

## Supporting Information

### Cyclization Reaction of Cyano-Substituted Unsaturated Esters

#### Prompted by Conjugate Addition of Organoborons

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**General.** Infrared spectra were recorded on a Shimadzu FTIR-8100 spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on a Varian Gemini 2000 ( $^1\text{H}$  at 300 MHz and  $^{13}\text{C}$  at 75 MHz), a Varian Mercury VX400 ( $^1\text{H}$  at 400 MHz and  $^{13}\text{C}$  at 100 MHz), or a JEOL JNM-ECA ( $^1\text{H}$  at 600 MHz and  $^{13}\text{C}$  at 150 MHz) spectrometer using  $\text{CHCl}_3$  ( $^1\text{H}$ ,  $\delta = 7.26$ ) and  $\text{CDCl}_3$  ( $^{13}\text{C}$ ,  $\delta = 77.0$ ) as an internal standard. High-resolution mass spectra were recorded on a JEOL JMS-SX102A spectrometer. All reactions were carried out under a nitrogen atmosphere. Column chromatography was performed with silica gel 60 N (Kanto). Preparative thin-layer chromatography was performed with silica gel 60 PF<sub>254</sub> (Merck).

#### (1a)<sup>1</sup>

IR (nujol): 2222, 1710  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta = 3.84$  (s, 3H), 6.61 (d,  $J = 15.9$  Hz, 1H), 7.48 (t,  $J = 7.7$  Hz, 1H), 7.62 (t,  $J = 7.8$  Hz, 1H), 7.72 (t,  $J = 7.2$  Hz, 2H), 7.98 (d,  $J = 15.9$  Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta = 52.1, 112.7, 117.1, 122.6, 126.9, 130.0, 132.9, 133.5, 137.3, 139.5, 166.2$ ; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{11}\text{H}_9\text{NO}_2$ ,  $M^+$  187.0633. Found  $m/z$  187.0634.

#### (1b)

IR (nujol): 2217, 1721, 1636  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta = 3.87$  (s, 3H), 6.74 (d,  $J = 15.9$  Hz, 1H), 7.60–7.80 (m, 3H), 7.91 (d,  $J = 8.1$  Hz, 1H), 8.05 (d,  $J = 8.4$  Hz, 1H), 8.22 (d,  $J = 15.9$  Hz, 1H), 8.28 (d,  $J = 8.4$  Hz, 1H);  $^{13}\text{C}$  NMR (75 Hz):  $\delta = 52.1, 111.0, 115.8, 122.2, 123.2, 126.0, 128.4, 128.5, 129.2, 132.6, 133.09, 133.15, 137.2, 139.9, 166.2$ ; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{15}\text{H}_{11}\text{NO}_2$ ,  $M^+$  237.0790. Found  $m/z$  237.0786.

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<sup>1</sup> Busacca, C. A.; Johnson, R. E. *Tetrahedron Lett.* **1992**, 33, 165.

**(1c)**

IR (KBr): 2203, 1713, 1642  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.86 (s, 3H), 3.88 (s, 3H), 7.10 (d,  $J$  = 16.2 Hz, 1H), 7.27–7.34 (m, 1H), 7.37–7.41 (m, 2H), 7.73–7.78 (m, 1H), 7.79 (d,  $J$  = 16.2 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 65.4, 70.7, 79.3, 85.4, 86.8, 87.8, 88.6, 88.8, 89.1, 89.8, 90.0, 92.1, 92.7, 99.5; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$ ,  $\text{M}^+$  240.0899. Found  $m/z$  240.0900.

**(1d)**

IR (KBr): 2230, 1725, 1632, 1312, 1175  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.82 (s, 3H), 6.47 (d,  $J$  = 15.9 Hz, 1H), 7.25 (d,  $J$  = 5.1 Hz, 1H), 7.41 (d,  $J$  = 5.7 Hz, 1H), 7.89 (d,  $J$  = 15.9 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 52.1, 112.0, 113.9, 121.6, 128.2, 129.8, 132.8, 147.7, 165.8; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_9\text{H}_7\text{O}_2\text{NS}$ ,  $\text{M}^+$  193.0197. Found  $m/z$  193.0201.

**(1e)<sup>2</sup>**

IR (KBr): 2247, 1713, 1634, 1320, 1169  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.82 (s, 3H), 3.86 (s, 2H), 6.40 (d,  $J$  = 15.9 Hz, 1H), 7.34–7.52 (m, 3H), 7.57–7.62 (m, 1H), 7.82 (d,  $J$  = 15.6 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 21.6, 51.8, 117.0, 121.4, 127.2, 128.7, 128.9, 129.1, 130.5, 133.1, 139.7, 166.5; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{12}\text{H}_{11}\text{NO}_2$ ,  $\text{M}^+$  201.0790. Found  $m/z$  201.0791.

**(1f)**

IR (nujol): 2224, 1653, 1611  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 1.81 (s, 6H), 3.83 (s, 3H), 6.30 (d,  $J$  = 15.6 Hz, 1H), 7.32–7.47 (m, 3H), 7.52–7.56 (m, 1H), 8.50 (d,  $J$  = 15.6 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 28.8, 35.4, 51.9, 121.4, 124.0, 125.4, 128.5, 129.2, 130.1, 133.9, 138.5, 142.7, 166.6; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{14}\text{H}_{15}\text{NO}_2$ ,  $\text{M}^+$  229.1103. Found  $m/z$  229.1106.

**(1g)<sup>3</sup>**

IR (neat): 2249, 1717, 1659, 1269, 1210, 1161  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 1.29 (t,  $J$  = 7.2 Hz, 3H), 2.45–2.60 (m, 4H), 4.20 (q,  $J$  = 7.2 Hz, 2H), 5.95 (d,  $J$  = 15.9 Hz, 1H), 6.90 (dt,  $J$  = 15.6, 6.2 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 13.6, 15.4, 27.1, 59.8, 118.2, 123.1, 143.1, 165.0; HRMS ( $\text{CI}^+$ ): Calcd for  $\text{C}_8\text{H}_{12}\text{NO}_2$ ,  $\text{M}+\text{H}^+$  154.0868. Found  $m/z$  154.0869.

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<sup>2</sup> Kolsaker, P.; Ellingsen, P. O. *Acta Chem. Scand., Series B: Organic Chemistry and Biochemistry* **1979**, B33, 138.

<sup>3</sup> Bhandal, H.; Howell, A. R.; Patel, V. F.; Pattenden, G. J. *Chem. Soc. Perkin Trans. 1: Organic and Bio-Organic Chemistry* **1990**, 2709.

**(1h)<sup>4</sup>**

IR (neat): 2247, 1719, 1655, 1271, 1198, 1156 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz):  $\delta$  = 1.28 (t,  $J$  = 7.1 Hz, 3H), 1.83 (quint,  $J$  = 7.2 Hz, 2H), 2.31–2.43 (m, 4H), 4.18 (q,  $J$  = 7.2 Hz, 2H), 5.88 (d,  $J$  = 15.9 Hz, 1H), 6.87 (dt,  $J$  = 15.6, 6.9 Hz, 1H); <sup>13</sup>C NMR (75 MHz):  $\delta$  = 14.2, 16.6, 23.8, 30.6, 60.4, 118.9, 123.2, 145.6, 166.1; HRMS (FAB<sup>+</sup>): Calcd for C<sub>9</sub>H<sub>14</sub>NO<sub>2</sub>, M+H<sup>+</sup> 168.1025. Found m/z 168.1025.

**General procedure:** To an oven-dried, N<sub>2</sub>-purged flask was added substrate **1** (0.3 mmol, 1.0 equiv), [Rh(OMe)(cod)]<sub>2</sub> (15  $\mu$ mol, 10 mol% of Rh), and a solution of *B*-Ar-9BBN (**2**, 0.6 mmol, 2.0 equiv) in toluene (3.0 mL). The resulting reaction mixture was stirred for 8~17 h at 110 °C. After the reaction mixture was cooled, water (5~10 mL) was added, and the aqueous layer was extracted with ethyl acetate (15 mL x 5). The combined extracts were washed with brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (dichloromethane) to give the product **3**. The second chromatography (hexane:ethyl acetate) was carried out in some cases to remove small amounts of impurities.

**(3aa)**

IR (nujol): 3499, 3349, 1659, 1628, 1536 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz):  $\delta$  = 3.62 (s, 3H), 4.81 (s, 1H), 6.11 (br s, 2H), 7.10–7.14 (m, 2H), 7.14–7.25 (m, 4H), 7.30–7.38 (m, 2H), 7.39–7.44 (m, 1H); <sup>13</sup>C NMR (75 MHz):  $\delta$  = 50.4, 52.1, 102.6, 118.8, 124.8, 126.2, 126.6, 127.5, 128.1, 129.4, 136.7, 141.4, 149.4, 156.9, 167.9; elemental analysis: Calcd for C<sub>17</sub>H<sub>15</sub>NO<sub>2</sub>: C 76.96, H 5.70; found: C 76.86, H 5.64.

**(3ab)**

IR (nujol): 3438, 3337, 1659, 1638, 1545, 1509 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz):  $\delta$  = 3.63 (s, 3H), 3.76 (s, 3H), 4.77 (s, 1H), 6.15 (br s, 2H), 6.80 (d,  $J$  = 8.4 Hz, 2H), 7.04 (d,  $J$  = 8.7 Hz, 2H), 7.16–7.21 (m, 1H), 7.29–7.35 (m, 2H), 7.38–7.44 (m, 1H); <sup>13</sup>C NMR (75 MHz):  $\delta$  = 50.4, 51.4, 55.1, 102.9, 113.6, 118.7, 124.8, 126.6, 128.5, 129.4, 133.4, 136.6, 149.7, 156.7, 158.0, 167.9; HRMS (EI<sup>+</sup>): Calcd for C<sub>18</sub>H<sub>17</sub>NO<sub>3</sub>, M<sup>+</sup> 295.1208. Found m/z 295.1207.

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<sup>4</sup> Rozema, M. J.; Sidduri, A.; Knochel, P. *J. Org. Chem.* **1992**, *57*, 1956.

**(3ac)**

IR (KBr): 3449, 3364, 1673, 1653, 1626  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.62 (s, 3H), 4.76 (s, 1H), 6.11 (br s, 2H), 7.04 (d,  $J$  = 8.1 Hz, 2H), 7.13–7.22 (m, 3H), 7.30–7.45 (m, 3H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 50.5, 51.5, 102.6, 118.9, 124.9, 127.0, 128.3, 129.0, 129.7, 131.9, 136.7, 140.1, 149.0, 156.9, 167.7; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{17}\text{H}_{14}\text{ClNO}_2$ ,  $\text{M}^+$  299.0713. Found  $m/z$  299.0712.

**(3ba)**

IR (KBr): 3403, 3305, 1653, 1628, 1522, 1262, 1100  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.63 (s, 3H), 4.83 (s, 1H), 6.68 (br s, 2H), 7.09–7.27 (m, 5H), 7.30 (d,  $J$  = 8.7 Hz, 1H), 7.52 (t,  $J$  = 7.7 Hz, 1H), 7.63 (t,  $J$  = 7.7 Hz, 1H), 7.80 (d,  $J$  = 8.4 Hz, 1H), 7.92 (d,  $J$  = 7.8 Hz, 1H), 8.33 (d,  $J$  = 8.1 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 50.4, 52.5, 104.7, 121.9, 122.9, 125.4, 126.4, 127.2, 127.8, 128.2, 128.5, 129.6, 130.6, 130.7, 133.3, 140.5, 150.1, 160.8, 168.0; HRMS ( $\text{CI}^+$ ): Calcd for  $\text{C}_{21}\text{H}_{18}\text{NO}_2$ ,  $\text{M}+\text{H}^+$  316.1338. Found  $m/z$  316.1335.

**(3ca)**

IR (KBr): 3391, 3301, 3214, 1624, 1489  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz):  $\delta$  = 3.42 (s, 3H), 3.57 (br s, 3H), 4.77 (s, 1H), 7.10–7.15 (m, 2H), 7.15–7.33 (m, 6H), 7.66–7.71 (m, 1H) ( $-\text{NH}_2$  missing);  $^{13}\text{C}$  NMR (150 MHz):  $\delta$  = 30.7, 46.4, 49.8, 98.5, 110.4, 114.8, 118.5, 120.9, 121.0, 121.6, 126.6, 128.1, 128.3, 138.8, 141.3, 156.7, 159.1, 167.7; HRMS ( $\text{CI}^+$ ): Calcd for  $\text{C}_{20}\text{H}_{19}\text{N}_2\text{O}_2$ ,  $\text{M}+\text{H}^+$  319.1447. Found  $m/z$  319.1445.

**(3da)**

IR (KBr): 3472, 3345, 1655, 1607, 1534  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 3.59 (s, 3H), 4.87 (s, 1H), 6.14 (br s, 2H), 7.03 (d,  $J$  = 4.8 Hz, 1H), 7.08–7.16 (m, 2H), 7.16–7.29 (m, 3H), 7.34 (d,  $J$  = 5.7 Hz, 1H);  $^{13}\text{C}$  NMR (150 MHz):  $\delta$  = 50.1, 50.2, 104.5, 116.9, 126.6, 127.4, 128.3, 129.7, 141.2, 141.8, 155.5, 156.0, 167.1; HRMS ( $\text{FAB}^+$ ): Calcd for  $\text{C}_{15}\text{H}_{13}\text{NO}_2\text{S}$ ,  $\text{M}^+$  271.0667. Found  $m/z$  271.0666.

**(3ea)**

IR (nujol): 3463, 3322, 1668, 1609  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz):  $\delta$  = 3.31 (d,  $J$  = 18.8 Hz, 1H), 3.70 (s, 3H), 3.83 (d,  $J$  = 18.8 Hz, 1H), 5.22 (s, 1H), 7.07–7.21 (m, 8H), 7.30–7.34 (m, 1H) ( $-\text{NH}_2$  missing);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 36.3, 45.4, 50.6, 95.0, 125.7, 126.0, 126.7, 127.2, 127.3, 128.1, 128.3, 131.8, 140.1, 145.9, 156.9, 169.5; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{18}\text{H}_{17}\text{NO}_2$ ,  $\text{M}^+$  279.1259. Found  $m/z$  279.1260.

**(3fa)** To an oven-dried, N<sub>2</sub>-purged flask was added a solution of **1f** (44.0 mg, 0.19 mmol) in 1,4-dioxane (2.0 mL), 2-phenyl[1,3,2]dioxaborolane **2a'** (59.0 mg, 0.40 mmol, 2.0 equiv), [Rh(OMe)(cod)]<sub>2</sub> (4.7 mg, 9.7 μmol, 10 mol% of Rh), and H<sub>2</sub>O (0.7 μL, 39 μmol, 0.2 equiv). The resulting reaction mixture was stirred for 13 h at 100 °C. After the reaction mixture was cooled, the reaction was quenched with water (5~10 mL). The aqueous layer was extracted with ethyl acetate (15 mL x 5). The combined extracts were washed with brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (dichloromethane) to give the product **3fa** (46.9 mg, 0.15 mmol, 80%): IR (nujol): 3426, 3312, 1653, 1624, 1522 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz): δ = 1.63 (s, 3H), 1.65 (s, 3H), 3.59 (s, 3H), 5.16 (s, 1H), 6.76 (br s, 2H), 7.04–7.14 (m, 2H), 7.15–7.23 (m, 6H), 7.33–7.39 (m, 1H); <sup>13</sup>C NMR (75 MHz): δ = 29.4, 31.9, 39.2, 44.5, 50.5, 93.1, 125.4, 125.8, 126.1, 126.2, 127.9, 128.0, 129.5, 136.9, 140.0, 147.9, 162.0, 170.3; HRMS (EI<sup>+</sup>): Calcd for C<sub>20</sub>H<sub>21</sub>NO<sub>2</sub>, M<sup>+</sup> 307.1572. Found m/z 307.1570.

**(3ga)**

[α]<sub>D</sub><sup>26</sup> – 9.51 (c 1.365, CHCl<sub>3</sub>) for the sample of 95% ee.

IR (KBr): 3478, 3335, 1651, 1620, 1566 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz): δ = 1.01 (t, *J* = 7.2 Hz, 3H), 1.70–1.82 (m, 1H), 2.30–2.50 (m, 2H), 2.62–2.77 (m, 1H), 3.91–4.05 (m, 2H), 4.06–4.13 (m, 1H), 5.83 (br s, 2H), 7.12–7.29 (m, 5H); <sup>13</sup>C NMR (75 MHz): δ = 14.2, 31.4, 33.6, 48.3, 58.4, 99.0, 125.4, 126.8, 127.8, 147.6, 162.6, 167.8; HRMS (EI<sup>+</sup>): Calcd for C<sub>14</sub>H<sub>17</sub>NO<sub>2</sub>, M<sup>+</sup> 231.1259. Found m/z 231.1257.

**(3gb)**

[α]<sub>D</sub><sup>23</sup> – 1.05 (c 1.045, CHCl<sub>3</sub>) for the sample of 89% ee.

IR (neat): 3446, 3320, 1651, 1634, 1557 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz): δ = 1.04 (t, *J* = 7.1 Hz, 3H), 1.65–1.78 (m, 1H), 2.26–2.48 (m, 2H), 2.58–2.77 (m, 1H), 3.77 (s, 3H), 3.93–4.08 (m, 3H), 5.71 (br s, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 7.09 (d, *J* = 8.7 Hz, 2H); <sup>13</sup>C NMR (75 MHz): δ = 14.4, 31.6, 33.6, 47.5, 55.2, 58.5, 99.4, 113.3, 127.8, 139.7, 157.5, 162.3, 167.9; HRMS (EI<sup>+</sup>): Calcd for C<sub>15</sub>H<sub>19</sub>NO<sub>3</sub>, M<sup>+</sup> 261.1365. Found m/z 261.1362.

**(3gc)**

[α]<sub>D</sub><sup>23</sup> – 5.36 (c 0.765, CHCl<sub>3</sub>) for the sample of 95% ee.

IR (KBr): 3422, 3314, 1659, 1624, 1547 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz): δ = 1.02 (t, *J* = 7.2 Hz, 3H),

1.63–1.75 (m, 1H), 2.28–2.52 (m, 2H), 2.61–2.76 (m, 1H), 3.91–4.09 (m, 3H), 5.76 (br s, 2H), 7.10 (d,  $J = 8.4$  Hz, 2H), 7.21 (d,  $J = 8.4$  Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta = 14.3, 31.3, 33.6, 47.8, 58.6, 98.7, 128.0, 128.2, 131.0, 146.2, 162.6, 167.7$ ; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{14}\text{H}_{16}\text{ClNO}_2$ ,  $\text{M}^+$  265.0870. Found  $m/z$  265.0877.

