

## Total Syntheses of Kealiinines A-C

Jayanta Das, Panduka B. Koswatta, J. Daniel Jones, Muhammed Yousufuddin<sup>‡</sup> and Carl J. Lovely\*

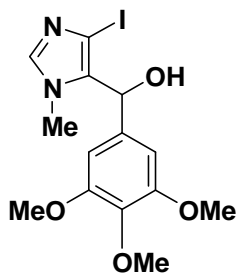
Department of Chemistry and Biochemistry, The University of Texas at Arlington, Arlington, TX 76019

<sup>‡</sup>Center for Nanostructured Materials, The University of Texas at Arlington, Arlington, TX 76019

[lovely@uta.edu](mailto:lovely@uta.edu)

1. Synthetic procedures and characterization data for compounds **14a-c**, **16a-c**, **18a-c**, **19b-c**, **20**, **21** and **8a-c**. S2-S20
2. Comparative listing of <sup>1</sup>H and <sup>13</sup>C NMR spectroscopic data. S21-S26
3. Mercury and ORTEP figures of X-ray structures of compounds **18c**, **20** and **8c**. S27-29
4. <sup>1</sup>H and <sup>13</sup>C NMR spectra for compounds **14a-c**, **16a-c**, **18a-c**, **19b-c**, **20**, **21** and **8a-c**. S30-S75
5. Comparative HPLC plots for natural and synthetic kealiinines A-C and isokealiinine C. S77-S87
6. 2D NMR spectra (ROESY, HSQC, HMBC) for compounds **8b** and **8c**. S88-S100

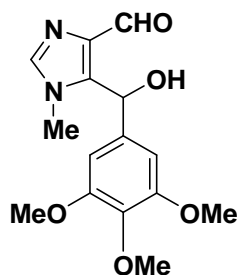
**(5-Iodo-3-methyl-3H-imidazol-4-yl)(3,4,5-trimethoxy-phenyl)-methanol (14c):**



EtMgBr (3.0 M solution in ether, 10.1 mL, 30.3 mmol) was added dropwise to compound **12** (10.0 g, 30.0 mmol) in anhydrous THF (150 mL) at 0 °C. After complete addition of the Grignard reagent, the reaction mixture was stirred for 1 h at room temperature. The aldehyde **13c** (5.87 g, 30.0 mmol) was added to the reaction mixture and then stirred for 5 h at room temperature. The reaction was quenched with aqueous NH<sub>4</sub>Cl solution (50 mL) then the organic layer was separated. The aqueous layer was extracted with EtOAc (3 x 150 mL) and then the combined organic solutions were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by silica gel chromatography (EtOAc) providing **14c** as a pale yellow solid (10.53 g, 87%). m.p. = 195-197 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 7.36 (s, 1H), 6.58 (s, 2H), 6.03 (d, *J* = 2.1 Hz, 1H), 3.83 (s, 3H), 3.81 (s, 6H), 3.47 (s, 3H), 3.39 (d, *J* = 2.1 Hz, 1H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 153.4, 141.3, 137.2, 136.0, 134.6, 102.4, 85.2, 67.5, 61.0, 56.3, 33.5; FT-IR (neat, cm<sup>-1</sup>): 3102, 2942, 1588, 1503, 1121, 1003, 962, 776, 723; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>14</sub>H<sub>17</sub>IN<sub>2</sub>O<sub>4</sub> 405.0306 found 405.0323.

**5-(Hydroxy(3,4,5-trimethoxyphenyl)methyl)-1-methyl-1H-imidazole-4-carbaldehyde (16c):**

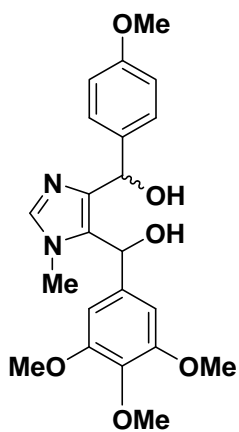
EtMgBr (3.0 M solution in ether, 5.0 mL, 15 mmol) was added dropwise to



compound **14c** (2.88 g, 7.13 mmol) in anhydrous THF (40 mL) at 0 °C. After complete addition of the Grignard reagent, the reaction mixture was stirred for an additional 2 h at room temperature. *N*-Methylformanilide **15** (1.2 mL, 10 mmol) was added to the reaction

mixture dropwise and then reaction mixture was stirred at room temperature for 16 h. The reaction mixture was quenched with water and extracted with ethyl acetate (3 x 120 mL), the combined organic extracts were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by column chromatography (acetone:hexane = 7:1) providing **16c** as a yellow solid (1.53 g, 70%). m.p. = 151-154 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ = 9.92 (s, 1H), 7.48 (s, 1H), 6.50 (s, 2H), 5.97 (d, *J* = 9.6 Hz, 1H), 5.82 (d, *J* = 9.6 Hz, 1H), 3.82 (s, 3H), 3.79 (s, 6H), 3.59 (s, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 189.4, 153.6, 141.6, 139.4, 138.4, 138.0, 136.1, 103.4, 67.8, 60.9, 56.2, 32.9; FT-IR (neat, cm<sup>-1</sup>): 3102, 2958, 1673, 1504, 1450, 1331, 1123, 1057, 712, 653; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>15</sub>H<sub>18</sub>N<sub>2</sub>O<sub>5</sub> 307.1288 found 307.1301.

**5-[Hydroxy-(3, 4, 5-trimethoxyphenyl)-methyl]-1-methyl-1*H*-imidazol-4-yl)-(4-methoxyphenyl)methanol (**17c**):** Magnesium turnings (0.25 g, 10.4 mmol) were

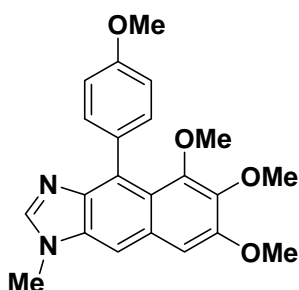


placed in a dry round bottom flask and suspended in dry THF (30 mL). *p*-Bromoanisole (1.30 mL, 10.5 mmol), dissolved in anhydrous THF (30 mL), was added dropwise over ca. 30 mins to maintain a controlled reflux. After complete addition of *p*-bromoanisole, the reaction mixture was refluxed for an additional 3 h. The reaction mixture was cooled to room temperature and compound **16c** (0.80 g, 2.61 mmol) was added to the Grignard

reagent, followed by heating the reaction mixture at reflux for 16 h. Finally the reaction was quenched with aqueous NH<sub>4</sub>Cl solution (60 mL). The reaction mixture was extracted with ethyl acetate (3 x 100 mL), the combined organic layers were washed

with brine solution, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum. The crude product **17c** (crude weight = 1.86 g) was used for next reaction without any further purification.

**5,6,7-Trimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-d]imidazole (18c):**



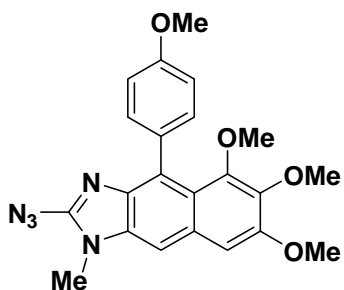
Concentrated HCl (1.5 mL, 18.0 mmol) was added dropwise to the crude diol **17c** (1.08 g, 2.61 mmol) in anhydrous  $\text{CH}_2\text{Cl}_2$  (60 mL) and then stirred at room temperature for 4 h. Water was added and the reaction mixture was extracted with  $\text{CH}_2\text{Cl}_2$  (3 x 50 mL) and the combined organic extracts were

washed with water followed by brine solution, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum. The crude product was purified by column chromatography (acetone: hexane = 4:1) providing **18c** as a light brown solid (0.64 g, 65%). m.p. = 168-170 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.91 (s, 1H), 7.65 (s, 1H), 7.39 (d,  $J$  = 8.5 Hz, 2H), 7.09 (s, 1H), 7.01 (d,  $J$  = 8.5 Hz, 2H), 4.01 (s, 3H), 3.91 (s, 3H), 3.89 (s, 3H), 3.88 (s, 3H), 3.33 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 158.1, 152.0, 150.2, 146.5, 143.3, 140.7, 134.4, 132.3, 130.7, 129.5, 119.6, 112.7, 103.9, 102.0, 70.7, 61.3, 60.7, 55.8, 55.4, 31.2; FT-IR (neat,  $\text{cm}^{-1}$ ): 3106, 2930, 1607, 1236, 1122, 1072, 998, 776; HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{22}\text{H}_{22}\text{N}_2\text{O}_4$  379.1652 found 379.1670.

**2-Azido-5,6,7-trimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-**

**d]imidazole (19c):** Compound **18c** (0.31 g, 0.82 mmol) in anhydrous THF (7 mL) was cooled to -78 °C and then BuLi (1.6 M solution in hexane, 1.1 mL, 1.7 mmol) was added

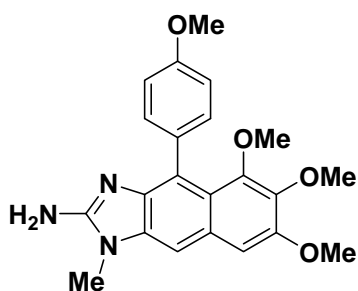




dropwise. After complete addition of the BuLi, the reaction was stirred for 3 h at  $-78\text{ }^{\circ}\text{C}$ , then trisyl azide (0.38 g, 1.23 mmol) dissolved in dry THF (2 mL) was added, followed by stirring the resulting solution for 2 h at room temperature. The reaction was quenched by the addition of satd.  $\text{NH}_4\text{Cl}$

solution (2 mL). The aqueous layer was extracted with EtOAc (3 x 15 mL) and the combined organic extracts were washed with brine solution and dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum and obtained crude material was purified by column chromatography (ethyl acetate: hexane = 2:8) to provide **19c** as a yellow crystalline solid (0.24 g, 69%). Since the product was relatively unstable at room temperature, no melting point was recorded.  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.44 (s, 1H), 7.38 (d,  $J$  = 8.5 Hz, 2H), 7.04 (s, 1H), 7.00 (d,  $J$  = 8.5 Hz, 2H), 4.00 (s, 3H), 3.90 (s, 3H), 3.89 (s, 3H), 3.60 (s, 3H), 3.28 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 158.1, 151.7, 150.2, 150.0, 140.9, 140.5, 135.2, 131.9, 131.1, 128.8, 125.5, 119.6, 112.5, 103.4, 102.2, 61.2, 60.7, 55.8, 55.4, 29.1.

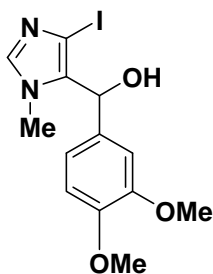
### 2-Amino-5,6,7-trimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-



**d]imidazole (Kealiinine C) (8c):** 10% Pd/C (0.022 g) was added to compound **19c** (0.21 g, 0.50 mmol) dissolved in MeOH (6 mL), the reaction vessel was then connected to a balloon containing hydrogen and the reaction mixture was stirred for 5 h at room temperature. After completion of reaction, the reaction mixture was filtered through a Celite

pad and the pad was washed with hot MeOH (3 x 50 mL) and the filtrate was concentrated to provide a green colored solid which was triturated with dry ether (2 x 10 mL) to produce compound **8c** as a brown colored solid (0.17 g, 85%). m.p. = 304-306 °C; <sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>): 7.36 (s, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.11 (s, 1H), 6.85 (d, *J* = 8.6 Hz, 2H), 6.67 (s, 2H), 3.84 (3H, s), 3.76 (3H, s), 3.69 (3H, s), 3.49 (3H, s), 3.06 (3H, s); <sup>13</sup>C NMR (125 MHz, DMSO-d<sub>6</sub>): δ = 157.9, 157.6, 150.1, 149.5, 142.9, 140.0, 136.3, 133.8, 131.5, 127.0, 120.7, 118.9, 112.3, 103.5, 102.1, 61.0, 60.6, 55.9, 55.5, 28.9; FT-IR (neat, cm<sup>-1</sup>): 3461, 3118, 2929, 1656, 1509, 1195, 857, 776, 545; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>22</sub>H<sub>23</sub>N<sub>3</sub>O<sub>4</sub> 394.1761 found 394.1767.

**(3,4-Dimethoxyphenyl)(4-iodo-1-methyl-1*H*-imidazol-5-yl)methanol (14b):** EtMgBr

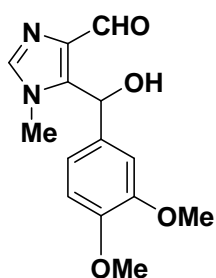


(3.0 M solution in ether, 22.5 mL, 67.4 mmol) was added dropwise to compound **12** (15.0 g, 44.9 mmol) in anhydrous THF (400 mL) at 0 °C. After complete addition of the Grignard reagent, the reaction mixture was stirred for 1 h at room temperature. The aldehyde **13b** (7.46 g, 44.9 mmol) was added

to the reaction mixture and stirred for 16 h at room temperature. The reaction mixture was quenched with aqueous NH<sub>4</sub>Cl solution (150 mL) then the organic layer was separated. The aqueous layer was extracted with ethyl acetate (3 x 250 mL), and then the combined organic layers were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by chromatography (ethyl acetate) providing **14b** as a pale yellow solid (12.07 g, 72%). m.p. = 171-174 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.34 (s, 1H), 6.94 (s, 1H), 6.82 (d, *J* = 8.6 Hz, 1H), 6.79 (d, *J* = 8.6 Hz, 1H), 6.05 (s, 1H), 3.86

(s, 3H), 3.85 (s, 3H), 3.43 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 149.1, 148.4, 141.3, 134.7, 132.7, 117.5, 111.0, 108.8, 85.3, 67.4, 56.0, 33.4; FT-IR (neat,  $\text{cm}^{-1}$ ): 3126, 2959, 2934, 2834, 1589, 1508, 1460, 1413, 1264, 1147, 1033, 975, 779, 752; HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{13}\text{H}_{15}\text{N}_2\text{O}_3$  375.0127 found 375.0178.

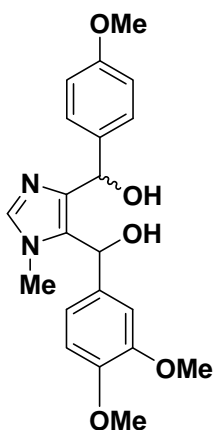
### 5-((3,4-Dimethoxyphenyl)(hydroxy)methyl)-1-methyl-1H-imidazole-4-carbaldehyde



**(16b)**: EtMgBr (3.0 M solution in ether, 10.0 mL, 30.1 mmol) was added dropwise to compound **14b** (5.00 g, 13.3 mmol) in anhydrous THF (100 mL) at 0 °C. After complete addition of Grignard reagent, the reaction mixture was stirred for an additional 2 h at room temperature. *N*-Methylformanilide **15** (1.7 mL, 13.4 mmol) was

added to the reaction mixture dropwise and the reaction mixture was stirred at room temperature for 16 h. The reaction mixture was quenched with water and extracted with ethyl acetate (3 x 200 mL), the combined organic extracts were washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated. The crude product was purified by column chromatography (acetone: hexane = 7:3) providing **16c** as yellow solid (2.6 g, 70%). m.p. = 141-144 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 9.87 (s, 1H), 7.42 (s, 1H), 6.92 (s, 1H), 6.74 (d,  $J$  = 8.0 Hz, 1H), 6.63 (d,  $J$  = 8.6 Hz, 1H), 6.01 (s, 1H), 3.82 (s, 3H), 3.81 (s, 3H), 3.54 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 189.4, 149.5, 149.1, 142.1, 139.2, 138.4, 133.1, 118.3, 111.0, 109.7, 67.7, 56.0, 32.8; FT-IR (neat,  $\text{cm}^{-1}$ ): 3367, 3151, 3093, 2957, 2837, 1683, 1593, 1464, 1349, 1265, 1060, 758, 586, HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{14}\text{H}_{16}\text{N}_2\text{O}_4$  277.1183 found 277.1152.

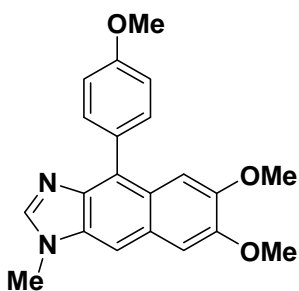
**5-[Hydroxy-(3, 4-dimethoxyphenyl)methyl]-1-methyl-1H-imidazol-4-yl)-(4-methoxyphenyl)methanol (17b):** Magnesium turnings (0.25 g, 10.4 mmol) were placed in a dry



round bottom flask and suspended in dry THF (30 mL). *p*-Bromoanisole (1.3 mL, 10.5 mmol), dissolved in anhydrous THF (30 mL), was added dropwise to maintain a controlled reflux over ca. 30 min. After complete addition of *p*-bromoanisole, the reaction mixture was heated at reflux for an additional 3 h. The reaction mixture was cooled to room temperature then compound **16b** (0.80 g, 2.89 mmol) was added to the Grignard reagent and the reaction mixture was

heated at reflux for 16 h. After cooling to room temperature the reaction was quenched with aqueous NH<sub>4</sub>Cl solution (50 mL). The reaction mixture was extracted with ethyl acetate (3 x 100 mL), the combined organic layers was washed with brine solution and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum. The crude product **17b** (crude weight = 1.86 g) was used for next reaction without any further purification.

**6, 7-Dimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-d]imidazole (18b):**

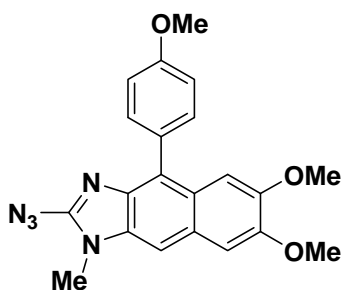


Concentrated HCl (1.43 mL, 17.2 mmol) was added dropwise to the crude diol **6** (1.78 g, 4.63 mmol) in anhydrous CH<sub>2</sub>Cl<sub>2</sub> (30 mL) and then stirred at room temperature for 4 h. Water was added and the reaction mixture was extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 x 70 mL) and the combined organic extracts were washed

with water followed by brine solution, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum and provided the crude product, which was purified by column

chromatography (acetone:hexane = 4:2) providing **18b** as a light brown solid (0.70 g, 70%). m.p. = 207-210 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.92 (s, 1H), 7.64 (s, 1H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.33 (s, 1H), 7.10 (d, *J* = 8.6 Hz, 2H), 4.03 (s, 3H), 3.90 (s, 6H), 3.89 (s, 3H), 3.83 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 159.0, 149.0, 148.2, 146.1, 141.4, 133.9, 132.1, 129.3, 128.1, 127.3, 124.1, 114.1, 105.7, 104.5, 103.2, 55.9, 55.8, 55.3, 31.2; FT-IR (neat, cm<sup>-1</sup>): 3009, 2958, 2827, 1740, 1607, 1511, 1269, 1122, 1050, 860, 787, 619; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>21</sub>H<sub>20</sub>N<sub>2</sub>O<sub>3</sub> 349.1547 found 349.1507.

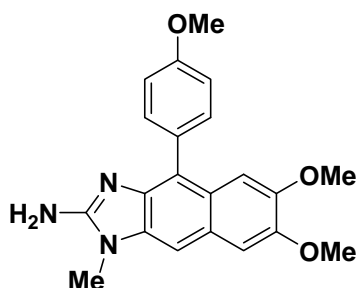
### 2-Azido-6,7-dimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazole



**(19b):** Compound **18b** (0.31 g, 0.89 mmol) in anhydrous THF (7 mL) was cooled to -78 °C and then BuLi (1.6 M solution in hexane, 1.1 mL, 1.7 mmol) was added dropwise. After complete addition of BuLi, the reaction mixture was stirred for 3 h at -78 °C, then trisyl azide (0.413 g, 1.33 mmol) dissolved in dry THF (2 mL) was added and the resulting solution was stirred for 2 h at room temperature. The reaction was quenched by addition of with satd. NH<sub>4</sub>Cl (2 mL). The aqueous layer was extracted with ethyl acetate (3 x 15 mL), and the combined organic extracts were washed with brine solution and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum and obtained crude material was purified by column chromatography (ethyl acetate:hexane = 2:8) to provide **19b** as a yellow solid (0.237 g, 69%). Since the product was relatively unstable at room temperature, no melting point was recorded. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.58 (d, *J* = 8.6 Hz, 2H), 7.44 (s, 1H), 7.31 (s, 1H), 7.21 (s, 1H), 7.09 (d, *J* = 8.6 Hz, 2H), 4.02 (s, 3H), 3.92 (s,

3H), 3.82 (s, 3H), 3.63 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 158.9, 150.0, 148.6, 148.1, 139.1, 134.9, 132.4, 129.0, 126.5, 125.9, 124.4, 113.9, 105.8, 104.7, 102.8, 55.9, 55.7, 55.4, 29.1.

### 6,7-Dimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazol-2-amine

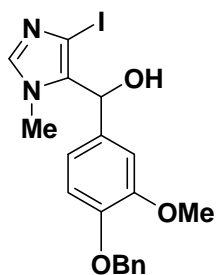


**(Kealiinine B) (8b):** 10% Pd/C (0.025 g) was added to compound **19b** (0.22 g, 0.56 mmol) dissolved in MeOH (6 mL), the reaction vessel was then connected to a balloon containing hydrogen and the reaction mixture was stirred for 5 h at room temperature. After completion of reaction, the mixture was filtered through a Celite pad and the pad

was washed with MeOH (3 x 50 mL) and the filtrate was concentrated to get a green colored solid which was triturated with dry ether (2 x 10 mL) to produce compound **8b** as a brown colored solid (0.179 g, 87%). m.p. = 282-284 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{DMSO-d}_6$ ): 7.40 (d,  $J$  = 8.6 Hz, 2H), 7.34 (s, 1H), 7.26 (s, 1H), 7.09 (s, 1H), 7.01 (d,  $J$  = 8.6 Hz, 2H), 6.63 (s, 2H), 3.82 (s, 3H), 3.80 (s, 3H), 3.60 (s, 3H), 3.51 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{DMSO-d}_6$ ):  $\delta$  = 158.4, 157.8, 147.4, 147.3, 141.3, 135.7, 132.7, 130.4, 124.5, 123.5, 121.2, 113.9, 107.2, 104.6, 101.6, 55.8, 55.6, 55.5, 28.6; FT-IR (neat,  $\text{cm}^{-1}$ ): 3456, 3308, 2987, 2958, 2803, 1657, 1505, 1245, 1031, 839, 775, 621, 566., HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{21}\text{H}_{21}\text{N}_3\text{O}_3$  364.1656 found 364.1614.

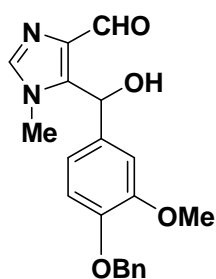
### (4-(Benzyloxy)-3-methoxyphenyl)(4-iodo-1-methyl-1*H*-imidazol-5-yl)methanol

**(14a):** EtMgBr (3.0 M solution in ether, 12.5 mL, 37.6 mmol) was added dropwise to



compound **12** (9.65 g, 28.9 mmol) in anhydrous THF (150 mL) at 0 °C. After complete addition of the Grignard reagent, the reaction mixture was stirred for 1 h at room temperature. The protected vanillin derivative **13a** (7.00 g, 28.9 mmol) was added to the reaction mixture and stirred for 5 h at room temperature. The reaction was quenched with aqueous NH<sub>4</sub>Cl solution (50 mL), and then the

organic layer was separated. The aqueous solution was extracted with ethyl acetate (3 x 200 mL) and then the combined organic extracts were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by chromatography (ethyl acetate) providing alcohol **14a** as a pale yellow solid (9.2 g, 70%). m.p. = 168-171 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.41 (d, *J* = 7.4 Hz, 2H), 7.36-7.32 (m, 3H), 7.29 (d, *J* = 7.4 Hz, 1H), 6.96 (s, 1H), 6.82 (d, *J* = 8.6 Hz, 1H), 6.72 (d, *J* = 8.6 Hz, 1H), 6.02 (s, 1H), 5.13 (s, 2H), 3.86 (s, 3H), 3.42 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 149.8, 147.5, 141.2, 137.1, 134.7, 133.3, 128.7, 127.9, 127.4, 117.5, 113.8, 109.4, 85.2, 71.2, 67.4, 56.1, 33.4; FT-IR (neat, cm<sup>-1</sup>): 3316, 3063, 2961, 2924, 2830, 1604, 1588, 1510, 1466, 1253, 1124, 1026, 995, 772, 745; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>19</sub>H<sub>19</sub>IN<sub>2</sub>O<sub>3</sub> 451.0508 found 451.0474.

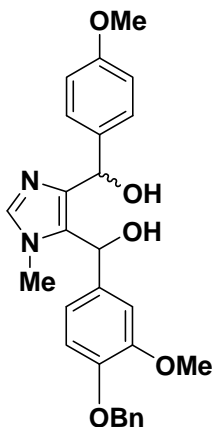


**5-((4-(Benzyloxy)-3-methoxyphenyl)(hydroxy)methyl)-1-methyl-1H-imidazole-4-carbaldehyde (16a):** EtMgBr (3.0 M solution in ether, 5.6 mL, 16.7 mmol) was added dropwise to alcohol **14a** (3.00 g, 6.66 mmol) in anhydrous THF (50 mL) at 0 °C. After complete addition of the Grignard reagent, the reaction mixture was stirred for

an additional 2 h at room temperature. *N*-Methylformanilide **15** (1.30 mL, 10.2 mmol) was added to the reaction mixture dropwise and the reaction mixture was stirred at room temperature for 16 h. The reaction mixture was quenched with water and extracted with ethyl acetate (3 x 120 mL), the combined organic extracts were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by column chromatography (acetone: hexane = 7:1) providing the corresponding aldehyde as yellow solid **16a** (1.65 g, 70%); m.p. = 152-155 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 9.86 (s, 1H), 7.40-7.38 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (d, *J* = 7.4 Hz, 1H), 6.95 (s, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 6.56 (d, *J* = 8.0 Hz, 1H), 6.01 (s, 2H), 5.10 (s, 2H), 3.83(s, 3H), 3.51 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 189.4, 150.1, 148.3, 142.1, 139.2, 138.3, 137.0, 133.6, 128.7, 128.0, 127.3, 118.3, 113.7, 110.2, 71.1, 67.6, 56.1, 32.8; FT-IR (neat, cm<sup>-1</sup>): 3290, 3100, 2964, 2866, 1667, 1512, 1465, 1344, 1254,1232, 1154, 995, 781, 730; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>20</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub> 353.1496 found 353.1488.

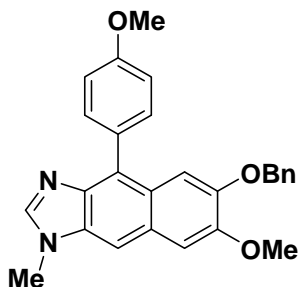
**5-[Hydroxy-(4-benzyloxy-3-methoxyphenyl)methyl]-1-methyl-1*H*-imidazol-4-yl)-(4-methoxyphenyl)methanol (17a):** Magnesium turnings (0.30 g, 12.5 mmol) were placed in a dry round bottom flask and suspended in dry THF (30 mL). *p*-Bromoanisole (1.6 mL, 12.5 mmol), dissolved in anhydrous THF (50 mL), was added dropwise under controlled reflux condition over ca. 30 mins to maintain a controlled reflux. After complete addition of *p*-bromoanisole, the reaction mixture was heated at reflux for an additional 2 h. The reaction mixture was cooled to room temperature and the hydroxyaldehyde derivative **16a** (1.10 g, 3.12 mmol) was added to the Grignard reagent





and the reaction mixture was heated at reflux for 16 h. Finally the reaction was quenched by the addition of aqueous  $\text{NH}_4\text{Cl}$  solution (5 mL). The reaction mixture was extracted with ethyl acetate (3 x 100 mL), the combined organic extracts were washed with brine solution and dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum. The crude product **17a** (crude weight = 2.21 g) was used directly in the next reaction without any purification.

### 8-(Benzyloxy)-7-methoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-

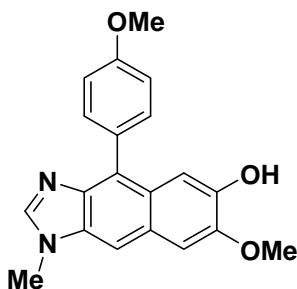


**d]imidazole (18a):** Concentrated HCl (1.0 mL, 12.5 mmol) was added dropwise to the crude diol **17a** (1.43 g, 3.12 mmol) in anhydrous  $\text{CH}_2\text{Cl}_2$  (40 mL) and then stirred at room temperature for 4 h. Water was added and the reaction mixture was extracted with  $\text{CH}_2\text{Cl}_2$  (3 x 50 mL) and the combined organic extracts were washed with water followed

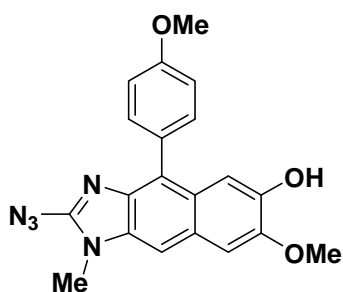
by brine solution, dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum to provide the crude product which was purified by column chromatography (ethyl acetate : hexane = 9:1) to afforded **18a** as a light brown solid (0.60 g, 65%); m.p. = 220-223 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.90 (s, 1H), 7.39 (d,  $J$  = 8.6 Hz, 2H), 7.37-7.27 (m, 6H), 7.04 (d,  $J$  = 8.6 Hz, 2H), 5.13 (s, 2H), 4.04 (s, 3H), 3.92 (s, 3H), 3.86 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 158.9, 149.3, 150.2, 146.9, 146.1, 141.3, 137, 134, 132.1, 129.1, 128.6, 128, 127.8, 127.5, 127.4, 123.9, 114.1, 107.2, 105.9, 103.1, 70.5, 56.0, 55.5,

31.2; FT-IR (neat,  $\text{cm}^{-1}$ ): 3024, 2960, 2830, 1606, 1511, 1486, 1269, 1212, 1024, 857, 786, 727.; HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{27}\text{H}_{24}\text{N}_2\text{O}_3$  425.1854 found 425.1827.

**7-Methoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-*d*]imidazol-6-ol (20):**



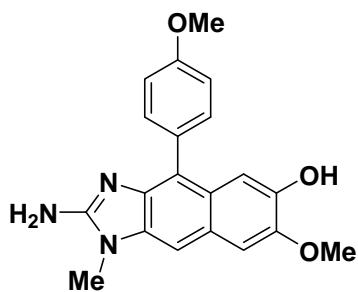
20%  $\text{Pd}(\text{OH})_2/\text{C}$  (0.035 g) was added to compound **18a** (0.35 g, 0.83 mmol) dissolved in MeOH (10 mL), the reaction vessel was then subjected to hydrogenation at 60 psi of hydrogen at 40 °C for 12 h. After completion of reaction, the reaction mixture was filtered through a Celite pad and the pad was washed with MeOH (3 x 20 mL) and the filtrate was concentrated to get a red-colored solid. **20** (0.245 g, 89%). m.p. = 235-238 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.93 (s, 1H), 7.62 (s, 1H), 7.50 (d,  $J$  = 8.6 Hz, 2H), 7.44 (s, 1H), 7.24 (s, 1H), 7.05 (d,  $J$  = 8.6 Hz, 2H), 5.88 (s, 1H), 4.04 (s, 3H), 3.88 (s, 3H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 159.0, 147.0, 146.2, 144.3, 141.2, 133.7, 132.1, 129.2, 127.9, 127.1, 124.8, 114.0, 107.6, 105.0, 103.4, 55.9, 55.5, 31.2; FT-IR (neat,  $\text{cm}^{-1}$ ): 3221, 3127, 2997, 2928, 2829, 1737, 1624, 1608, 1521, 1267, 1038, 846, 794, 635, 594; HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{20}\text{H}_{18}\text{N}_2\text{O}_3$  335.139 found 335.1355.



**2-Azido-7-methoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-d]imidazol-6-ol (21):**

Compound **20** (0.30 g, 0.89 mmol) in anhydrous THF (7 mL) was cooled to -78 °C and then *n*-BuLi (1.6 M solution in hexane, 1.2 mL, 1.9 mmol) was added dropwise. After complete addition of *n*-BuLi, the reaction was stirred for 2 h at -78 °C, then trisyl azide (0.413 g, 1.33 mmol) dissolved in dry THF (4 mL) was added dropwise and the resulting solution was stirred for 2 h at room temperature. The reaction was quenched by addition of with satd. NH<sub>4</sub>Cl solution (4 mL). The aqueous layer was extracted with ethyl acetate (3 x 20 mL), and combined organic extracts were washed with brine solution and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum and obtained crude material was purified by column chromatography (ethyl acetate: hexane = 4:6) to provide **21** as a light yellow solid (0.144 g, 43%). Since the product was relatively unstable at room temperature, no melting point was recorded. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.52 (d, *J* = 8.6 Hz, 2H), 7.43 (s, 1H), 7.42 (s, 1H), 7.20 (s, 1H), 7.06 (d, *J* = 8.6 Hz, 2H), 5.75 (s, 1H), 4.03 (s, 3H), 3.90 (s, 3H), 3.62 (s, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>): δ = 158.90, 150.2, 146.6, 144.2, 139.2, 134.8, 132.4, 128.9, 126.3, 126.0, 125.1, 113.9, 107.8, 105.2, 102.9, 55.9, 55.4, 29.1.

**2-amino-7-methoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-d]imidazol-6-ol (Kealiinine A) (8a):** 10% Pd/C (0.012 g) was added to compound **21** (0.12 g, 0.31 mmol) dissolved in MeOH (5 mL), the reaction vessel was then connected to a ballon containing hydrogen and the reaction mixture was stirred for 5 h at room temperature.

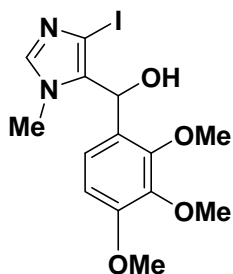


After completion of reaction, the reaction was filtered through Celite pad and the pad was washed with MeOH (3 x 15 mL) and the filtrate was concentrated to get a green-colored solid which was triturated with dry ether (2 x 10 mL) to produce compound **8a** as a green-colored

solid (0.10 g, 90%). m.p. = 276-279 °C; <sup>1</sup>H NMR (500 MHz, DMSO-d<sub>6</sub>): δ = 8.82 (s, 1H), 7.31 (d, *J* = 8.5 Hz, 2H), 7.28 (s, 1H), 7.20 (s, 1H), 7.01 (s, 1H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.57 (s, 2H), 3.82 (3H, s), 3.79 (3H, s), 3.50 (3H, s); <sup>13</sup>C NMR (125 MHz, DMSO-d<sub>6</sub>): δ = 158.3, 157.7, 147.3, 144.9, 141.2, 135.0, 132.6, 130.9, 124.4, 124.0, 120.4, 113.8, 107.8, 107.1, 101.3, 55.8, 55.7, 28.9; FT-IR (neat, cm<sup>-1</sup>): 3448, 3425, 2997, 2929, 2831, 1656, 1508, 1243, 1029, 840, 770, 622, 566; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>20</sub>H<sub>19</sub>N<sub>3</sub>O<sub>3</sub> 350.1499, found 350.1456.

**(5-Iodo-3-methyl-3H-imidazol-4-yl)-(3,4,5-trimethoxyphenyl)methanol:** EtMgBr

(10.1 mL, 30.3 mmol) was added dropwise to compound **12** (10.0 g, 24.9 mmol) in anhydrous THF (150 mL) at 0 °C. After complete addition of Grignard reagent, the reaction mixture was stirred for 1 h at room temperature. The aldehyde (5.87 g, 30.0 mmol) was added to the reaction mixture and stirred for 5 h at room temperature. The reaction was quenched with aqueous NH<sub>4</sub>Cl



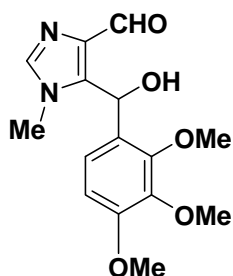
solution (50 mL) then the organic layer was separated. The aqueous layer was extracted with EtOAc (3x150 mL), and then the combined organic solutions were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated. The crude product was purified by

chromatography (EtOAc) providing the alcohol as a pale yellow solid (10.1 g, 83%). m.p. = 190-192 °C; <sup>1</sup>H NMR (500 MHz): δ = 7.37 (s, 1H), 6.82 (d, *J* = 8.6 Hz, 1H), 6.61 (d, *J* = 8.6 Hz, 1H), 6.12 (d, *J* = 2.9 Hz, 1H), 3.86 (s, 6H), 3.85 (s, 3H), 3.56 (s, 3H); 3.37 (d, *J* = 3.5 Hz, 1H); <sup>13</sup>C NMR (125 MHz): δ = 154.0, 151.2, 143.7, 141.1, 132.9, 126.3, 121.3, 106.6, 65.7, 60.9, 56.1, 33.7 ; FT-IR (neat, cm<sup>-1</sup>): 3109, 2955, 2833, 2668, 1667, 1534, 1502, 1203, 1196, 1156, 1024, 938, 751, 636 ; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>14</sub>H<sub>17</sub>N<sub>2</sub>O<sub>4</sub> 405.0306 found 405.0301.

### 5-(Hydroxy-(3,4,5-trimethoxyphenyl)methyl)-1-methyl-1H-imidazole-4-

**carbaldehyde):** 3M EtMgBr (3.96 mL, 11.9 mmol) was added dropwise to alcohol (2.00

g, 4.95 mmol) in anhydrous THF (40 mL) at 0 °C. After complete



addition of Grignard reagent, the reaction mixture was stirred for an

additional 2 h at room temperature. *N*-Methylformanilide (0.74 mL,

5.94 mmol) was added to the reaction mixture dropwise and the

reaction mixture was stirred at room temperature for 16 h. The

reaction mixture was quenched with water and extracted with ethyl

acetate (3 x 120 mL), the combined organic extracts were washed with brine, dried over

anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated .The crude product was purified by column

chromatography (acetone: hexane = 7:1) providing the hydroxy aldehyde as yellow solid

(1.4 g, 92%). m.p. = 152-154 °C; <sup>1</sup>H NMR (500 MHz): δ = 9.87 (s, 1H), 7.43 (s, 1H),

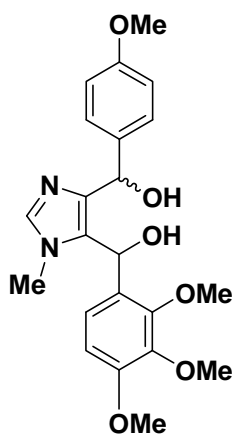
6.85 (d, *J* = 8.6 Hz, 1H), 6.59 (d, *J* = 8.6 Hz, 1H), 6.19 (d, *J* = 10.3 Hz, 1H), 6.15 (d, *J* =

10.3 Hz, 1H), 3.91 (s, 3H), 3.86 (s, 3H), 3.82 (s, 3H); 3.57 (s, 3H), 3.48 (d, *J* = 5.1 Hz,

1H); <sup>13</sup>C NMR (125 MHz): δ = 189.7, 153.6, 150.9, 142.7, 141.6, 139.2, 138.6, 126.1,

122.3, 108.4, 63.2, 61.9, 56.2, 32.8; FT-IR (neat,  $\text{cm}^{-1}$ ): 3037, 2968, 2873, 1668, 1600, 1524, 1461, 1435, 1310, 1203, 975, 708; HR-MS ( $m/z$ ): calc for  $[\text{M}+\text{H}]^+$   $\text{C}_{15}\text{H}_{18}\text{N}_2\text{O}_5$  307.1288 found 307.1275.

**5-[Hydroxy-(3, 4, 5-trimethoxyphenyl)methyl]-1-methyl-1H-imidazol-4-yl)-(4-methoxyphenyl)methanol:** Magnesium turnings (407 mg, 16.9 mmol) were placed in a

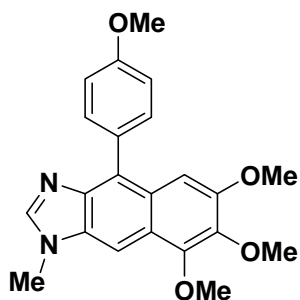


dry round bottom flask and suspended in dry THF (50 mL). *p*-Bromoanisole (2.14 mL, 17.0 mmol), dissolved in anhydrous THF (30 mL), was added dropwise over ca. 30 mins to maintain a controlled reflux. After complete addition of *p*-bromoanisole, the reaction mixture was heated at reflux for an additional 3 h. The reaction mixture was cooled to room temperature and the aldehyde prepared above (1.3 g, 4.24 mmol) was added to the

Grignard reagent and the reaction mixture was heated at reflux for 16 h. Finally the cooled reaction was quenched with aqueous  $\text{NH}_4\text{Cl}$  solution. The reaction mixture was extracted with ethyl acetate (3x100 mL), the combined organic layers was washed with brine solution and dried over anhydrous  $\text{Na}_2\text{SO}_4$  and concentrated under vacuum. The crude product (0.9 g) was used for next reaction without any purification.

**5,6,7-Trimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-*d*]imidazole:**

Concentrated HCl (0.56 mL, 6.76 mmol) was added to the reaction mixture dropwise to the crude diol **6** (0.7 g, 1.69 mmol) in anhydrous  $\text{CH}_2\text{Cl}_2$  (60 mL) and then stirred at room temperature for 4 h. Water was added and the reaction mixture was extracted

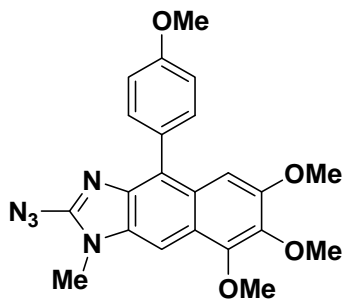


with CH<sub>2</sub>Cl<sub>2</sub> (3x50 mL) and the combined organic extracts were washed with water followed by brine solution, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum and provided crude product was purified by column chromatography (acetone:hexane = 4:1) providing the naphthimidazole as a light brown solid (410 mg, 65%). m.p. =

172-175 °C; <sup>1</sup>H NMR (500 MHz): δ = 8.02 (s, 1H), 7.96 (s, 1H), 7.55 (d, *J* = 8.6 Hz, 2H), 7.14 (s, 1H), 7.10 (d, *J* = 8.6 Hz, 2H), 4.14 (s, 3H), 4.01 (s, 3H), 3.93 (s, 3H), 3.90 (s, 3H), 3.81 (s, 3H); <sup>13</sup>C NMR (125 MHz): δ = 158.7, 150.9, 146.8, 142.3, 140.4, 133.5, 132.1, 129.3, 128.72, 125.7, 122.9, 114.2, 101.4, 99.3, 61.2, 55.8, 55.3, 31.3; FT-IR (neat, cm<sup>-1</sup>): 3627, 2998, 2958, 2830, 1607, 1541, 1311, 1198, 835, 770; HR-MS (*m/z*): calc for [M+H]<sup>+</sup> C<sub>22</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub> 379.1652 found 379.1655.

