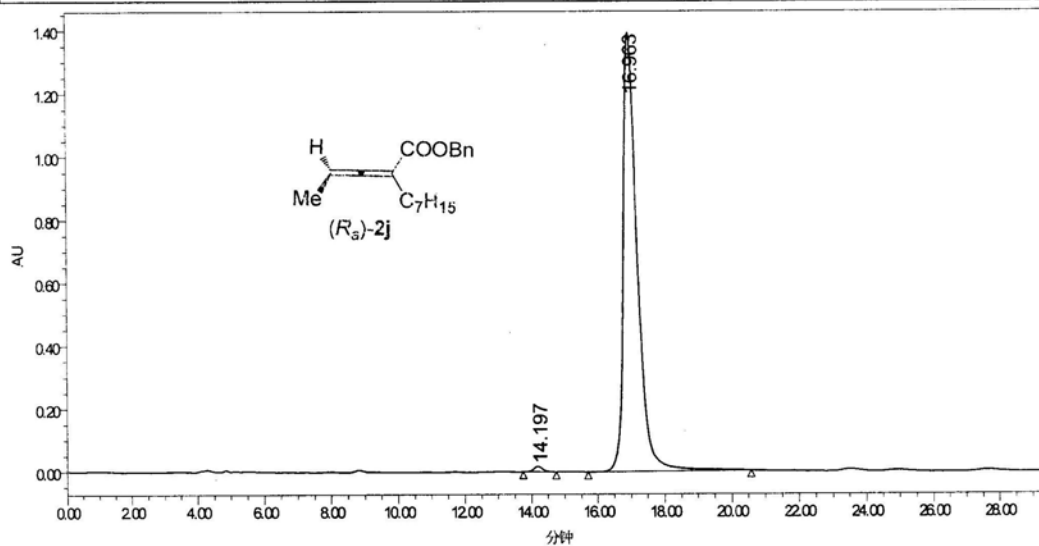






## SAMPLE INFORMATION

Sample Name:	zwf-1-129-whale-200-1-0.5-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/11/5 10:20:14 CST
Vial:	1	Acq. Method:	zgj100
Injection #:	4	Date Processed:	2013/11/5 15:18:11 CST
Injection Volume:	10.00 $\mu$ l	Channel Name:	V0489 ChA
Run Time:	60.00 Minutes	Sample Set Name:	

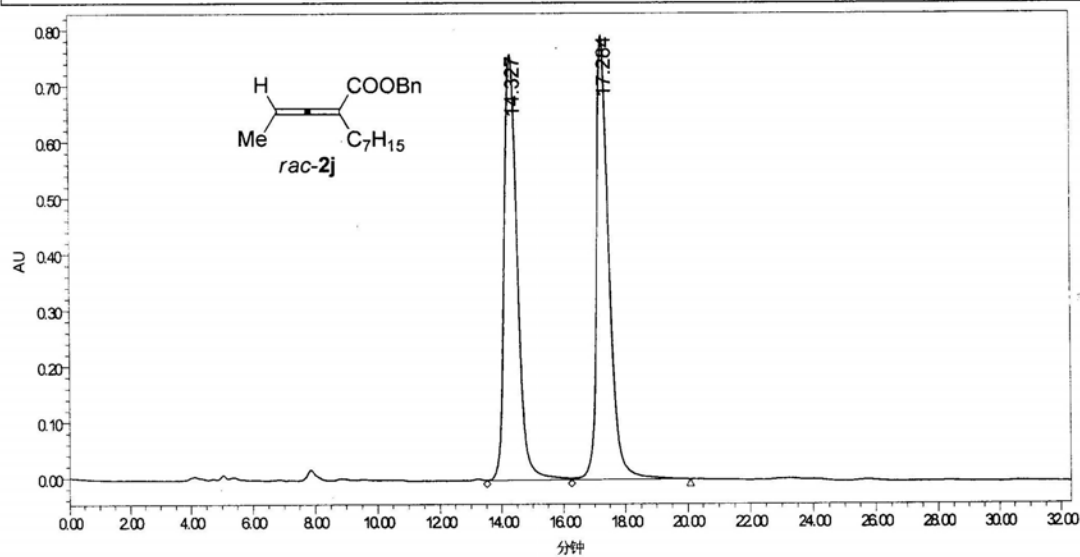


	RT (min)	Area (#sec)	%Area	Height (#)	% Height
1	14.197	343930	0.83	17755	1.26
2	16.963	40940710	99.17	139867	98.74

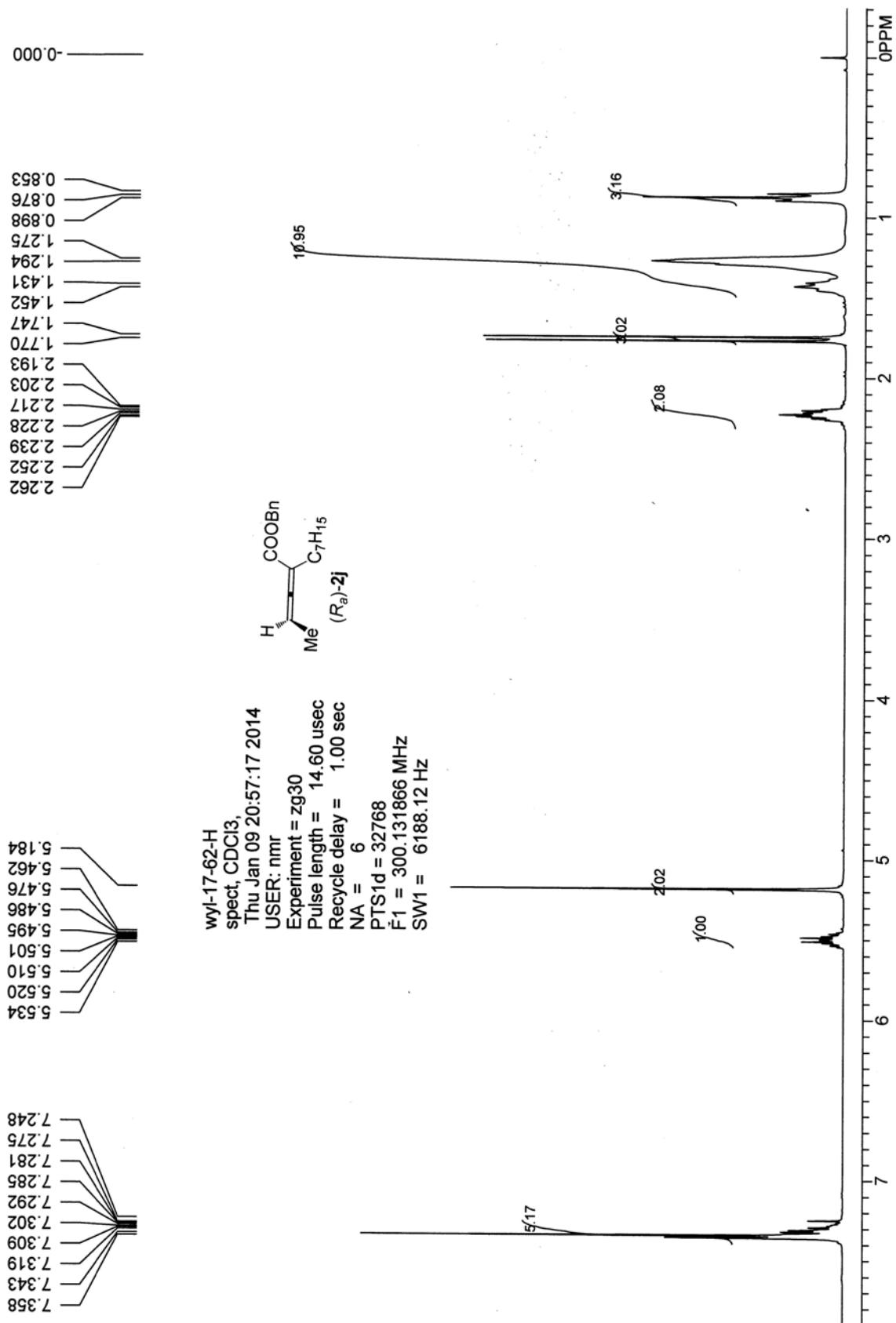
## SAMPLE INFORMATION

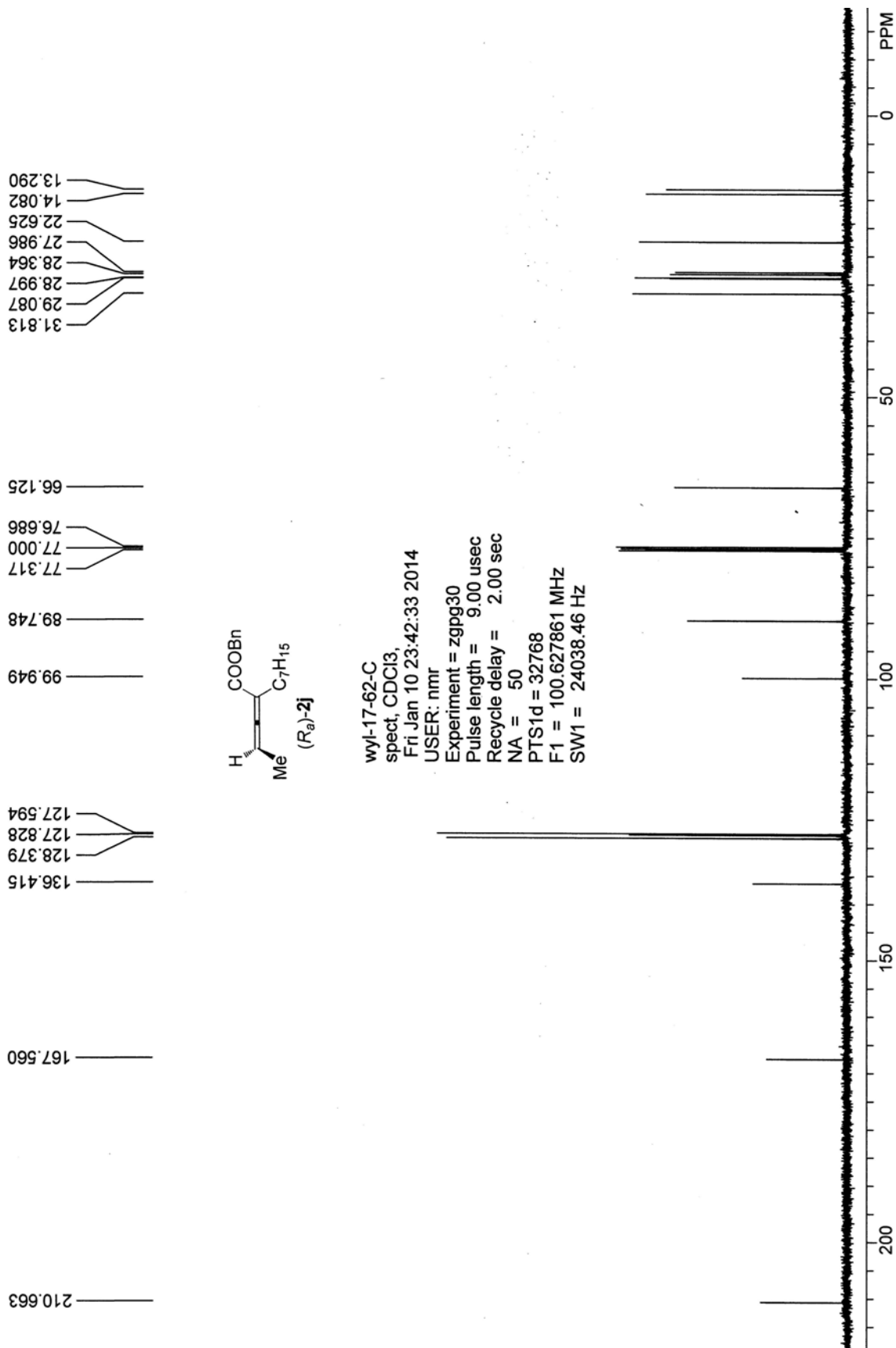
Sample Name: zwl-1-130-whale-200-1-05-214  
Sample Type: 未知  
Vial: 1  
Injection #: 6  
Injection Volume: 10.00  $\mu$ l  
Run Time: 60.00 Minutes