**(4ha)<sup>5</sup>**

keto:enol=45:55; IR (neat): 2940, 1744, 1715, 1647, 1617  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz):  $\delta = 0.94$  (t,  $J = 7.2$  Hz, 1.65H), 1.04 (t,  $J = 7.2$  Hz, 1.35H), 1.50–2.00 (m, 3H), 2.04–2.62 (m, 3H), 3.38 (dt,  $J = 11.7, 3.7$  Hz, 0.45H), 3.67 (d,  $J = 12.6$  Hz, 0.45H), 3.86–3.93 (m, 0.55H), 3.93–4.08 (m, 2H), 7.12–7.34 (m, 5H), 12.57 (s, 0.55H);  $^{13}\text{C}$  NMR (150 MHz):  $\delta = 13.8, 13.9, 17.2, 25.4, 29.1, 31.6, 33.0, 38.6, 41.1, 47.6, 60.0, 60.7, 63.7, 100.0, 125.6, 126.98, 127.01, 127.5, 127.9, 128.6, 142.2, 146.4, 168.7, 172.4, 173.6, 205.3$ ; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_3$ ,  $\text{M}^+$  246.1256. Found  $m/z$  246.1253.

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<sup>5</sup> Bunce, R. A.; Harris, C. R. *J. Org. Chem.* **1992**, *57*, 6981.

**Asymmetric procedure:** To an oven-dried, N<sub>2</sub>-purged flask was added [RhCl(C<sub>2</sub>H<sub>4</sub>)<sub>2</sub>]<sub>2</sub> (3.9 mg, 10 μmol, 10 mol% of Rh), (*R*)-H<sub>8</sub>-BINAP (12.6 mg, 20 μmol, 10 mol%), KOH (5.48 mg, 98 μmol, 0.5 equiv), and a solution of *B*-Ph-9BBN (**2a**, 77.8 mg, 0.39 mmol, 2.0 equiv) in toluene (1.0 mL). The resulting reaction mixture was stirred for 30 min at 70 °C, and then a solution of **1g** (30.5 mg, 0.2 mmol) in toluene (1.0 mL) was added. After stirring for 12 h at 70 °C, the reaction was quenched with water (5~10 mL). The aqueous layer was extracted with ethyl acetate (15 mL x 5). The combined extracts were washed with brine and dried over MgSO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (dichloromethane). The second chromatography (hexane:ethyl acetate=3:1) was carried out to remove small amounts of impurities, leading to the product **3ga** (30.2 mg, 0.13 mmol, 66%, 95% ee).

**3ga:** The ee was determined on a Daicel Chiralcel OD-H column with hexane:isopropanol = 9:1, flow rate = 0.6 mL/min, λ = 220 nm. Retention times: 13.0 min, 14.6 min.

**3gb:** The ee was determined on a Daicel Chiralcel AS-H column with hexane:isopropanol = 9:1, flow rate = 0.6 mL/min, λ = 220 nm. Retention times: 15.8 min, 18.6 min.

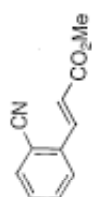
**3gc:** The ee was determined on a Daicel Chiralcel OD-H column with hexane:isopropanol = 9:1, flow rate = 0.6 mL/min, λ = 220 nm. Retention times: 14.2 min, 15.5 min.

(6) To an oven-dried, N<sub>2</sub>-purged flask was added a solution of **3ga** (49.7 mg, 0.22 mmol) in DMF (2 mL) and NaH (9.3 mg, 0.39 mmol, 1.8 equiv) at 0 °C. The resulting reaction mixture was stirred for 50 min at 0 °C, and then phenyl isocyanate (36.0 mg, 0.3 mmol, 1.4 equiv) was added at 0 °C. After stirring for 1 h at room temperature, the reaction was quenched with water (5~10 mL). The solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (dichloromethane) to give the product **6** (52.2 mg, 0.17 mmol, 80%): IR (KBr): 3200, 1721, 1650, 1418 cm<sup>-1</sup>; <sup>1</sup>H NMR (600 MHz): δ = 1.92–2.00 (m, 1H), 2.38–2.44 (m, 1H), 2.46–2.55 (m, 1H), 2.58–2.66 (m, 1H), 4.24–4.29 (m, 1H), 7.14–7.21 (m, 5H), 7.25–7.29 (m, 2H), 7.35–7.39 (m, 1H), 7.42–7.46 (m, 2H), 10.13 (br s, 1H); <sup>13</sup>C NMR (150 MHz): δ = 30.5, 32.0, 46.6, 114.3, 126.5, 126.9, 128.4, 128.5, 128.6, 129.1, 134.9, 143.8, 153.3, 154.9, 160.8; HRMS (EI<sup>+</sup>): Calcd for C<sub>19</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>, M<sup>+</sup> 304.1212. Found m/z 304.1212.

(7) To an oven-dried, N<sub>2</sub>-purged flask was added a solution of **3ea** (49.0 mg, 0.175 mmol) in benzene and DDQ (60.2 mg, 0.265 mmol, 1.5 equiv). The resulting reaction mixture was stirred

for 2 h at 80 °C. After the reaction mixture was cooled, the solvent was removed under reduced pressure and the residue was purified by preparative thin-layer chromatography (dichloromethane) to give the product **7** (29.6 mg, 0.11 mmol, 61%): IR (KBr): 3457, 3370, 1715, 1622  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz):  $\delta$  = 3.40 (s, 3H), 4.57 (br s, 2H), 7.08 (s, 1H), 7.10–7.17 (m, 1H), 7.29–7.49 (m, 7H), 7.61 (d,  $J$  = 8.1 Hz, 1H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 51.7, 109.9, 120.8, 123.0, 125.7, 126.4, 127.3, 127.4, 127.6, 127.8, 129.7, 135.6, 139.2, 141.4, 142.2, 169.6; HRMS ( $\text{EI}^+$ ): Calcd for  $\text{C}_{18}\text{H}_{15}\text{NO}_2$ ,  $\text{M}^+$  277.1103. Found  $m/z$  277.1104.





1a

STANDARD IN OBSERVE

Pulse sequence: zgpg30

Relaxed: 0.013

Acquired temperature

000120-21000 "VET1002"

Solvent delay: 1.000 sec

Pulse: 45.0 degrees

Acq. time: 3.260 sec

Width: 9000.0 Hz

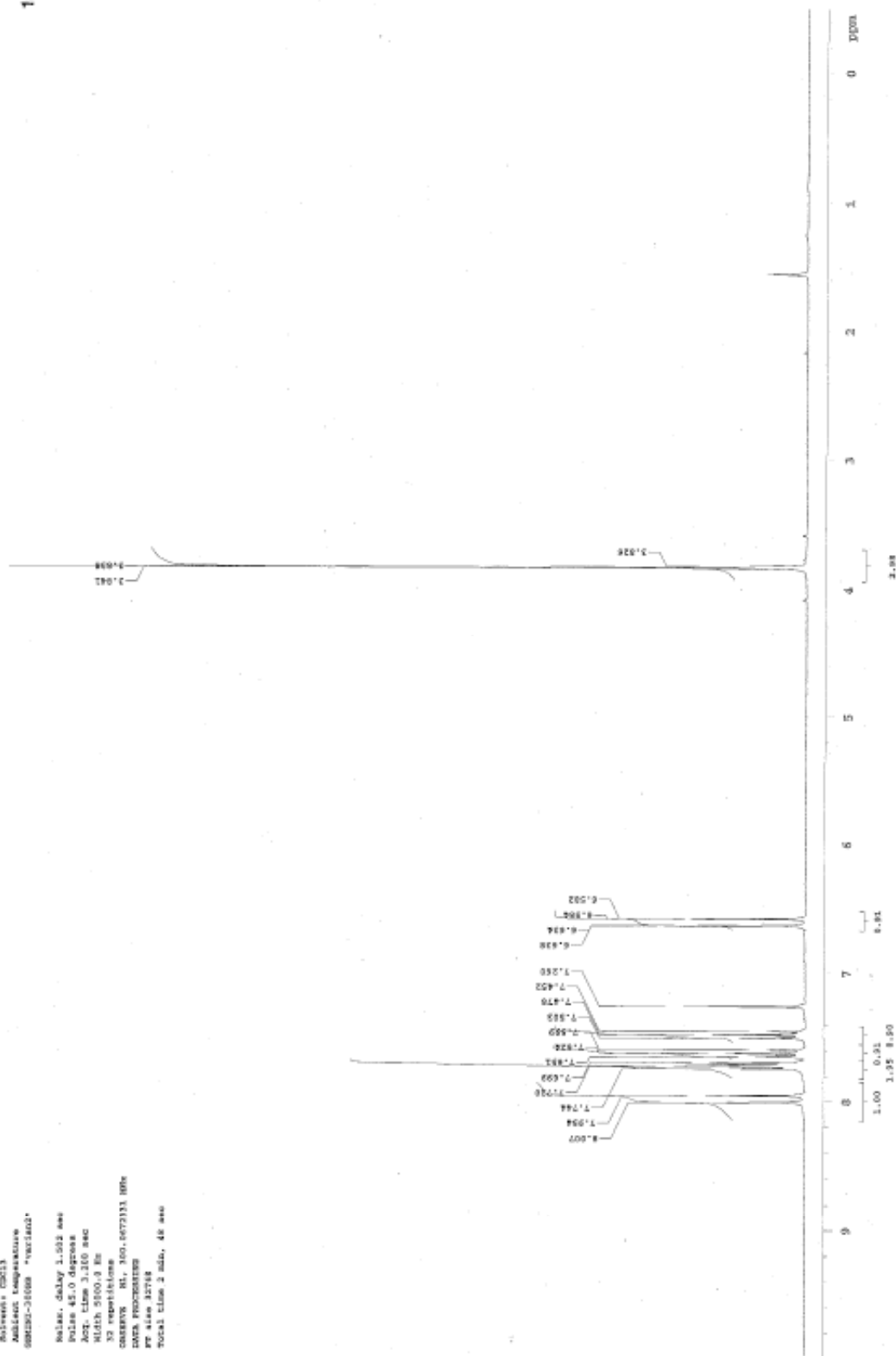
32 repetitions

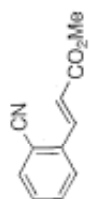
CHURN: 01, 160, 0072133 000

DATA PROCESSING

PT: also 12714

Total time: 3 min, 48 sec





1a

13C QM824VW

Pulse sequence: zgpg30

Solvent: CDCl3

Acquire temperature:

CDCl3-1008 "various"

Relax. delay 1.116 sec

Pulse 45.0 degree

Acq. time 8.642 sec

Relax 1008.0 Hz

1324 repetitions

CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"

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CDCl3-1008 "various"

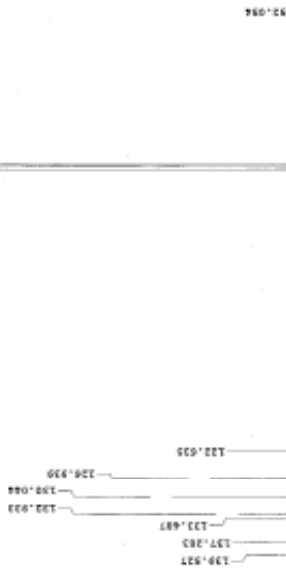
CDCl3-1008 "various"

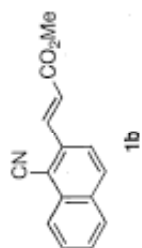
CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"

CDCl3-1008 "various"





# STANDARD 1H NMR

Pulse sequence: zgpg30

Solvent: CDCl<sub>3</sub>

Acidant temperature

CHCl<sub>3</sub>-d<sub>3</sub> 100% "varian2"

Relax. delay: 1.500 sec

Pulse: zgpg30

Acq. time: 3.200 sec

Width: 10000.0 Hz

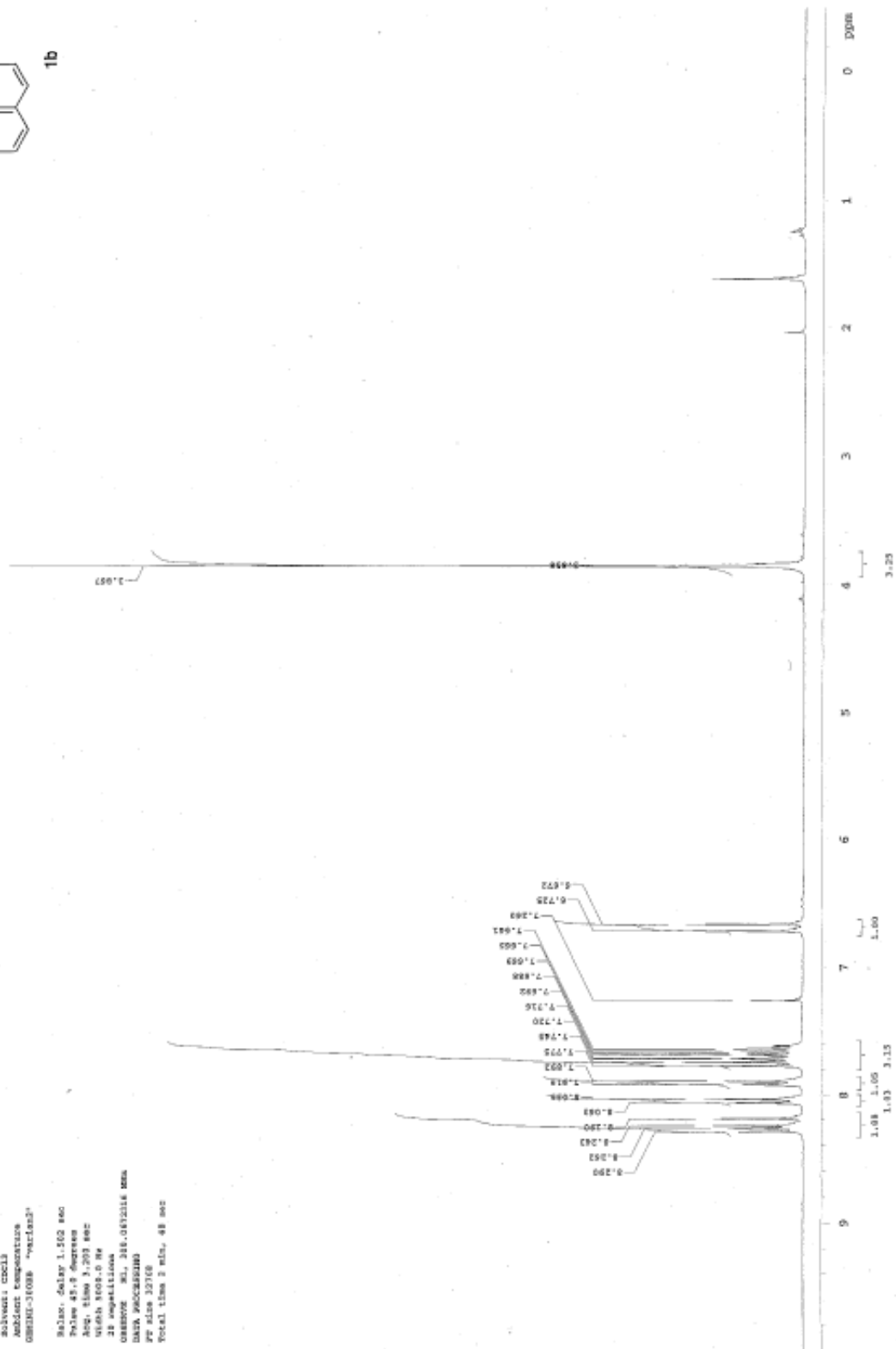
28 acquisitions

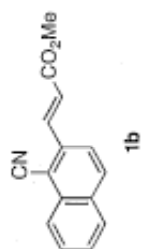
OBSERVE: M1, 316.067216 MHz

DATA PROCESSING

FT size: 32768

Total time: 2 min, 48 sec





11C C0080478

Pulse sequence: zgpg30

Software: CNO13

Acquisition temperature

000000-000000 "Varian2"