### 2-azido-5,6,7-trimethoxy-4-(4-methoxyphenyl)-1-methyl-1*H*-naphtho[2,3-

*d*]imidazole: The naphthimidazole (250 mg, 0.66 mmol) in anhydrous THF (7 mL) was



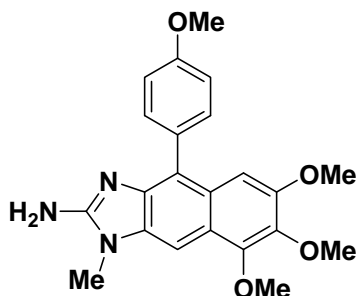
cooled to -78 °C and then BuLi (1.6 M in hexane, 1.28 mL, 2.05 mmol) was added dropwise. After complete addition of the BuLi, the reaction was stirred for 3 h at -78 °C, then trisyl azide (307 mg, 0.99 mmol) was added and the resulting solution was stirred for 2 h at room temperature.

The reaction was quenched by addition of satd. NH<sub>4</sub>Cl (2 mL). The aqueous layer was extracted with EtOAc (3 x 15 mL), and the combined organic extracts were washed with brine solution and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and concentrated under vacuum

and obtained crude material was purified by column chromatography (20% EtOAc:Hexane) to provide the azide as a yellow crystalline solid (215 mg, 78%).  $^1\text{H}$  NMR (300 MHz):  $\delta$  = 7.81 (s, 1H), 7.55 (d,  $J$  = 8.1 Hz, 2H), 7.11 (s, 1H), 7.09 (d,  $J$  = 8.1 Hz, 2H), 4.12 (s, 3H), 3.99 (s, 3H), 3.92 (s, 3H), 3.80 (s, 3H), 3.66 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 158.7, 150.6, 147.9, 146.1, 140.3, 139.4, 135.4, 131.8, 131.7, 128.8, 124.8, 124.3, 122.1, 113.9, 101.3, 98.3, 61.2, 56.2, 55.3, 29.3.

**5,6,7-Trimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-d]imidazol-2-**

**amine (Isokealiinine C):** 10% Pd/C (20 mg) was added to the azide (200 mg, 0.48



mmol) dissolved in MeOH (6 mL), the reaction vessel was

then connected to a balloon containing hydrogen for 12 h.

After completion of reaction, the reaction mixture was

filtered through a Celite pad and the pad was washed with

hot MeOH (3 x 50 mL); the filtrate was concentrated to get

a green colored solid which was triturated with dry ether (2 x 10 mL) to produce

isokealiinine C as a brown-colored solid (180 mg, 96%). m.p. = 276-280 °C.  $^1\text{H}$  NMR

(DMSO- $d_6$ , 500 MHz): 7.49 (s, 1H), 7.38 (d,  $J$  = 8.6 Hz, 2H), 7.02 (d,  $J$  = 8.6 Hz, 2H),

6.91 (s, 1H), 6.75 (s, 2H), 3.96 (s, 3H), 3.79 (s, 3H), 3.63 (s, 3H), 3.55 (s, 3H), 3.33 (s,

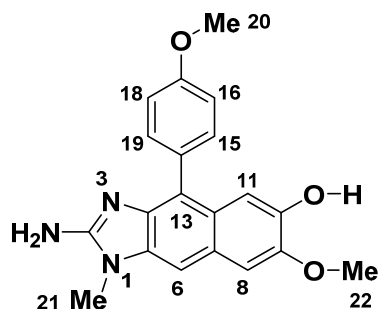
3H);  $^{13}\text{C}$  NMR (75 MHz):  $\delta$  = 158.4, 150.1, 147.4, 142.8, 137.7, 135.9, 132.7, 130.1,

125.3, 121.5, 119.2, 113.9, 100.2, 96.4, 70.1, 61.6, 61.2, 55.7, 55.6, 28.9; FT-IR (neat,

$\text{cm}^{-1}$ ): 3464, 2935, 2824, 1653, 1548, 1498, 1103, 1030, 833, 734; HR-MS ( $m/z$ ): calc

for  $[\text{M}+\text{H}]^+$   $\text{C}_{22}\text{H}_{23}\text{N}_3\text{O}_4$  394.1761 found 394.1773.



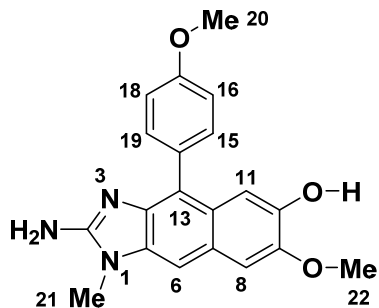


**Table S1:**  $^1\text{H}$  NMR spectroscopic data for kealiinine A ( $\text{DMSO-}d_6$ )

Assignment	Kealiinine A (Nat) <sup>a</sup>	Assignment	Kealiinine A (Synth) <sup>b</sup>
NH (C2)	8.30		
NH (C3)	12.10		
H10	9.49	H10 (OH)	8.82
H6	7.71	H15/H19	7.31
H8	7.40	H6	7.28
H15/19	7.35	H8	7.20
H16/H18	7.18	H11	7.01
H11	6.95	H16/H18	7.00
		NH <sub>2</sub>	6.57
H22	3.90	H22	3.82
H20	3.88	H20	3.79
H21	3.67	H21	3.50

a. Based on assignments from *J. Nat. Prod.* **2004**, 67, 817.

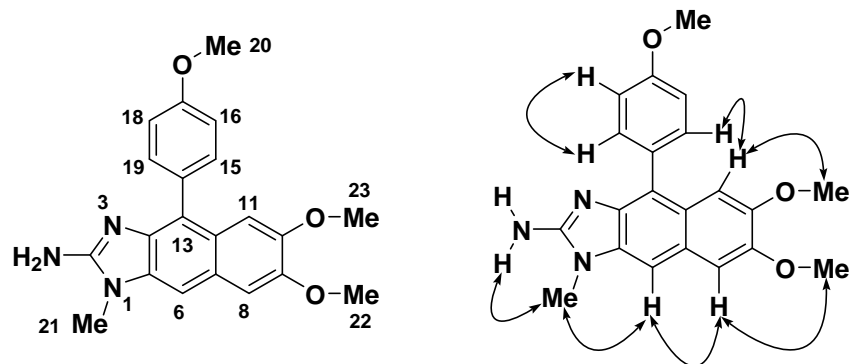
b. Assignments based on analogy with kealiinine B.



**Table S2:**  $^{13}\text{C}$  NMR spectroscopic data for kealiinine A (DMSO- $d_6$ )

Assignment	Kealiinine A (Nat) <sup>a</sup>	Assignment	Kealiinine A (Synth) <sup>b</sup>
C17	159.0	C17	158.3
C2	152.0	C2	157.7
C9	148.1	C9	147.3
C10	146.5	C10	144.9
C15/19	131.5	C4	141.2
C5	129.5	C5	135.0
C14	127.0	C15/19	132.6
C4	125.0	C14	130.9
C7	125.0	C12	124.4
C12	124.8	C7	124.0
C13	118.5	C13	120.4
C16/18	114.2	C16/18	113.8
C11	107.0	C8	107.8
C8	106.5	C11	107.1
C6	104.4	C6	101.3
C22	55.8	C22	55.8
C20	55.5	C20	55.7
C21	28.1	C21	28.9

- a. Based on assignments from *J. Nat. Prod.* **2004**, 67, 817.  
 b. Assignments based on analogy with kealiinine B.



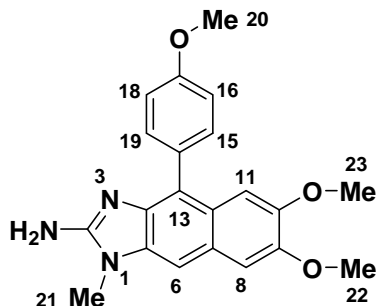
**Table S3:**  $^1\text{H}$  NMR spectroscopic data for kealiinine B (DMSO- $d_6$ )

Assignment	Kealiinine B (Nat) <sup>a</sup>	Assignment	Kealiinine B (Syn) <sup>b</sup>	Kealiinine B (Syn) <sup>c</sup>
H6	7.64	H15/H19	7.40	7.44
H8	7.32	H6	7.34	7.37
H15/H19	7.22	H8	7.26	7.29
H11	7.10	H11	7.09	7.13
H16/H18	6.97	H16/H18	7.01	7.05
		NH <sub>2</sub>	6.63	6.68
H22	3.91	H22	3.82	3.86
H20	3.89	H20	3.80	3.83
H21	3.74	H23	3.60	3.64
H23	3.72	H21	3.51	3.55

a. Based on assignments from *J. Nat. Prod.* **2004**, 67, 817.

b. Assignments based HSQC, HMBC and ROESY experiments (NOE's shown above)

c. Data reported by Looper and coworkers *Org. Lett.* **2012**, 14, 4734.

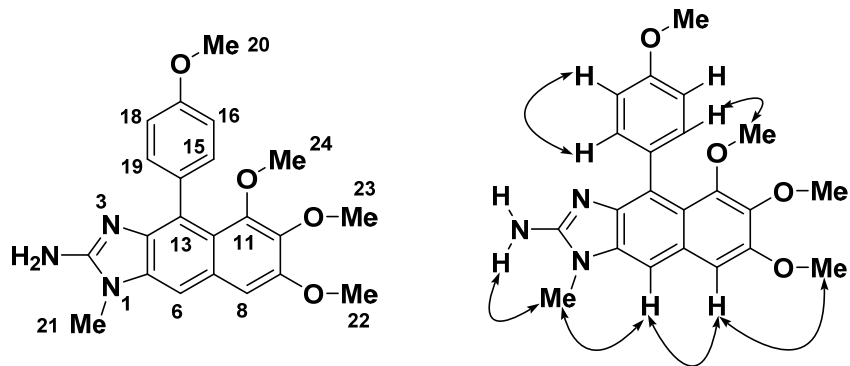


**Table S4:**  $^{13}\text{C}$  NMR spectroscopic data for kealiinine B ( $\text{DMSO-}d_6$ )

Assignment	Kealiinine B (Syn) <sup>a</sup>	Kealiinine B (Syn) <sup>b</sup>
C17	158.4	157.9
C2	157.8	157.4
C9	147.4	147.0
C10	147.3	146.9
C4	141.3	140.8
C5	135.7	135.2
C15/19	132.7	132.2
C14	130.4	130.0
C12	124.5	124.1
C7	123.5	123.1
C13	121.2	120.8
C16/18	113.9	113.4
C8	107.2	106.8
C11	104.6	104.2
C6	101.6	101.1
C22	55.8	55.3
C20	55.6	55.1
C23	55.5	55.0
C21	28.6	28.5

a. Assignments based HSQC, HMBC and ROESY experiments (NOE's shown above)

b. Data reported by Looper and coworkers *Org. Lett.* **2012**, *14*, 4734; assignments are based on analogy to our data.



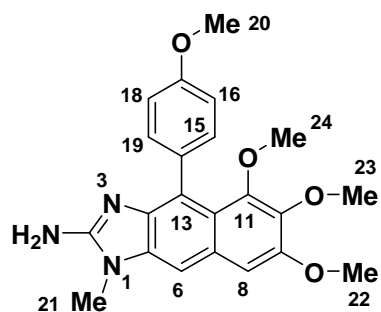
**Table S5:**  $^1\text{H}$  NMR spectroscopic data for kealiinine C (DMSO- $d_6$ )

Assignment	Kealiinine C (Nat) <sup>a</sup>	Assignment	Kealiinine C (Syn) <sup>b</sup>	Kealiinine C (Syn) <sup>c</sup>
H6	7.66	H6	7.36	7.85
H15/H19	7.33	H15/H19	7.13	7.26
H8	7.19	H8	7.11	7.32
H16/H18	7.09	H16/H19	6.85	7.06
		NH <sub>2</sub>	6.67	
H22	3.99	H22	3.84	3.92
H20	3.85	H20	3.76	3.85
H23	3.83	H23	3.69	3.76
H21	3.73	H21	3.49	3.68
H24	3.40	H24	3.06	3.16

a. Based on assignments from *J. Nat. Prod.* **2004**, 67, 817.

b. Assignments based HSQC, HMBC and ROESY experiments (NOE's shown above)

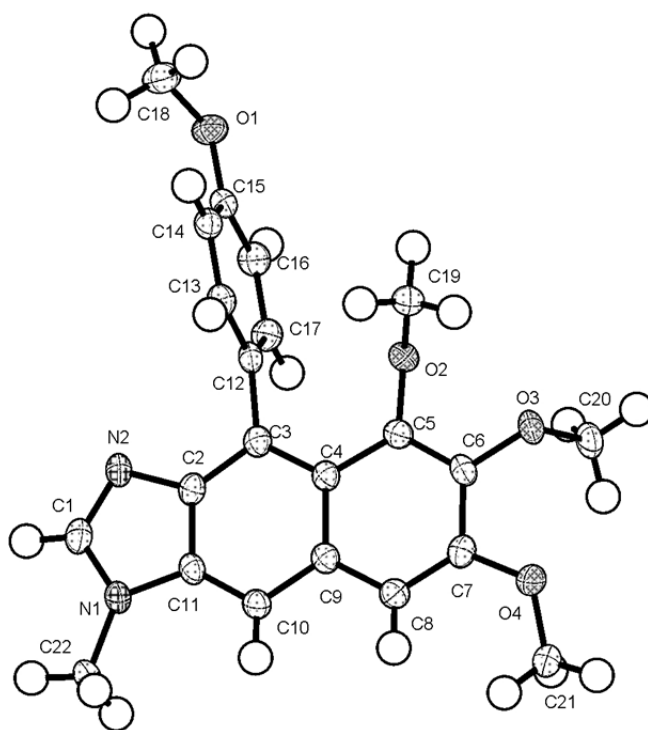
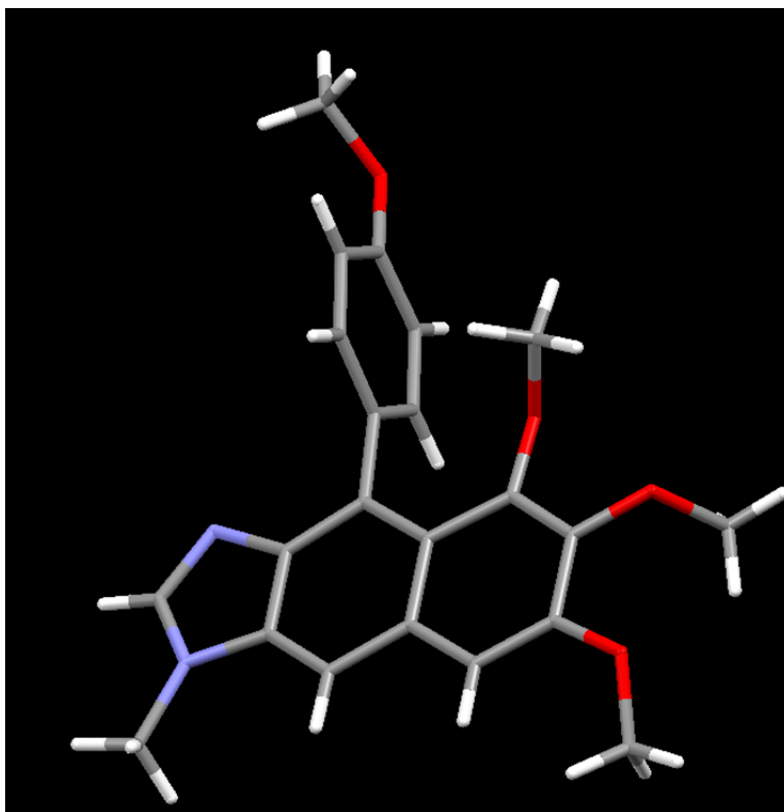
c. Data reported for the TFA salt by Looper and coworkers *Org. Lett.* **2012**, 14, 4734.



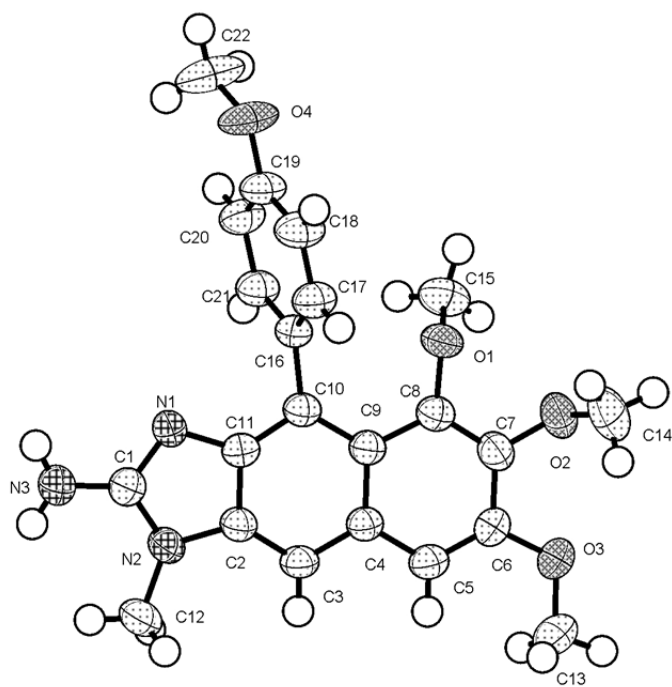
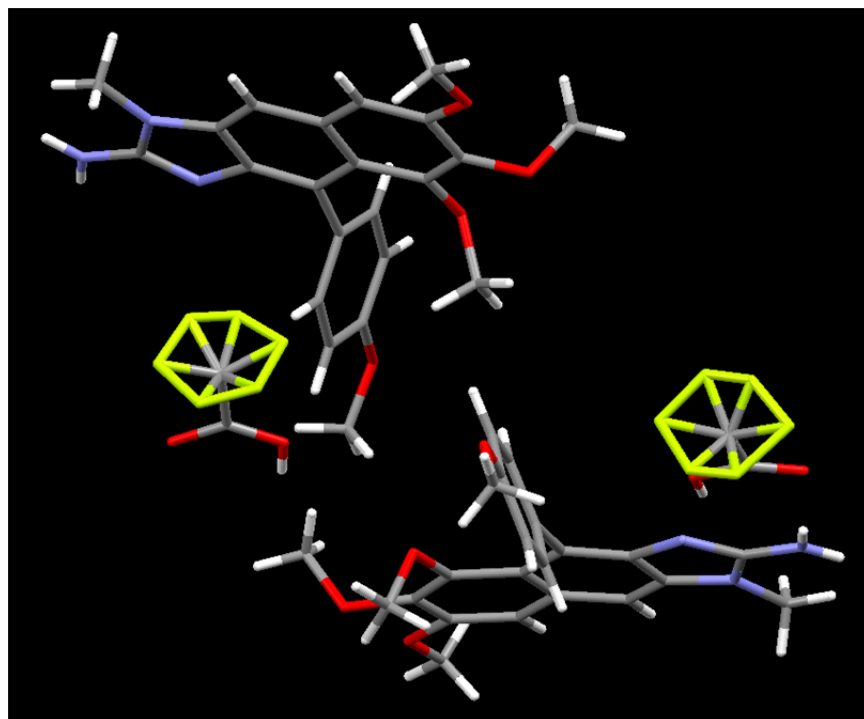
**Table S6:**  $^{13}\text{C}$  NMR spectroscopic data for kealiinine B (DMSO- $d_6$ )

Assignment	Kealiinine C (Syn)
C2	157.9
C17	157.6
C9	150.1
C11	149.5
C4	142.9
C10	140.0
C5	136.3
C14	133.8
C15/C19	131.5
C13	127.0
C7	120.7
C12	118.9
C16/C18	112.3
C8	103.5
C6	102.1
C23	61.0
C24	60.6
C22	55.9
C20	55.5
C21	28.9

a. Assignments based HSQC, HMBC and ROESY experiments (NOE's shown above)

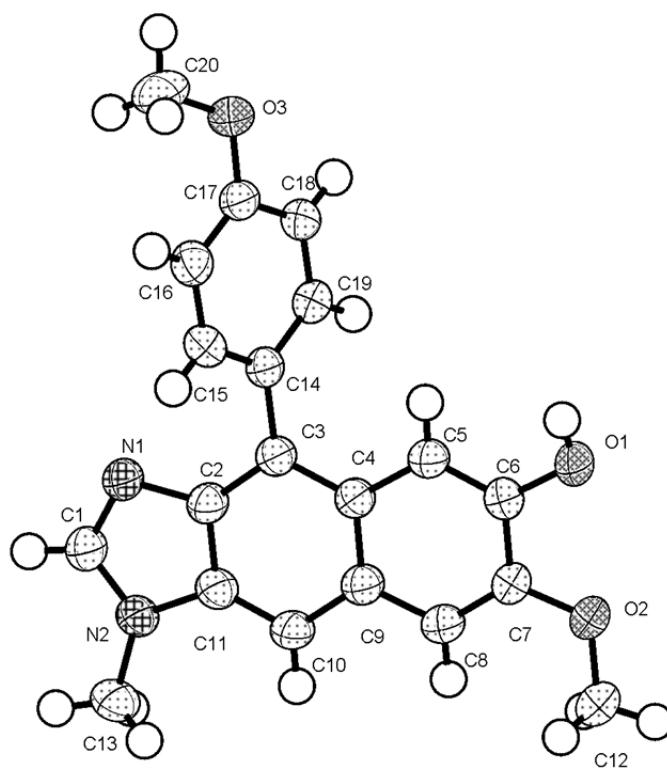
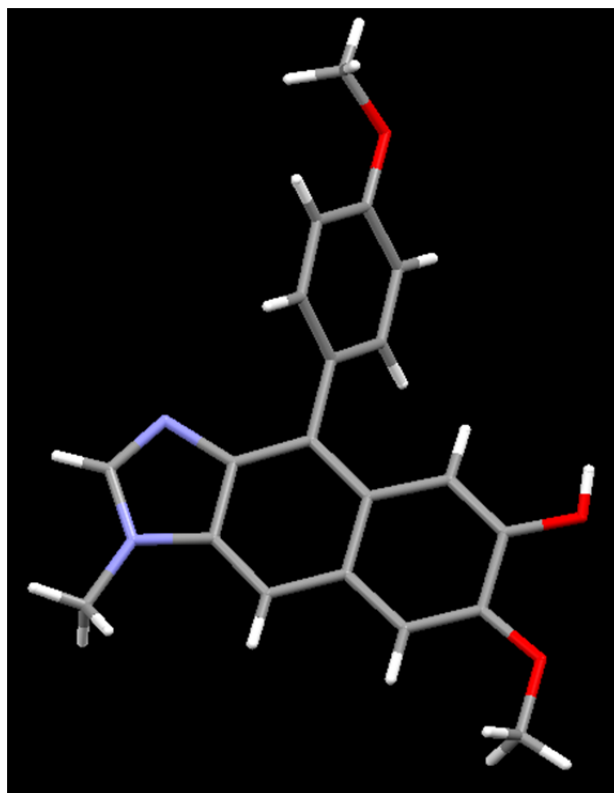


**Figure S1:** X-ray crystal structure of Friedel-Crafts product **18c** (ORTEP plot at 50% probability level)



**Figure S2:** X-ray crystal structure of synthetic kealiinine C (**8c**) (Upper plot shows two independent molecules in the unit cell with two disordered molecules of TFA)





**Figure S3:** X-ray crystal structure of deprotected Friedel-Crafts product **21**.

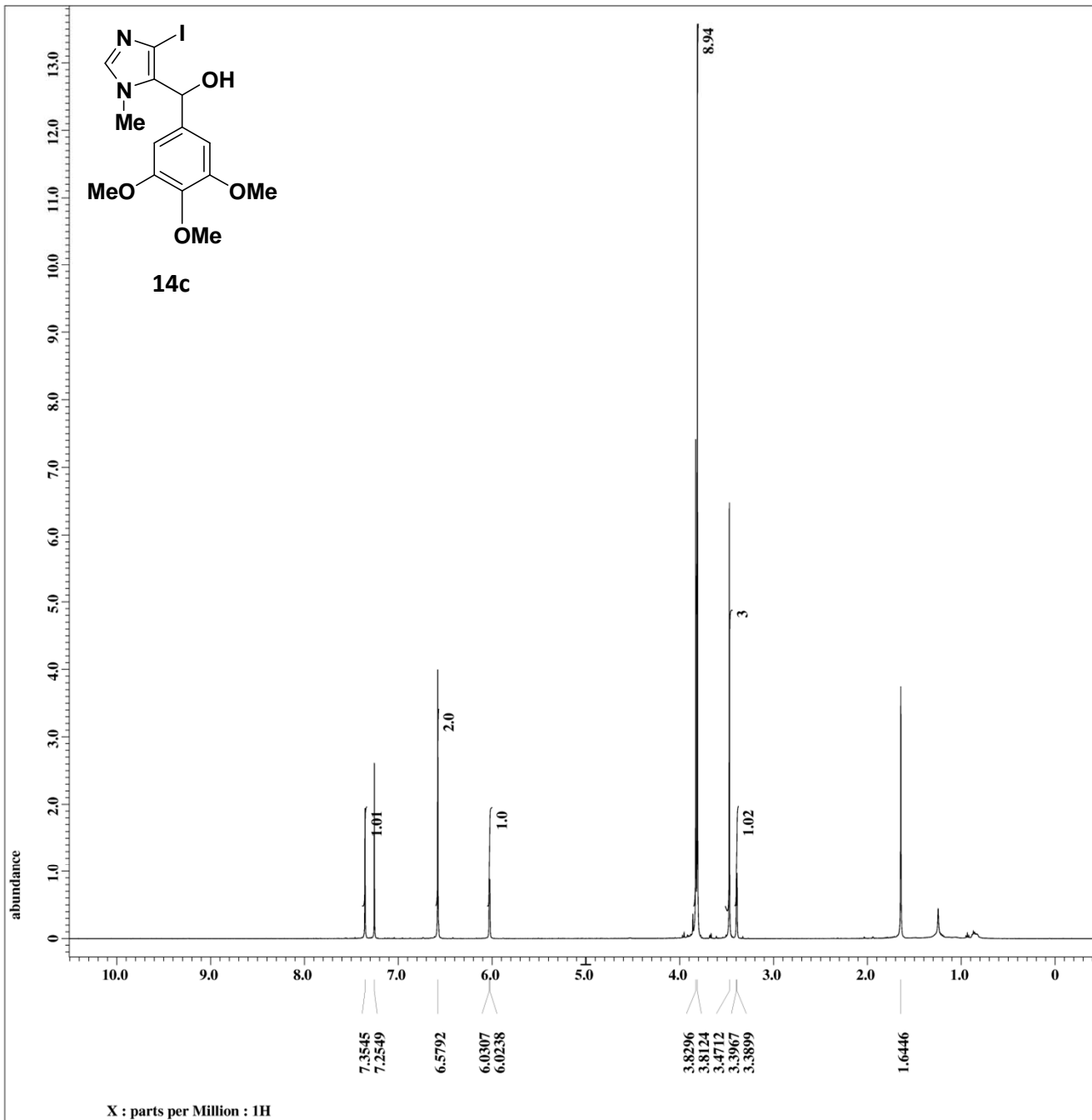


Filename = JKD\_I\_13\_MONO\_ALCOHOL  
Author = delta  
Experiment = single\_pulse.ex2  
Sample\_id = S#412504  
Solvent = CHLOROFORM-D  
Creation\_time = 18-MAY-2012 01:19:40  
Revision\_time = 19-MAY-2012 20:23:05  
Current\_time = 19-MAY-2012 20:23:50

Comment = single\_pulse  
Data\_format = 1D COMPLEX  
Dim\_size = 13107  
Dim\_title = 1H  
Dim\_units = [ppm]  
Dimensions = X  
Site = ECA 500  
Spectrometer = JNM-ECA500

Field\_strength = 11.7473579 [T] (500 [MH  
X\_acq\_duration = 1.74587904 [s]  
X\_domain = 1H  
X\_freq = 500.15991521 [MHz]  
X\_offset = 5.0 [ppm]  
X\_points = 16384  
X\_prescans = 0  
X\_resolution = 0.57277737 [Hz]  
X\_sweep = 9.38438438 [kHz]  
Irr\_domain = 1H  
Irr\_freq = 500.15991521 [MHz]  
Irr\_offset = 5.0 [ppm]  
Tri\_domain = 1H  
Tri\_freq = 500.15991521 [MHz]  
Tri\_offset = 5.0 [ppm]  
Clipped = FALSE  
Mod\_return = 1  
Scans = 24  
Total\_scans = 24

X\_90\_width = 12.54 [us]  
X\_acq\_time = 1.74587904 [s]  
X\_angle = 45 [deg]  
X\_atn = 4 [dB]  
X\_pulse = 6.27 [us]  
Irr\_mode = Off  
Tri\_mode = Off  
Dante\_presat = FALSE  
Initial\_wait = 1 [s]  
Recvr\_gain = 46  
Relaxation\_delay = 5 [s]  
Repetition\_time = 6.74587904 [s]  
Temp\_get = 22 [dC]



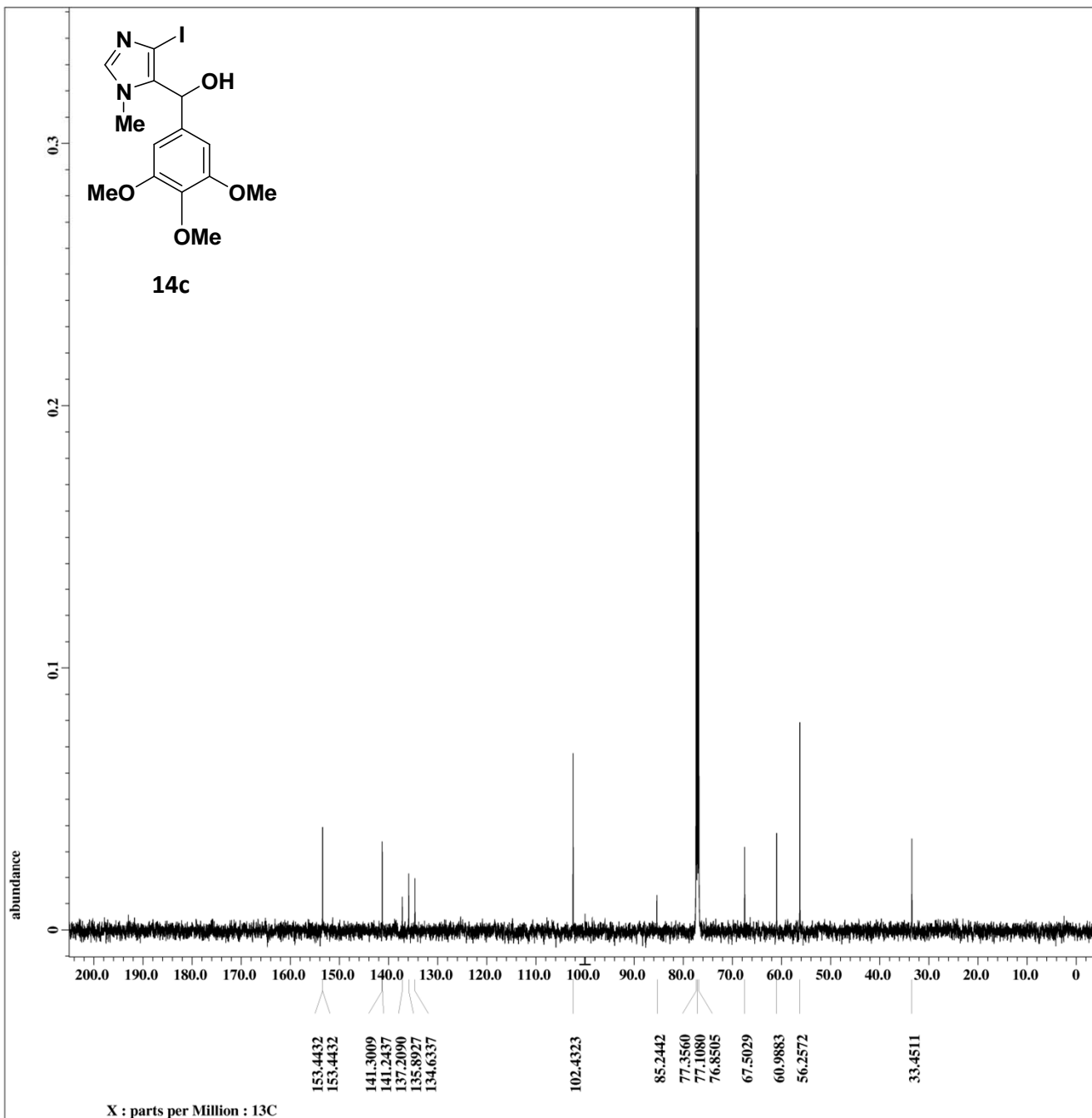


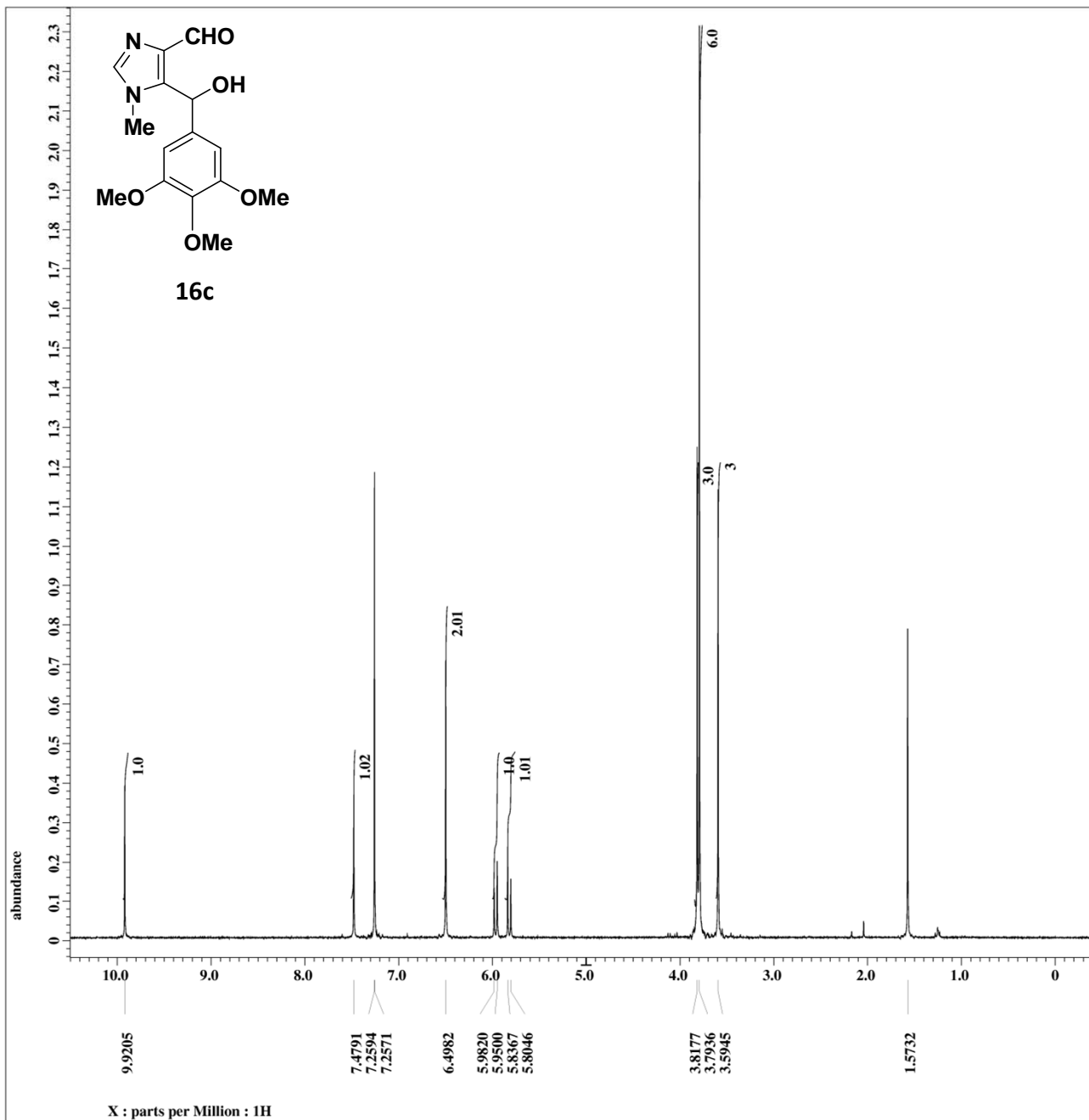
Filename = JKD\_I\_13\_MONO\_ALCOHOL  
Author = delta  
Experiment = single\_pulse\_dec  
Sample\_id = S#414709  
Solvent = CHLOROFORM-D  
Creation\_time = 18-MAY-2012 01:41:44  
Revision\_time = 19-MAY-2012 20:50:25  
Current\_time = 19-MAY-2012 20:52:14

Comment = single pulse decouple  
Data\_format = 1D COMPLEX  
Dim\_size = 26214  
Dim\_title = 13C  
Dim\_units = [ppm]  
Dimensions = X  
Site = ECA 500  
Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
X\_acq\_duration = 0.83361792[s]  
X\_domain = 13C  
X\_freq = 125.76529768[MHz]  
X\_offset = 100[ppm]  
X\_points = 32768  
X\_prescans = 4  
X\_resolution = 1.19959034[Hz]  
X\_sweep = 39.3081761[kHz]  
Irr\_domain = 1H  
Irr\_freq = 500.15991521[MHz]  
Irr\_offset = 5.0[ppm]  
Clipped = FALSE  
Mod\_return = 10  
Scans = 450  
Total\_scans = 450

X\_90\_width = 10.73[us]  
X\_acq\_time = 0.83361792[s]  
X\_angle = 30[deg]  
X\_atn = 9[dB]  
X\_pulse = 3.57666667[us]  
Irr\_atn\_dec = 20[dB]  
Irr\_atn\_noe = 20[dB]  
Irr\_noise = WALTZ  
Decoupling = TRUE  
Initial\_wait = 1[s]  
Noe = TRUE  
Noe\_time = 2[s]  
Recvr\_gain = 50  
Relaxation\_delay = 2[s]  
Repetition\_time = 2.83361792[s]  
Temp\_get = 22.6[dC]





```

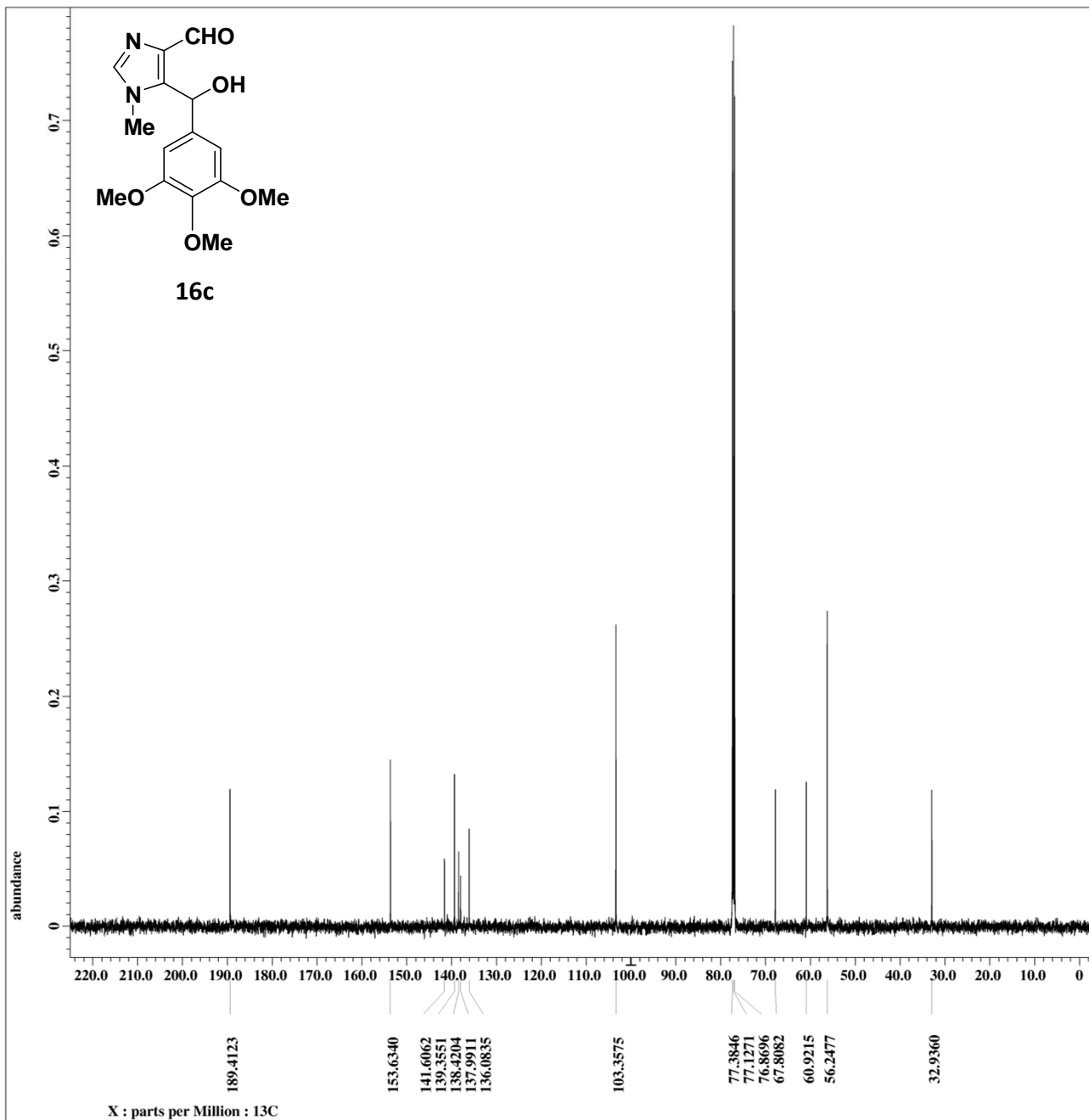
Filename      = jkd_I_15_PURE-6.jdf
Author       = delta
Experiment    = single_pulse.ex2
Sample_id     = S#664297
Solvent      = CHLOROFORM-D
Creation_time = 30-MAR-2010 17:11:50
Revision_time = 20-MAY-2012 18:44:20
Current_time  = 20-MAY-2012 18:44:54

Comment      = single_pulse
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECX 300
Spectrometer = DELTA2_NMR

Field_strength = 7.0586013[T] (300[MHz])
X_acq_duration = 2.90717696[s]
X_domain       = 1H
X_freq         = 300.52965592[MHz]
X_offset       = 5[ppm]
X_points       = 16384
X_prescans     = 0
X_resolution   = 0.34397631[Hz]
X_sweep        = 5.63570784[kHz]
Irr_domain     = 1H
Irr_freq       = 300.52965592[MHz]
Irr_offset     = 5[ppm]
Tri_domain     = 1H
Tri_freq       = 300.52965592[MHz]
Tri_offset     = 5[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 24
Total_scans    = 24

X_90_width    = 13.01[us]
X_acq_time     = 2.90717696[s]
X_angle        = 45[deg]
X_atn          = 4[dB]
X_pulse        = 6.505[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 46
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get       = 21[dC]