Acquired By: Breeze  
Date Acquired: 2013/11/5 11:36:35 CST  
Acq. Method: zg100  
Date Processed: 2013/11/5 15:16:24 CST  
Channel Name: V0489 ChA  
Sample Set Name:



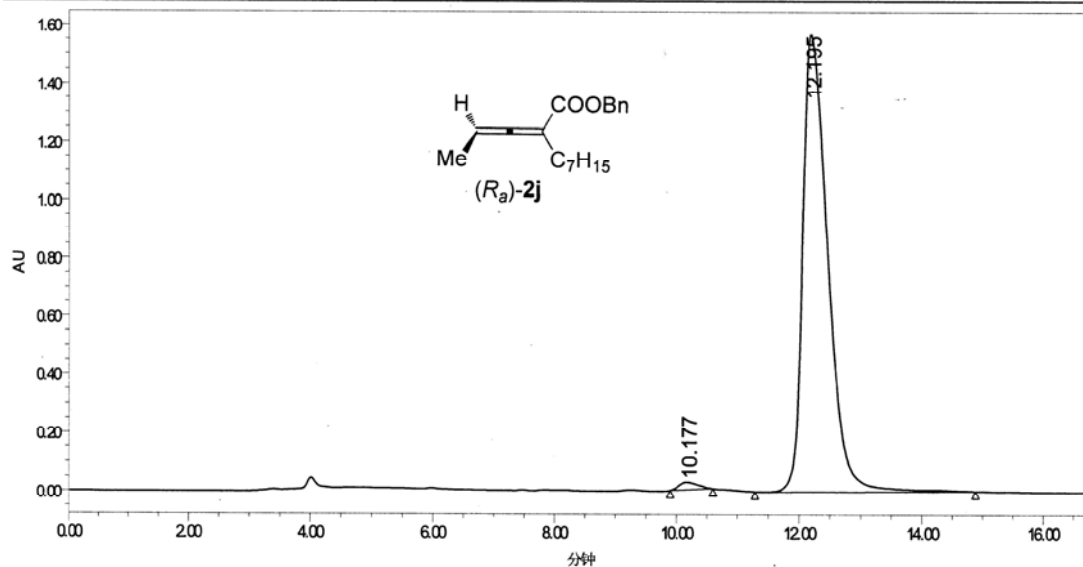
	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	14.327	22230993	49.79	760131	48.93
2	17.284	22415126	50.21	793233	51.07





## SAMPLE INFORMATION

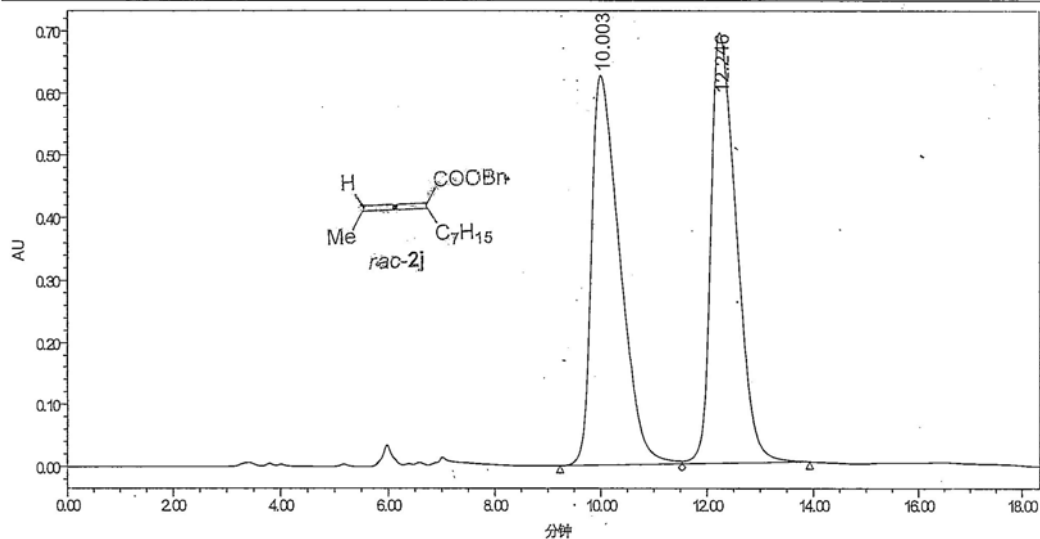
Sample Name:	wyl-17-62-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/12/31 12:41:58 CST
Vial:	1	Acq. Method:	zg100
Injection#:	16	Date Processed:	2013/12/31 13:55:17 CST
Injection Volume:	10.00 u	Channel Name:	V0489 ChA
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (msec)	%Area	Hight (mm)	% Hight
1	10.177	579242	1.29	26623	1.67
2	12.196	44349320	98.71	1567366	98.33

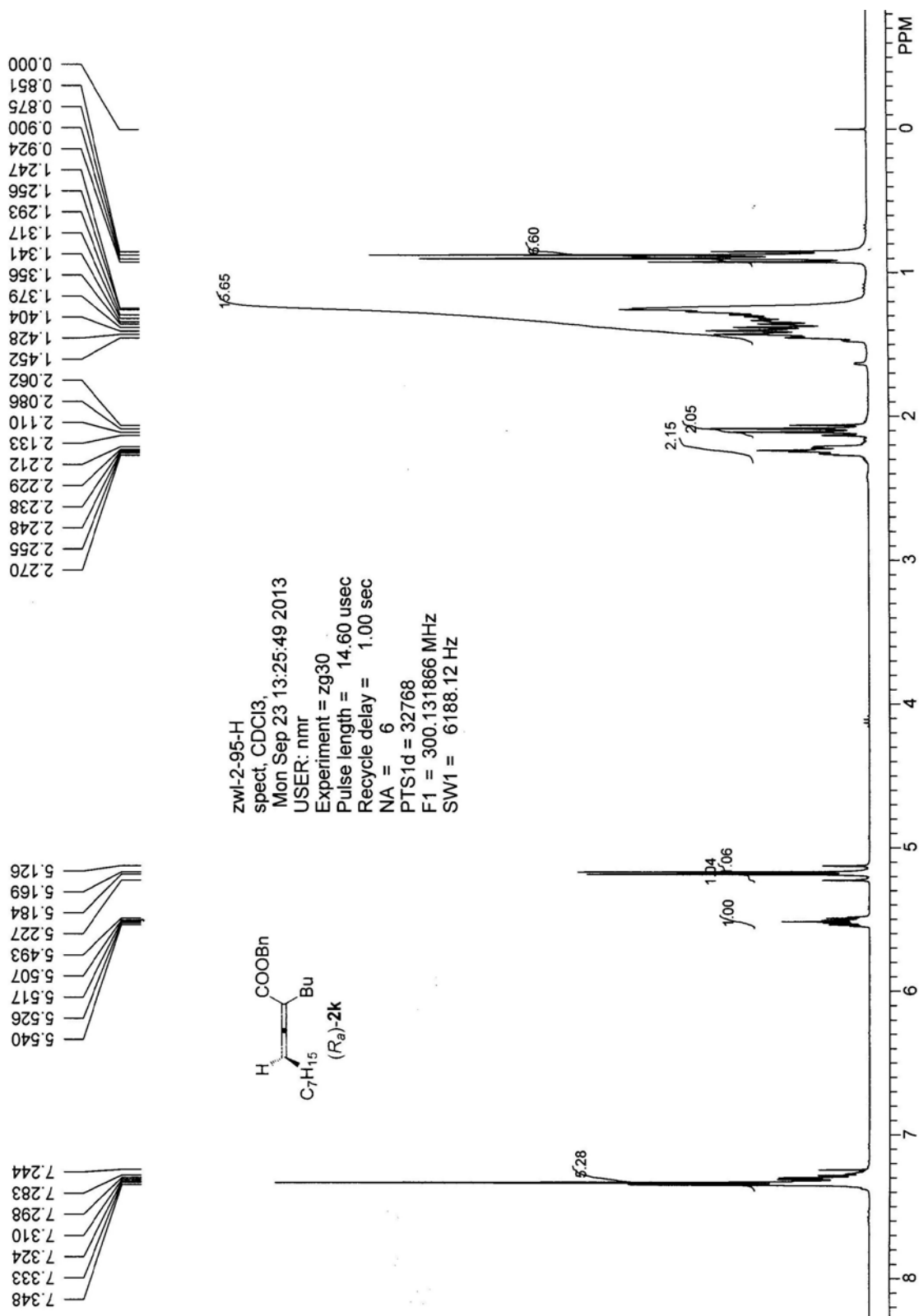
# SAMPLE INFORMATION

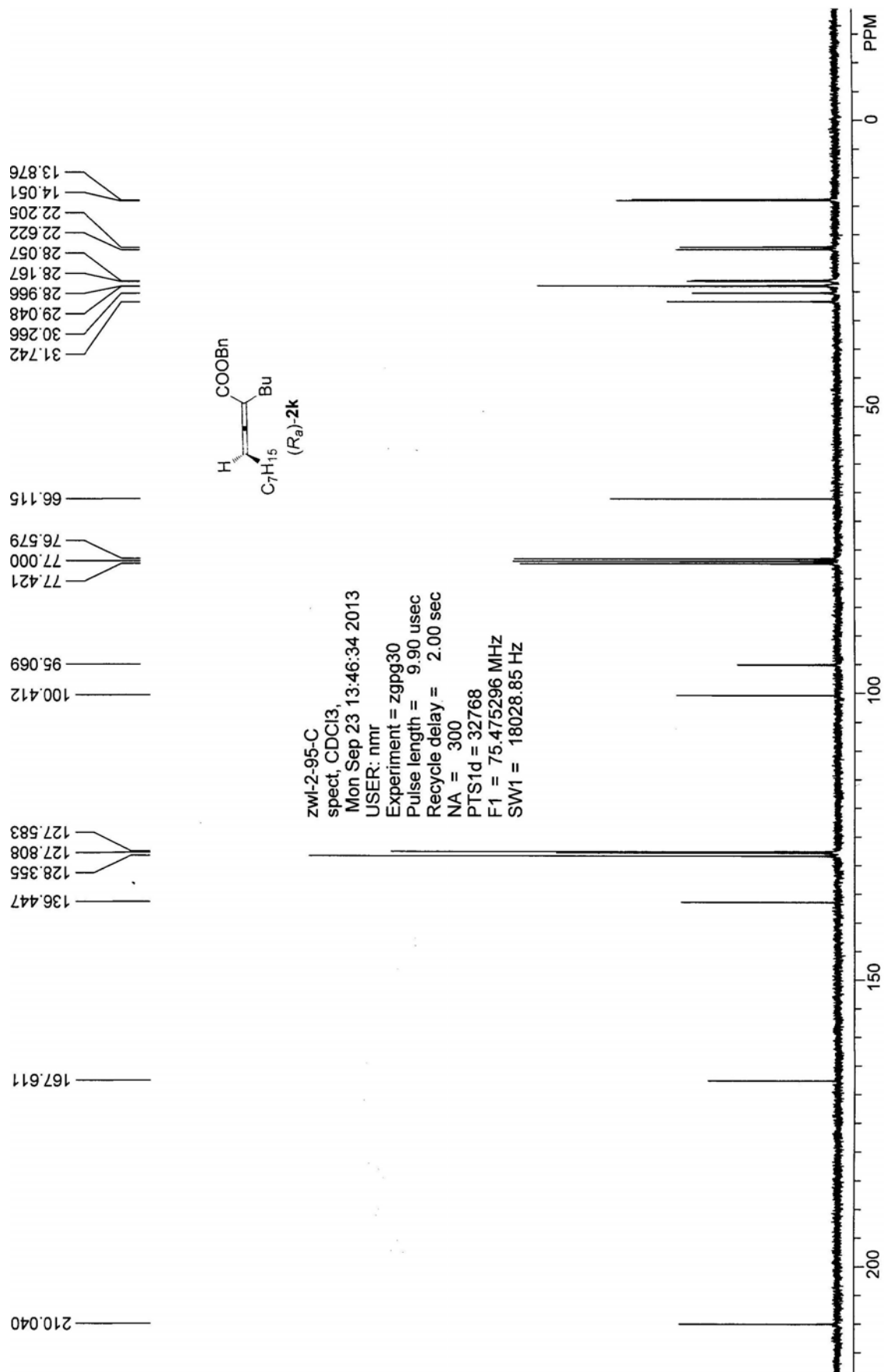
Sample Name:	zwf-1-130-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/12/31 11:59:56 CST
Vial:	1	Acq. Method:	zg100
Injection #:	14	Date Processed:	2013/12/31 16:46:25 CST
Injection Volume:	10.00 uL	Channel Name:	V2489 CHA
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (msec)	%Area	Hight (msec)	% Hight
1	10.003	23241379	50.18	626142	47.51
2	12.246	23076326	49.82	691879	52.49