Pulse delay 1.100 sec

Relax 45.0 degrees

Acq. time 0.042 sec

WZCH 10000.0 Hz

1300 repetitions

00000000 033. 75.451675 MHz

00000000 033. 310.000135 MHz

Power 30 dB

continuously on

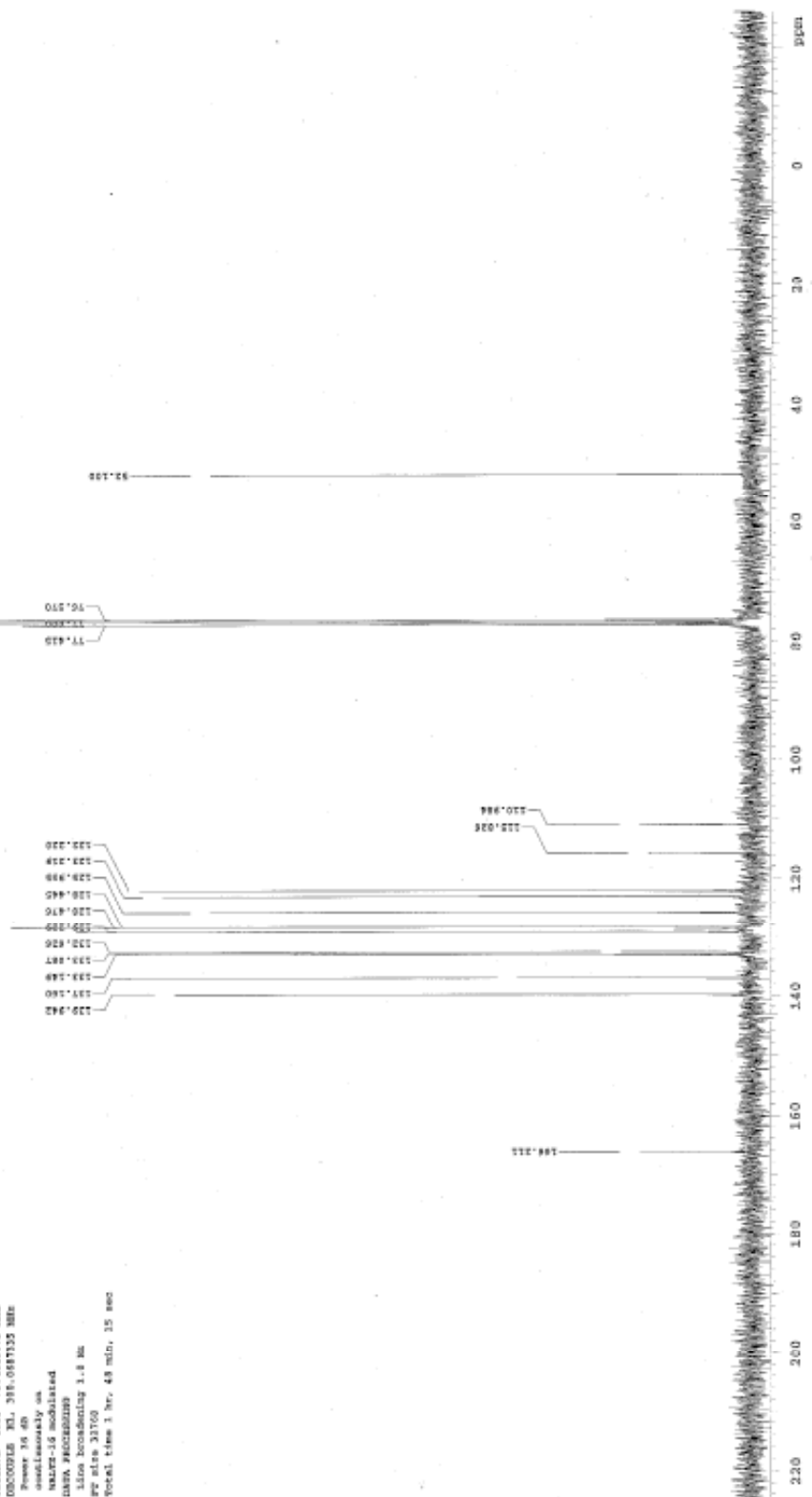
NAME: 15 mobilized

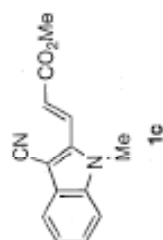
DATA PROCESSING

Line resolution 1.0 Hz

Processing 32768

Total time 1 hr. 40 min. 35 sec





STANDARD IN CHROMAT

Pulse Sequence: zgpg30

solvent: CDCl3

Acquisition Date: 20030313

Acquisition Time: 10:00:00

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Acquisition Time: 10:00:00

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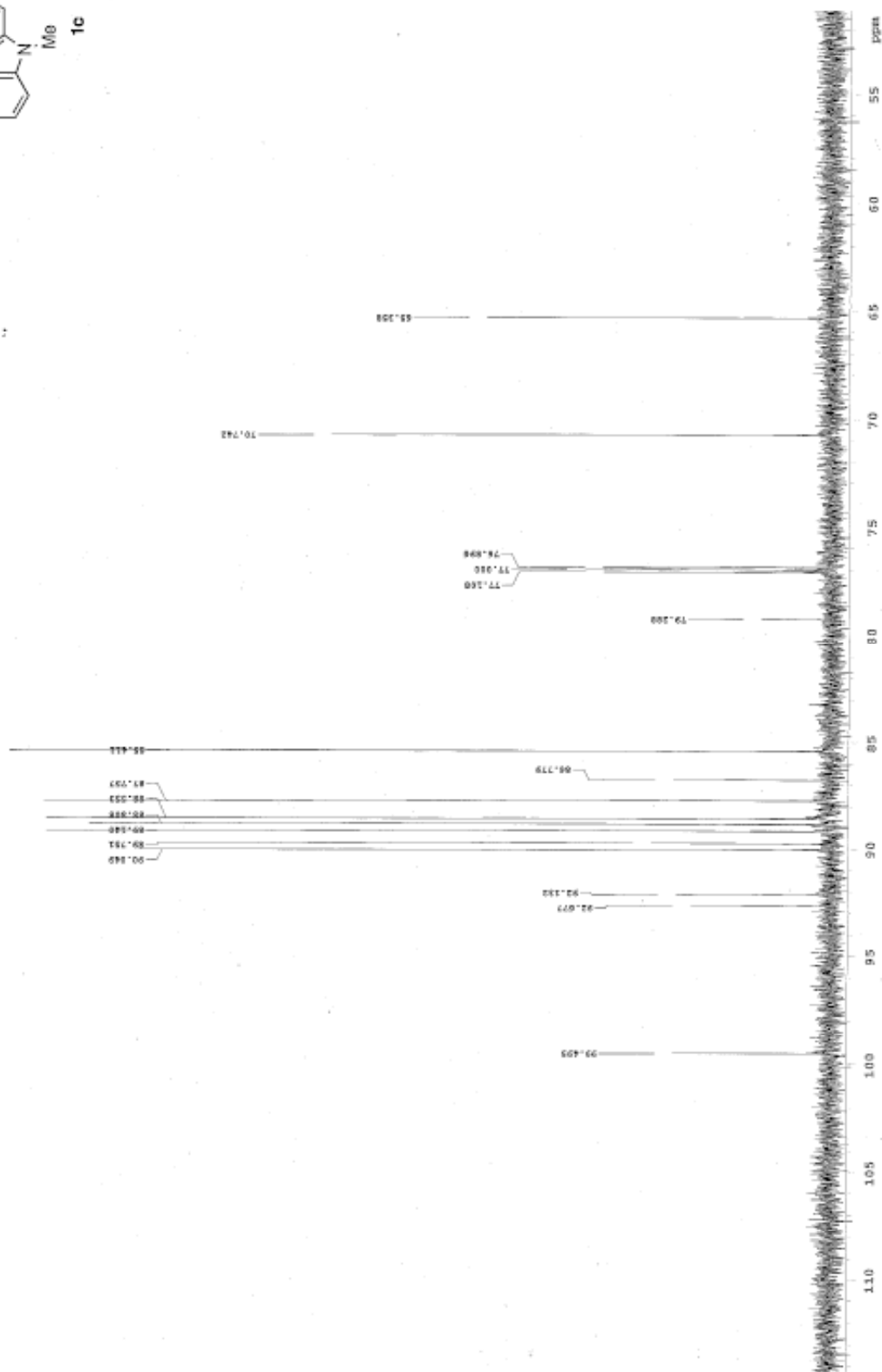
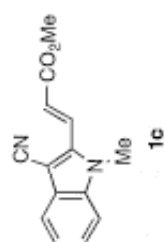
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Acquisition Date: 20030313

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Acquisition Time: 10:00:00

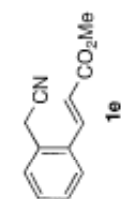












13C NMR

Pulse Sequence: zgpg30

Solvent: CDCl<sub>3</sub>

Acquire Temperature: 300.2 K

NAME: 1e

EXPNO: 300

F2 - Acquisition Date: 20130801

F2 - Acquisition Time: 14.00

F2 - Acquisition Date: 20130801

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F2 - Acquisition Date: 20130801

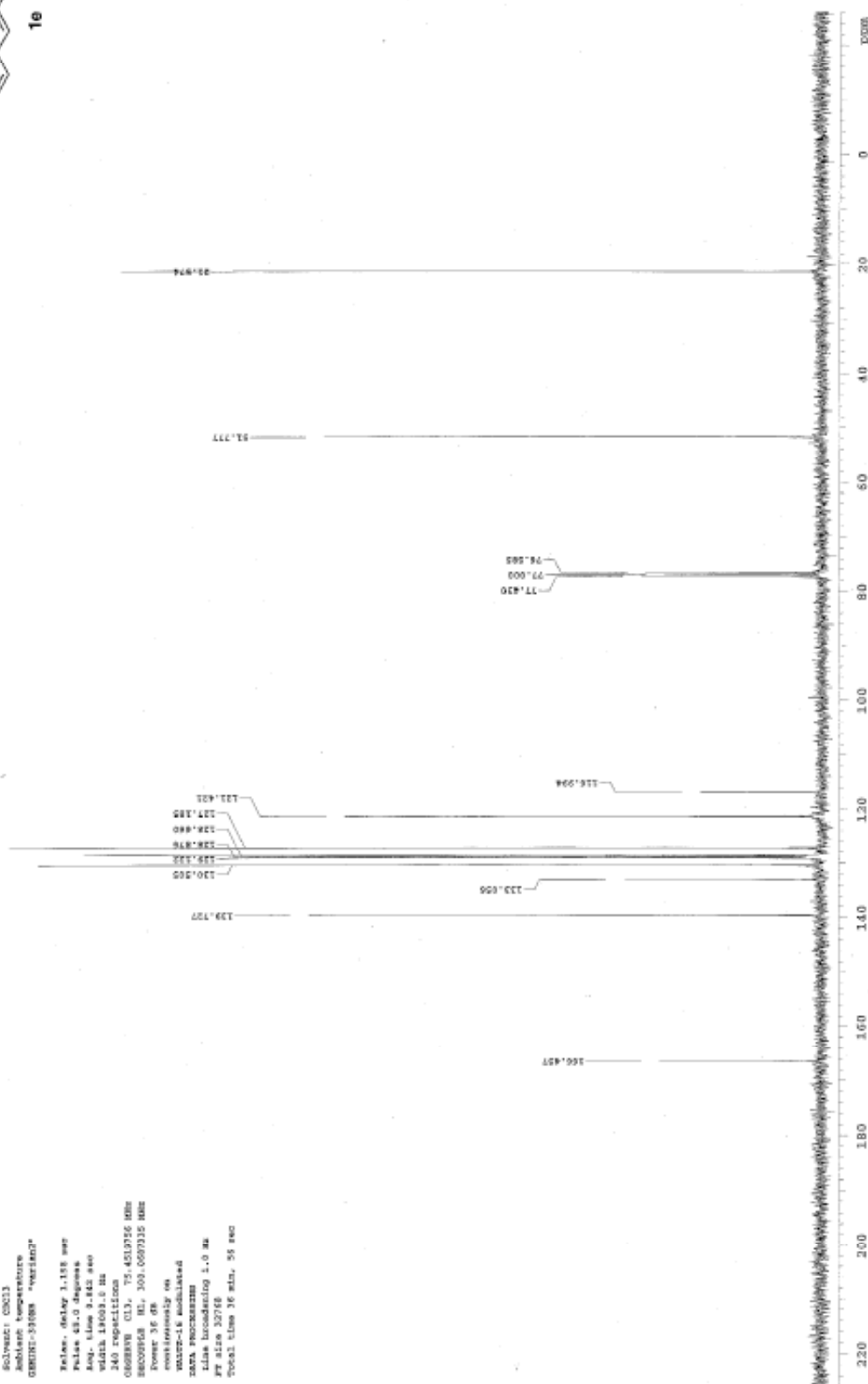
F2 - Acquisition Time: 14.00

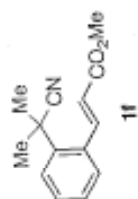
F2 - Acquisition Date: 20130801

F2 - Acquisition Time: 14.00

F2 - Acquisition Date: 20130801

F2 - Acquisition Time: 14.00





STANDARD IN OBSERVE

Pulse sequence: zgpg30

Solvent: CDCl<sub>3</sub>

Acquire temperature:

40001-10000 "various"

Relax. delay: 1.000 sec

Invert: 45.0 degrees

Acq. time: 3.280 sec

Width: 16000.0 Hz

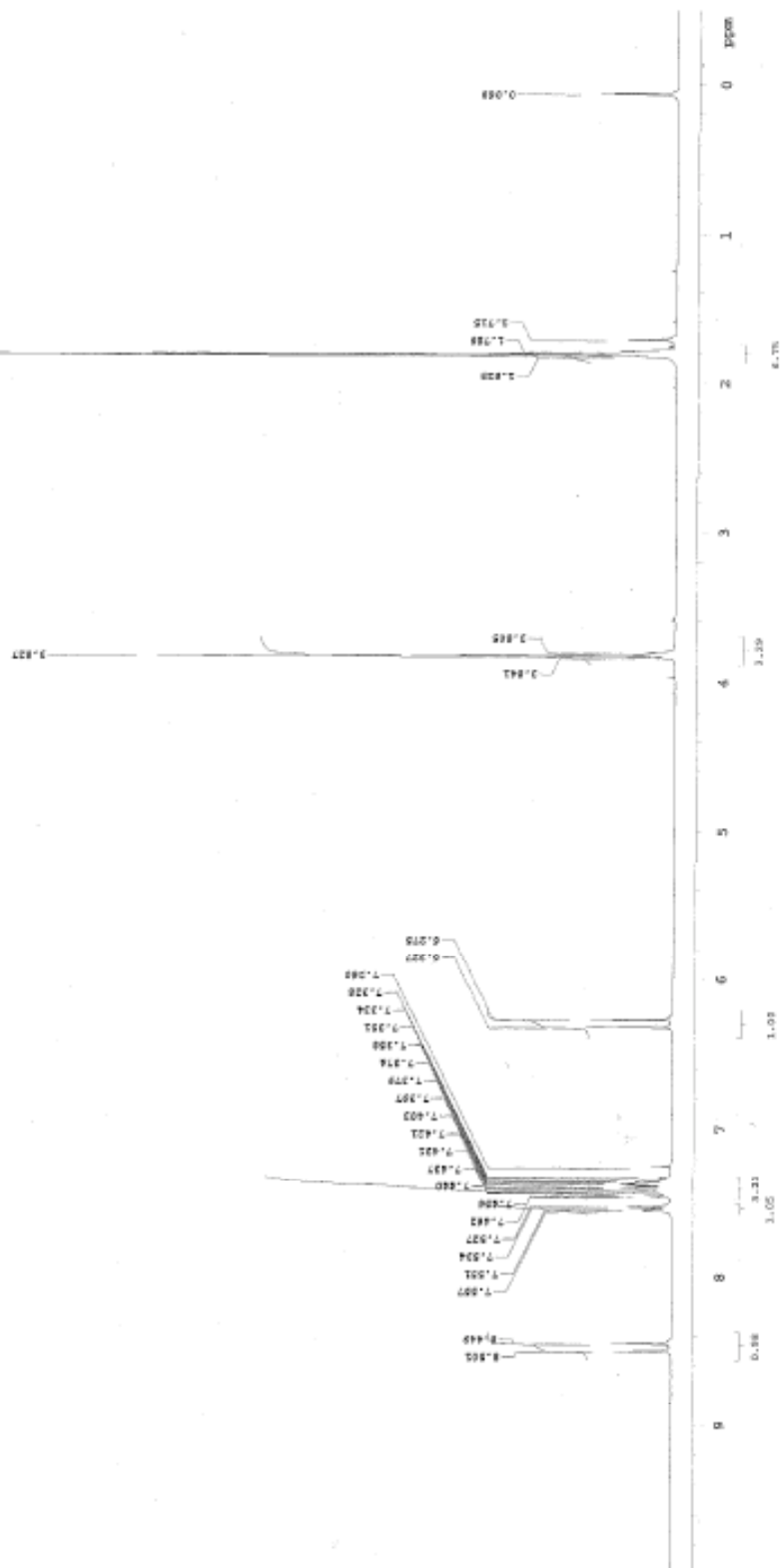
24 experiments

Completed: 11/15/2009 09:40:33:00

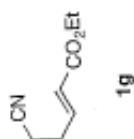
DATA PROCESSING

FF file 12108

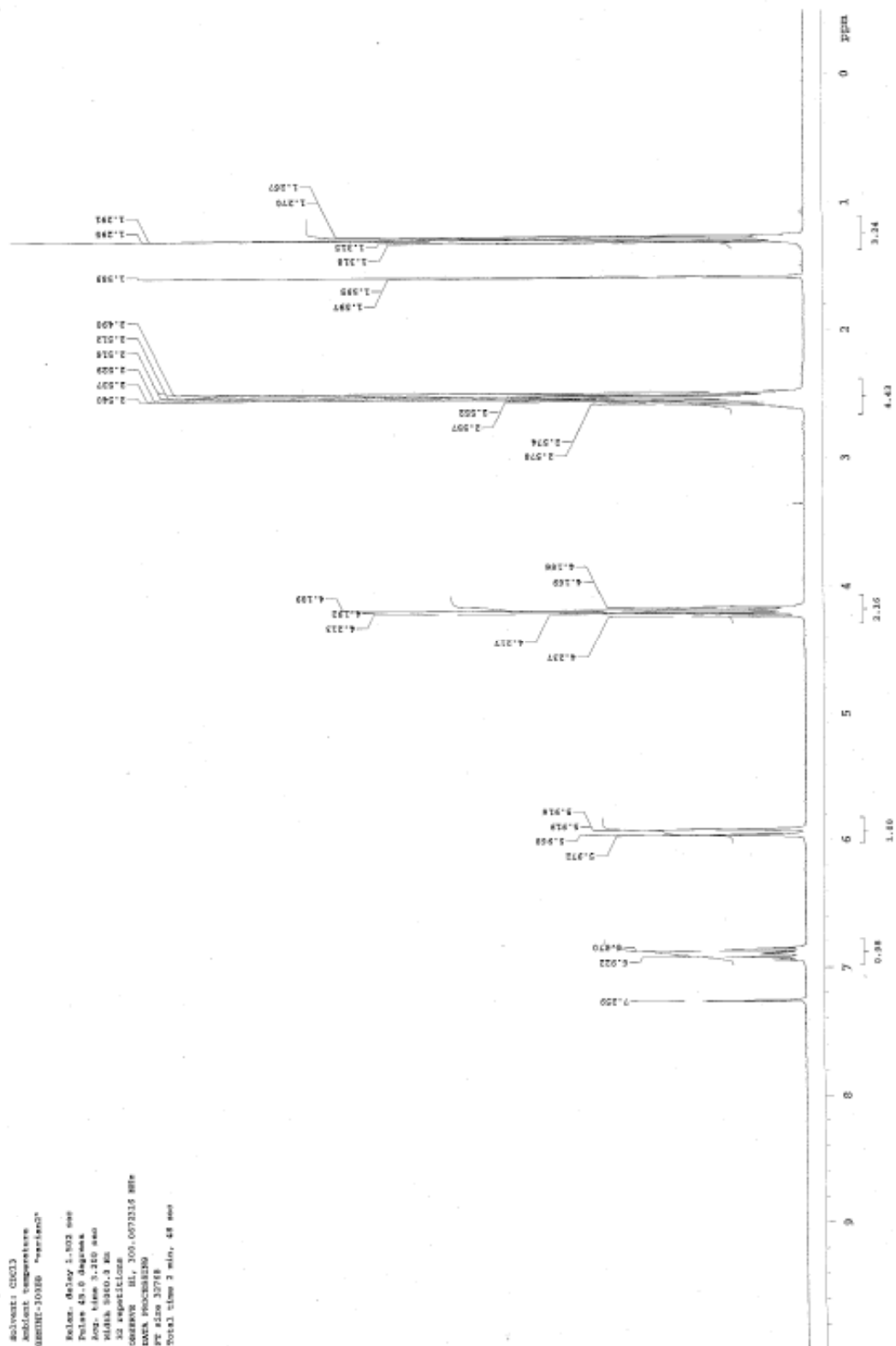
Total time 5 min, 37 sec

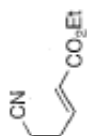






INSTRUMENT 3H CQ500N  
 Pulse Sequence: zgpg30  
 Solvent: CDCl3  
 Acquisition Temperature: 300.2 K  
 QNP-1H1000  
 Relax. Delay: 2.0000 sec  
 Pulse 45.0 degrees  
 Acq. time 0.200 sec  
 Lock 500.136 MHz  
 32 Spectral Lines  
 CDCl3 (H1, 100.6272315 MHz)  
 EPOCH PROCESSING  
 PR size 32768  
 Total time 2 min, 48 sec





19

13c: 00000000

Delta Sequence: 2400

solvent: CDCl<sub>3</sub>

solvent temperature

GENESIS-300B "varian2"