```



```

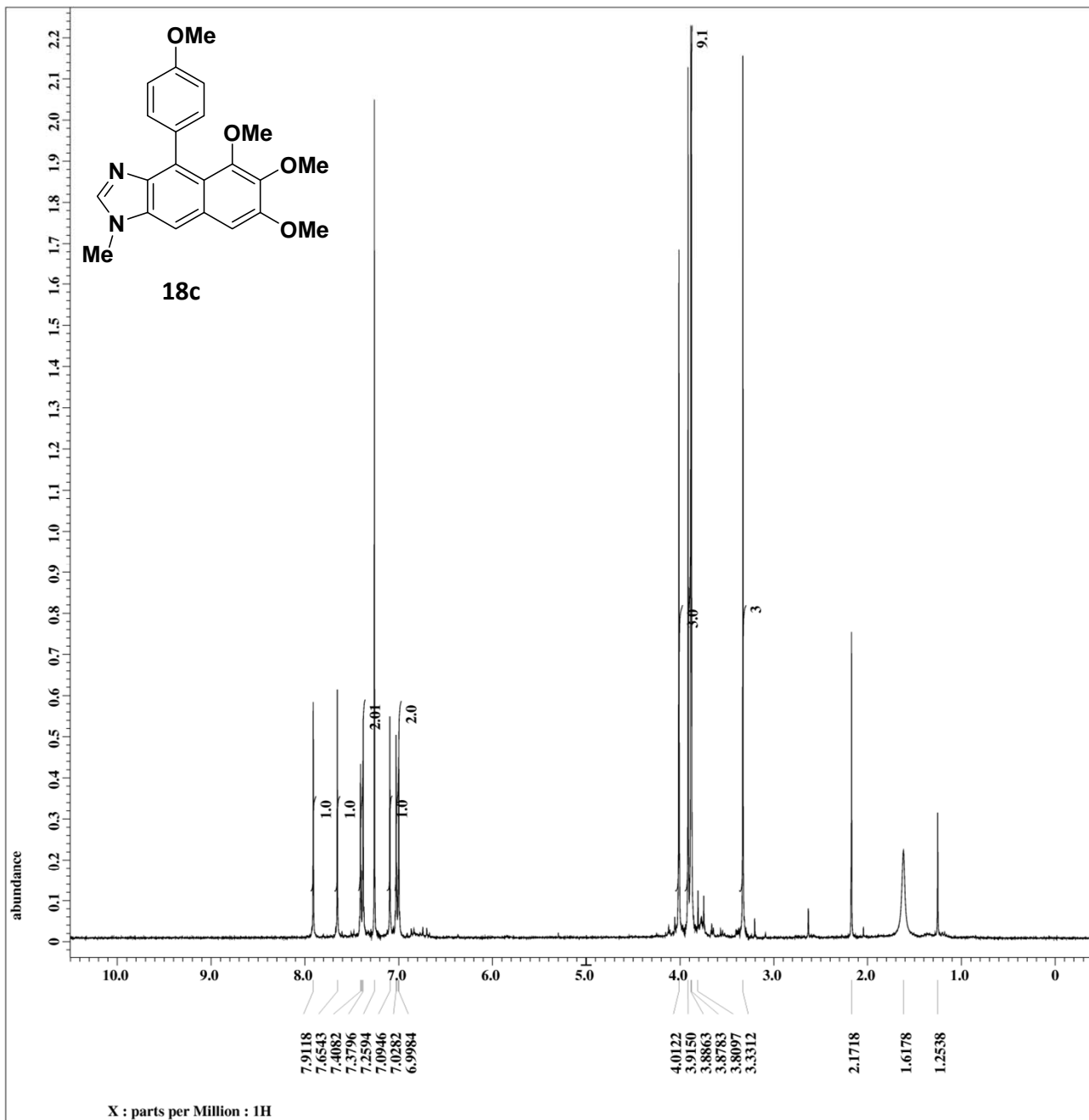
Filename      = JKD_I_ALDEHYDE-3.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#579867
Solvent      = CHLOROFORM-D
Creation_time = 21-MAY-2012 06:03:48
Revision_time = 20-MAY-2012 18:48:16
Current_time = 20-MAY-2012 18:48:49

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 170
Total_scans    = 170

X_90_width    = 10.73 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 9 [dB]
X_pulse        = 3.57666667 [us]
Irr_atn_dec    = 20 [dB]
Irr_atn_noe    = 20 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get       = 22.8 [dC]

```

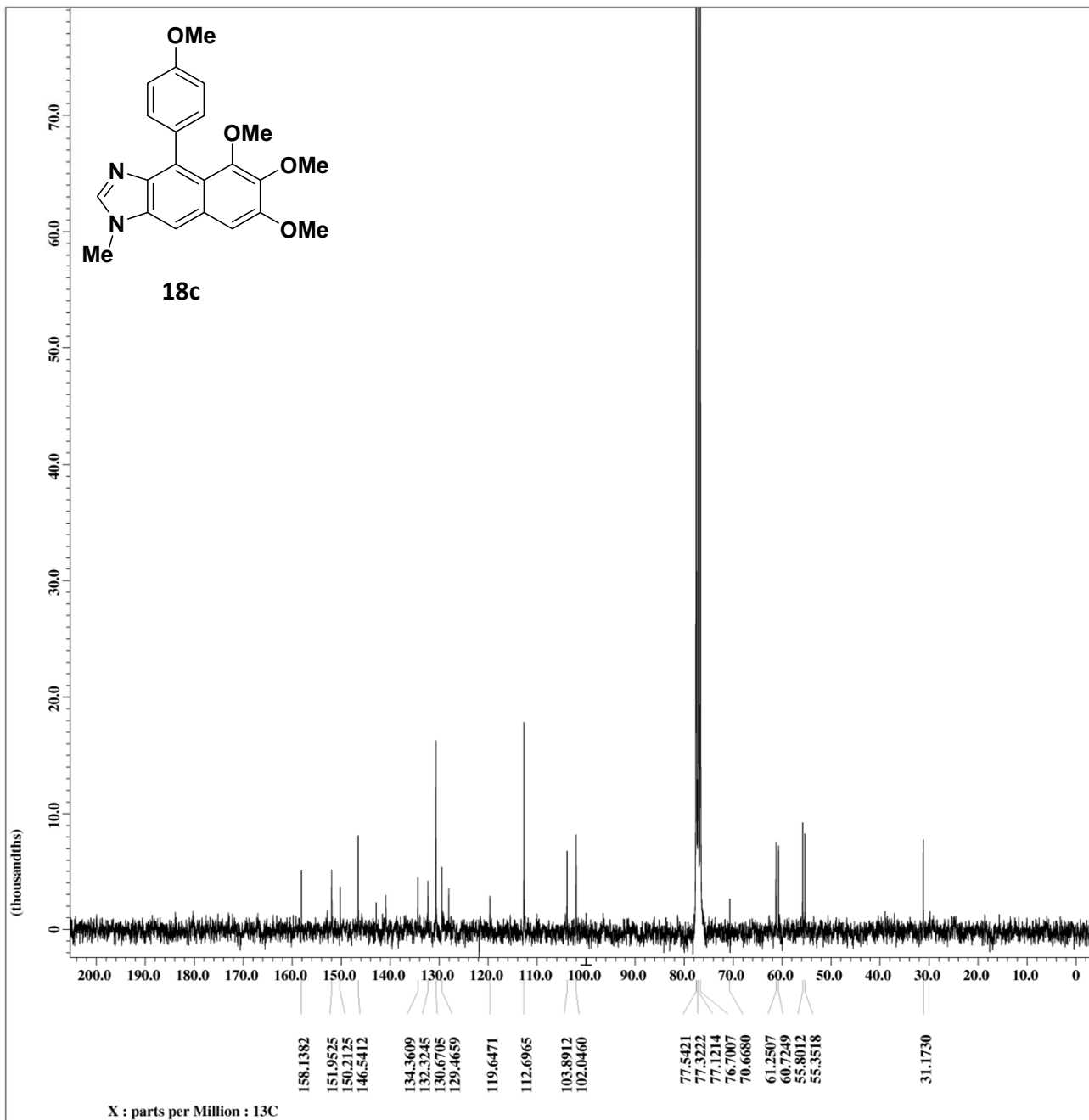


Filename = jkd\_I\_16\_CYCLIC-4.jdf  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = j  
 Solvent = CHLOROFORM-D  
 Creation\_time = 2-APR-2010 16:22:15  
 Revision\_time = 19-MAY-2012 20:34:07  
 Current\_time = 19-MAY-2012 20:34:44

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECX 300  
 Spectrometer = DELTA2\_NMR

Field\_strength = 7.0586013[T] (300[MHz])  
 X\_acq\_duration = 2.90717696[s]  
 X\_domain = 1H  
 X\_freq = 300.52965592[MHz]  
 X\_offset = 5[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.34397631[Hz]  
 X\_sweep = 5.63570784[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 300.52965592[MHz]  
 Irr\_offset = 5[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 300.52965592[MHz]  
 Tri\_offset = 5[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 13.01[us]  
 X\_acq\_time = 2.90717696[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.505[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 48  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 7.90717696[s]  
 Temp\_get = 21.1[dC]

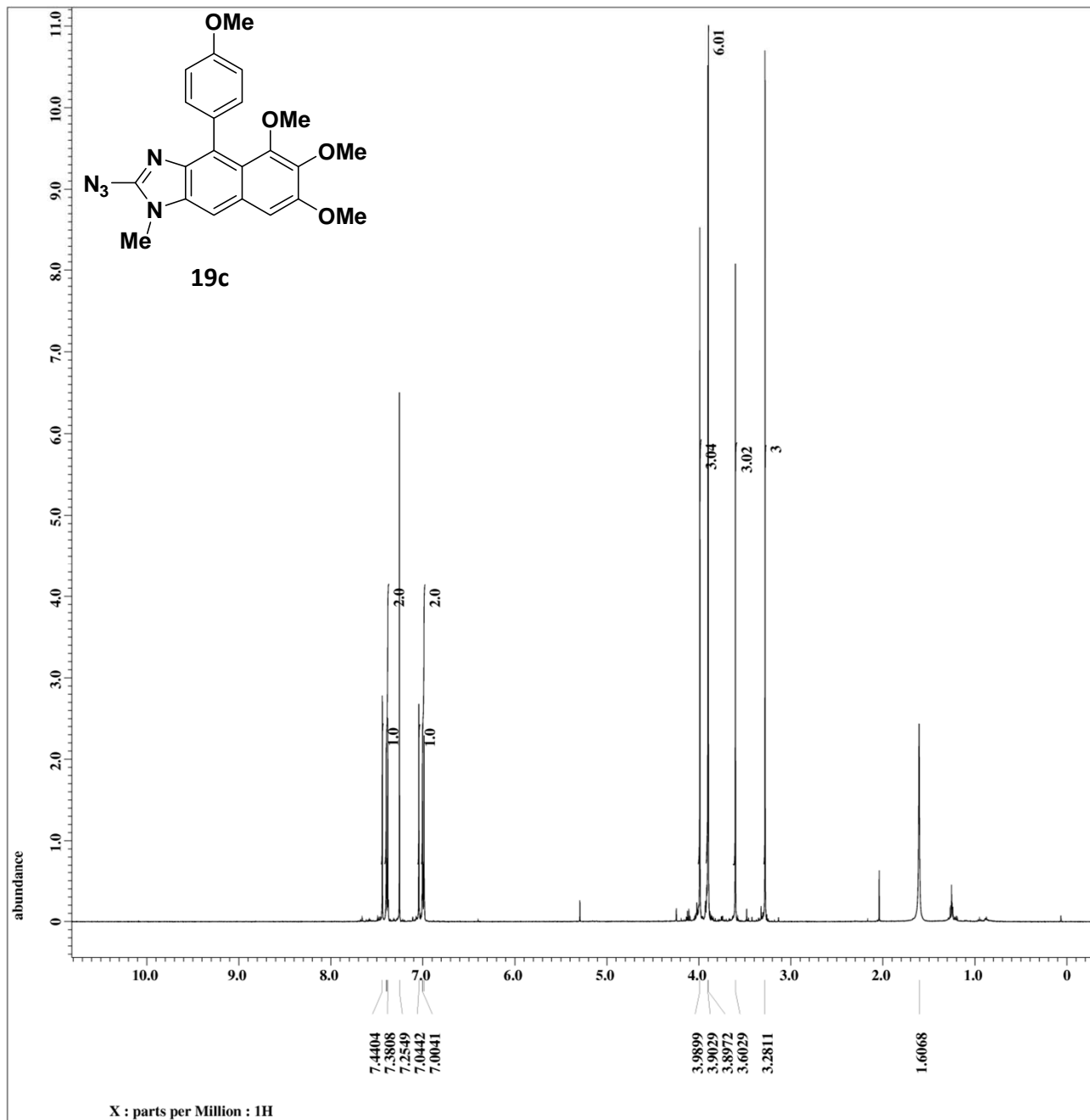


Filename = JKD\_I\_16\_CYCLIC-4.jdf  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#534289  
 Solvent = CHLOROFORM-D  
 Creation\_time = 19-MAY-2010 17:22:37  
 Revision\_time = 19-MAY-2012 20:53:23  
 Current\_time = 19-MAY-2012 20:54:12

Comment = single pulse decouple  
 Data\_format = 1D COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECX 300  
 Spectrometer = DELTA2\_NMR

Field\_strength = 7.0586013[T] (300[MHz])  
 X\_acq\_duration = 1.38412032[s]  
 X\_domain = 13C  
 X\_freq = 75.56823426[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 0.72248054[Hz]  
 X\_sweep = 23.67424242[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 300.52965592[MHz]  
 Irr\_offset = 5[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 2980  
 Total\_scans = 2980

X\_90\_width = 9.75[us]  
 X\_acq\_time = 1.38412032[s]  
 X\_angle = 30[deg]  
 X\_atn = 8[dB]  
 X\_pulse = 3.25[us]  
 Irr\_atn\_dec = 25[dB]  
 Irr\_atn\_noe = 25[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 3.38412032[s]  
 Temp\_get = 21.3[degC]



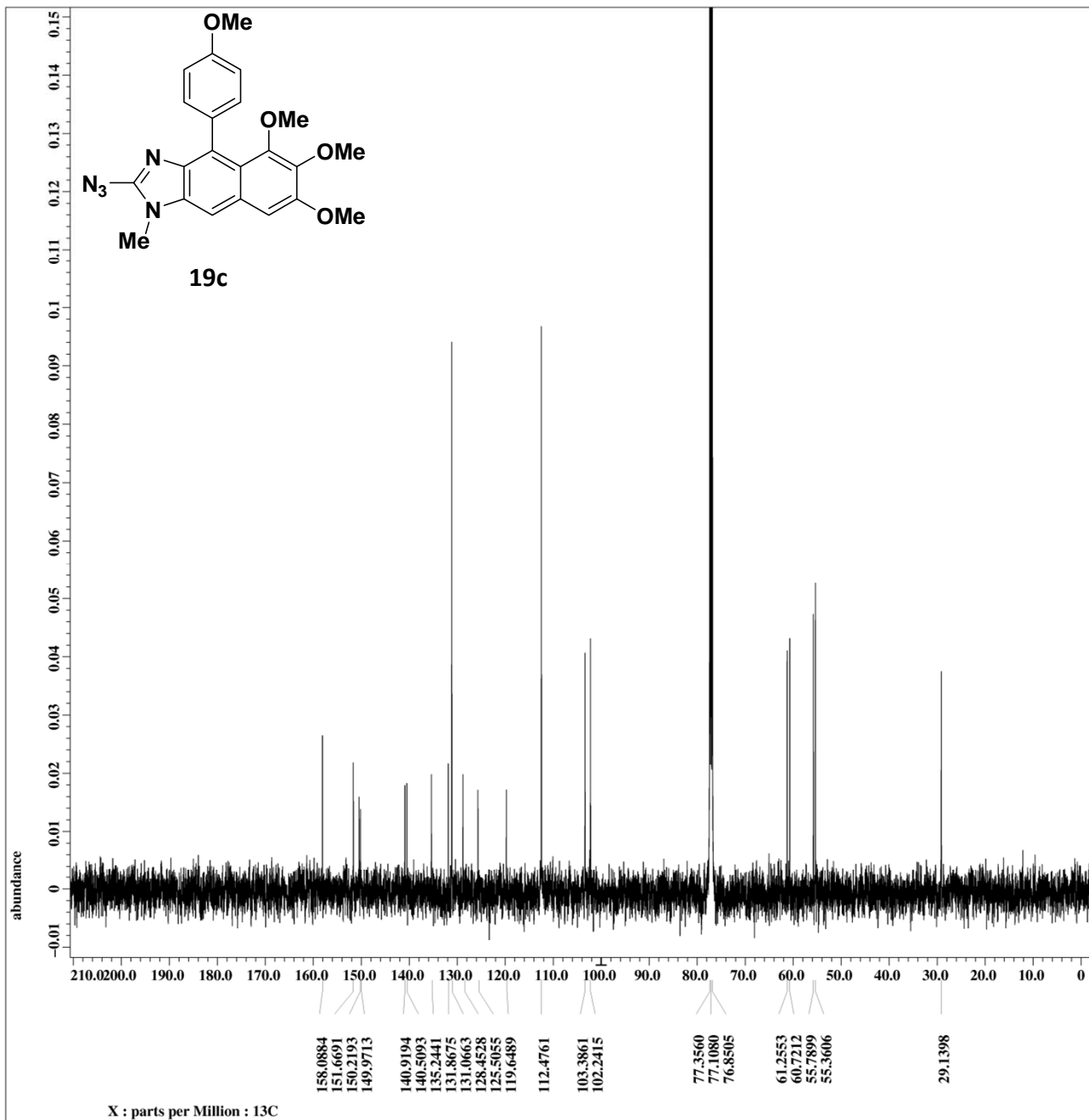
Filename = JKD\_I\_KC\_AZIDE\_PURE-4  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#503070  
 Solvent = CHLOROFORM-D  
 Creation\_time = 10-JUL-2012 03:52:01  
 Revision\_time = 8-AUG-2012 08:37:20  
 Current\_time = 8-AUG-2012 08:43:22

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 15  
 Total\_scans = 15

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 48  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]





```

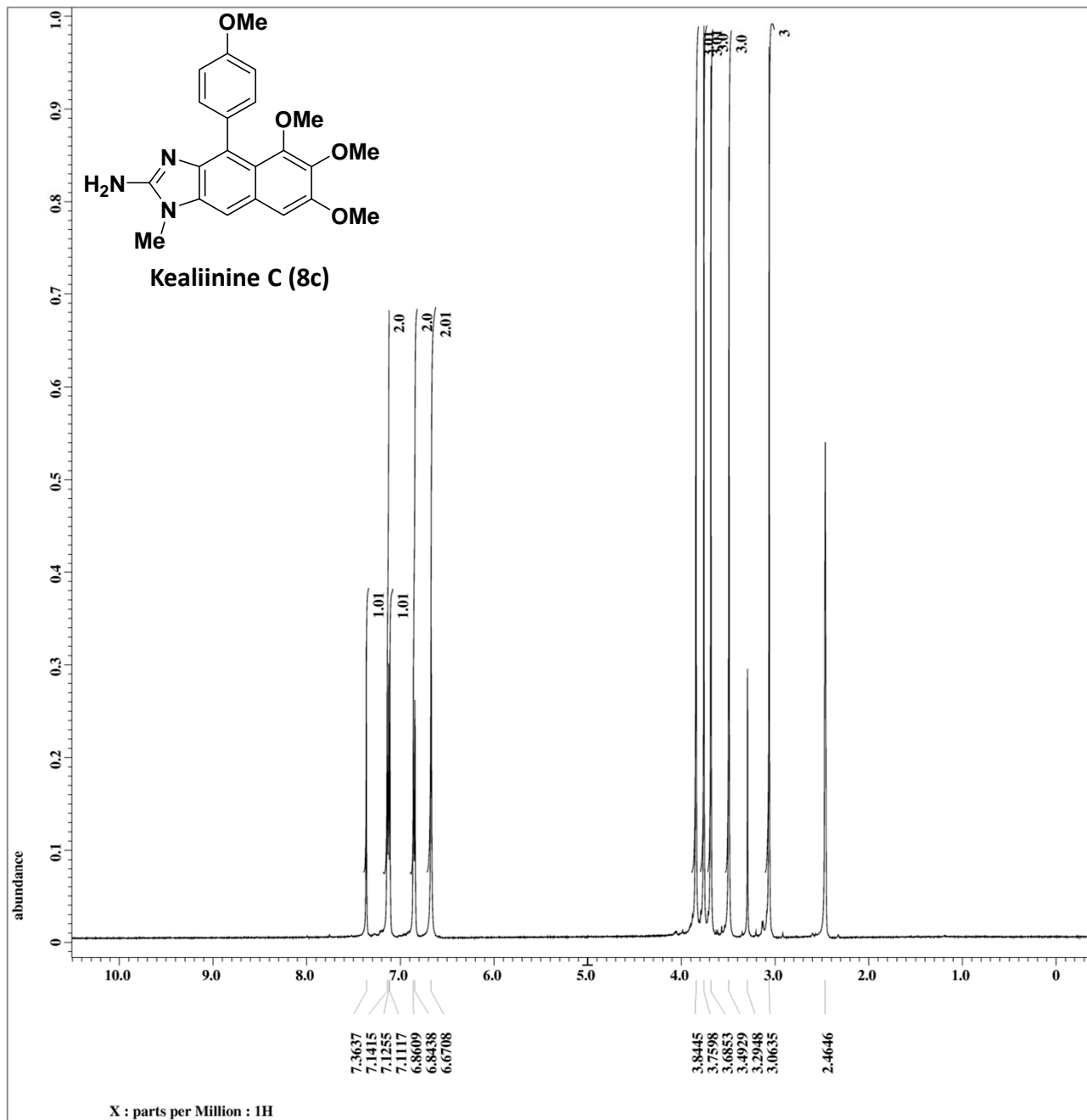
Filename      = JKD_I_KC_AZIDE_PURE-6
Author        = delta
Experiment    = single_pulse_dec
Sample_id     = S#506745
Solvent       = CHLOROFORM-D
Creation_time = 10-JUL-2012 04:18:57
Revision_time = 8-AUG-2012 08:52:15
Current_time  = 8-AUG-2012 08:52:35

Comment       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECA 500
Spectrometer  = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 480
Total_scans    = 480

X_90_width    = 10.73 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 9 [dB]
X_pulse        = 3.57666667 [us]
Irr_atn_dec    = 20 [dB]
Irr_atn_noe    = 20 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get       = 22.6 [dC]

```



```

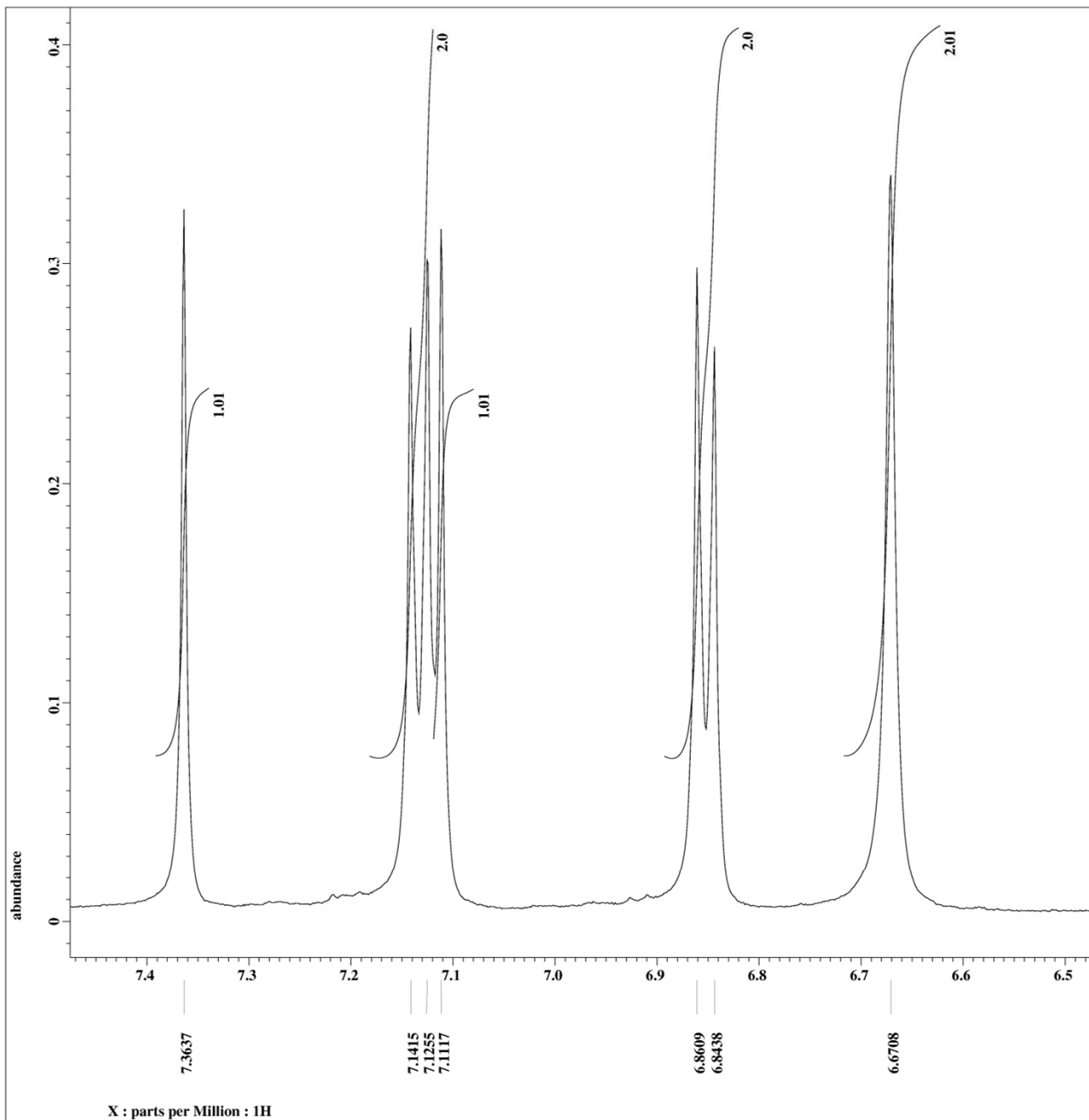
Filename          = JKD_I_KC-4. jdf
Author           = delta
Experiment        = single_pulse.ex2
Sample_id         = S#551055
Solvent           = DMSO-D6
Creation_time     = 28-APR-2010 05:23:53
Revision_time    = 19-MAY-2012 20:43:20
Current_time      = 19-MAY-2012 20:43:51

Comment          = single_pulse
Data_format       = 1D COMPLEX
Dim_size         = 13107
Dim_title        = 1H
Dim_units        = [ppm]
Dimensions       = X
Site             = ECA 500
Spectrometer     = JNM-ECA500

Field_strength   = 11.7473579[T] (500[MH
X_acq_duration   = 1.74587904[s]
X_domain         = 1H
X_freq           = 500.15991521[MHz]
X_offset         = 5.0[ppm]
X_points         = 16384
X_prescans       = 0
X_resolution     = 0.57277737[Hz]
X_sweep         = 9.38438438[kHz]
Irr_domain       = 1H
Irr_freq         = 500.15991521[MHz]
Irr_offset       = 5.0[ppm]
Tri_domain       = 1H
Tri_freq         = 500.15991521[MHz]
Tri_offset       = 5.0[ppm]
Clipped         = FALSE
Mod_return       = 1
Scans            = 16
Total_scans     = 16

X_90_width      = 14.63[us]
X_acq_time       = 1.74587904[s]
X_angle         = 45[deg]
X_atn           = 2[dB]
X_pulse         = 7.315[us]
Irr_mode        = Off
Tri_mode        = Off
Dante_presat    = FALSE
Initial_wait    = 1[s]
Recvr_gain      = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get        = 22.2[dC]

```

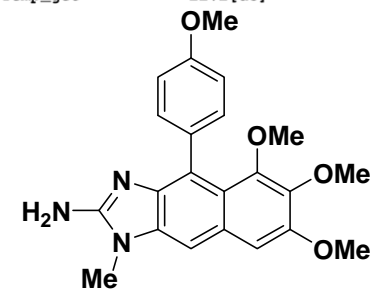


Filename = JKD\_I\_KC-4.jdf  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#551055  
 Solvent = DMSO-D6  
 Creation\_time = 28-APR-2010 05:23:53  
 Revision\_time = 19-MAY-2012 20:43:20  
 Current\_time = 19-MAY-2012 20:44:33

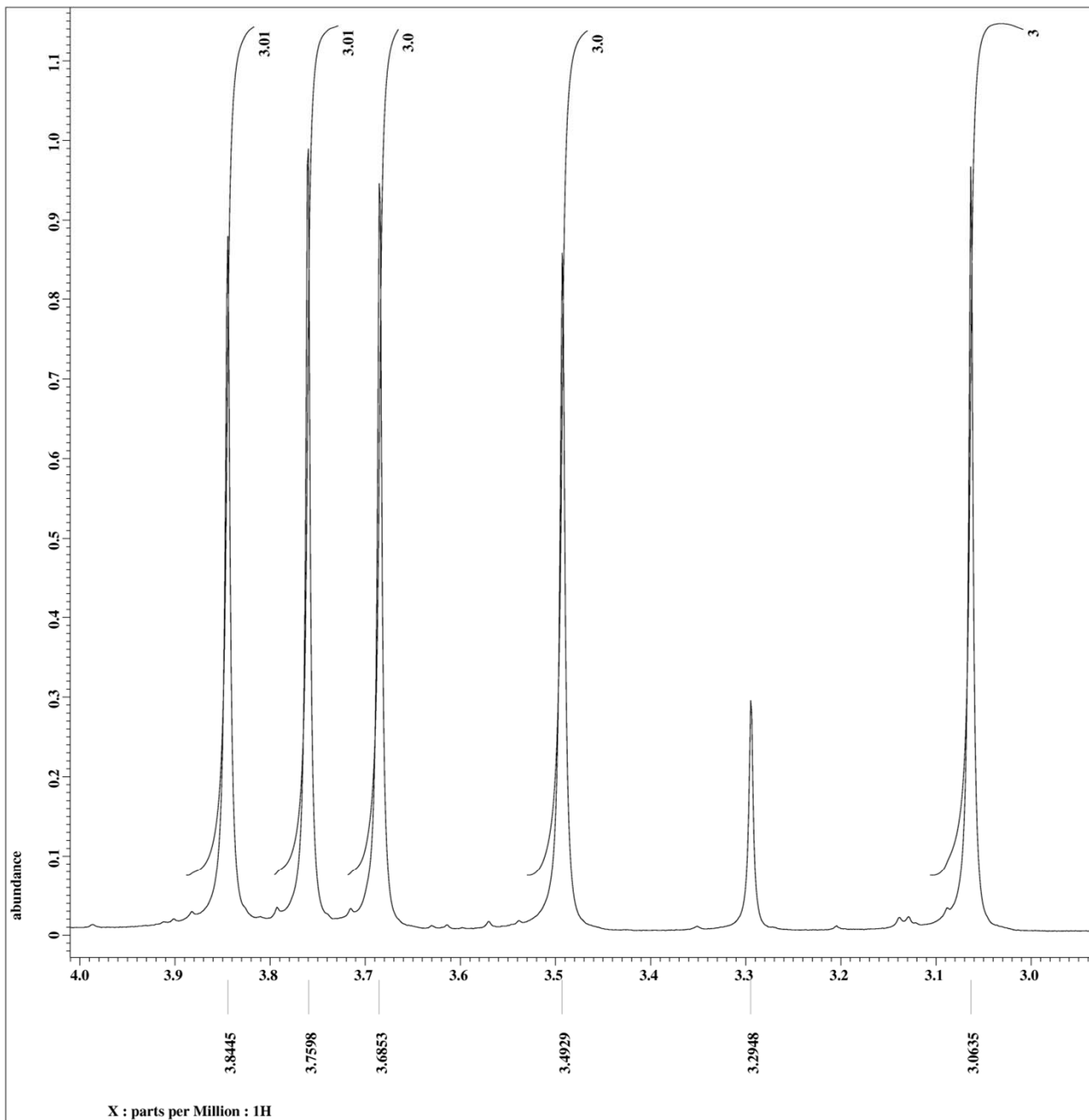
Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 16  
 Total\_scans = 16

X\_90\_width = 14.63[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 2[dB]  
 X\_pulse = 7.315[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 36  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.2[dC]



Kealiinine C (8c)



```

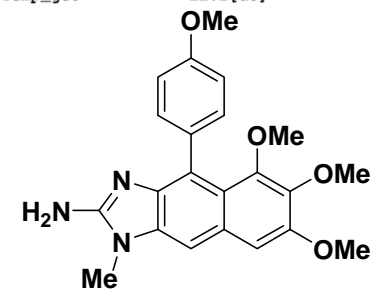
Filename      = JKD_I_KC-4.jdf
Author       = delta
Experiment   = single_pulse.ex2
Sample_id    = S#551055
Solvent      = DMSO-D6
Creation_time = 28-APR-2010 05:23:53
Revision_time = 19-MAY-2012 20:43:20
Current_time  = 19-MAY-2012 20:45:27

Comment      = single_pulse
Data_format  = 1D COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

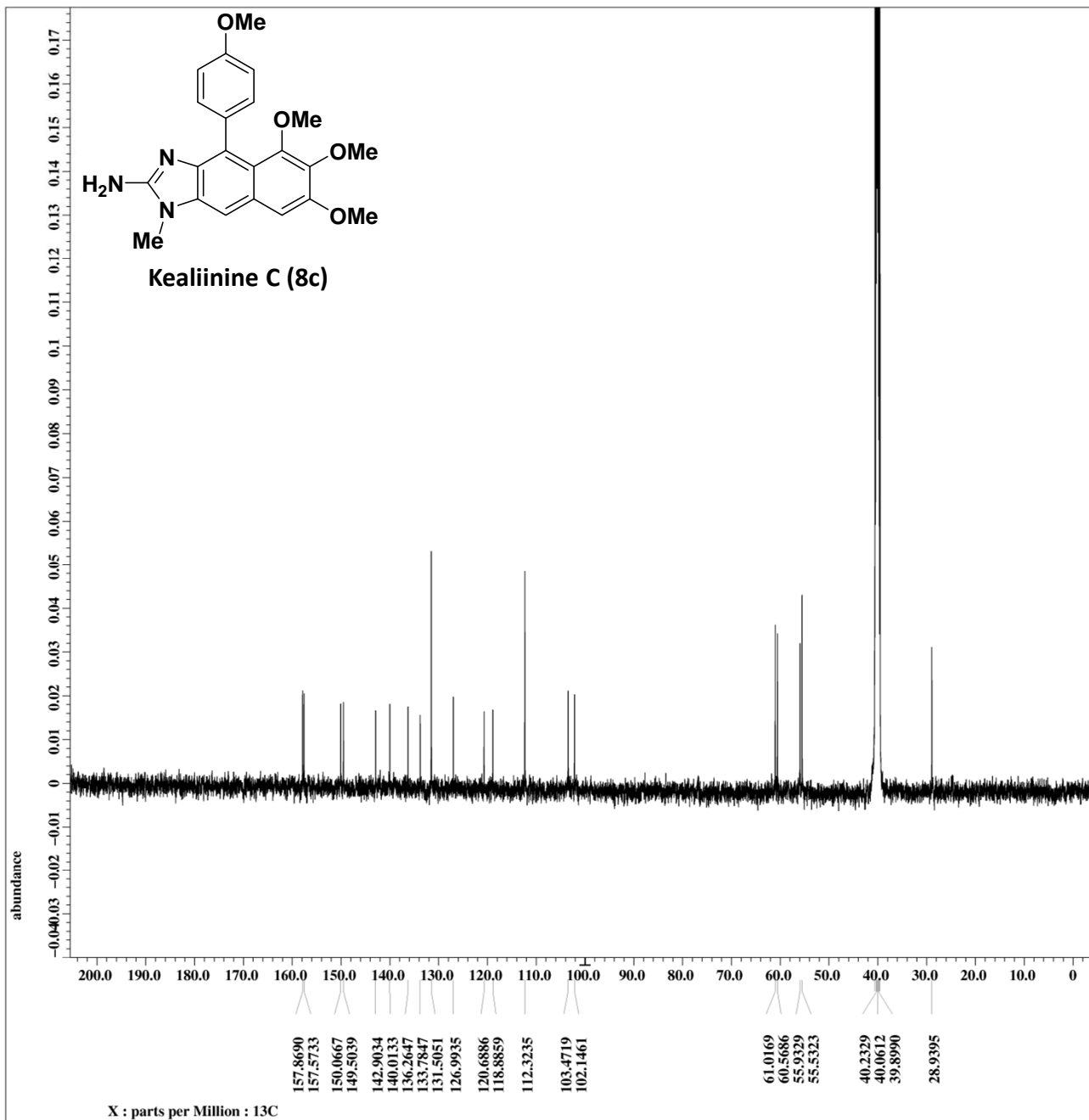
Field_strength = 11.7473579[T] (500[MH]
X_acq_duration = 1.74587904[s]
X_domain       = 1H
X_freq         = 500.15991521[MHz]
X_offset       = 5.0[ppm]
X_points       = 16384
X_prescans     = 0
X_resolution   = 0.57277737[Hz]
X_sweep        = 9.38438438[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521[MHz]
Tri_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 16
Total_scans    = 16

X_90_width    = 14.63[us]
X_acq_time    = 1.74587904[s]
X_angle       = 45[deg]
X_atn         = 2[dB]
X_pulse       = 7.315[us]
Irr_mode      = Off
Tri_mode      = Off
Dante_presat  = FALSE
Initial_wait  = 1[s]
Recvr_gain    = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get      = 22.2[dC]

```



Kealiinine C (8c)



```

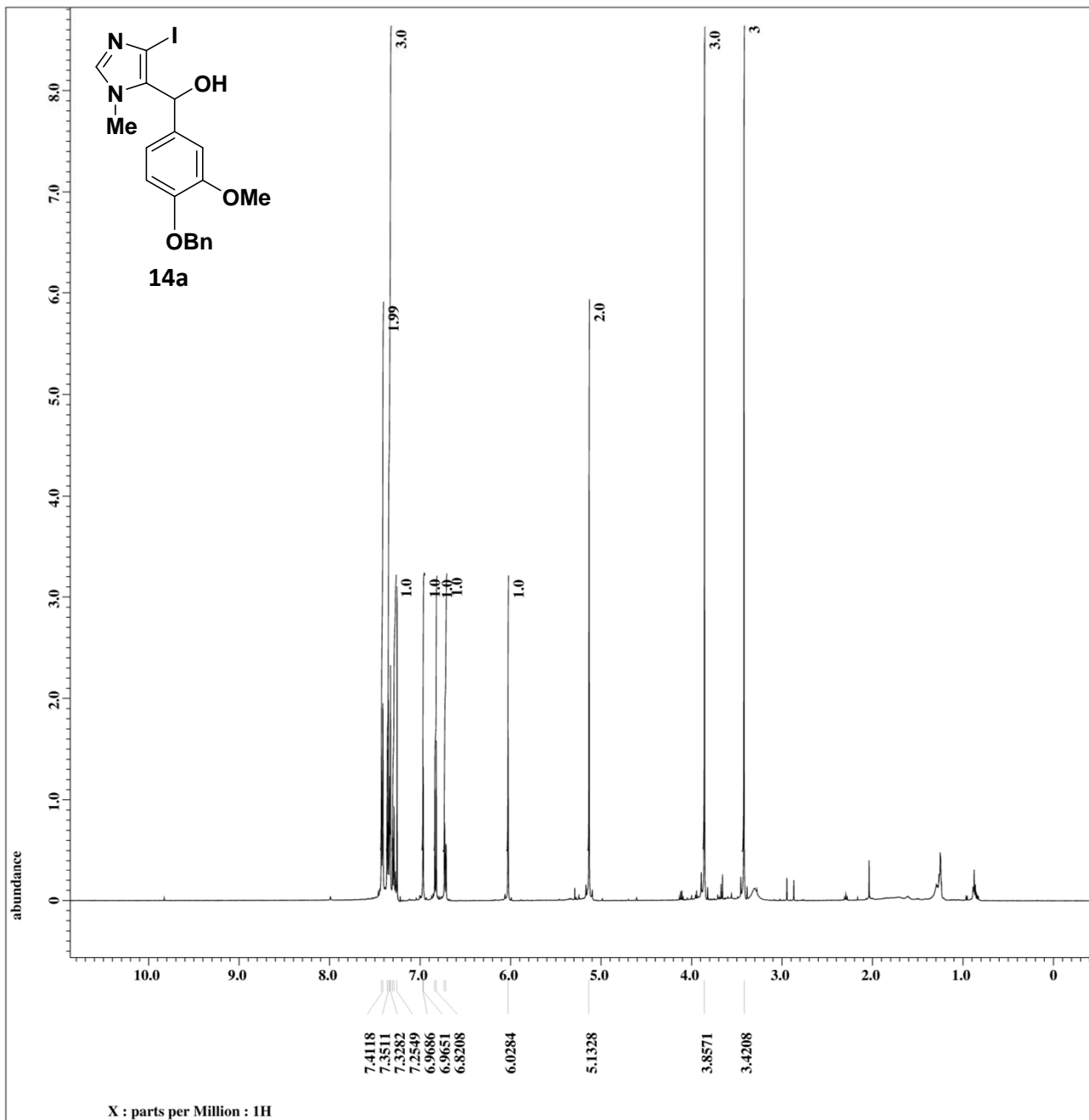
Filename      = JKD_I_KC-3.jdf
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#553126
Solvent      = DMSO-D6
Creation_time = 28-APR-2010 07:12:28
Revision_time = 19-MAY-2012 20:55:19
Current_time  = 19-MAY-2012 20:56:04

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768 [MHz]
X_offset      = 100[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution   = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521 [MHz]
Irr_offset    = 5.0[ppm]
Clipped       = FALSE
Mod_return    = 10
Scans         = 940
Total_scans   = 940

X_90_width   = 13.3[us]
X_acq_time   = 0.83361792[s]
X_angle      = 30[deg]
X_atn        = 9[dB]
X_pulse      = 4.43333333[us]
Irr_atn_dec  = 16.5[dB]
Irr_atn_noe  = 16.5[dB]
Irr_noise    = WALTZ
Decoupling   = TRUE
Initial_wait = 1[s]
Noe          = TRUE
Noe_time     = 6[s]
Recvr_gain   = 50
Relaxation_delay = 6[s]
Repetition_time = 6.83361792[s]
Temp_get     = 23.7[dC]