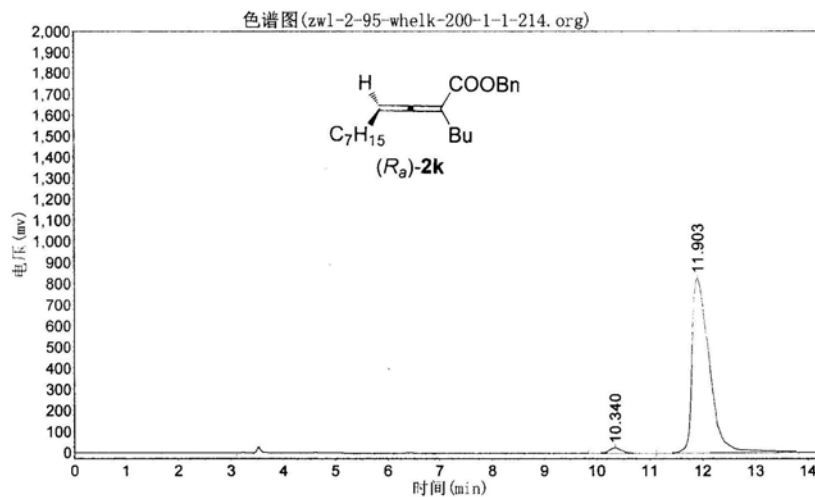
# zw1-2-95-whelk-200-1-1-214

实验时间: 2013/9/24, 9:54:02

报告时间: 2013/9/24, 11:27:38

谱图文件: D:\zhuguangjiong\zw1\20130924\zw1-2-95-whelk-200-1-1-214.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		10.340	26013.137	398905.344	2.1085
2		11.903	824633.313	18519628.000	97.8915
总计			850646.449	18918533.344	100.0000

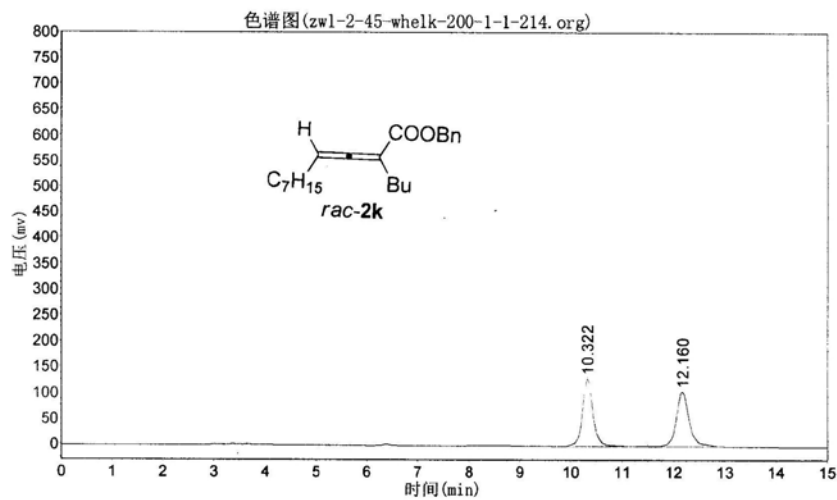
# zw1-2-45-whelk-200-1-1-214

实验时间: 2013/9/24, 9:35:48

报告时间: 2013/9/24, 11:29:02

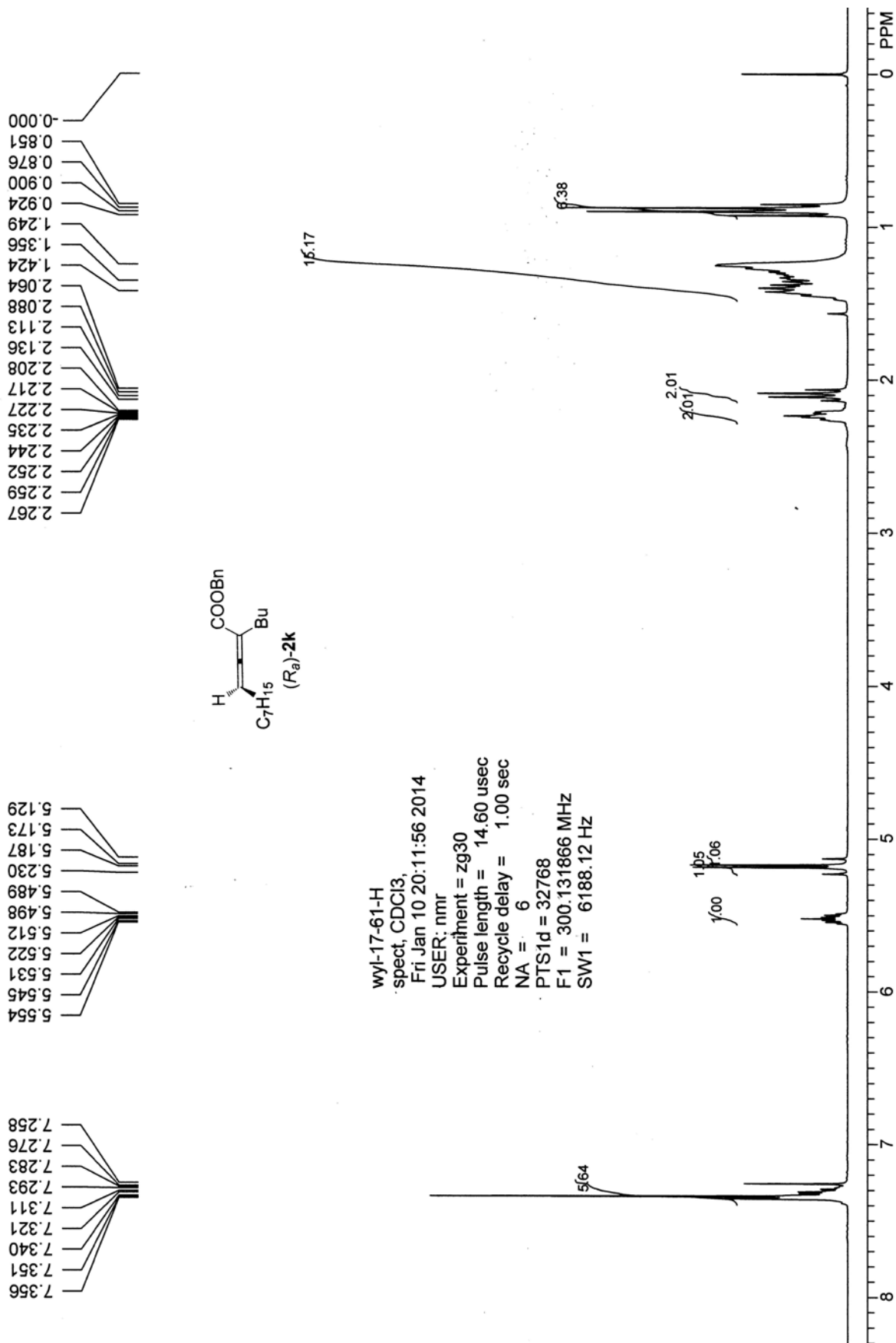
谱图文件: D:\zhuguangjiong\zw1\20130924\zw1-2-45-whelk-200-1-1-214.org

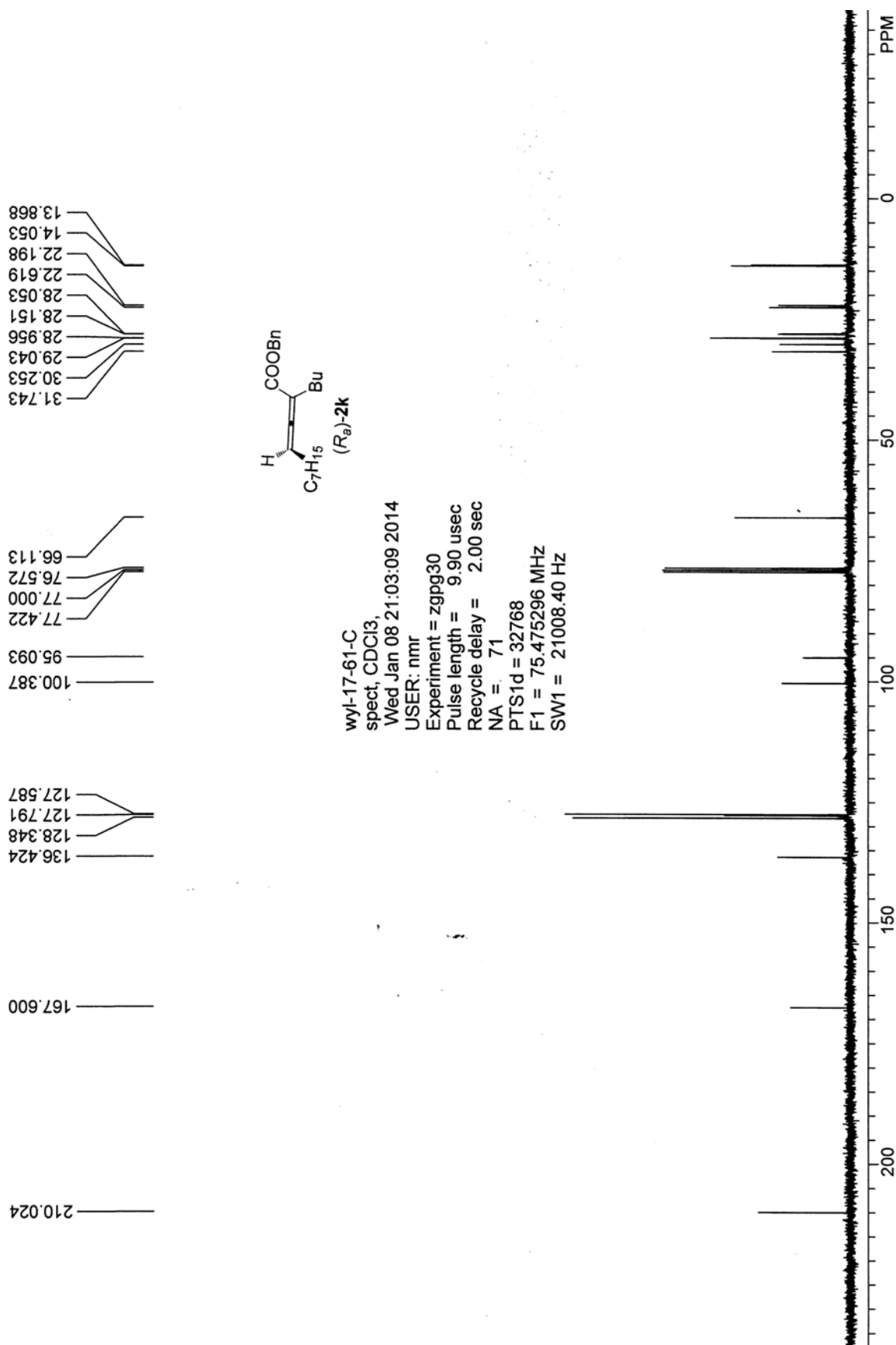
实验内容简介:



分析结果表

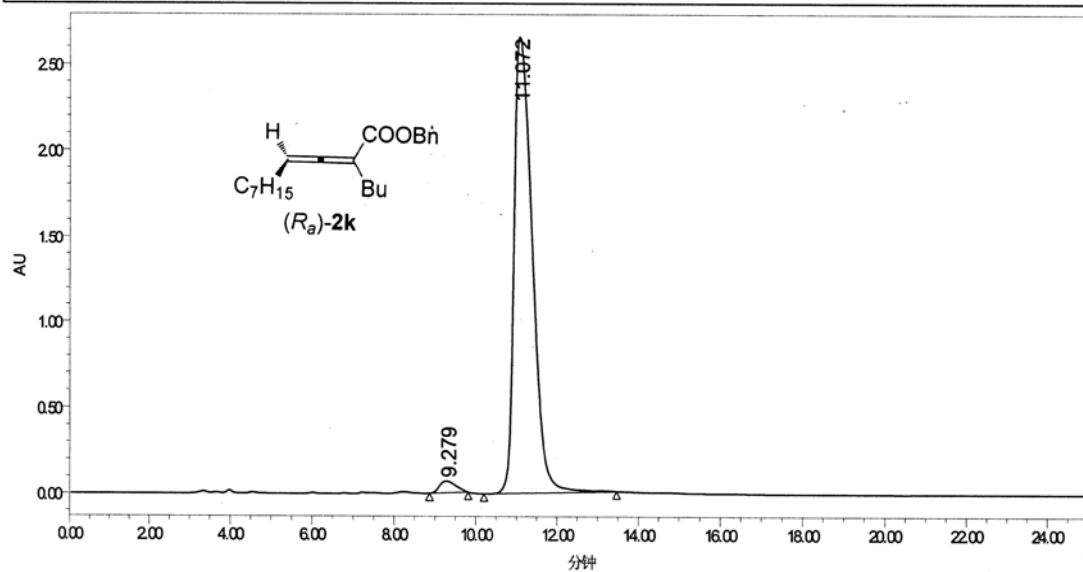
峰号	峰名	保留时间	峰高	峰面积	含量
1		10.322	129877.438	1893951.375	50.1008
2		12.160	105808.227	1886327.500	49.8992
总计			235685.664	3780278.875	100.0000





## SAMPLE INFORMATION

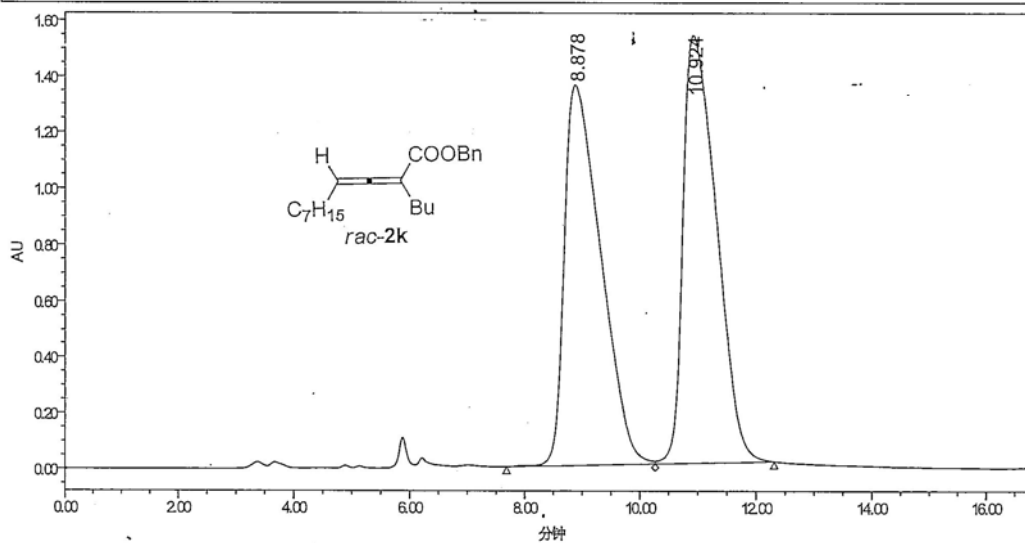
Sample Name:	wyl-17-61-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/12/31 10:07:13 CST
Vial:	1	Acq. Method:	zg100
Injection#:	8	Date Processed:	2013/12/31 12:34:16 CST
Injection Volume:	10.00 uL	Channel Name:	V0489 ChA
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (msec)	%Area	Height (msec)	% Height
1	9.279	1883123	2.22	68076	2.50
2	11.072	82828456	97.78	2654040	97.50

