2,3-Dimethoxyindolines: A Latent Electrophile for S_NAr Reactions Triggered by Indium-Catalyst

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SUPPORTING INFORMATION

Contents

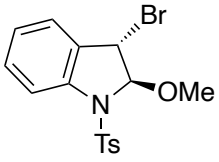
Experimental Section

1. General Experimental

Melting points were recorded with a Yanaco MP3 or Yamato MP-21 and are uncorrected. High-resolution MS spectra were recorded with a JEOL JMS-T100LP mass spectrometers. IR spectra were measured with a Shimadzu IR Affinity-1 spectrometer. The NMR experiments were performed with a JEOL JNM-ECA500 (500 MHz) spectrometer, and chemical shifts are expressed in ppm (δ) using residual solvent as an internal reference ($CDCl_3$, ^{1}H NMR: δ 7.25, ^{13}C NMR: δ 77.1). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, br = broad. Flash column chromatography was performed on silica gel (Silica Gel 60N, Kanto Chemical Co., Ltd.). The N-protected indoles **1a–1j** were prepared by reported methods.[S1]

2. Experimental Procedure

Bromomethoxylation of indoles with NBS and MeOH (Scheme 3)

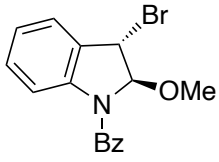


trans **2a**

trans-3-Bromo-2-methoxy-1-tosylindoline (2a)

To a solution of N-Ts indole **1a** (13.565 g, 50 mmol) in MeOH (500 mL) was added NBS (9.79 g, 55 mmol). The mixture was stirred at room temperature for 10 min. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2a**.

16.36 g, 86% yield. colorless solid; >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.69 (d, J = 8.6 Hz, 2H), 7.65 (d, J = 8.6 Hz, 1H), 7.32 (ddd, J = 1.2, 8.0, 8.0 Hz, 1H), 7.26 (d, J = 8.0 Hz, 1H), 7.17 (d, J = 8.1 Hz, 2H), 7.09 (ddd, J = 1.2, 7.5, 7.5 Hz, 1H), 5.59 (s, 1H), 4.94 (s, 1H), 3.59 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.6, 140.6, 135.3, 131.4, 130.6, 129.6, 127.8, 126.3, 125.4, 117.0, 99.9, 56.4, 47.3, 21.7; HRMS (ESI) *m/z*: 403.9931, 405.9912 (Calcd for $C_{16}H_{16}BrNO_3SNa$ $[M+Na]^+$: 403.9932, 405.9912).



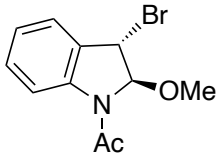
trans **2b**

trans-3-Bromo-2-methoxy-1-benzoylindoline (2b)

To a solution of N-Bz indole **1b** (1.01 g, 4.6 mmol) in MeOH (46 mL) was added NBS (890 mg, 5 mmol). The mixture was stirred at room temperature for 10 min. The resulting precipitate was separated by filtration, washed

with MeOH, and dried *in vacuo* to give **2b**.

1.05 g, 69% yield. colorless solid; >300 °C (decomp.); IR ($CHCl_3$): 3014, 1683, 1101, 1086 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.64 (d, *J* = 7.5 Hz, 2H), 7.54–7.51 (m, 1H), 7.487.42 (m, 3H), 7.27 (br s, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 5.55 (br s, 1H), 5.14 (s, 1H), 3.28 (br s, 3H) (one proton was disappeared.); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.0, 142.0, 135.5, 131.0, 130.6, 130.4, 128.7, 127.7, 126.0, 125.1, 117.9, 98.3, 55.6, 46.9; HRMS (ESI) *m/z*: 354.0104, 356.0083 (Calcd for $C_{16}H_{14}BrNO_2Na$ $[M+Na]^+$:354.0106, 356.0085).

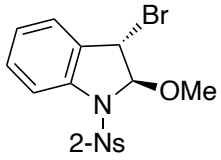


trans **2d**

trans-3-Bromo-2-methoxy-1-acetylindoline (2d)

To a solution of N-Ac indole **1d** (796 mg, 5 mmol) in MeOH (50 mL) was added NBS (979 mg, 5.5 mmol). The mixture was stirred at room temperature for 2 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/8–1/5) to give **2d**.

686 mg, 51% yield. colorless solid; mp 216–221 °C; IR ($CHCl_3$): 3019, 1717, 1223, 1207 cm^{-1};^1H NMR (500 MHz, $CDCl_3$) δ: 8.16 (d, *J* = 8.0 Hz, 1H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.33 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 5.60 (s, 1H), 5.29 (s, 1H), 3.38 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.6, 142.3, 130.8, 129.0, 125.8, 124.8, 117.2, 98.2, 53.8, 46.8; HRMS (ESI) *m/z*: 291.9947, 293.9928 (Calcd for $C_{11}H_{12}BrNO_2Na$ $[M+Na]^+$: 291.9949, 293.9929).



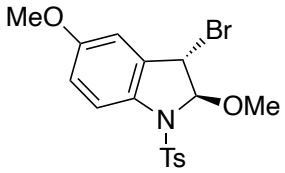
trans **2e**

trans-3-Bromo-2-methoxy-1-(2-nitrobenzenesulfonyl)indoline (2e)

To a solution of N-Ns indole **1e** (907 mg, 3 mmol) in MeOH (30 mL) was added NBS (587 mg, 3.3 mmol). The mixture was stirred at room temperature for 0.5 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 150 mL), washed with brine (100 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was recrystallized from $CHCl_3$/MeOH and the resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2e**.

786 mg, 64% yield. colorless solid; >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.76 (dd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.64 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.54 (dd, *J* = 1.2, 8.0 Hz, 1H), 7.49 (ddd, *J* = 1.7, 7.4, 7.4 Hz, 1H), 7.39 (ddd, *J* = 1.1, 8.0, 8.0 Hz, 1H), 7.35 (d, *J* = 7.5 Hz, 1H), 7.20 (dd, *J* = 1.2, 7.4 Hz, 1H), 5.84 (s, 1H), 4.99 (s, 1H), 3.57 (s, 3H),; ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.6,

140.1, 134.5, 131.7, 131.5, 131.3, 130.9, 129.8, 126.4, 126.2, 124.0, 117.5, 98.9, 57.0, 47.1; HRMS (ESI) *m/z*: 434.9625, 436.9605 (Calcd for $C_{15}H_{13}BrN_2O_5Na$ $[M+Na]^+$: 434.9626, 436.9606).

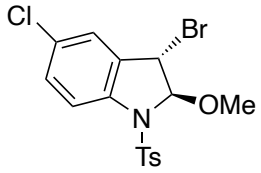


trans **2f**

***trans*-3-Bromo-2,5-dimethoxy-1-tosylindoline (2f)**

To a solution of 5-MeO-N-Ts indole **1f** (6.03 g, 20 mmol) in MeOH (200 mL) was added NBS (3.92 g, 22 mmol). The mixture was stirred at room temperature for 0.5 h. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2f**.

7.03 g, 85% yield. colorless solid; 298–300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.64 (d, J = 8.0 Hz, 2H), 7.60 (d, J = 9.2 Hz, 1H), 7.18 (d, J = 8.0 Hz, 2H), 6.89 (dd, J = 2.3, 8.6 Hz, 1H), 6.76 (d, J = 2.3 Hz, 1H), 5.52 (s, 1H), 4.85 (s, 1H), 3.76 (s, 3H), 3.59 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.7, 144.5, 135.2, 134.1, 132.8, 129.6, 127.8, 118.5, 117.0, 110.7, 100.2, 56.4, 55.8, 47.6, 21.6; HRMS (ESI) *m/z*: 434.0036, 436.0016 (Calcd for $C_{17}H_{18}BrNO_4SNa$ $[M+Na]^+$: 434.0038, 436.0017).

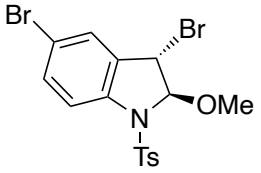


trans **2g**

***trans*-3-Bromo-5-chloro-2-methoxy-1-tosylindoline (2g)**

To a solution of 5-Cl-N-Ts indole **1g** (2.75 g, 9 mmol) in MeOH (90 mL) was added NBS (1.75 g, 9.9 mmol). The mixture was stirred at room temperature for 10 min. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2g**.

2.11 g, 56% yield. colorless solid; >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.66 (d, J = 8.7 Hz, 2H), 7.59 (d, J = 9.2 Hz, 1H), 7.28 (dd, J = 1.7, 8.6 Hz, 1H), 7.23 (d, J = 2.3 Hz, 1H), 7.20 (d, J = 8.0 Hz, 2H), 5.55 (s, 1H), 4.85 (s, 1H), 3.59 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.9, 139.2, 135.0, 133.2, 130.7, 130.6, 129.7, 127.8, 126.3, 118.1, 100.2, 56.5, 46.1, 21.7; HRMS (ESI) *m/z*: 437.9544, 439.9515, 439.9524, 441.9496 (Calcd for $C_{16}H_{15}BrClNO_3SNa$ $[M+Na]^+$: 437.9542, 439.9513, 439.9522, 441.9492).

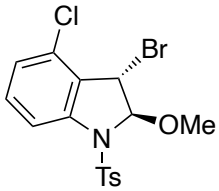


trans **2h**

trans-3,5-dibromo-2-methoxy-1-tosylindoline (2h)

To a solution of 5-Br-N-Ts indole **1h** (3.5 g, 10 mmol) in MeOH (100 mL) was added NBS (1.96 g, 11 mmol). The mixture was stirred at room temperature for 1.5 h. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2h**.

4.18 g, 91% yield. colorless solid; >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.6 Hz, 2H), 7.53 (d, J = 8.6 Hz, 1H), 7.43 (dd, J = 2.3, 8.6 Hz, 1H), 7.38 (d, J = 2.3 Hz, 1H), 7.21 (d, J = 8.0 Hz, 2H), 5.54 (s, 1H), 4.85 (s, 1H), 3.58 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.9, 139.7, 135.0, 133.6, 129.7, 129.3, 127.8, 118.5, 118.0, 100.1, 56.5, 46.0, 21.7; HRMS (ESI) *m/z*: 481.9039, 483.9017, 485.8994 (Calcd for $C_{16}H_{15}Br_2NO_3SNa$ $[M+Na]^+$: 481.9037, 483.9017, 485.8996).

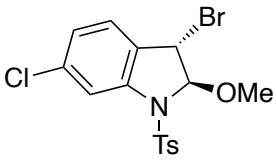


trans **2i**

trans-3-Bromo-4-chloro-2-methoxy-1-tosylindoline (2i)

To a solution of 4-Cl-N-Ts indole **1i** (3.06 g, 10 mmol) in MeOH (100 mL) was added NBS (1.96 g, 11 mmol). The mixture was stirred at room temperature for 1 h. The resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2i**.

3.52 g, 85% yield. colorless solid; mp >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.70 (d, J = 8.6 Hz, 2H), 7.56 (d, J = 8.6 Hz, 1H), 7.28 (t, J = 8.6 Hz, 1H), 7.20 (d, J = 8.0 Hz, 2H), 7.05 (d, J = 9.2 Hz, 1H), 5.60 (s, 1H), 4.95 (s, 1H), 3.60 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.9, 141.9, 135.1, 132.1, 132.0, 129.7, 129.4, 127.8, 125.5, 115.1, 99.9, 56.5, 45.9, 21.7; HRMS (ESI) *m/z*: 437.9543, 439.9510, 439.9524, 441.9496 (Calcd for $C_{16}H_{15}BrClNO_3SNa$ $[M+Na]^+$: 437.9542, 439.9513, 439.9522, 441.9492).



trans **2j**

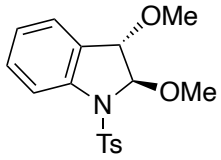
trans-3-Bromo-6-chloro-2-methoxy-1-tosylindoline (2g)

To a solution of 6-Cl-N-Ts indole**1j** (915 mg, 3 mmol) in MeOH (30 mL) was added NBS (587 mg, 3.3 mmol). The mixture was stirred at room temperature for 2 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was recrystallized from MeOH and the resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **2j**.

976 mg, 78% yield. colorless solid; mp 272–275 °C; IR ($CHCl_3$): IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.71 (d, J = 8.6 Hz, 2H), 7.66 (d, J = 1.7 Hz, 1H), 7.22 (d, J = 8.6 Hz, 2H), 7.18 (d, J = 8.0 Hz, 1H),

7.07 (dd, J = 1.7, 8.0 Hz, 1H), 5.57 (s, 1H), 4.89 (s, 1H), 3.59 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.0, 141.7, 136.6, 135.1, 130.0, 129.8, 127.8, 127.0, 125.6, 117.3, 100.4, 56.5, 46.5, 21.7; HRMS (ESI) *m/z*: 437.9541, 439.9514, 439.9525, 441.9493 (Calcd for $C_{16}H_{15}BrClNO_3SNa$ $[M+Na]^+$: 437.9542, 439.9513, 439.9522, 441.9492).

Synthesis of 2,3-dimethoxyindolines using MeOH (Scheme 3)

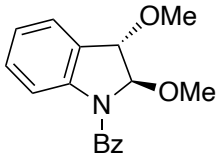


trans **3a**

trans-2,3-dimethoxy-1-tosylindoline (3a)

A solution of **2i** (17.3 g, 45.2 mmol) in MeOH (700 mL) was stirred at 100 °C for 20 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 400 mL), washed with brine (300 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was recrystallized from MeOH and the resulting precipitate was separated by filtration, washed with MeOH, and dried *in vacuo* to give **3a**.

8.74 g, 58% yield. colorless solid; mp 250–251 °C; IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (d, J = 8.0 Hz, 1H), 7.58 (d, J = 8.6 Hz, 2H), 7.33 (ddd, J = 1.2, 7.5, 7.5 Hz, 1H), 7.27 (t, J = 9.2 Hz, 1H), 7.13 (d, J = 8.6 Hz, 2H), 7.07 (t, J = 7.5 Hz, 1H), 5.33 (s, 1H), 4.25 (s, 1H), 3.59 (s, 3H), 3.17 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ:144.1, 141.9, 135.2, 130.6, 130.4, 129.3, 127.6, 126.8, 124.8, 117.4, 96.4, 83.9, 56.2, 56.0, 21.6; HRMS (ESI) *m/z*: 356.0932 (Calcd for $C_{17}H_{19}NO_4SNa$ $[M+Na]^+$: 356.0933).

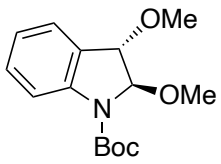


trans **3b**

trans-2,3-dimethoxy-1-benzoylindoline (3b)

A solution of **2b** (166 mg, 0.5 mmol) in MeOH (20 mL) was stirred at 100 °C for 14 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/2) to give **3b**.

45 mg, 31% yield. colorless solid; mp 243–250 °C; IR ($CHCl_3$): 3014, 1655, 1101, 1086 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (d, J = 7.5 Hz, 2H), 7.51–7.48 (m, 1H), 7.45 (d, J = 7.5 Hz, 2H), 7.42 (d, J = 6.9 Hz, 2H), 7.32 (br s, 1H), 7.12 (t, J = 7.5 Hz, 1H), 5.25 (br s, 1H), 4.48 (s, 1H), 3.42 (s, 3H), 3.25 (br s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 170.1, 143.2, 136.1, 130.7, 130.3, 129.0, 128.5, 127.6, 126.6, 124.3, 94.9, 82.1, 56.2, 54.9; HRMS (ESI) *m/z*: 306.1103 (Calcd for $C_{17}H_{17}NO_3Na$ $[M+Na]^+$: 306.1106).

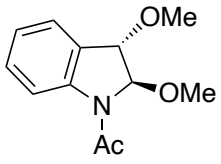


trans **3c**

trans-2,3-dimethoxy-1-(*tert*-butoxycarbonyl)indoline (3c)

To a solution of N-Boc indole **1c** (1.09 g, 5 mmol) in MeOH (50 mL) was added NBS (979 mg, 5.5 mmol). The mixture was stirred at room temperature for 5 min. After addition of H_2O, the whole was extracted with AcOEt (3 x 100 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The resulting crude material was used without purification. A solution of the crude material in MeOH (40 mL) was stirred at 100 °C for 20 min. After addition of H_2O, the whole was extracted with AcOEt (3 x 50 mL), washed with brine (30 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **3c**.

146 mg, 11% yield. colorless oil; IR ($CHCl_3$): 3019, 1705, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.84 (br s, 1H), 7.36 (d, J = 7.5 Hz, 1H), 7.33 (t, J = 8.1 Hz, 1H), 7.03 (t, J = 6.9 Hz, 1H), 5.41 (s, 1H), 4.40 (s, 1H), 3.49 (s, 3H), 3.43 (s, 3H), 1.58 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.4, 142.8, 130.4, 128.2, 126.4, 122.9, 116.4, 93.9, 83.2, 82.0, 56.2, 28.4; HRMS (ESI) *m/z*: 302.1369 (Calcd for $C_{15}H_{21}NO_4Na$ $[M+Na]^+$: 302.1368).

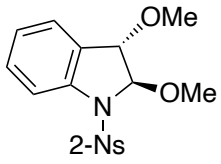


trans **3d**

trans-2,3-dimethoxy-1-acetylindoline (3d)

A solution of **2d** (328 mg, 1 mmol) in MeOH (30 mL) was stirred at 100 °C for 0.5 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/2) to give **3d**.

81 mg, 29% yield. colorless solid; mp 190–196 °C; IR ($CHCl_3$): 3019, 1670, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.21 (d, J = 8.1 Hz, 1H), 7.38 (d, J = 7.5 Hz, 1H), 7.35 (ddd, J = 1.2, 7.5, 7.5 Hz, 1H), 7.09 (t, J = 7.5 Hz, 1H), 5.27 (s, 1H), 4.62 (s, 1H), 3.44 (s, 3H), 3.32 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.9, 143.6, 130.7, 127.4, 126.1, 123.9, 117.2, 94.7, 81.9, 56.0, 53.0, 23.2; HRMS (ESI) *m/z*: 244.0950 (Calcd for $C_{12}H_{15}NO_3Na$ $[M+Na]^+$: 244.0950).

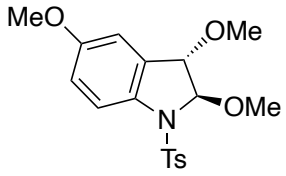


trans **3e**

trans-2,3-dimethoxy-1-(2-nitrobenzenesulfonyl)indoline (3e)

A solution of **2e** (146 mg, 0.4 mmol) in MeOH (20 mL) was stirred at 100 °C for 15 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/3) to give **3b**.

17 mg, 13% yield. colorless solid; mp 278–281 °C; IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.67 (d, J = 8.0 Hz, 1H), 7.61 (t, J = 8.0 Hz, 2H), 7.59 (d, J = 7.5 Hz, 1H), 7.54 (d, J = 8.0 Hz, 1H), 7.43 (t, J = 7.5 Hz, 1H), 7.39–7.36 (m, 2H), 7.17 (t, J = 7.5 Hz, 1H), 5.56 (s, 1H), 4.31 (s, 1H), 3.57 (s, 3H), 3.30 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.7, 141.0, 134.0, 131.3, 130.9, 130.8, 130.7, 130.1, 127.1, 125.6, 123.9, 117.4, 95.9, 84.1, 57.0, 56.5; HRMS (ESI) *m/z*: 387.0626 (Calcd for $C_{16}H_{16}N_2O_6SNa$ $[M+Na]^+$: 387.0627).

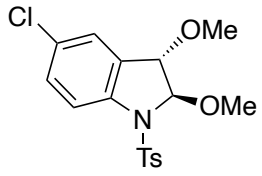


trans **3f**

trans-2,3,5-trimethoxy-1-1-tosylindoline (3f)

A solution of **2f** (412 mg, 1.0 mmol) in MeOH (20 mL) was stirred at 100 °C for 14 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/3) to give **3f**.

265 mg, 72% yield. colorless solid; mp 240–242 °C; IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.56 (d, J = 9.2 Hz, 1H), 7.52 (d, J = 8.6 Hz, 2H), 7.13 (d, J = 8.6 Hz, 2H), 6.88 (dd, J = 2.3, 8.6 Hz, 1H), 6.80 (d, J = 2.9 Hz, 1H), 5.25 (s, 1H), 4.16 (s, 1H), 3.75 (s, 3H), 3.59 (s, 3H), 3.13 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.4, 143.9, 135.2, 135.0, 131.9, 129.3, 127.6, 118.9, 116.5, 111.8, 96.6, 84.3, 56.3, 56.0, 55.7, 21.6; HRMS (ESI) *m/z*: 386.1035 (Calcd for $C_{18}H_{21}NO_5SNa$ $[M+Na]^+$: 386.1038).



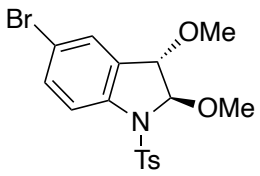
trans **3g**

trans-5-chloro-2,3-dimethoxy-1-tosylindoline (3g)

A solution of **2g** (208 mg, 0.5 mmol) in MeOH (20 mL) was stirred at 100 °C for 14 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/2) to give **3g**.

27 mg, 15% yield. colorless solid; mp 260–264 °C; IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz,

$CDCl_3$) δ: 7.56 (d, J = 8.6 Hz, 2H), 7.55 (d, J = 9.2 Hz, 1H), 7.29 (dd, J = 2.3, 9.2 Hz, 2H), 7.24 (d, J = 2.3 Hz, 1H), 7.15 (d, J = 8.0 Hz, 2H), 5.30 (s, 1H), 4.20 (s, 1H), 3.58 (s, 3H), 3.16 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.3, 140.5, 134.9, 132.2, 130.6, 130.1, 129.4, 127.6, 127.0, 118.6, 96.6, 83.6, 56.4, 56.1, 21.6; HRMS (ESI) *m/z*: 390.0544, 392.0514 (Calcd for $C_{17}H_{18}ClNO_4SNa$ $[M+Na]^+$: 390.0543, 392.0513).

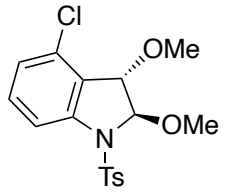


trans **3h**

***trans*-5-bromo-2,3-dimethoxy-1-tosylindoline (3h)**

A solution of **2h** (922 mg, 2 mmol) in MeOH (50 mL) was stirred at 100 °C for 16 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/8–1/2) to give **3h** and a recovery of **2h** (533 mg, 58%).

95 mg, 12% yield (27%, brsm). colorless oil; IR ($CHCl_3$): 3022, 1165, 1109 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.56 (d, J = 8.0 Hz, 2H), 7.49 (d, J = 8.6 Hz, 1H), 7.44 (dd, J = 2.3, 8.6 Hz, 2H), 7.39 (d, J = 1.7 Hz, 1H), 7.15 (d, J = 8.6 Hz, 2H), 5.29 (s, 1H), 4.20 (s, 1H), 3.58 (s, 3H), 3.17 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.4, 141.0, 134.9, 133.5, 132.6, 129.9, 129.5, 127.6, 119.0, 117.6, 96.6, 83.5, 56.4, 56.1, 21.7; HRMS (ESI) *m/z*: 434.0038, 436.0019 (Calcd for $C_{17}H_{18}BrNO_4SNa$ $[M+Na]^+$: 434.0038, 436.0017).

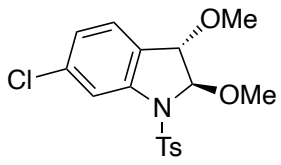


trans **3i**

***trans*-4-chloro-2,3-dimethoxy-1-tosylindoline (3i)**

A solution of **2i** (208 mg, 0.5 mmol) in MeOH (20 mL) was stirred at 100 °C for 15 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/1) to give **3i** and a recovery of **2i** (107 mg, 51%).

60 mg, 32% yield (65%, brsm). colorless solid; mp >300 °C (decomp.); IR ($CHCl_3$): 3019, 1223, 1207 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.60 (d, J = 8.6 Hz, 2H), 7.48 (d, J = 8.6 Hz, 1H), 7.24 (t, J = 8.6 Hz, 1H), 7.16 (d, J = 8.0 Hz, 2H), 7.03 (d, J = 8.0 Hz, 1H), 5.34 (s, 1H), 4.39 (s, 1H), 3.59 (s, 3H), 3.29 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.4, 143.1, 135.1, 132.8, 131.8, 129.5, 129.1, 127.6, 125.2, 115.4, 96.1, 82.9, 57.3, 56.1, 21.6; HRMS (ESI) *m/z*: 390.0540, 392.0512 (Calcd for $C_{17}H_{18}ClNO_4SNa$ $[M+Na]^+$: 390.0543, 392.0513).



trans **3j**

trans-6-chloro-2,3-dimethoxy-1-tosylindoline (3j)

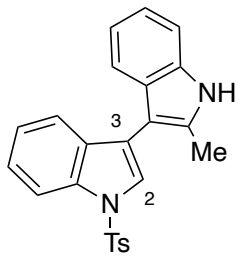
A solution of **2j** (417 mg, 1 mmol) in MeOH (30 mL) was stirred at 100 °C for 15 h. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/3) to give **3j** and a recovery of **2i** (107 mg, 51%).

253mg, 69% yield. colorless solid; mp 293–296 °C; IR ($CHCl_3$): 3018, 1223, 1207 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.63 (d, *J* = 1.7 Hz, 1H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 7.17 (d, *J* = 8.6 Hz, 2H), 7.04 (dd, *J* = 2.3, 8.0 Hz, 1H), 5.32 (s, 1H), 4.21 (s, 1H), 3.57 (s, 3H), 3.16 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.4, 143.1, 136.4, 135.0, 129.5, 128.9, 127.6, 124.9, 117.7, 96.8, 86.3, 56.2, 56.1, 21.7; HRMS (ESI) *m/z*: 390.0542, 392.0513 (Calcd for $C_{17}H_{18}ClNO_4SNa$ $[M+Na]^+$: 390.0543, 392.0513).

General Procedure for the Indium-mediated S_NAr Reaction of 3 with 4a (Scheme 3)

To a solution of **3** (0.5 mmol) and **4a** (98.4 mg, 0.75 mmol) in MeCN (10 mL) was added $In(OTf)_3$ (28.1 mg, 0.05 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (15 mL). The whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/3, and/or $CHCl_3$/hexane = 1/2–5/1) to give **6**.

The ratio of **5**/**6** was determined by ^1H-NMR analysis of the crude material.

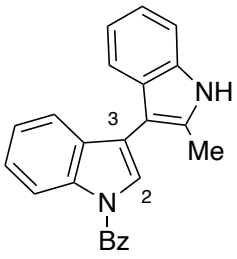


6aa

3-(2-Methylindol-3-yl)-1-tosylindole (6aa)

187 mg, 93% yield. colorless solid; mp 79–81 °C; IR ($CHCl_3$): 3468, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.07 (d, *J* = 8.6 Hz, 1H), 8.06 (br s, 1H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.57 (s, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.33-7.38 (m, *J* = 3H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.17 (ddd, *J* = 1.2, 6.7, 6.7 Hz, 1H), 7.06 (ddd, *J* = 1.2, 6.7, 6.7 Hz, 1H), 2.44 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.9, 135.5, 135.4, 135.3, 132.9, 131.3, 130.0, 128.4, 127.0, 124.7, 124.0, 123.2, 121.7, 121.5, 119.9, 119.3, 117.2, 113.9, 110.5, 105.1, 21.7, 12.7; HRMS

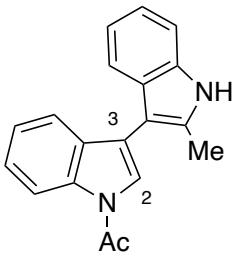
(ESI) *m/z*: 423.1142 (Calcd for $C_{24}H_{20}N_2O_2SNa$ $[M+Na]^+$: 423.1143).