```

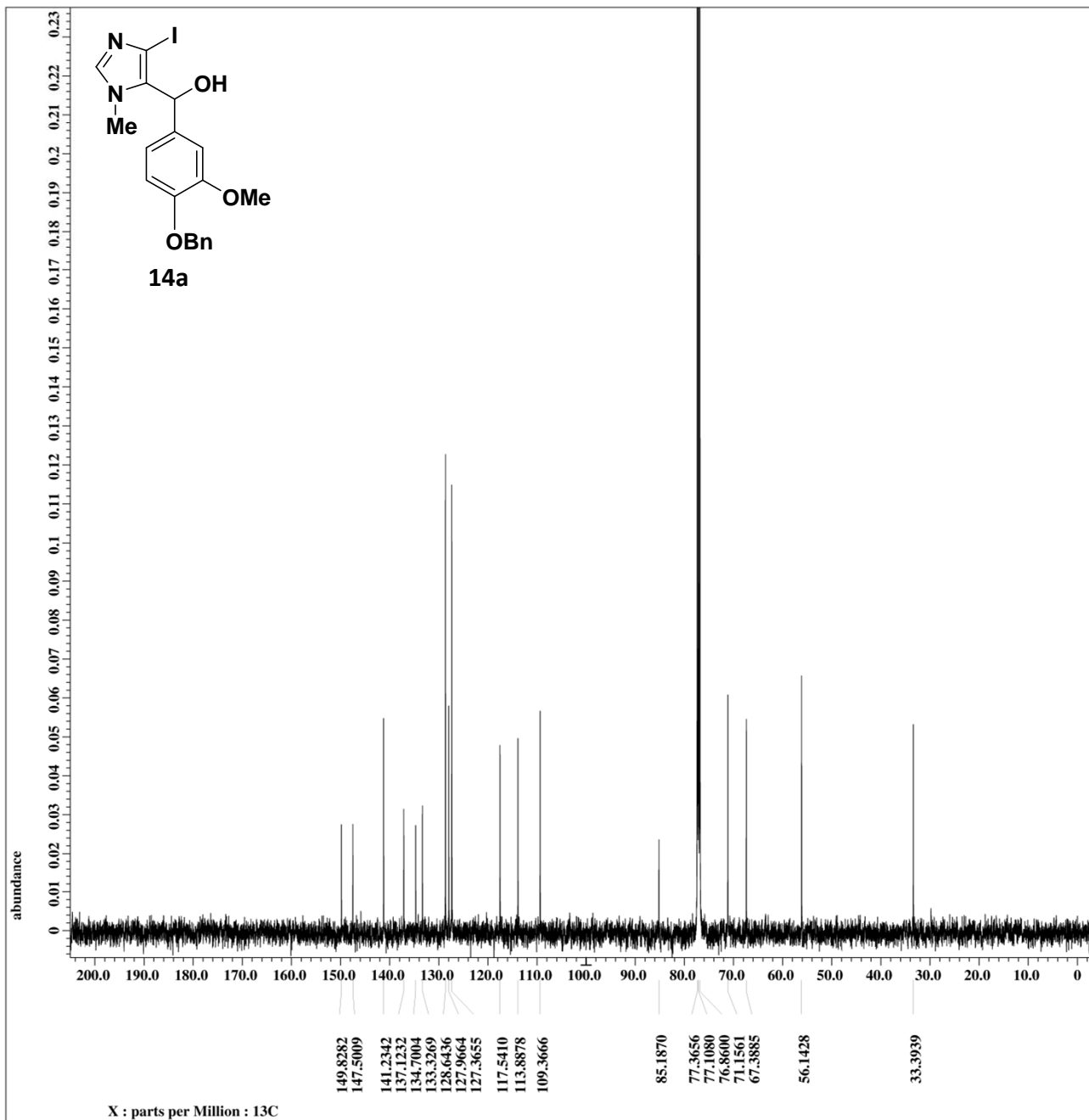


Filename = JKD\_I\_102\_MONO\_ALCOHO  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#539182  
 Solvent = CHLOROFORM-D  
 Creation\_time = 29-NOV-2011 05:43:21  
 Revision\_time = 25-JUL-2012 00:25:28  
 Current\_time = 25-JUL-2012 00:27:54

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 46  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.9[dc]



```

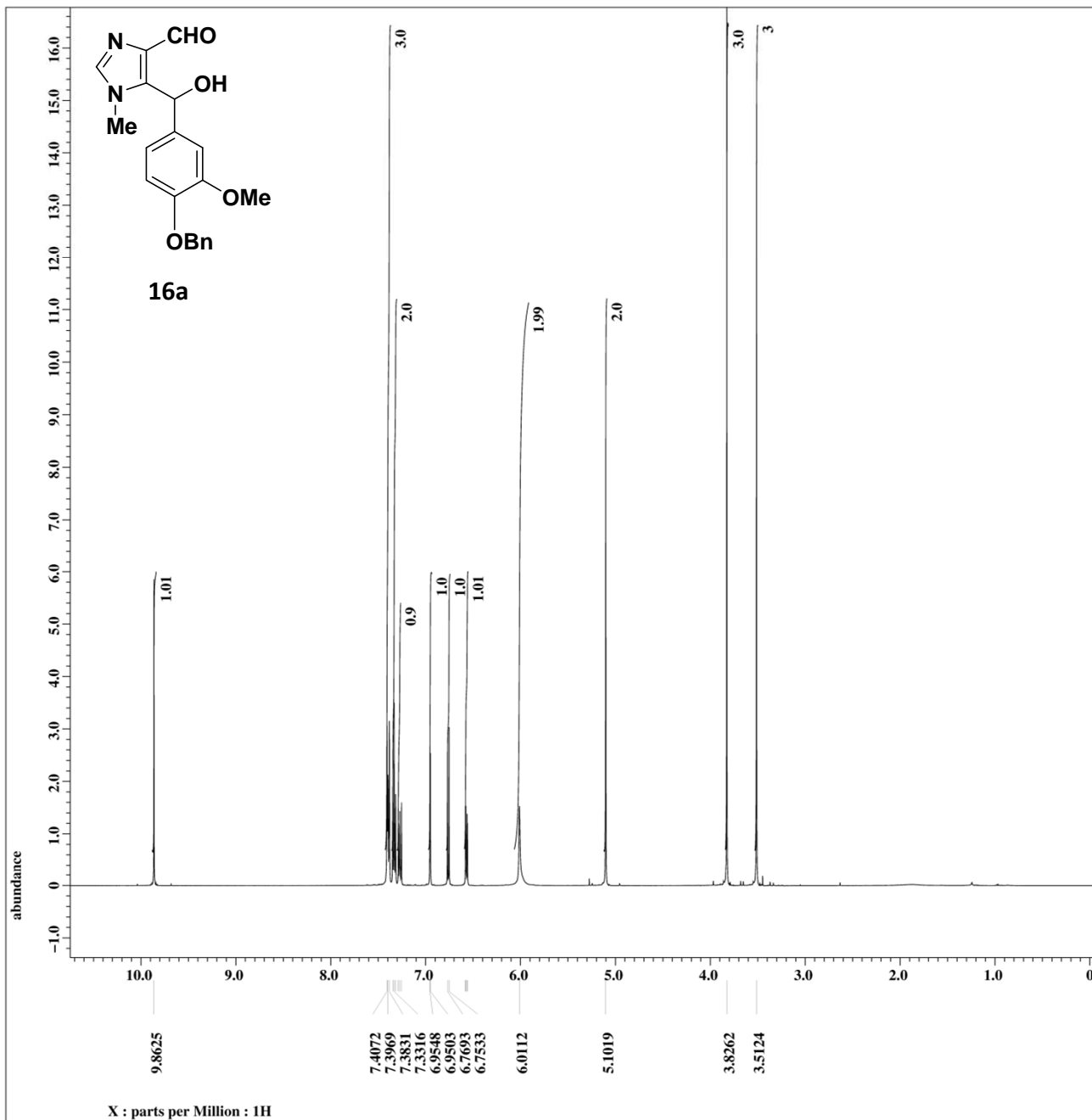
Filename      = JKD_I_102_MONO_ALCOHO
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#541801
Solvent      = CHLOROFORM-D
Creation_time = 29-NOV-2011 06:12:57
Revision_time = 25-JUL-2012 00:32:44
Current_time  = 25-JUL-2012 00:33:31

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 600
Total_scans    = 600

X_90_width    = 10.73 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 9 [dB]
X_pulse        = 3.57666667 [us]
Irr_atn_dec    = 20 [dB]
Irr_atn_noe    = 20 [dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get       = 22.2 [dC]

```



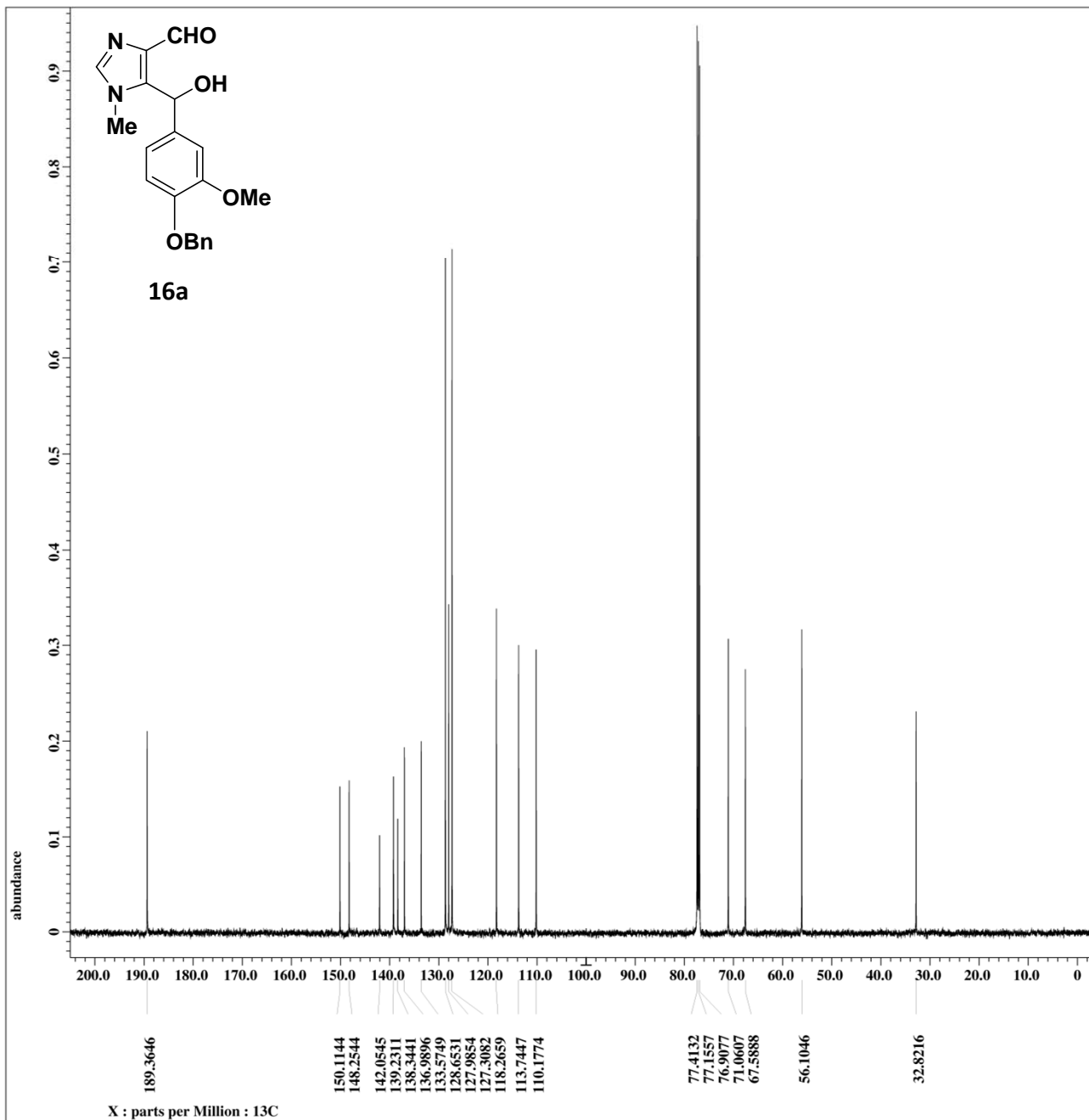
Filename = JKD\_I\_KA\_ALDEHYDE-3.j  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#483586  
 Solvent = CHLOROFORM-D  
 Creation\_time = 12-JAN-2012 04:11:21  
 Revision\_time = 25-JUL-2012 00:38:21  
 Current\_time = 25-JUL-2012 00:42:05

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 8  
 Total\_scans = 8

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 36  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.9[dC]



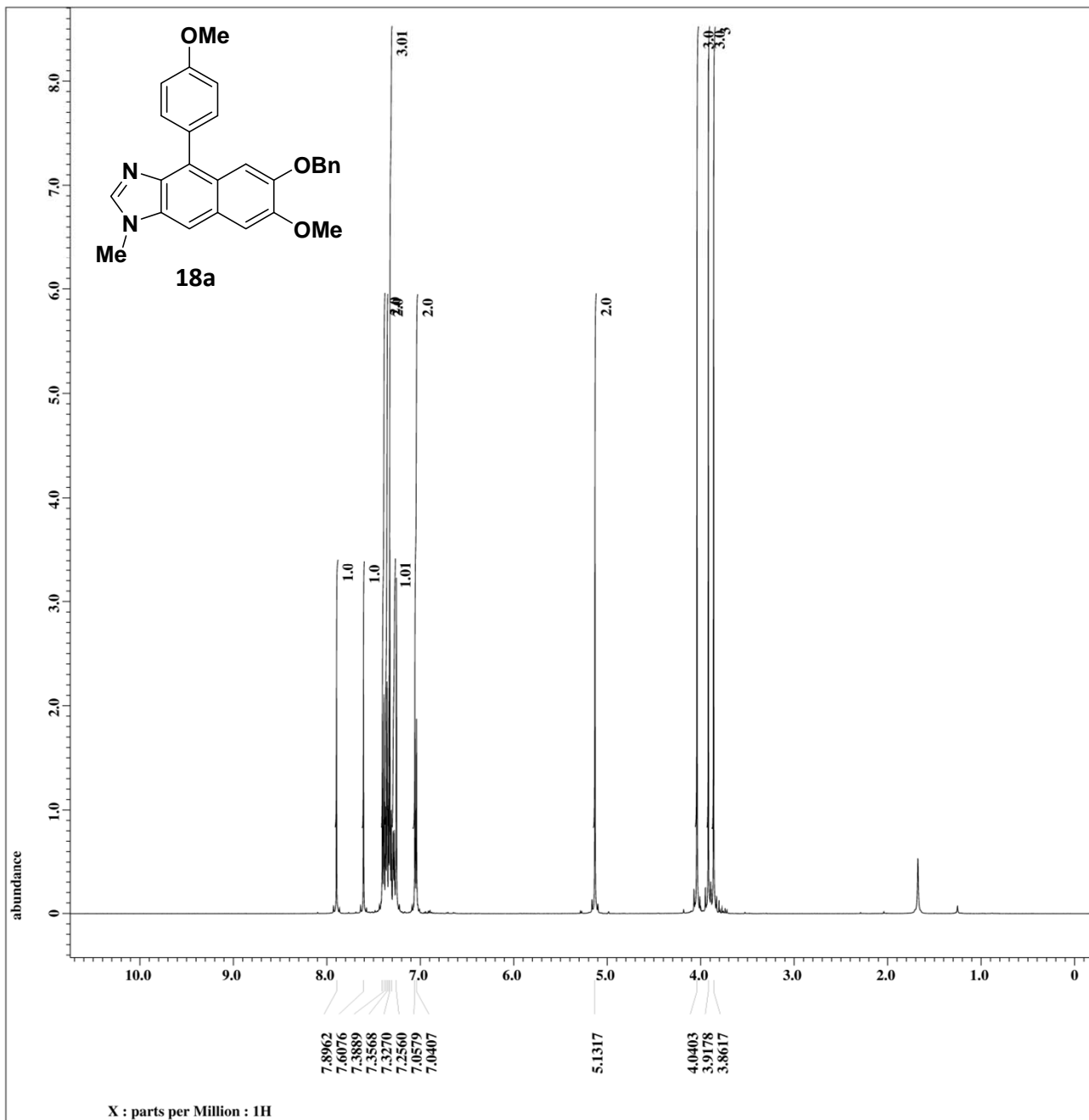


Filename = JKD\_I\_KA\_ALDEHYDE-3.j  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#485010  
 Solvent = CHLOROFORM-D  
 Creation\_time = 12-JAN-2012 04:54:18  
 Revision\_time = 25-JUL-2012 00:45:17  
 Current\_time = 25-JUL-2012 00:45:48

Comment = single pulse decouple  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 870.0  
 Total\_scans = 870.0

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 22.1[dC]

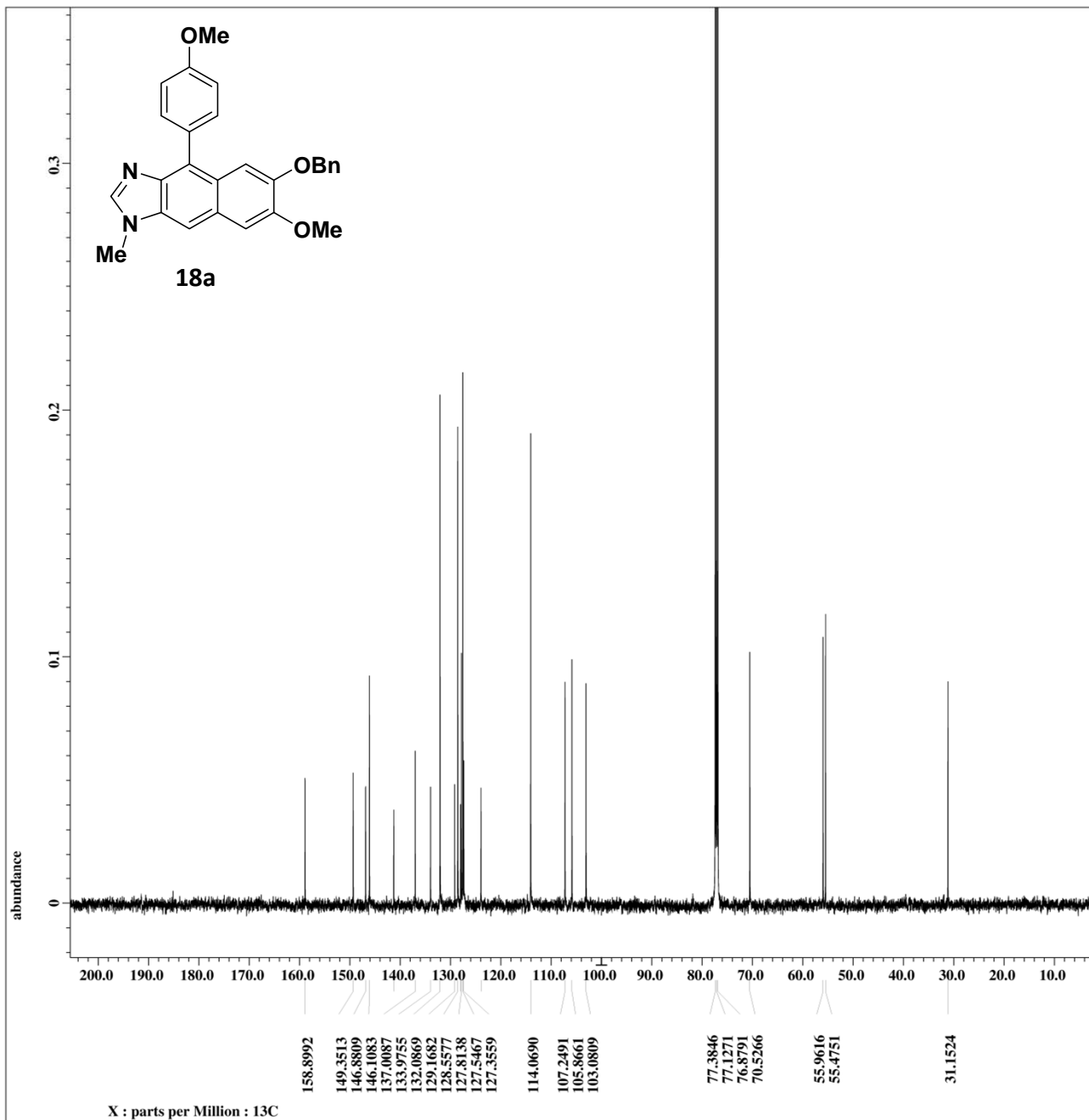


Filename = JKD\_I\_106\_CYCLIC\_KEAL  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#815256  
 Solvent = CHLOROFORM-D  
 Creation\_time = 5-DEC-2011 13:24:17  
 Revision\_time = 25-JUL-2012 00:50:02  
 Current\_time = 25-JUL-2012 00:52:11

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_preset = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 42  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]

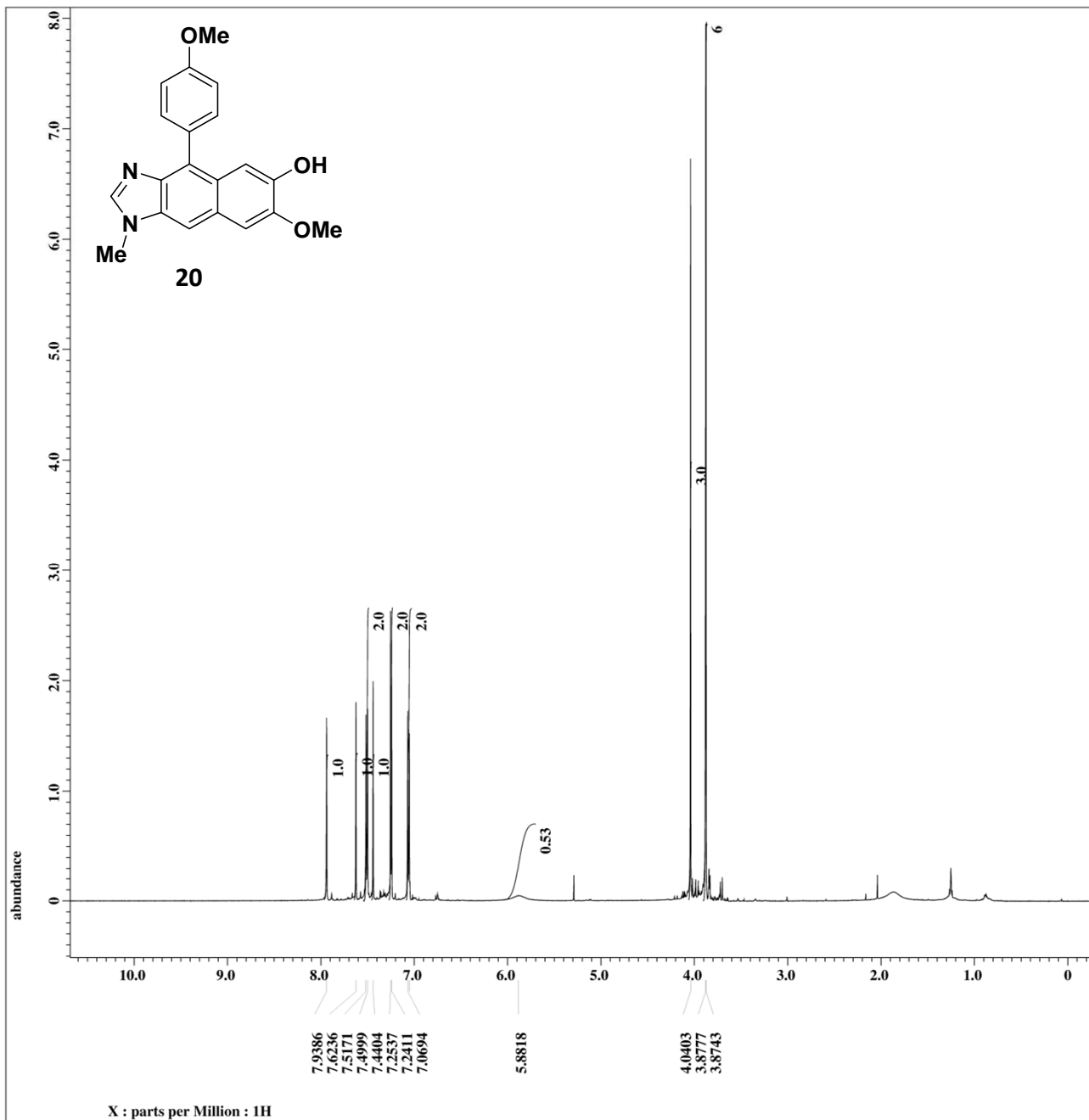


Filename = JKD\_I\_106\_CYCLIC\_KEAL  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = JKD\_I\_106  
 Solvent = CHLOROFORM-D  
 Creation\_time = 5-DEC-2011 14:12:30  
 Revision\_time = 25-JUL-2012 00:53:53  
 Current\_time = 25-JUL-2012 00:54:51

Comment = single pulse decouple  
 Data\_format = 1D COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 1000  
 Total\_scans = 1000

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 22.4[dC]

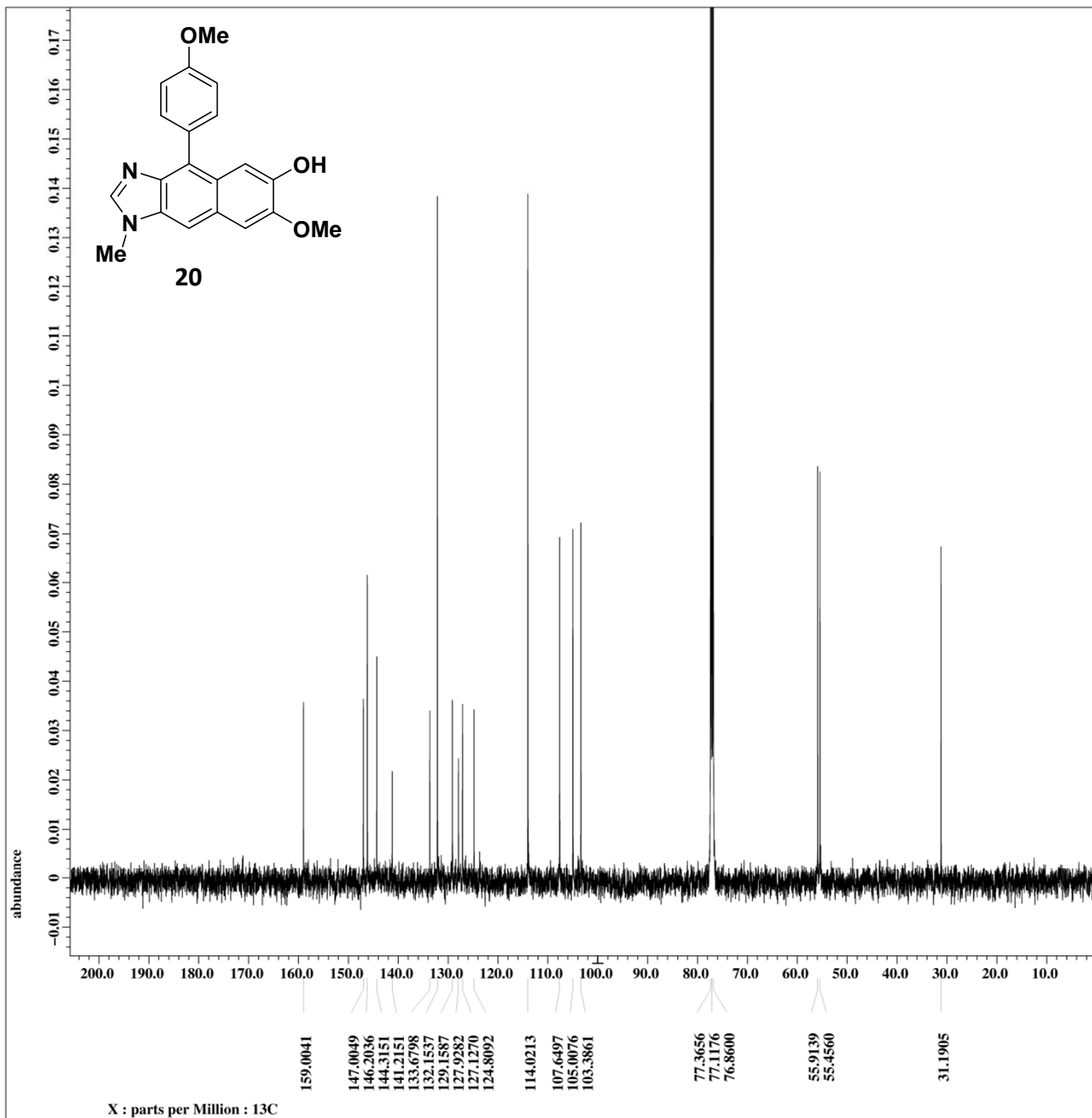


Filename = JKD\_I\_KA\_CYCLIC\_DEPRO  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#692498  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JUL-2012 09:07:52  
 Revision\_time = 25-JUL-2012 09:22:09  
 Current\_time = 25-JUL-2012 09:23:14

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 16  
 Total\_scans = 16

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 44  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.2[dC]

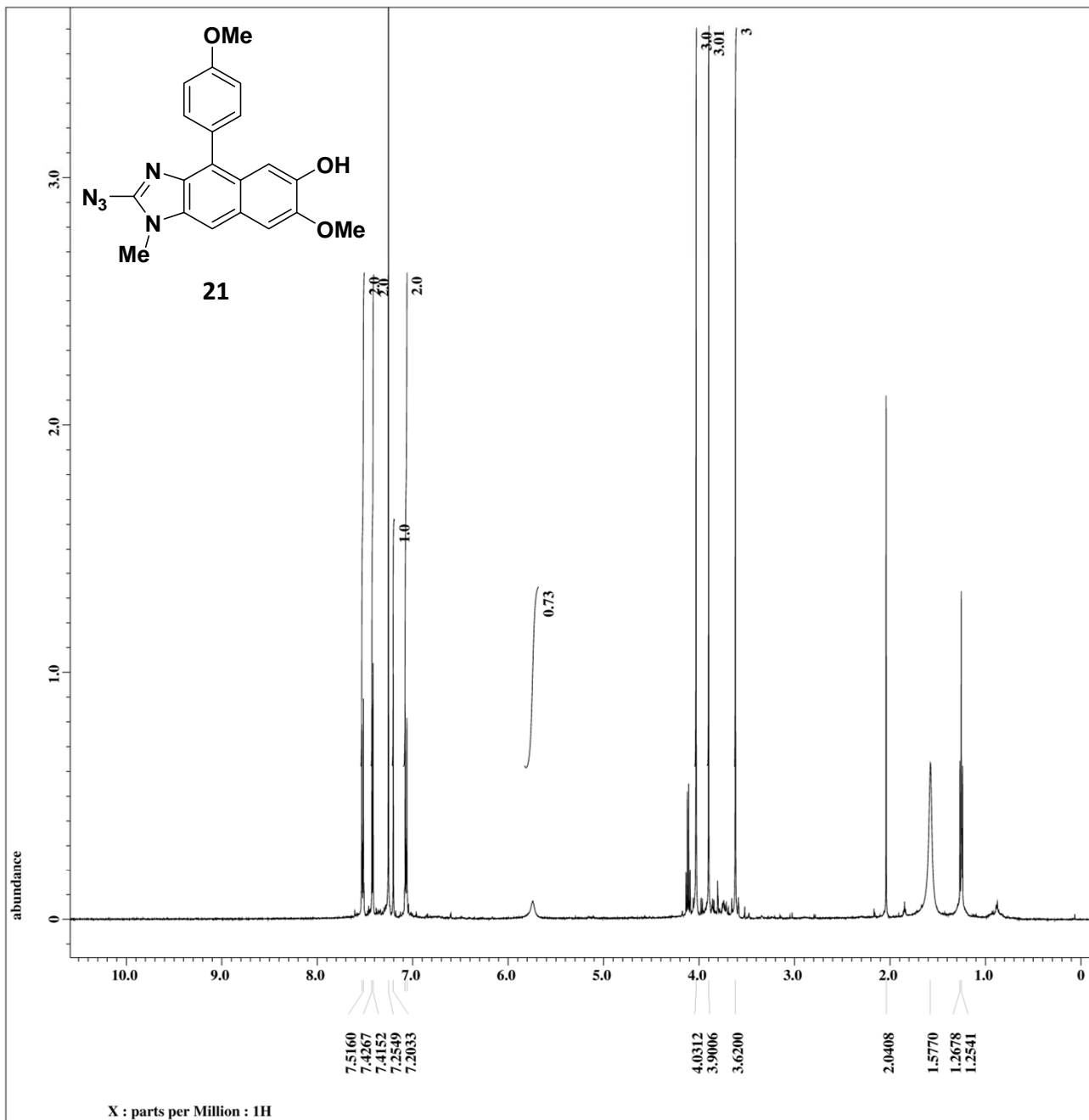


Filename = JKD-I\_KA\_CYCLIC\_DEPRO  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#650342  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JUL-2012 08:42:52  
 Revision\_time = 25-JUL-2012 09:25:17  
 Current\_time = 25-JUL-2012 09:26:21

Comment = single pulse decouple  
 Data\_format = 1D COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768 [MHz]  
 X\_offset = 100 [ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034 [Hz]  
 X\_sweep = 39.3081761 [kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521 [MHz]  
 Irr\_offset = 5.0 [ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 1000  
 Total\_scans = 1000

X\_90\_width = 10.73 [us]  
 X\_acq\_time = 0.83361792 [s]  
 X\_angle = 30 [deg]  
 X\_atn = 9 [dB]  
 X\_pulse = 3.57666667 [us]  
 Irr\_atn\_dec = 20 [dB]  
 Irr\_atn\_noe = 20 [dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1 [s]  
 Noe = TRUE  
 Noe\_time = 2 [s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2 [s]  
 Repetition\_time = 2.83361792 [s]  
 Temp\_get = 22.8 [dC]

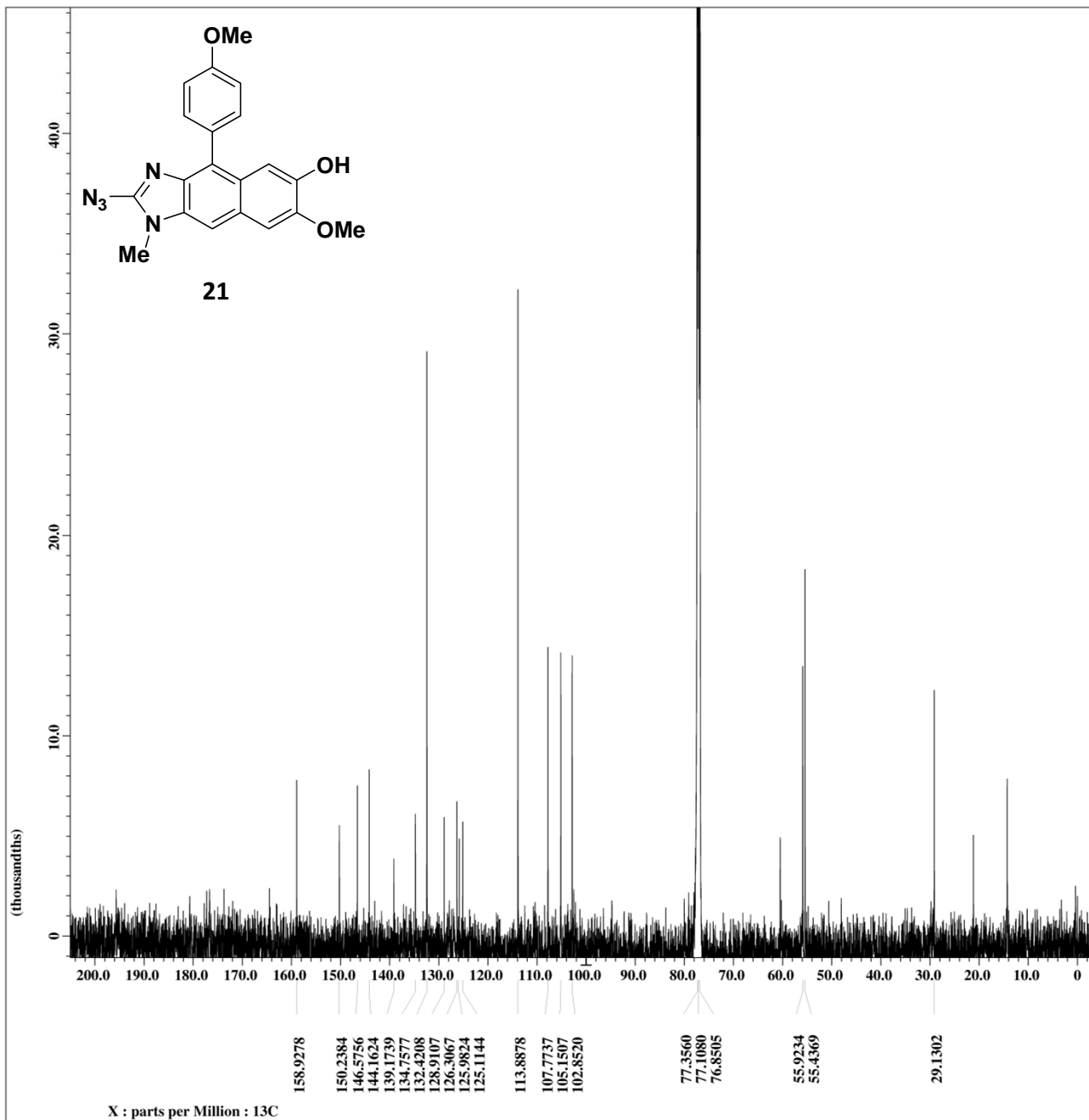


Filename = JKD\_I\_KA\_AZIDE\_METHOD  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#601411  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JUL-2012 06:36:45  
 Revision\_time = 25-JUL-2012 09:31:52  
 Current\_time = 25-JUL-2012 09:32:40

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_preset = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]



```

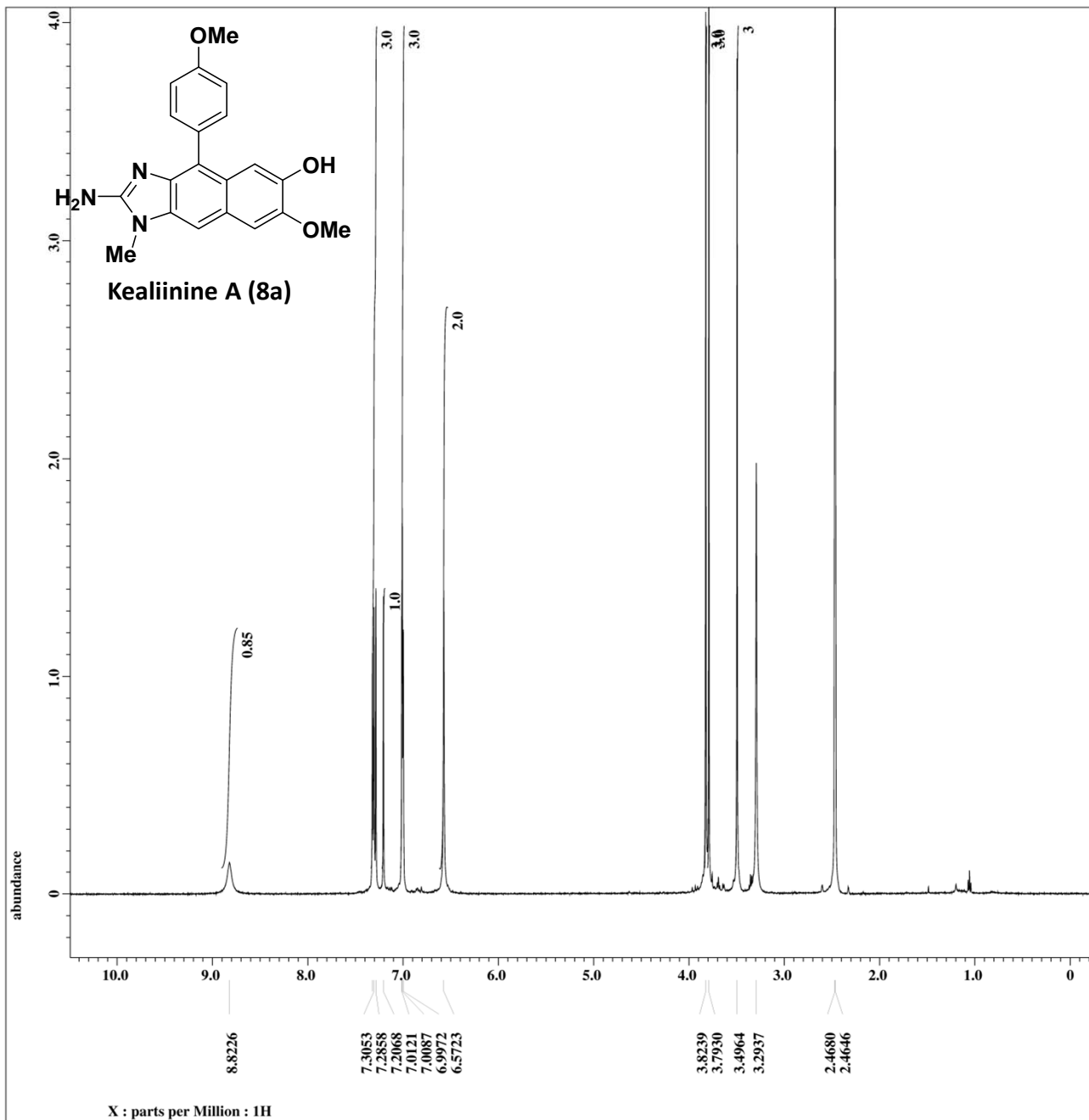
Filename      = JKD_I_KA_AZIDE_M 2-4.
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#32009
Solvent      = CHLOROFORM-D
Creation_time = 16-JUL-2012 17:54:01
Revision_time = 25-JUL-2012 09:40:26
Current_time  = 25-JUL-2012 09:42:01

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 4000
Total_scans    = 4000

X_90_width    = 10.73 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 9 [dB]
X_pulse        = 3.57666667 [us]
Irr_atn_dec   = 20 [dB]
Irr_atn_noe   = 20 [dB]
Irr_noise     = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get       = 22.6 [dC]