Pulse: delay 1.100 sec

Pulse 2: 1.100 sec

Pulse 3: 1.100 sec

Pulse 4: 1.100 sec

Pulse 5: 1.100 sec

Pulse 6: 1.100 sec

Pulse 7: 1.100 sec

Pulse 8: 1.100 sec

Pulse 9: 1.100 sec

Pulse 10: 1.100 sec

Pulse 11: 1.100 sec

Pulse 12: 1.100 sec

Pulse 13: 1.100 sec

Pulse 14: 1.100 sec

Pulse 15: 1.100 sec

Pulse 16: 1.100 sec

Pulse 17: 1.100 sec

Pulse 18: 1.100 sec

Pulse 19: 1.100 sec

Pulse 20: 1.100 sec

Pulse 21: 1.100 sec

Pulse 22: 1.100 sec

Pulse 23: 1.100 sec

Pulse 24: 1.100 sec

Pulse 25: 1.100 sec

Pulse 26: 1.100 sec

Pulse 27: 1.100 sec

Pulse 28: 1.100 sec

Pulse 29: 1.100 sec

Pulse 30: 1.100 sec

Pulse 31: 1.100 sec

Pulse 32: 1.100 sec

Pulse 33: 1.100 sec

Pulse 34: 1.100 sec

Pulse 35: 1.100 sec

Pulse 36: 1.100 sec

Pulse 37: 1.100 sec

Pulse 38: 1.100 sec

Pulse 39: 1.100 sec

Pulse 40: 1.100 sec

Pulse 41: 1.100 sec

Pulse 42: 1.100 sec

Pulse 43: 1.100 sec

Pulse 44: 1.100 sec

Pulse 45: 1.100 sec

Pulse 46: 1.100 sec

Pulse 47: 1.100 sec

Pulse 48: 1.100 sec

Pulse 49: 1.100 sec

Pulse 50: 1.100 sec

Pulse 51: 1.100 sec

Pulse 52: 1.100 sec

Pulse 53: 1.100 sec

Pulse 54: 1.100 sec

Pulse 55: 1.100 sec

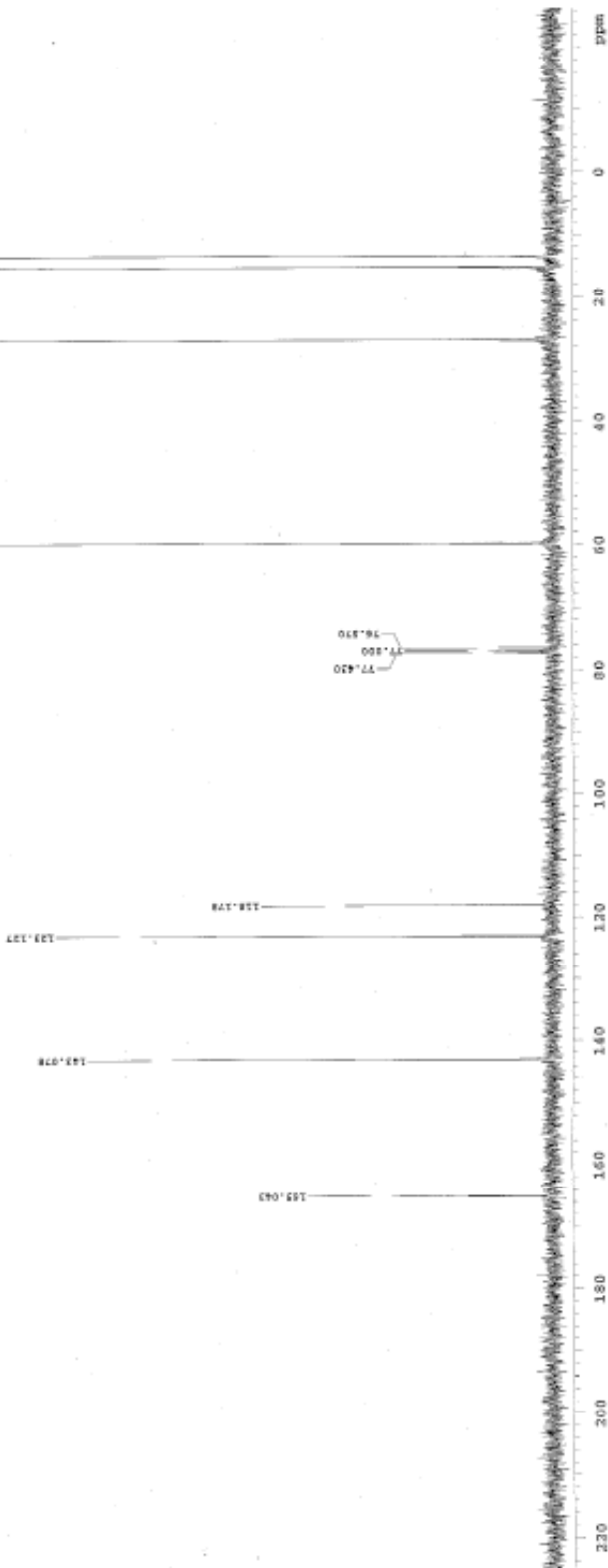
Pulse 56: 1.100 sec

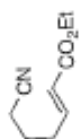
Pulse 57: 1.100 sec

Pulse 58: 1.100 sec

Pulse 59: 1.100 sec

Pulse 60: 1.100 sec





1h

STANDARD 1H NMR

Pulse Sequence: zgpg30

Acquire: 02013

Acquire: 02013

Acquire: 02013

Acquire: 02013

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Acquire: 02013

Acquire: 02013

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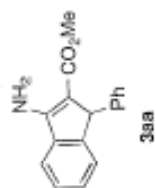
Acquire: 02013

Acquire: 02013

Acquire: 02013



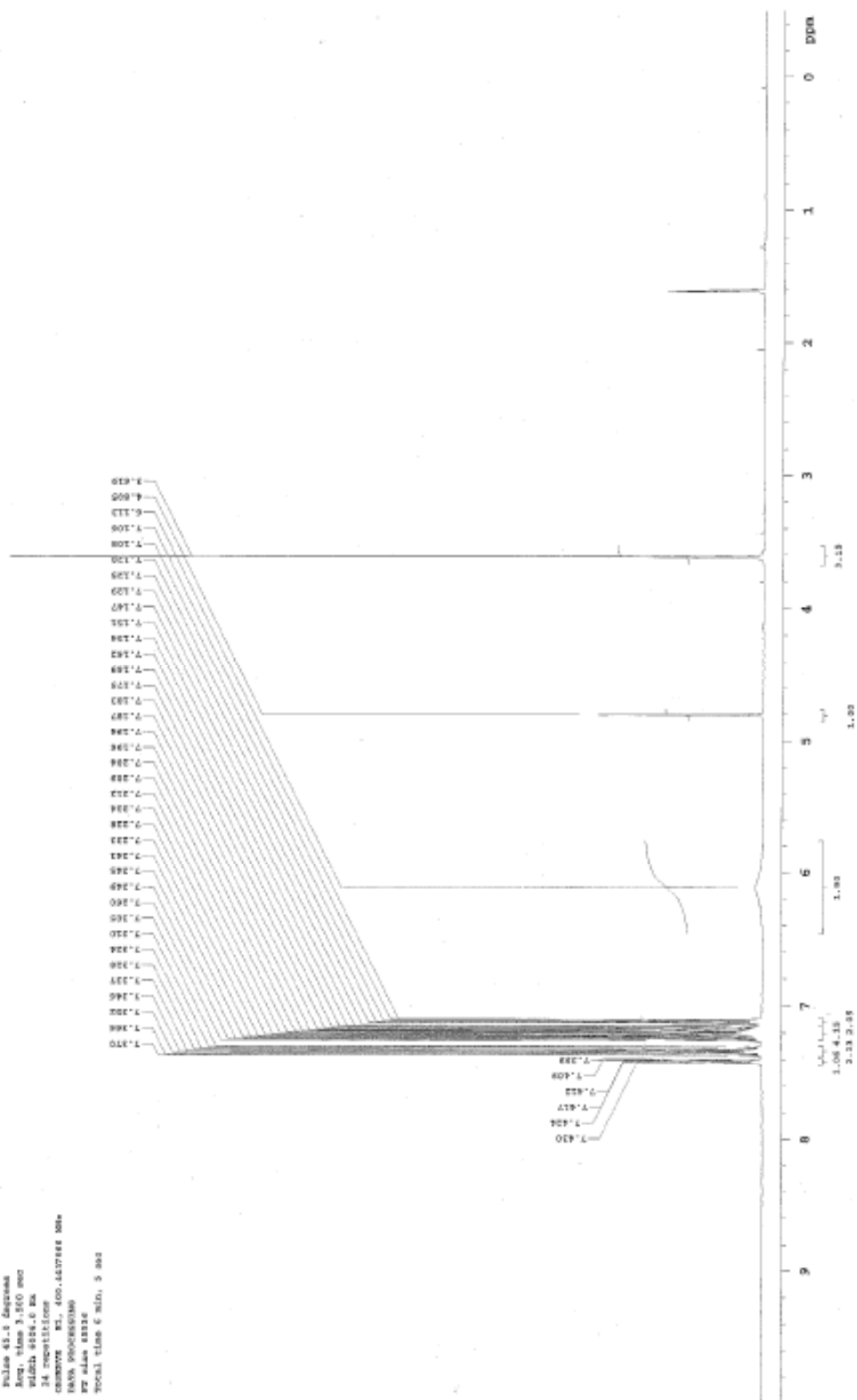


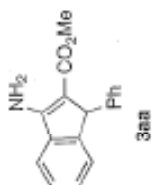


STANDARD 1E OBSERVE

Pulse Program: zgpg30  
 nucleus: 13C13  
 Aquisition: 13C13  
 Aquisition: 13C13  
 Aquisition: 13C13

Relax. delay: 3.000 sec  
 Pulse 45.0 degrees  
 Acq. time 3.000 sec  
 Width 4494.6 Hz  
 34 repetitions  
 CHANNEL F2, 400.6407888 MHz  
 100% 900000000  
 PR 4444 4444  
 Total time 6 min, 5 sec





LIN: C0000000

Peak Sequence: 11001

Substrate: C0013

Acidic Temperature

CHIRAL-10000 "various"

Notes: delay 1.150 sec

Peak 45.0 degrees

Acq. time 9.843 sec

11001 10000.0 Hz

256 repetitions

CHIRAL-10000 75.431557 Hz

CHIRAL-10000 311.667111 Hz

Power 16 W

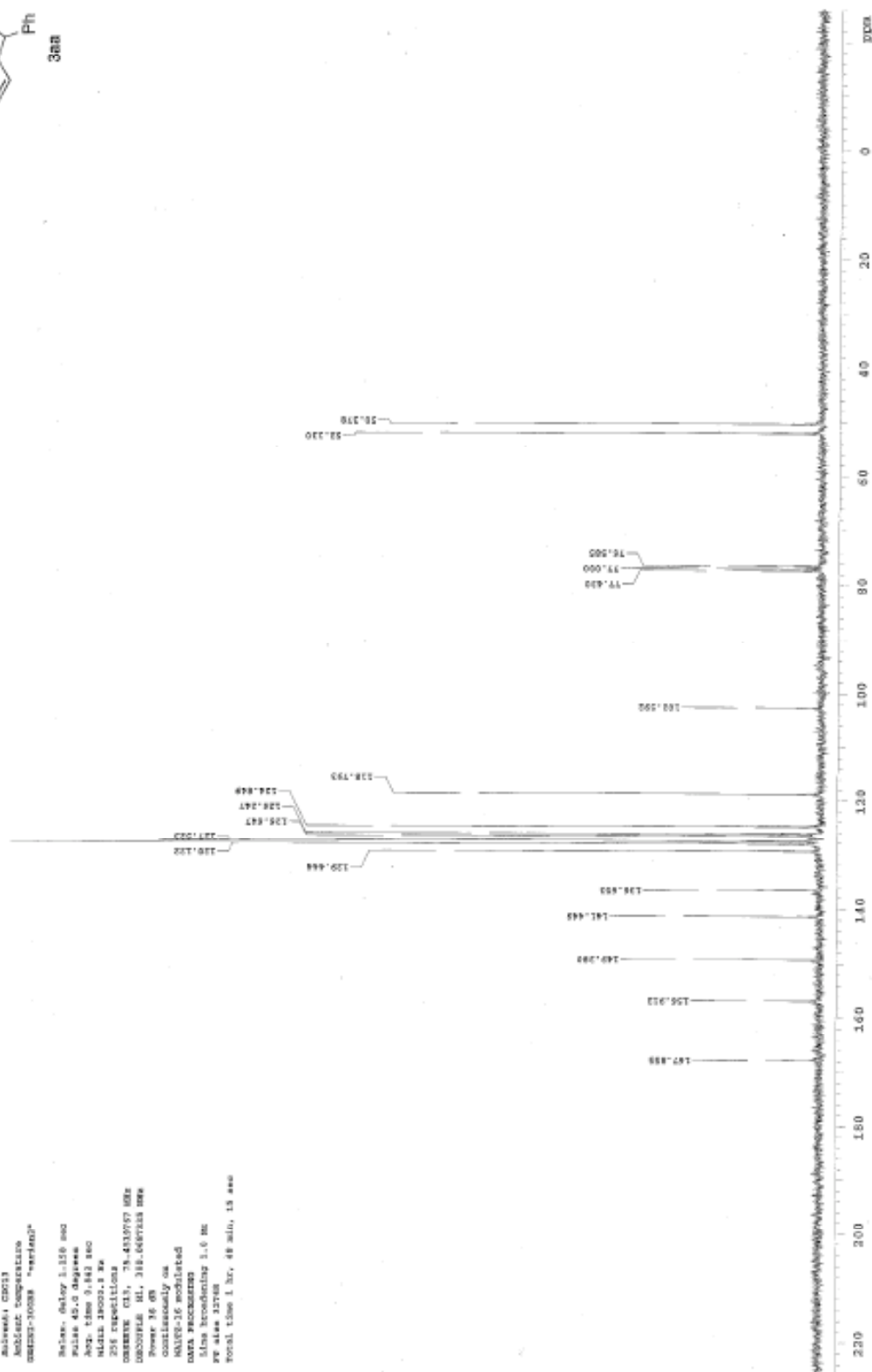
CHIRAL-10000 10000.0 Hz

DATA PROCESSING

Also processing 1.0 Hz

PE size 12716

Total time 1 hr, 48 min, 13 sec





Public Engagement: 02/04/23

6728 J. Neurosci., July 26, 2006 • 26(30):6723–6730

Adult-Like Components

00000-10000

Volume: 40, Number 1, 1993

value 45.0 degrees  
nom. value 1.300 mm

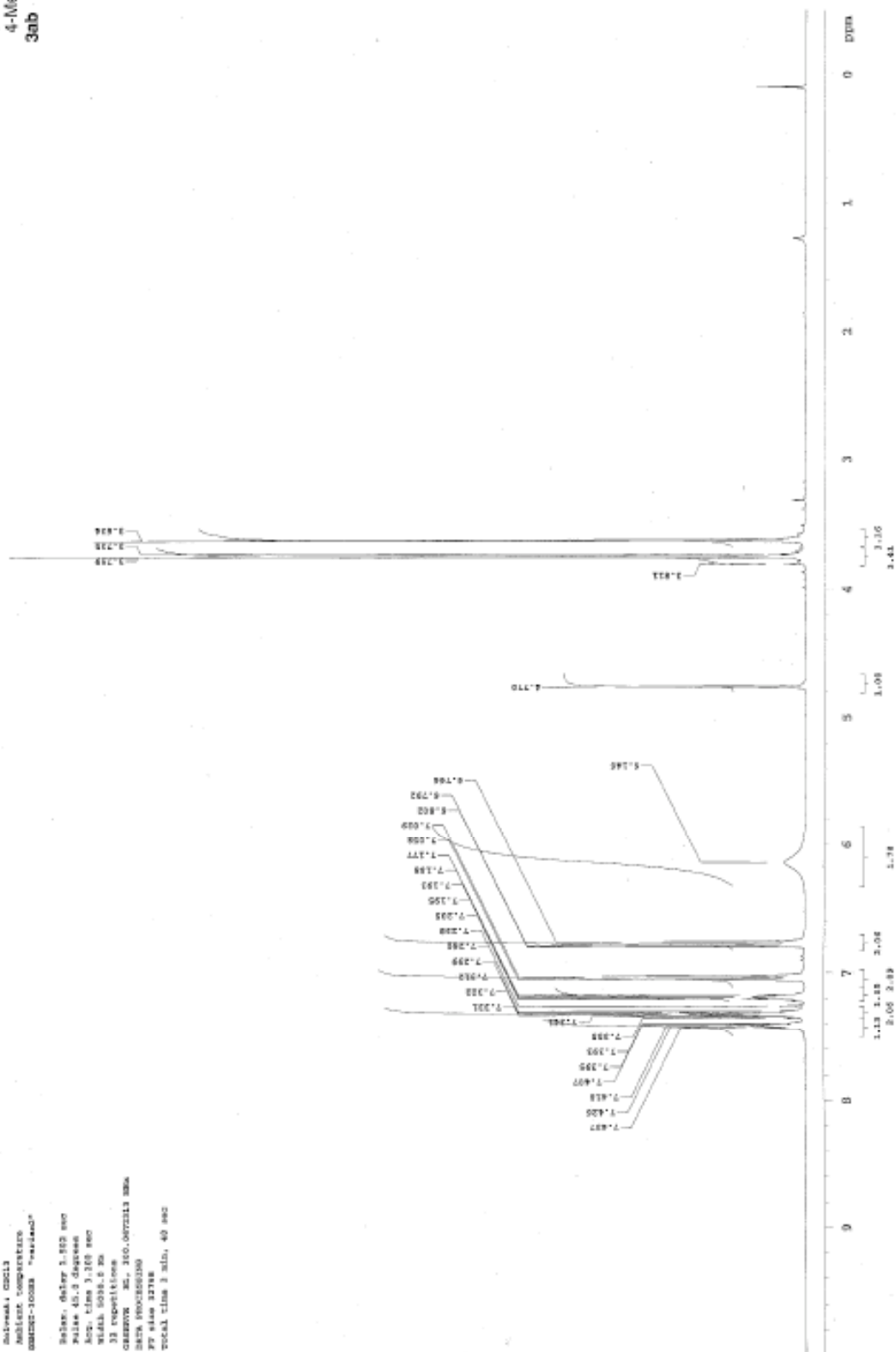
with 9-800-477-1111

32 repetitions

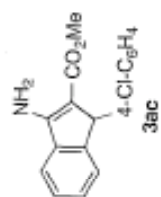
CHARTERED BY THE U.S. GOVERNMENT

[illegible]

total time 3 min, 40 sec







# STRUCTURE IN COMMENTS

Pulse Sequence: zgpg30

Solvent: (CDCl<sub>3</sub>)