6ba

3-(2-Methylindol-3-yl)-1-benzoylindole (6ba)

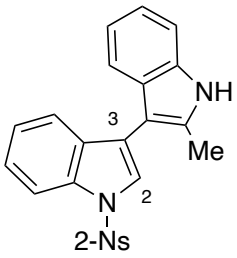
166 mg, 95% yield. colorless solid; mp 245–248 °C; IR ($CHCl_3$): 3468, 2976, 1680 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.48 (d, *J* = 8.6 Hz, 1H), 8.06 (br s, 1H), 7.82–7.80 (m, 2H), 7.61–7.58 (m, 1H), 7.54–7.51 (m, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.43 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 7.33 (s, 1H), 7.31 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 7.17 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.08 (t, *J* = 8.0 Hz, 1H), 2.45 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 168.7, 136.5, 135.5, 134.9, 132.9, 131.9, 131.3, 129.3, 128.7, 128.6, 125.4, 125.2, 123.9, 121.7, 121.0, 120.0, 119.3, 116.6, 116.5, 110.5, 105.3, 12.8; HRMS (ESI) *m/z*: 373.1317 (Calcd for $C_{24}H_{18}N_2ONa$ $[M+Na]^+$: 373.1317).



6ca

3-(2-Methylindol-3-yl)-1-acetylindole (6ca)

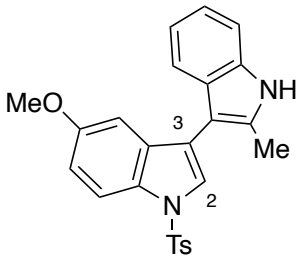
139 mg, 96% yield. colorless solid; mp 270275°C; R ($CHCl_3$): 3468, 3022, 1701cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.52 (d, *J* = 6.3 Hz, 1H), 8.10 (br s, 1H), 7.48 (d, *J* = 8.0 Hz, 2H), 7.44 (s, 1H), 7.40 (t, *J* = 8.6 Hz, 1H), 7.37 (d, *J* = 8.6 Hz, 1H), 7.27 (t, *J* = 7.4 Hz, 1H), 7.18 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 7.30-7.25 (m, 2H), 7.21-7.19 (m, 3H), 2.69 (s, 3H), 2.48 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 168.6, 136.1, 135.5, 132.9, 131.0, 128.7, 125.4, 123.6, 123.2, 121.8, 121.0, 120.0, 119.3, 117.1, 116.8, 110.5, 105.3, 24.2, 12.8; HRMS (ESI) *m/z*: 311.1161 (Calcd for $C_{19}H_{16}N_2ONa$ $[M+Na]^+$: 311.1160).



6ea

3-(2-Methylindol-3-yl)-1-(2-nitrobenzenesulfonyl)indole (6ea)

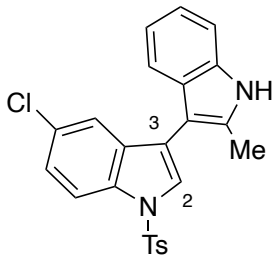
57 mg, 26% yield. yellow solid; mp >300 °C (decomp.); IR ($CHCl_3$): 3447, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.10 (br s, 1H), 7.98 (d, *J* = 7.5 Hz, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.74 (s, 1H), 7.72 (dd, *J* = 1.1, 5.2 Hz, 1H), 7.697.65 (m, 1H), 7.62 (s, 1H), 7.53 (d, *J* = 7.4 Hz, 1H), 7.47 (d, *J* = 8.5 Hz, 1H), 7.38–7.35 (m, 2H), 7.27 (t, *J* = 8.0 Hz, 1H), 7.18 (ddd, *J* = 1.1, 6.9, 6.9 Hz, 1H), 7.09 (ddd, *J* = 1.2, 6.9, 6.9 Hz, 1H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 148.1, 135.5, 135.0, 134.9, 133.3, 132.4, 132.1, 131.3, 130.1, 128.5, 125.0, 124.7, 123.7, 122.0, 121.8, 120.0, 119.4, 117.0, 113.5, 110.5, 104.7, 12.7; HRMS (ESI) *m/z*: 454.0837 (Calcd for $C_{23}H_{17}N_3O_4SNa$ $[M+Na]^+$: 454.0838).



6fa

5-Methoxy-3-(2-methylindol-3-yl)-1-tosylindole (6fa)

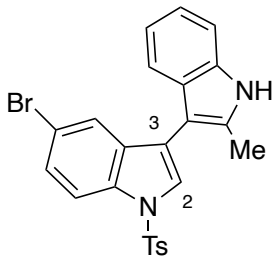
186 mg, 86% yield. colorless solid; mp 234–237 °C; IR ($CHCl_3$): 3468, 3019, 1173 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.07 (br s, 1H), 7.96 (d, *J* = 9.2 Hz, 1H), 7.78 (d, *J* = 8.6 Hz, 2H), 7.51 (s, 1H), 7.36 (t, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 8.0 Hz, 1H), 7.06 (t, *J* = 6.9 Hz, 1H), 6.96 (dd, *J* = 2.9, 9.2 Hz, 1H), 6.86 (d, *J* = 2.3 Hz, 1H), 3.70 (s, 3H), 2.44 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.6, 145.0, 135.6, 135.2, 133.1, 132.5, 130.2, 130.0, 128.3, 126.9, 124.9, 121.7, 119.9, 119.3, 117.7, 114.9, 114.1, 110.7, 104.9, 103.8, 55.7, 21.7, 12.6; HRMS (ESI) *m/z*: 453.1249 (Calcd for $C_{25}H_{22}N_2O_3SNa$ $[M+Na]^+$: 453.1249).



6ga

5-Chloro-3-(2-methylindol-3-yl)-1-tosylindole (6ga)

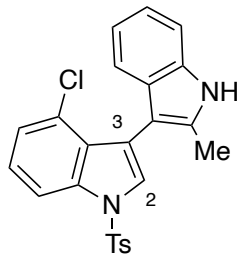
195 mg, 90% yield. colorless solid; mp 165–166 °C; IR ($CHCl_3$): 3468, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.09 (br s, 1H), 7.99 (d, *J* = 8.6 Hz, 1H), 7.89 (d, *J* = 8.6 Hz, 2H), 7.57 (s, 1H), 7.39 (d, *J* = 2.3 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.30 (dd, *J* = 1.7, 8.6 Hz, 1H), 7.25 (d, *J* = 8.6 Hz, 2H), 7.18 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 7.08 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 2.42 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.4, 135.5, 135.0, 133.8, 133.1, 132.7, 130.1, 129.3, 128.3, 126.9, 125.4, 125.1, 121.9, 121.1, 120.2, 119.0, 116.9, 115.0, 110.6, 104.3, 21.7, 12.6; HRMS (ESI) *m/z*: 457.0756, 459.0723 (Calcd for $C_{24}H_{19}ClN_2O_2SNa$ $[M+Na]^+$: 457.0754, 459.0724).



6ha

5-Bromo-3-(2-methylindol-3-yl)-1-tosylindole (6ha)

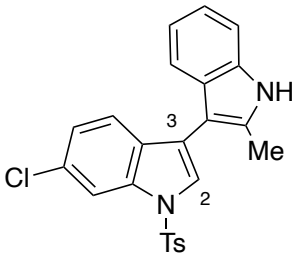
203 mg, 85% yield. colorless solid; mp 239–243 °C; IR ($CHCl_3$): 3468, 3017, 2976 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.09 (br s, 1H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.79 (d, *J* = 8.6 Hz, 2H), 7.55 (d, *J* = 2.9 Hz, 1H), 7.55 (s, 1H), 7.43 (d, *J* = 1.7, 8.6 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 6.7 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.18 (ddd, *J* = 1.2, 7.4, 7.4 Hz, 1H), 7.08 (ddd, *J* = 1.2, 7.4, 7.4 Hz, 1H), 2.42 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.3, 135.5, 135.0, 134.1, 133.1, 130.1,, 128.3, 127.7, 126.9, 125.2, 124.1, 121.9, 120.2, 119.0, 116.9, 116.8, 115.3, 110.6, 104.3, 21.7, 12.6; HRMS (ESI) *m/z*: 501.0251, 503.0233 (Calcd for $C_{24}H_{19}BrN_2O_2SNa$ $[M+Na]^+$: 501.0248, 503.0228).



6ia

4-Chloro-3-(2-methylindol-3-yl)-1-tosylindole (6ia)

126 mg, 58% yield. Colorless oil; IR ($CHCl_3$): 3447, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.01 (br s, 1H), 8.00 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.6 Hz, 2H), 7.56 (s, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 1H), 7.17 (t, *J* = 8.1 Hz, 2H), 7.14 (t, *J* = 6.9 Hz, 1H), 7.04 (ddd, *J* = 1.2, 7.5, 7.5 Hz, 1H), 2.39 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.4, 136.6, 135.1, 135.0, 134.1, 130.4, 130.1, 128.7, 127.4, 127.0, 126.9, 125.3, 124.8, 121.4, 119.8, 119.1, 116.0, 112.4, 110.2, 105.1, 21.7, 12.5; HRMS (ESI) *m/z*: 457.0751, 459.0723 (Calcd for $C_{24}H_{19}ClN_2O_2SNa$ $[M+Na]^+$: 457.0754, 459.0724).



6ja

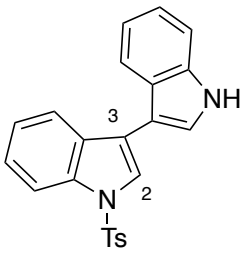
6-Chloro-3-(2-methylindol-3-yl)-1-tosylindole (6ja)

131 mg, 60% yield. colorless oil; IR ($CHCl_3$): 3447, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.09 (br s, 2H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.54 (s, 1H), 7.35 (d, *J* = 8.6 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.19-7.15 (m, 2H), 7.06 (t, *J* = 8.0 Hz, 1H), 2.43 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.3, 135.7, 135.4, 135.1, 133.0, 130.8, 130.1, 129.8, 128.2, 127.0, 124.4, 123.9, 122.3, 121.9, 120.1, 119.1, 117.1, 114.0, 110.6, 104.6, 21.7, 12.7; HRMS (ESI) *m/z*: 457.0755, 459.0721 (Calcd for $C_{24}H_{19}ClN_2O_2SNa$ $[M+Na]^+$: 457.0754, 459.0724).

General Procedure for the Indium-mediated S_NAr Reaction of 3a with indoles 4 (Scheme 4)

To a solution of **3a** (166.7 mg, 0.5 mmol) and **4** (0.75 mmol) in MeCN (10 mL) was added $In(OTf)_3$ (28.1 mg, 0.05 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (15 mL). The whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/3, and/or $CHCl_3$/hexane = 1/2–5/1) to give **6**.

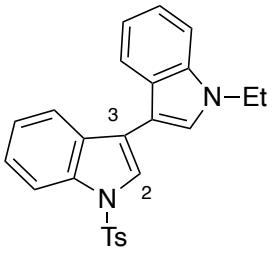
The ratio of **5**/**6** was determined by ^1H-NMR analysis of the crude material.



6ab

3-(Indol-3-yl)-1-tosylindole (6ab)

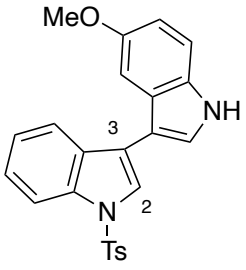
173 mg, 90% yield. colorless solid; mp 270–271 °C; IR ($CHCl_3$): 3476, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.31 (br s, 1H), 8.08 (d, *J* = 8.6 Hz, 1H), 7.82 (d, *J* = 9.2 Hz, 2H), 7.81 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.49 (d, *J* = 2.3 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.30-7.25 (m, 2H), 7.21-7.19 (m, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.9, 136.4, 135.5, 135.4, 130.5, 130.0, 127.0, 126.4, 124.9, 123.4, 122.9, 122.5, 120.9, 120.5, 120.1, 117.4, 114.0, 111.5, 108.9, 21.6; HRMS (ESI) *m/z*: 409.0986 (Calcd for $C_{23}H_{18}N_2O_2SNa$ $[M+Na]^+$: 409.0987).



6ac

3-(1-Ethylindol-3-yl)-1-tosylindole (6ac)

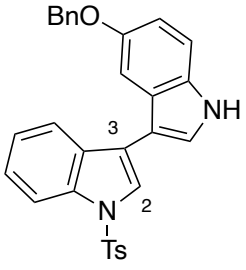
58 mg, 28% yield. colorless solid; mp 82–83 °C; IR ($CHCl_3$): 3482, 3018 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.10 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.81 (s, 1H), 7.75 (d, *J* = 7.5 Hz, 1H), 7.43 (d, *J* = 6.9 Hz, 1H), 7.42 (s, 1H), 7.38 (t, *J* = 8.6 Hz, 1H), 7.31 (t, *J* = 6.9 Hz, 1H), 7.29 (t, *J* = 7.4 Hz, 1H), 7.21 (t, *J* = 6.9 Hz, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 4.25 (q, *J* = 7.5 Hz, 2H), 2.32 (s, 3H), 1.53 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 1444.9, 136.3, 135.6, 135.4,130.5, 129.9, 126.9, 125.5, 124.9, 123.4, 122.3, 122.2, 121.0, 120.3, 120.0, 117.7, 114.0, 109.8, 41.2, 21.7, 15.6; HRMS (ESI) *m/z*: 437.1300 (Calcd for $C_{25}H_{22}N_2O_2SNa$ $[M+Na]^+$: 437.1300).



6ae

3-(5-Methoxyindol-3-yl)-1-tosylindole (6ae)

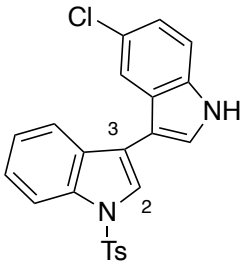
184 mg, 88% yield. colorless solid; mp 209–210 °C; IR ($CHCl_3$): 3476, 3013, 2976, 1173 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.25 (br s, 1H), 8.10 (d, *J* = 8.1 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.78 (s, 1H), 7.70 (d, *J* = 7.5 Hz, 1H), 7.40 (d, *J* = 2.3 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 9.2 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.20 (s, 1H), 7.19 (d, *J* = 8.6 Hz, 2H), 6.94 (dd, *J* = 1.8, 8.6 Hz, 1H), 3.85 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 154.8, 145.0, 135.5, 135.3, 131.5, 130.6, 130.0, 126.9, 126.8, 125.0, 123.5, 122.3, 121.0, 117.6, 114.0, 113.0, 112.3, 108.5, 101.9, 56.1, 21.6; HRMS (ESI) *m/z*: 439.1092 (Calcd for $C_{24}H_{20}N_2O_3SNa$ $[M+Na]^+$: 439.1092).



6af

3-(5-Benzyloxyindol-3-yl)-1-tosylindole (6af)

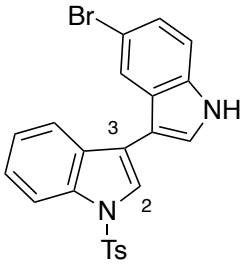
208 mg, 84% yield. colorless solid; mp 179–181 °C; IR ($CHCl_3$): 3480, 3019, 1074 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.20 (br s, 1H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.73 (s, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 2H), 7.43-7.38 (m, 3H), 7.36-7.31 (m, 3H), 7.27-7.24 (m, 3H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.01 (dd, *J* = 1.7, 8.6 Hz, 1H), 5.10 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.9, 144.9, 137.6, 135.5, 135.4, 131.7, 130.5, 130.0, 128.7, 127.9, 127.7, 127.0, 126.8, 124.9, 123.4, 123.3, 122.3, 120.9, 117.5, 114.0, 113.8, 112.2, 108.7, 103.6, 71.1, 21.6; HRMS (ESI) *m/z*: 515.1405 (Calcd for $C_{30}H_{24}N_2O_3SNa$ $[M+Na]^+$: 515.1405).



6ag

3-(5-Chloroindol-3-yl)-1-tosylindole (6ag)

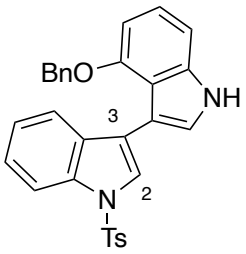
137mg, 65% yield. colorless solid; mp 180–183 °C; IR ($CHCl_3$): 3474, 3017, 2976 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.36 (br s, 1H), 8.08 (d, *J* = 8.6 Hz, 1H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.75 (s, 1H), 7.67 (d, *J* = 1.7 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 2.3 Hz, 1H), 7.39-7.35 (m, 2H), 7.29-7.21 (m, 4H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.1, 135.5, 135.3, 134.7, 130.3, 130.0, 127.6, 127.0, 126.3, 125.1, 123.9, 123.5, 123.2, 122.7, 120.7, 119.5, 116.7, 114.0, 112.5, 108.7, 21.7; HRMS (ESI) *m/z*: 443.0596, 445.0567 (Calcd for $C_{23}H_{17}ClN_2O_2SNa$ $[M+Na]^+$: 443.0597, 445.0568).



6ah

3-(5-Bromoindol-3-yl)-1-tosylindole (6ah)

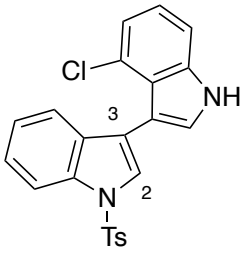
125 mg, 54% yield. colorless solid; mp 98–99 °C; IR ($CHCl_3$): 3472, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.37 (br s, 1H), 8.09 (d, *J* = 8.0 Hz, 1H), 7.83 (s, 1H), 7.82 (d, *J* = 5.8 Hz, 2H), 7.74 (s, 1H), 7.64 (d, *J* = 8.1 Hz, 1H), 7.44 (s, 1H), 7.39-7.26 (m, 4H), 7.22 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.1, 135.5, 135.3, 135.0, 130.3, 130.0, 128.2, 126.9, 125.8, 125.1, 123.8, 123.6, 122.7, 122.5, 120.7, 116.7, 114.0, 113.8, 113.0, 108.6, 21.7; HRMS (ESI) *m/z*: 487.0091, 489.0070 (Calcd for $C_{23}H_{17}BrN_2O_2SNa$ $[M+Na]^+$: 487.0092, 489.0071).



6ai

3-(4-Benzyloxyindol-3-yl)-1-tosylindole (6ai)

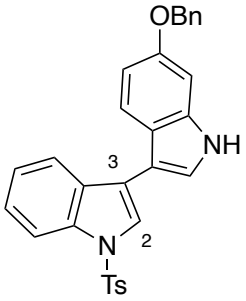
185 mg, 75% yield. colorless solid; mp 160–165 °C; IR ($CHCl_3$): 3476, 3011, 2959, 1174, 1126 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.29 (br s, 1H), 7.99 (d, *J* = 8.6 Hz, 1H), 7.74 (s, 1H), 7.71 (d, *J* = 8.6 Hz, 2H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.27-7.23 (m, 2H), 7.16-7.10 (m, 2H), 7.08-7.05 (m, 4H), 7.01 (t, *J* = 7.5 Hz, 2H), 6.82 (d, *J* = 7.5 Hz, 2H), 6.60 (d, *J* = 8.0 Hz, 1H), 5.00 (s, 2H), 2.24 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 153.5, 144.6, 138.3, 137.0, 135.6, 135.1, 132.1, 129.8, 128.2, 127.2, 126.8, 126.4, 124.4, 123.5, 123.2, 122.7, 121.6, 118.2, 117.2, 113.5, 107.9, 105.1, 102.3, 70.0, 21.6; HRMS (ESI) *m/z*: 515.1406 (Calcd for $C_{30}H_{24}N_2O_3SNa$ $[M+Na]^+$: 515.1405).



6aj

3-(4-Chloroindol-3-yl)-1-tosylindole (6aj)

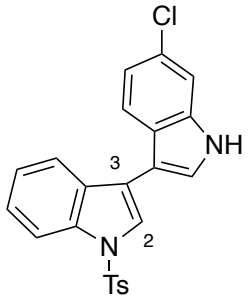
133 mg, 63% yield. colorless solid; mp 110–112 °C; IR ($CHCl_3$): 3472, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.52 (br s, 1H), 8.06 (d, *J* = 8.6 Hz, 1H), 7.79 (d, *J* = 8.6 Hz, 2H), 7.64 (s, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.33 (t, *J* = 8.6 Hz, 2H), 7.25-7.19 (m, 4H), 7.13 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.5 Hz, 1H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.0, 137.6, 135.5, 134.9, 132.9, 129.9, 126.9, 126.5, 125.7, 125.5, 124.7, 124.3, 123.4, 123.1, 121.4, 121.2, 117.3, 113.7, 110.4, 107.5, 21.7; HRMS (ESI) *m/z*: 443.0595, 445.0566 (Calcd for $C_{23}H_{17}ClN_2O_2SNa$ $[M+Na]^+$: 443.0597, 445.0568).



6ak

3-(6-Benzyloxyindol-3-yl)-1-tosylindole (6ak)

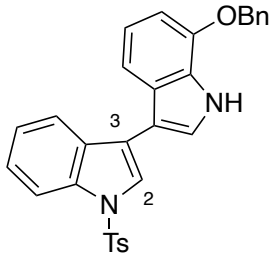
201 mg, 82% yield. colorless solid; mp 179–180 °C; IR ($CHCl_3$): 3447, 3019, 1175 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.14 (br s, 1H), 8.08 (d, *J* = 8.6 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 2H), 7.79 (s, 1H), 7.70 (d, *J* = 7.5 Hz, 1H), 7.47 (d, *J* = 7.5 Hz, 2H), 7.41-7.31 (m, 5H), 7.26 (t, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.97-6.96 (m, 2H), 5.12 (s, 2H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 156.1, 144.9, 137.4, 137.1, 135.5, 135.3, 130.4, 130.0, 128.7, 128.0, 127.6, 126.9, 124.9, 123.4, 122.3, 121.5, 120.9, 120.7, 117.5, 114.0, 111.2, 108.8, 96.3, 70.7, 21.6; HRMS (ESI) *m/z*: 515.1404 (Calcd for $C_{30}H_{24}N_2O_3SNa$ $[M+Na]^+$: 515.1405).



6al

3-(6-Chloroindol-3-yl)-1-tosylindole (6al)

120 mg, 57% yield. colorless solid; mp 176–180 °C; IR ($CHCl_3$): 3472, 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.29 (br s, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.77 (s, 1H), 7.66 (t, *J* = 8.1 Hz, 2H), 7.45 (d, *J* = 2.9 Hz, 1H), 7.44 (d, *J* = 1.8 Hz, 1H), 7.37 (ddd, *J* = 1.2, 8.2, 8.2 Hz, 1H), 7.22 (d, *J* = 8.6 Hz, 2H), 7.16 (dd, *J* = 1.7, 8.6 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.1, 136.7, 135.4, 135.3, 130.3, 130.0, 128.7, 126.9, 125.1, 125.0, 123.5, 123.2, 122.6, 121.2, 120.9, 120.8, 116.9, 114.0, 111.5, 109.0, 21.7; HRMS (ESI) *m/z*: 443.0596, 445.0569 (Calcd for $C_{23}H_{17}ClN_2O_2SNa$ $[M+Na]^+$: 443.0597, 445.0568).



6am

3-(7-Benzyloxyindol-3-yl)-1-tosylindole (6am)

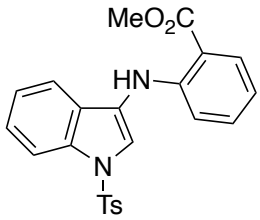
215 mg, 87% yield. colorless solid; mp 150–154 °C; IR ($CHCl_3$): 3474, 3017, 2976, 1174 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.56 (br s, 1H), 8.08 (d, *J* = 8.6 Hz, 1H), 7.81 (d, *J* = 8.6 Hz, 2H), 7.80 (s, 1H), 7.72 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 6.9 Hz, 2H), 7.45-7.34 (m, 6H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.11 (t, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 8.1Hz, 1H), 5.25 (s, 2H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.6, 144.9, 137.0, 135.5, 135.4, 130.5, 130.0, 128.8, 128.3, 128.0, 127.8, 127.2, 126.9, 124.9, 123.4, 122.5, 122.1, 120.9, 120.8, 117.6, 114.0, 113.0, 109.3, 103.9, 70.5, 21.6; HRMS (ESI) *m/z*: 515.1404 (Calcd for $C_{30}H_{24}N_2O_3SNa$ $[M+Na]^+$: 515.1405).

Gram-scale synthesis of 6am

To a solution of **3a** (1.67 g, 5 mmol) and **4** (1.68 g, 7.5 mmol) in MeCN (80 mL) was added $In(OTf)_3$ (281 mg, 0.5 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (50 mL). The whole was extracted with AcOEt (3 x 80 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/3, and/or $CHCl_3$/hexane = 1/2–5/1) to give **6am** (2.054 g, 4.17 mmol, 83%).

Amination of 3a (Scheme 6)

To a solution of **3a** (333.4 mg, 1 mmol) and methyl anthranilate (202.7 mg, 1.5 mmol) in MeCN (20 mL) was added $In(OTf)_3$ (56.2 mg, 0.1 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (30 mL). The whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/3, and $CHCl_3$/hexane = 1/1) to give **7**.



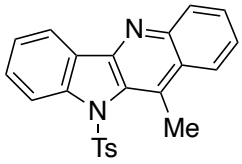
7

Methyl 2-((1-tosylindol-3-yl)amino)benzoate (7)

342 mg, 81% yield. colorless solid; mp 268–269 °C; IR ($CHCl_3$): 3482, 3019, 1684 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 9.67 (br s, 1H), 8.05 (d, J = 8.6 Hz, 1H), 8.00 (dd, J = 1.2, 8.1 Hz, 1H), 7.75 (d, J = 8.6 Hz, 2H), 7.51 (s, 1H), 7.47 (d, J = 8.0 Hz, 1H), 7.38 (ddd, J = 1.2, 8.0, 8.0 Hz, 1H), 7.36 (ddd, J = 1.2, 8.0, 8.0 Hz, 1H), 7.24 (t, J = 7.5 Hz, 1H), 7.20 (d, J = 8.6 Hz, 2H), 7.03 (d, J = 8.6 Hz, 1H), 6.77 (t, J = 7.5 Hz, 1H), 3.92 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 169.2, 147.9, 144.9, 134.9, 134.7, 134.4, 131.7, 129.9, 127.5, 126.9, 125.6, 124.4, 123.4, 118.9, 117.4, 115.0, 114.4, 114.3, 111.8, 52.0, 21.7; HRMS (ESI) *m/z*: 443.1046 (Calcd for $C_{23}H_{20}N_2O_4SNa$ $[M+Na]^+$: 443.1042).

Amination/cyclization of 3a and 2-aminoacetophenone (Scheme 6)

To a solution of **3a** (333.4 mg, 1 mmol) and 2-aminoacetophenone (202.7 mg, 1.5 mmol) in MeCN (20 mL) was added $In(OTf)_3$ (56.2 mg, 0.1 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (30 mL). The whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/5–1/3, and $CHCl_3$/hexane = 1/1) to give **8**.



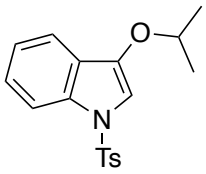
8

11-Methyl-10-tosyl-10*H*-indolo[3,2-*b*]quinoline (8)

235 mg, 61% yield. Pale yellow solid; mp >300 °C; IR ($CHCl_3$): 3019 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.22 (d, *J* = 8.6 Hz, 2H), 8.14 (d, *J* = 8.6 Hz, 1H), 8.03 (d, *J* = 7.5 Hz, 1H), 7.75 (ddd, *J* = 1.8, 7.5, 7.5 Hz, 1H), 7.64 (ddd, *J* = 1.2, 7.4, 7.4 Hz, 1H), 7.56 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.39 (t, *J* = 7.4 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 2H), 6.76 (d, *J* = 8.6 Hz, 2H), 3.18 (s, 3H), 2.13 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.1, 146.8, 144.8, 144.7, 136.3, 132.9, 131.4, 130.4, 129.5, 129.4, 129.3, 129.1, 129.0, 127.9, 127.2, 126.5, 126.3, 124 9, 121.7, 120.1, 21.5, 17.7; HRMS (ESI) *m/z*: 409.0986 (Calcd for $C_{23}H_{18}N_2O_2SNa$ $[M+Na]^+$: 409.0987).