```



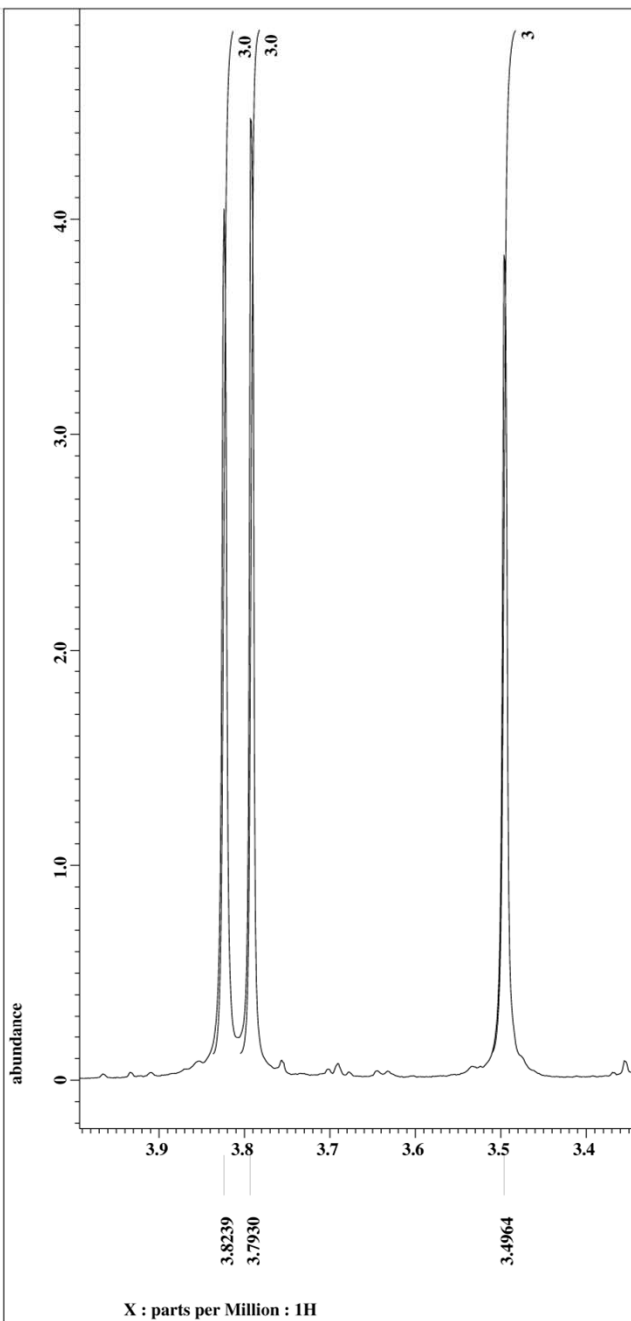
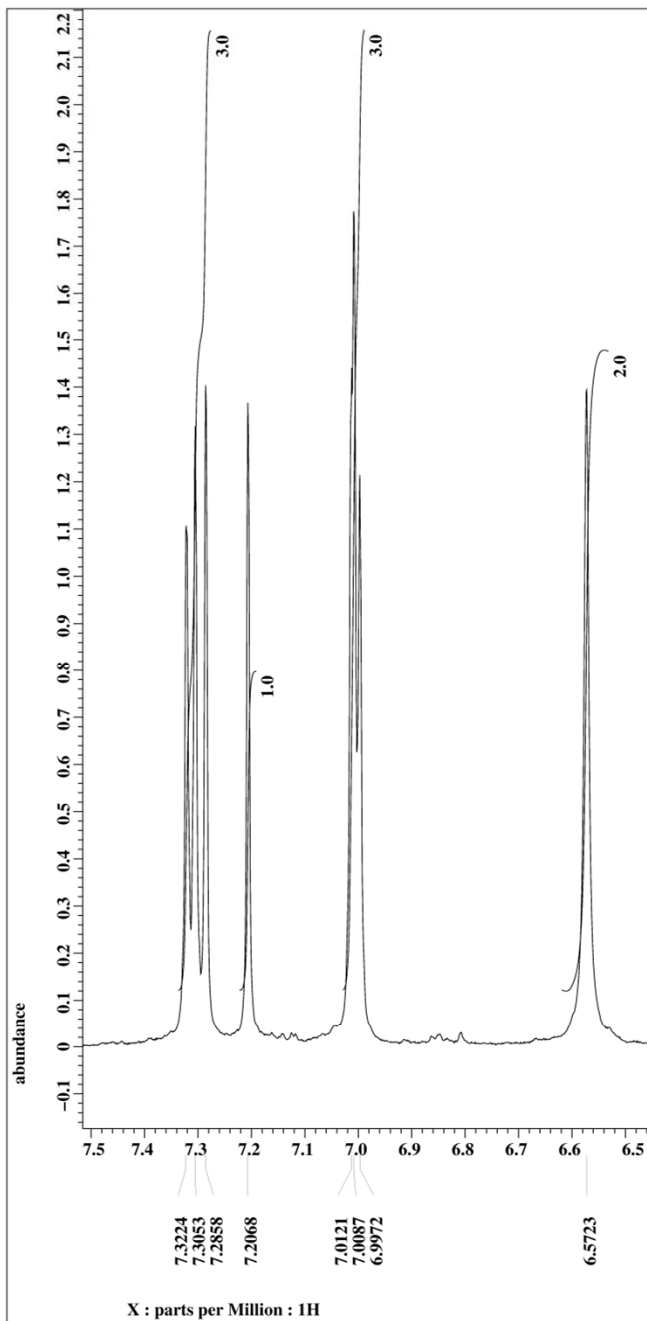
Filename = JKD\_I\_KA\_FINAL METHOD  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#525061  
 Solvent = DMSO-D6  
 Creation\_time = 10-JUL-2012 04:29:27  
 Revision\_time = 25-JUL-2012 09:48:33  
 Current\_time = 25-JUL-2012 09:49:44

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_preset = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]



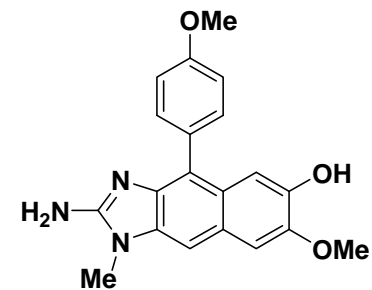


Filename = JKD\_I\_KA\_FINAL\_METHOD  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#525061  
 Solvent = DMSO-D6  
 Creation\_time = 10-JUL-2012 04:29:27  
 Revision\_time = 25-JUL-2012 09:48:33  
 Current\_time = 25-JUL-2012 09:50:38

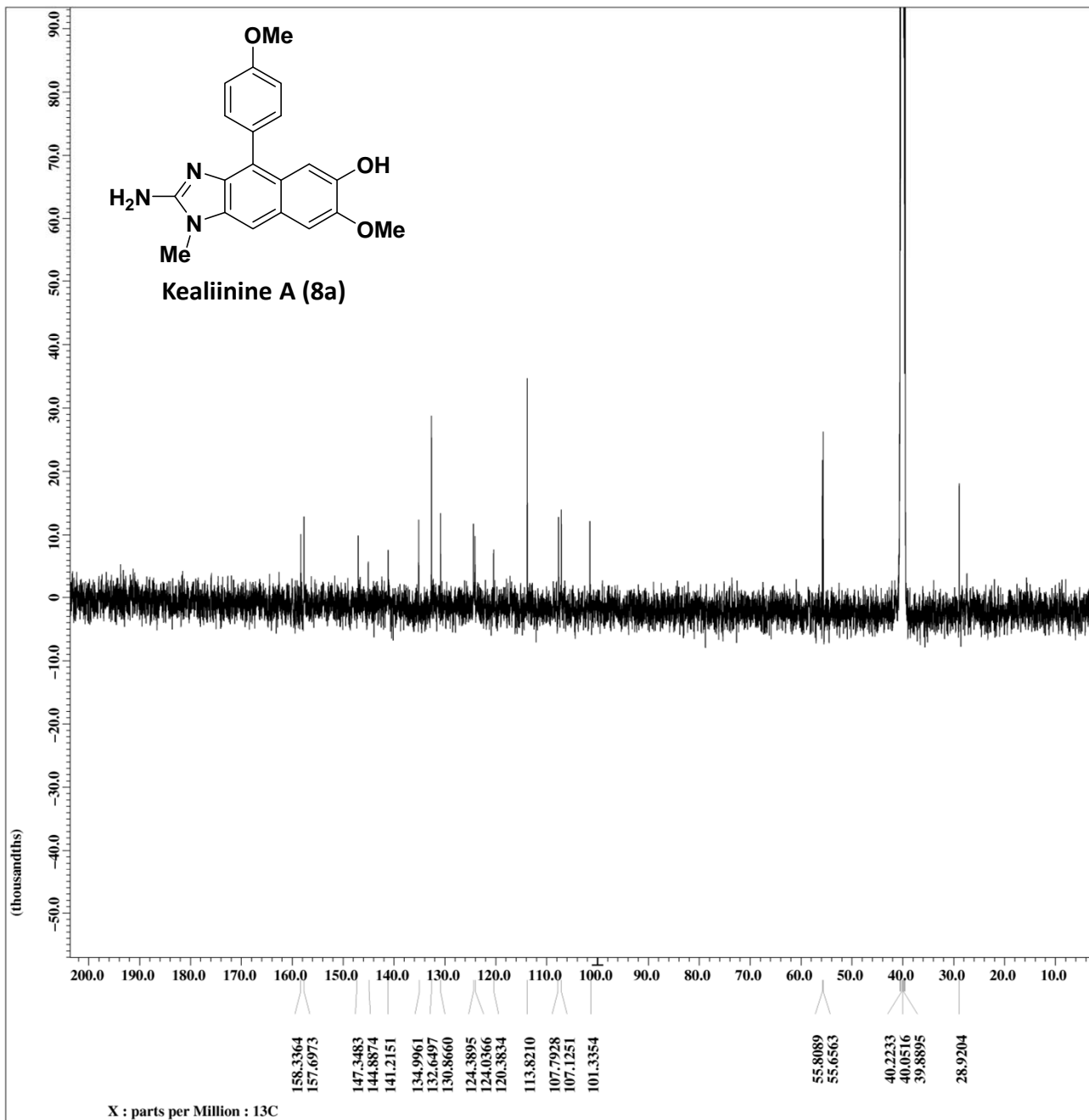
Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]



Kealiinine A (8a)



```

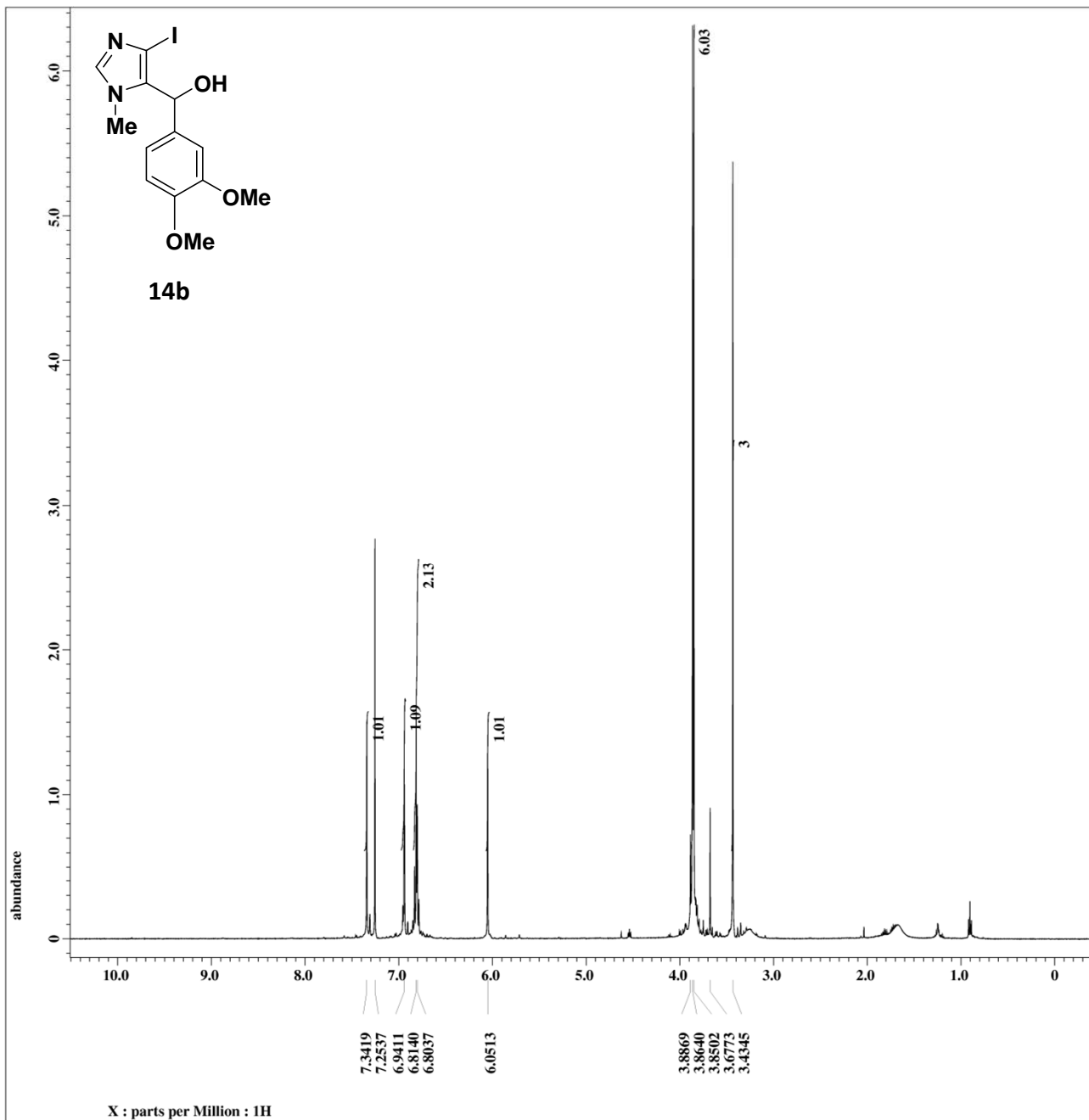
Filename      = JKD_I_KA_FINAL_METHOD
Author       = delta
Experiment    = single_pulse_dec
Sample_id    = S#527329
Solvent      = DMSO-D6
Creation_time = 10-JUL-2012 05:09:47
Revision_time = 31-JUL-2012 05:35:28
Current_time  = 31-JUL-2012 05:37:51

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 830
Total_scans    = 830

X_90_width    = 10.73[us]
X_acq_time     = 0.83361792[s]
X_angle        = 30[deg]
X_atn          = 9[dB]
X_pulse        = 3.57666667[us]
Irr_atn_dec    = 20[dB]
Irr_atn_noe    = 20[dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1[s]
Noe            = TRUE
Noe_time       = 2[s]
Recvr_gain     = 50
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get       = 22.5[dC]

```

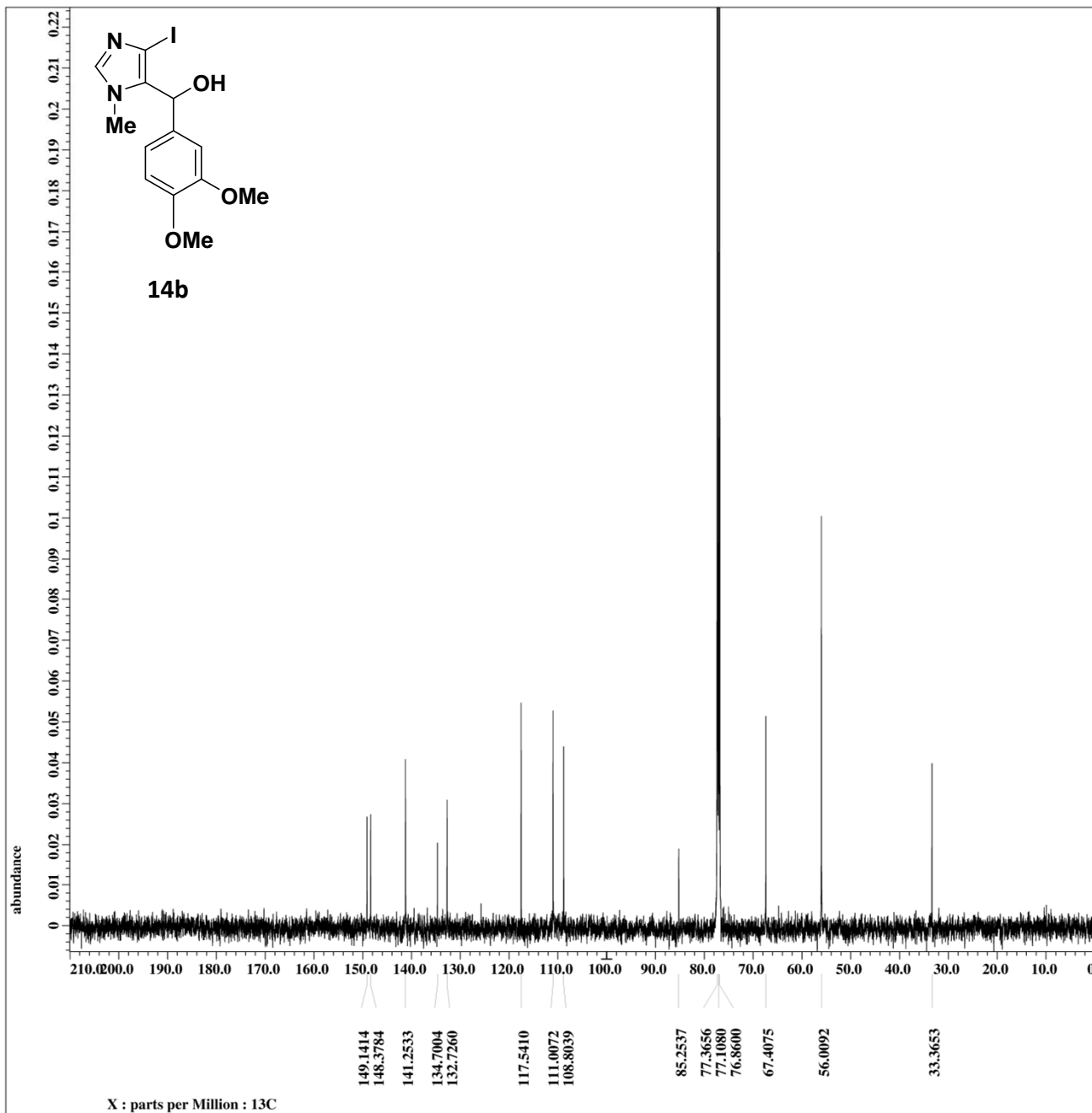


Filename = JKD\_I\_82\_MONO\_ALCOHOL  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#492810  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JAN-2012 04:28:20  
 Revision\_time = 18-JUL-2012 22:58:29  
 Current\_time = 18-JUL-2012 23:01:21

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 9616  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 46  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.6[dC]

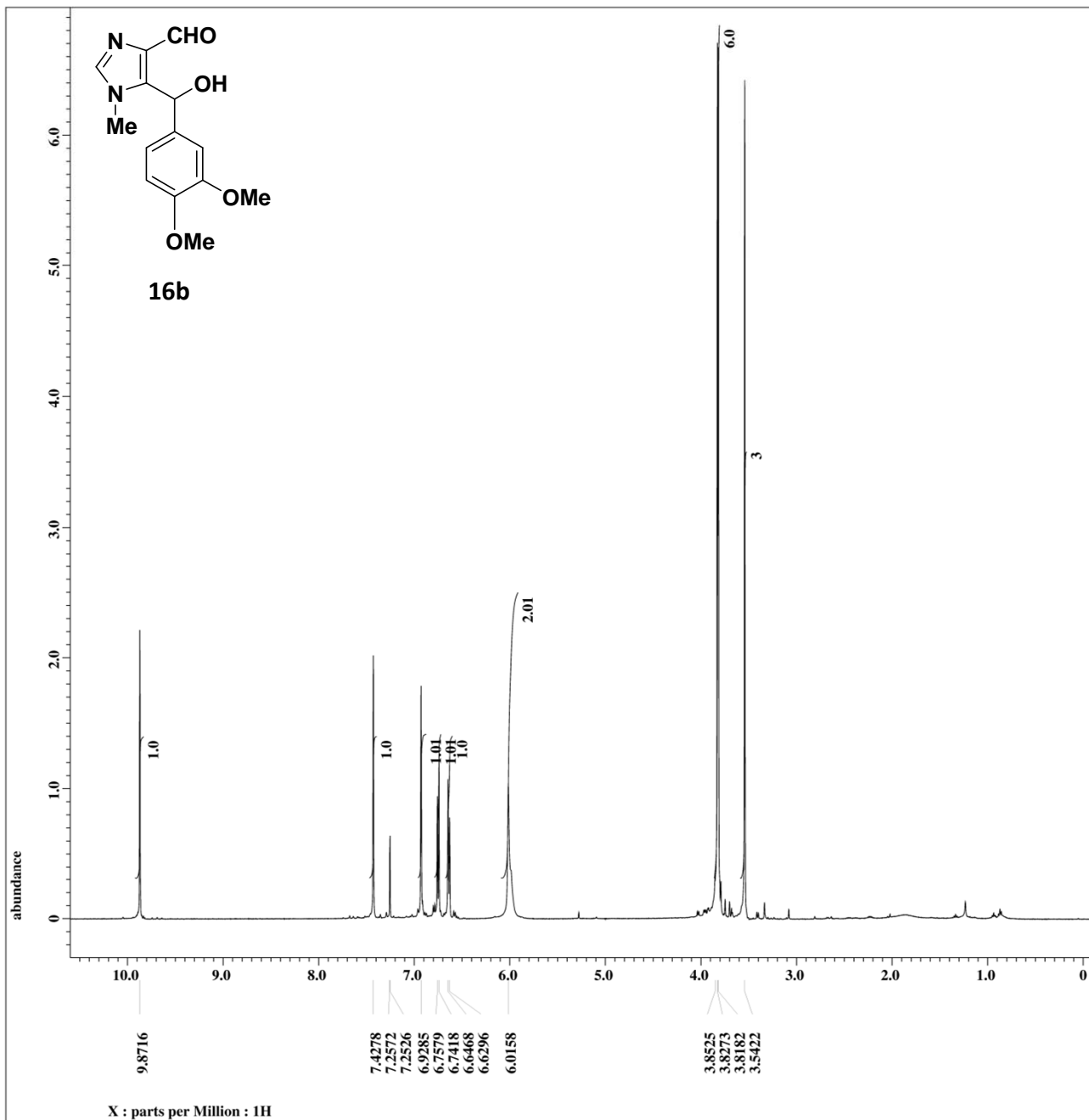


Filename = JKD\_I\_82\_MONO\_ALCOHOL  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#495227  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JAN-2012 05:16:42  
 Revision\_time = 19-JUL-2012 00:28:56  
 Current\_time = 19-JUL-2012 00:29:53

Comment = single pulse decouple  
 Data\_format = 1D COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 1000  
 Total\_scans = 1000

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 22.3[dC]

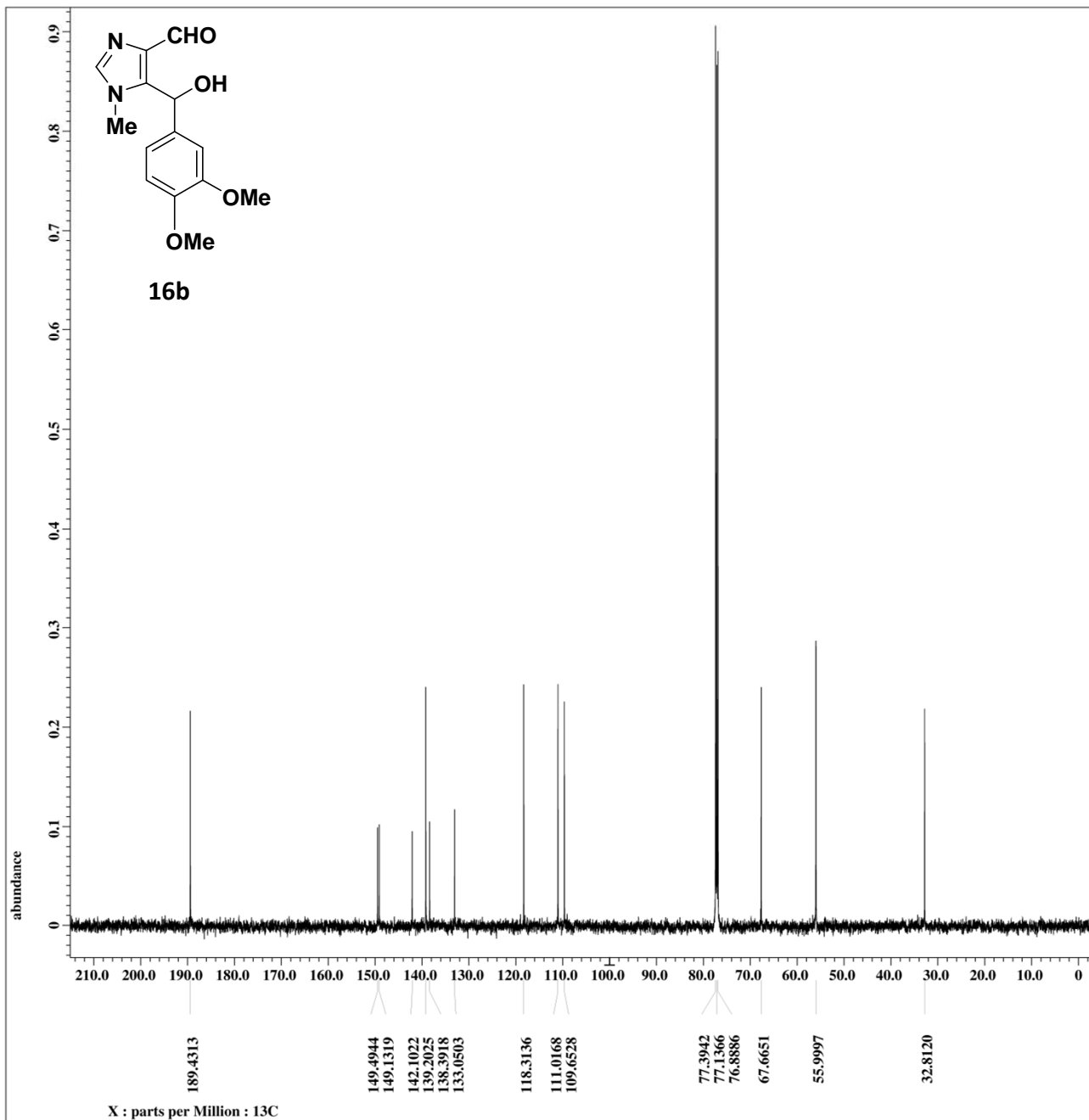


Filename = JKD\_I\_83\_ALDEHYDE\_KB-  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#481632  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JAN-2012 04:07:46  
 Revision\_time = 18-JUL-2012 23:57:01  
 Current\_time = 18-JUL-2012 23:57:47

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 5  
 Total\_scans = 5

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 38  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.7[dC]

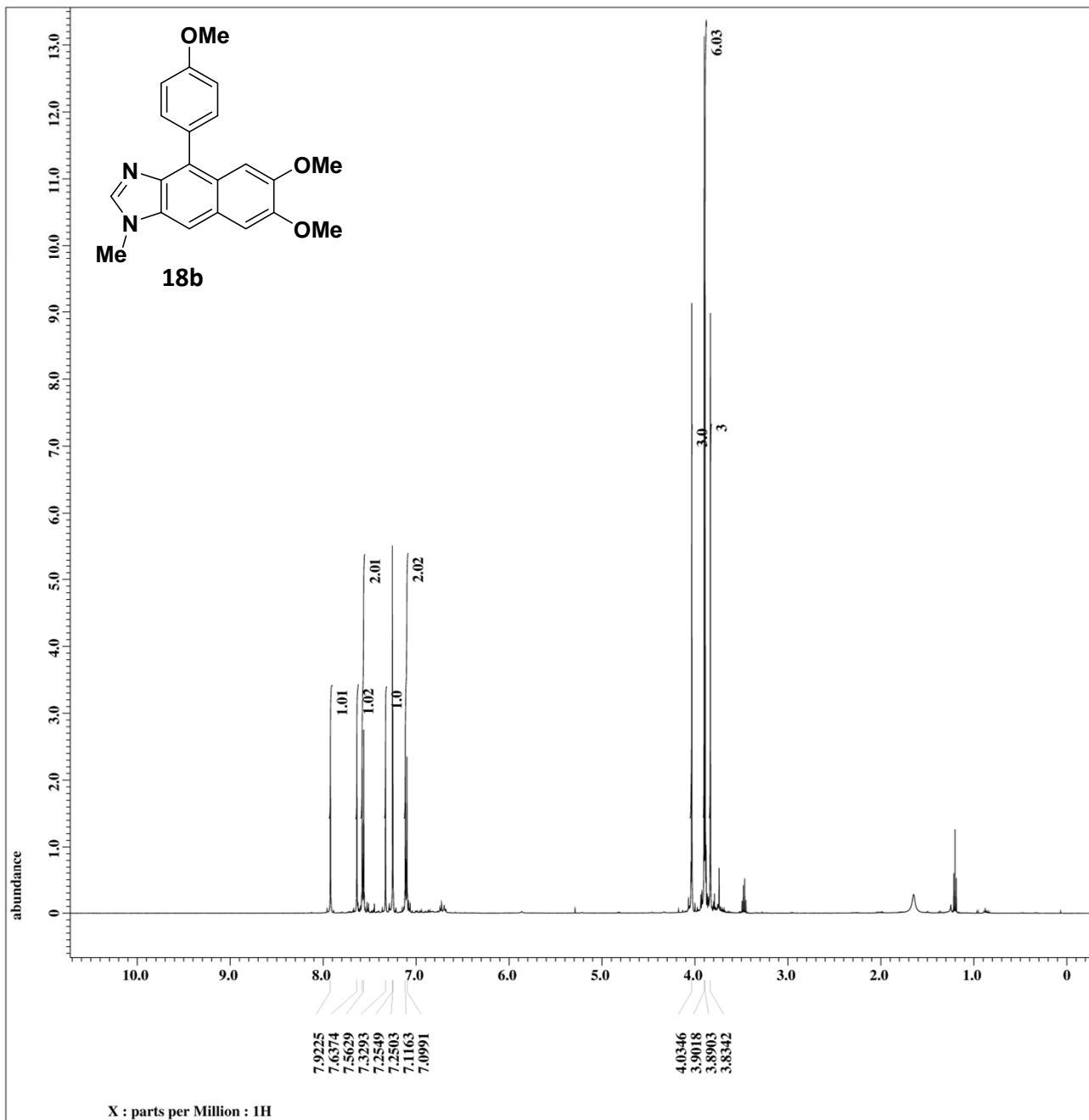


Filename = JKD\_I\_83\_ALDEHYDE\_KB-  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#482687  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JAN-2012 04:19:26  
 Revision\_time = 19-JUL-2012 00:30:35  
 Current\_time = 19-JUL-2012 00:31:25

Comment = single pulse decouple  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768 [MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521 [MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 230.0  
 Total\_scans = 230.0

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 22[dC]

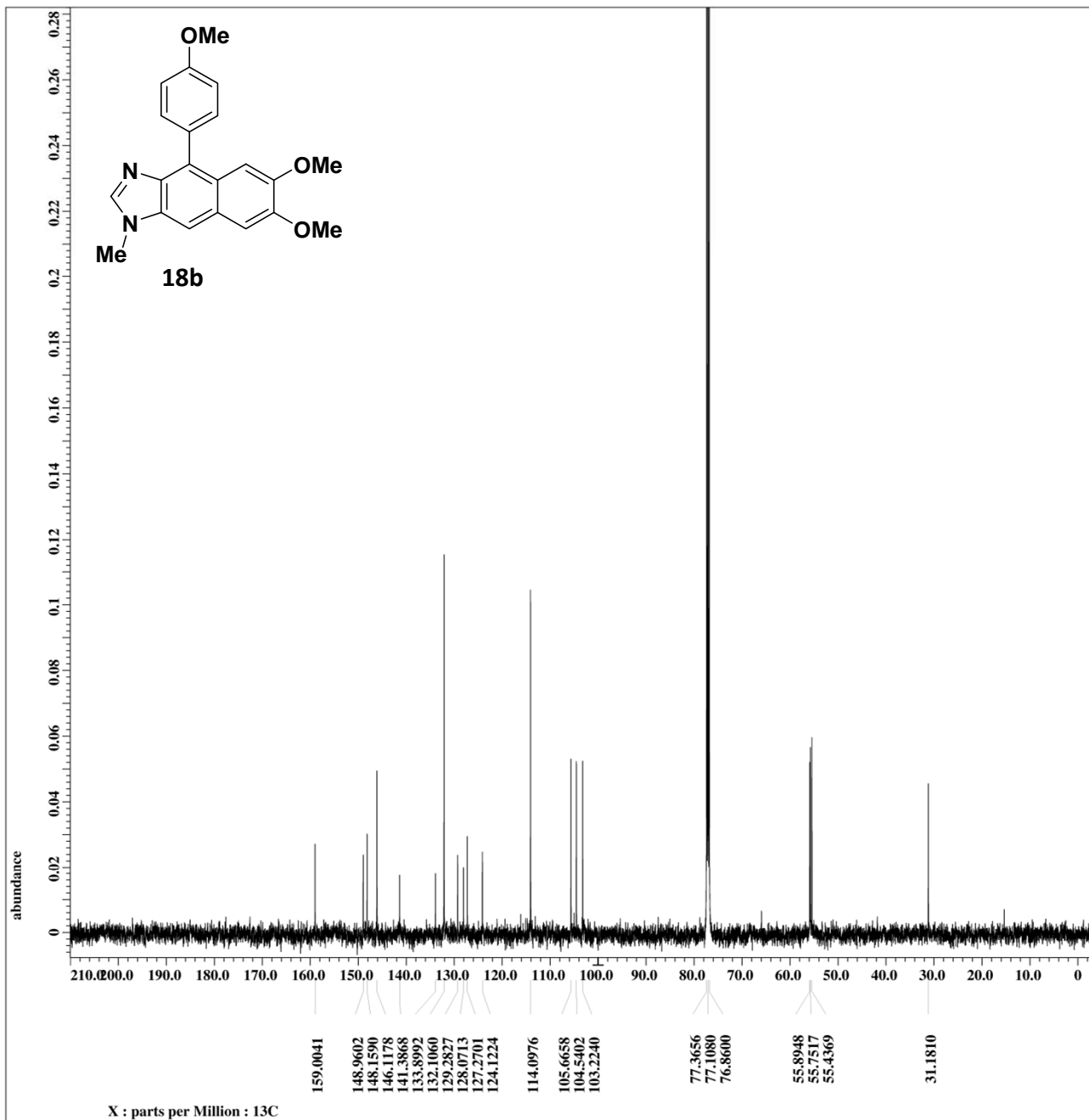


Filename = JKD\_I\_89\_CYCLIC\_KEALI  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#462755  
 Solvent = CHLOROFORM-D  
 Creation\_time = 27-OCT-2011 02:35:43  
 Revision\_time = 19-JUL-2012 00:03:24  
 Current\_time = 19-JUL-2012 00:04:07

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 46  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.3[dC]



```

Filename      = JKD_I_CYCLIC_KEALIINI
Author        = delta
Experiment    = single_pulse_dec
Sample_id     = S#465420
Solvent       = CHLOROFORM-D
Creation_time = 27-OCT-2011 03:10:08
Revision_time = 19-JUL-2012 00:32:04
Current_time  = 19-JUL-2012 00:33:15

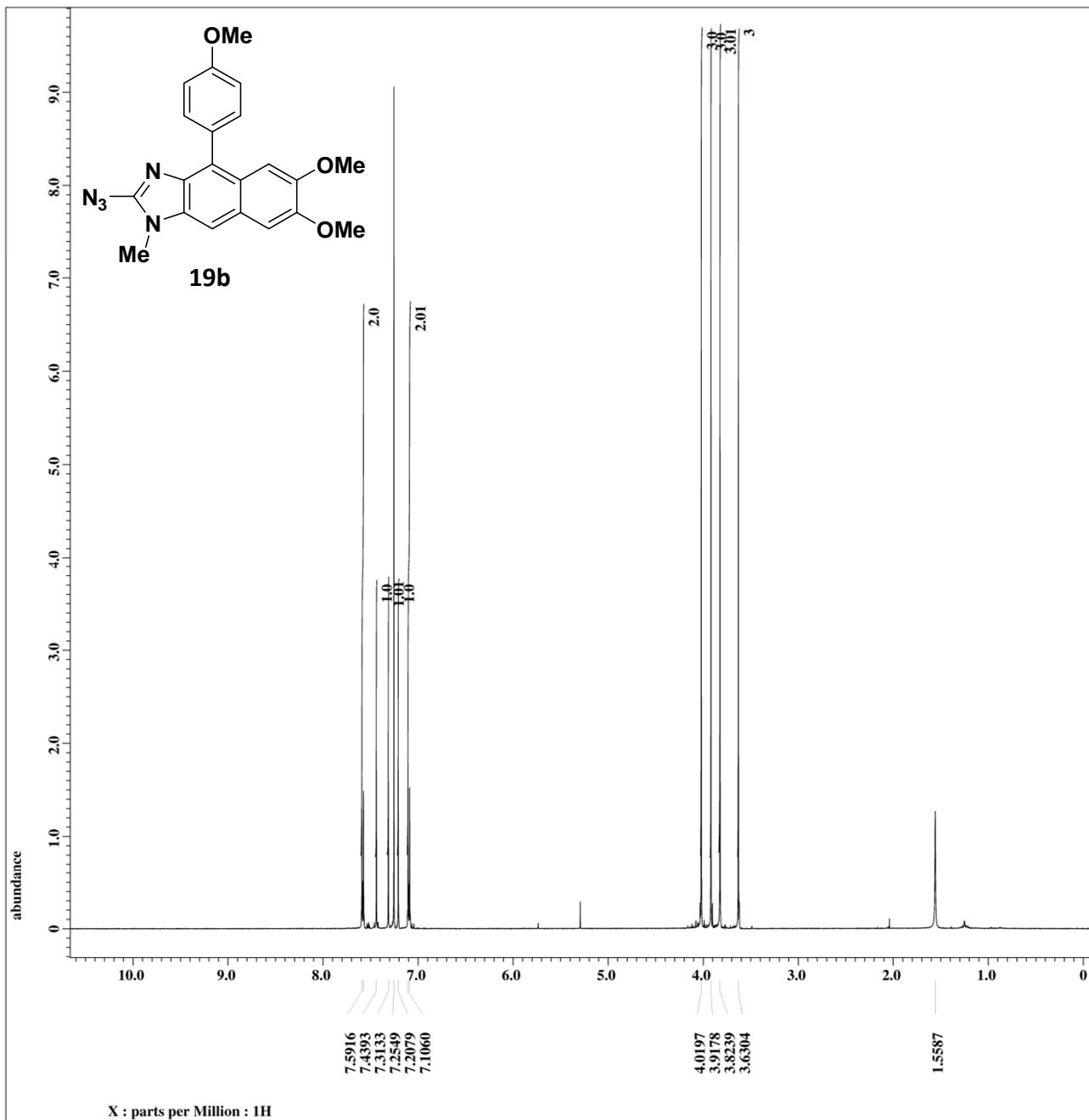
Comment       = single pulse decouple
Data_format   = 1D COMPLEX
Dim_size      = 26214
Dim_title     = 13C
Dim_units     = [ppm]
Dimensions    = X
Site          = ECA 500
Spectrometer  = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100 [ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034 [Hz]
X_sweep        = 39.3081761 [kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0 [ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 700
Total_scans    = 700

X_90_width    = 10.73 [us]
X_acq_time     = 0.83361792 [s]
X_angle        = 30 [deg]
X_atn          = 9 [dB]
X_pulse        = 3.57666667 [us]
Irr_atn_dec   = 20 [dB]
Irr_atn_noe   = 20 [dB]
Irr_noise     = WALTZ
Decoupling     = TRUE
Initial_wait   = 1 [s]
Noe            = TRUE
Noe_time       = 2 [s]
Recvr_gain     = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get       = 22.8 [dC]