Acquisition Temperature: 300.2 K

Reference: TMS

Relax. Delay: 1.500 sec

Pulse: 45.0 degrees

Acq. time: 3.000 sec

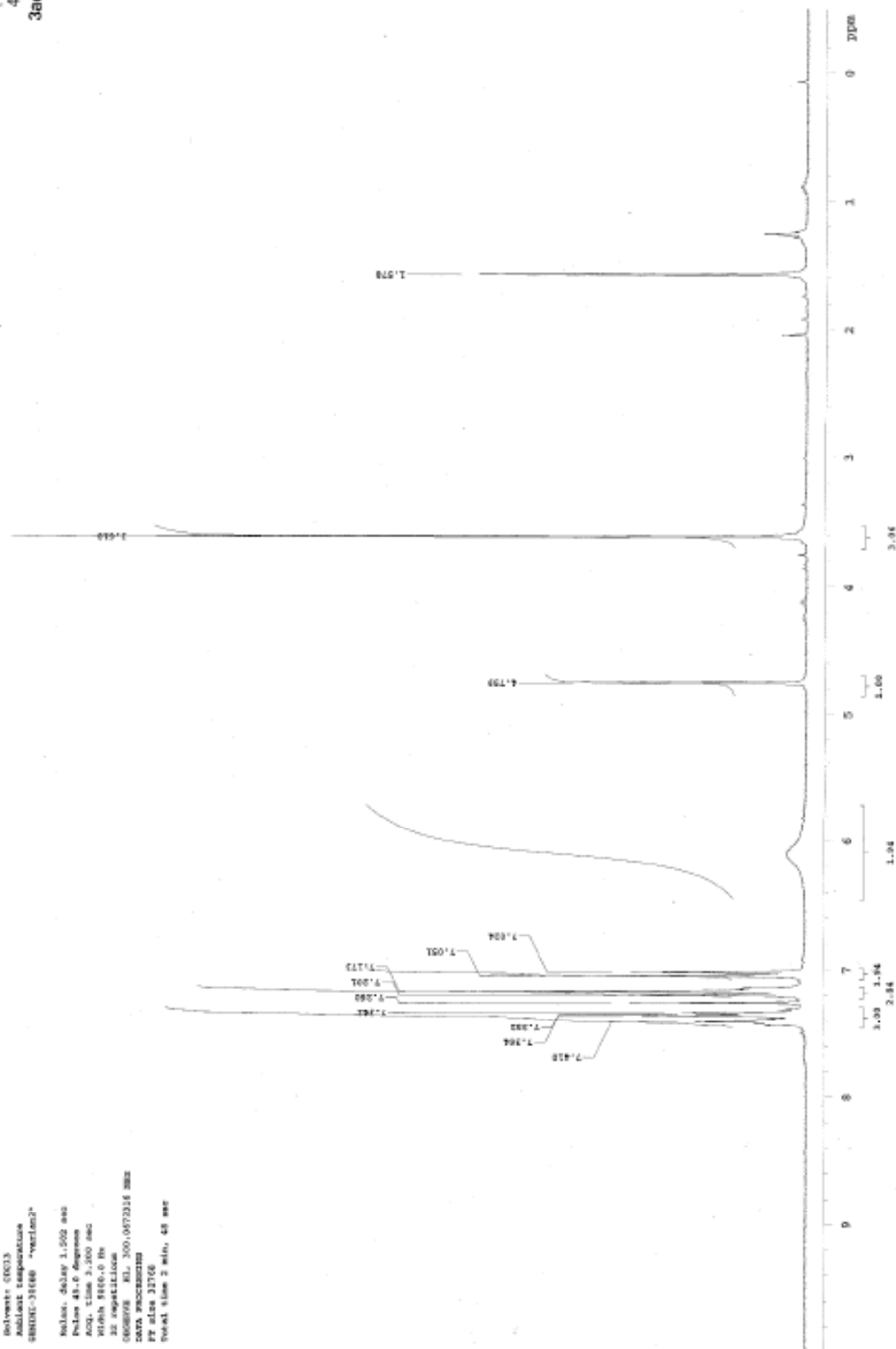
File: 11000000

Processing: 11000000

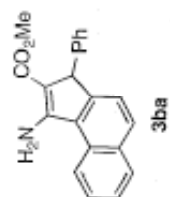
DATA: 11000000

PPM: 377.60

Total time: 2 min, 40 sec







STANDARD IN CHARGE

Pulse sequence: zgpg30

solvent: cpd13

Acquire time: 1.201 sec

Acquire time: 1.201 sec

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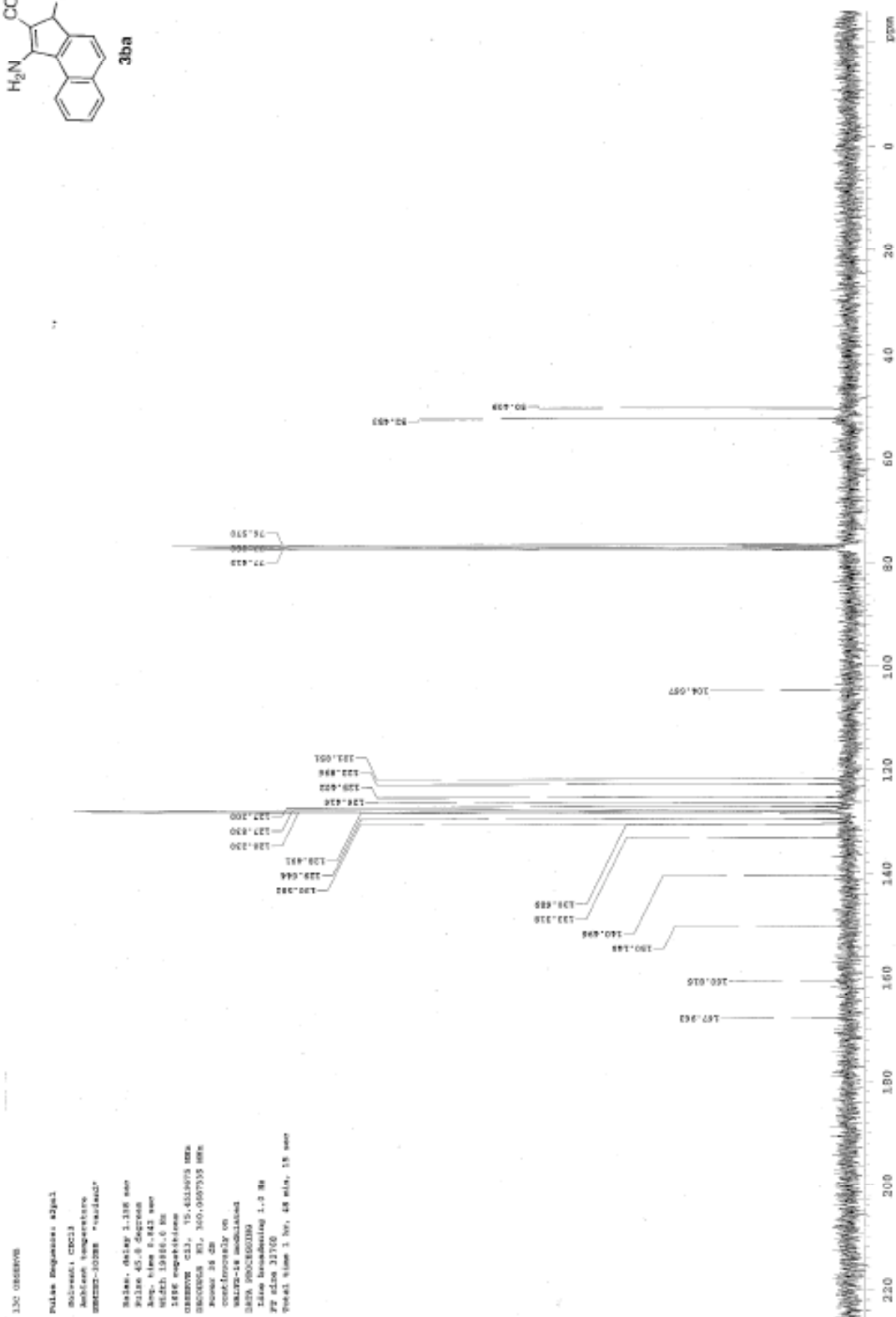
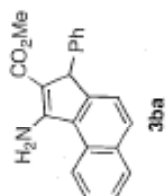
Acquire time: 1.201 sec

Acquire time: 1.201 sec

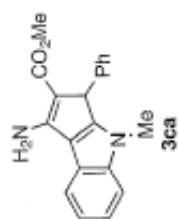
Acquire time: 1.201 sec

Acquire time: 1.201 sec

Acquire time: 1.201 sec







UNRECORDED 1H NMR

Pulse Program: zgpg30

Solvent: CDCl3

Acquire Temperature: 300.2 K

Reference: TMS

Relax. delay: 1.500 sec

Pulse: 65.0 degrees

Acq. time: 3.000 sec

Width: 6560.0 Hz

20 repetitions

Current: 10.00 A

RF: 400.443793 MHz

RF2: 101.625000 MHz

RF3: 101.625000 MHz

RF4: 101.625000 MHz

RF5: 101.625000 MHz

RF6: 101.625000 MHz

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RF99: 101.625000 MHz

RF100: 101.625000 MHz

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RF197: 101.625000 MHz

RF198: 101.625000 MHz

RF199: 101.625000 MHz

RF200: 101.625000 MHz

RF201: 101.625000 MHz

RF202: 101.625000 MHz

RF203: 101.625000 MHz

RF204: 101.625000 MHz

RF205: 101.625000 MHz

RF206: 101.625000 MHz

RF207: 101.625000 MHz

RF208: 101.625000 MHz

RF209: 101.625000 MHz

RF210: 101.625000 MHz

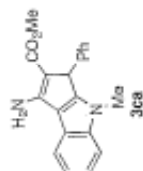
RF211: 101.625000 MHz

RF212: 101.625000 MHz

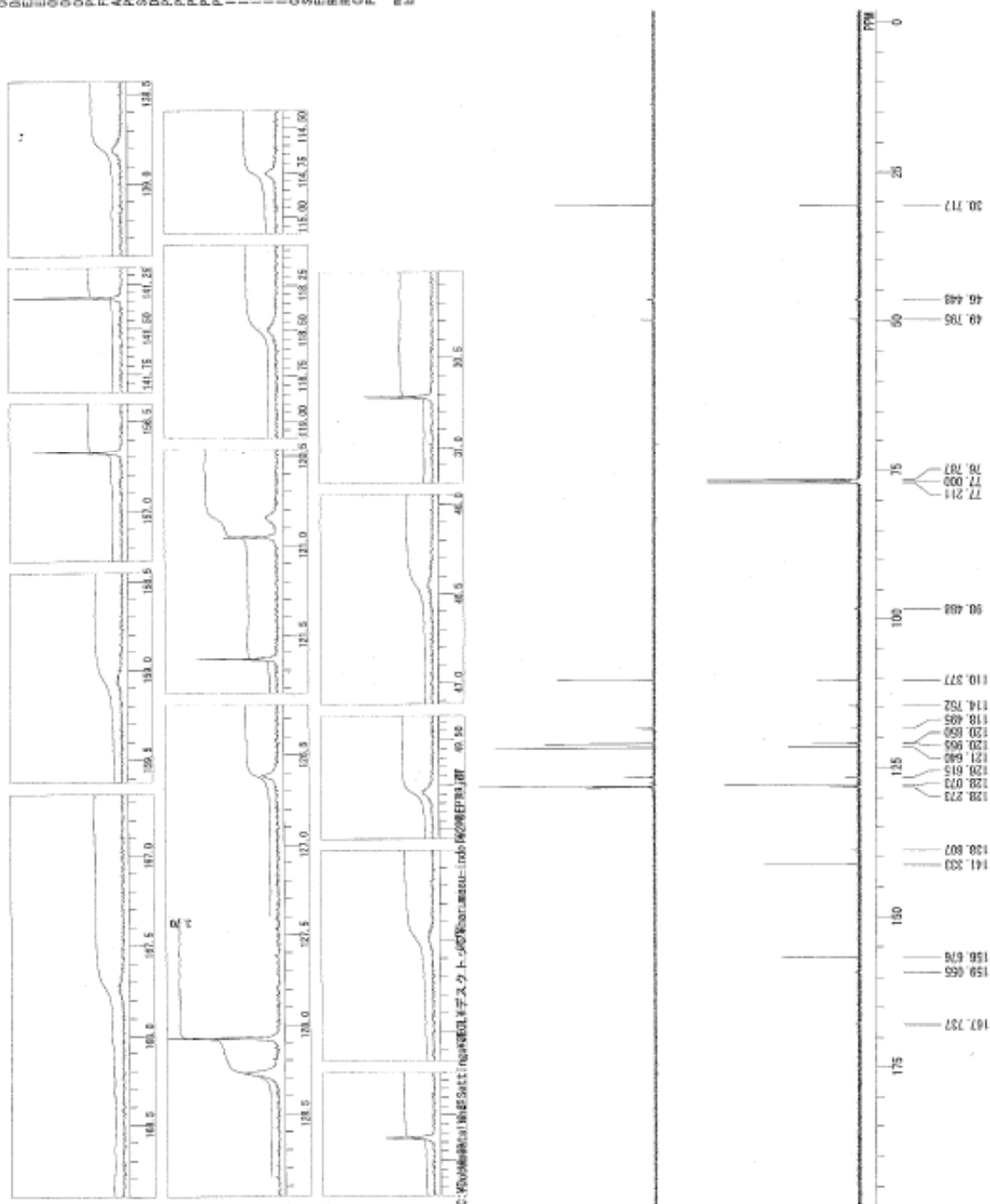
RF213: 101.625000 MHz

RF214: 101.625000 MHz

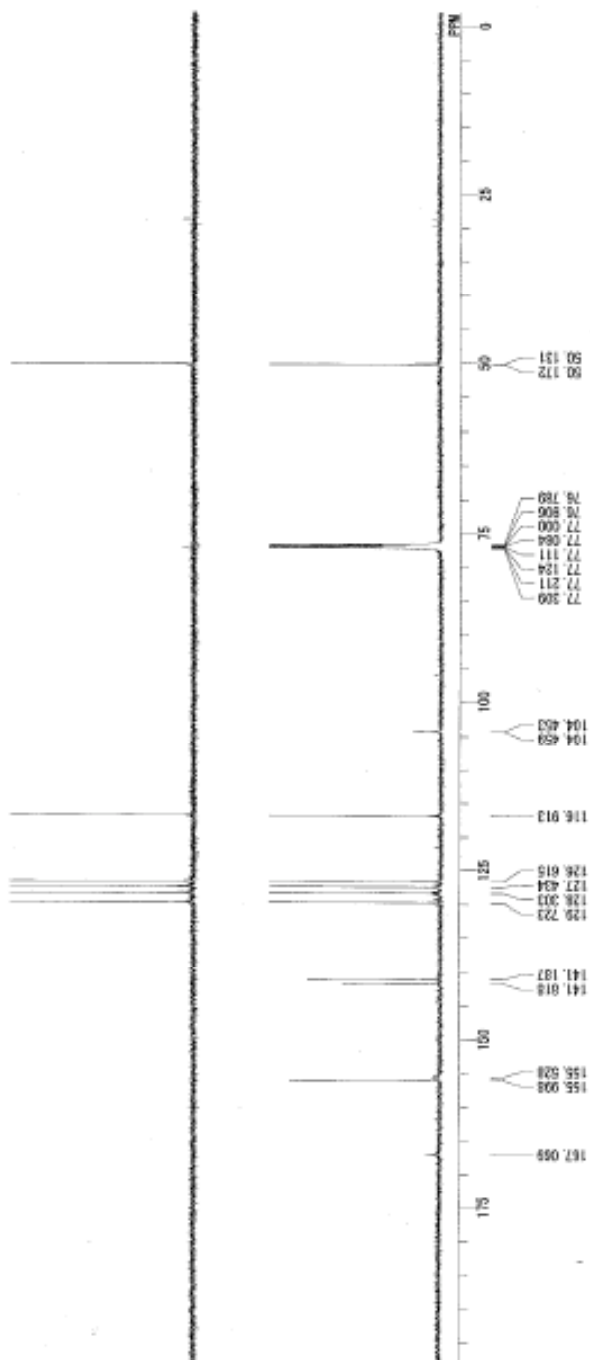
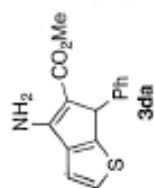
RF215: 101.625000 MHz

[illegible]

Measurement by FUJITA

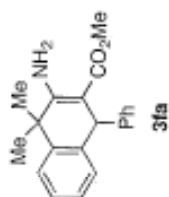












07/26/2010 11:08:38

Peak: 3fa

File: 0023

Method: 10000

Acq. time: 10000

Acq. date: 07/26/2010

Acq. time: 10000

Acq. date: 07/26/2010

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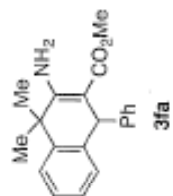
Acq. time: 10000

Acq. date: 07/26/2010

Acq. time: 10000

Acq. date: 07/26/2010

Acq. time: 10000



L30: 0888008

Twice Regression: elgel

solvent: CDCl<sub>3</sub>

solvent temperature

CHMINT-3328 "VAR1A02"

Time: delay 1.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

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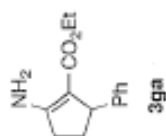
Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec

Time: 41.100 sec





STANDARD 33 C080709

Pulse sequence: zgpg30  
Solvent: CHCl<sub>3</sub>  
Acquired temperature: 300.2 K  
SOLVENT-100% "variaa2"