Methoxy-alkoxy exchange reaction of 3a with 2-propanol (Scheme 6)

To a solution of **3a** (333.4 mg, 1 mmol) and 2-propanol (0.77 mL, 10 mmol) in MeCN (20 mL) was added $In(OTf)_3$ (56.2 mg, 0.1 mmol). The mixture was stirred at 100 °C for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (30 mL). The whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **9**.



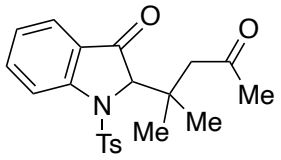
9

3-Isopropoxy-1-tosylindole (9)

152 mg, 46% yield. colorless solid; mp 98-100 °C; R ($CHCl_3$): 3019, 2978, 1168, 1092 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 8.6 Hz, 1H), 7.67 (d, *J* = 8.6 Hz, 2H), 7.49 (d, *J* = 8.1 Hz, 1H), 7.31 (ddd, *J* = 1.2, 8.0, 8.0 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.87 (s, 1H), 4.39-4.34 (m, 1H), 2.31 (s, 3H), 1.38 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 144.6, 144.2, 134.8, 134.2, 129.7, 126.8, 125.8, 125.7, 123.2, 118.8, 114.3, 105.4, 73.4, 21.8, 21.6; HRMS (ESI) *m/z*: 352.0982 (Calcd for $C_{18}H_{19}NO_3SNa$ $[M+Na]^+$: 352.0983).

C2 Alkylation of 3a in acetone (Scheme 6)

To a solution of **3a** (333.4 mg, 1 mmol) in acetone (10 mL) was added $In(OTf)_3$ (56.2 mg, 0.1 mmol). The mixture was stirred at 100 °C (oil-bath temperature) for 16 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (30 mL). The whole was extracted with AcOEt (3 x 50 mL), washed with brine (50 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **10**.



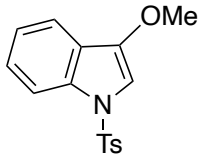
10

2-(2-Methyl-4-oxopentan-2-yl)-1-tosylindolin-3-one (10)

167 mg, 43% yield. colorless solid; mp 249–251 °C; IR ($CHCl_3$): 3019, 1717 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.02(d, *J* = 8.0 Hz, 1H), 7.66 (t, *J* = 6.9 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 4.64 (s, 1H), 2.92 (d, *J* = 18.9 Hz, 1H), 2.58 (d, *J* = 18.9 Hz, 1H), 2.29 (s, 3H), 2.19 (s, 3H), 1.01 (s, 3H), 0.98 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 207.7, 200.1, 154.5, 144.9, 136.5, 132.1, 129.8, 128.7, 127.8, 126.1, 123.5, 120.6, 71.2, 51.6, 38.1, 31.3, 25.4, 23.0, 21.6; HRMS (ESI) *m/z*: 408.1243 (Calcd for $C_{21}H_{23}NO_4SNa$ $[M+Na]^+$: 408.1246).

Control Experiment (Scheme 7)

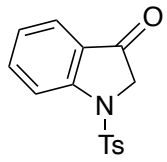
To a solution of **3a** (333.4 mg, 1 mmol) in MeCN (10 mL) was added $In(OTf)_3$ (562 mg, 1 mmol). The mixture was stirred at 100 °C for 1 h. After reaction, the mixture was cooled to room temperature and then quenched by H_2O (15 mL). The whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. After filtration, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **11** and **12**.



11

3-Methoxy-1-tosylindole (11)

216 mg, 72% yield. colorless solid; mp 148-150 °C; IR ($CHCl_3$): 3019, 1172 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 7.99 (d, J = 8.6 Hz, 1H), 7.69 (d, J = 8.6 Hz, 2H), 7.49 (d, J = 7.5 Hz, 1H), 7.33 (ddd, J = 1.2, 8.0, 8.0 Hz, 1H), 7.21 (t, J = 8.1 Hz, 1H), 7.16 (d, J = 8.6 Hz, 2H), 6.90 (s, 1H), 3.87 (s, 3H), 2.31 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 146.8, 144.7, 134.8, 134.3, 129.7, 126.8, 125.8, 124.7, 123.3, 118.6, 114.3, 104.0, 57.9, 21.6; HRMS (ESI) *m/z*: 324.0668 (Calcd for $C_{16}H_{15}NO_3SNa$ $[M+Na]^+$: 324.0670).



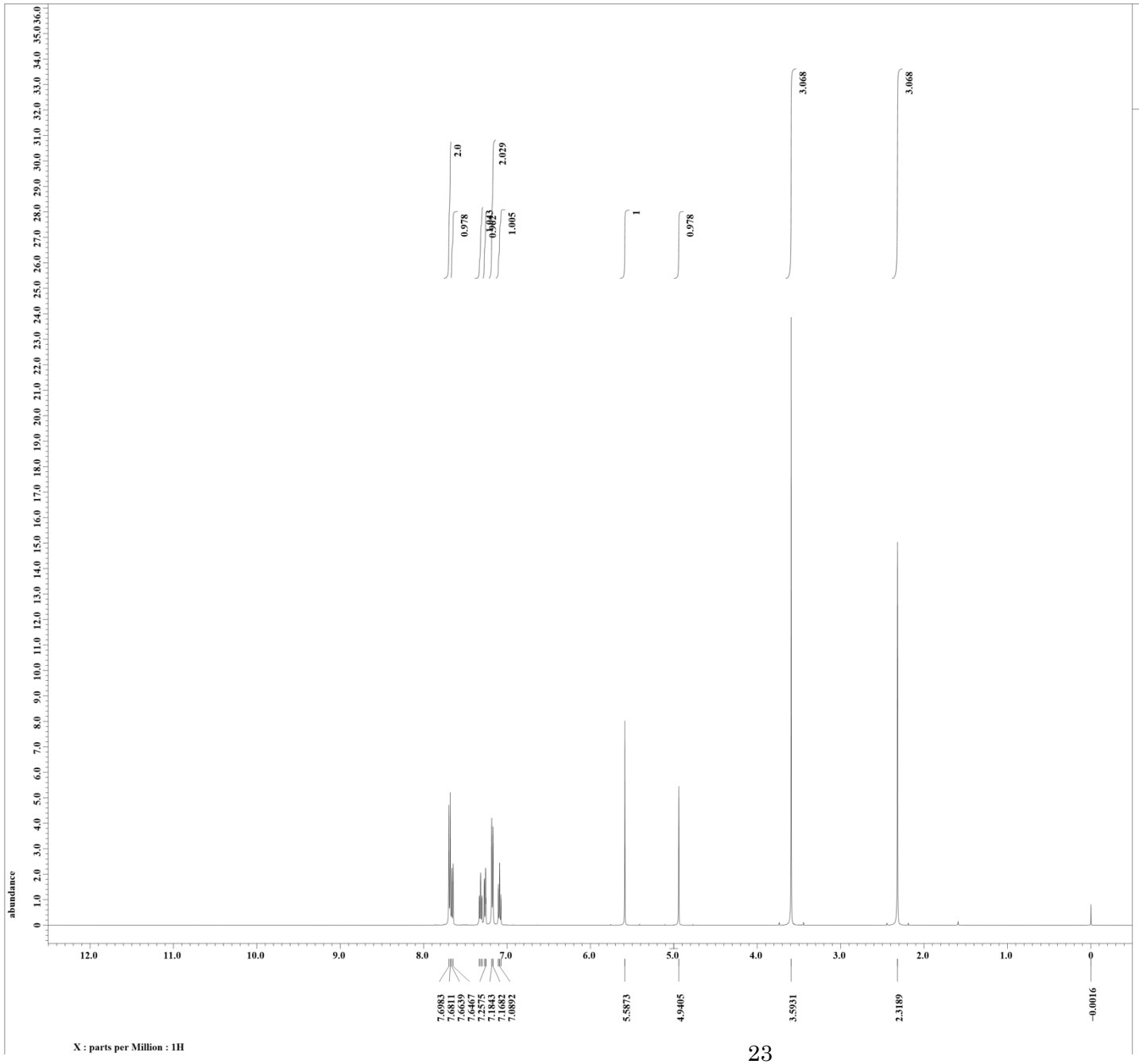
12

1-Tosylindolin-3-one (12)

21 mg, 7% yield. colorless solid; mp 170-174 °C; IR ($CHCl_3$): 3019, 1719 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ: 8.03 (d, J = 8.6 Hz, 1H), 7.71 (d, J = 8.0 Hz, 2H), 7.65 (t, J = 8.1 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H), 7.17 (t, J = 7.5 Hz, 1H), 4.13 (s, 2H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 194.9, 153.7, 145.3, 137.4, 133.6, 130.2, 127.2, 125.1, 124.5, 124.1, 116.0, 56.2, 21.7; HRMS (ESI) *m/z*: 310.0517 (Calcd for $C_{15}H_{13}NO_3SNa$ $[M+Na]^+$: 317.0514).

2. Supplementary References

(S1) Hodson, H. F.; Madge, D. J.; Slawin, A. N. Z.; Widdowson, D. A.; Williams, D. J. *Tetrahedron* **1994**, *50*, 1899.



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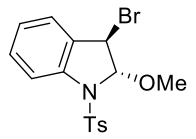

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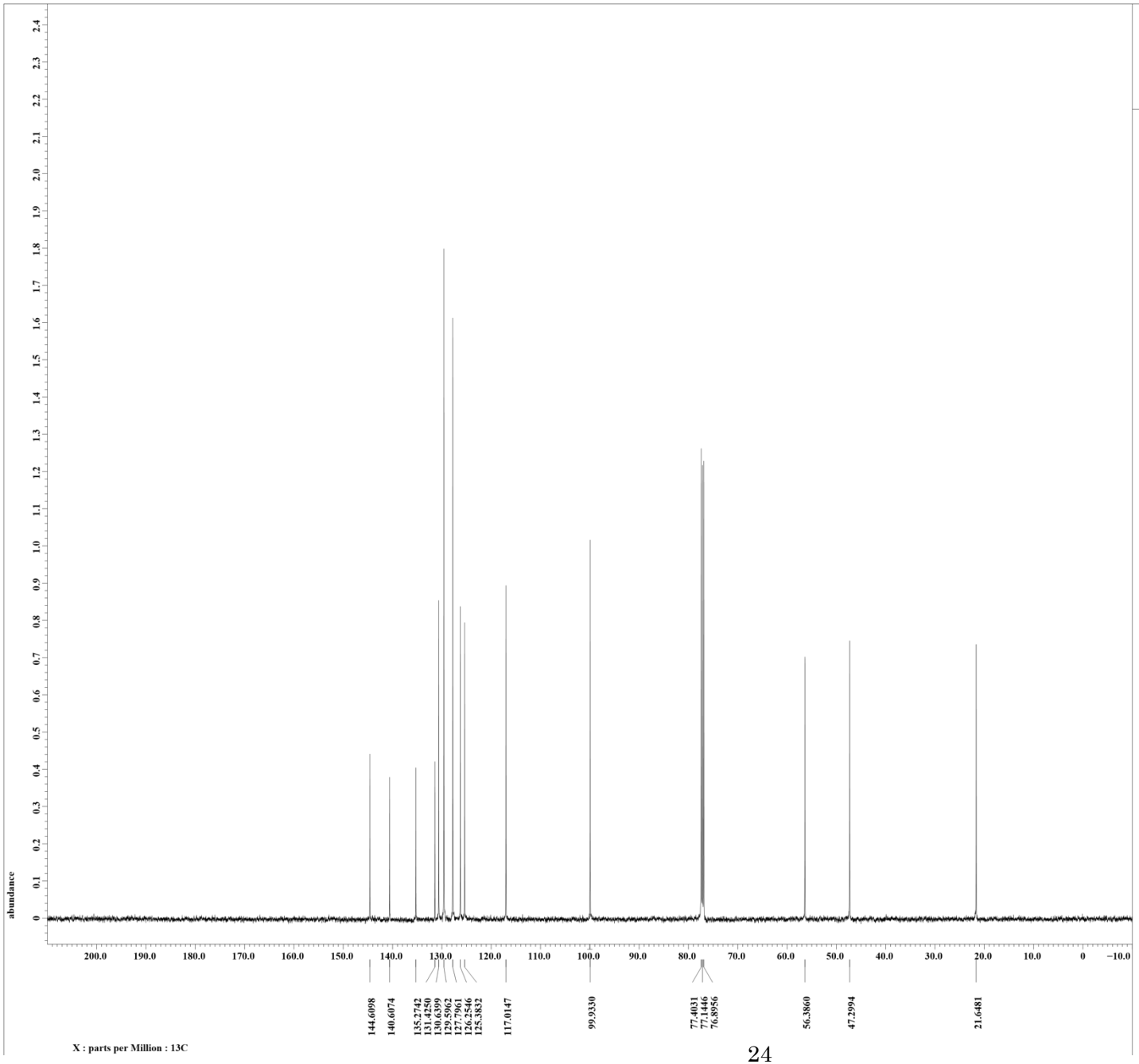

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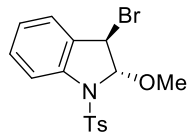

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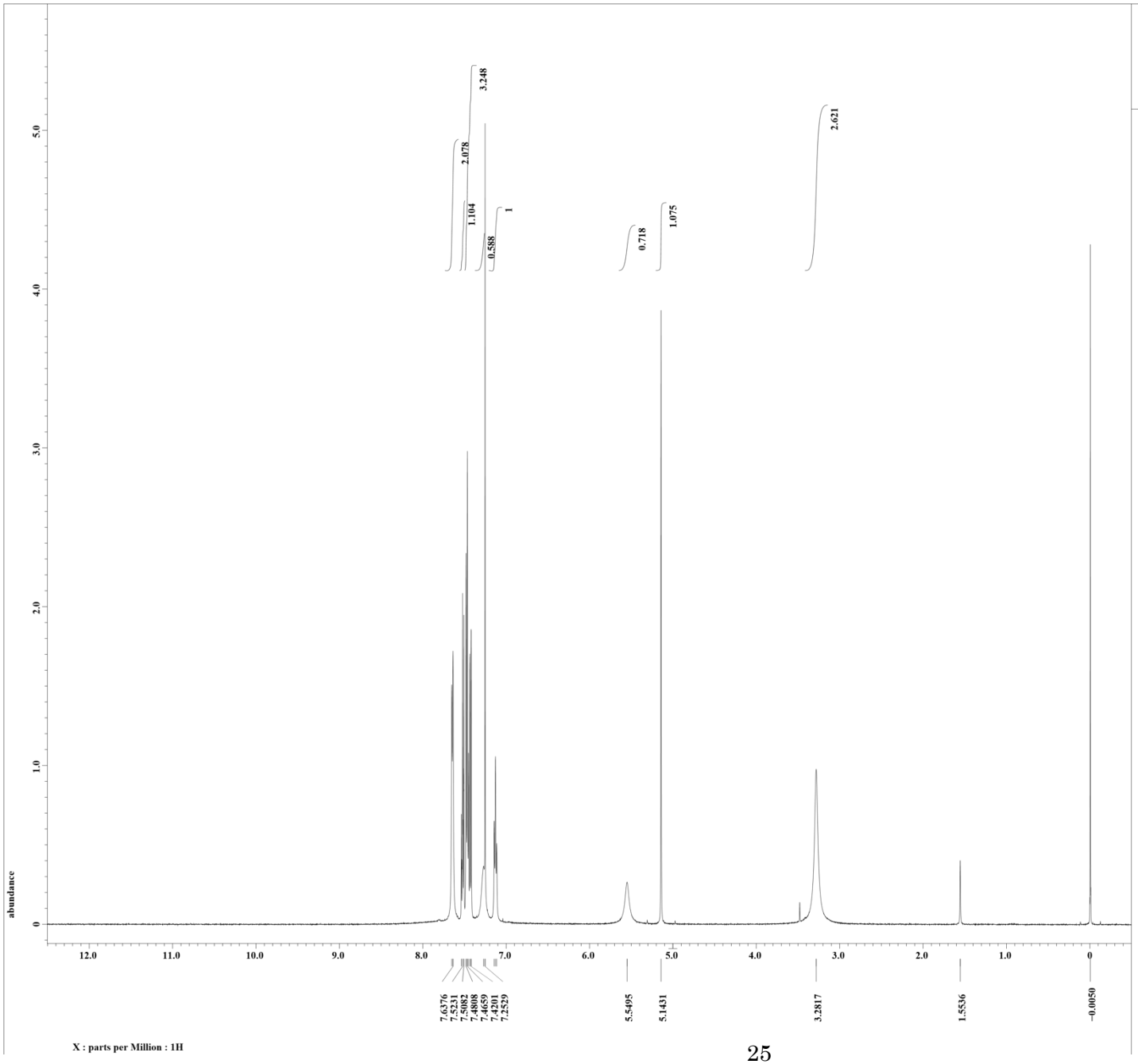

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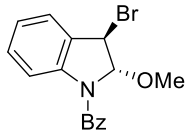
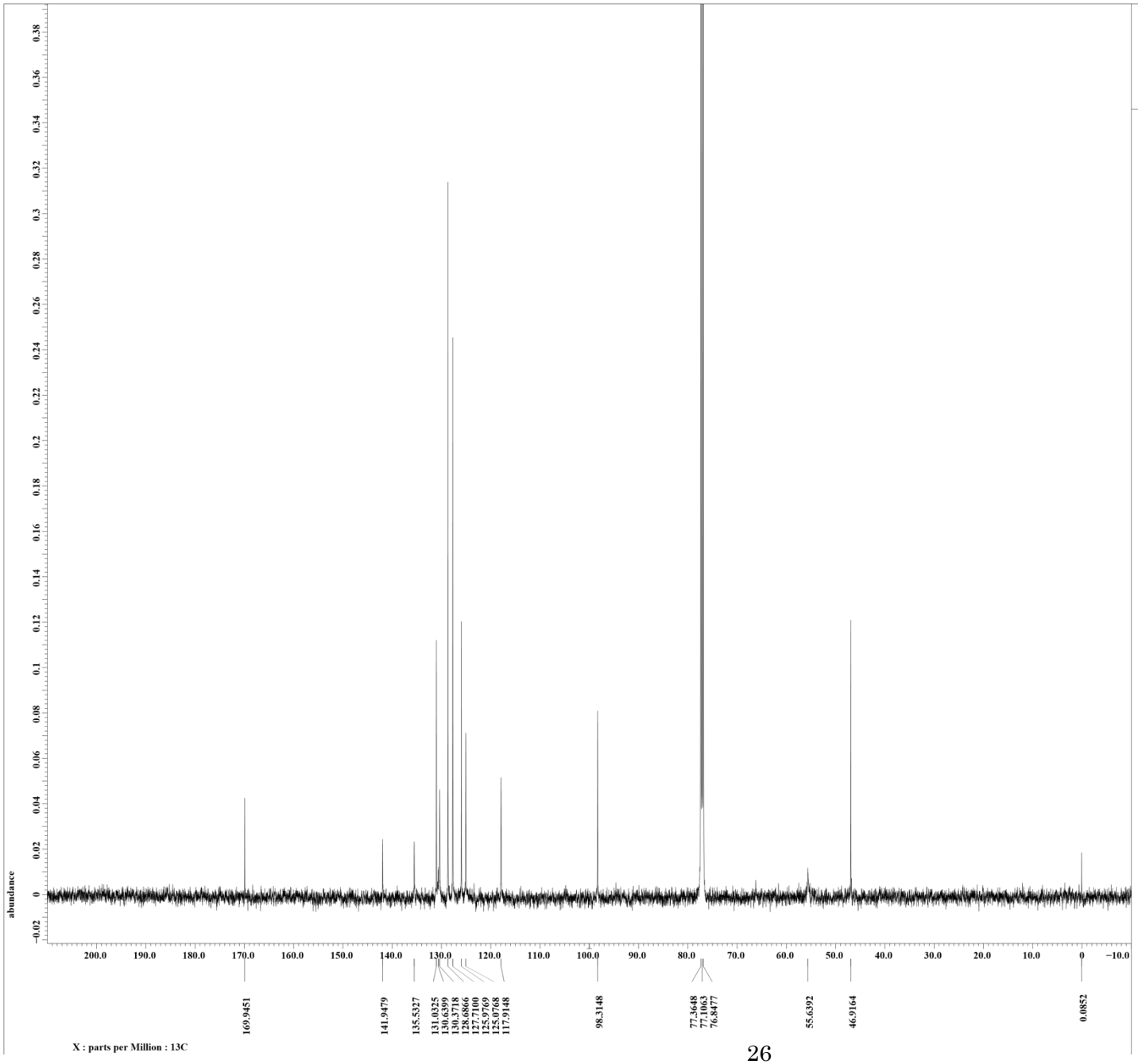


2b



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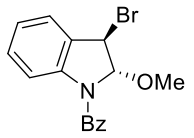

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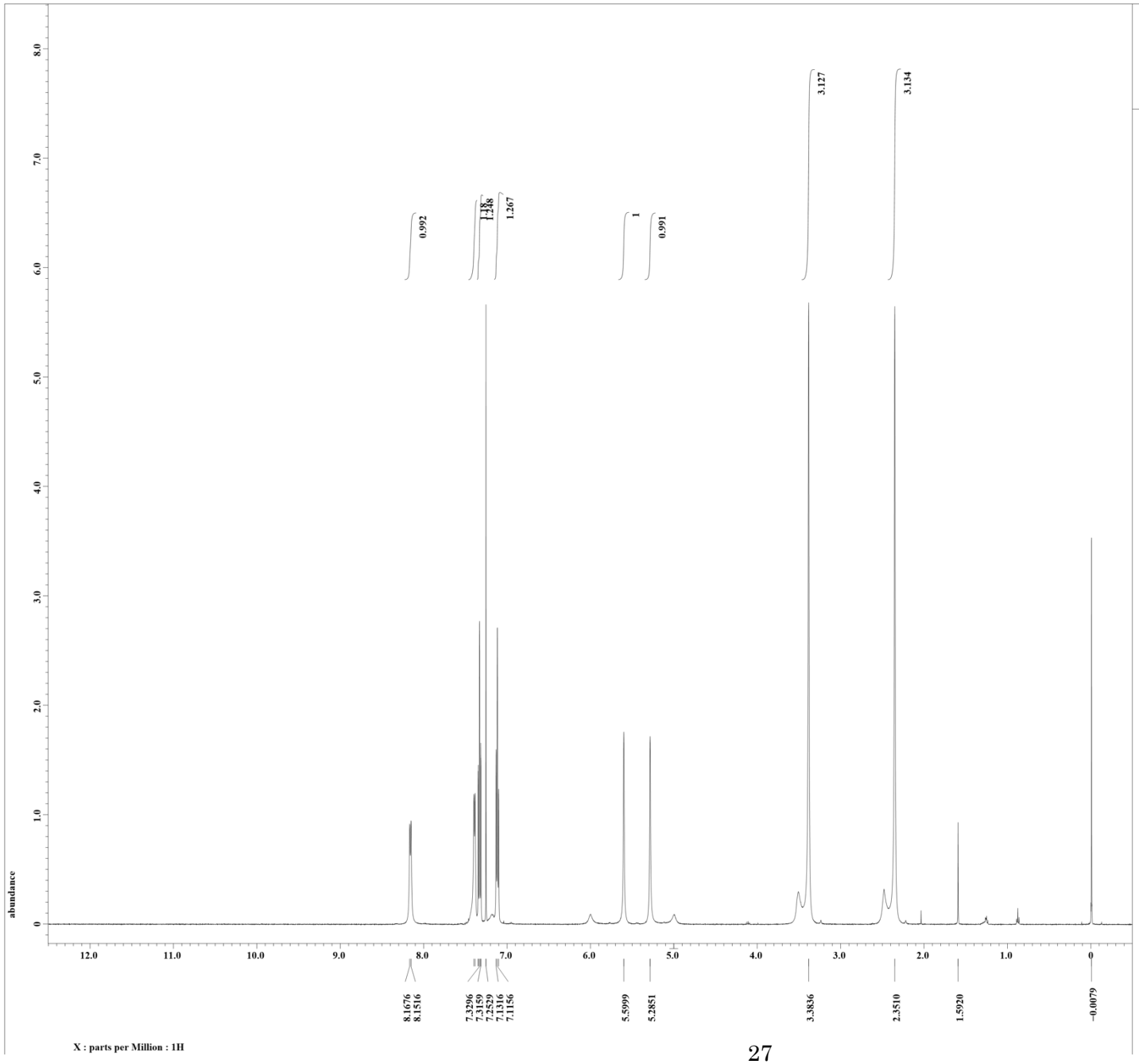

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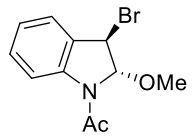
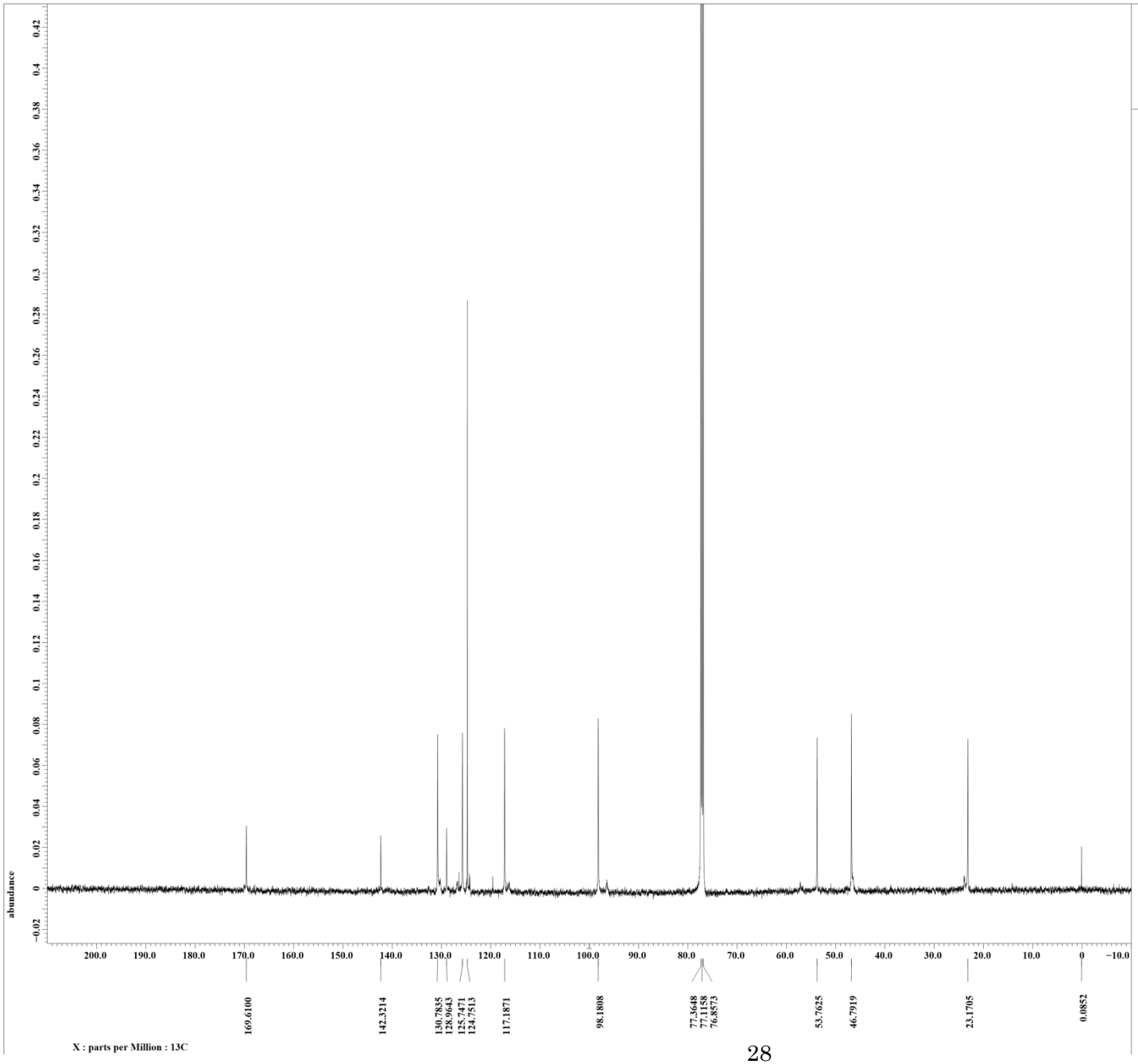
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