```



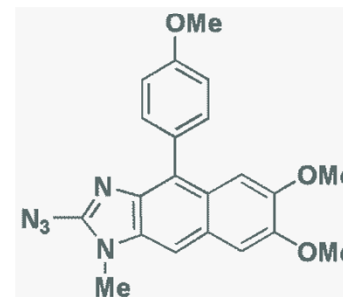


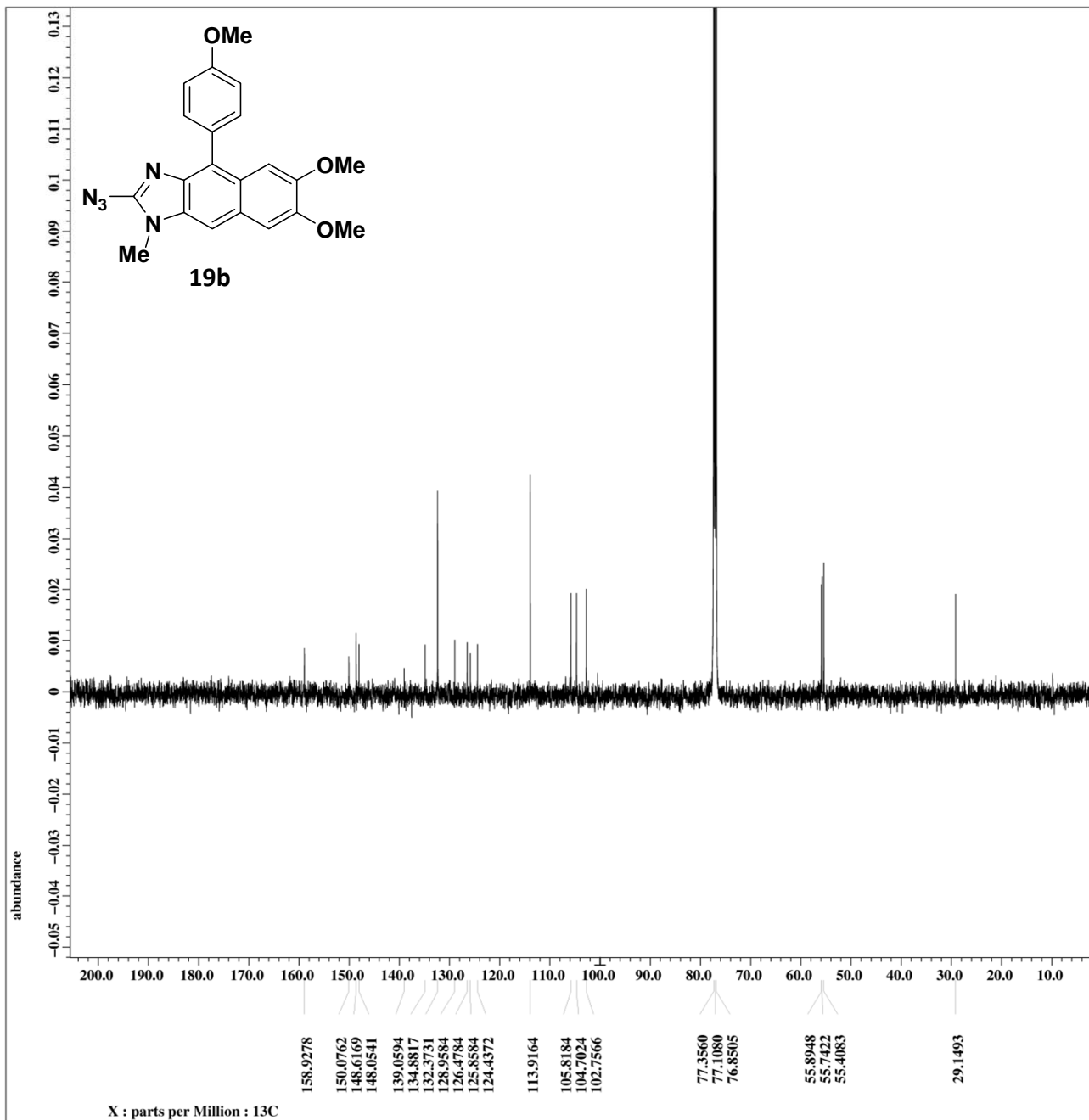
Filename = JKD\_I\_KB\_AZIDE-4.jdf  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#626023  
 Solvent = CHLOROFORM-D  
 Creation\_time = 14-JUL-2012 07:17:46  
 Revision\_time = 19-JUL-2012 00:11:57  
 Current\_time = 19-JUL-2012 00:12:39

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.1[dC]





```

Filename      = JKD_I_KB_AZIDE_1-4.jd
Author        = delta
Experiment    = single_pulse_dec
Sample_id     = S#571696
Solvent       = CHLOROFORM-D
Creation_time = 17-JUL-2012 07:19:05
Revision_time = 25-JUL-2012 00:12:05
Current_time  = 25-JUL-2012 00:14:22
  
```

```

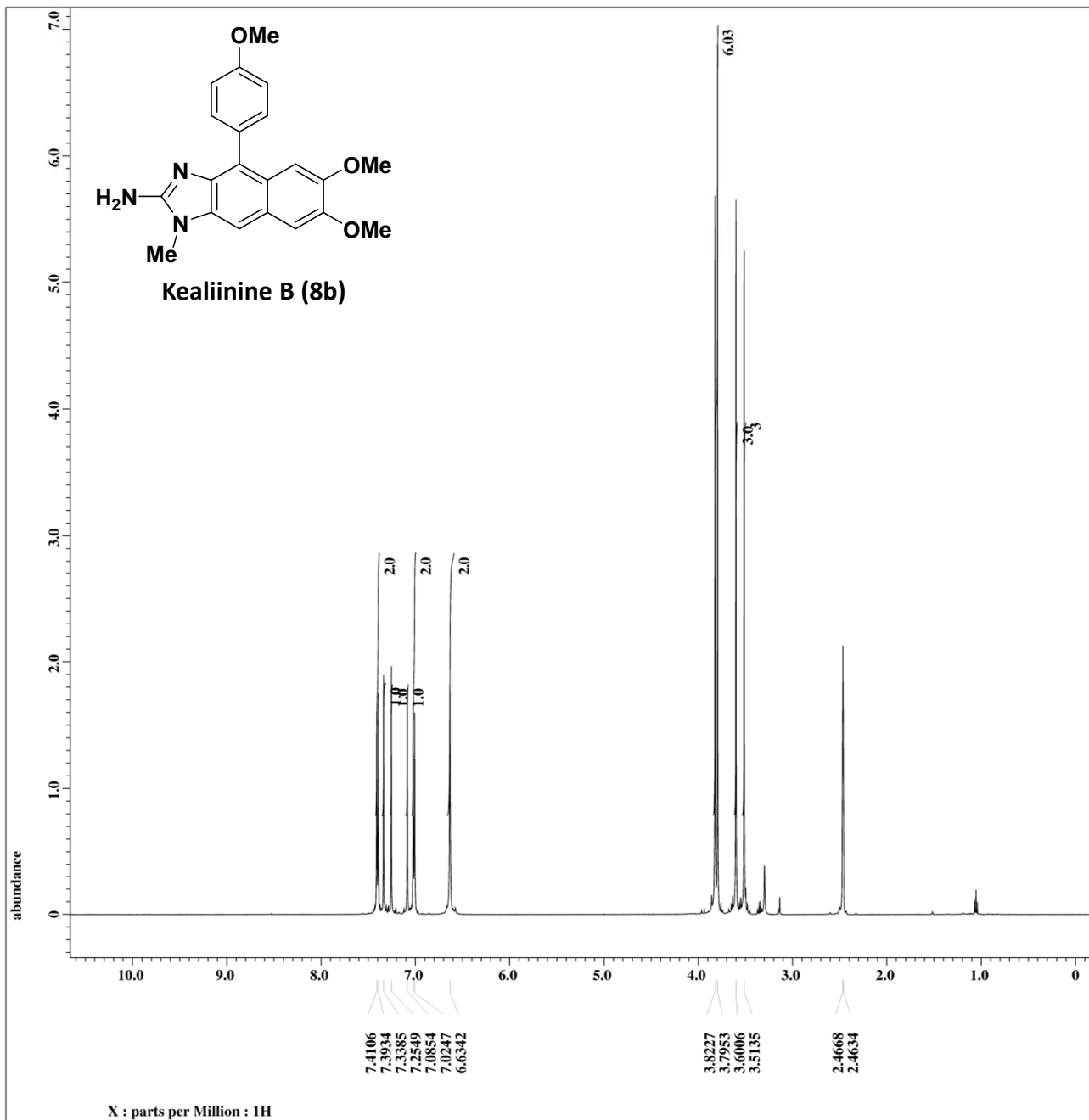
Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500
  
```

```

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 2000
Total_scans    = 2000
  
```

```

X_90_width    = 10.73[us]
X_acq_time     = 0.83361792[s]
X_angle        = 30[deg]
X_atn          = 9[dB]
X_pulse        = 3.57666667[us]
Irr_atn_dec    = 20[dB]
Irr_atn_noe    = 20[dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1[s]
Noe            = TRUE
Noe_time       = 2[s]
Recvr_gain     = 50
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get       = 22.6[dC]
  
```

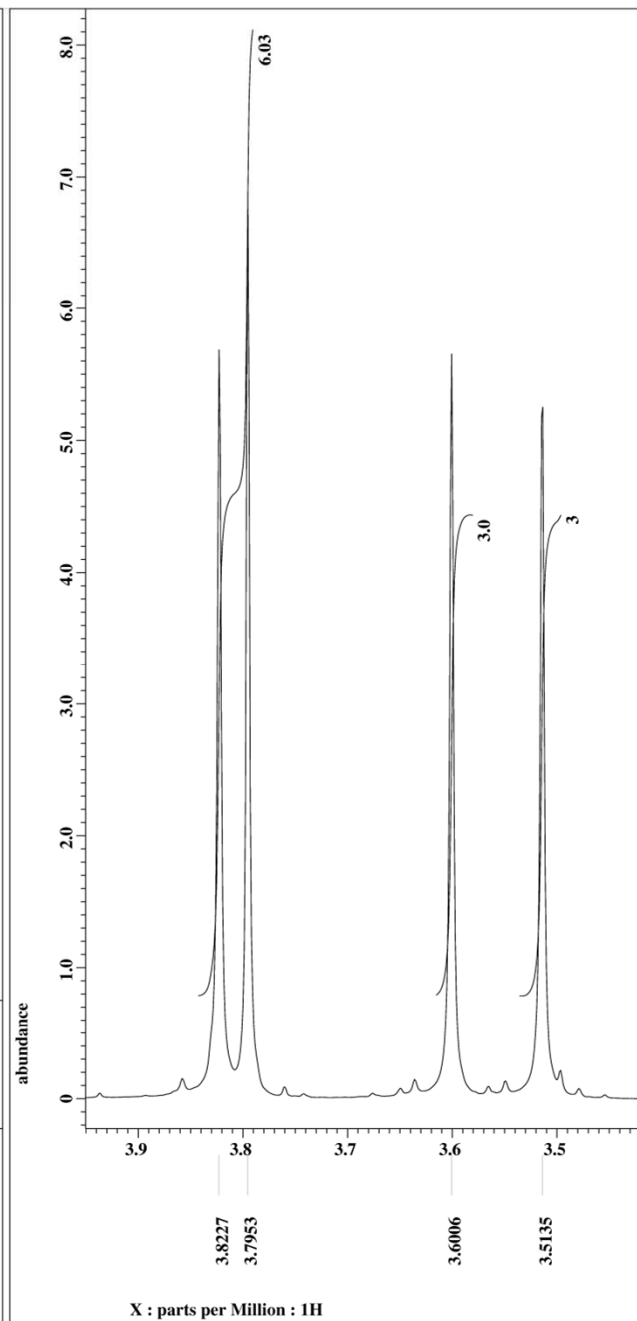
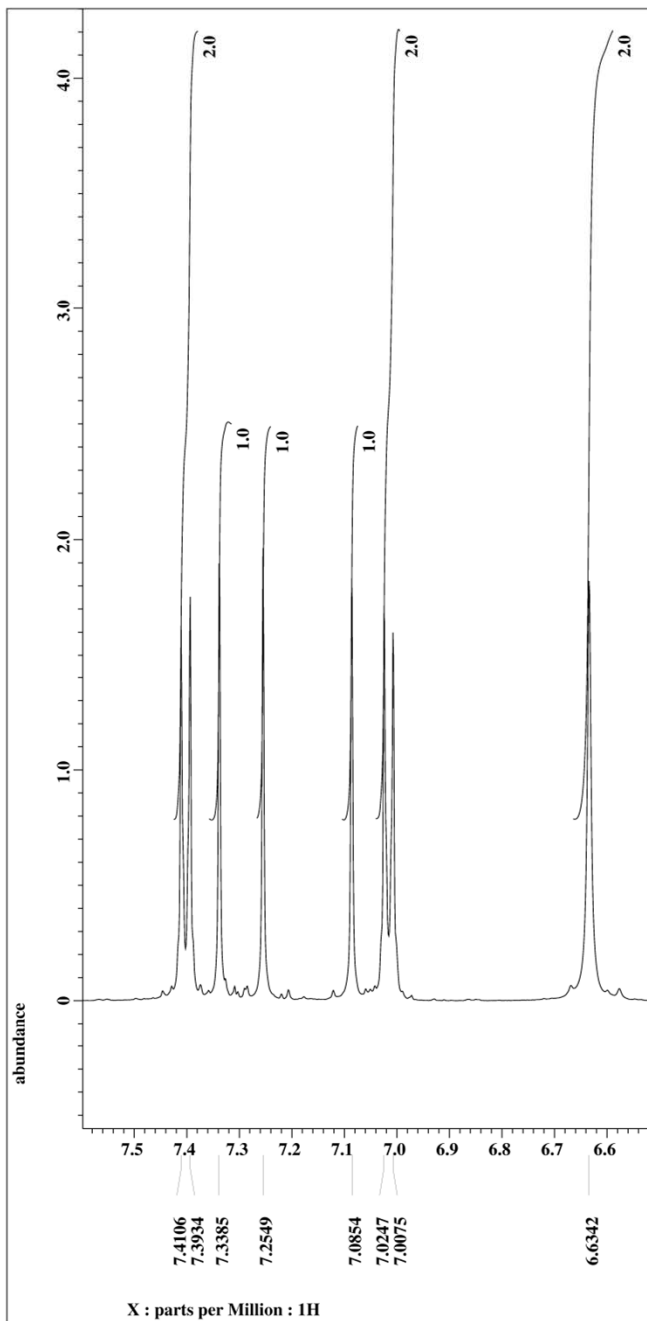


Filename = JKD\_I\_92\_KEALIININE B  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#548518  
 Solvent = DMSO-D6  
 Creation\_time = 1-NOV-2011 04:58:42  
 Revision\_time = 19-JUL-2012 00:18:46  
 Current\_time = 19-JUL-2012 00:19:41

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 42  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.3[dC]

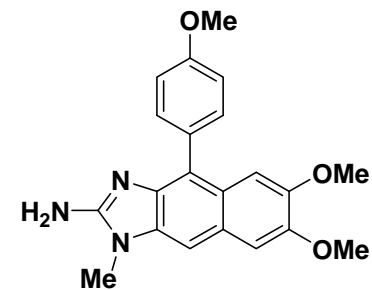


Filename = JKD\_I\_92\_KEALIININE B  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#548518  
 Solvent = DMSO-D6  
 Creation\_time = 1-NOV-2011 04:58:42  
 Revision\_time = 19-JUL-2012 00:18:46  
 Current\_time = 19-JUL-2012 00:21:41

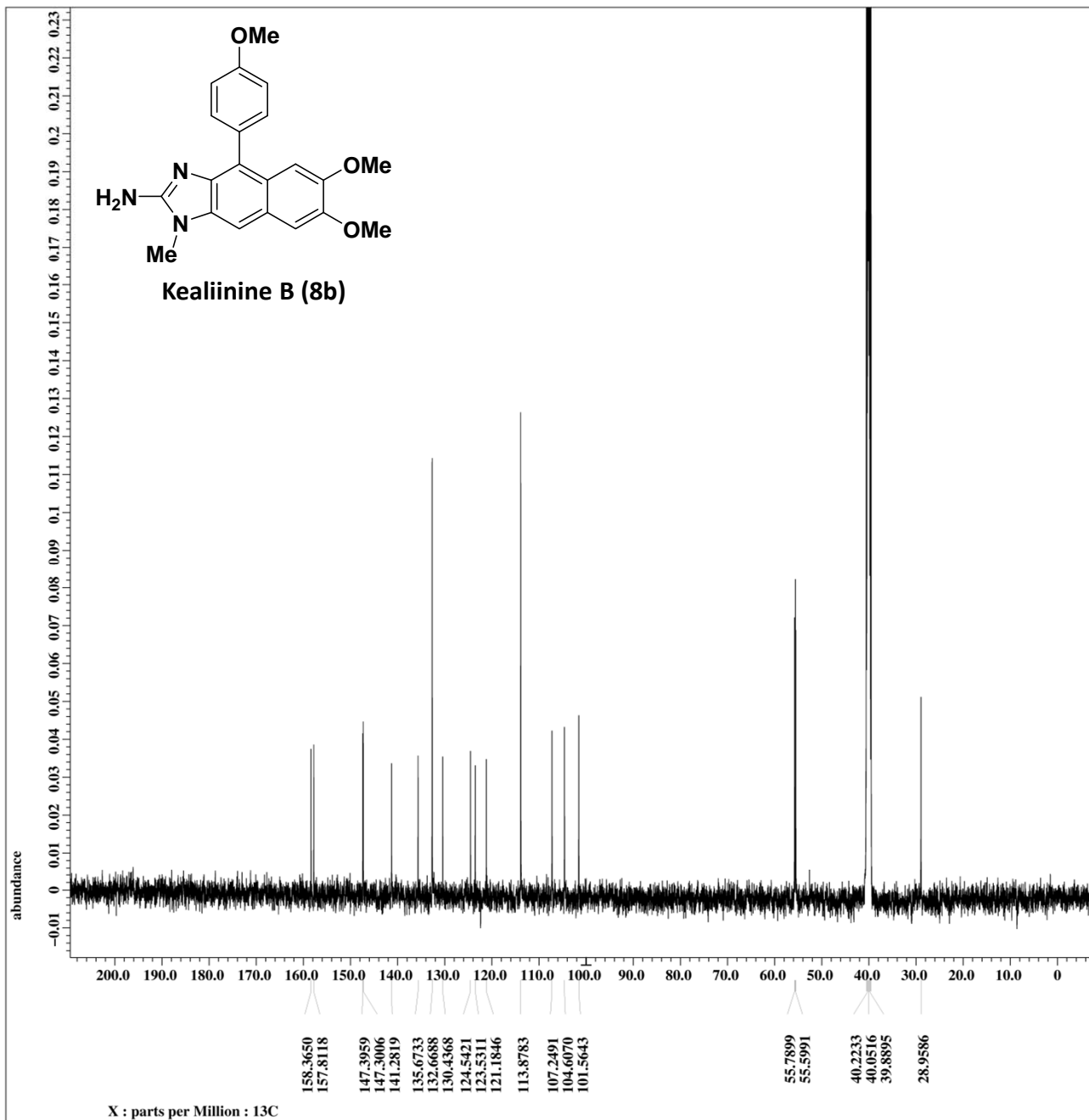
Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 42  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 22.3[dc]



Kealiinine B (8b)

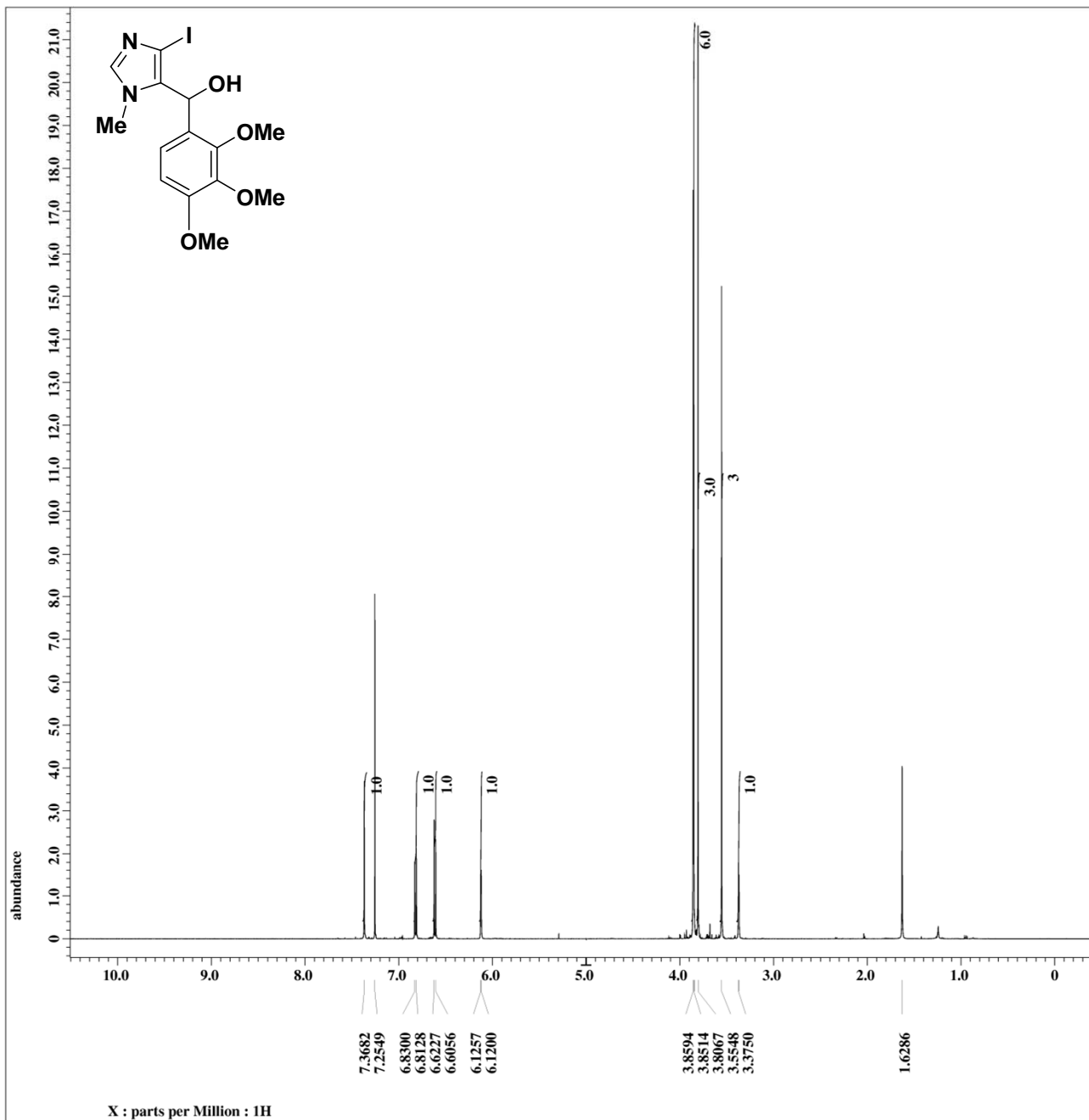


Filename = JKD\_I\_92\_KEALIININE B  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#551393  
 Solvent = DMSO-D6  
 Creation\_time = 1-NOV-2011 05:31:33  
 Revision\_time = 19-JUL-2012 00:36:00  
 Current\_time = 19-JUL-2012 00:36:39

Comment = single pulse decouple  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 660  
 Total\_scans = 660

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 22.3[dC]

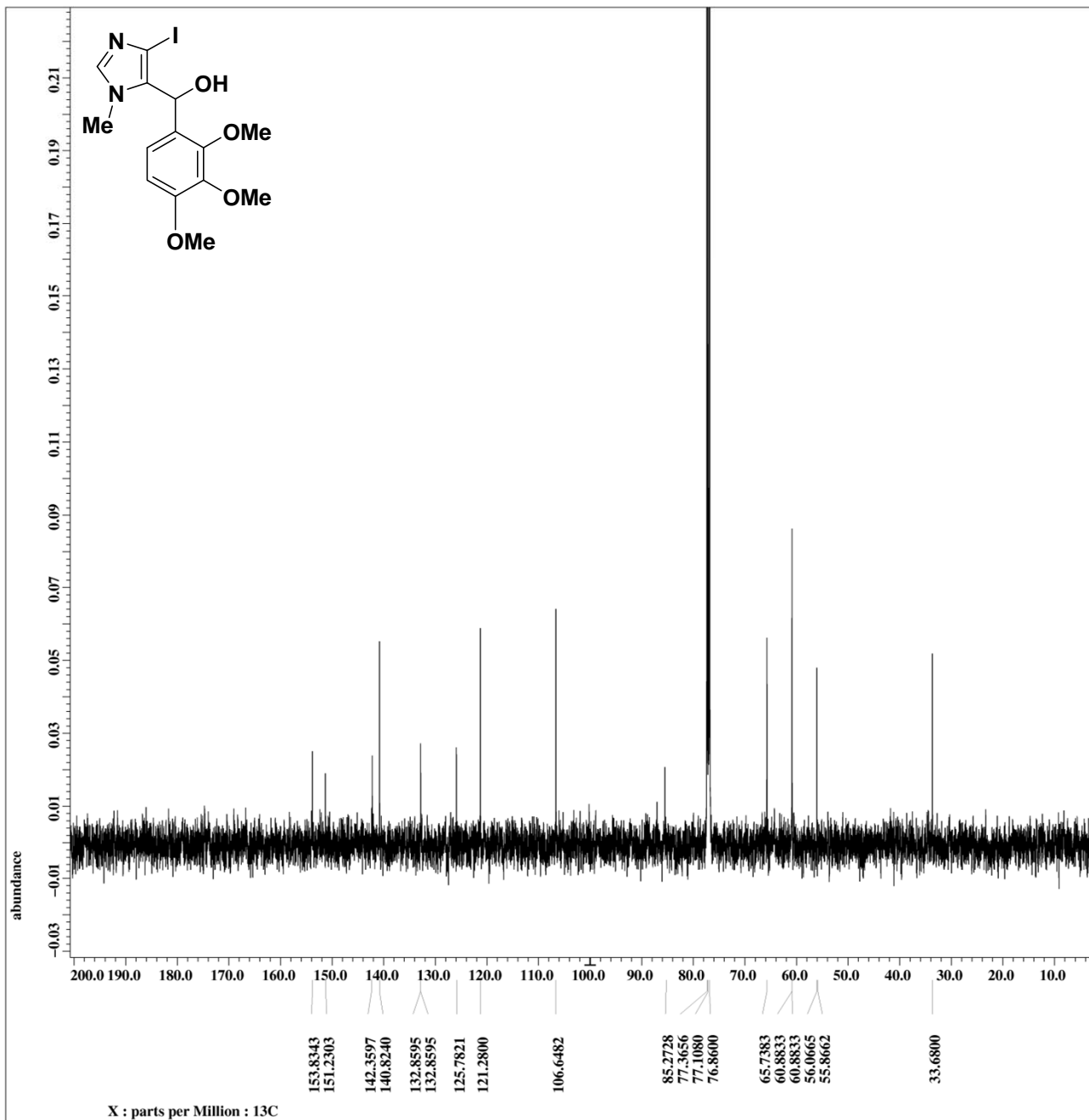


Filename = JKD\_I\_58\_MONO ALCOHOL  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#627449  
 Solvent = CHLOROFORM-D  
 Creation\_time = 30-APR-2011 07:32:36  
 Revision\_time = 17-MAY-2012 00:36:59  
 Current\_time = 17-MAY-2012 00:37:52

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.1[dC]



```

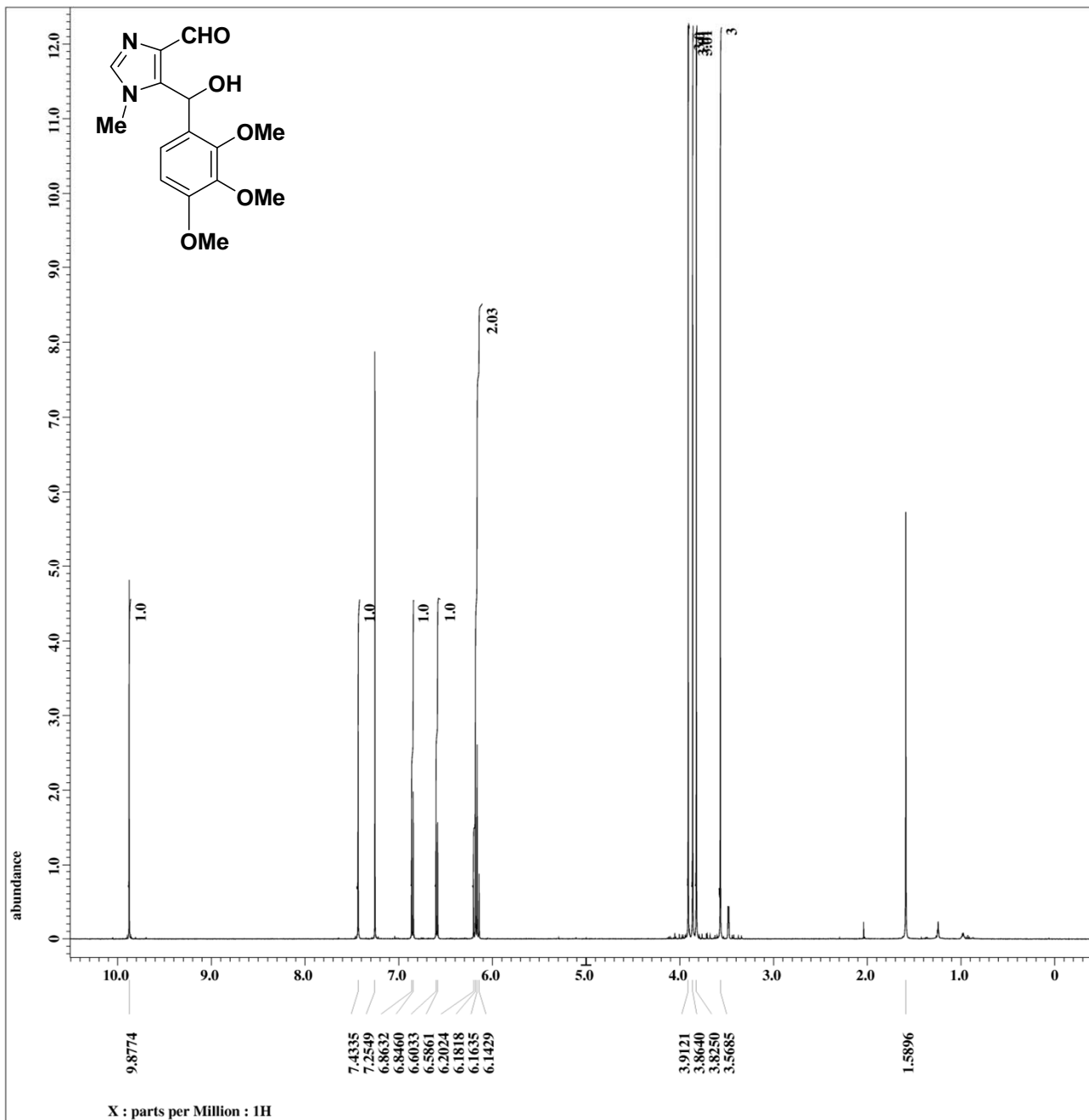
Filename      = JKD_I_MONO ALCOHOL-4.
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#629625
Solvent      = CHLOROFORM-D
Creation_time = 30-APR-2011 07:43:01
Revision_time = 17-MAY-2012 01:18:54
Current_time  = 17-MAY-2012 01:20:05

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768 [MHz]
X_offset      = 100 [ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.19959034 [Hz]
X_sweep       = 39.3081761 [kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521 [MHz]
Irr_offset    = 5.0 [ppm]
Clipped       = FALSE
Mod_return    = 10
Scans         = 200
Total_scans   = 200

X_90_width   = 10.73 [us]
X_acq_time   = 0.83361792 [s]
X_angle      = 30 [deg]
X_atn        = 9 [dB]
X_pulse      = 3.57666667 [us]
Irr_atn_dec  = 20 [dB]
Irr_atn_noe  = 20 [dB]
Irr_noise    = WALTZ
Decoupling   = TRUE
Initial_wait = 1 [s]
Noe          = TRUE
Noe_time     = 2 [s]
Recvr_gain   = 50
Relaxation_delay = 2 [s]
Repetition_time = 2.83361792 [s]
Temp_get     = 21.7 [dC]

```



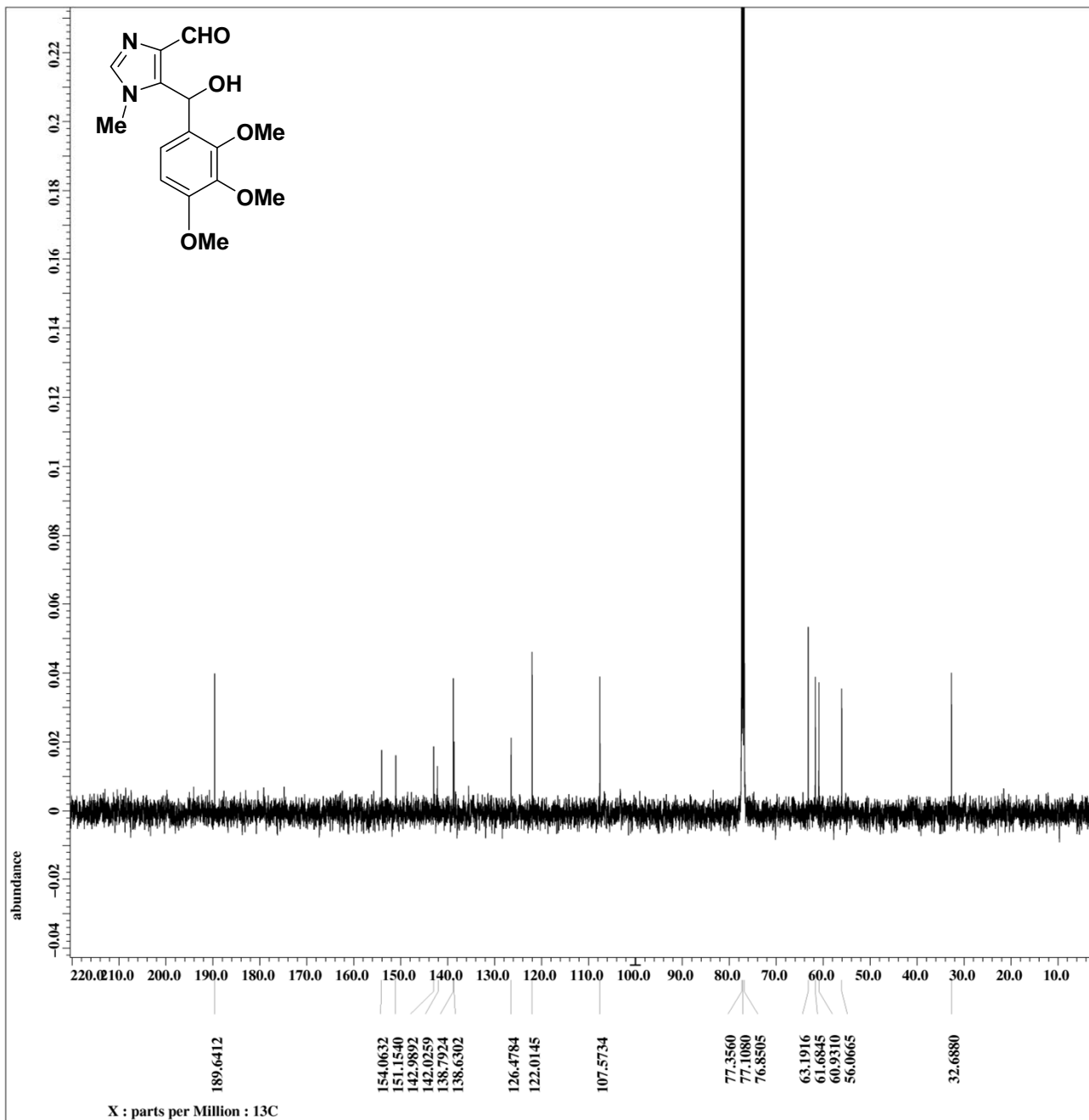
Filename = JKD\_I\_59-MONO\_ALDEHYD  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#690859  
 Solvent = CHLOROFORM-D  
 Creation\_time = 4-MAY-2011 09:18:19  
 Revision\_time = 17-MAY-2012 01:01:57  
 Current\_time = 17-MAY-2012 01:02:32

Comment = single\_pulse  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.3[dC]



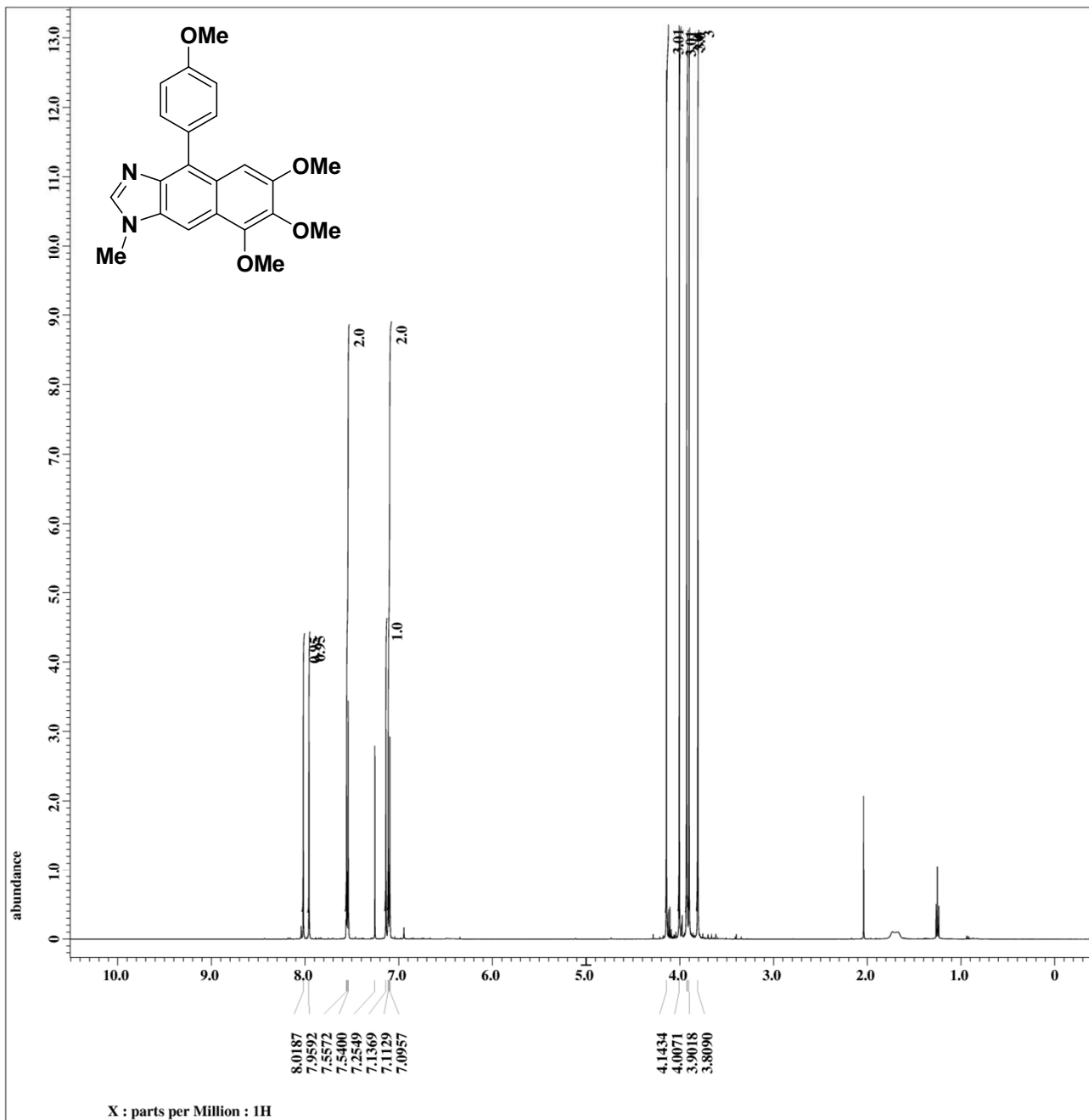


Filename = JKD\_I\_59\_MONO\_ALDEHYD  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#693148  
 Solvent = CHLOROFORM-D  
 Creation\_time = 4-MAY-2011 09:42:34  
 Revision\_time = 17-MAY-2012 01:22:23  
 Current\_time = 17-MAY-2012 01:27:14

Comment = single pulse decouple  
 Data\_format = 1D COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768 [MHz]  
 X\_offset = 100 [ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034 [Hz]  
 X\_sweep = 39.3081761 [kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521 [MHz]  
 Irr\_offset = 5.0 [ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 488  
 Total\_scans = 488

X\_90\_width = 10.73 [us]  
 X\_acq\_time = 0.83361792 [s]  
 X\_angle = 30 [deg]  
 X\_atn = 9 [dB]  
 X\_pulse = 3.57666667 [us]  
 Irr\_atn\_dec = 20 [dB]  
 Irr\_atn\_noe = 20 [dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1 [s]  
 Noe = TRUE  
 Noe\_time = 2 [s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2 [s]  
 Repetition\_time = 2.83361792 [s]  
 Temp\_get = 21.8 [dC]