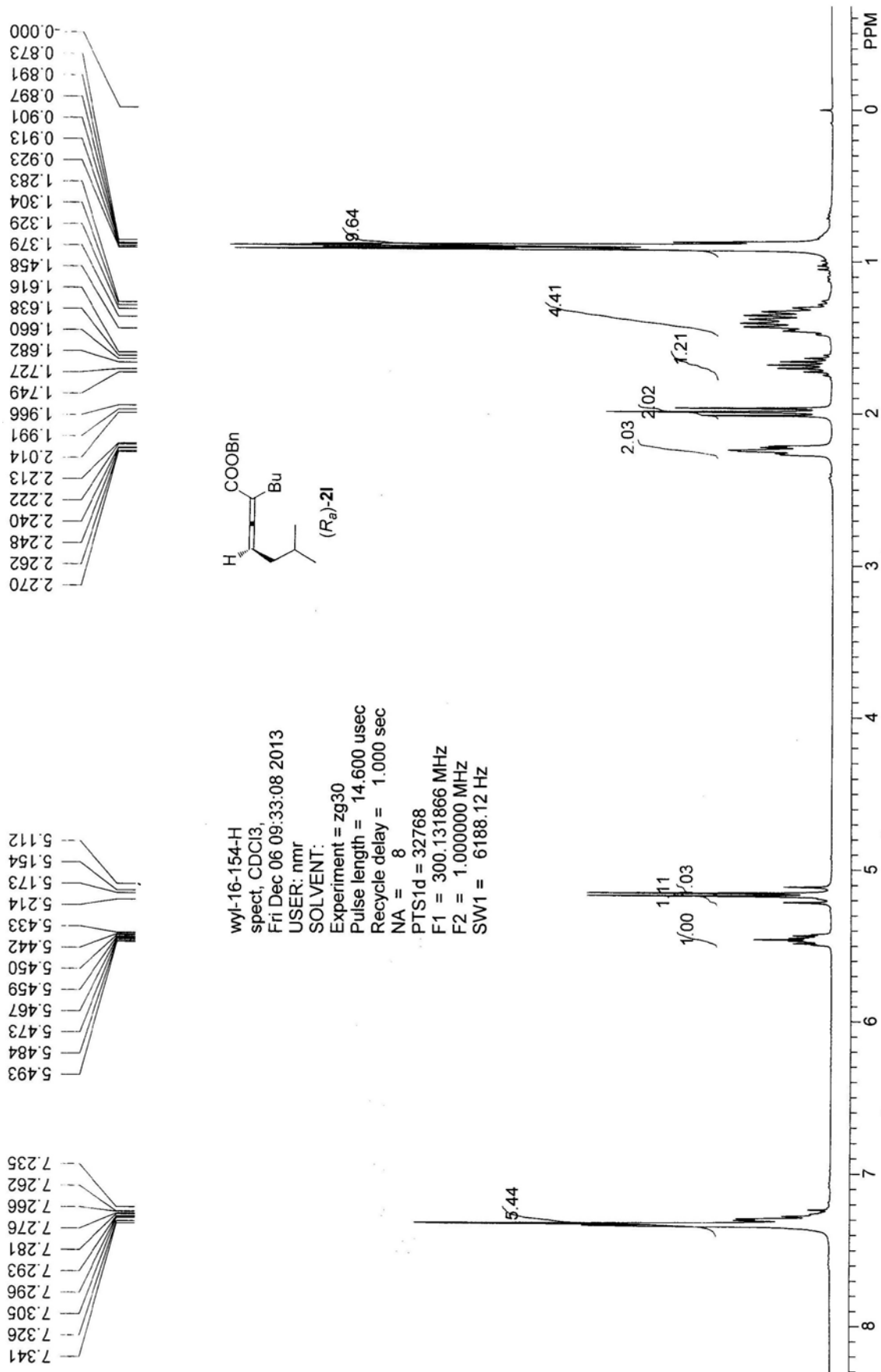
## SAMPLE INFORMATION

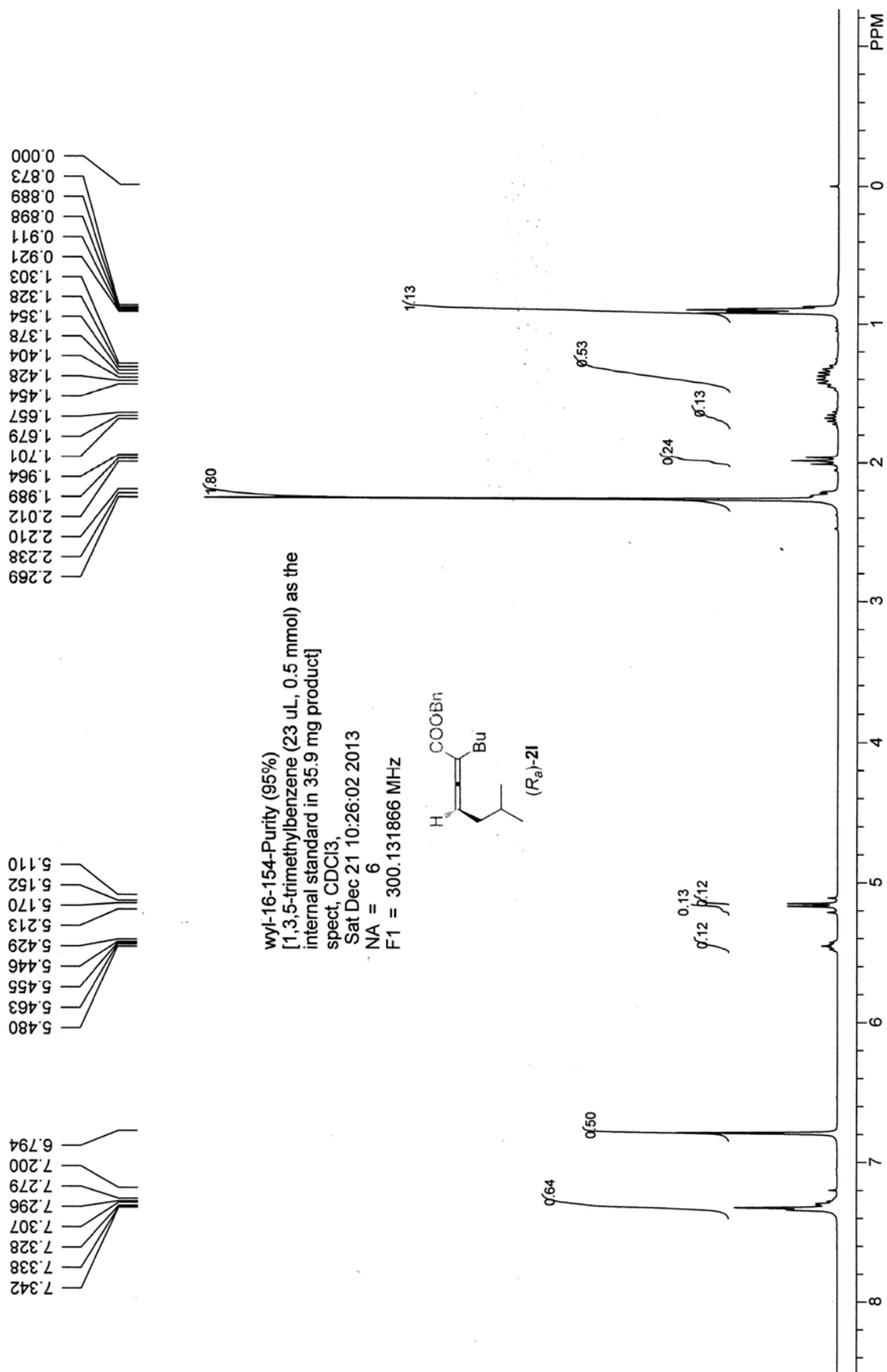
Sample Name:	zwl-2-45-whale-200-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/12/31 9:28:30 CST
Vial:	1	Acq. Method:	zg100
Injection #:	6	Date Processed:	2013/12/31 16:47:05 CST
Injection Volume:	10.00 $\mu$ l	Channel Name:	V2489 ChA
Run Time:	60.00 Minutes	Sample Set Name:	

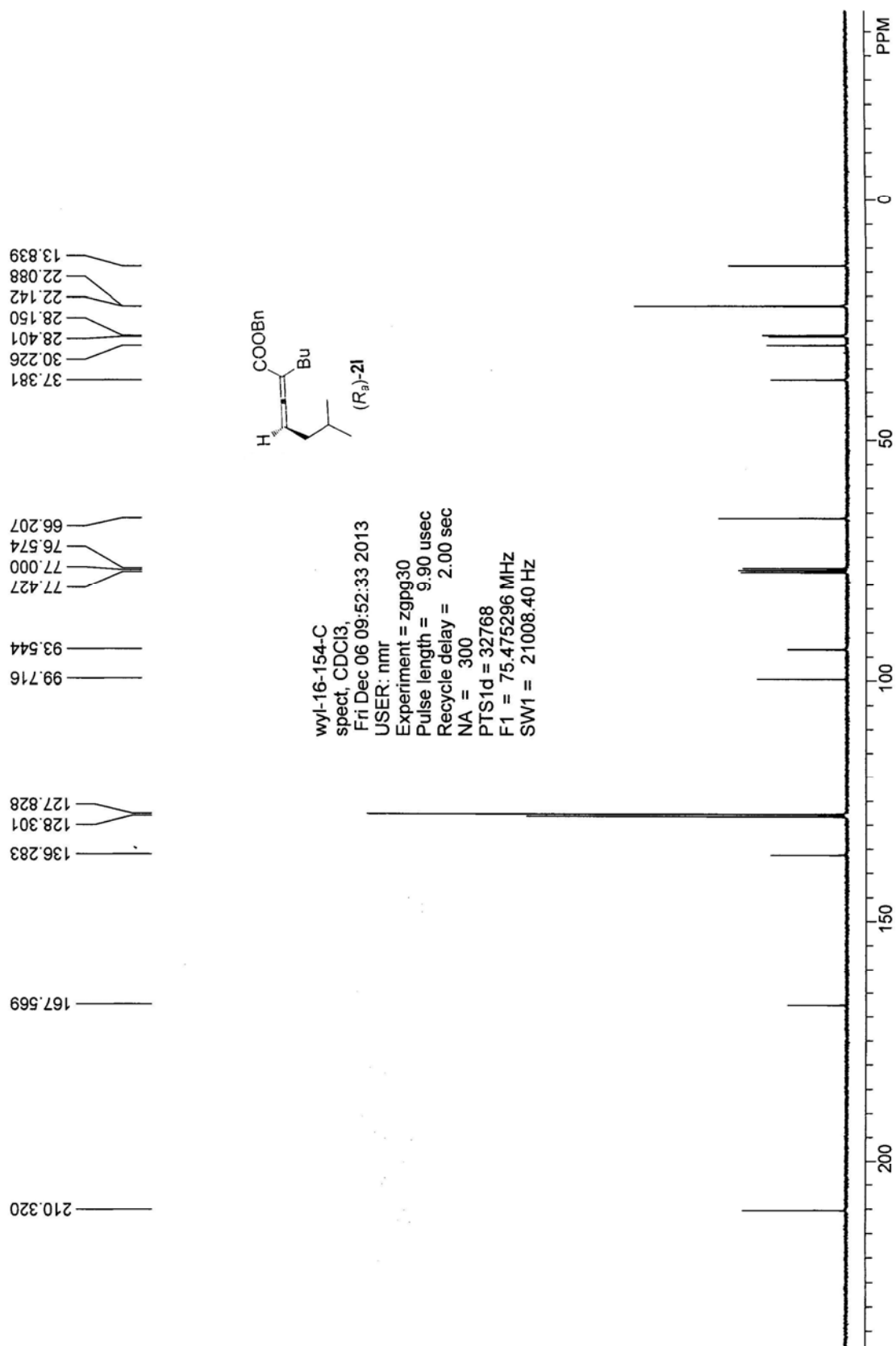


	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	8.878	59740630	49.51	1357028	47.05
2	10.924	60920528	50.49	1527175	52.95









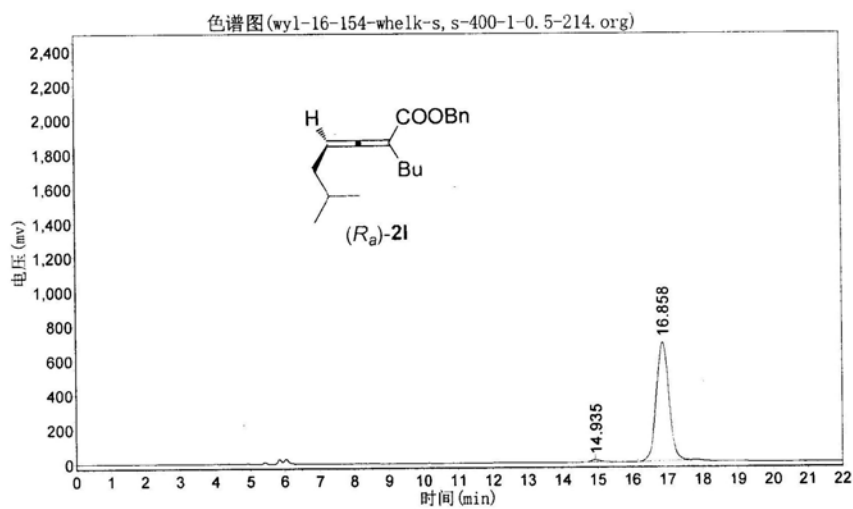
# wyl-16-154-whelk-s, s-400-1-0.5-214

实验时间: 2013/6/28, 15:16:47

报告时间: 2013/6/28, 15:57:30

谱图文件: D:\zhuguangjiong\wyl\20130628\wyl-16-154-whelk-s, s-400-1-0.5-214.org

实验内容简介:  
whelk-s, s 400+1  
0.5ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.935	13307.122	215480.500	1.2561
2		16.858	690324.813	16939586.000	98.7439
总计			703631.935	17155066.500	100.0000

# wyl-16-135-whelk-s, s-400-1-0.5-214

实验时间: 2013/6/28, 15:53:16

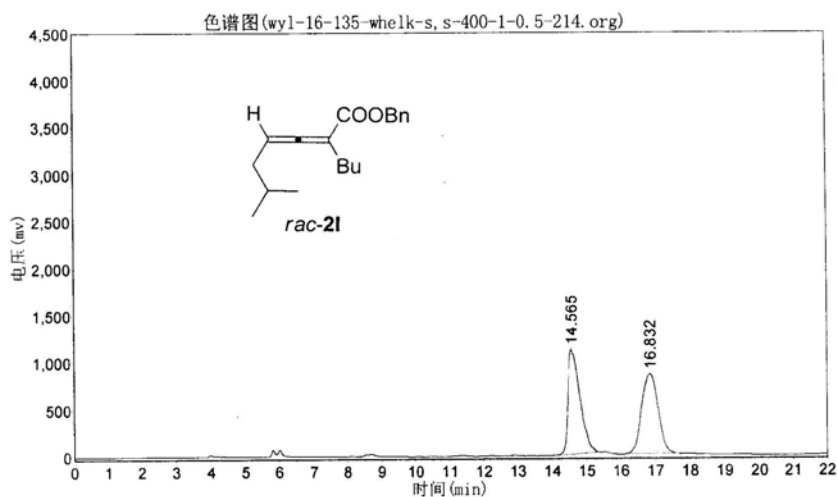
报告时间: 2013/6/28, 16:18:54

谱图文件: D:\zhuguangjiong\wyl\20130628\wyl-16-135-whelk-s, s-400-1-0.5-214.org

实验内容简介:

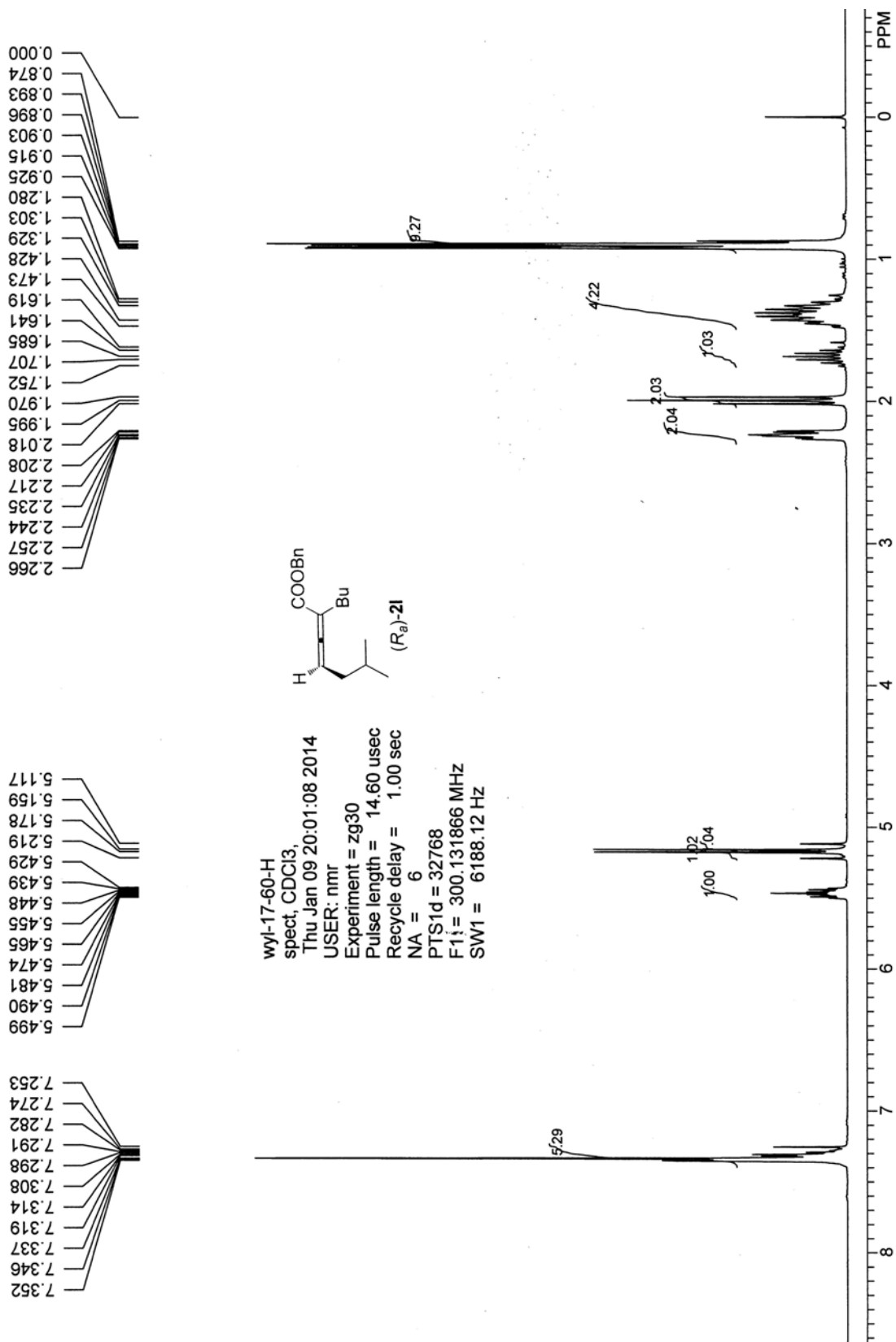
whelk-s, s 400+1

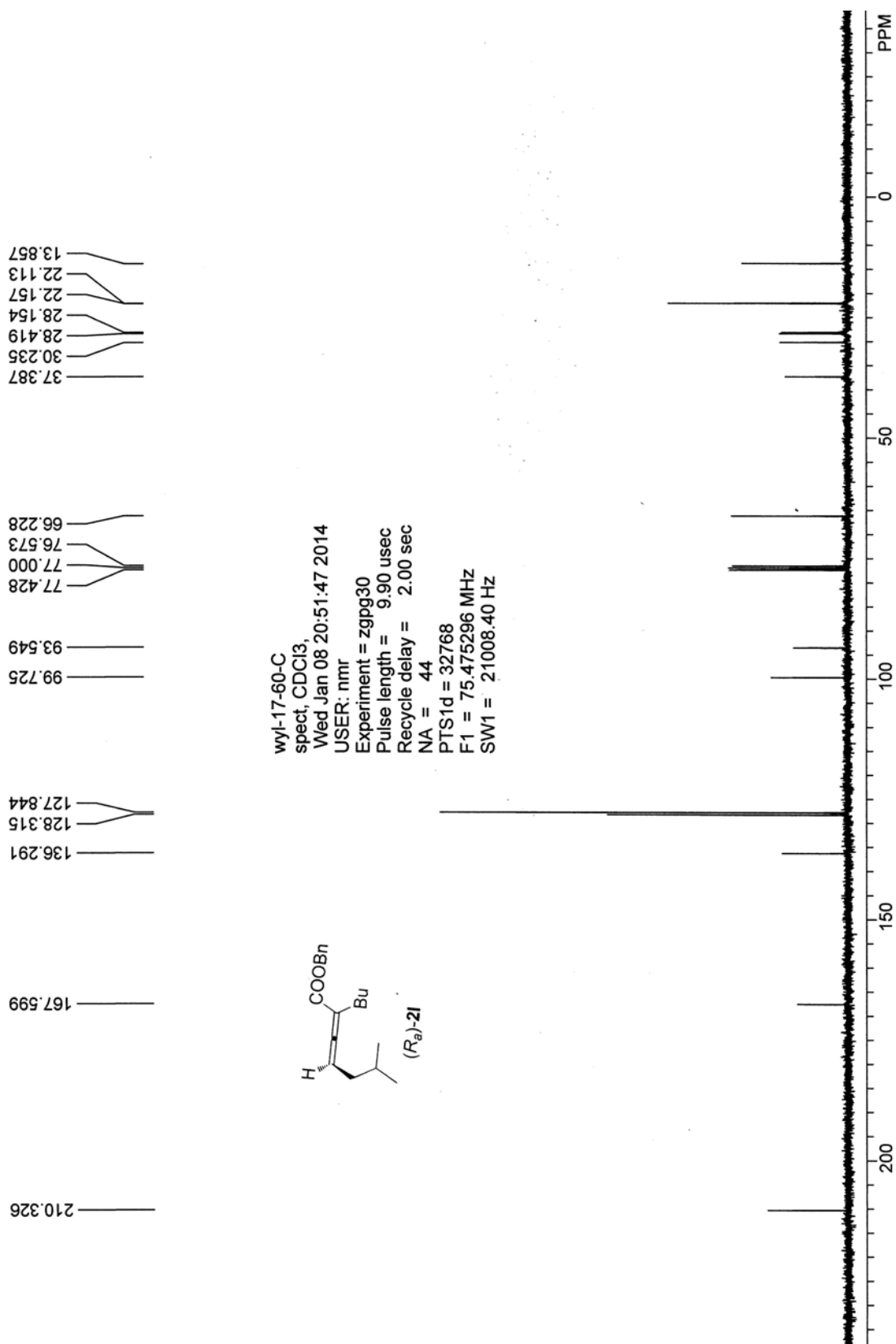
0.5ml/min 214nm



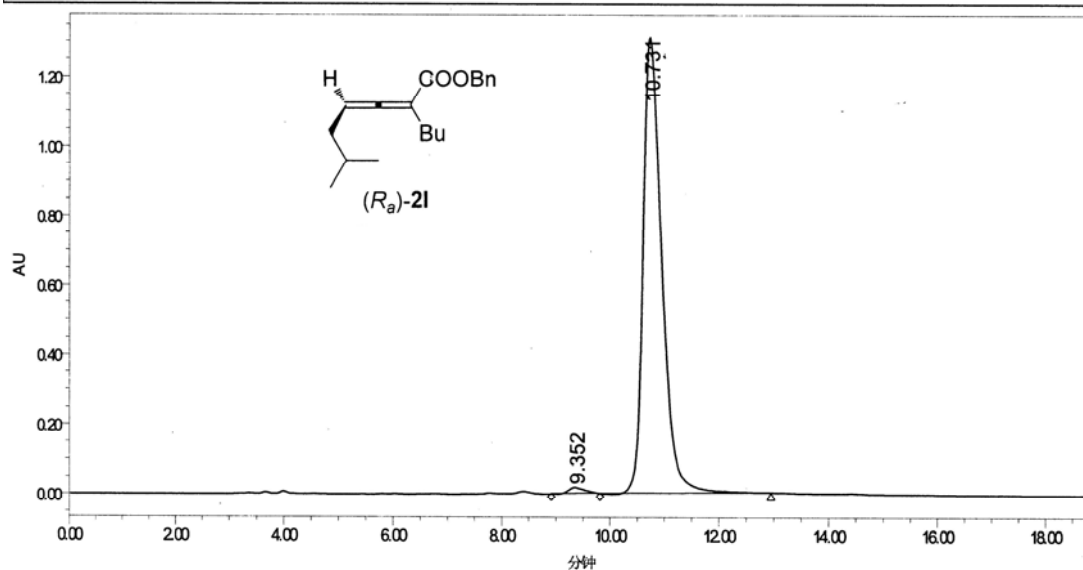
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		14.565	1107220.375	27030012.000	49.5097
2		16.832	842960.688	27565426.000	50.4903
总计			1950181.063	54595438.000	100.0000





## SAMPLE INFORMATION

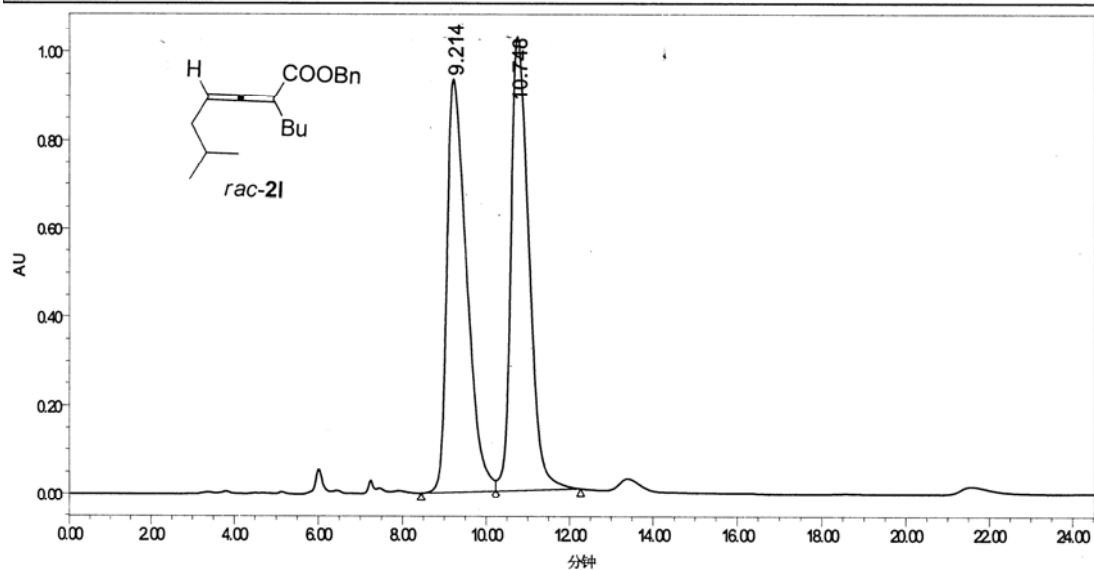
Sample Name: wyl-17-60-whale-400-1-1-214  
Sample Type: 未知  
Vial: 1  
Injection #: 12  
Injection Volume: 10.00  $\mu$ l  
Run Time: 20.00 MinutesAcquired By: Breeze  
Date Acquired: 2013/12/31 11:37:04 CST  
Acq. Method: zg100  
Date Processed: 2013/12/31 12:35:33 CST  
Channel Name: V02489 ChA  
Sample Set Name:

	RT (min)	Area (峰面积)	%Area	Height (峰高)	% Height
1	9.352	344067	1.08	16448	1.24
2	10.731	31370880	98.92	1307711	98.76