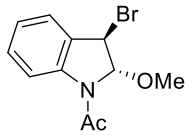
X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.4[dC]



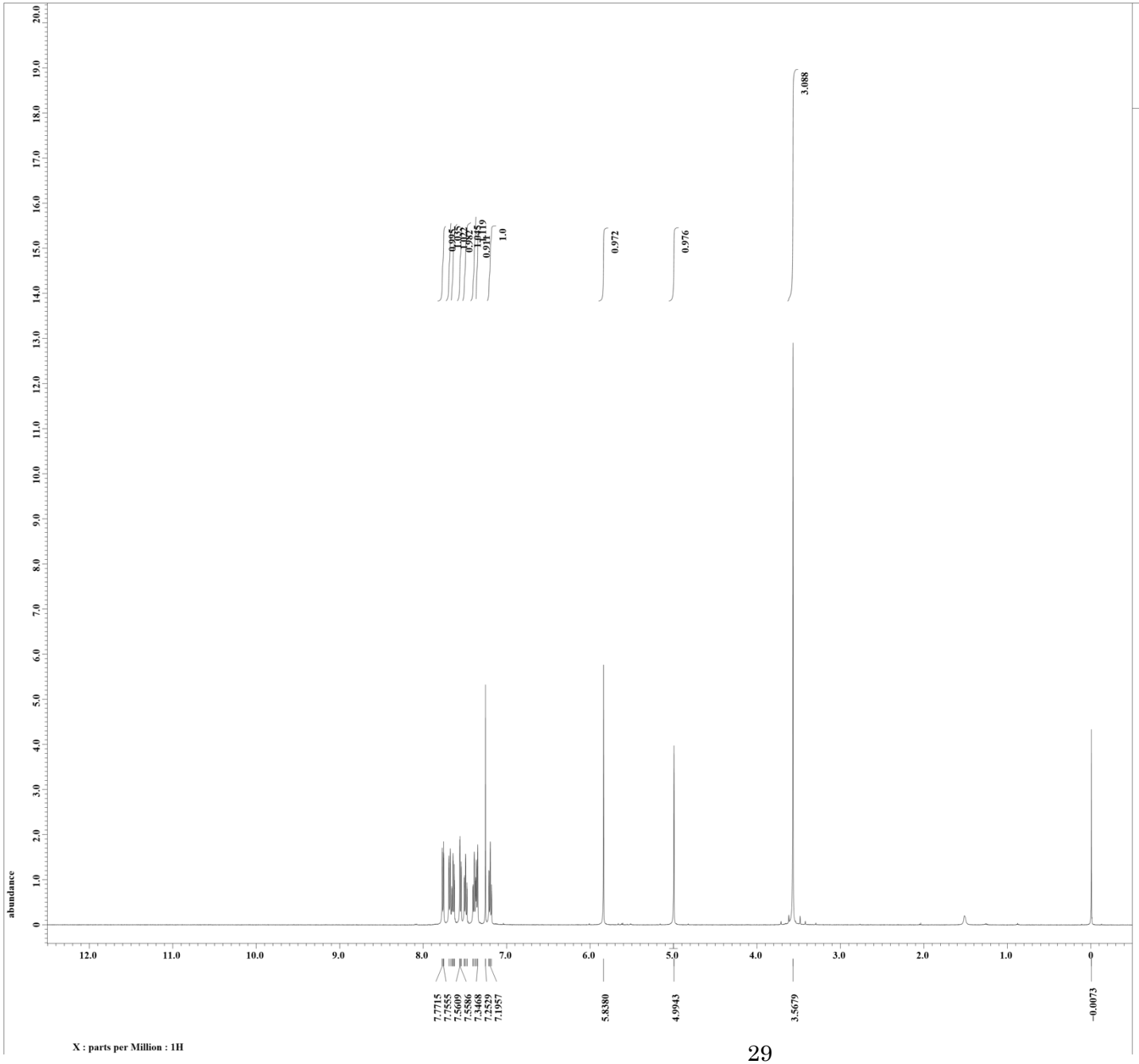
2d




```
Filename          = YT-20-01-27-6-12.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 01276
Solvent           = CHLOROFORM-D
Creation_time     = 28-JAN-2020 08:22:30
Revision_time     = 30-JAN-2020 04:06:28
Current_time      = 30-JAN-2020 04:06:48

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 16016
Total_scans       = 16016

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 23.8[dC]
```



2d

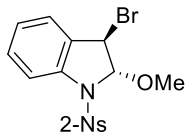


Filename = YT-20-01-29-6-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = Ns
Solvent = CHLOROFORM-D
Creation_time = 29-JAN-2020 12:25:18
Revision_time = 30-JAN-2020 03:16:22
Current_time = 30-JAN-2020 03:16:37

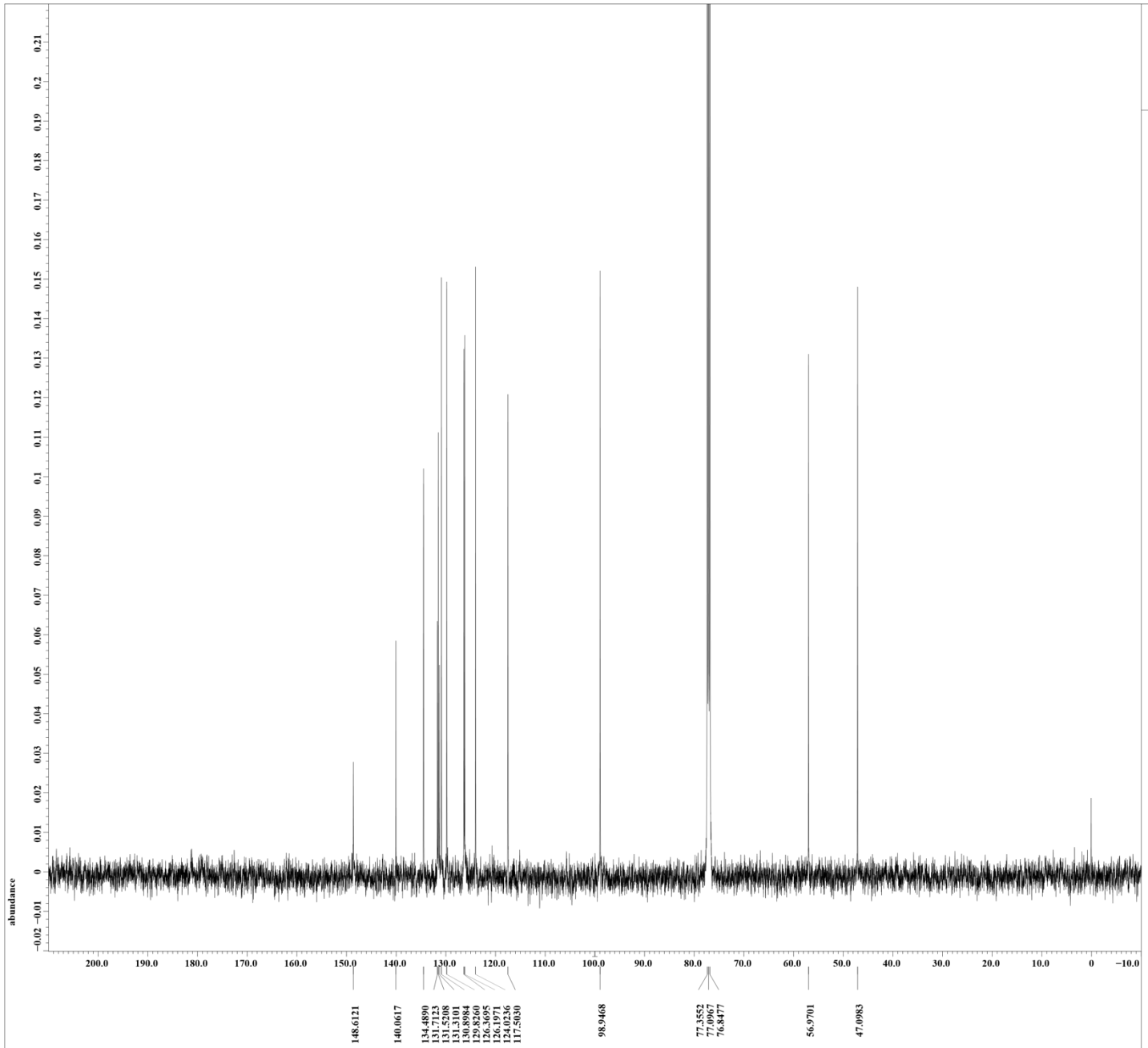
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

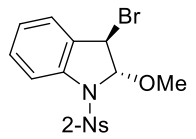
X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.5[dC]



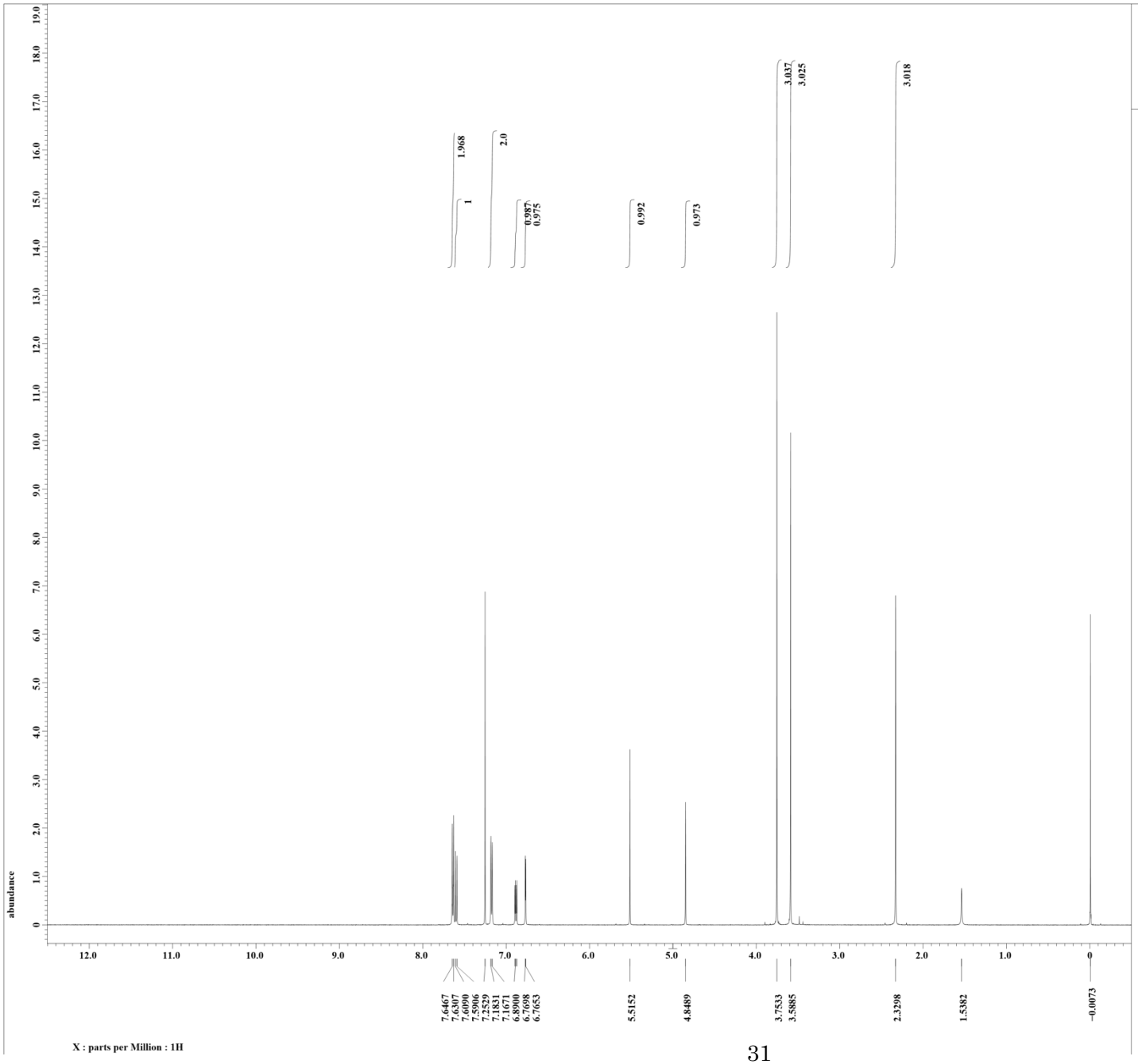
2e



```
Filename          = YT-20-01-29-6-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = Ns
Solvent           = CHLOROFORM-D
Creation_time     = 29-JAN-2020 14:34:10
Revision_time     = 30-JAN-2020 03:19:30
Current_time      = 30-JAN-2020 03:19:40

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 2695
Total_scans       = 2695

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 24.3[dC]
```



2e

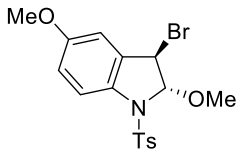


Filename = YT-20-01-14-2-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 5OMe
Solvent = CHLOROFORM-D
Creation_time = 14-JAN-2020 08:36:25
Revision_time = 30-JAN-2020 03:27:56
Current_time = 30-JAN-2020 03:28:29

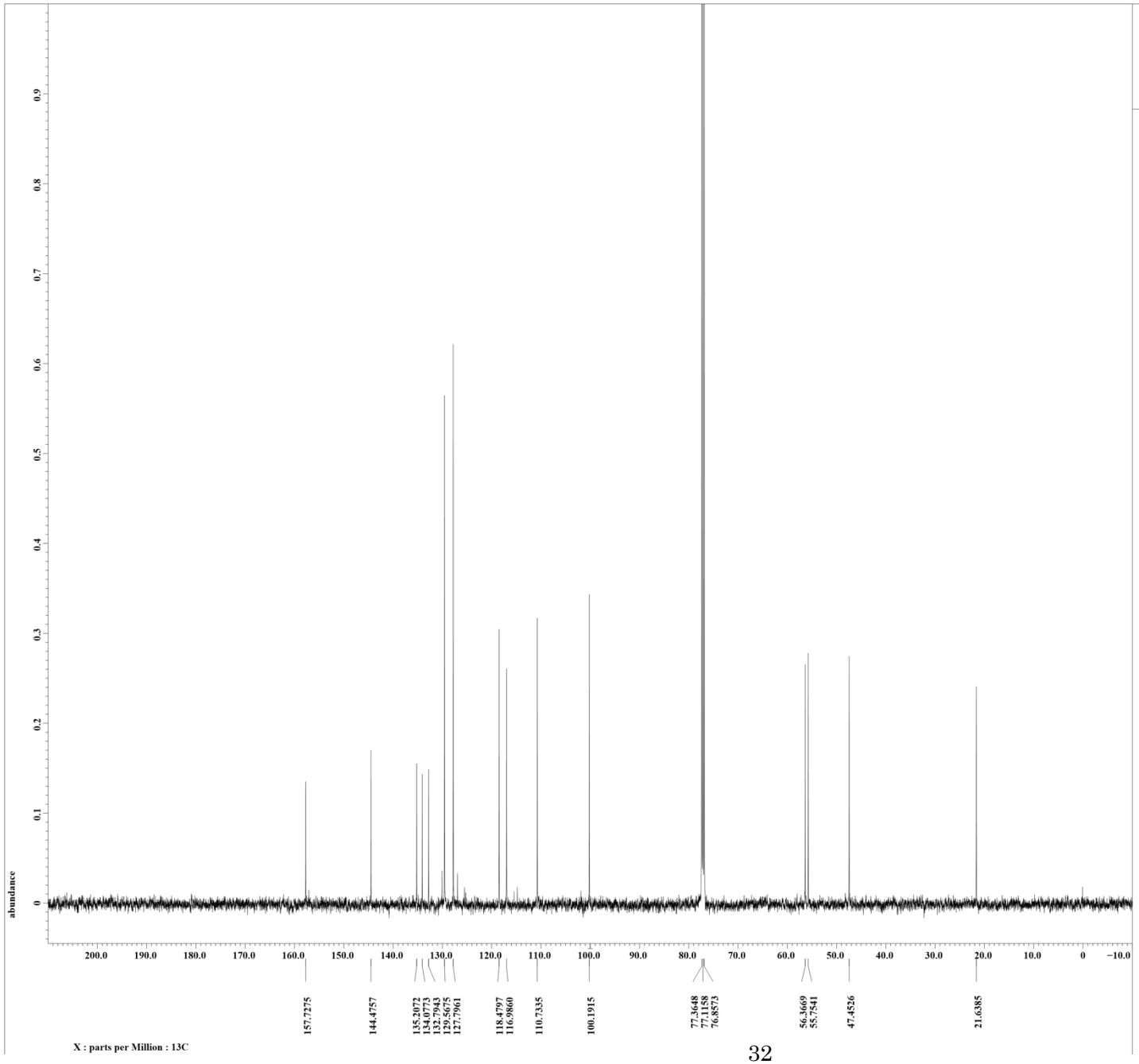
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23[dC]



2f

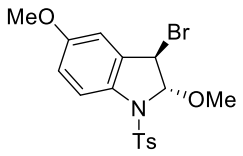


Filename = YT-20-01-28-6-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 5OMe
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 15:30:56
Revision_time = 30-JAN-2020 03:29:31
Current_time = 30-JAN-2020 03:30:51

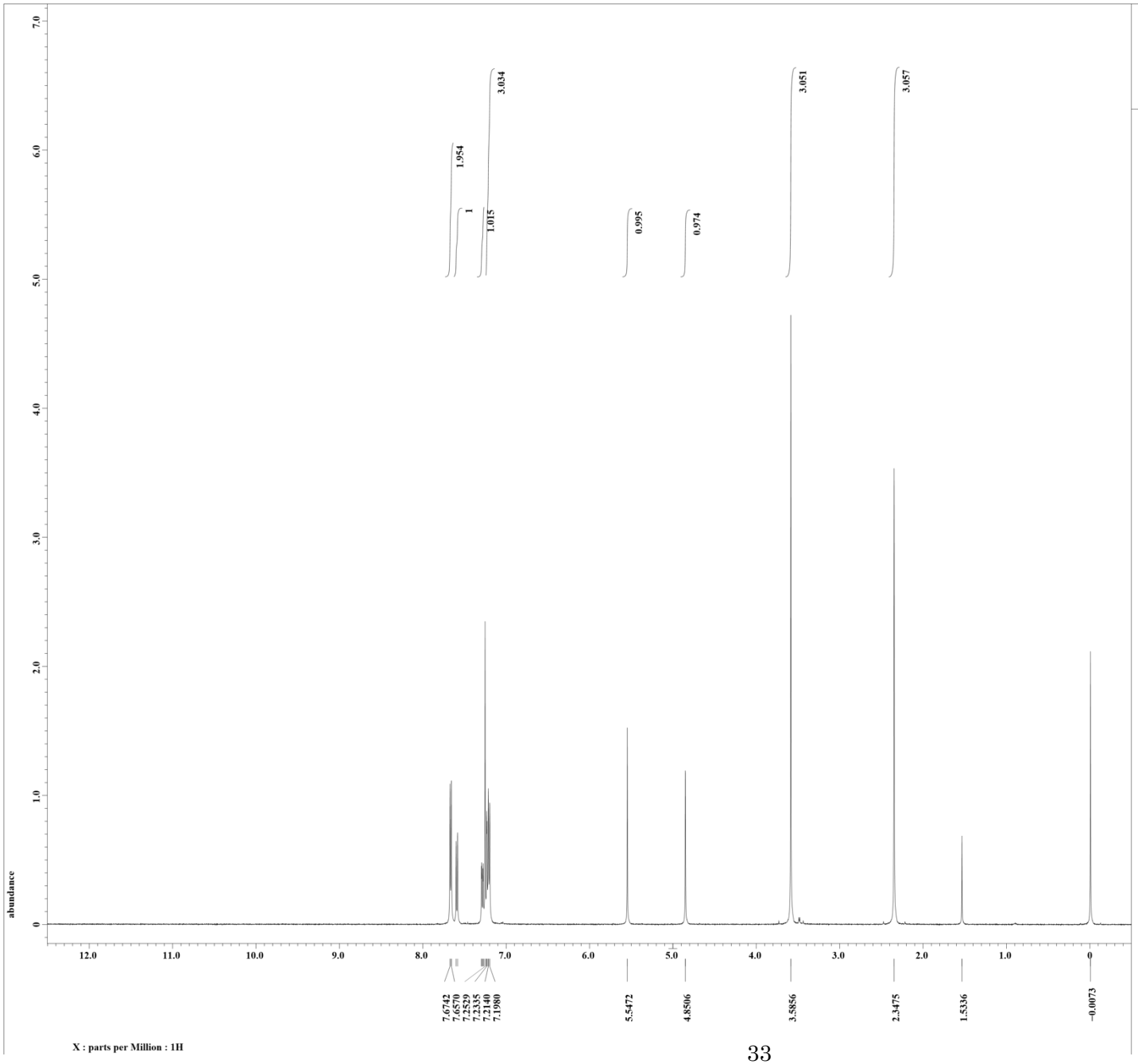
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 871
Total_scans = 871

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
RecVr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.1[dC]



2f

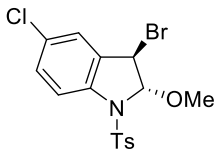


Filename = YT-20-01-16-6-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SC1
Solvent = CHLOROFORM-D
Creation_time = 16-JAN-2020 18:36:53
Revision_time = 30-JAN-2020 03:36:44
Current_time = 30-JAN-2020 03:36:54

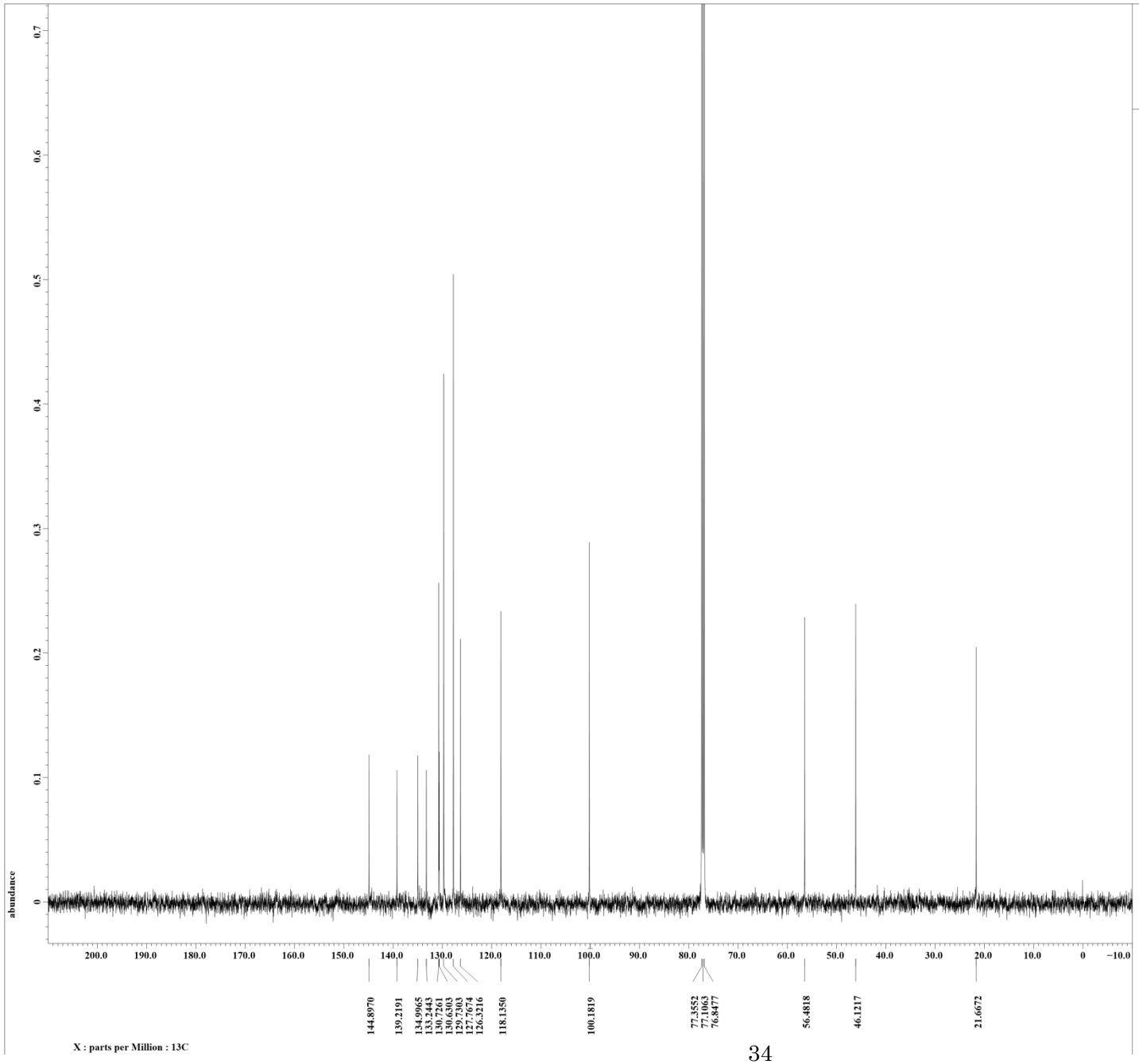
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.2[dC]



2g

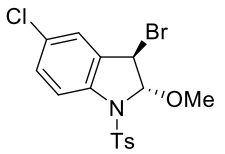


Filename = YT-20-01-28-5-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 5C1
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 14:44:39
Revision_time = 30-JAN-2020 03:38:09
Current_time = 30-JAN-2020 03:38:21

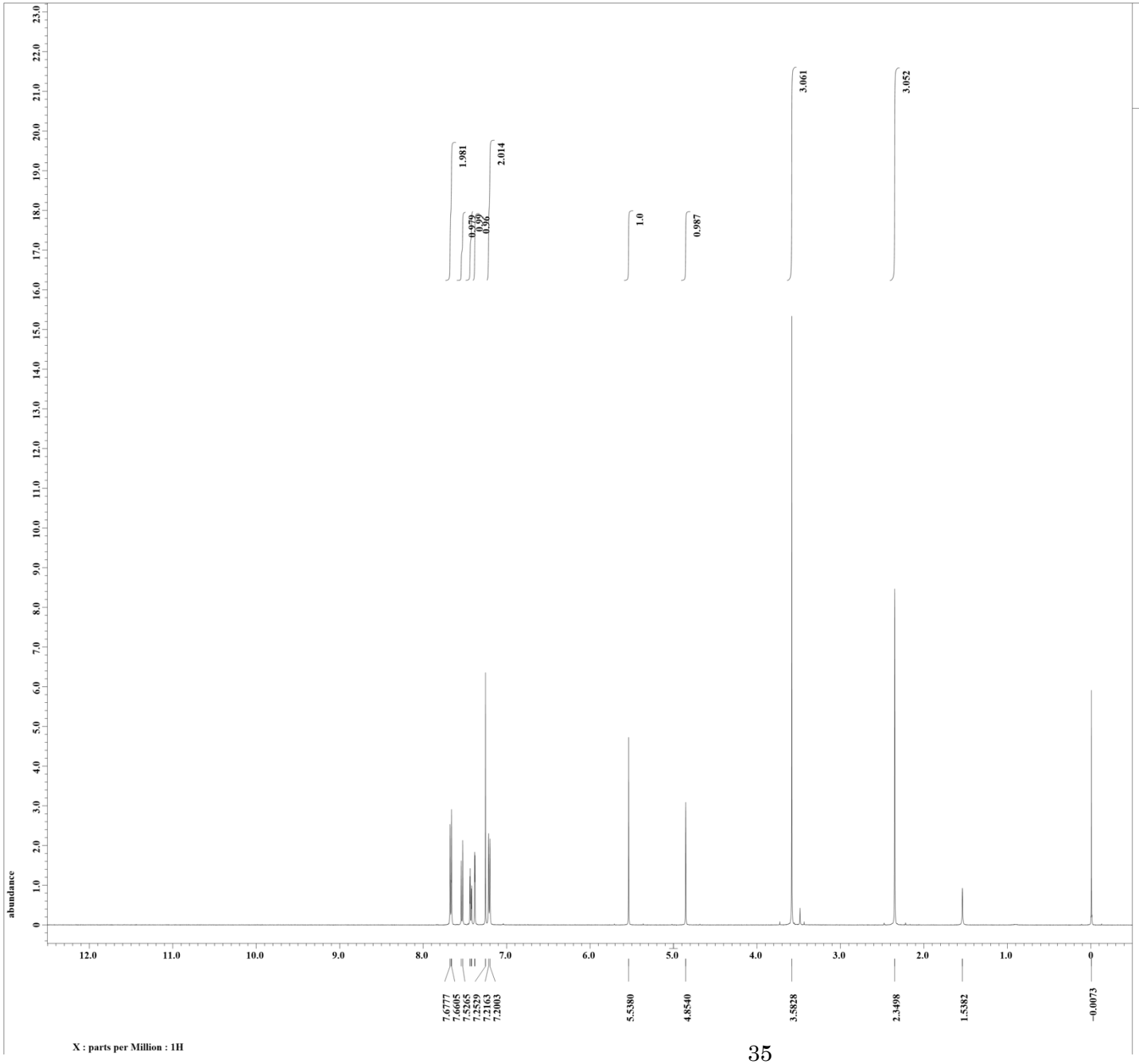
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 783
Total_scans = 783