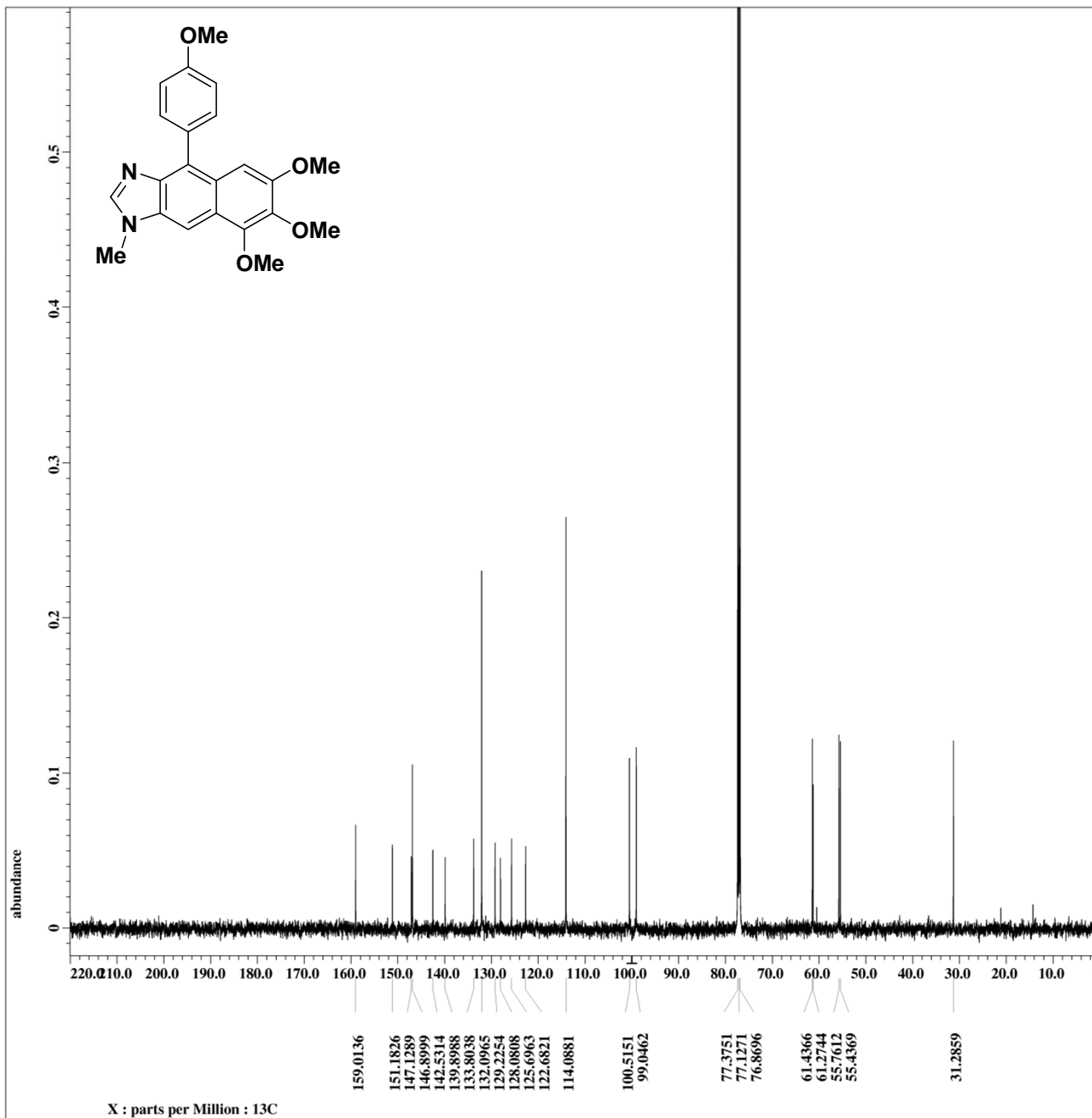


Filename = JKD\_I\_61\_CYCLIC\_KC-4.  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#508278  
 Solvent = CHLOROFORM-D  
 Creation\_time = 16-MAY-2011 04:14:33  
 Revision\_time = 16-MAY-2012 03:04:45  
 Current\_time = 16-MAY-2012 03:05:23

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 42  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.2[dC]

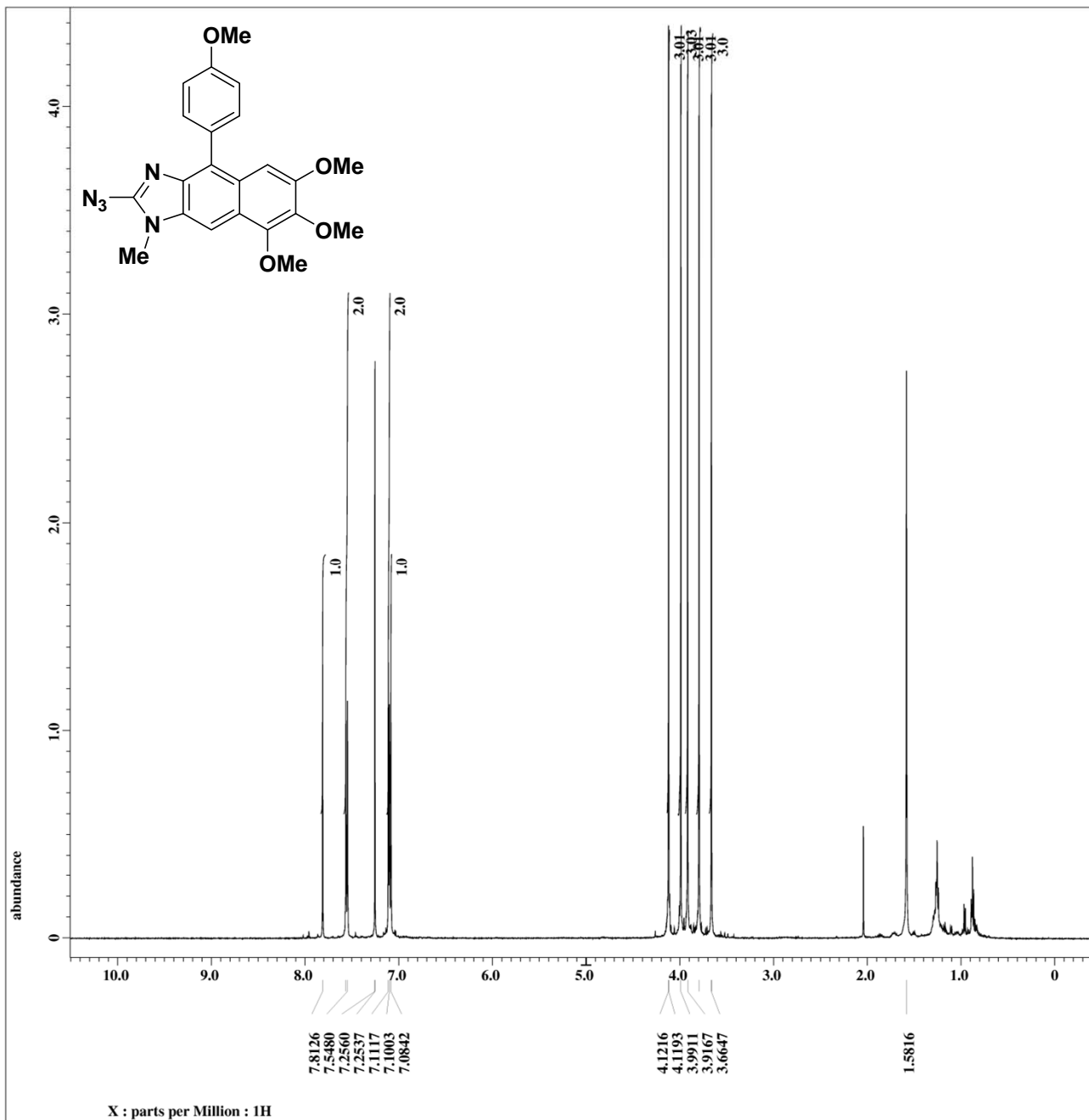


Filename = JKD\_I\_61\_CYCLIC\_KC-3.  
 Author = delta  
 Experiment = single\_pulse\_dec  
 Sample\_id = S#511308  
 Solvent = CHLOROFORM-D  
 Creation\_time = 16-MAY-2011 04:34:28  
 Revision\_time = 17-MAY-2012 01:28:00  
 Current\_time = 17-MAY-2012 01:29:15

Comment = single pulse decouple  
 Data\_format = 1D\_COMPLEX  
 Dim\_size = 26214  
 Dim\_title = 13C  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 0.83361792[s]  
 X\_domain = 13C  
 X\_freq = 125.76529768[MHz]  
 X\_offset = 100[ppm]  
 X\_points = 32768  
 X\_prescans = 4  
 X\_resolution = 1.19959034[Hz]  
 X\_sweep = 39.3081761[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 10  
 Scans = 380  
 Total\_scans = 380

X\_90\_width = 10.73[us]  
 X\_acq\_time = 0.83361792[s]  
 X\_angle = 30[deg]  
 X\_atn = 9[dB]  
 X\_pulse = 3.57666667[us]  
 Irr\_atn\_dec = 20[dB]  
 Irr\_atn\_noe = 20[dB]  
 Irr\_noise = WALTZ  
 Decoupling = TRUE  
 Initial\_wait = 1[s]  
 Noe = TRUE  
 Noe\_time = 2[s]  
 Recvr\_gain = 50  
 Relaxation\_delay = 2[s]  
 Repetition\_time = 2.83361792[s]  
 Temp\_get = 21.7[dC]

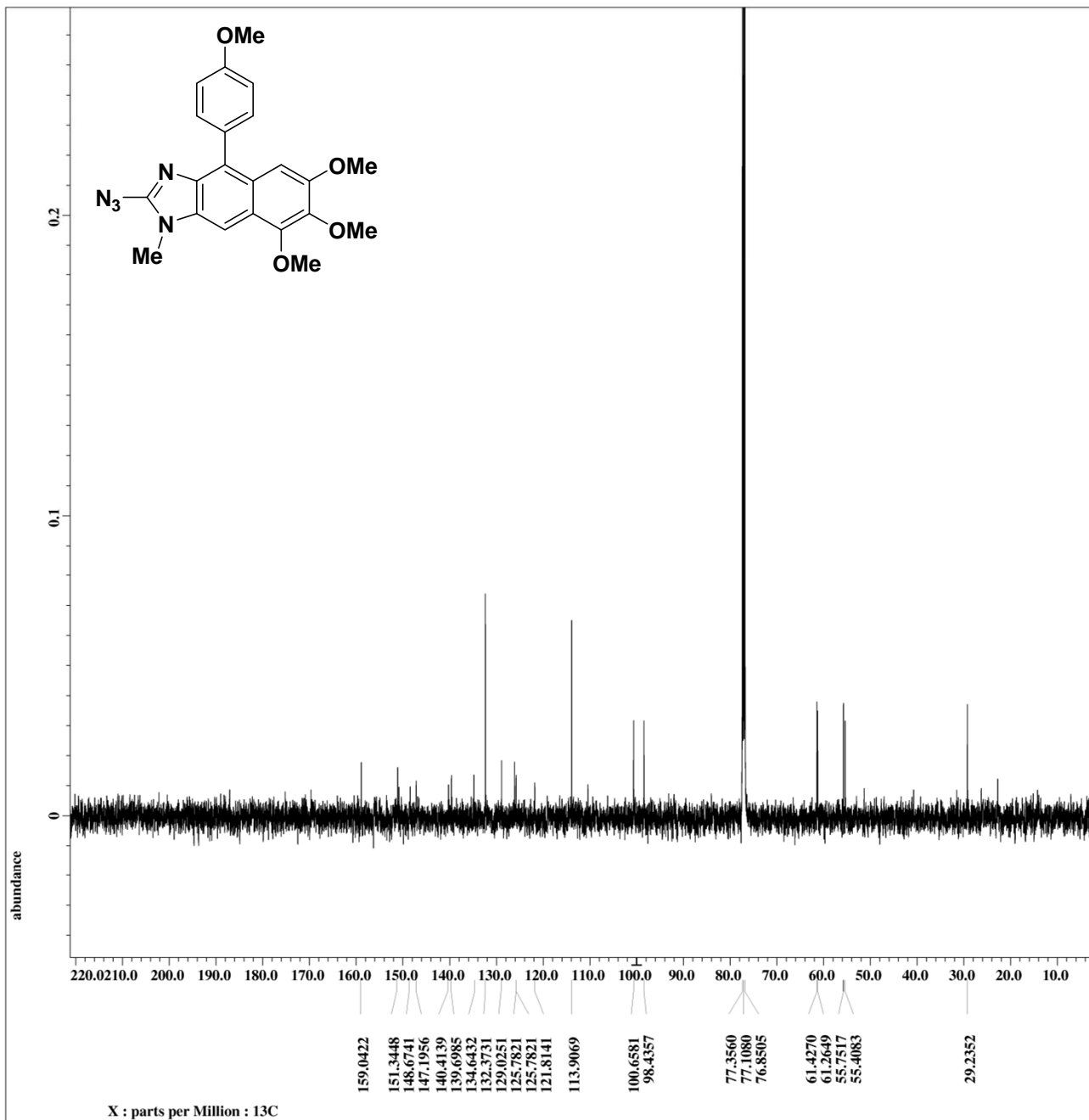


Filename = JKD\_I\_62\_AZIDE\_KC-4.j  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id =  
 Solvent = CHLOROFORM-D  
 Creation\_time = 18-MAY-2011 10:25:07  
 Revision\_time = 17-MAY-2012 00:49:55  
 Current\_time = 17-MAY-2012 00:50:35

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579[T] (500[MH  
 X\_acq\_duration = 1.74587904[s]  
 X\_domain = 1H  
 X\_freq = 500.15991521[MHz]  
 X\_offset = 5.0[ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737[Hz]  
 X\_sweep = 9.38438438[kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521[MHz]  
 Irr\_offset = 5.0[ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521[MHz]  
 Tri\_offset = 5.0[ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54[us]  
 X\_acq\_time = 1.74587904[s]  
 X\_angle = 45[deg]  
 X\_atn = 4[dB]  
 X\_pulse = 6.27[us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1[s]  
 Recvr\_gain = 48  
 Relaxation\_delay = 5[s]  
 Repetition\_time = 6.74587904[s]  
 Temp\_get = 21.4[dC]



```

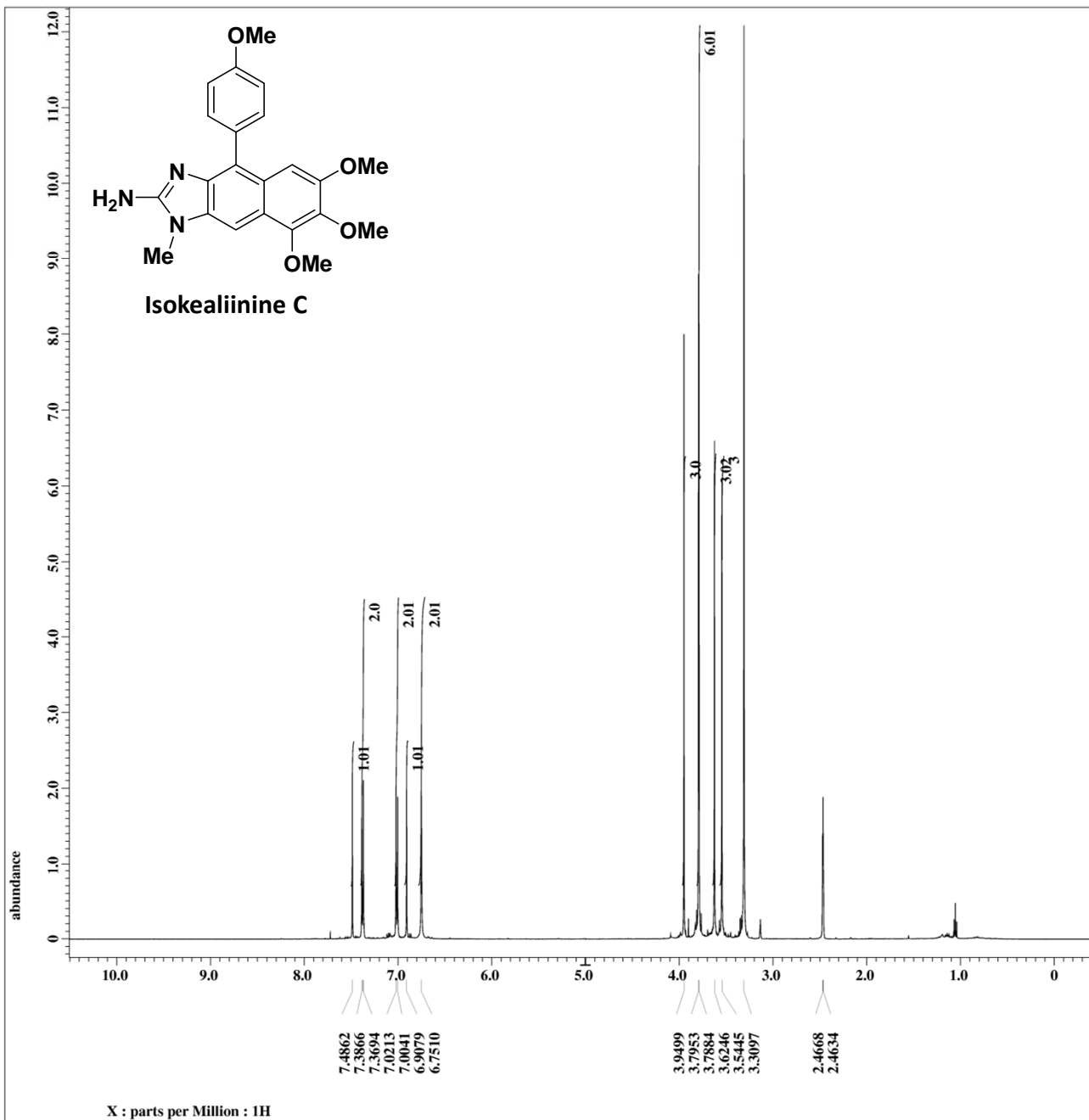
Filename      = JKD_I_62_AZIDE_KC-4.j
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#733107
Solvent      = CHLOROFORM-D
Creation_time = 18-MAY-2011 10:40:22
Revision_time = 17-MAY-2012 01:35:45
Current_time = 17-MAY-2012 01:36:40

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 300
Total_scans    = 300

X_90_width    = 10.73[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.57666667[us]
Irr_atn_dec   = 20[dB]
Irr_atn_noe   = 20[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 2[s]
Recvr_gain    = 50
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get      = 21.9[dC]

```

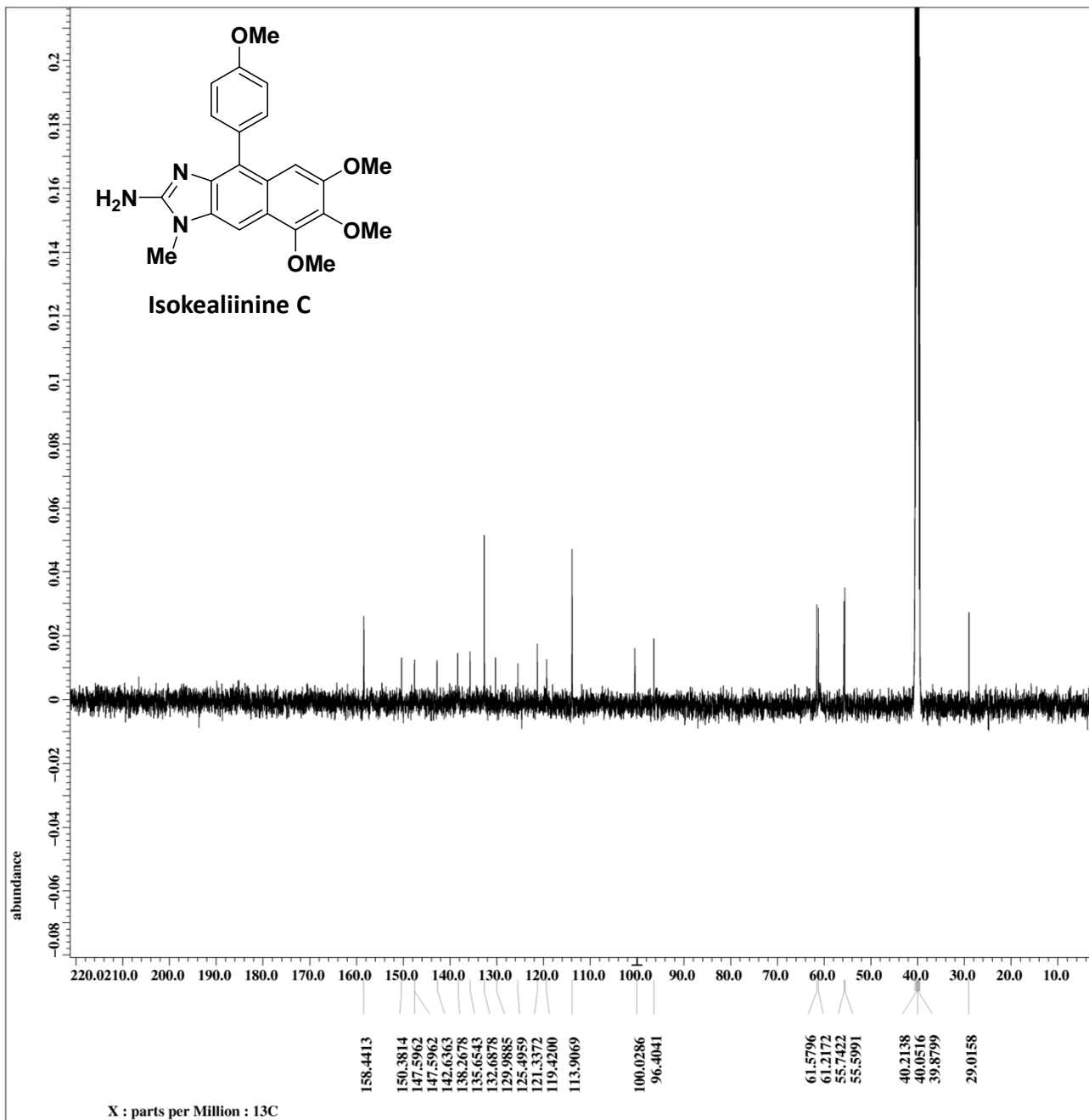


Filename = JKD\_I\_63\_KC\_FINAL\_CON  
 Author = delta  
 Experiment = single\_pulse.ex2  
 Sample\_id = S#669023  
 Solvent = DMSO-D6  
 Creation\_time = 23-MAY-2011 08:42:46  
 Revision\_time = 17-MAY-2012 01:10:41  
 Current\_time = 17-MAY-2012 01:11:33

Comment = single\_pulse  
 Data\_format = 1D COMPLEX  
 Dim\_size = 13107  
 Dim\_title = 1H  
 Dim\_units = [ppm]  
 Dimensions = X  
 Site = ECA 500  
 Spectrometer = JNM-ECA500

Field\_strength = 11.7473579 [T] (500 [MH  
 X\_acq\_duration = 1.74587904 [s]  
 X\_domain = 1H  
 X\_freq = 500.15991521 [MHz]  
 X\_offset = 5.0 [ppm]  
 X\_points = 16384  
 X\_prescans = 0  
 X\_resolution = 0.57277737 [Hz]  
 X\_sweep = 9.38438438 [kHz]  
 Irr\_domain = 1H  
 Irr\_freq = 500.15991521 [MHz]  
 Irr\_offset = 5.0 [ppm]  
 Tri\_domain = 1H  
 Tri\_freq = 500.15991521 [MHz]  
 Tri\_offset = 5.0 [ppm]  
 Clipped = FALSE  
 Mod\_return = 1  
 Scans = 24  
 Total\_scans = 24

X\_90\_width = 12.54 [us]  
 X\_acq\_time = 1.74587904 [s]  
 X\_angle = 45 [deg]  
 X\_atn = 4 [dB]  
 X\_pulse = 6.27 [us]  
 Irr\_mode = Off  
 Tri\_mode = Off  
 Dante\_presat = FALSE  
 Initial\_wait = 1 [s]  
 Recvr\_gain = 44  
 Relaxation\_delay = 5 [s]  
 Repetition\_time = 6.74587904 [s]  
 Temp\_get = 21.1 [dC]



```

Filename      = JKD_I_63_KC_FINAL-4.j
Author       = delta
Experiment   = single_pulse_dec
Sample_id    = S#519751
Solvent      = DMSO-D6
Creation_time = 19-MAY-2011 04:49:31
Revision_time = 17-MAY-2012 01:32:45
Current_time  = 17-MAY-2012 01:33:46

Comment      = single pulse decouple
Data_format  = 1D COMPLEX
Dim_size     = 26214
Dim_title    = 13C
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = JNM-ECA500

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768 [MHz]
X_offset       = 100[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521 [MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 10
Scans          = 400
Total_scans    = 400

X_90_width    = 10.73[us]
X_acq_time     = 0.83361792[s]
X_angle        = 30[deg]
X_atn          = 9[dB]
X_pulse        = 3.57666667[us]
Irr_atn_dec    = 20[dB]
Irr_atn_noe    = 20[dB]
Irr_noise      = WALTZ
Decoupling     = TRUE
Initial_wait   = 1[s]
Noe            = TRUE
Noe_time       = 2[s]
Recvr_gain     = 50
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get       = 21.6[dC]
  
```

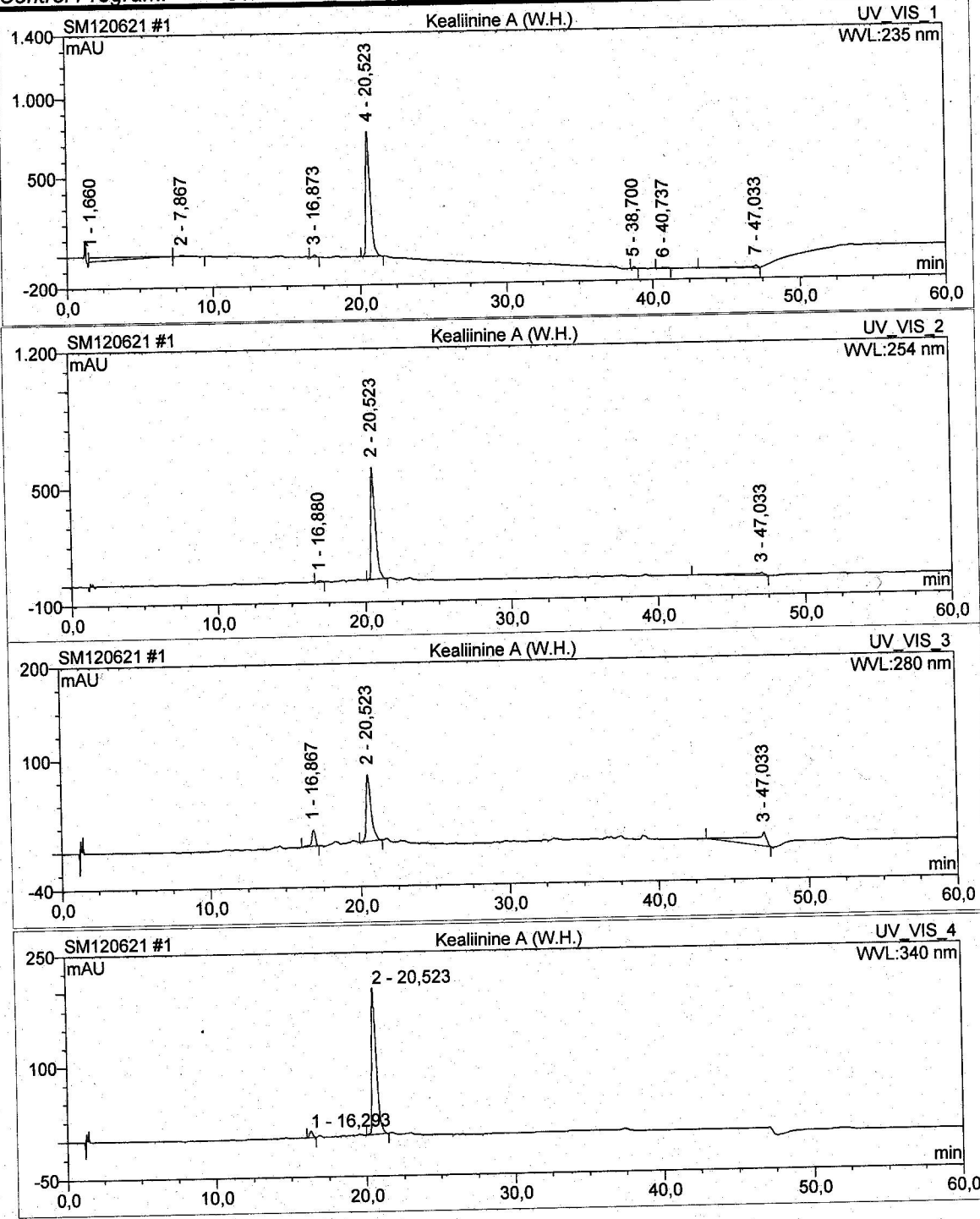
## HPLC Measurements

The HPLC plots were obtained by the laboratory of Prof. Peter Proksch at the Universität Düsseldorf.

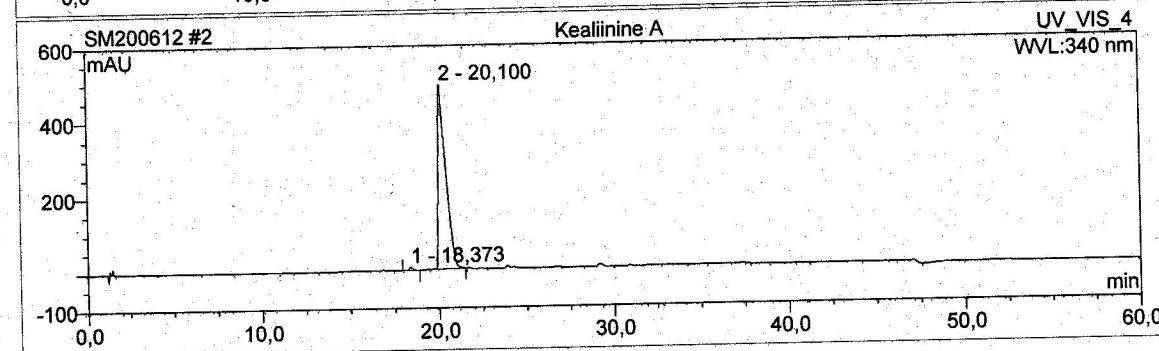
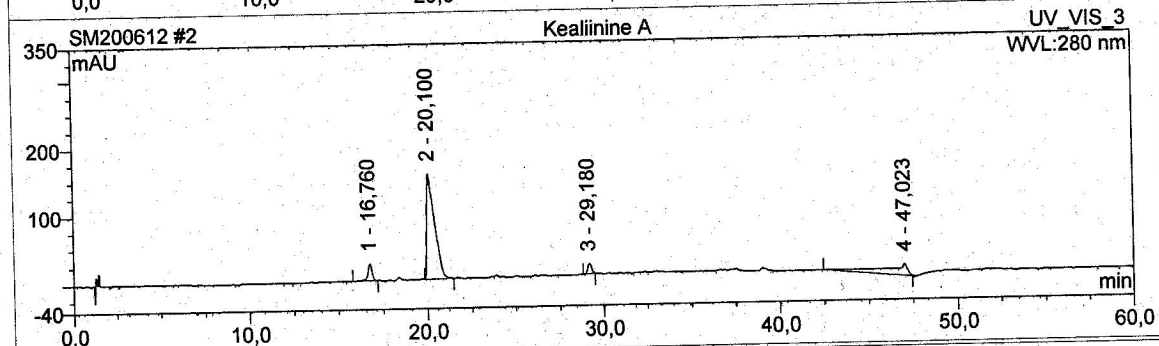
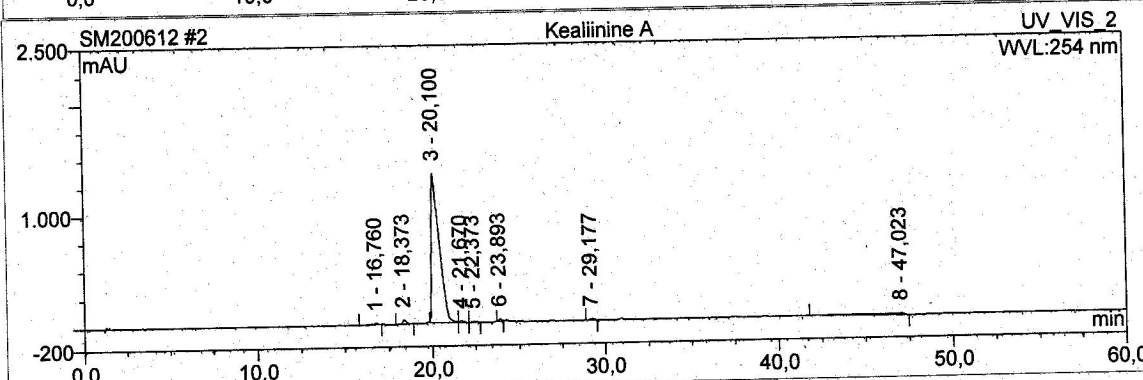
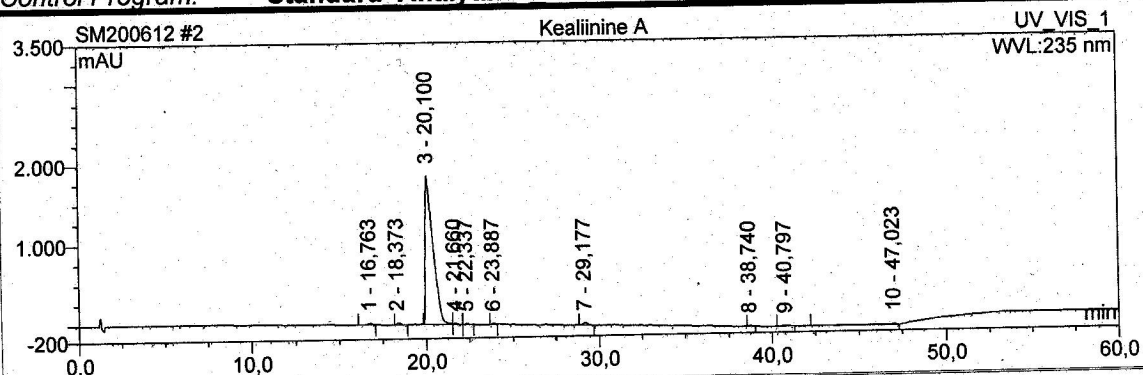
HPLC analysis was performed using a HPLC (Dionex P580) system coupled to a UV-Vis detector (UVD340S). Routine detection was at 235, 254, 280, and 340 nm. The separation column (125 × 4 mm, L × ID) was prefilled with Eurospher-10 C18 (Knauer, Germany) using a linear gradient of MeOH and 0.1 % HCOOH in H<sub>2</sub>O and a flow rate of 1 mL/min.



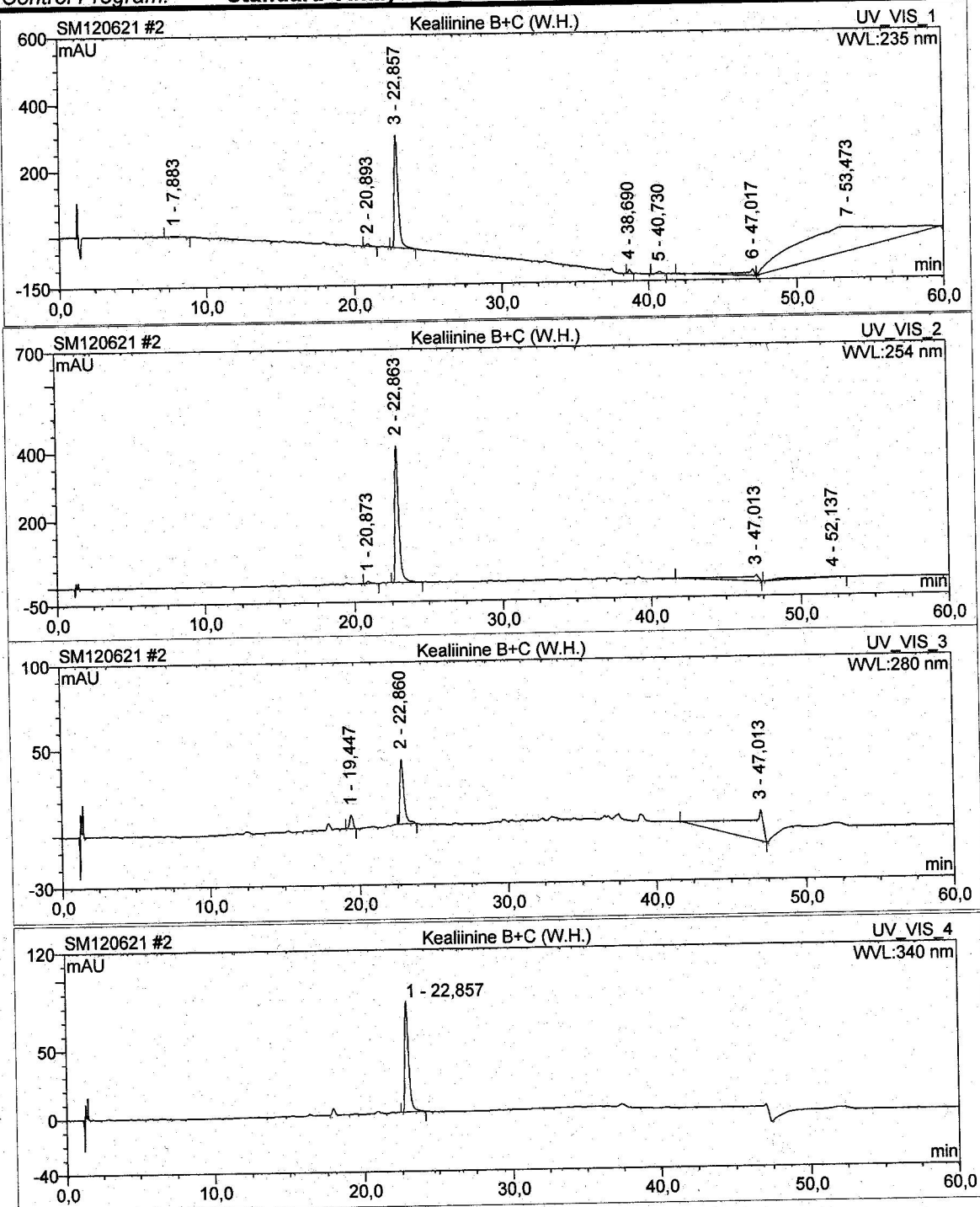
<b>1 Kealinine A (W.H.)</b> Natural kealinine A			
Sequence Name	SM120621	Injection Volume:	20,0
Vial Number:	RA9	Recording Time:	21.6.2012 10:59
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		



<b>2 Kealinine A</b> ✓ <i>USA</i> Synthetic kealinine A			
Sequence Name	SM200612	Injection Volume:	20,0
Vial Number:	RA1	Recording Time:	21.6.2012 6:55
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		

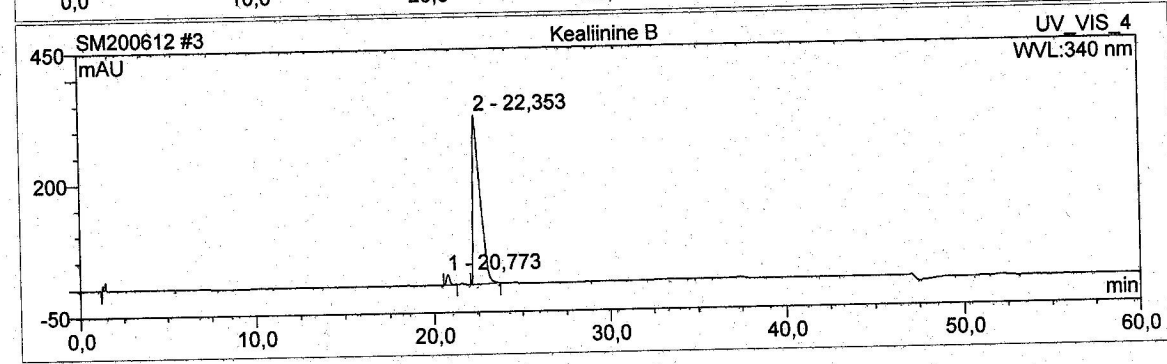
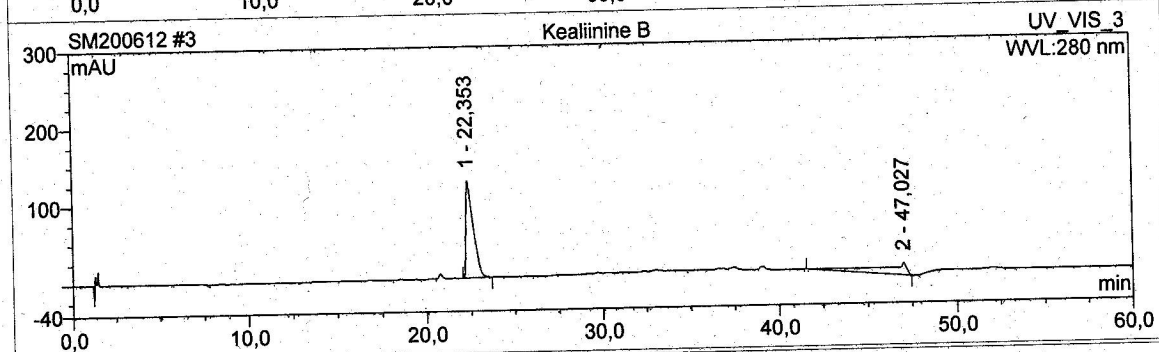
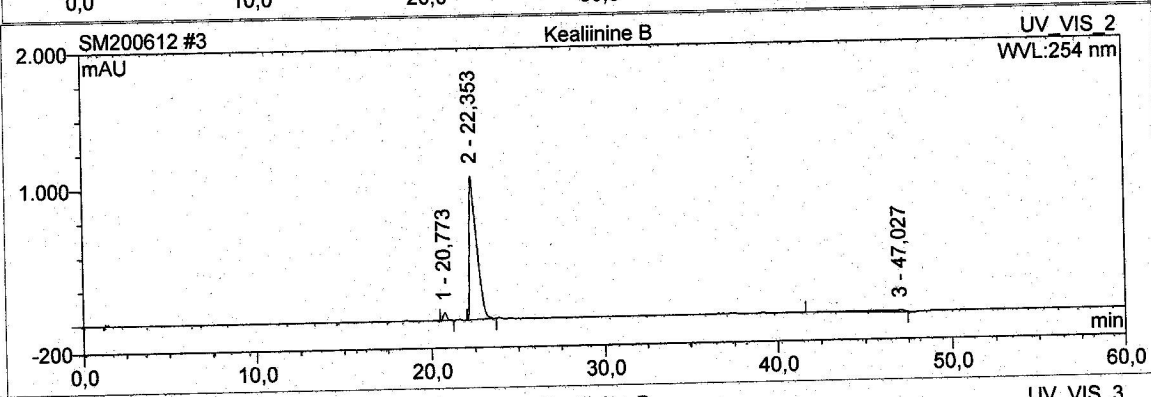
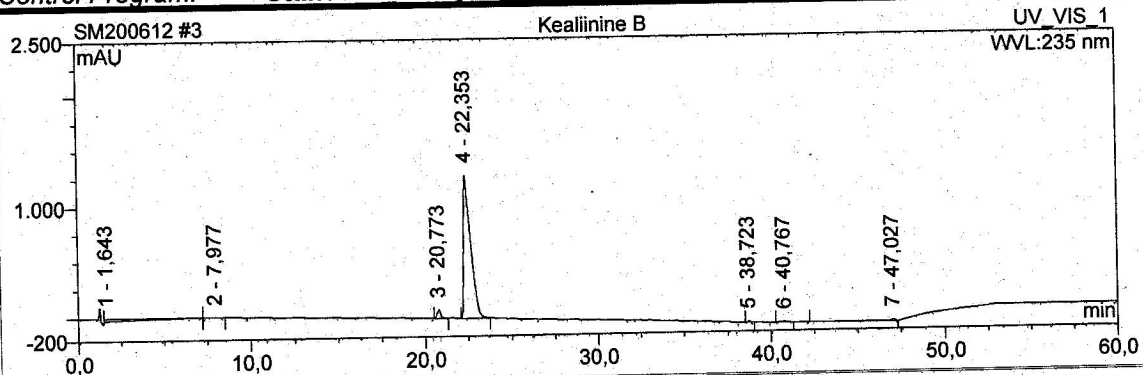