Relax. delay: 1.500 sec

Pulses: 45.0 degrees

Acq. time: 1.200 sec

FID: 2000.0 Hz

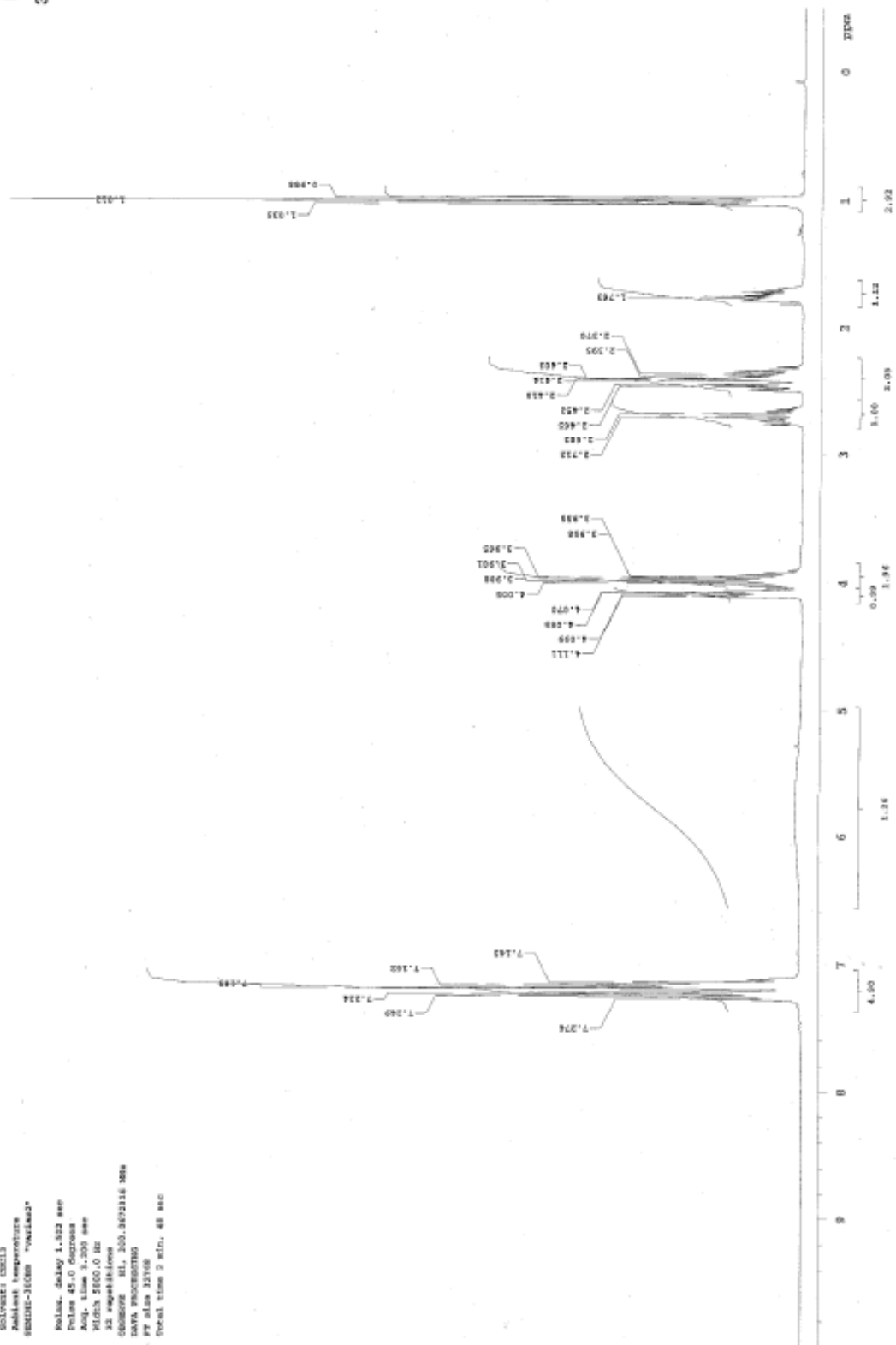
32 segments

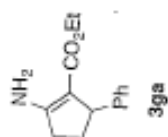
COMPOUND: 33, 300.3672116 MHz

DATA PROCESSING

PP: 100.000

Total time: 2 min, 48 sec





13C CHANNEL

Pulse Program: zgpg30

Software: CMC13

NUC1: 13C

PROBHD: 5mmBBO

QNP13: 125.76 MHz

P1: 12.00 us

P2: 12.00 us

P3: 12.00 us

P4: 12.00 us

P5: 12.00 us

P6: 12.00 us

P7: 12.00 us

P8: 12.00 us

P9: 12.00 us

P10: 12.00 us

P11: 12.00 us

P12: 12.00 us

P13: 12.00 us

P14: 12.00 us

P15: 12.00 us

P16: 12.00 us

P17: 12.00 us

P18: 12.00 us

P19: 12.00 us

P20: 12.00 us

P21: 12.00 us

P22: 12.00 us

P23: 12.00 us

P24: 12.00 us

P25: 12.00 us

P26: 12.00 us

P27: 12.00 us

P28: 12.00 us

P29: 12.00 us

P30: 12.00 us

P31: 12.00 us

P32: 12.00 us

P33: 12.00 us

P34: 12.00 us

P35: 12.00 us

P36: 12.00 us

P37: 12.00 us

P38: 12.00 us

P39: 12.00 us

P40: 12.00 us

P41: 12.00 us

P42: 12.00 us

P43: 12.00 us

P44: 12.00 us

P45: 12.00 us

P46: 12.00 us

P47: 12.00 us

P48: 12.00 us

P49: 12.00 us

P50: 12.00 us

P51: 12.00 us

P52: 12.00 us

P53: 12.00 us

P54: 12.00 us

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P56: 12.00 us

P57: 12.00 us

P58: 12.00 us

P59: 12.00 us

P60: 12.00 us

P61: 12.00 us

P62: 12.00 us

P63: 12.00 us

P64: 12.00 us

P65: 12.00 us

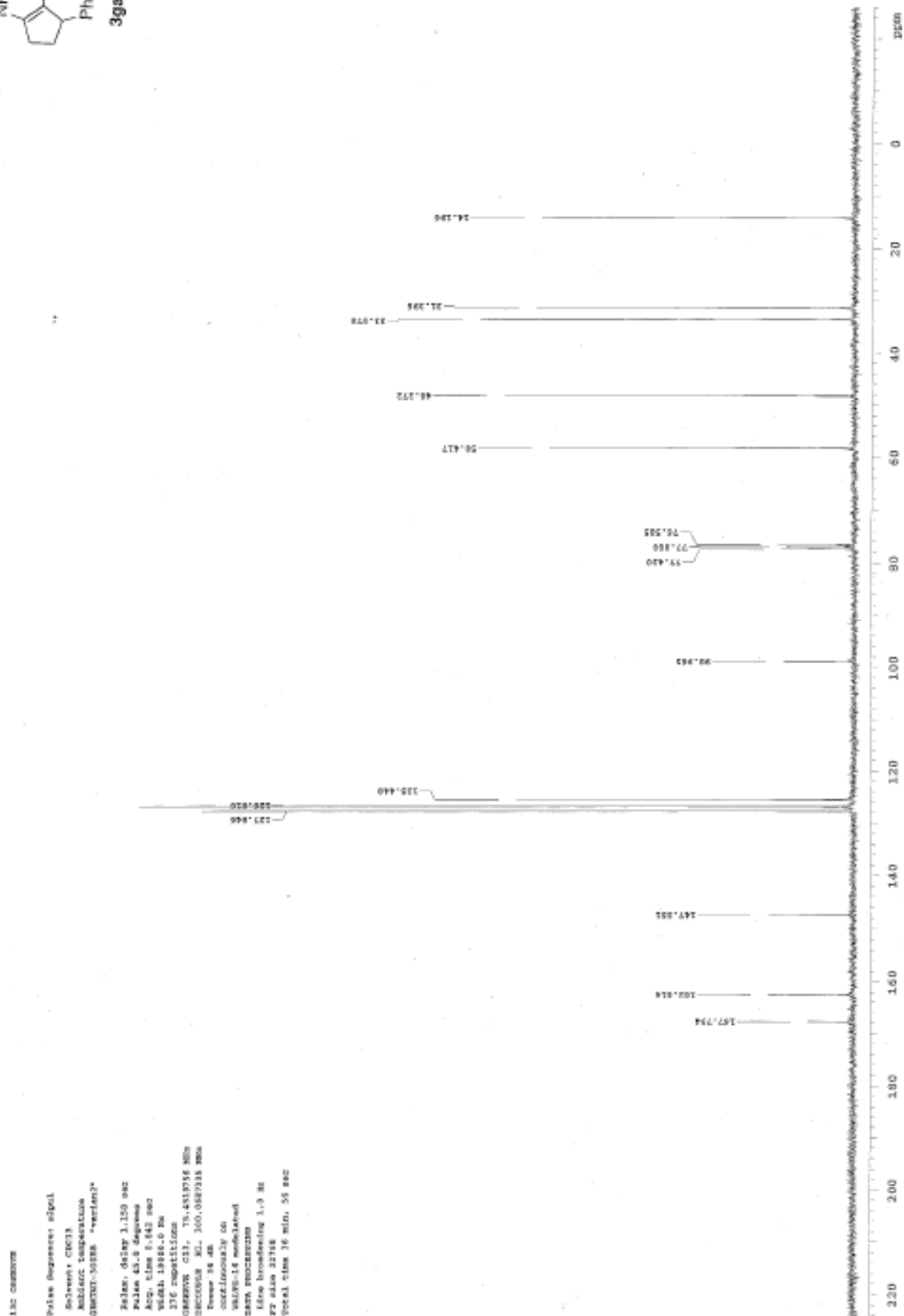
P66: 12.00 us

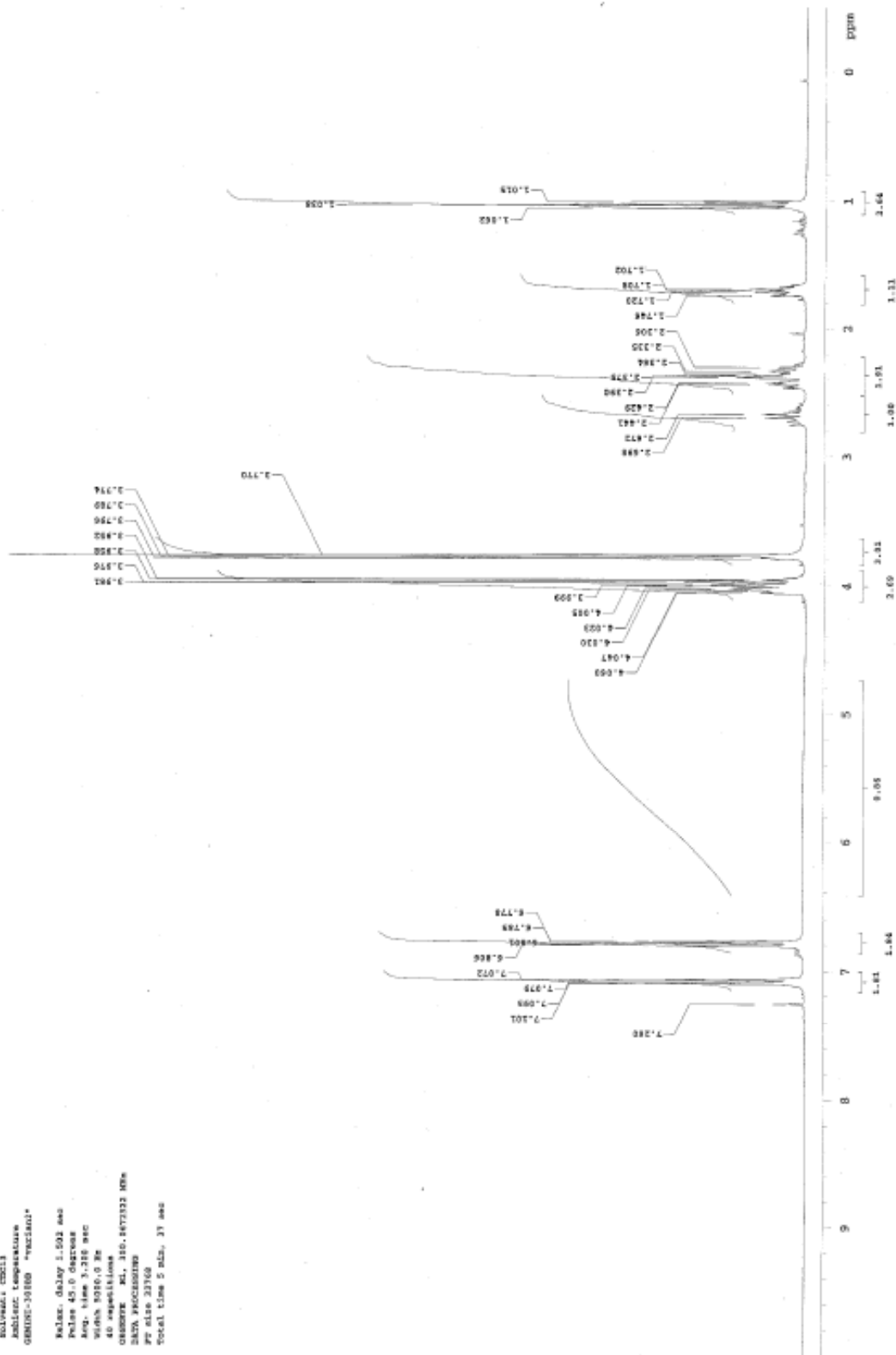
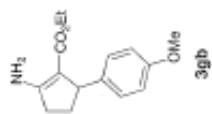
P67: 12.00 us

P68: 12.00 us

P69: 12.00 us

P70: 12.00 us





STATIONED IN GERMANY

Pulse Sequences: zgpg30  
solvent: crn3  
ambient temperature  
GEMINI-1000 "variable"

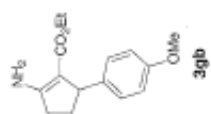
**Author:** Andrew J. Nathan

```

Relax. delay 1.502 sec
Pulse 45.0 degrees
Acq. time 3.286 sec
Width 9280.0 Hz
40 repetitions
NAME: 01, 310.867392

```

ORIGIN: MI, 380.867322 MM  
DATA PROCESSING  
FW size 23760  
Total time 5 min, 37 sec



13C NMR

Pulse Program: zgpg30

Solvent: CDCl3

Acquisition Temperature: 300K

Chemical Shift Reference: TMS

Pulse delay: 1.150 sec

Relaxation delay: 45.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

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Acq. time: 5.000 sec

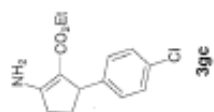
Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec

Acq. time: 5.000 sec



STANDARD 111 CEMEX/VE

Pulse Program: 42901

Solvent: CDCl3

Ambient temperature

CEMEX-10000 "various"

Relax. delay: 1.000 sec

Pulse: 40.0 degrees

Acq. time: 1.000 sec

Width: 5000.0 Hz

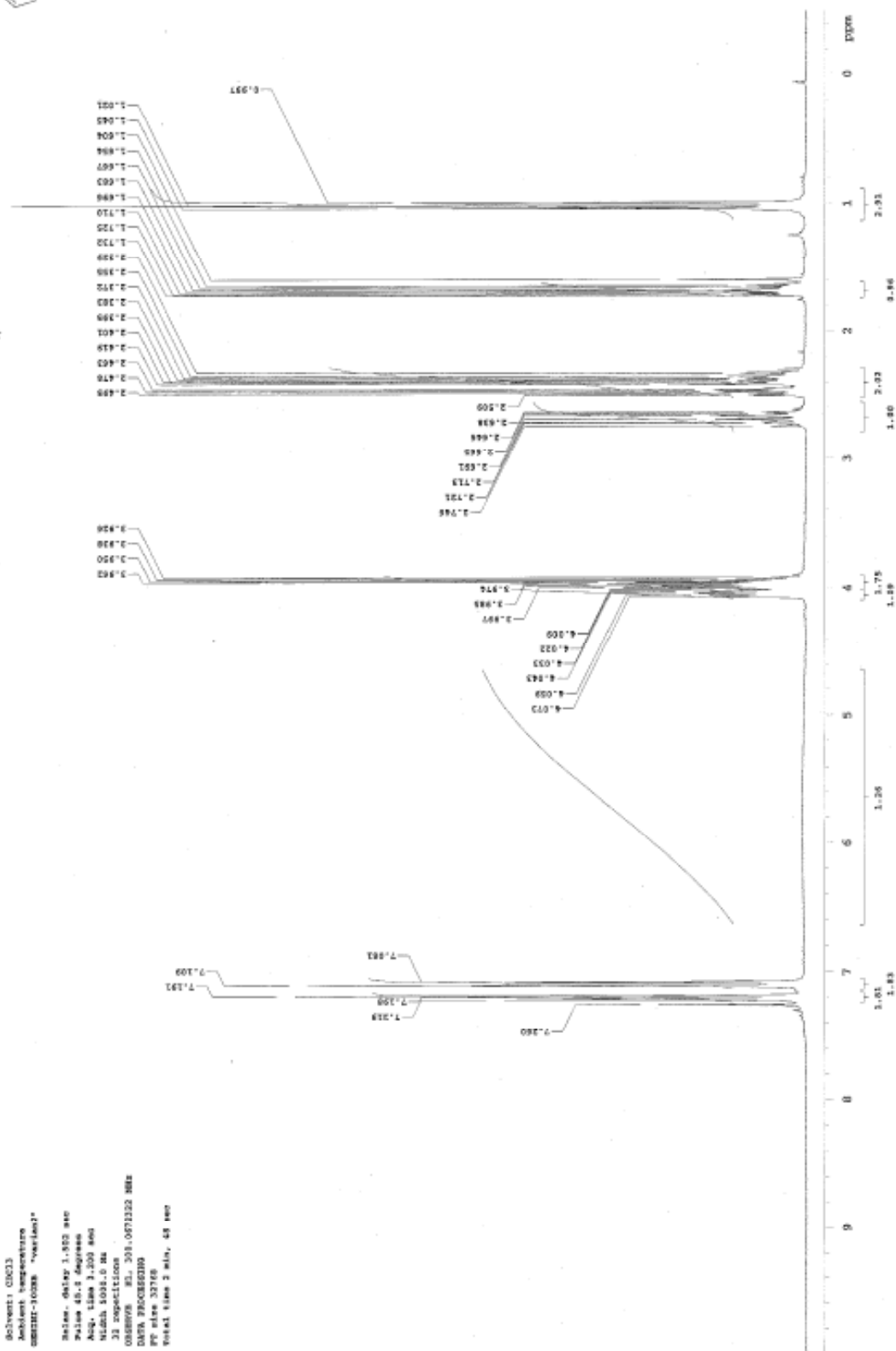
31 repetitions

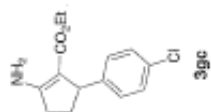
CEMEX/VE 31.001.0071222 MHz

DATA F200003100

PROBHD 5mm

Total time: 2 min, 48 sec

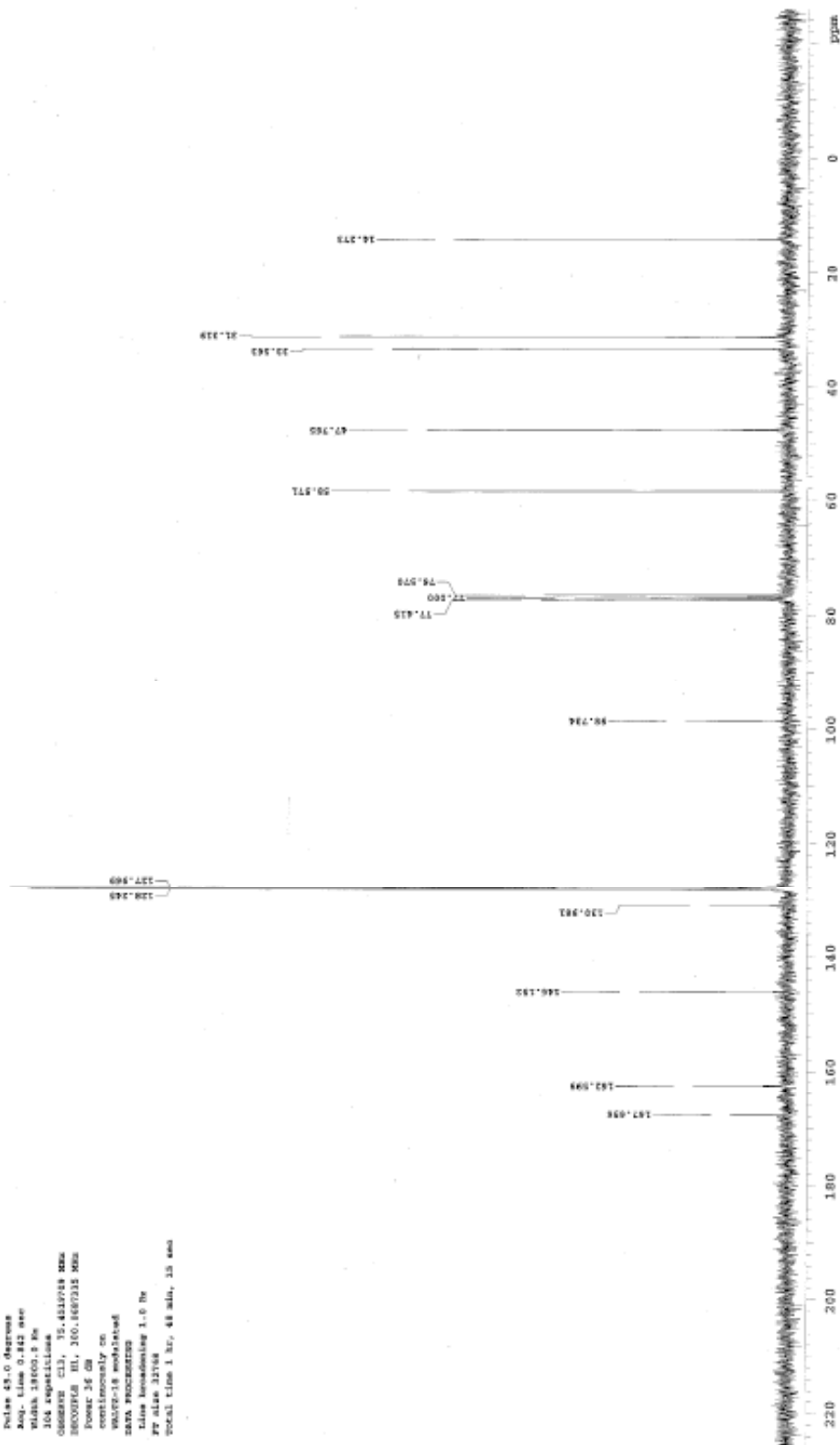




13C NMR

Pulse sequence: zgpg30  
Solvent: CDCl<sub>3</sub>  
Reference: TMS  
Acquisition: 300 MHz, 125 MHz  
Chemical shift: 125.0 MHz