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
RecVr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24[dC]



2g

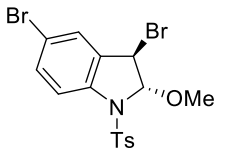


Filename = YT-20-01-14-3-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 5Br
Solvent = CHLOROFORM-D
Creation_time = 14-JAN-2020 12:31:44
Revision_time = 30-JAN-2020 03:21:41
Current_time = 30-JAN-2020 03:21:52

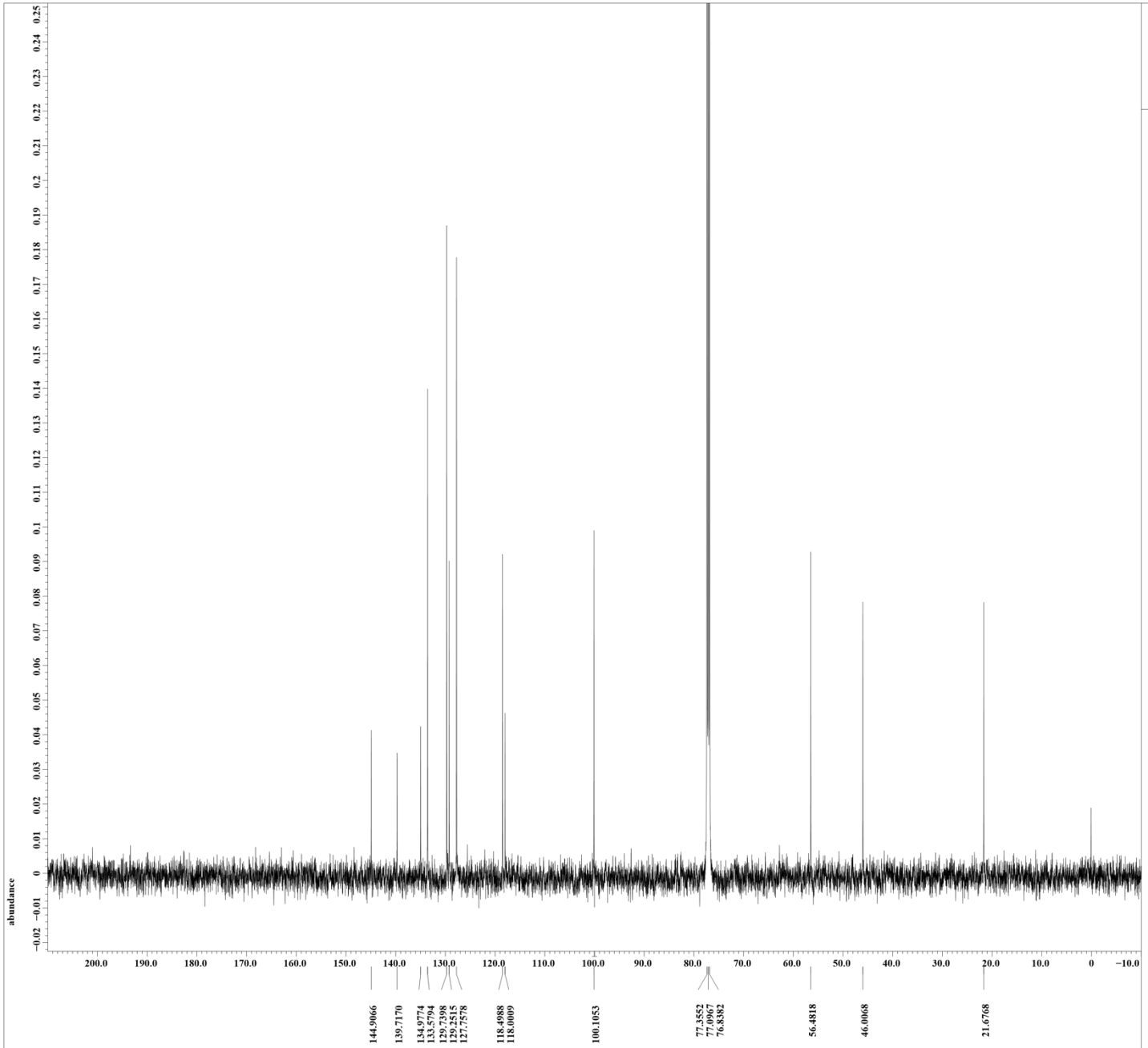
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.1[dC]



2h

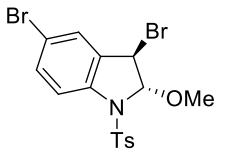


Filename = YT-20-01-14-3-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 5Br
Solvent = CHLOROFORM-D
Creation_time = 14-JAN-2020 14:07:42
Revision_time = 30-JAN-2020 03:22:35
Current_time = 30-JAN-2020 03:22:49

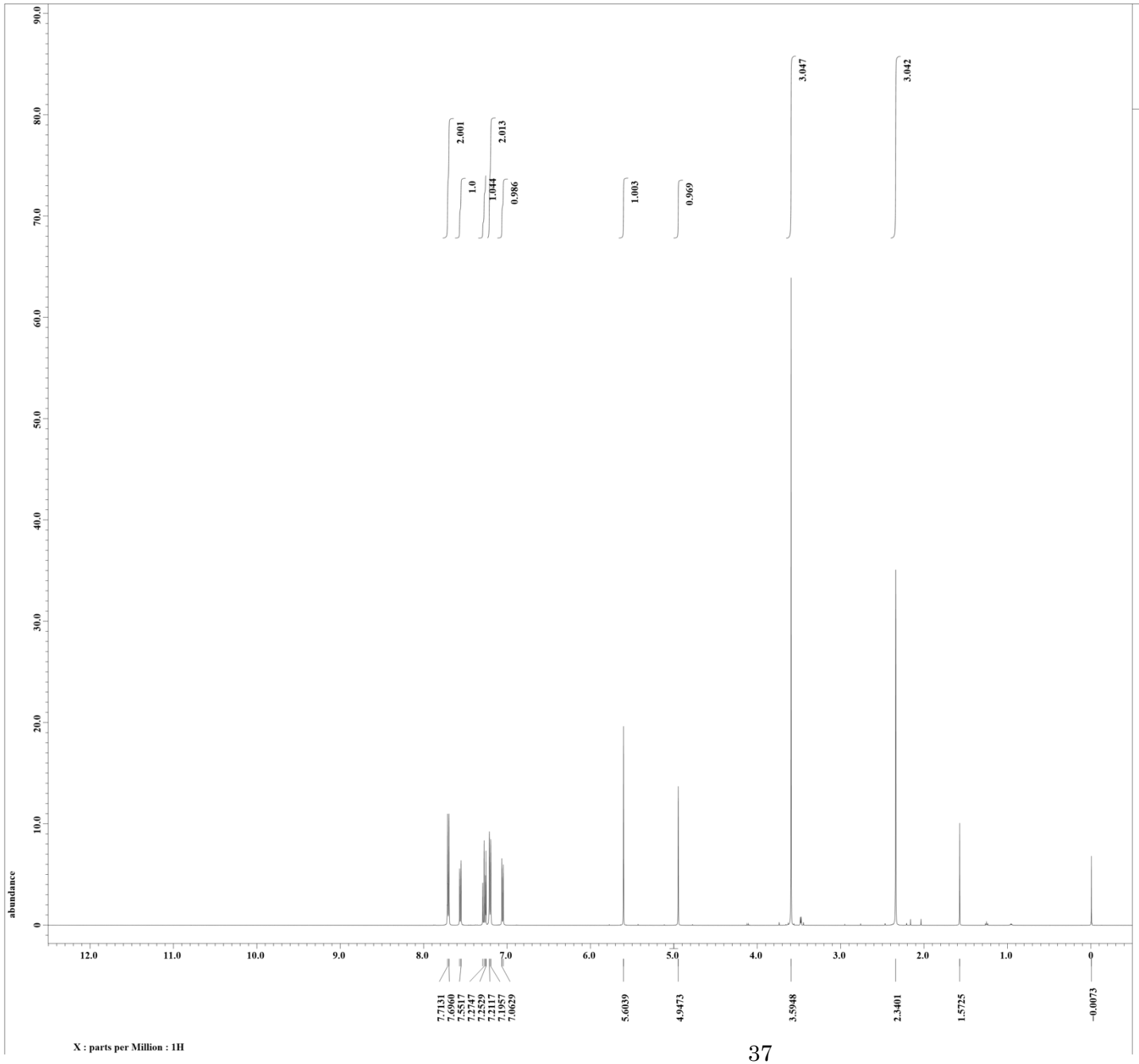
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 2000
Total_scans = 2000

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 23.9[dC]



2h

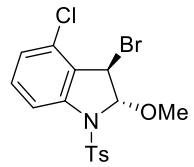


Filename = YT-20-01-18-4-9.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 4Cl
Solvent = CHLOROFORM-D
Creation_time = 18-JAN-2020 16:45:13
Revision_time = 30-JAN-2020 03:34:21
Current_time = 30-JAN-2020 03:34:35

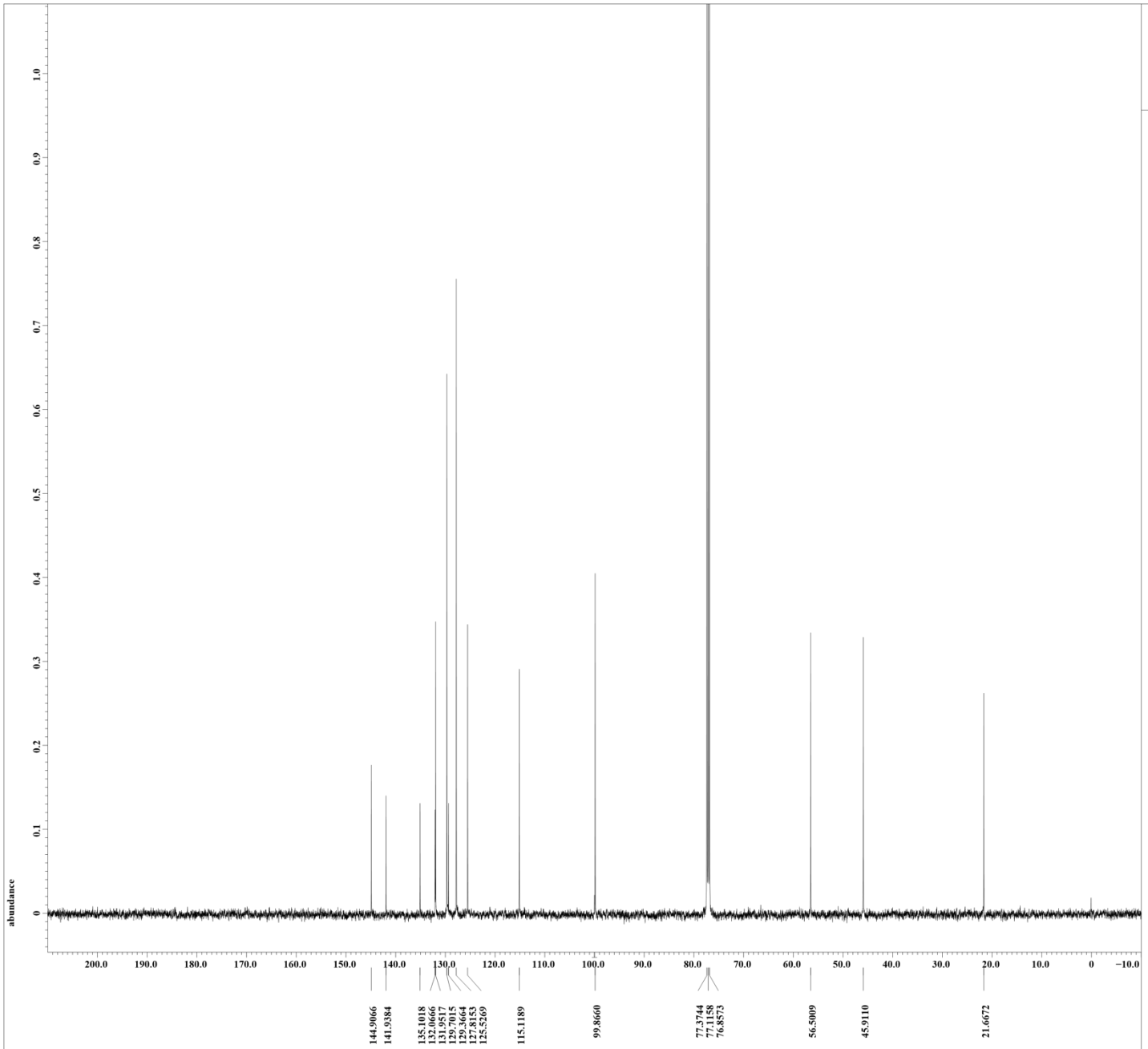
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 8
Total_scans = 8

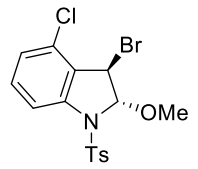
X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.3[dC]



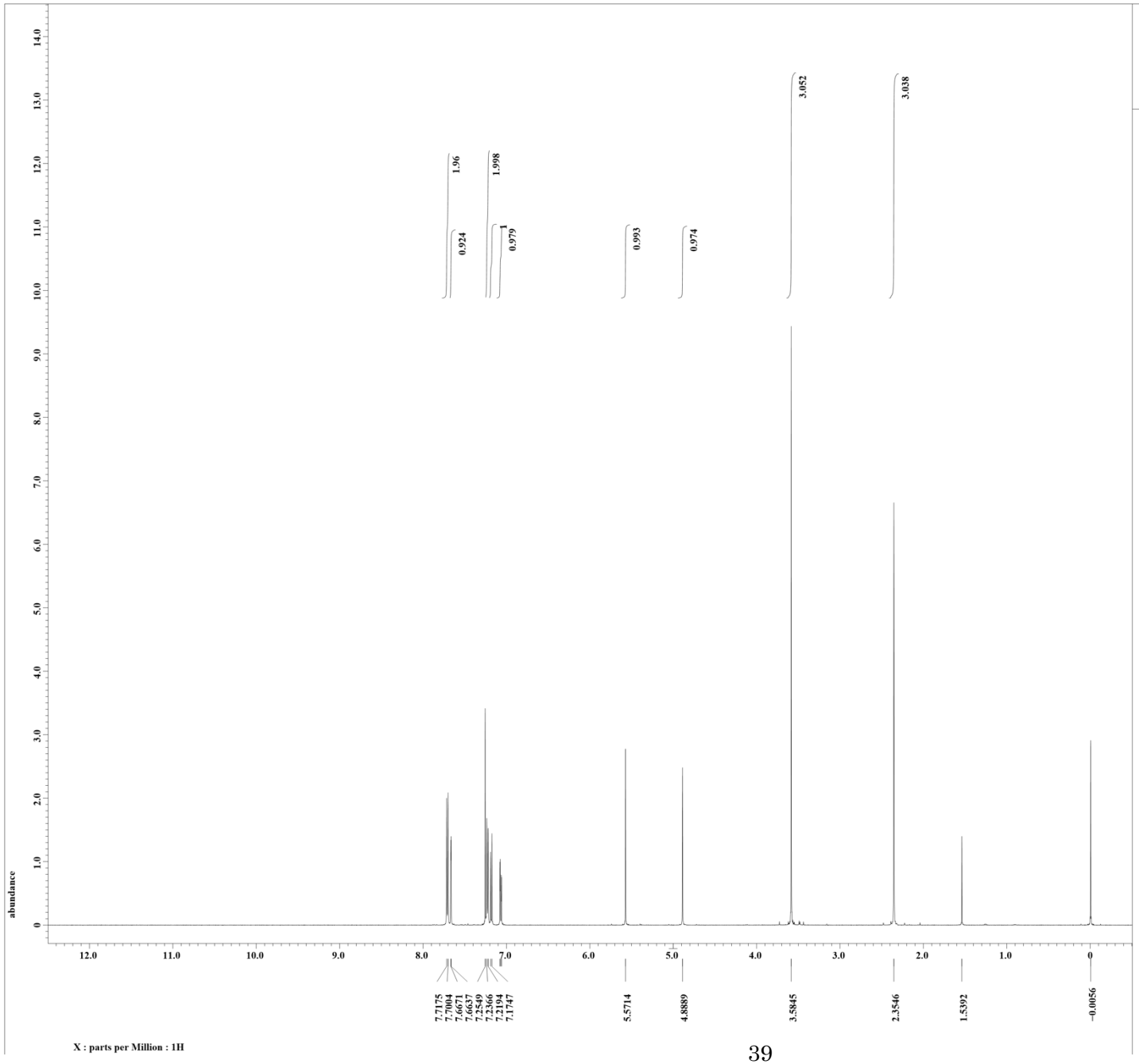
2i



```
Filename          = YT-20-01-18-4-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 4Cl
Solvent           = CHLOROFORM-D
Creation_time     = 18-JAN-2020 17:53:58
Revision_time     = 30-JAN-2020 03:35:19
Current_time      = 30-JAN-2020 03:35:31

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1418
Total_scans       = 1418

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 24[dC]
```

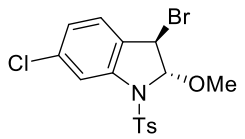


2i

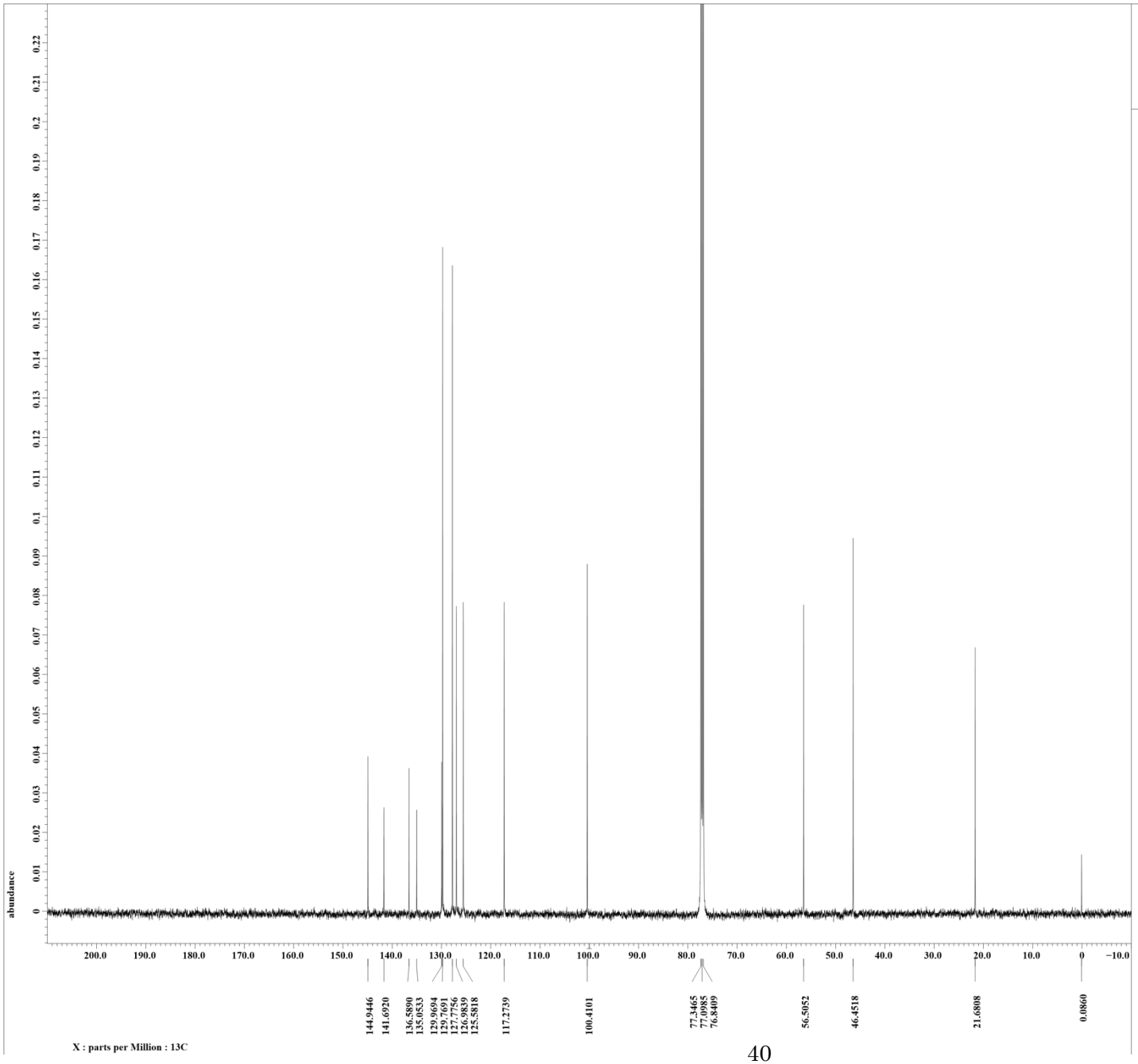


JEOL

```
Filename          = YT-20-01-21-10-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = 6 1cr
Solvent           = CHLOROFORM-D
Creation_time     =  7-APR-2000 18:59:15
Revision_time     = 30-JAN-2020 03:46:13
Current_time      = 30-JAN-2020 03:46:21

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 22.8[dC]
```

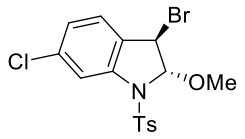


2j

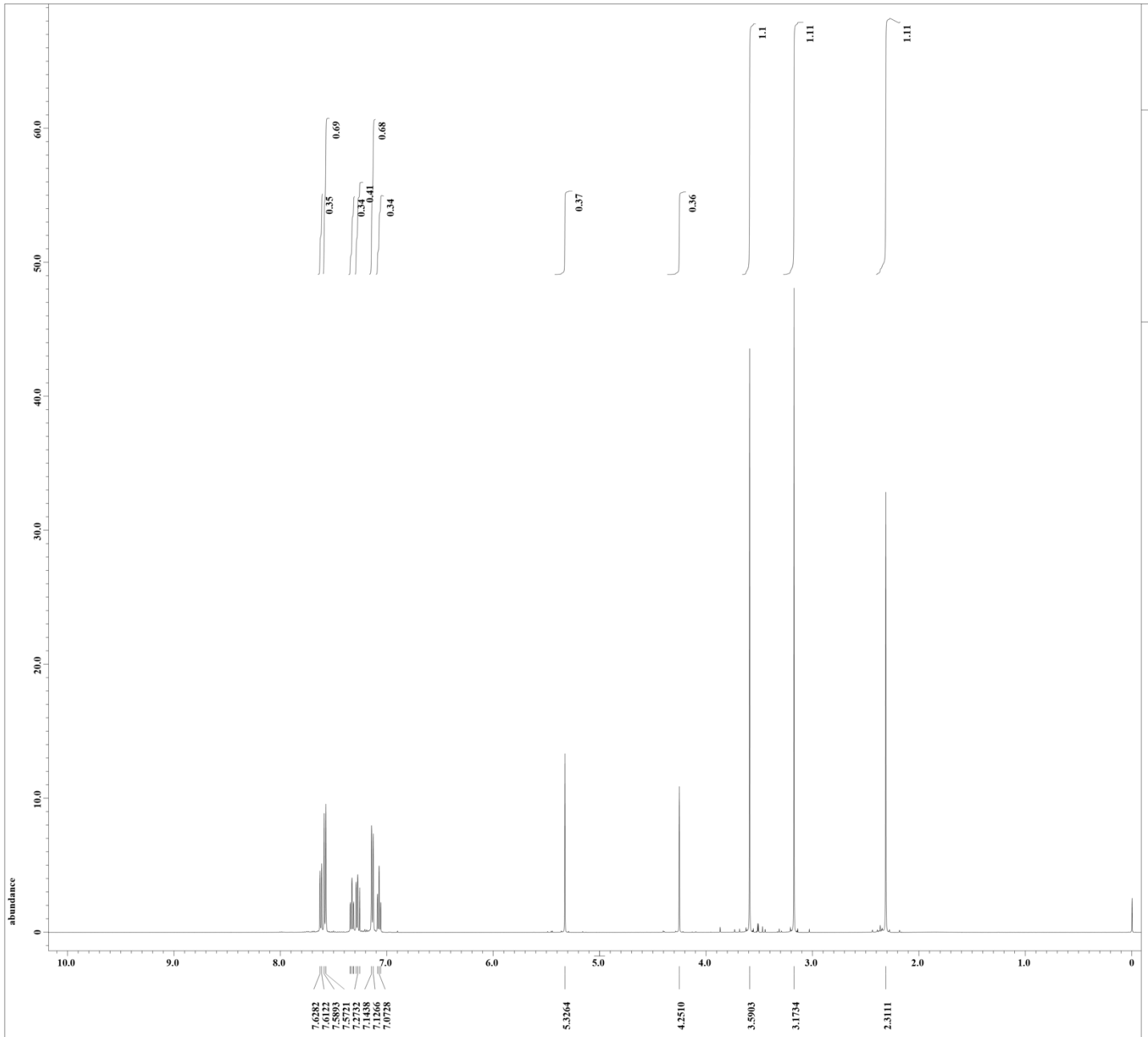


JEOL

```
Filename          = YT-20-01-21-10-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 6 1or
Solvent           = CHLOROFORM-D
Creation_time     =  8-APR-2000 10:31:13
Revision_time     = 30-JAN-2020 03:44:51
Current_time      = 30-JAN-2020 03:45:12

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 19723
Total_scans       = 19723

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 25[dC]
```