<b>2 Kealiinine B+C (W.H.)</b> Mixture of natural kealiinine B and C			
Sequence Name	SM120621	Injection Volume:	20,0
Vial Number:	RA10	Recording Time:	21.6.2012 11:59
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		



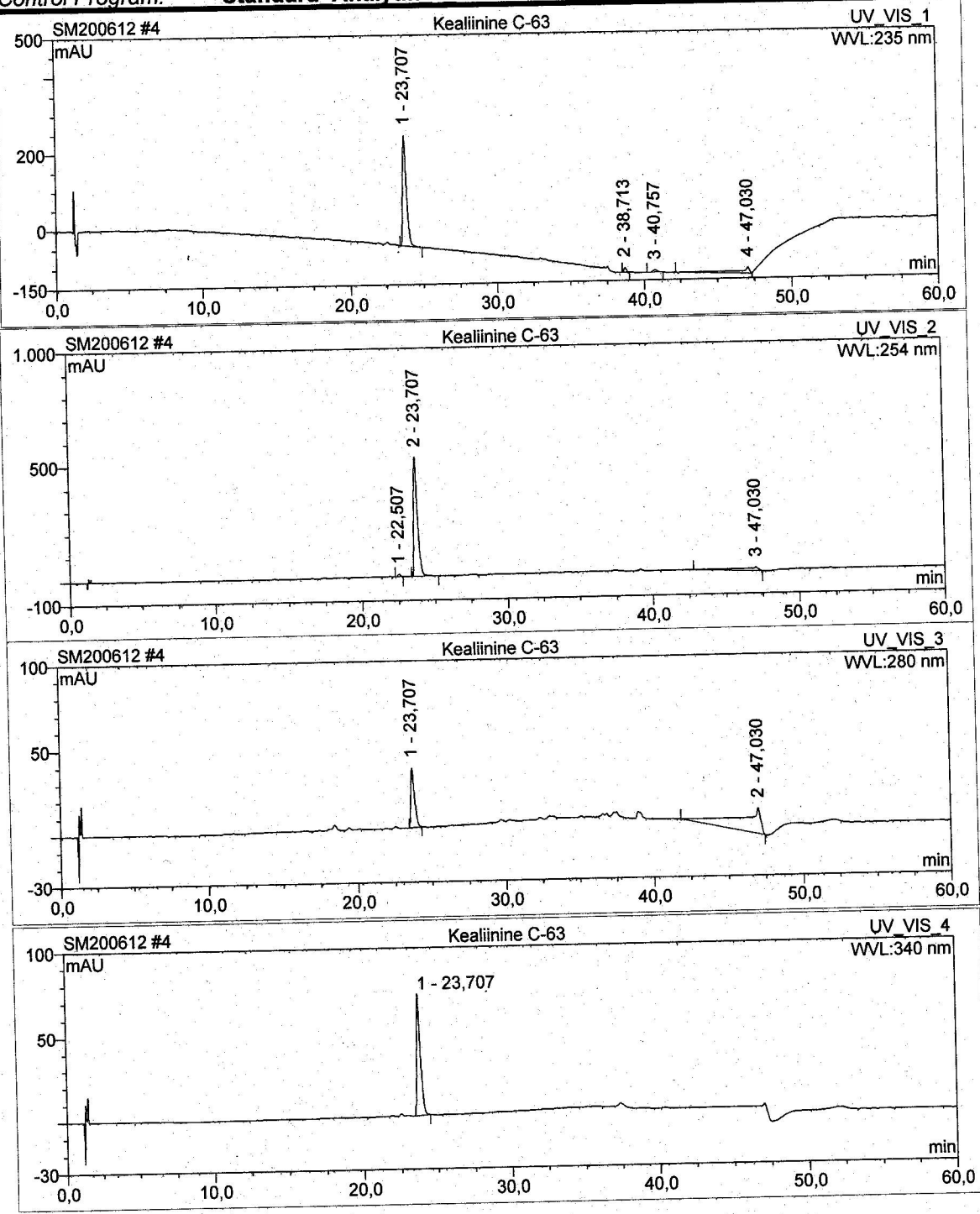
**3 Kealinine B** ✓ USA Synthetic kealinine B

Sequence Name	SM200612	Injection Volume:	20,0
Vial Number:	RA2	Recording Time:	21.6.2012 7:56
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		



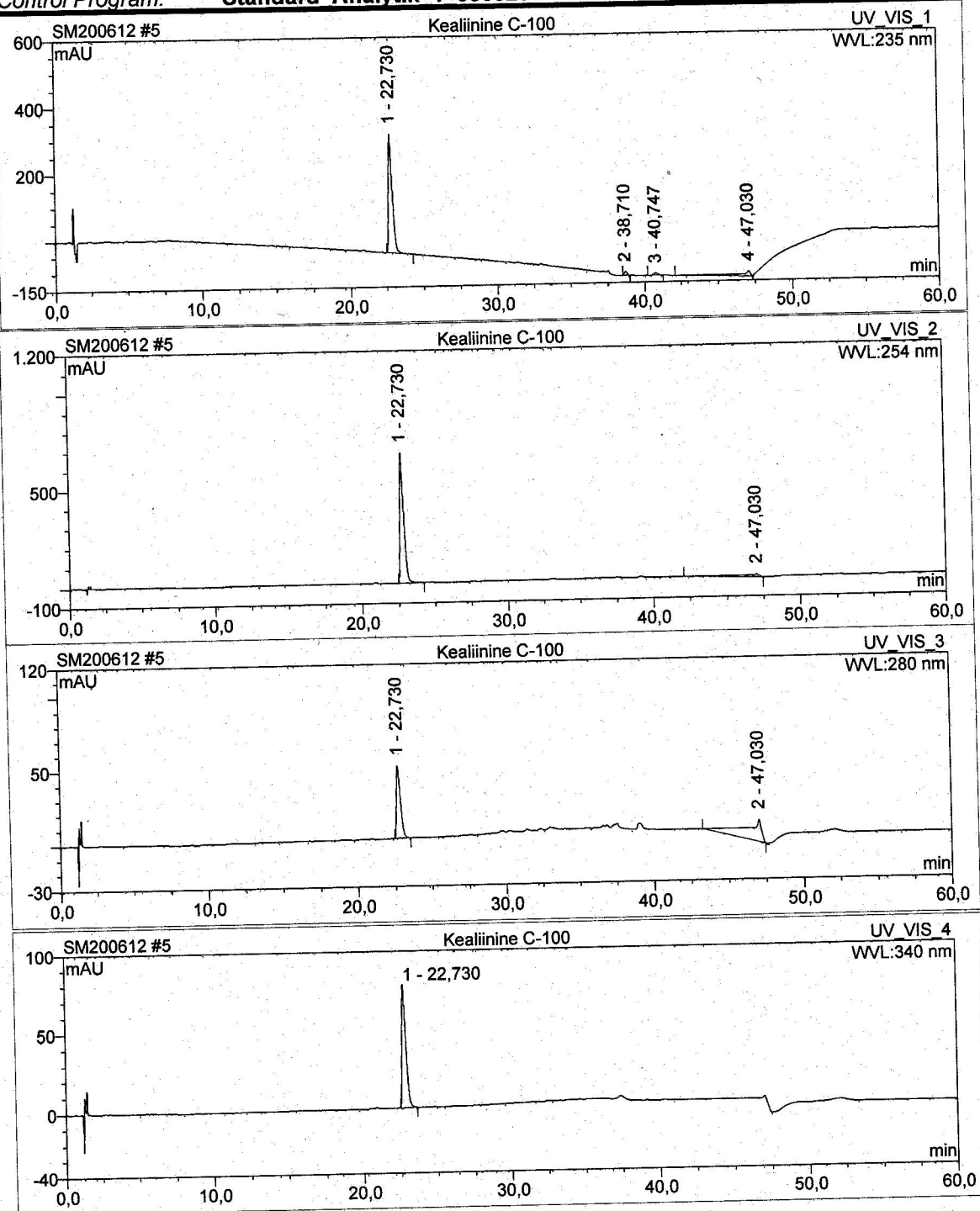
**4 Kealinine C-63 ? USA Isokealinine C**

Sequence Name	SM200612	Injection Volume:	20,0
Vial Number:	RA3	Recording Time:	21.6.2012 8:57
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		

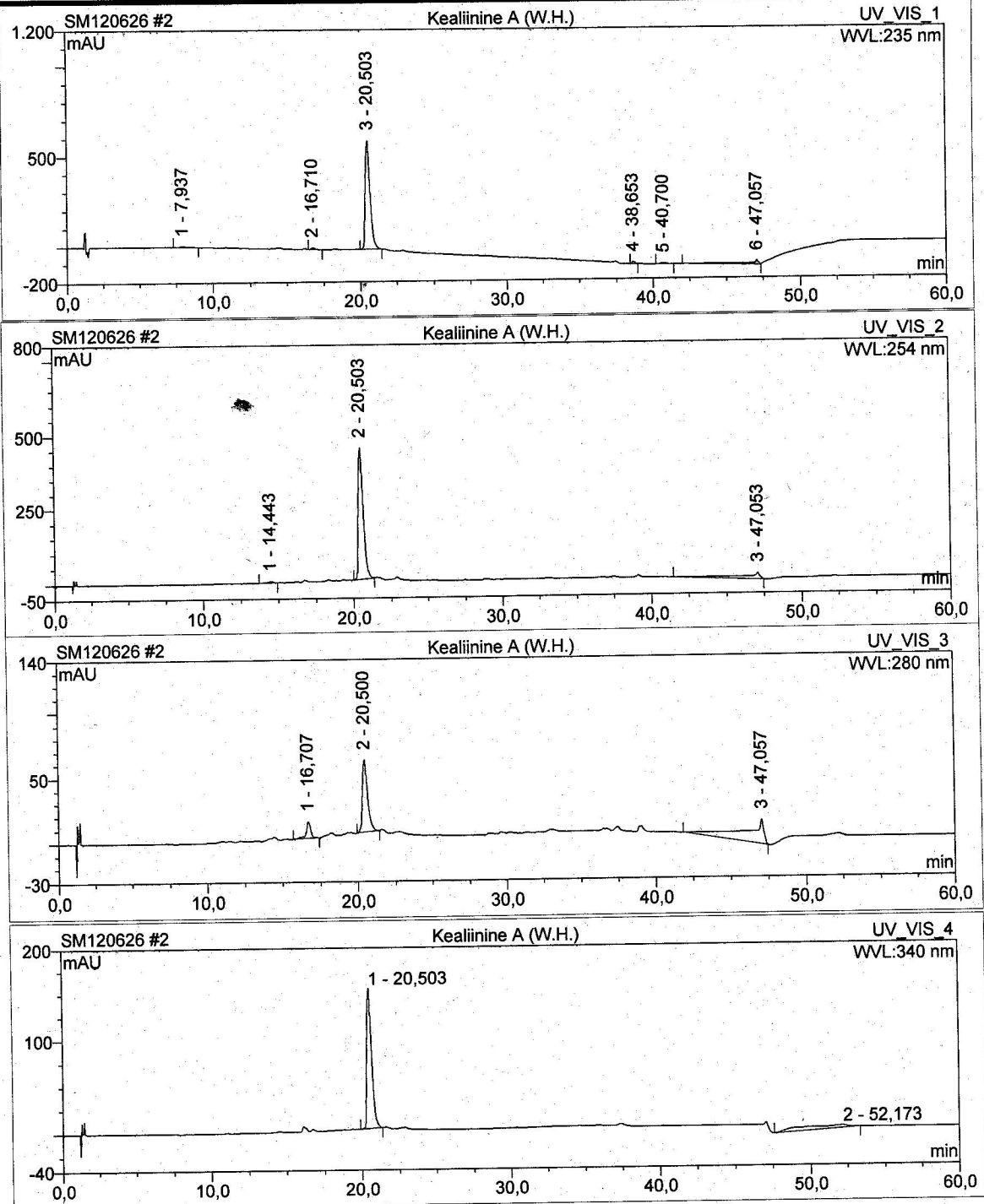


**5 Kealiinine C-100** ✓ **USA** Synthetic Kealiinine C

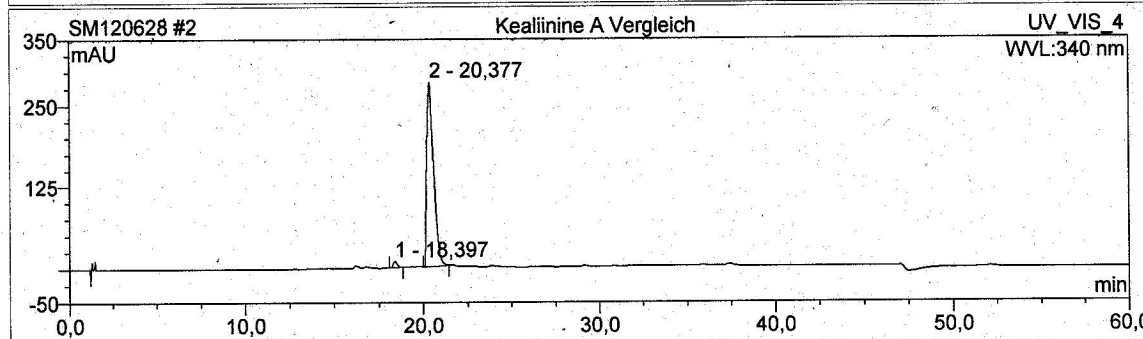
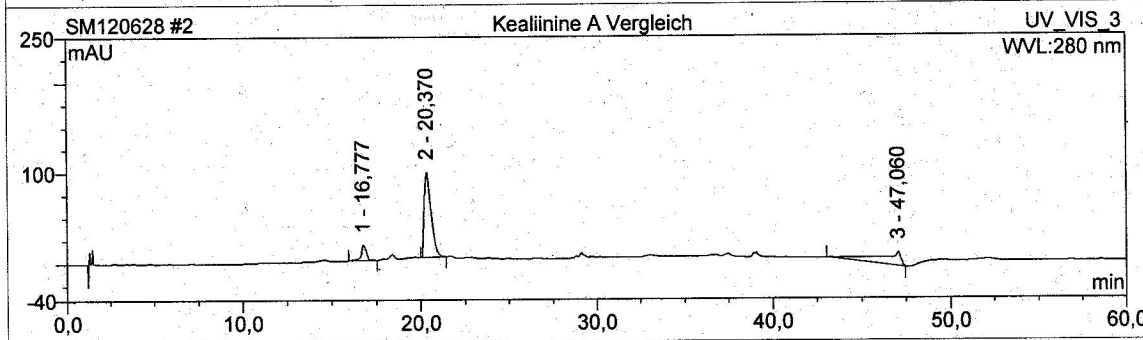
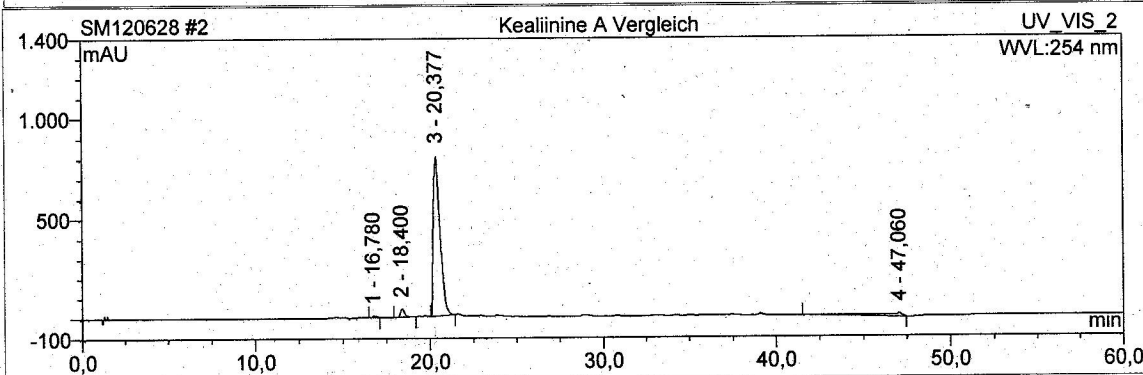
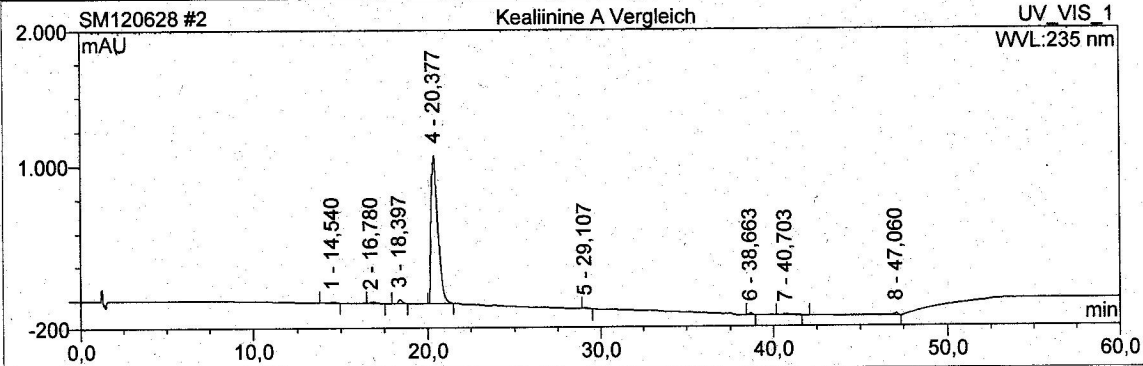
Sequence Name	SM200612	Injection Volume:	20,0
Vial Number:	RA4	Recording Time:	21.6.2012 9:58
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		



<b>2 Kealiinine A (W.H.)</b>		Natural kealiinine A	
Sequence Name	SM120626	Injection Volume:	20,0
Vial Number:	RB1	Recording Time:	27.6.2012 11:07
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		

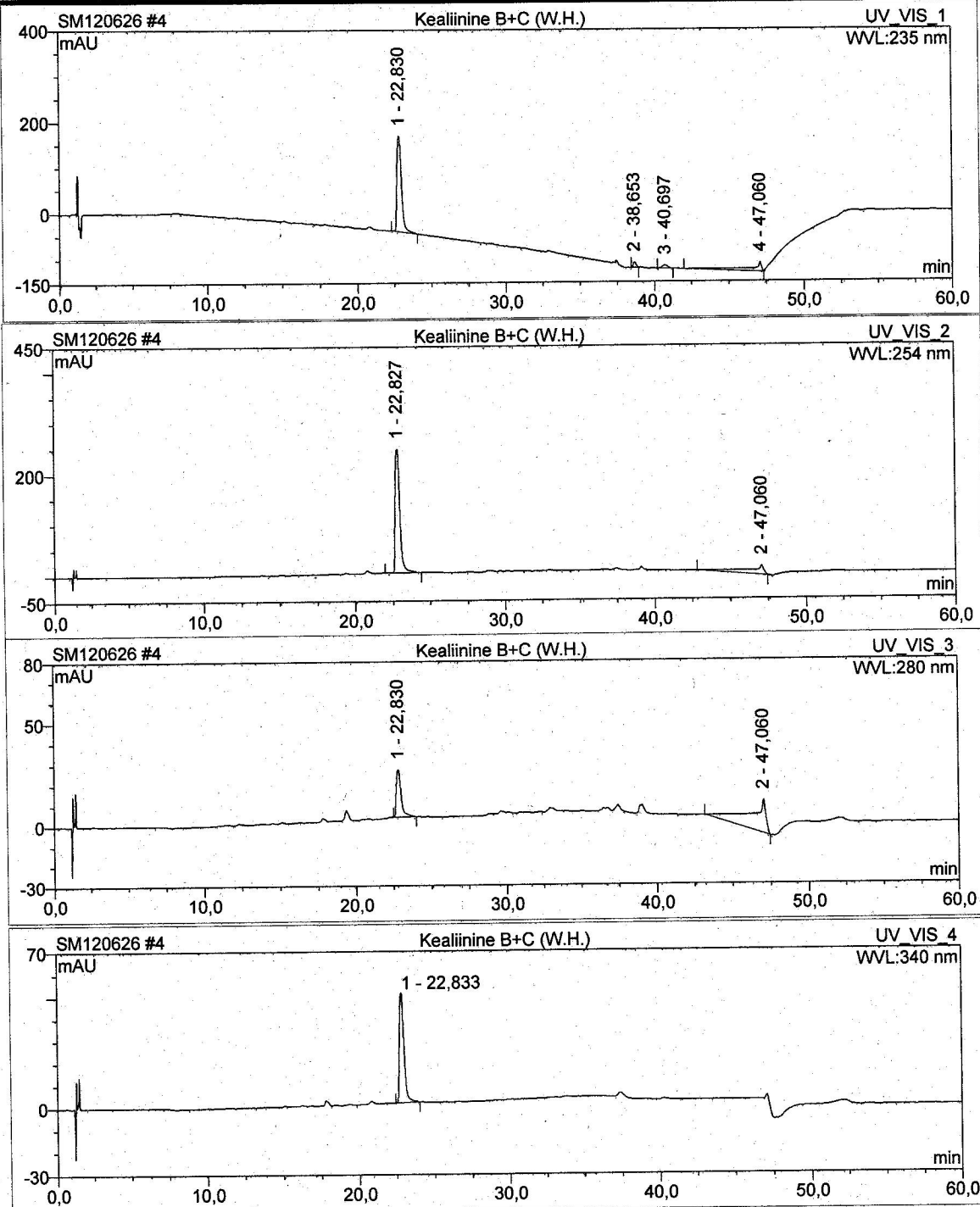


<b>2 Kealiinine A Vergleich</b>			
Co-injection of natural and synthetic kealiinine A			
Sequence Name	SM120628	Injection Volume:	20,0
Vial Number:	RB1	Recording Time:	28.6.2012 14:31
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		



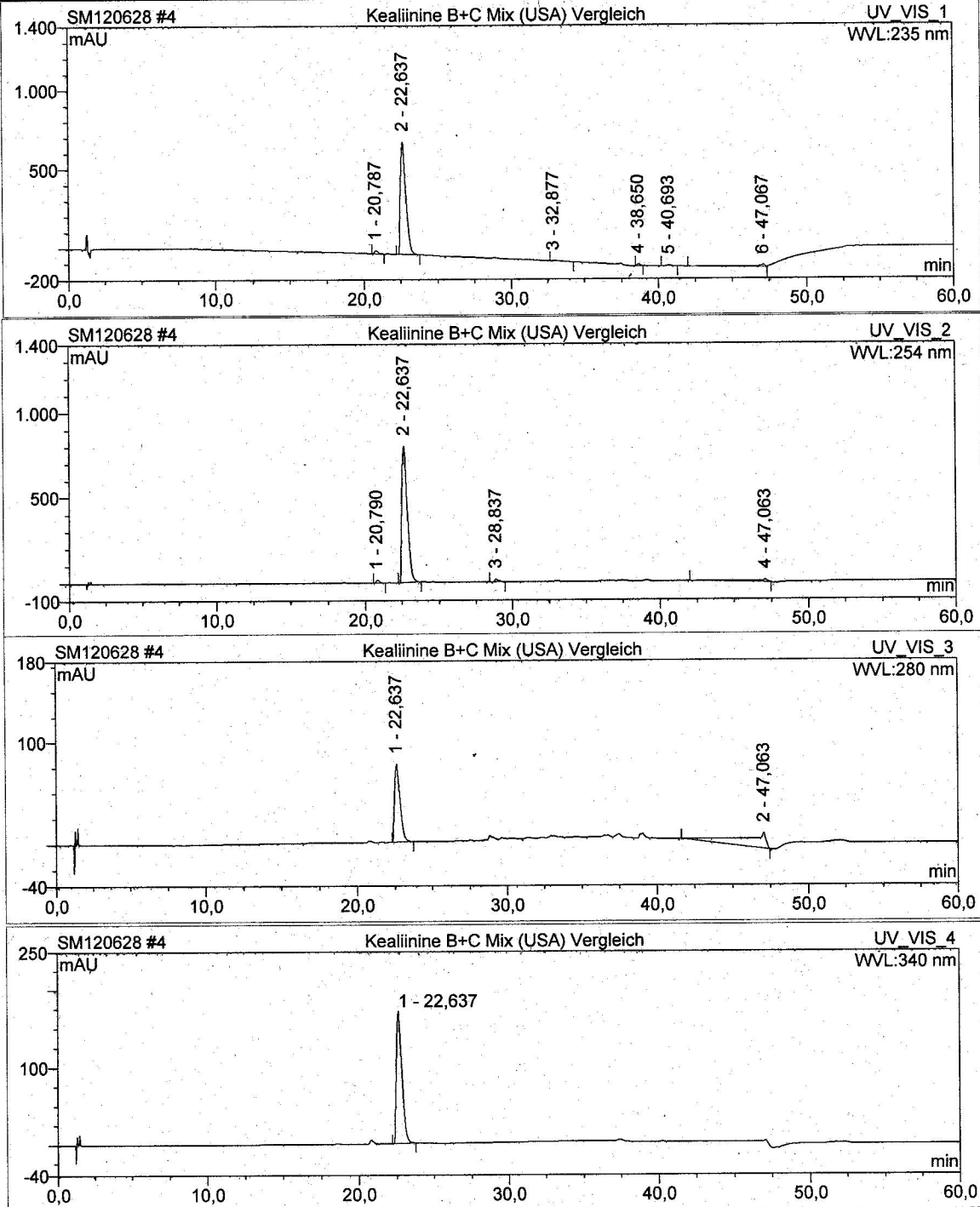


<b>4 Kealiinine B+C (W.H.)</b>			
Mixture of natural kealiinines B and C			
Sequence Name	SM120626	Injection Volume:	20,0
Vial Number:	RB3	Recording Time:	27.6.2012 13:09
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		

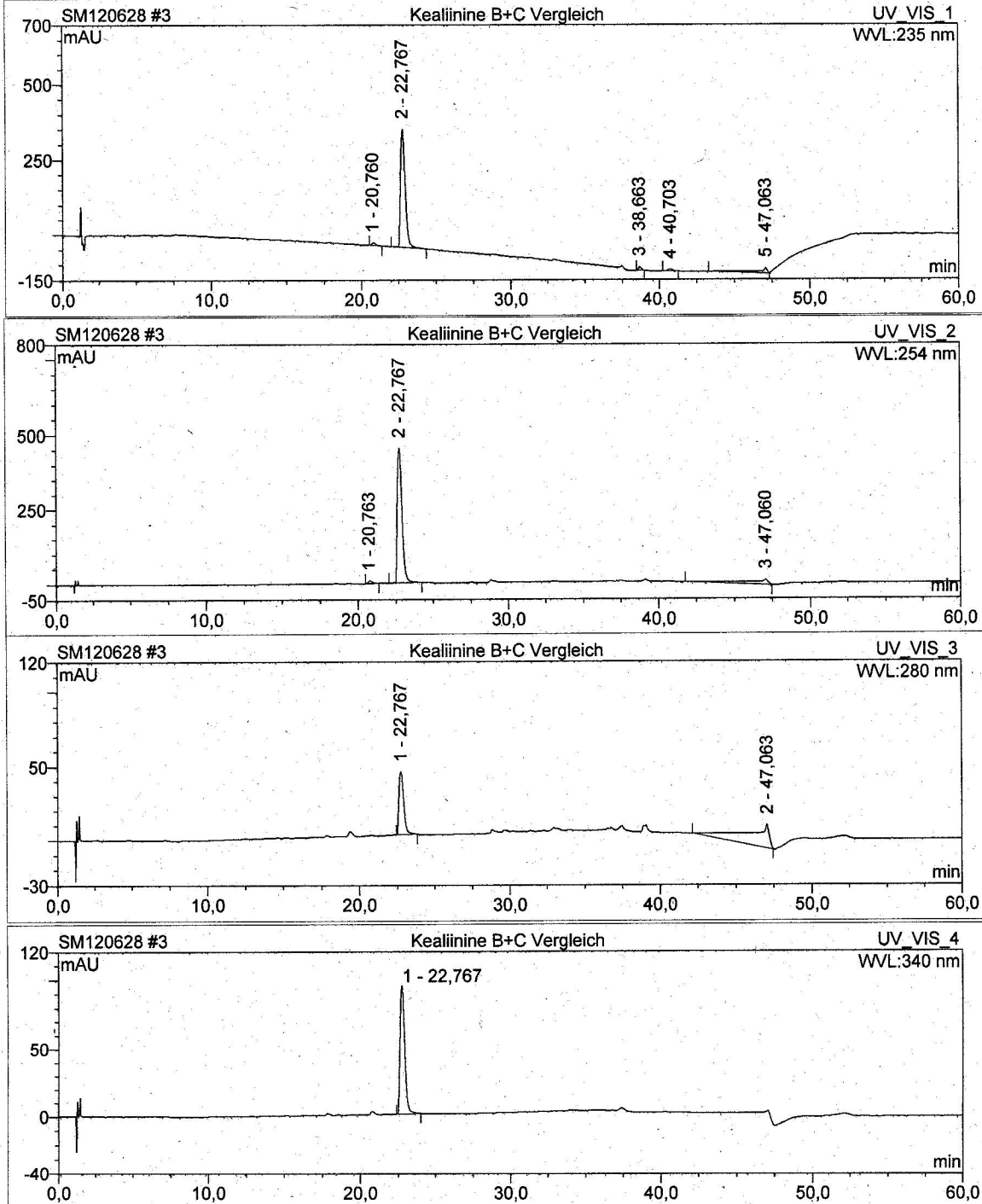


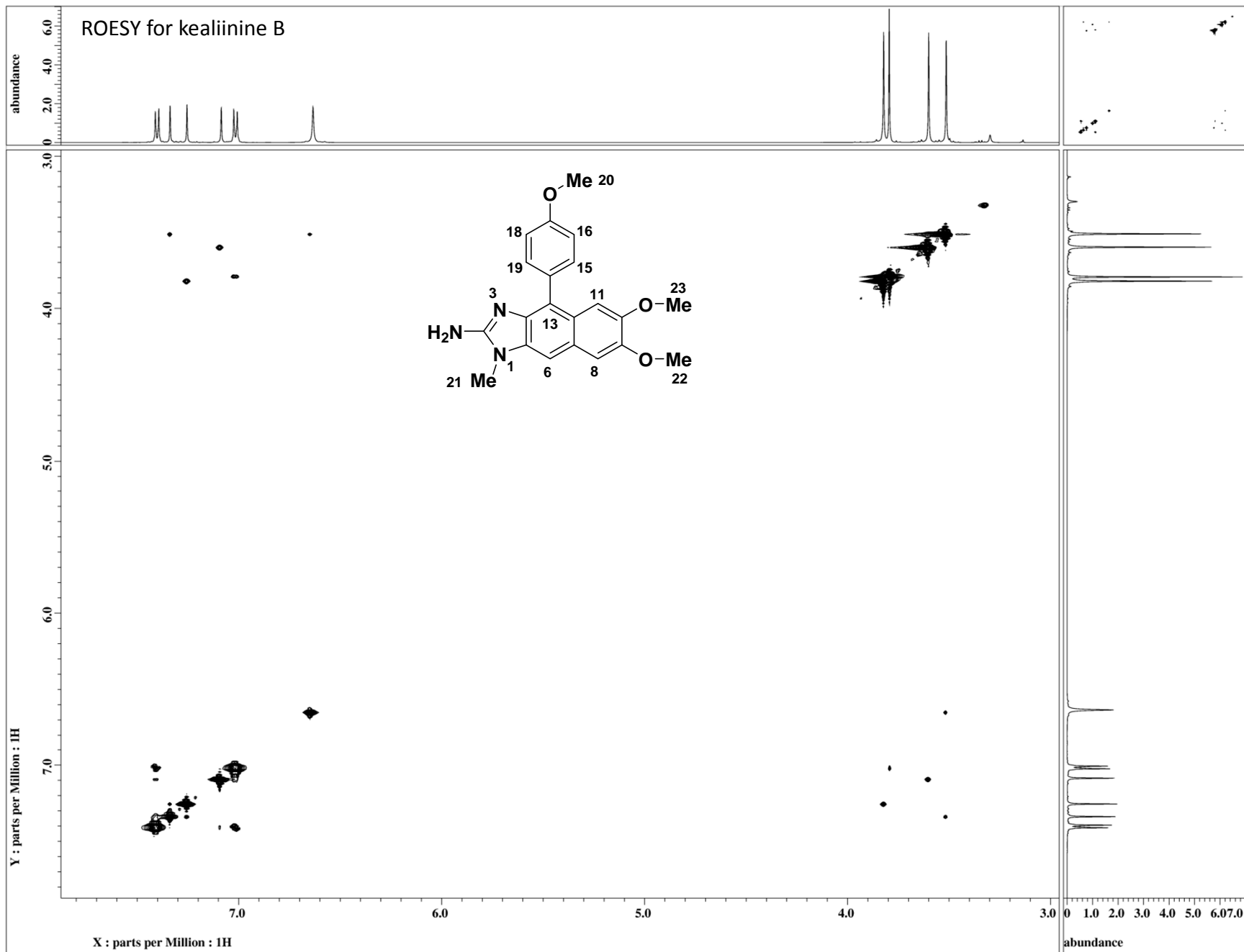
**4 Kealiinine B+C Mix (USA)**  
Co-injection of mixture of natural B and C with synthetic kealiinine B

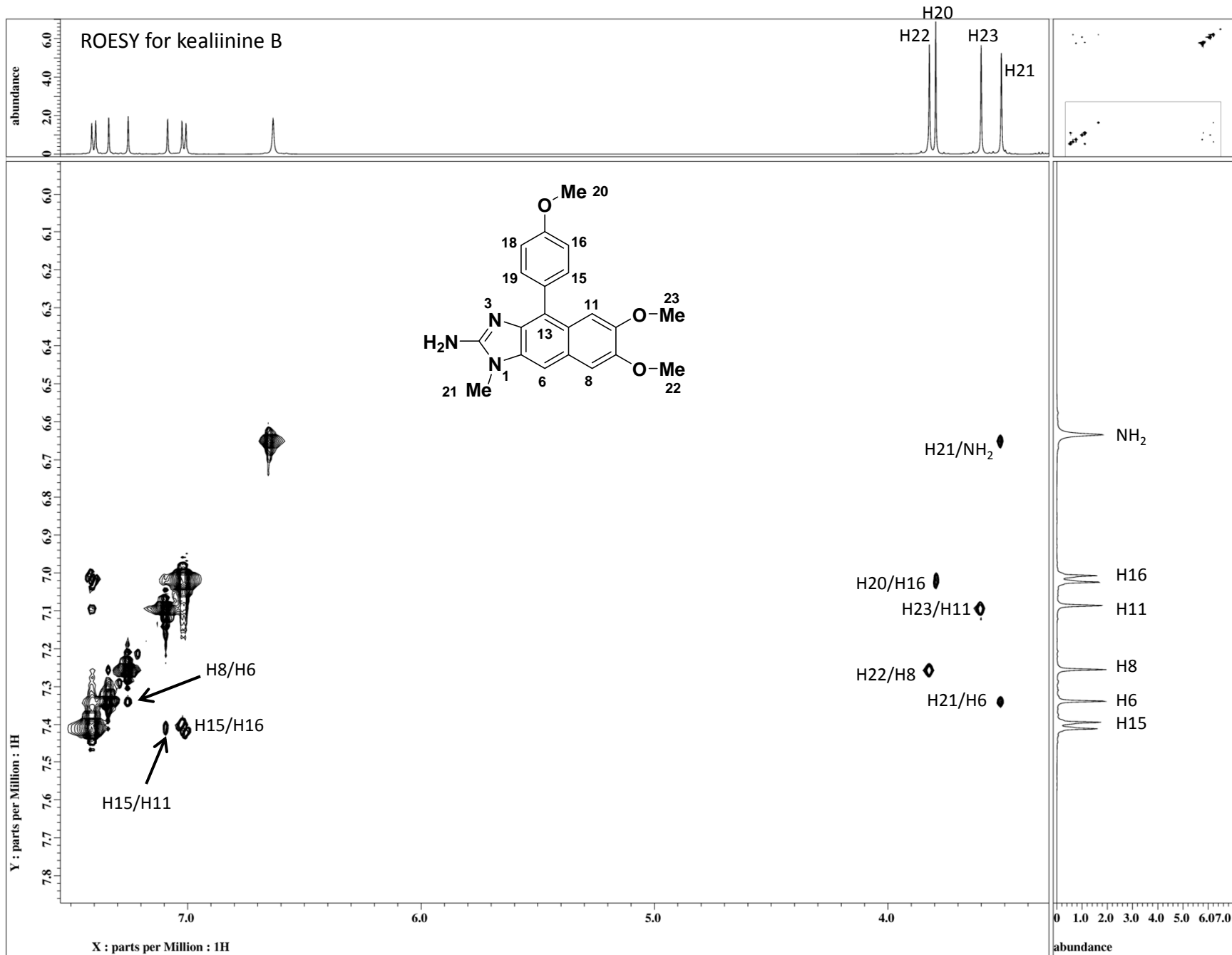
Sequence Name	SM120628	Injection Volume:	20,0
Vial Number:	RB3	Recording Time:	28.6.2012 16:33
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		

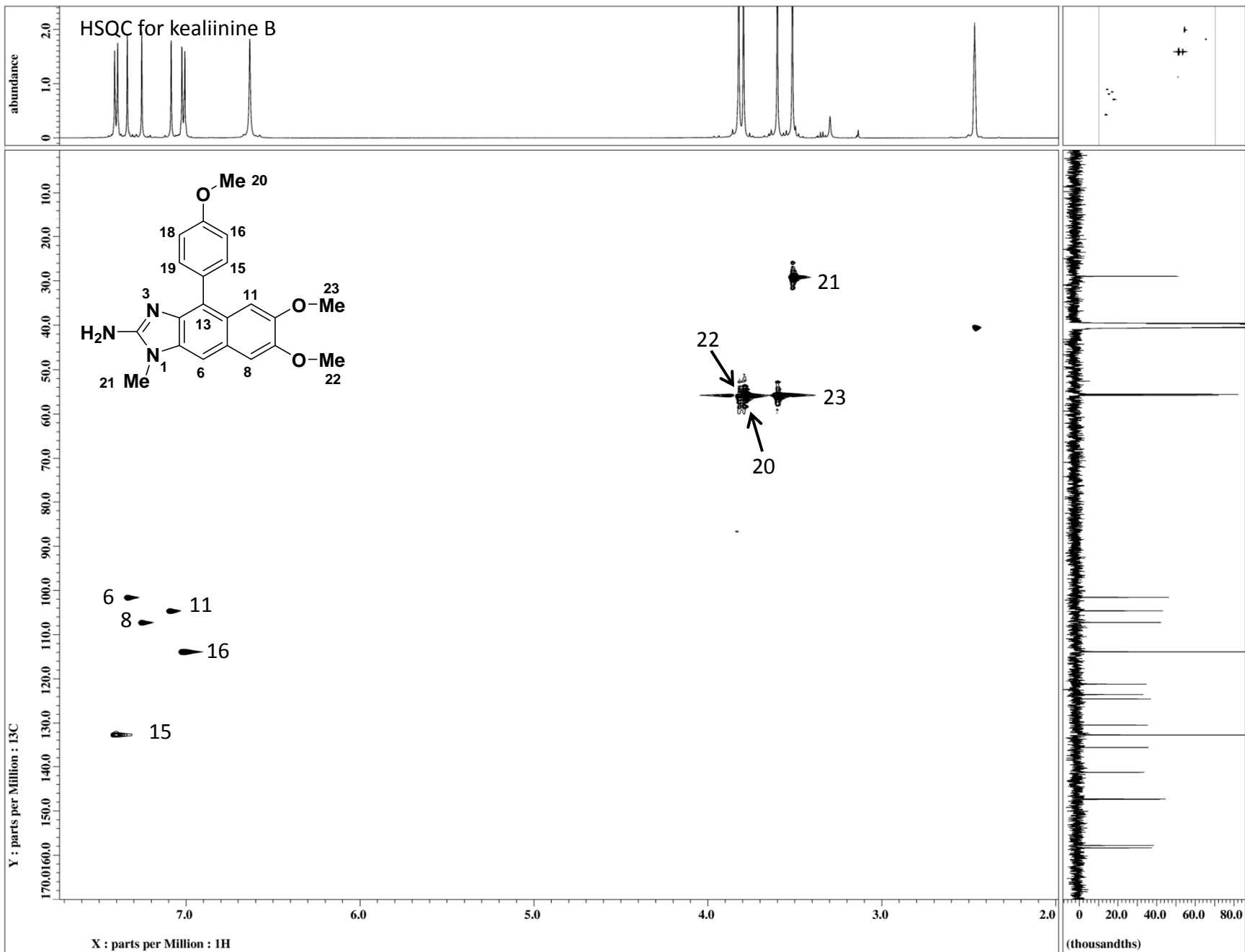


<b>3 Kealiinine B+C Vergleich</b>			
Co-injection of natural B and C mixture with synthetic kealiinine C			
Sequence Name	SM120628	Injection Volume:	20,0
Vial Number:	RB2	Recording Time:	28.6.2012 15:32
Sample Type:	unknown		
Control Program:	Standard Analytik 1 08052012		









H23