Relax. delay: 3.158 sec  
Pulse: 45.0 degrees  
Acq. time: 0.442 sec  
F2: 125.0 MHz  
F1: 125.0 MHz  
104 Experiments  
Spectrum: 13, 75.433/719 MHz  
SFO00168 H1, 100.626/735 MHz  
Power: 35.00  
Continuously on  
VNUC-18 modulated  
DATA PROCESSING  
Time: 1.00 Hz  
FT also 31768  
Total time: 1 hr, 48 min, 15 sec





RECORDED IN CDCl<sub>3</sub>

Pulse Sequence: zgpg30

Solvent: CDCl<sub>3</sub>

Acquisition Temperature: 300 K

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

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NAME: 4ha

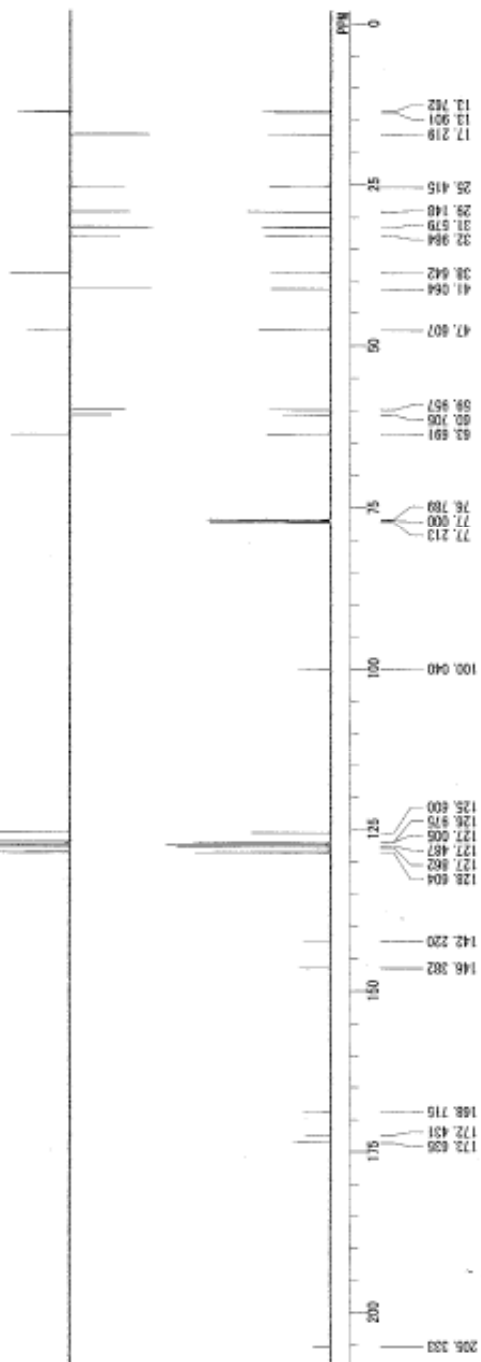
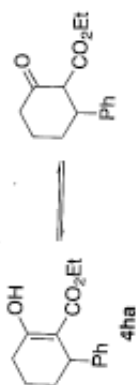
NAME: 4ha

NAME: 4ha

NAME: 4ha

NAME: 4ha

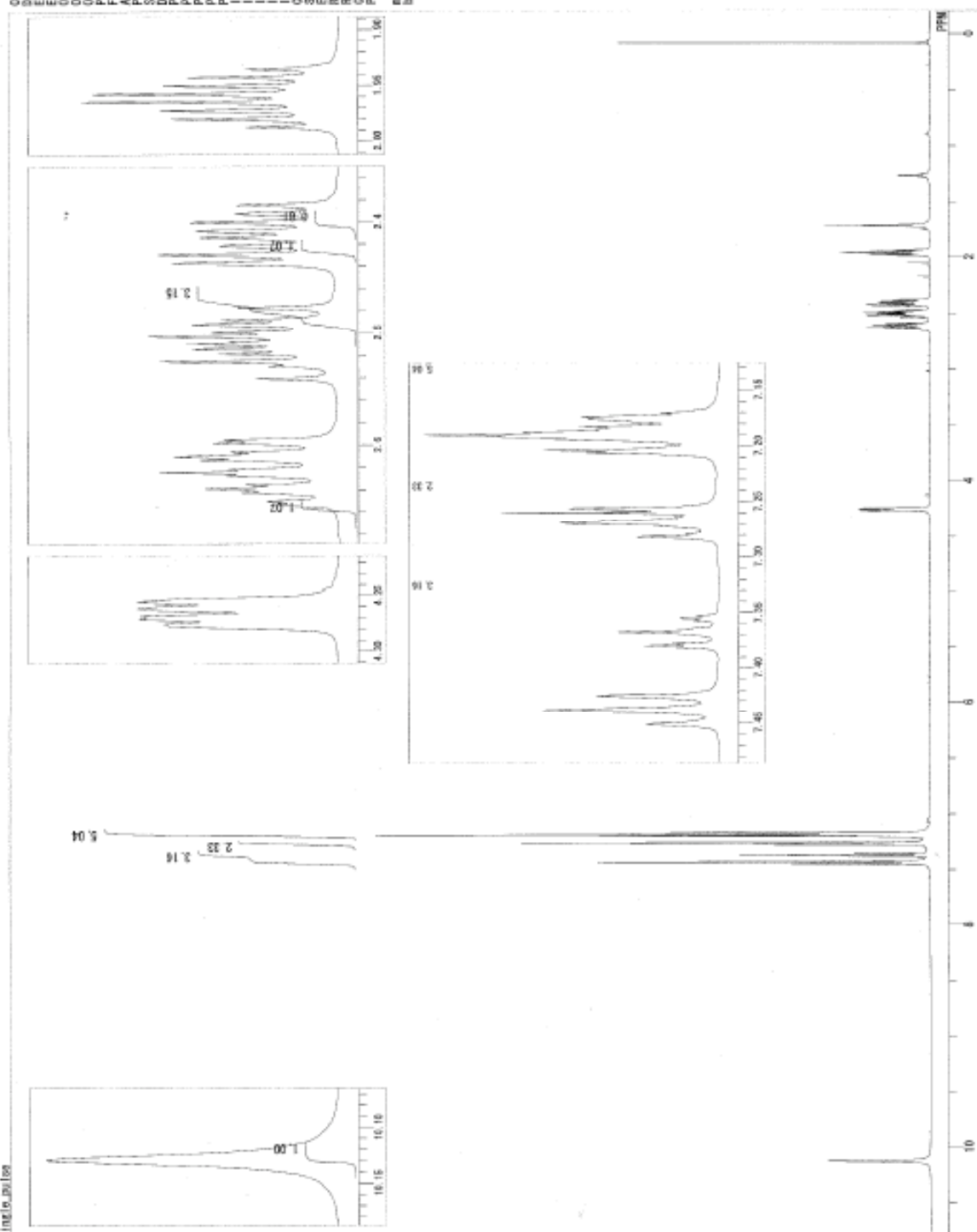
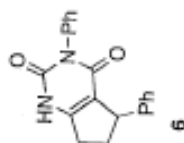
NAME: 4ha

[illegible]

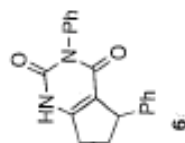


COMET single\_pulse  
SATIM 20-11-2006 16:23:02  
EXM00 single\_pulse.ac2  
EXPOR 1H  
CDEPC 600.17 MHz  
CDEPC 5.17 Hz  
F2F2T 9245.78 Hz  
F2F2T 3.6394 sec  
AQTIM 3.0000 sec  
PD 3.0000 sec  
SCANS 4  
DUMMY 2  
PUL 6.75 usec  
PR2 0.00 usec  
PR3 0.00 usec  
P1 0.0000 usec  
P12 0.0000 usec  
P13 0.0000 usec  
IRFWD 10000 1H  
IRFWD 600.17 MHz  
IRSET 5.30 MHz  
IRATN 0  
CTEMP 30.0 C  
SLVIT C00.3  
EXREF 7.25 ppm  
RGAIN 50  
RESOL 0.27 Hz  
CTEMP 30.0 C  
PRICE

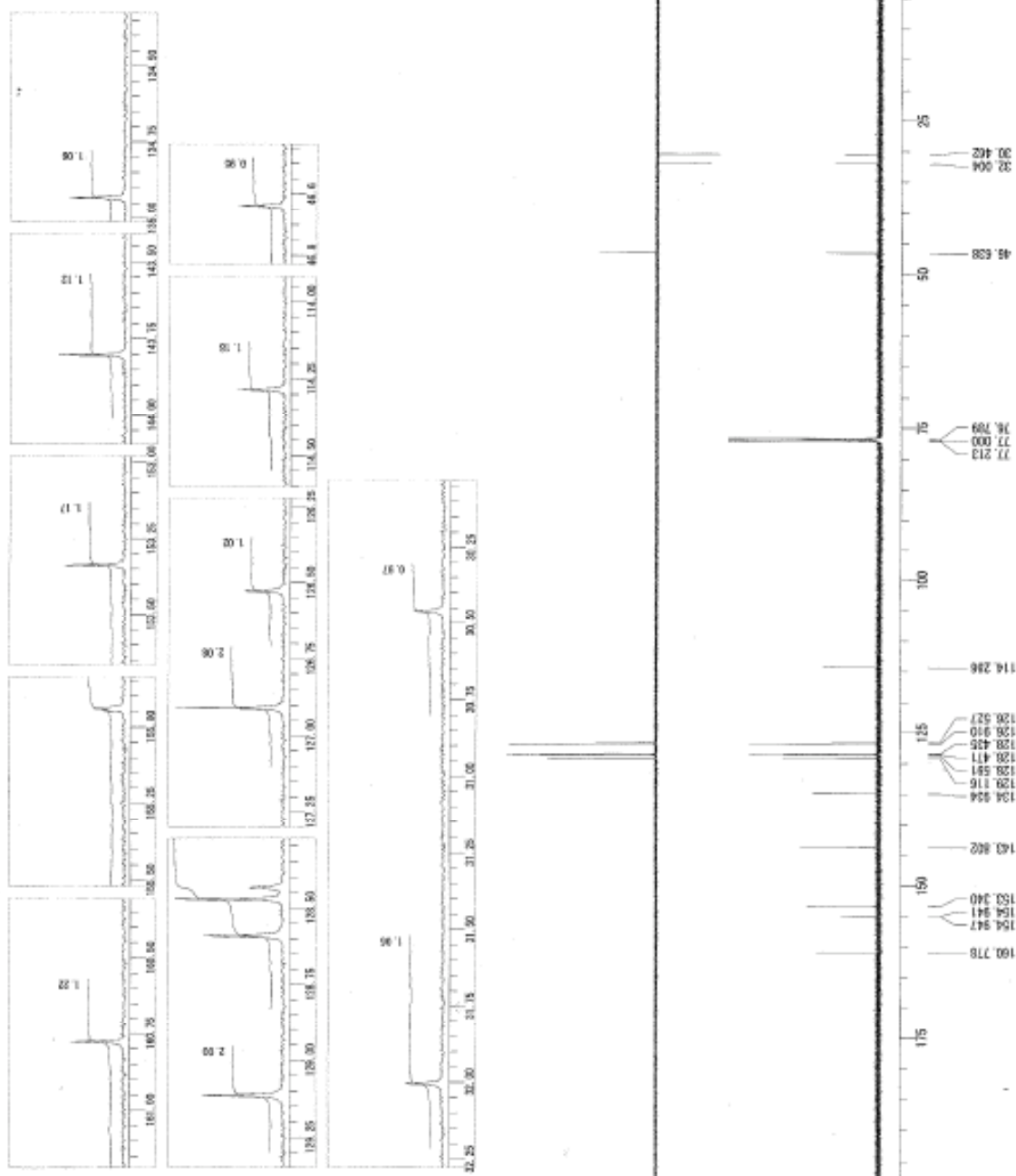
measurement no  
by RUIITA



C:\NMR\PMW\Fujita\Pharmaceuticals\1-NHEtL.a1.s



20-11-2004 16:07:11  
 single pulse dec  
 130 100.32 MHz  
 104.876 Hz  
 33332.82 Hz  
 1.5739 sec  
 6.0000 sec  
 800  
 4  
 3.75 usec  
 0.00 usec  
 0.00 usec  
 0.00 usec  
 0.0000 sec  
 0.0000 sec  
 1H  
 600.17 MHz  
 5.30 MHz  
 0  
 36.0 g  
 003.3  
 77.00 ppm  
 60  
 0.32 Hz  
 36.0 g  
 measurement no  
 by FUJITA