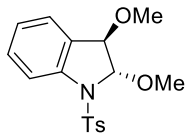


2j

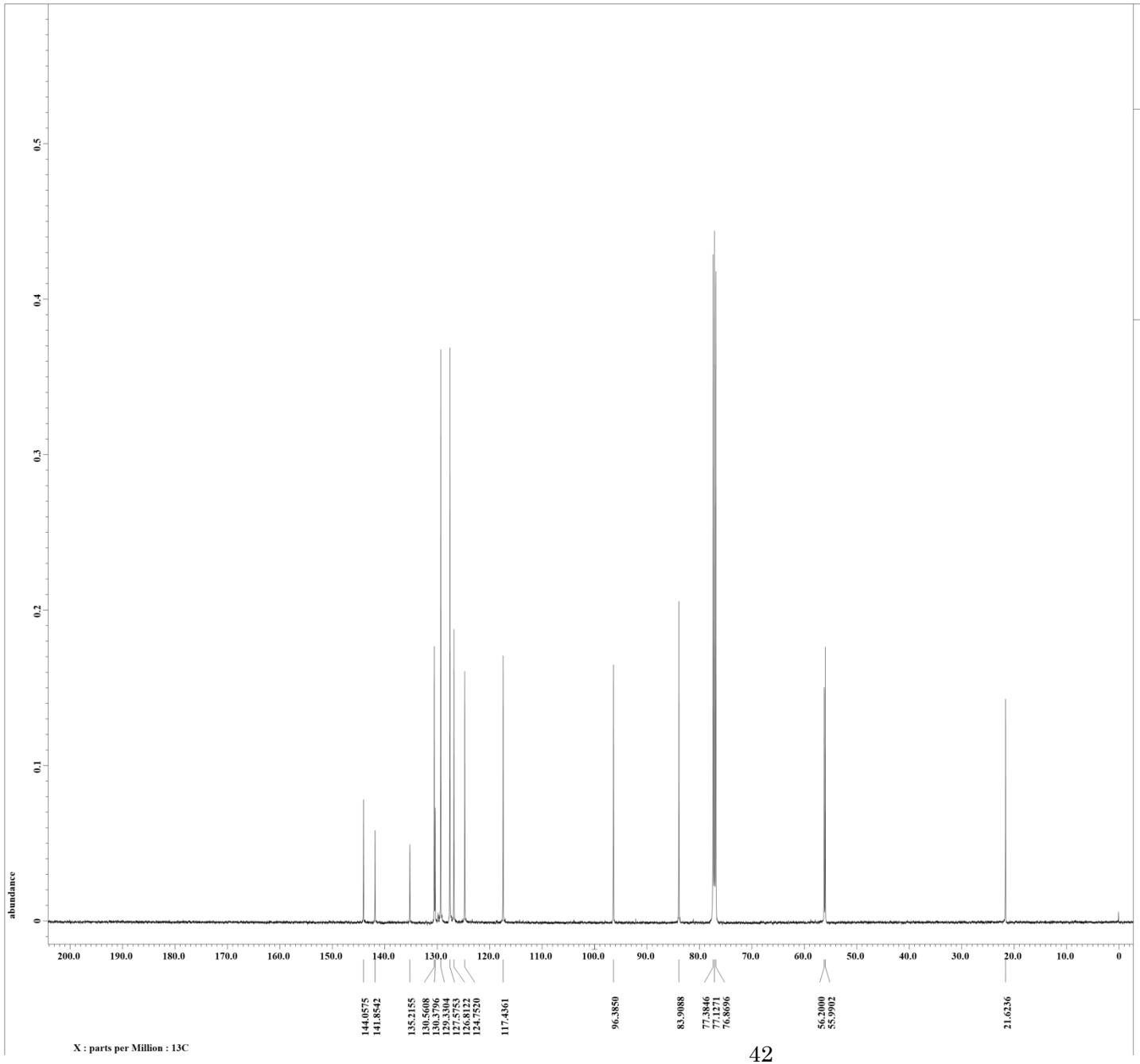


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2019-1224-1.jdf
```

```
Filename          = TA2019-1224-12.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#599878
Solvent           = CHLOROFORM-D
Creation_time     = 10-MAR-2000 18:29:32
Revision_time     =  2-FEB-2020 11:51:44
Current_time      =  2-FEB-2020 11:52:09

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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           = TRUE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 22.2[dC]
```

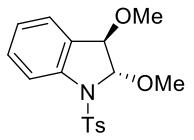


3a

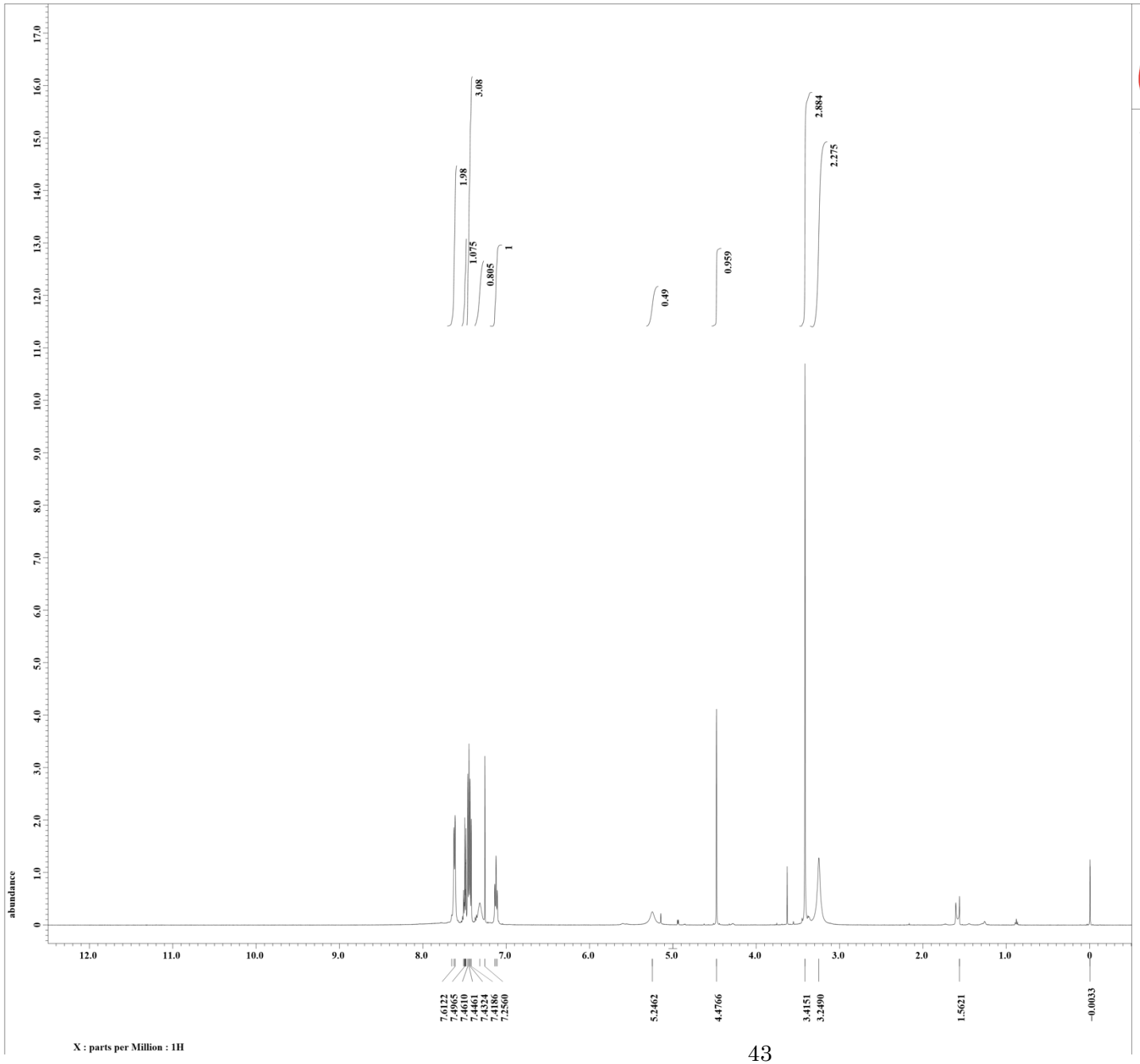



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2019-1224-5.jdf
```

```
Filename          = TA2019-1224-10.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#600565
Solvent           = CHLOROFORM-D
Creation_time     = 11-MAR-2000 08:55:17
Revision_time     =  2-FEB-2020 11:52:48
Current_time      =  2-FEB-2020 11:53:16

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 18315
Total_scans       = 18315

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.3[dC]
```



3a

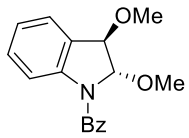


Filename = YT-20-01-31-4-12.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = Bz
Solvent = CHLOROFORM-D
Creation_time = 17-APR-2000 20:27:37
Revision_time = 2-FEB-2020 15:21:10
Current_time = 2-FEB-2020 15:21:28

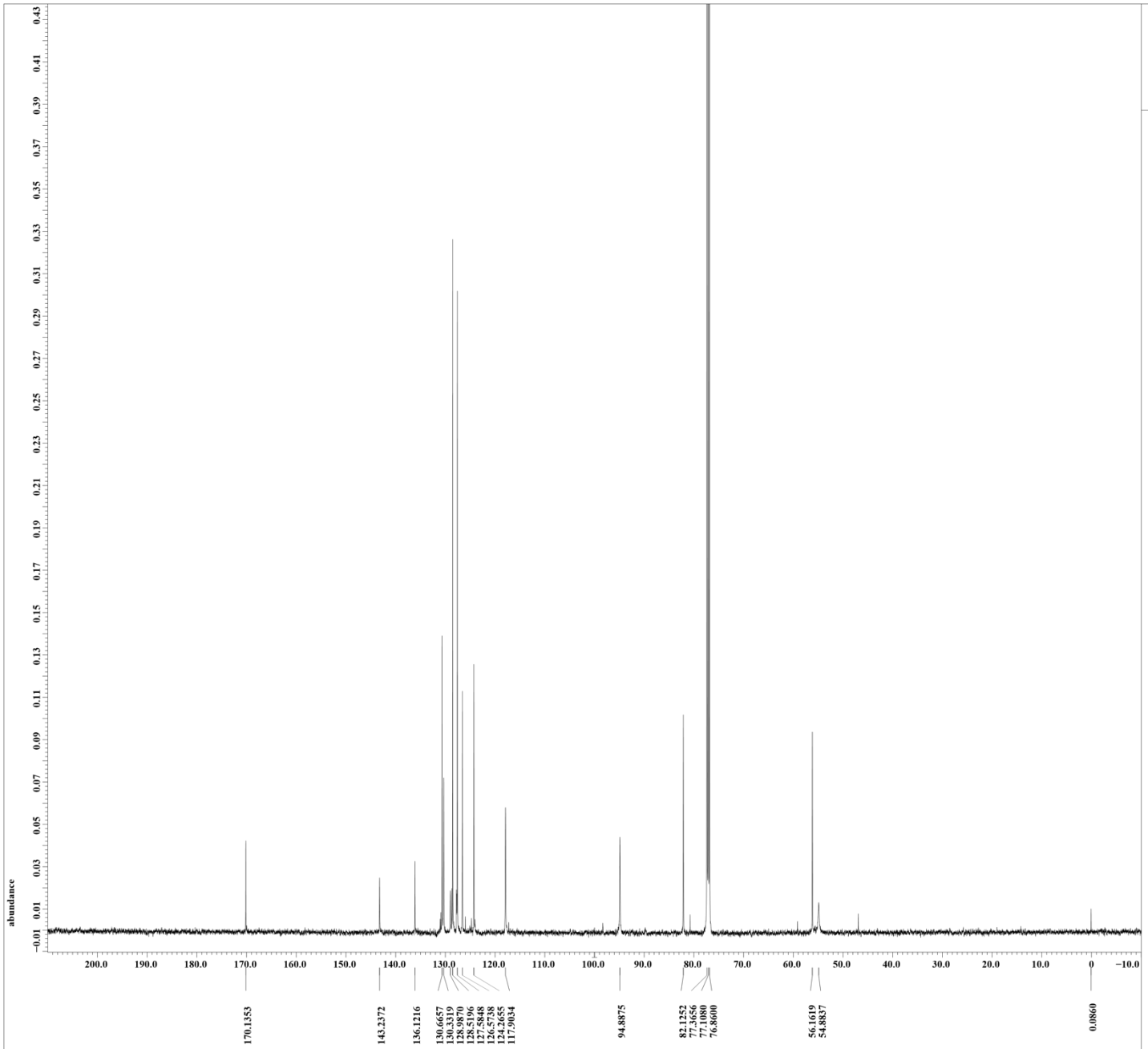
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 22.4[dC]



3b



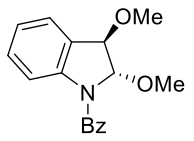
JEOL

Filename = YT-20-01-31-4-12.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = Bz
Solvent = CHLOROFORM-D
Creation_time = 18-APR-2000 09:53:36
Revision_time = 2-FEB-2020 15:09:21
Current_time = 2-FEB-2020 15:09:54

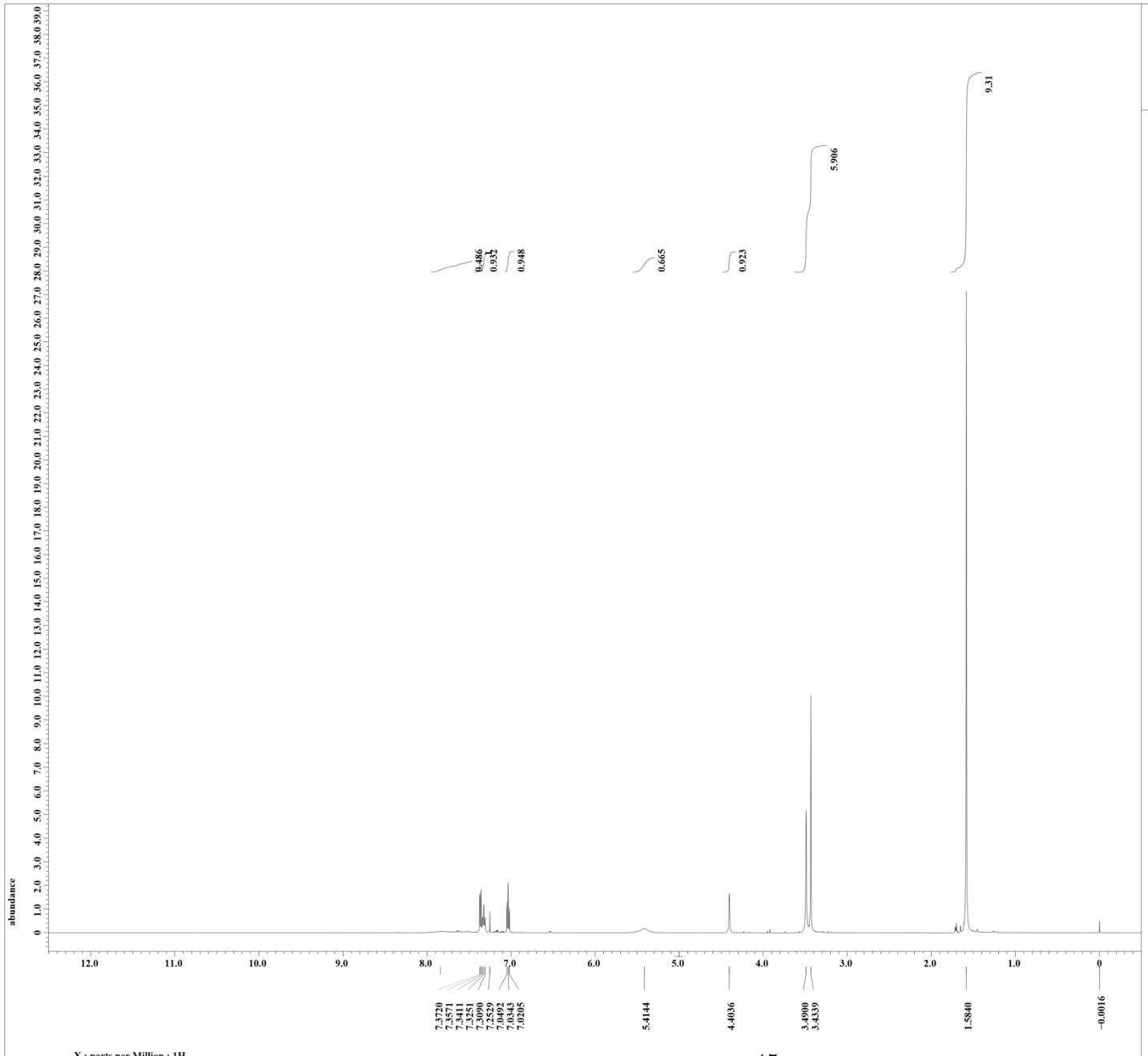
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = TRUE
Mod_return = 1
Scans = 14996
Total_scans = 14996

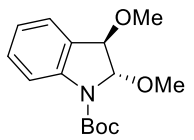
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.1[dC]



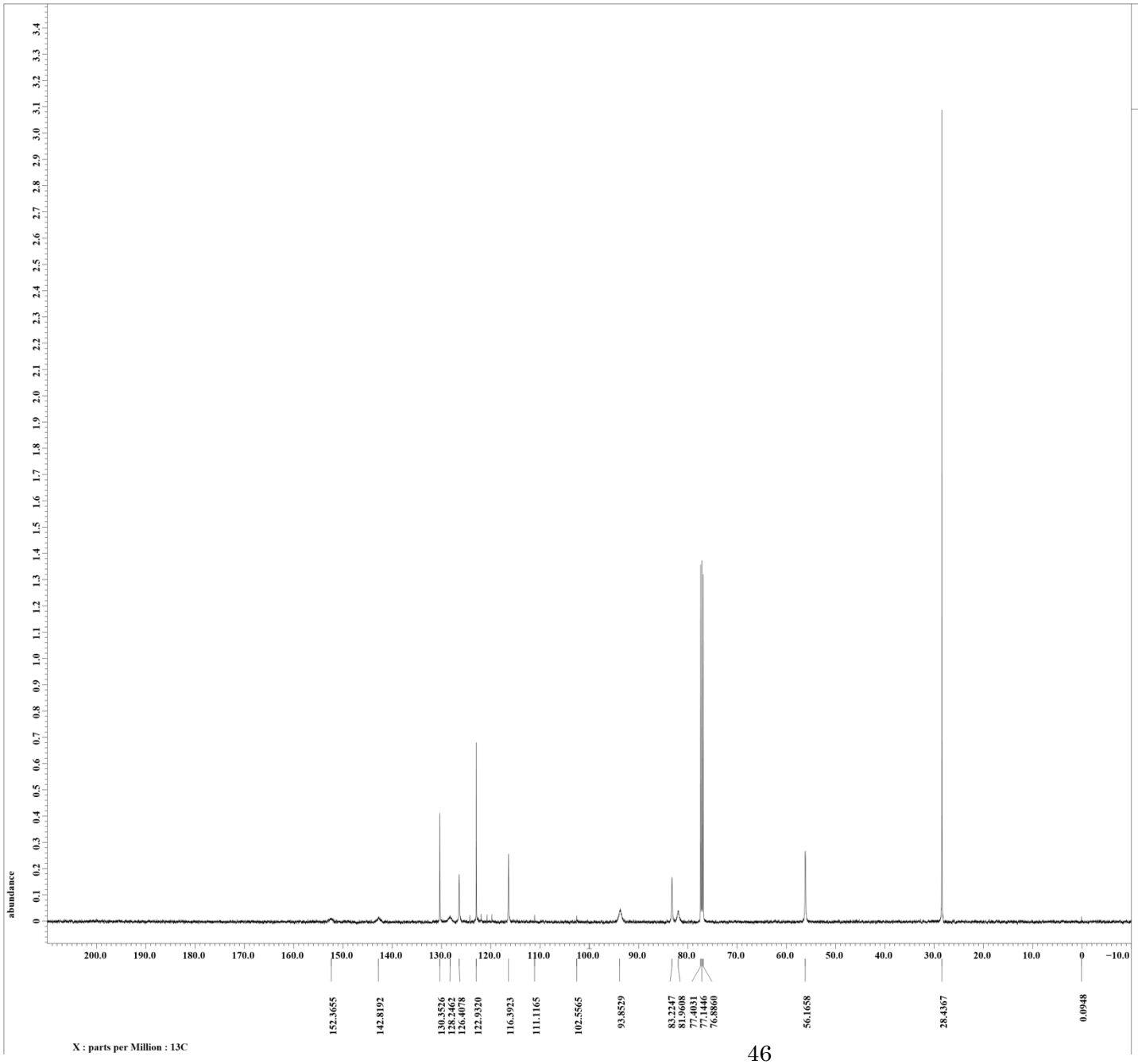
3b



```
Filename          = YT-20-01-25-9-15.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = fr2
Solvent           = CHLOROFORM-D
Creation_time     = 25-JAN-2020 15:52:50
Revision_time     = 30-JAN-2020 03:54:11
Current_time      = 30-JAN-2020 03:54:30

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.2[dC]
```

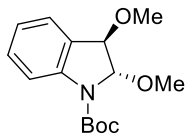


3c

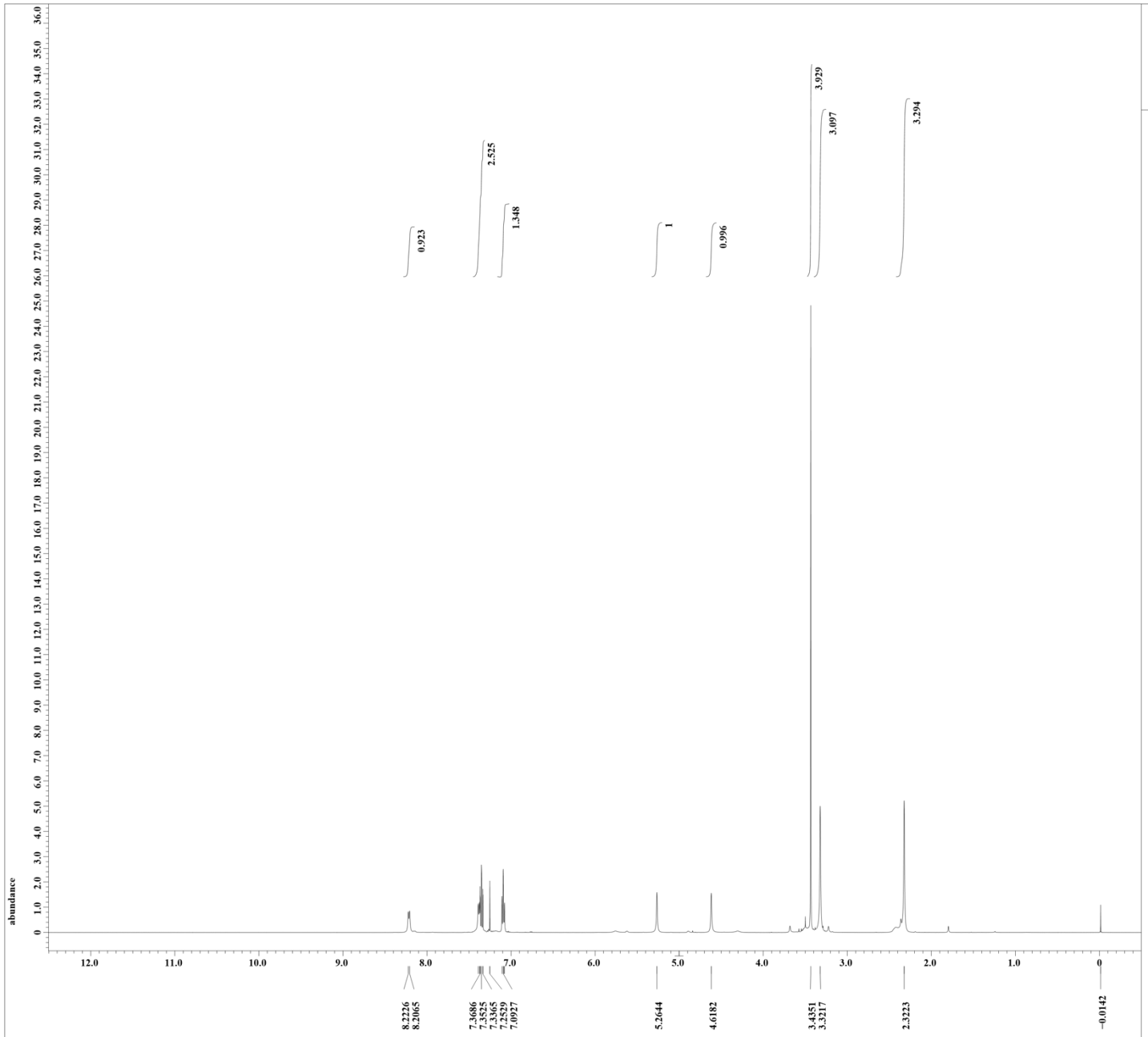


JEOL

```
Filename          = YT-20-01-25-9-17.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = fr2
Solvent           = CHLOROFORM-D
Creation_time     = 25-JAN-2020 17:05:17
Revision_time     = 25-JAN-2020 19:45:12
Current_time      = 25-JAN-2020 19:46:09

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1504
Total_scans       = 1504

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 23.9[dC]
```



3c

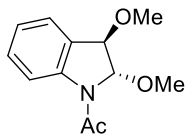


Filename = YT-20-01-27-3-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = ac2
Solvent = CHLOROFORM-D
Creation_time = 27-JAN-2020 15:28:14
Revision_time = 30-JAN-2020 04:10:45
Current_time = 30-JAN-2020 04:10:56

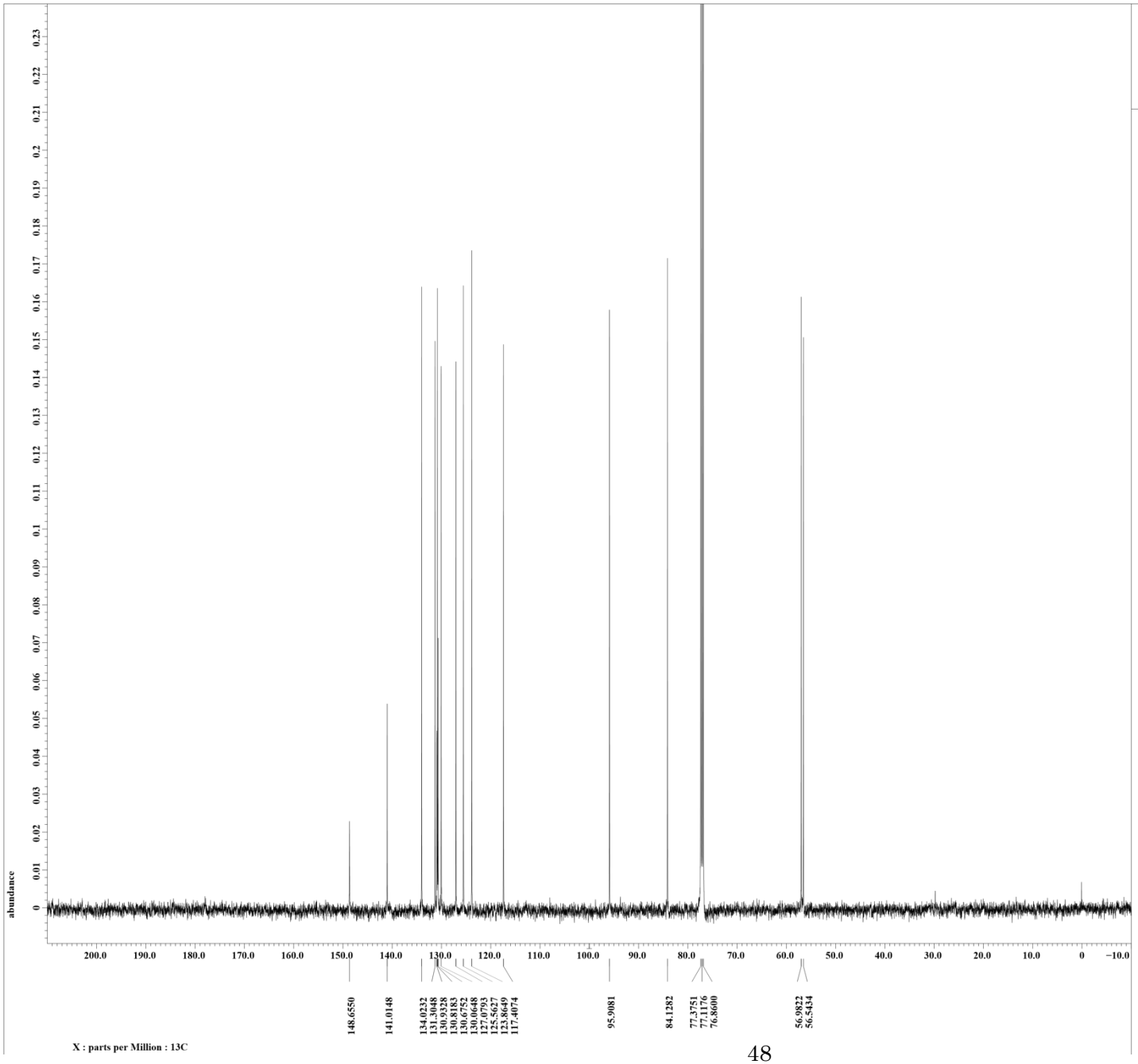
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.4[dC]



3d

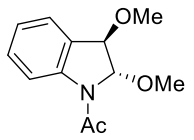


Filename = YT-20-01-21-8-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = nsdi
Solvent = CHLOROFORM-D
Creation_time = 7-APR-2000 18:44:10
Revision_time = 30-JAN-2020 03:48:50
Current_time = 30-JAN-2020 03:49:11