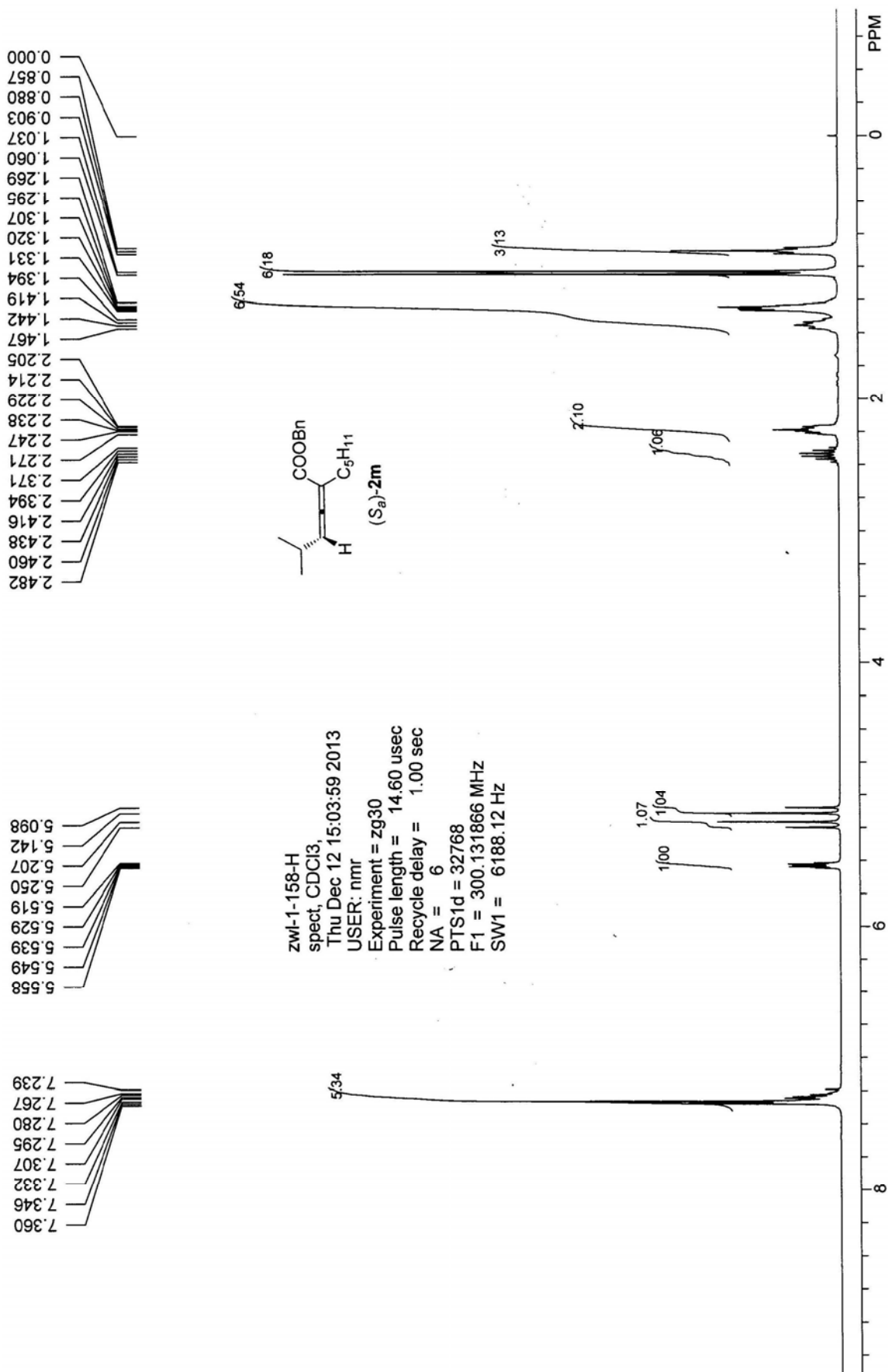


## SAMPLE INFORMATION

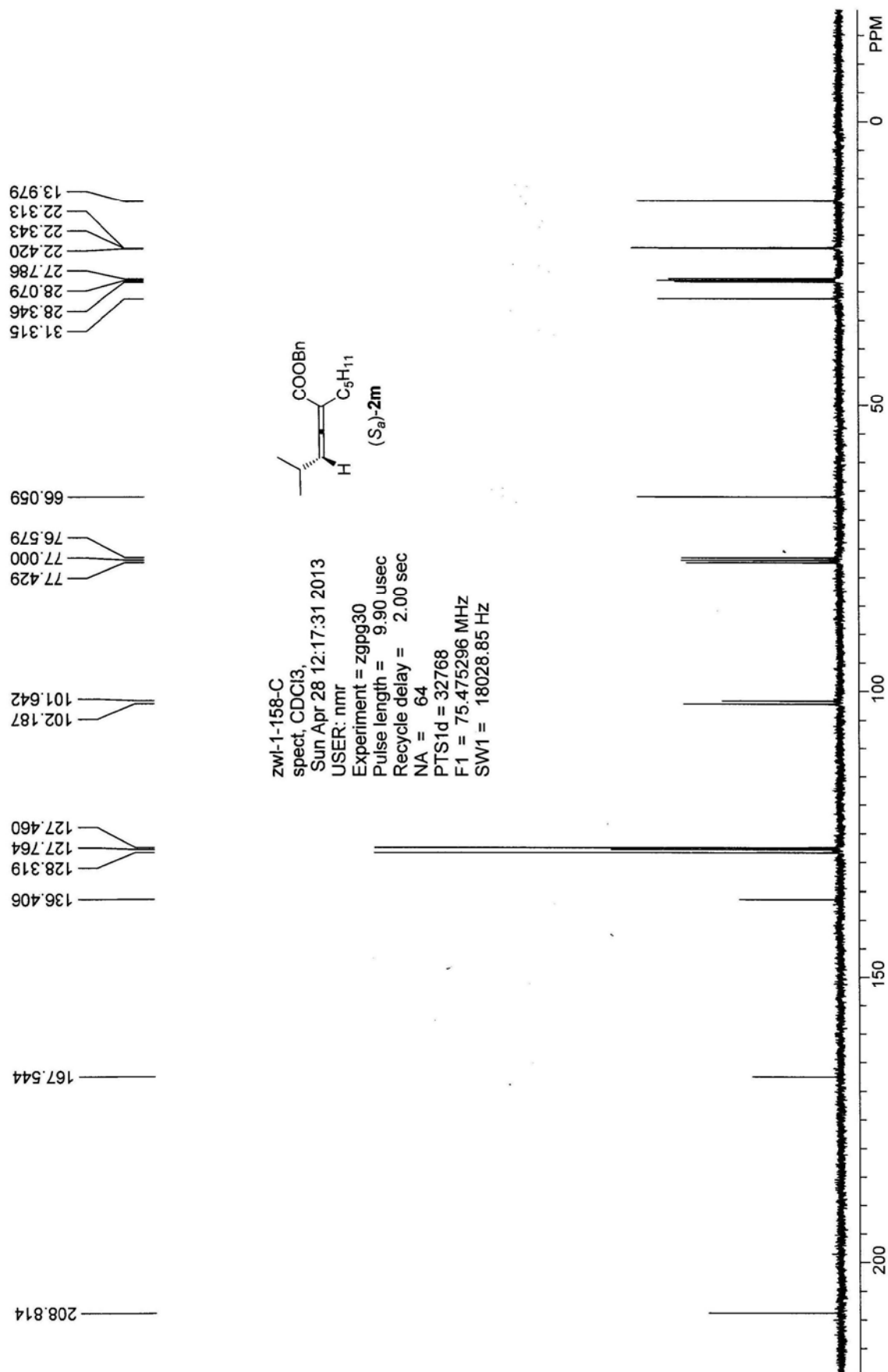
Sample Name:	wyl-16-135-whalk-400-1-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2013/12/31 10:33:43 CST
Vial:	1	Acq. Method:	zg100
Injection#:	9	Date Processed:	2013/12/31 16:46:47 CST
Injection Volume:	10.00 u	Channel Name:	V02489 ChA
Run Time:	60.00 Minutes	Sample Set Name:	



	RT (min)	Area (msec)	%Area	Height (msec)	% Height
1	9.214	30609681	49.79	935143	47.70
2	10.748	30870399	50.21	1025188	52.30



zwl-1-158-H  
 spect, CDCl<sub>3</sub>,  
 Thu Dec 12 15:03:59 2013  
 USER: nmr  
 Experiment = zg30  
 Pulse length = 14.60 usec  
 Recycle delay = 1.00 sec  
 NA = 6  
 PTS1d = 32768  
 F1 = 300.131866 MHz  
 SW1 = 6188.12 Hz



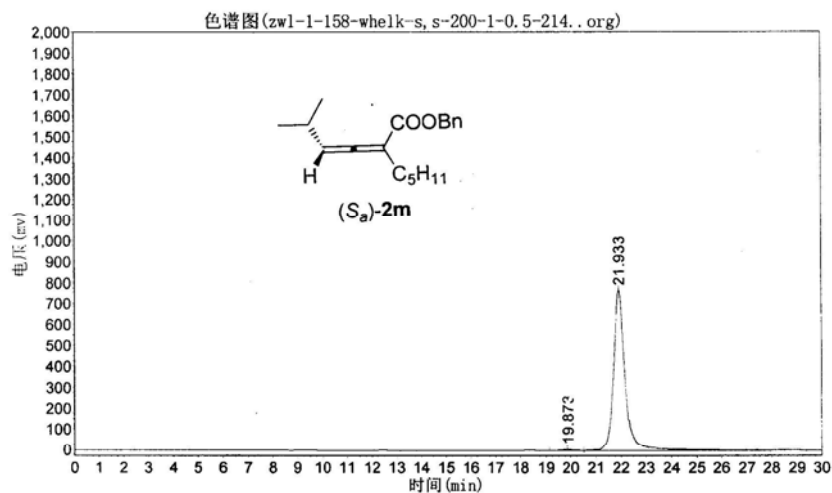
# zw1-1-158-whelk-s, s-200-1-0.5-214

实验时间: 2013-04-28, 13:51:31

报告时间: 2013-04-28, 14:28:39

谱图文件: D:\zhuguangjiong\zw1\20130428\zw1-1-158-whelk-s, s-200-1-0.5-214..org

实验内容简介:  
whelk-s, s 200+1  
0.5ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		19.873	6011.043	160463.172	0.6266
2		21.933	767171.063	25447982.000	99.3734
总计			773182.105	25608445.172	100.0000