STIMULATING THE CONSUMER

Polymer Temperature: 210°C  
 Solvent: *m*-Cresol  
 Ambient Temperature  
 Duration: 40 min "steady"

```

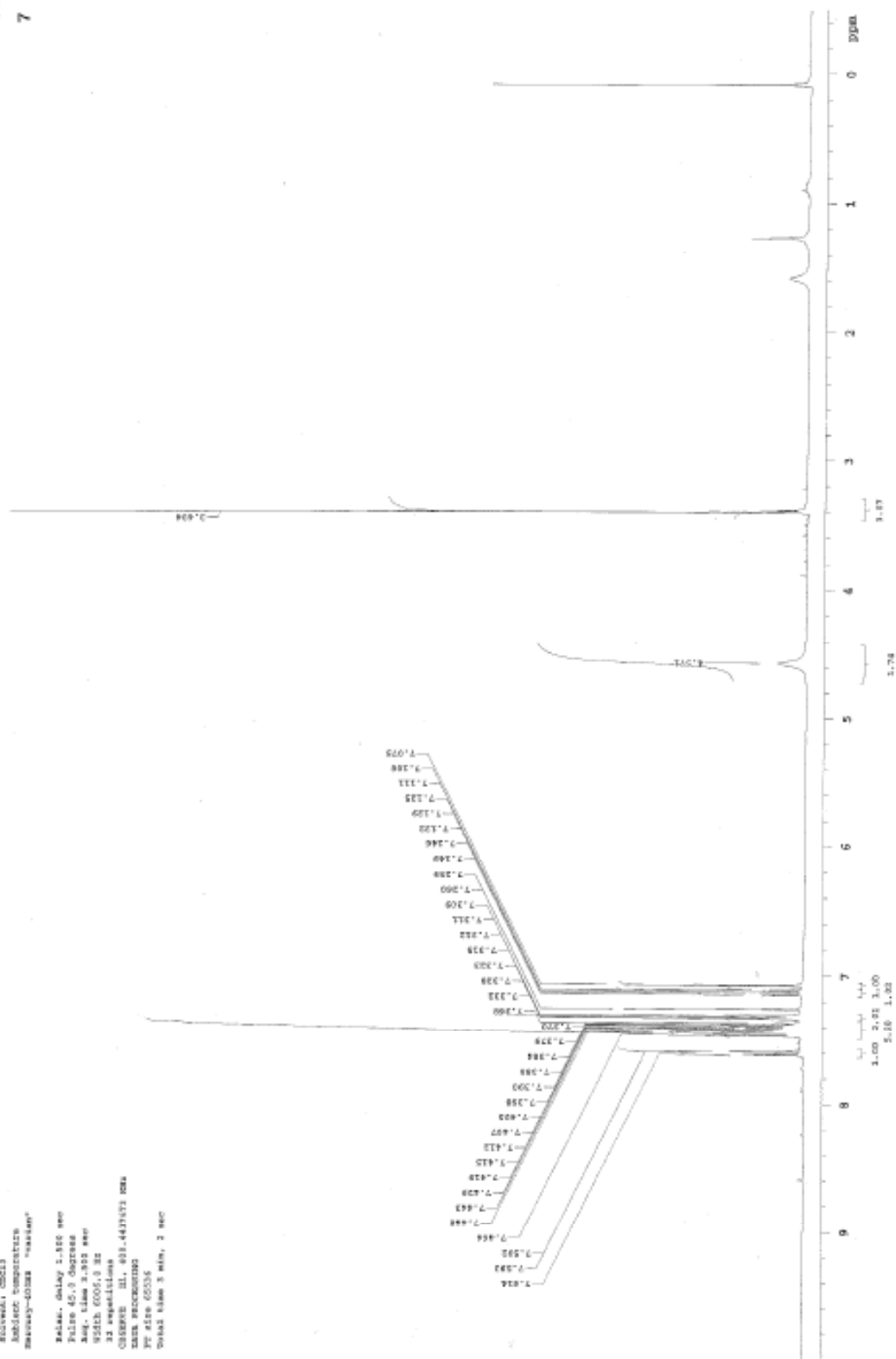
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false: false 45.0 degrees
freq: freq 3.000 sec
width: width 600.0 Hz

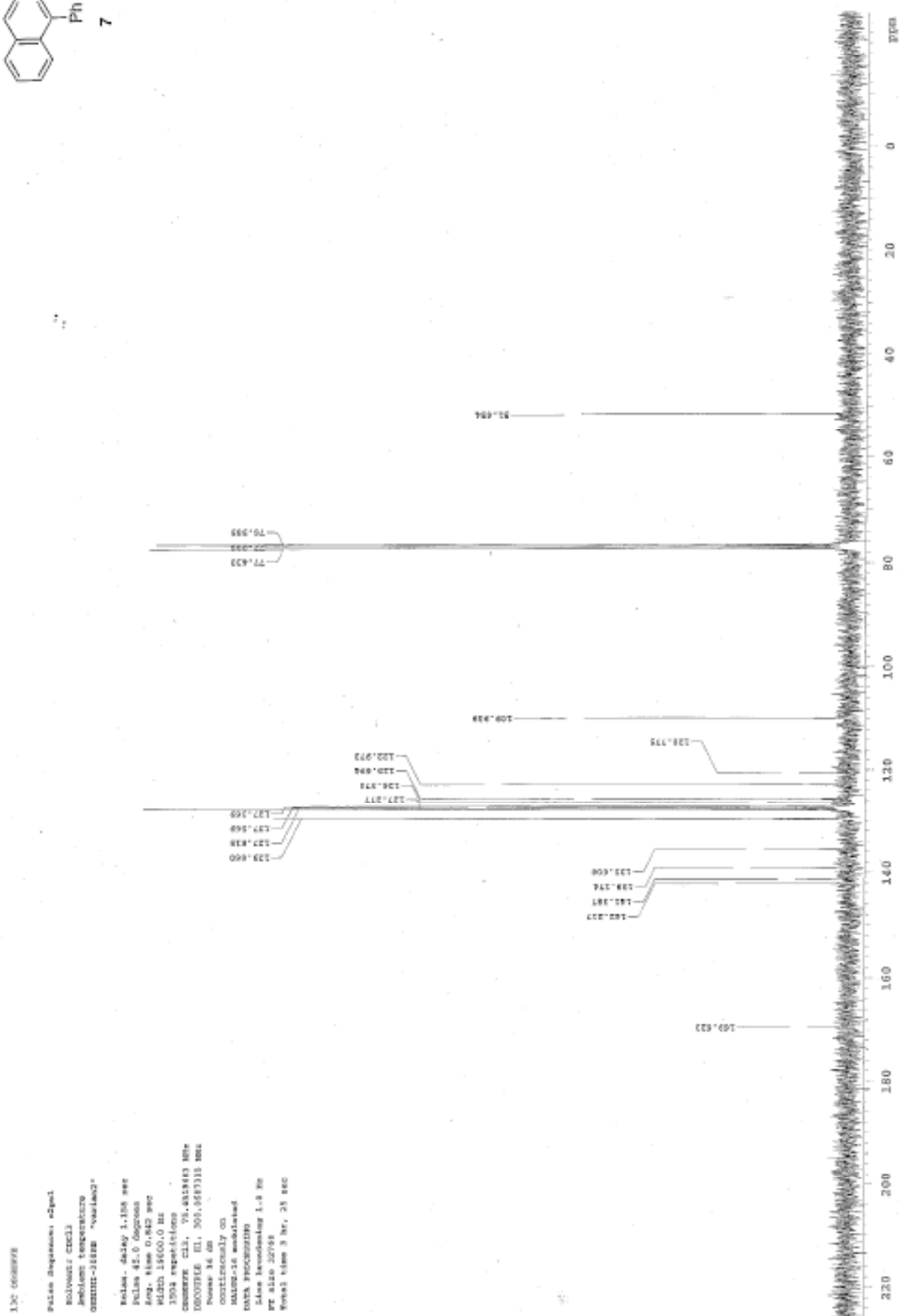
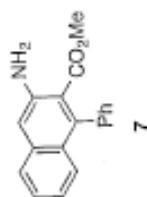
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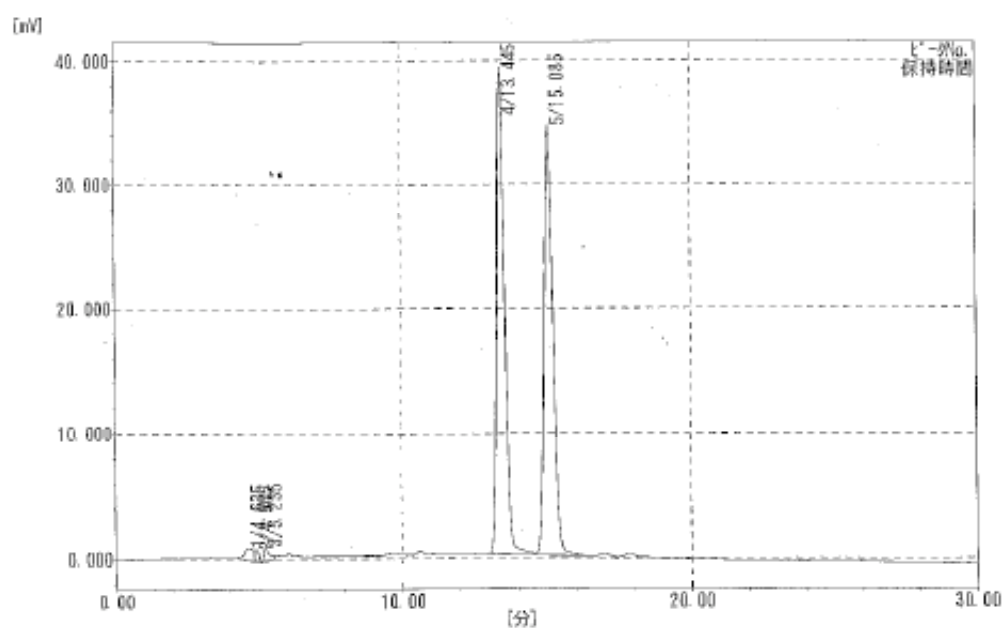
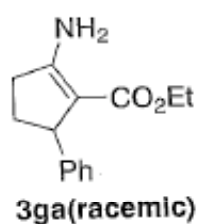
2050000 31, 400, 443, 673 2006

COINTEGRATING NEW

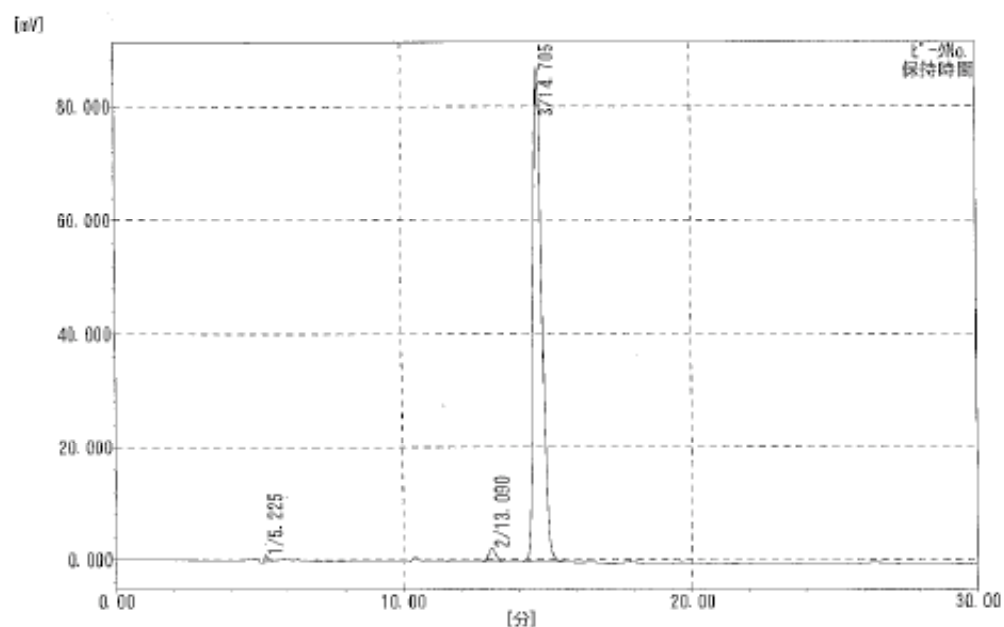
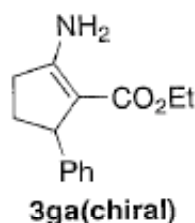
WT size 65536  
Total time 3 min, 3 sec



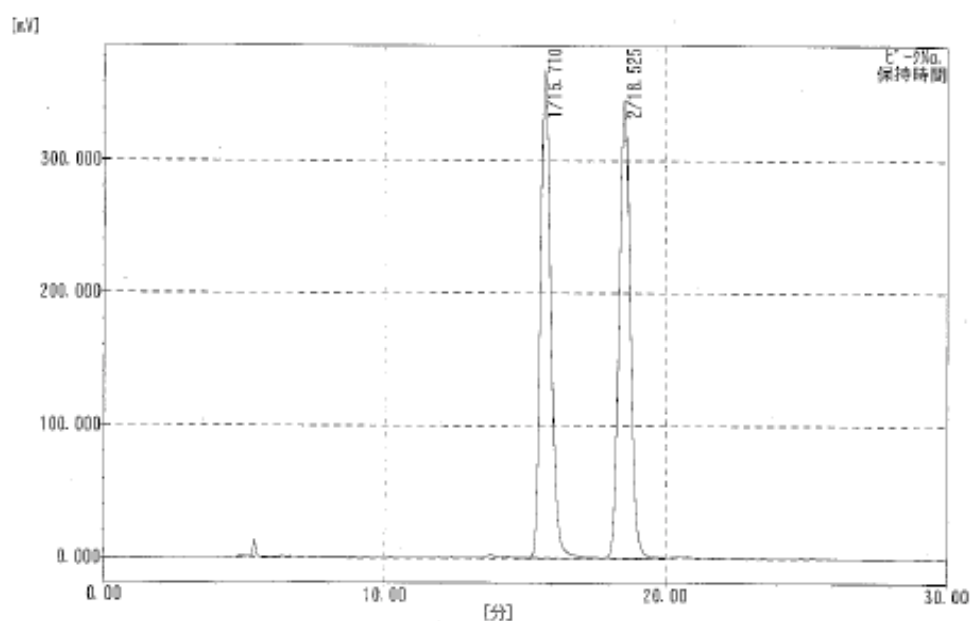
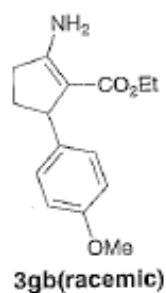




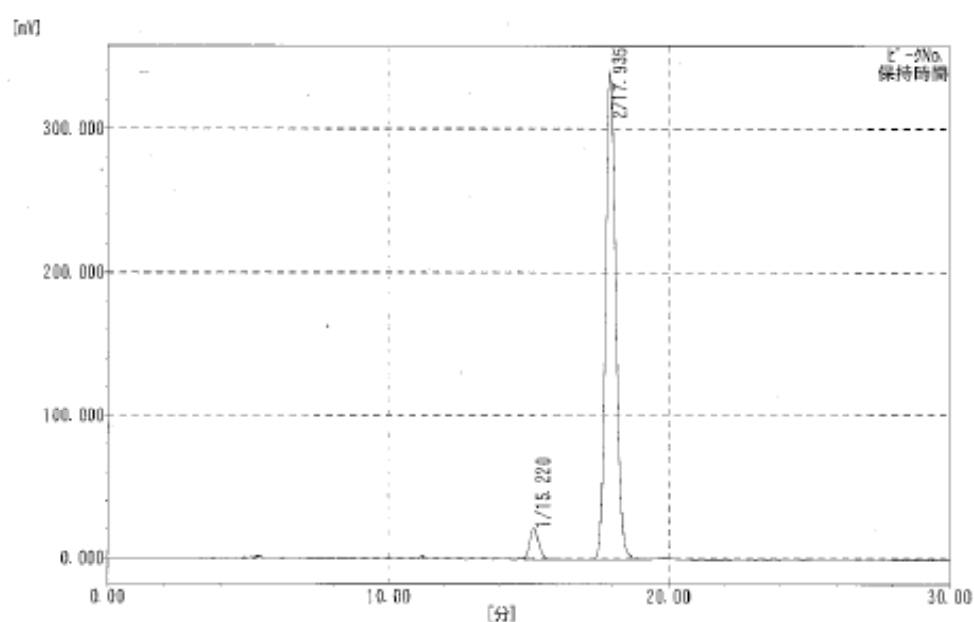
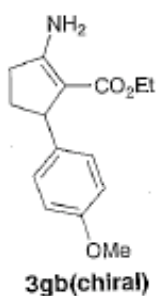
| No. | Ch | 時間[分]  | 高さ[mV] | 半値幅[秒] | 面積[mV×秒] | 面積%    | 理論段数  | 分離能   | 非対称係数 |
|-----|----|--------|--------|--------|----------|--------|-------|-------|-------|
| 1   | 1  | 4.635  | 1.00   | 0.00   | 20.89    | 1.39   | 0     | 0.00  | 0.00  |
| 2   | 1  | 4.905  | 1.02   | 0.00   | 10.66    | 0.71   | 0     | 0.00  | 0.00  |
| 3   | 1  | 5.235  | 1.21   | 11.10  | 16.17    | 1.08   | 4435  | 0.00  | 0.00  |
| 4   | 1  | 13.445 | 39.23  | 16.85  | 728.33   | 48.59  | 12696 | 20.76 | 1.17  |
| 5   | 1  | 15.085 | 34.51  | 19.27  | 722.99   | 48.23  | 12222 | 3.21  | 1.15  |
|     |    | 76.98  |        |        | 1499.04  | 100.00 |       |       |       |



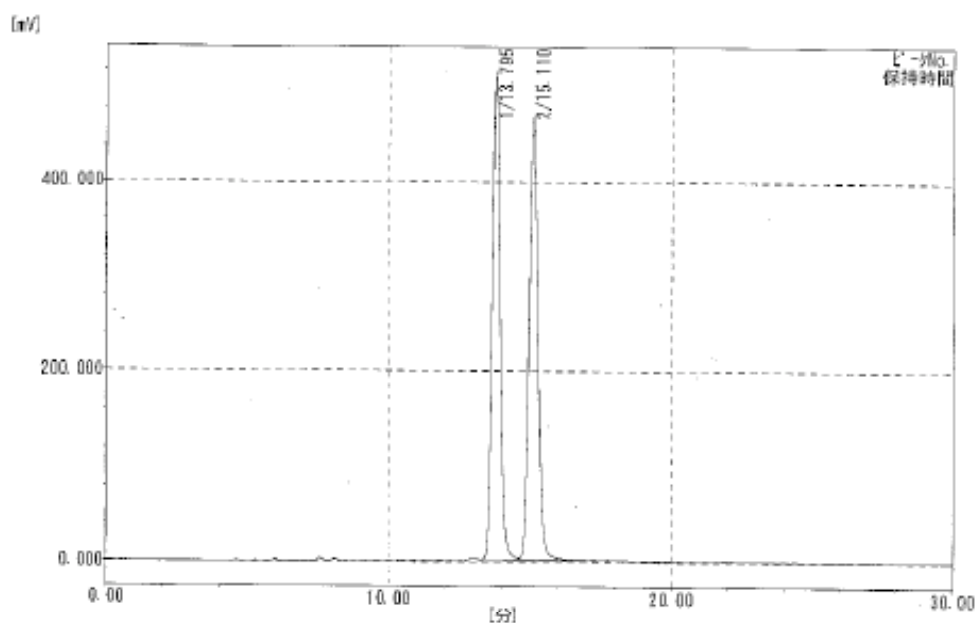
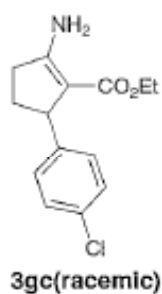
| No. | Ch | 時間[分]  | 高さ[mV] | 半値幅[秒] | 面積[mV×秒] | 面積%    | 理論段数  | 分離能   | 非対称係数 |
|-----|----|--------|--------|--------|----------|--------|-------|-------|-------|
| 1   | 1  | 5.225  | 1.28   | 9.28   | 14.91    | 0.81   | 6319  | 0.00  | 2.27  |
| 2   | 1  | 13.090 | 2.57   | 16.20  | 44.98    | 2.46   | 13014 | 21.81 | 1.07  |
| 3   | 1  | 14.705 | 87.26  | 18.78  | 1771.72  | 96.73  | 12226 | 3.26  | 1.15  |
|     |    | 91.12  |        |        | 1831.62  | 100.00 |       |       |       |



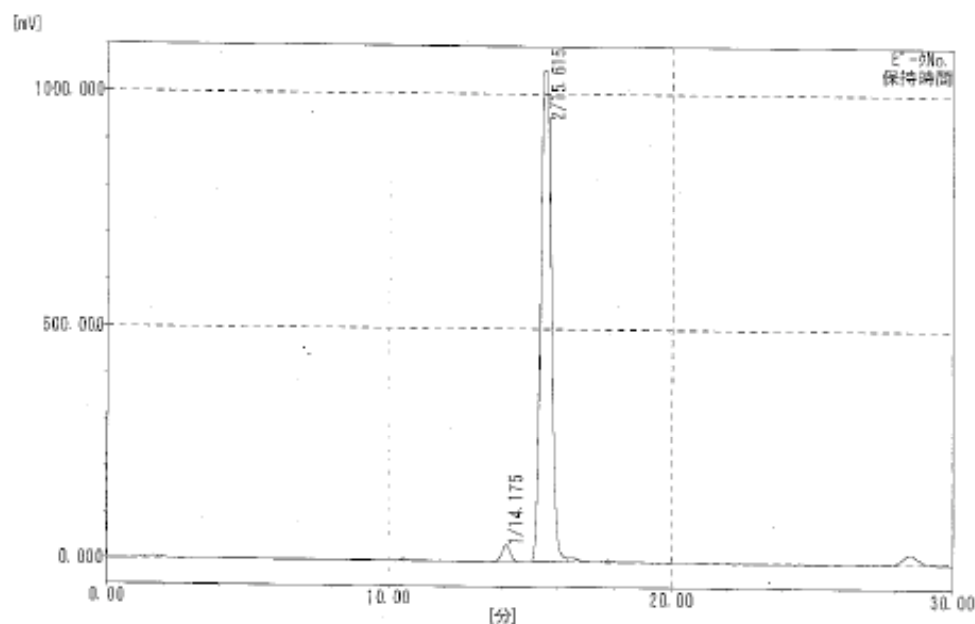
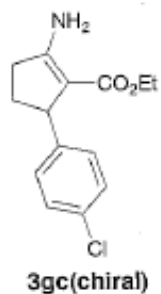
| No. | Ch | 時間 [分] | 高さ [mV] | 半値幅 [秒] | 面積 [mVx秒] | 面積%    | 理論段数  | 分離能  | 非対称係数 |
|-----|----|--------|---------|---------|-----------|--------|-------|------|-------|
| 1   | 1  | 15.710 | 368.73  | 23.46   | 9530.38   | 50.02  | 8943  | 0.00 | 1.56  |
| 2   | 1  | 18.525 | 346.01  | 25.40   | 9521.54   | 49.98  | 10612 | 4.07 | 1.24  |
|     |    | 714.74 |         |         | 19051.91  | 100.00 |       |      |       |



| No. | Ch | 時間 [分] | 高さ [mV] | 半値幅 [秒] | 面積 [mVx秒] | 面積%    | 理論段数  | 分離能  | 非対称係数 |
|-----|----|--------|---------|---------|-----------|--------|-------|------|-------|
| 1   | 1  | 15.220 | 21.80   | 20.93   | 459.46    | 5.43   | 10544 | 0.00 | 1.23  |
| 2   | 1  | 17.935 | 341.11  | 23.53   | 8690.33   | 94.57  | 11582 | 4.32 | 1.20  |
|     |    | 362.91 |         |         | 9189.79   | 100.00 |       |      |       |



| No. | Ch | 時間[分]  | 高さ[mV] | 半値幅[秒] | 面積[mVx秒] | 面積%    | 理論段数  | 分離能  | 非対称係数 |
|-----|----|--------|--------|--------|----------|--------|-------|------|-------|
| 1   | 1  | 13.795 | 518.28 | 17.63  | 9992.03  | 49.73  | 12214 | 0.00 | 1.22  |
| 2   | 1  | 15.110 | 468.90 | 19.60  | 10099.12 | 50.27  | 11855 | 2.50 | 1.23  |
|     |    | 987.18 |        |        | 20091.15 | 100.00 |       |      |       |



| No. | Ch | 時間[分]   | 高さ[mV]  | 半値幅[秒] | 面積[mVx秒] | 面積%    | 理論段数  | 分離能  | 非対称係数 |
|-----|----|---------|---------|--------|----------|--------|-------|------|-------|
| 1   | 1  | 14.175  | 34.39   | 18.69  | 727.03   | 2.58   | 11477 | 0.00 | 1.04  |
| 2   | 1  | 15.615  | 1048.45 | 23.96  | 27406.12 | 97.42  | 8470  | 2.39 | 0.84  |
|     |    | 1082.84 |         |        | 28133.15 | 100.00 |       |      |       |