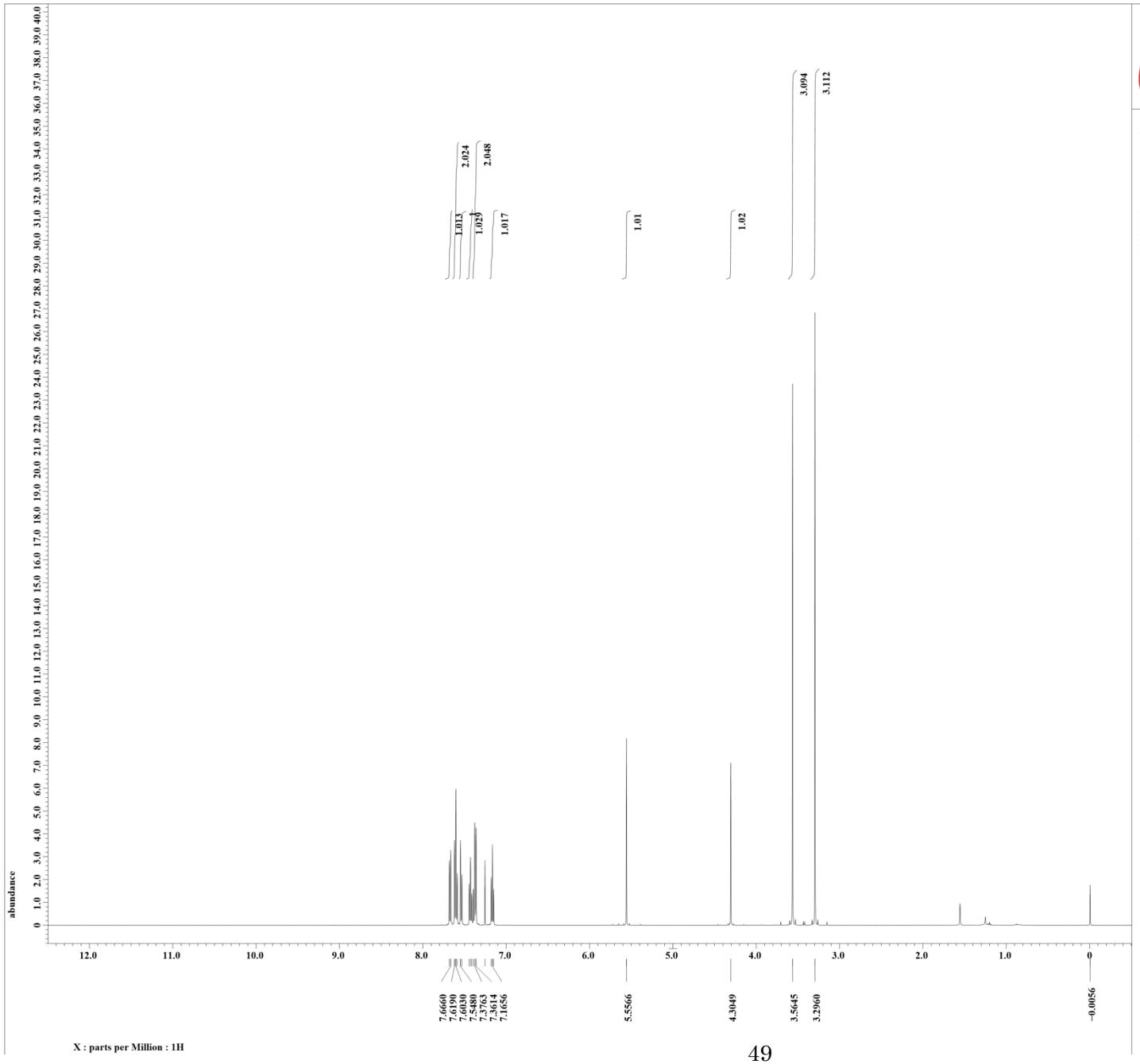
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
Scans = 1637
Total_scans = 1637

X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.5[dC]



3d

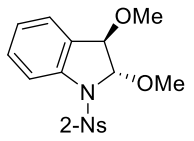


Filename = YT-20-01-21-8-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = nsdi
Solvent = CHLOROFORM-D
Creation_time = 7-APR-2000 17:25:44
Revision_time = 30-JAN-2020 03:48:00
Current_time = 30-JAN-2020 03:48:07

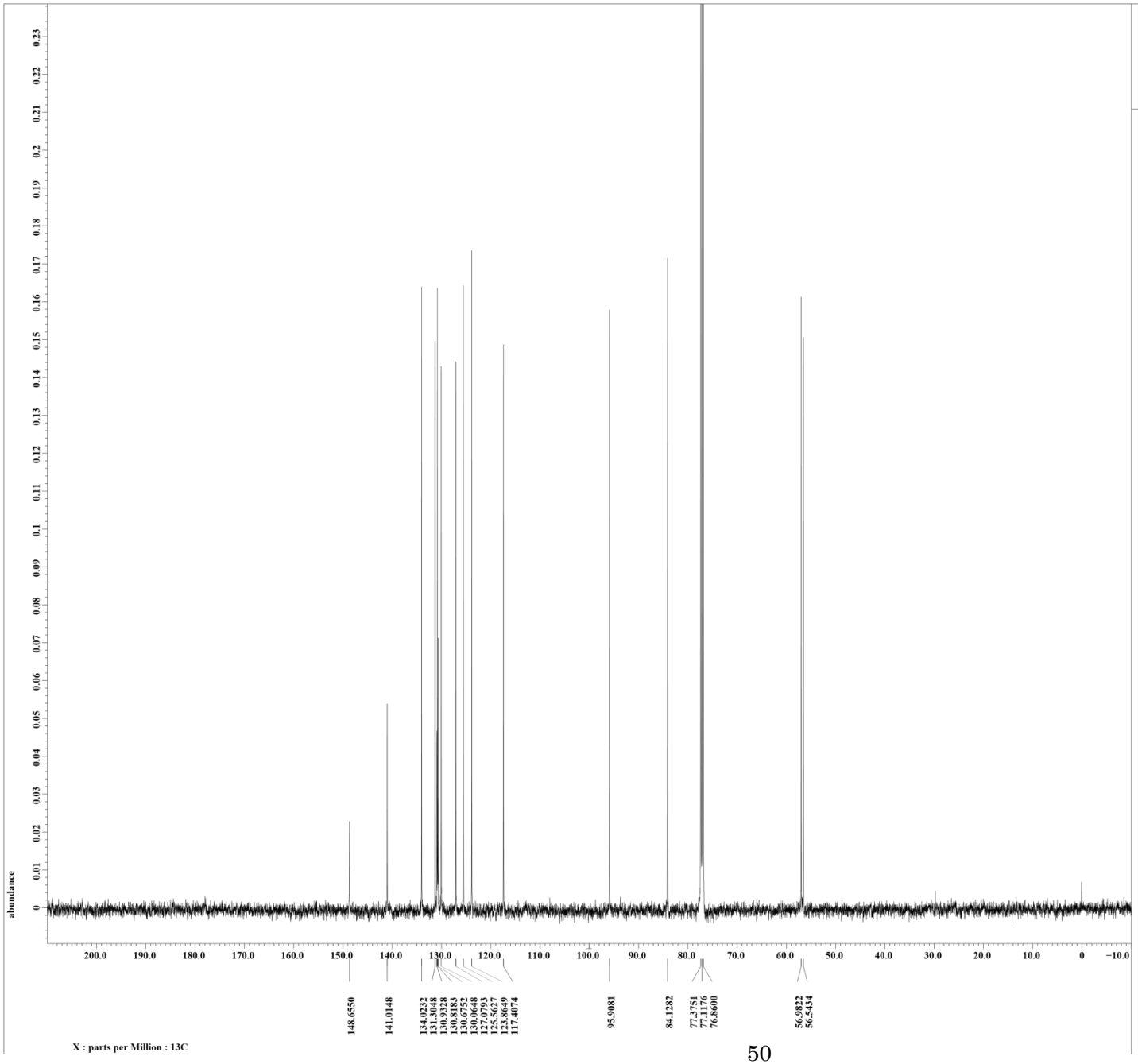
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 22.9[dC]

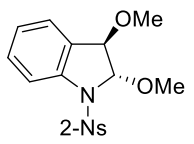


3e

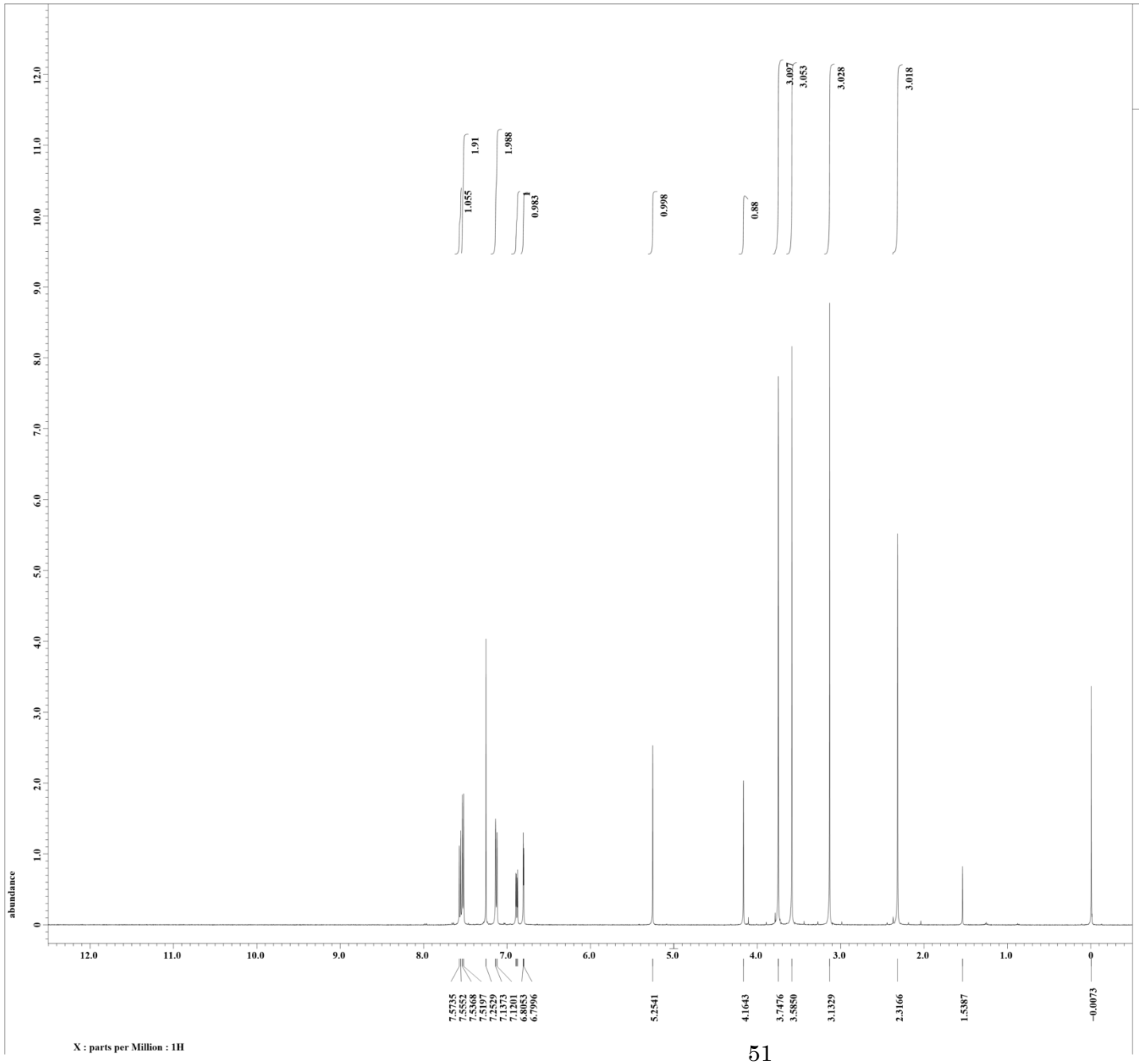


JEOL

```
Filename          = YT-20-01-21-8-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = nsdi
Solvent           = CHLOROFORM-D
Creation_time     =  7-APR-2000 18:44:10
Revision_time     = 30-JAN-2020 03:48:50
Current_time      = 30-JAN-2020 03:49:11

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 1637
Total_scans       = 1637

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 54
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 23.5[dC]
```



3e

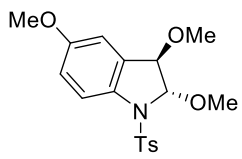


Filename = YT-20-01-28-1-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 01281
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 09:41:12
Revision_time = 28-JAN-2020 10:38:07
Current_time = 28-JAN-2020 14:16:37

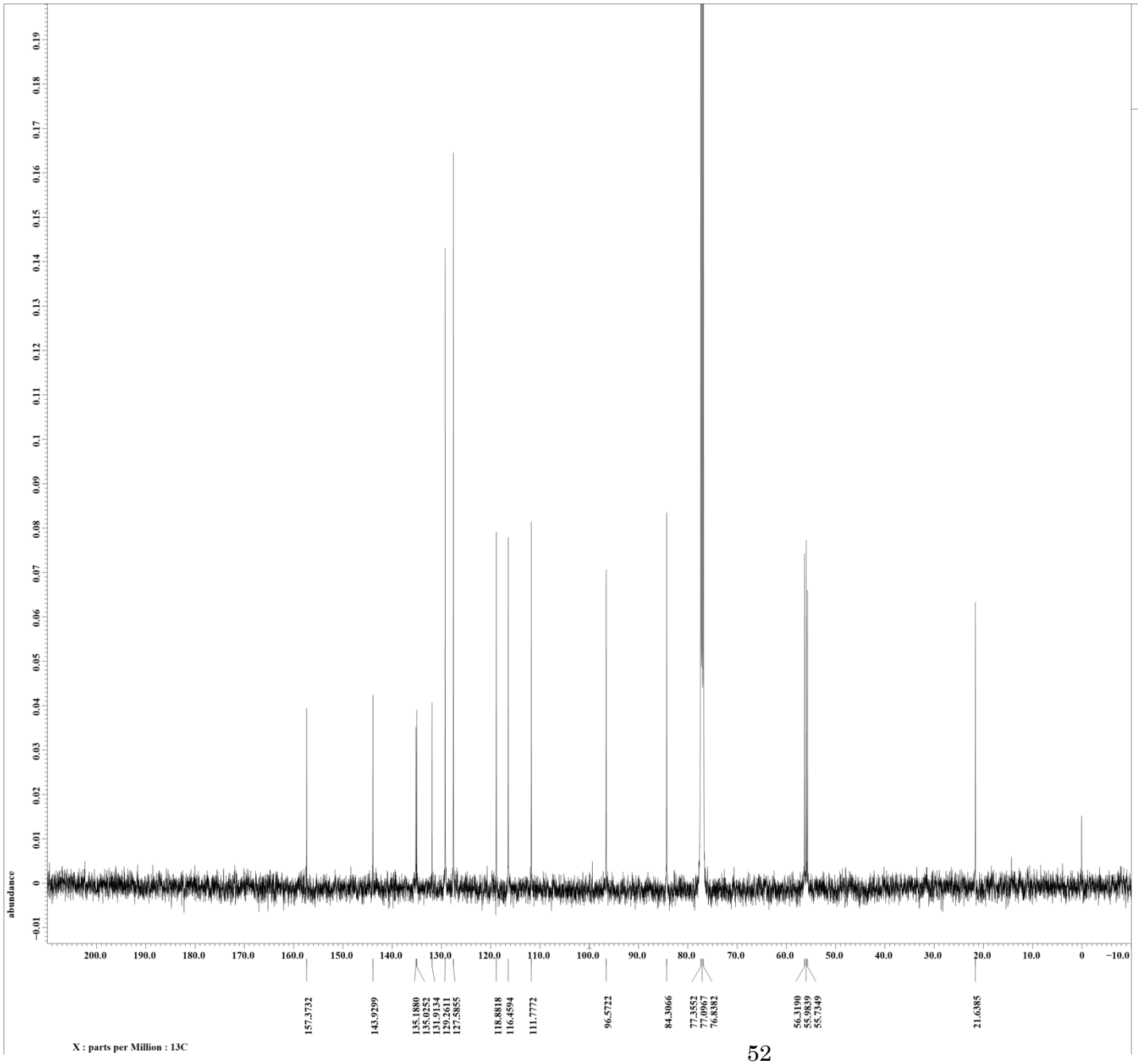
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

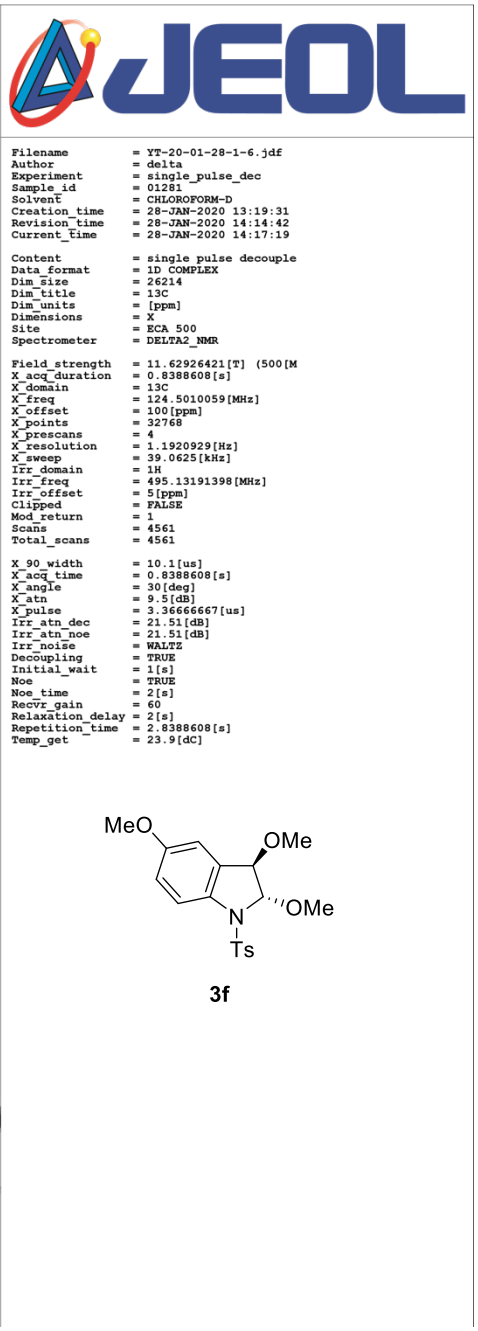
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.2[dC]



3f





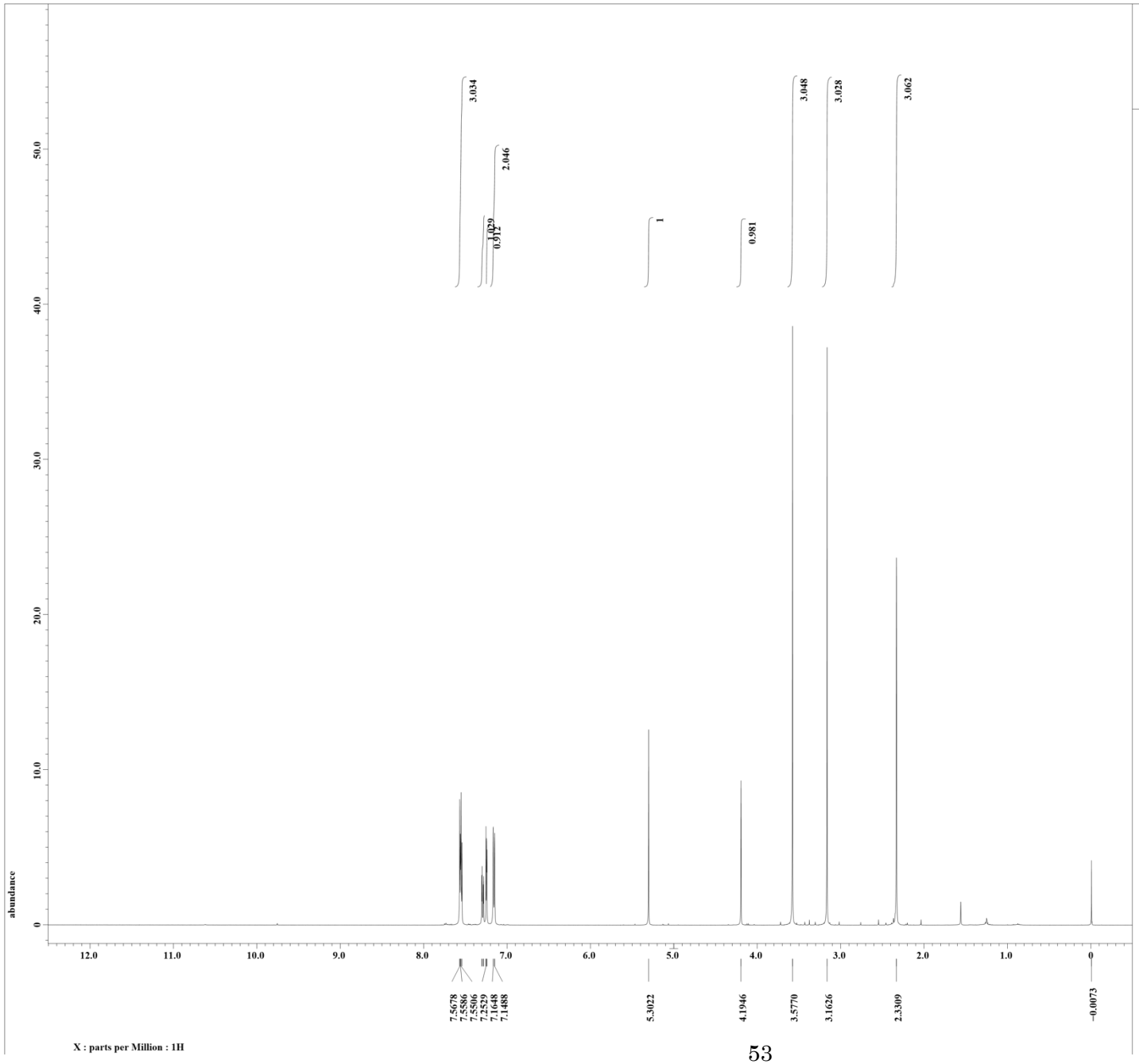
Filename = YT-20-01-28-1-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 01281
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 13:19:31
Revision_time = 28-JAN-2020 14:14:42
Current_time = 28-JAN-2020 14:17:19

Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 4561
Total_scans = 4561

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 23.9[dC]

3f

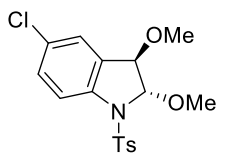


Filename = YT-20-01-18-3-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = fr2
Solvent = CHLOROFORM-D
Creation_time = 18-JAN-2020 16:40:42
Revision_time = 30-JAN-2020 03:39:27
Current_time = 30-JAN-2020 03:39:36

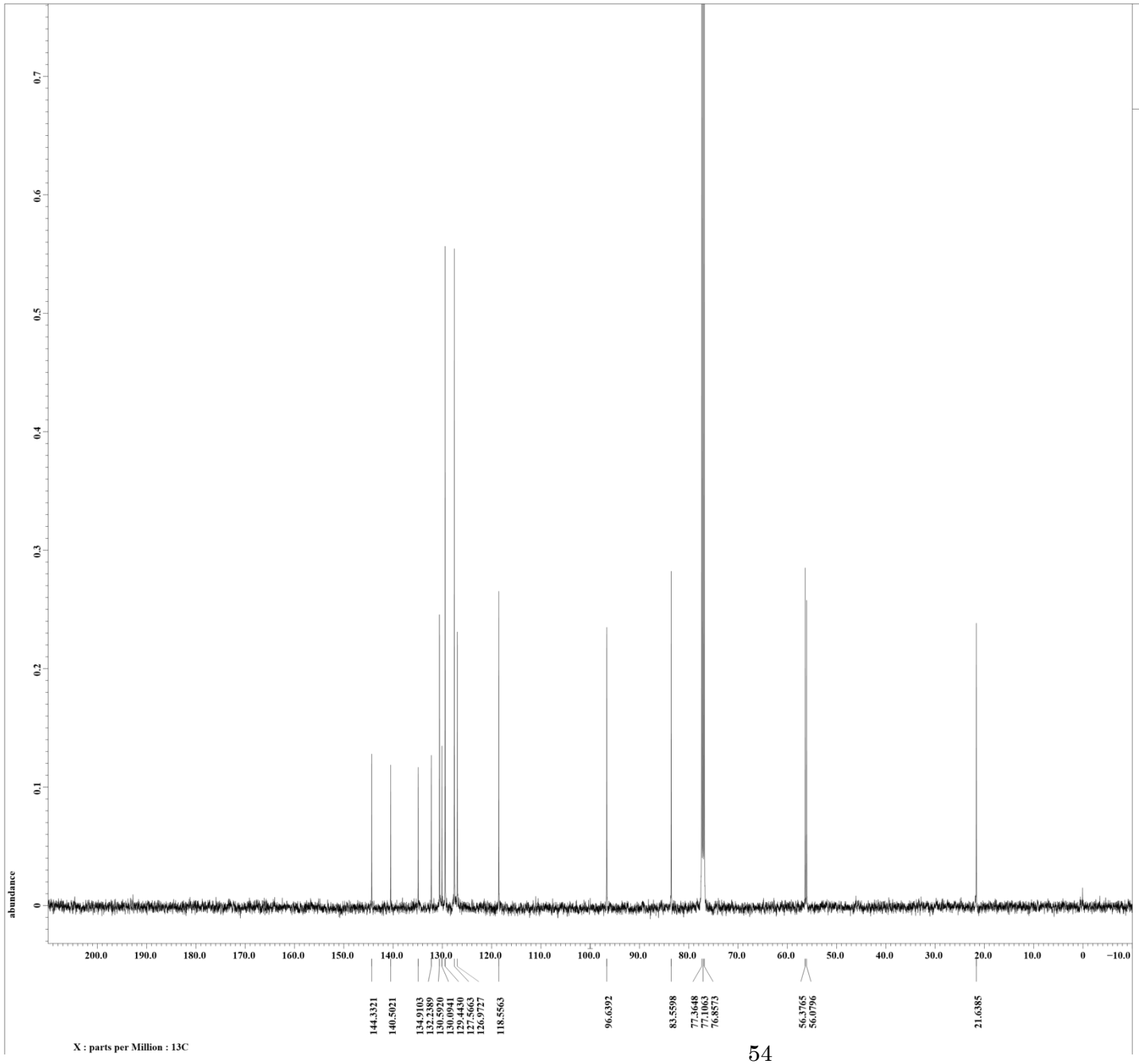
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.2[dC]



3g

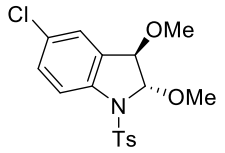


Filename = YT-20-01-18-3-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = fr2
Solvent = CHLOROFORM-D
Creation_time = 18-JAN-2020 19:20:12
Revision_time = 30-JAN-2020 03:40:06
Current_time = 30-JAN-2020 03:40:19