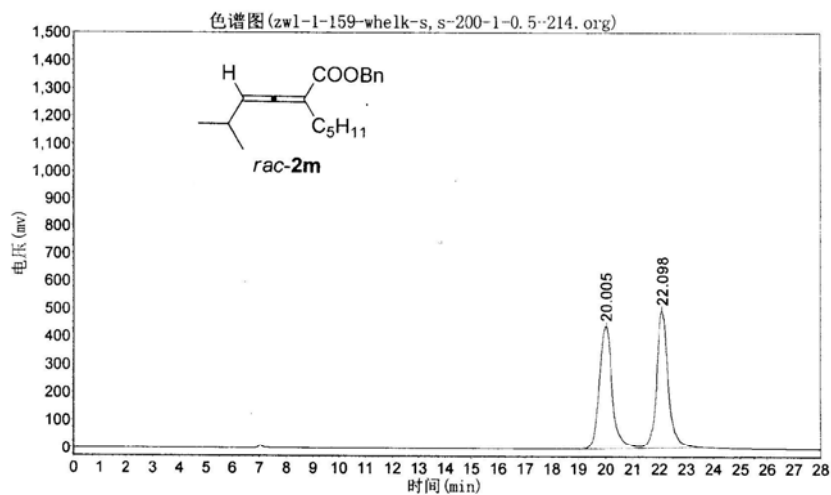
# zw1-1-159-whelk-s, s-200-1-0.5-214

实验时间: 2013-04-28, 12:43:50

报告时间: 2013-04-28, 13:42:19

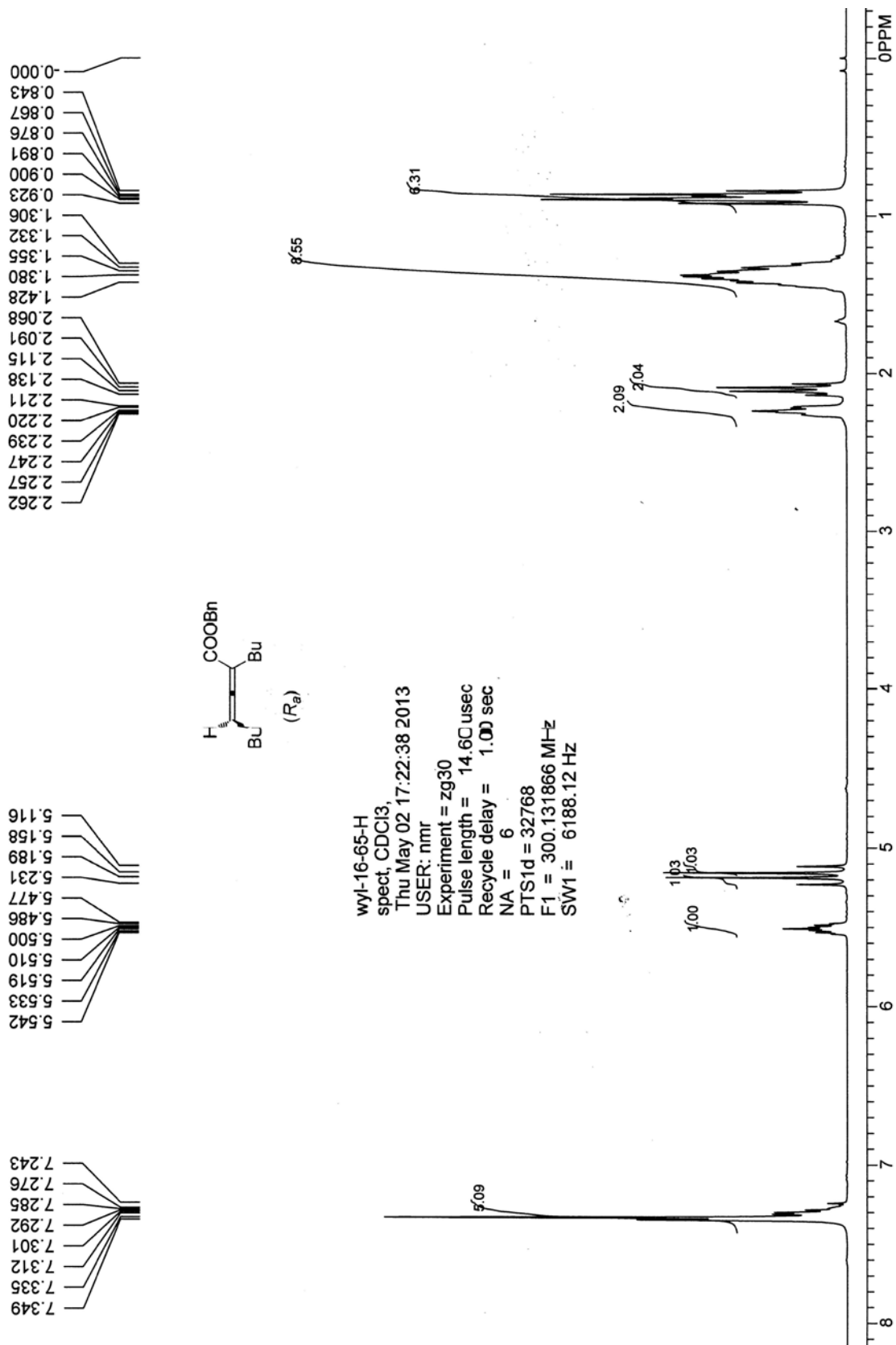
谱图文件: D:\zhuguangjiong\zw1\20130428\zw1-1-159-whelk-s, s-200-1-0.5-214. org

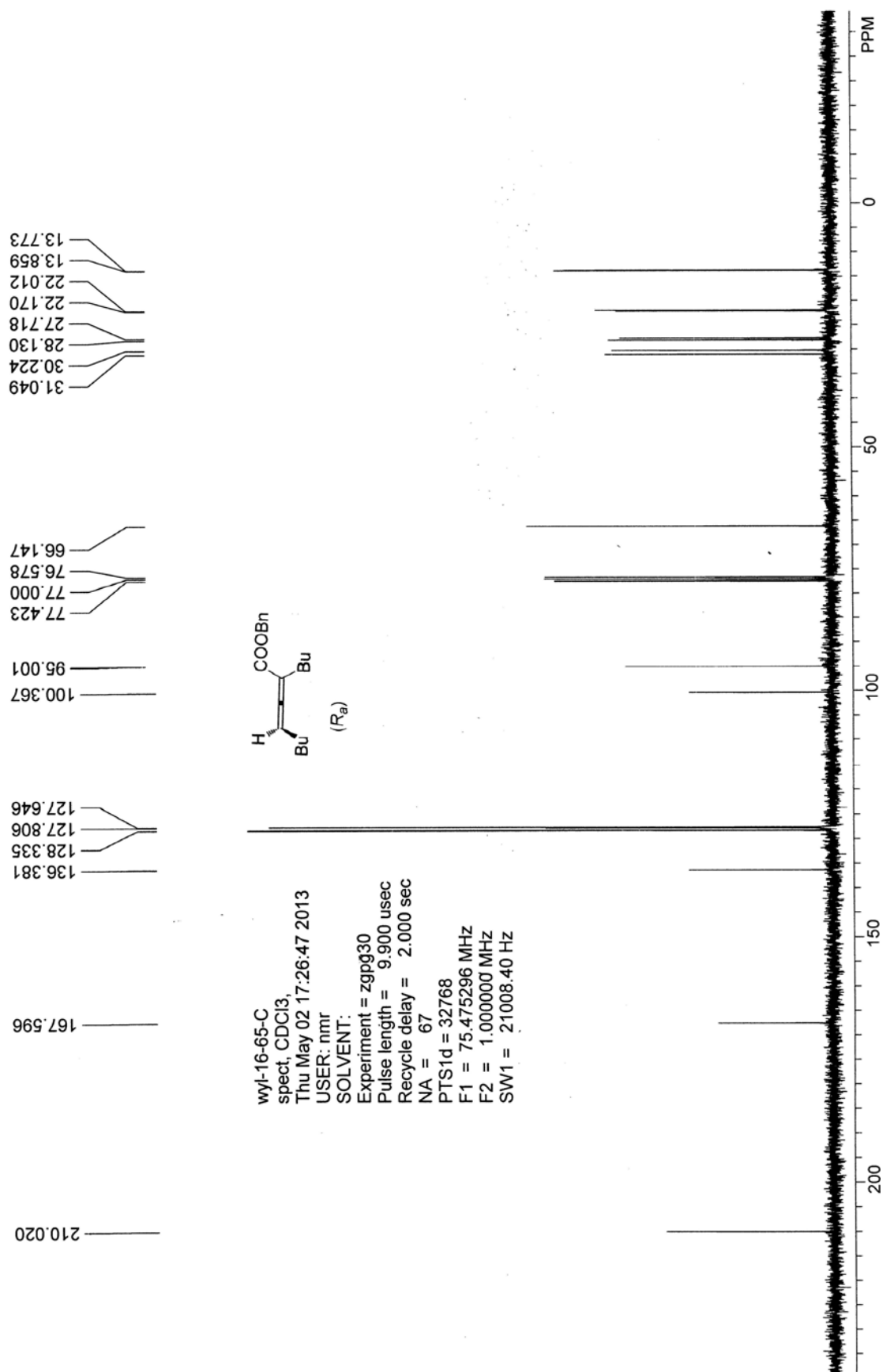
实验内容简介:  
whelk-s, s 200+1  
0.5ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.005	439468.469	14792792.000	49.5197
2		22.098	492610.281	15079771.000	50.4803
总计			932078.750	29872563.000	100.0000



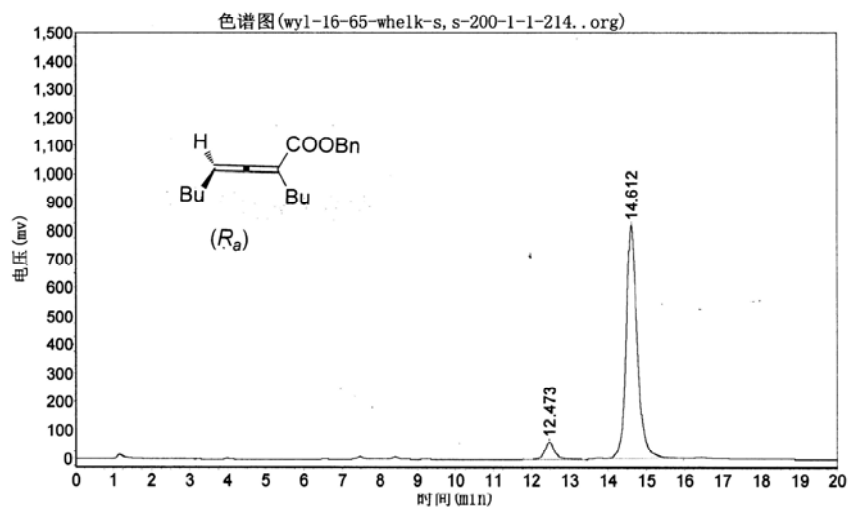


实验时间: 2013-05-02, 11:28:26

报告时间: 2013-05-02, 13:31:47

谱图文件: D:\zhuguangjiong\wyl\20130502\wyl-16-65-wheelk-s, s-200-1-1-214. .org

实验内容简介:  
wheelk-s, s 200+1  
1ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.473	63753.641	1192039.375	6.6915
2		14.612	818372.250	16622253.000	93.3085
总计			882125.891	17814292.375	100.0000

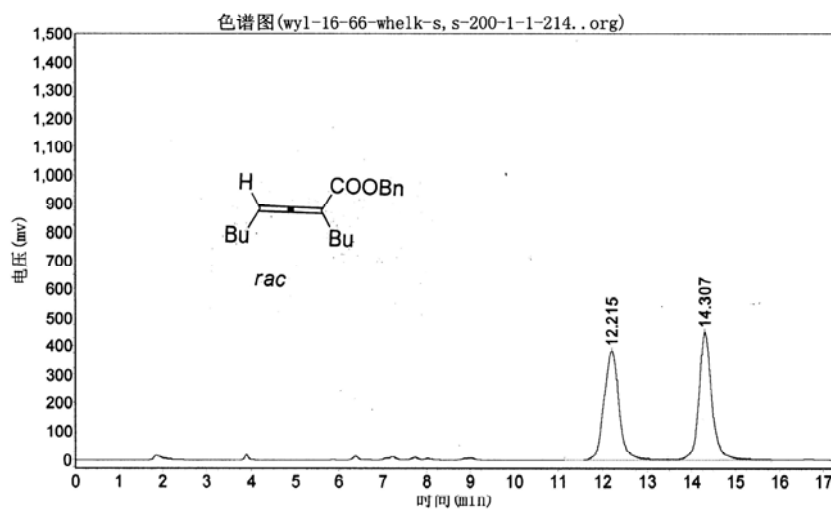


实验时间: 2013-05-02, 12:10:06

报告时间: 2013-05-02, 13:30:09

谱图文件: D:\zhuguangjiong\wyl\20130502\wyl-16-66-wheelk-s, s-200-1-1-214. .org

实验内容简介:  
wheelk-s, s 200+1  
1ml/min 214nm



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.215	381762.719	9162743.000	49.9487
2		14.307	449652.094	9181581.000	50.0513
总计			831414.813	18344324.000	100.0000