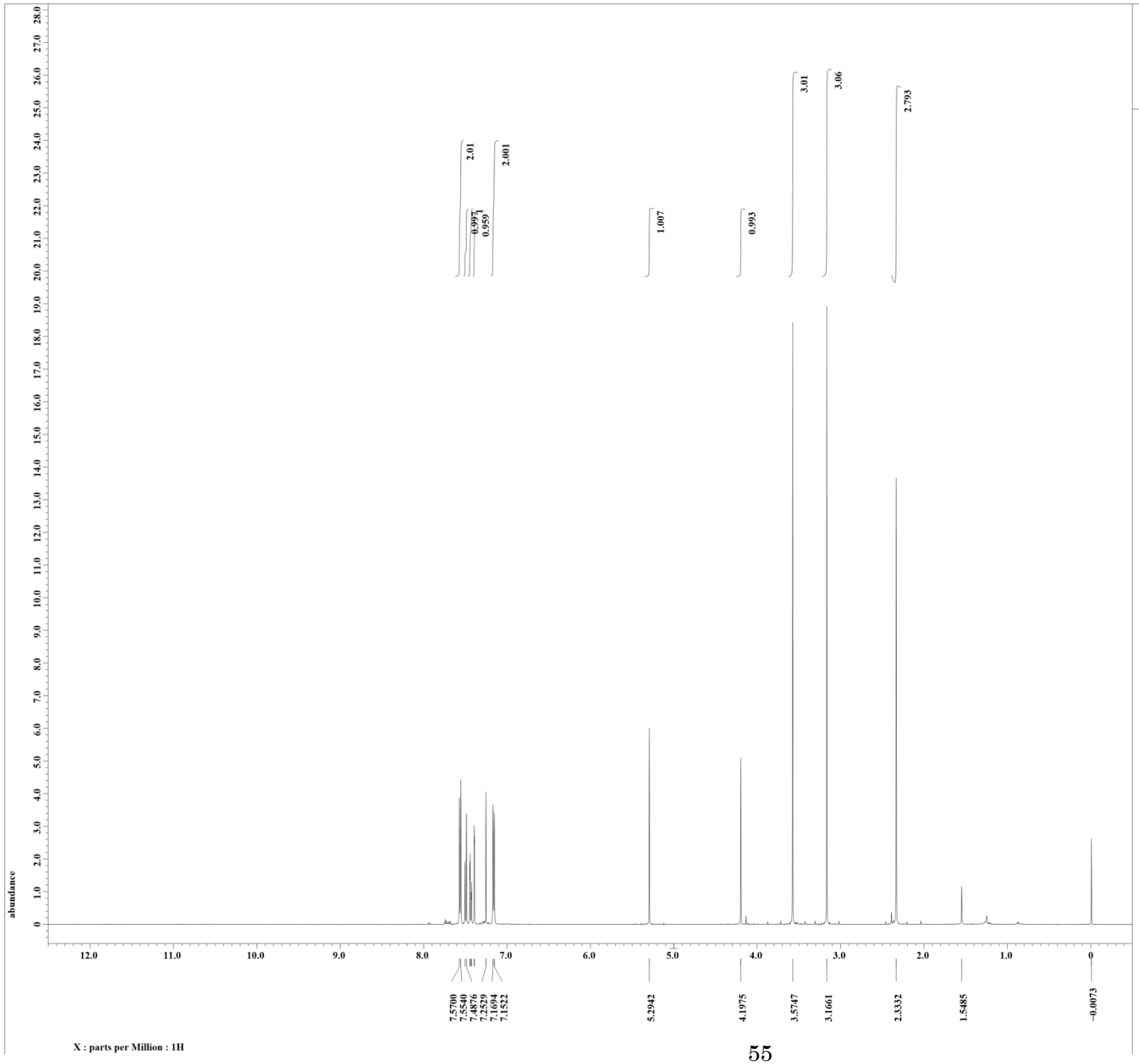
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1711
Total_scans = 1711

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24[dC]



3g

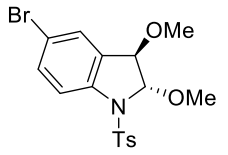


Filename = YT-20-01-27-5-8.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 01275
Solvent = CHLOROFORM-D
Creation_time = 27-JAN-2020 18:34:03
Revision_time = 30-JAN-2020 04:07:47
Current_time = 30-JAN-2020 04:07:55

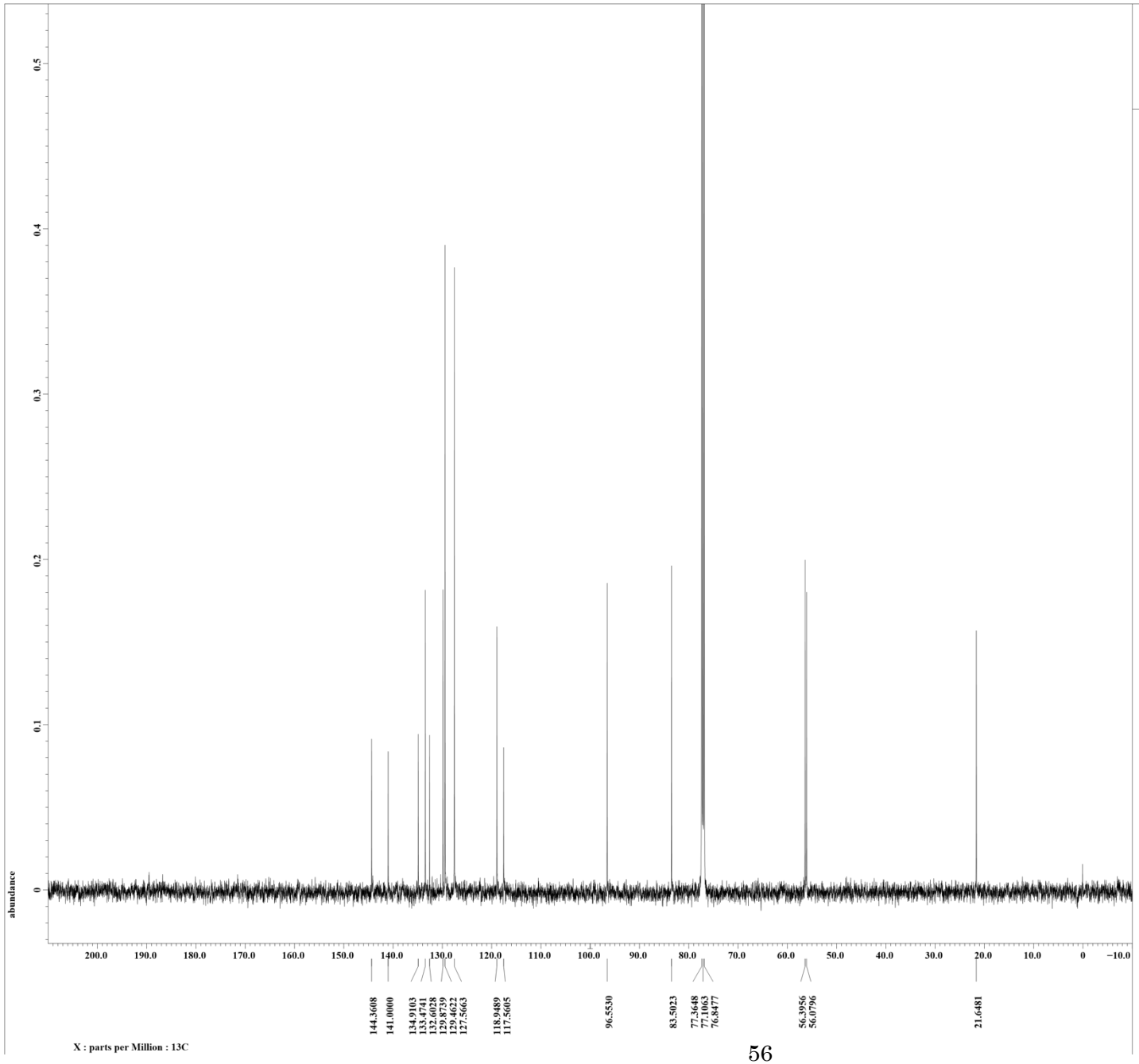
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.3[dC]



3h

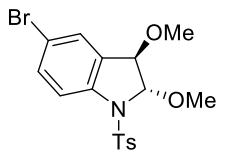


Filename = YT-20-01-27-5-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 01275
Solvent = CHLOROFORM-D
Creation_time = 27-JAN-2020 19:37:58
Revision_time = 30-JAN-2020 04:08:40
Current_time = 30-JAN-2020 04:08:52

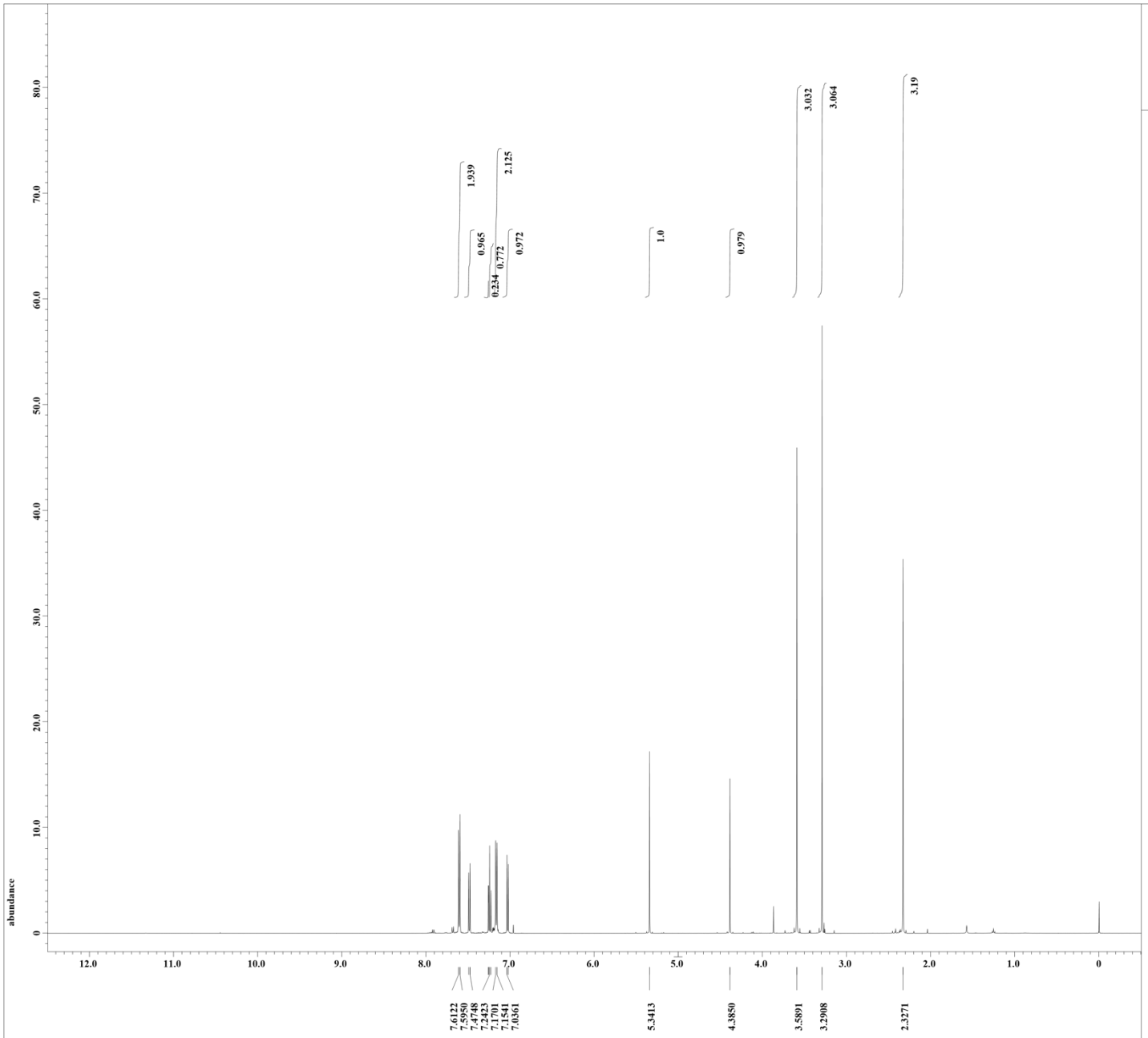
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1321
Total_scans = 1321

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 23.9[dC]



3h

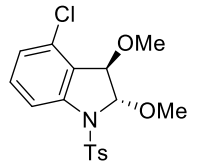


Filename = YT-20-01-20-2-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 4 fr2
Solvent = CHLOROFORM-D
Creation_time = 6-APR-2000 11:45:48
Revision_time = 30-JAN-2020 03:42:35
Current_time = 30-JAN-2020 03:42:52

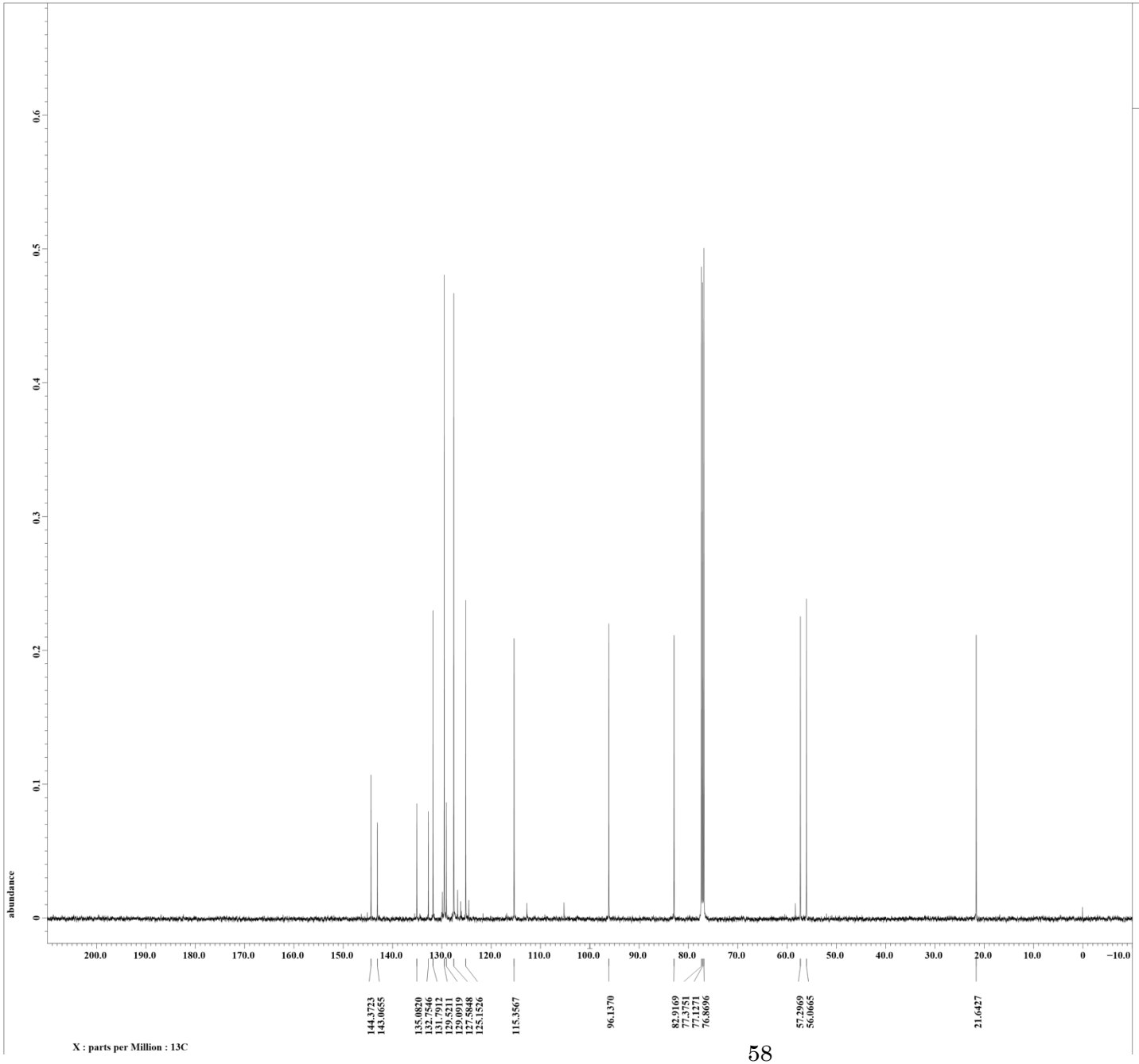
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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 = TRUE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 23.7[dC]



3i

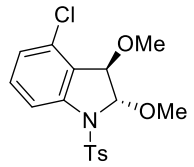


Filename = YT-20-01-20-2-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 4 fr2
Solvent = CHLOROFORM-D
Creation_time = 6-APR-2000 13:11:52
Revision_time = 30-JAN-2020 03:43:46
Current_time = 30-JAN-2020 03:43:57

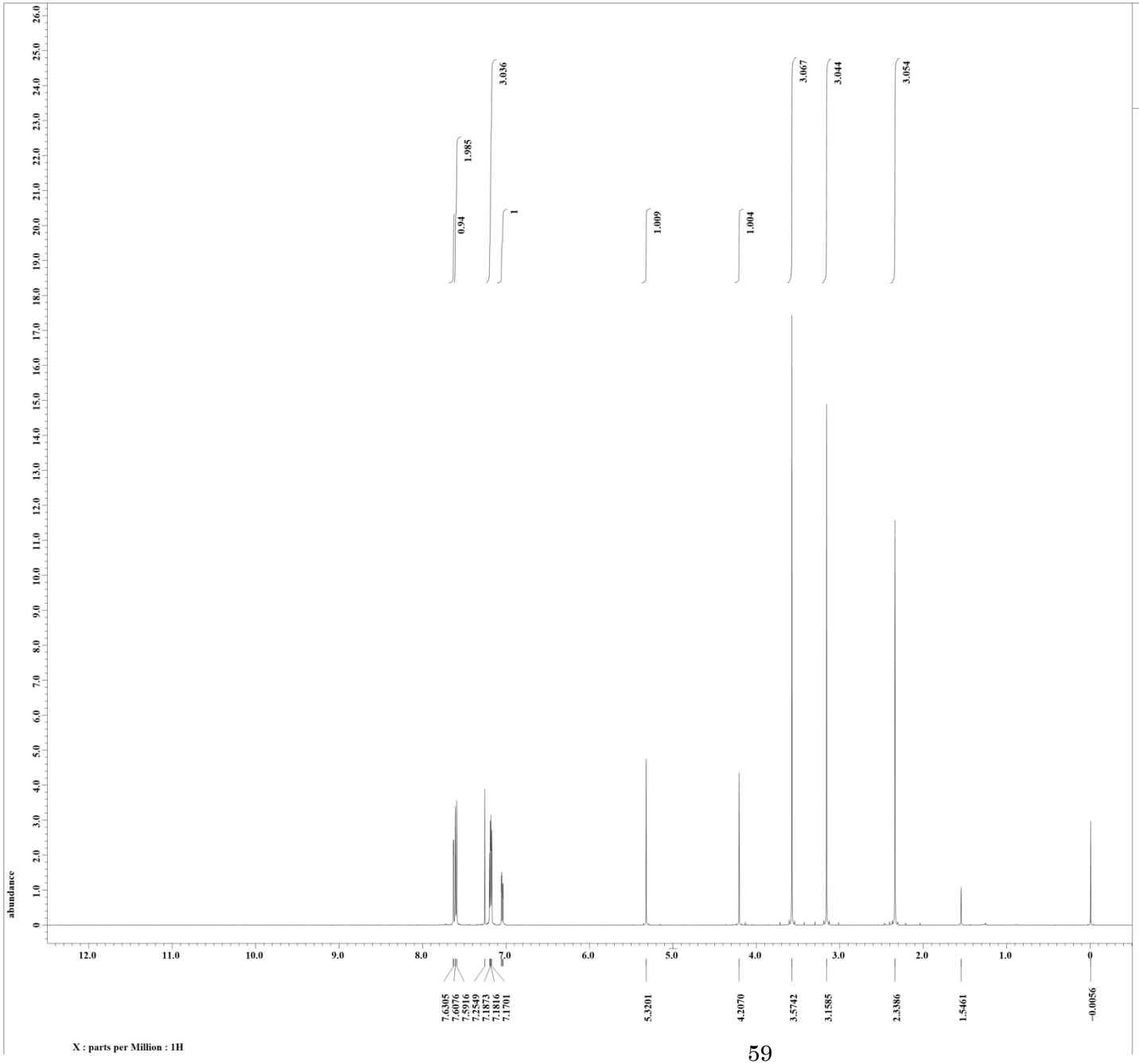
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
Scans = 1809
Total_scans = 1809

X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.5[dC]



3i

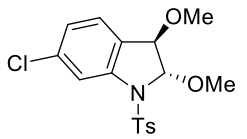


Filename = YT-20-01-24-2-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = 161 fr1
Solvent = CHLOROFORM-D
Creation_time = 10-APR-2000 13:17:57
Revision_time = 30-JAN-2020 03:56:19
Current_time = 30-JAN-2020 03:56:27

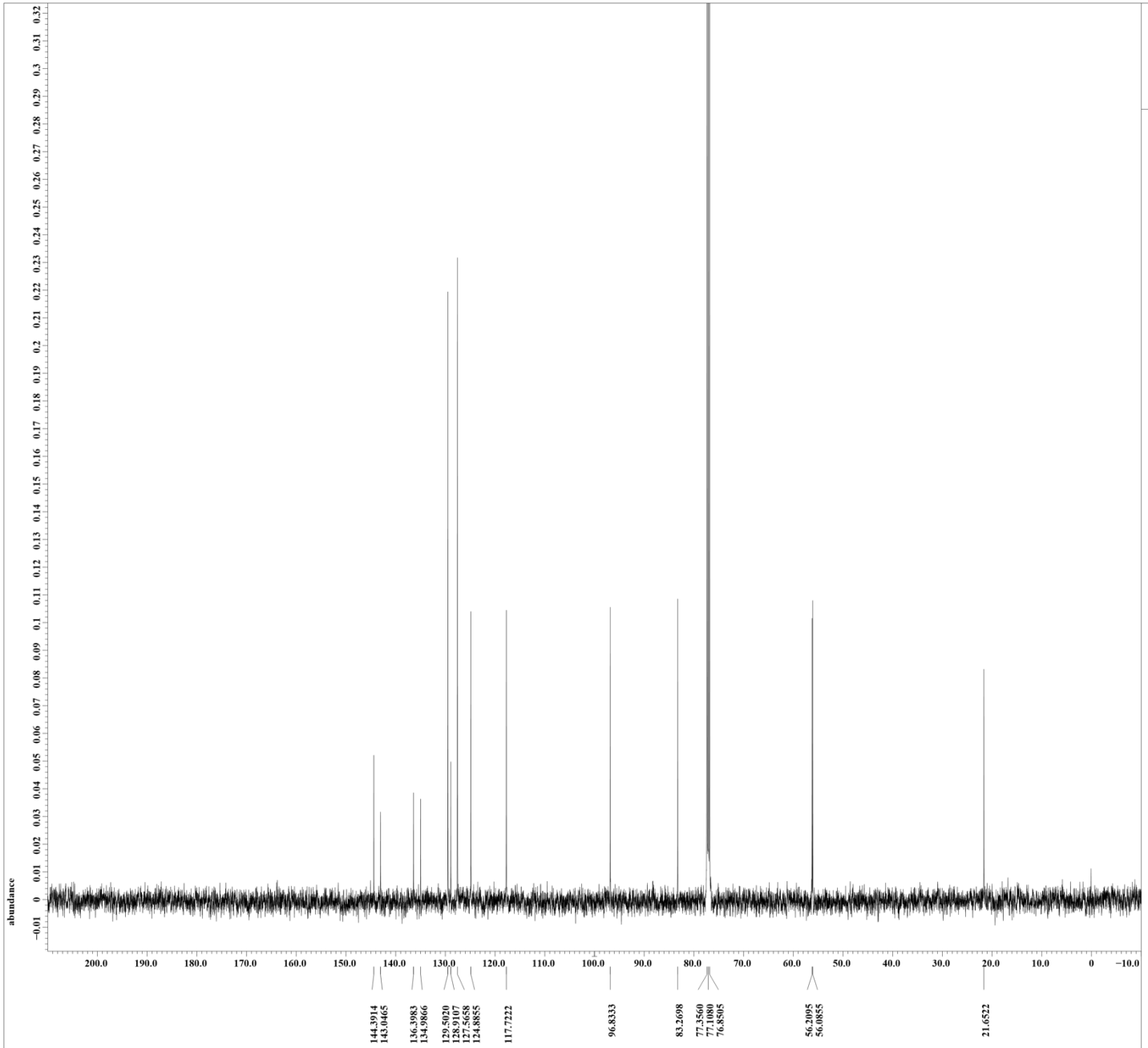
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 22.7[dC]

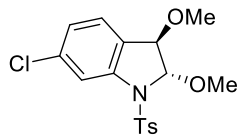


3j

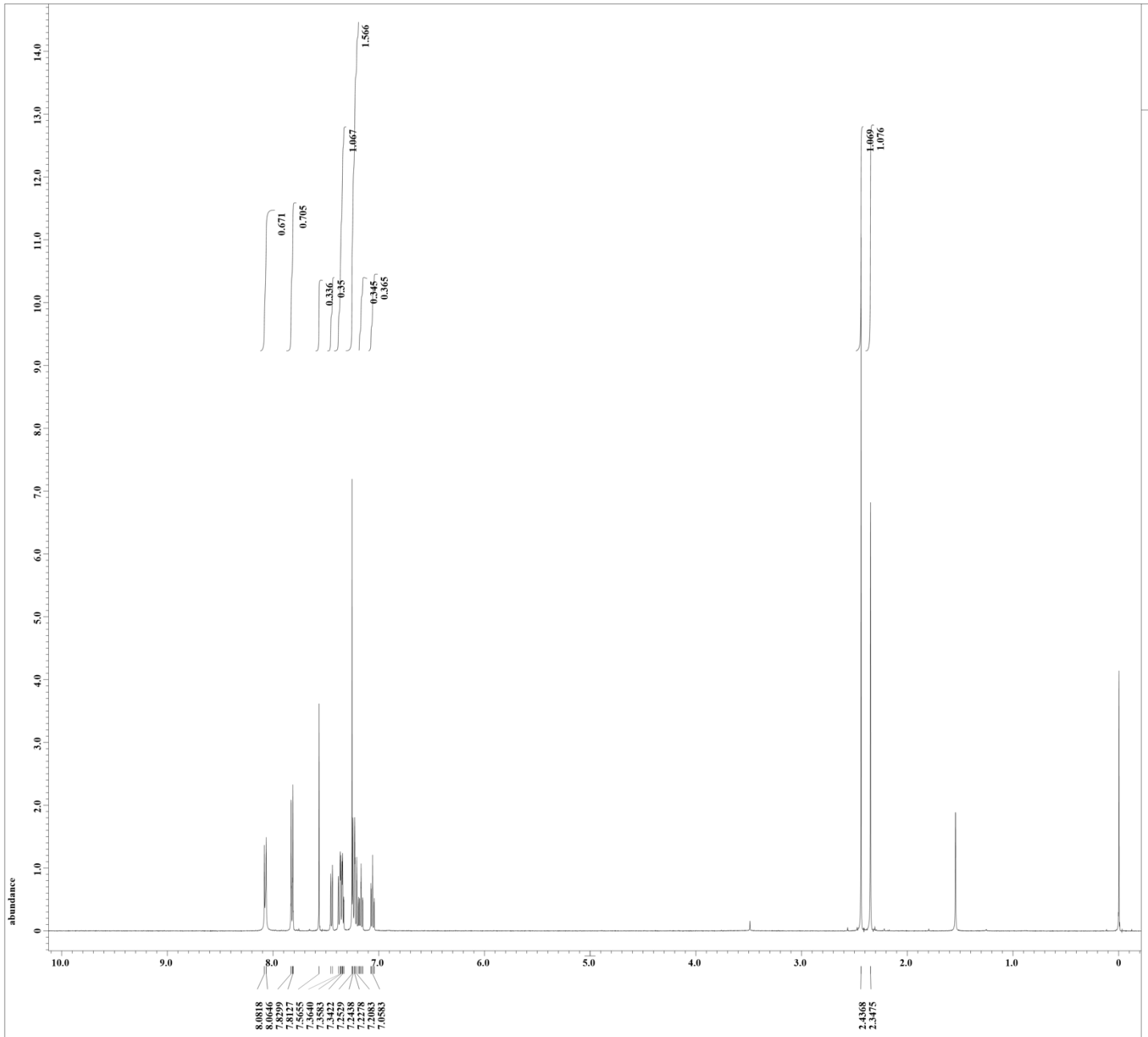


JEOL

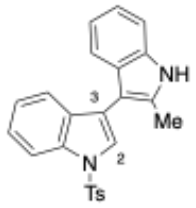
```
Filename          = YT-20-01-24-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = 161 fr1
Solvent           = CHLOROFORM-D
Creation_time     = 10-APR-2000 13:41:12
Revision_time     = 30-JAN-2020 03:57:01
Current_time      = 30-JAN-2020 03:57:18

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 469
Total_scans       = 469

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 23.6[dC]
```



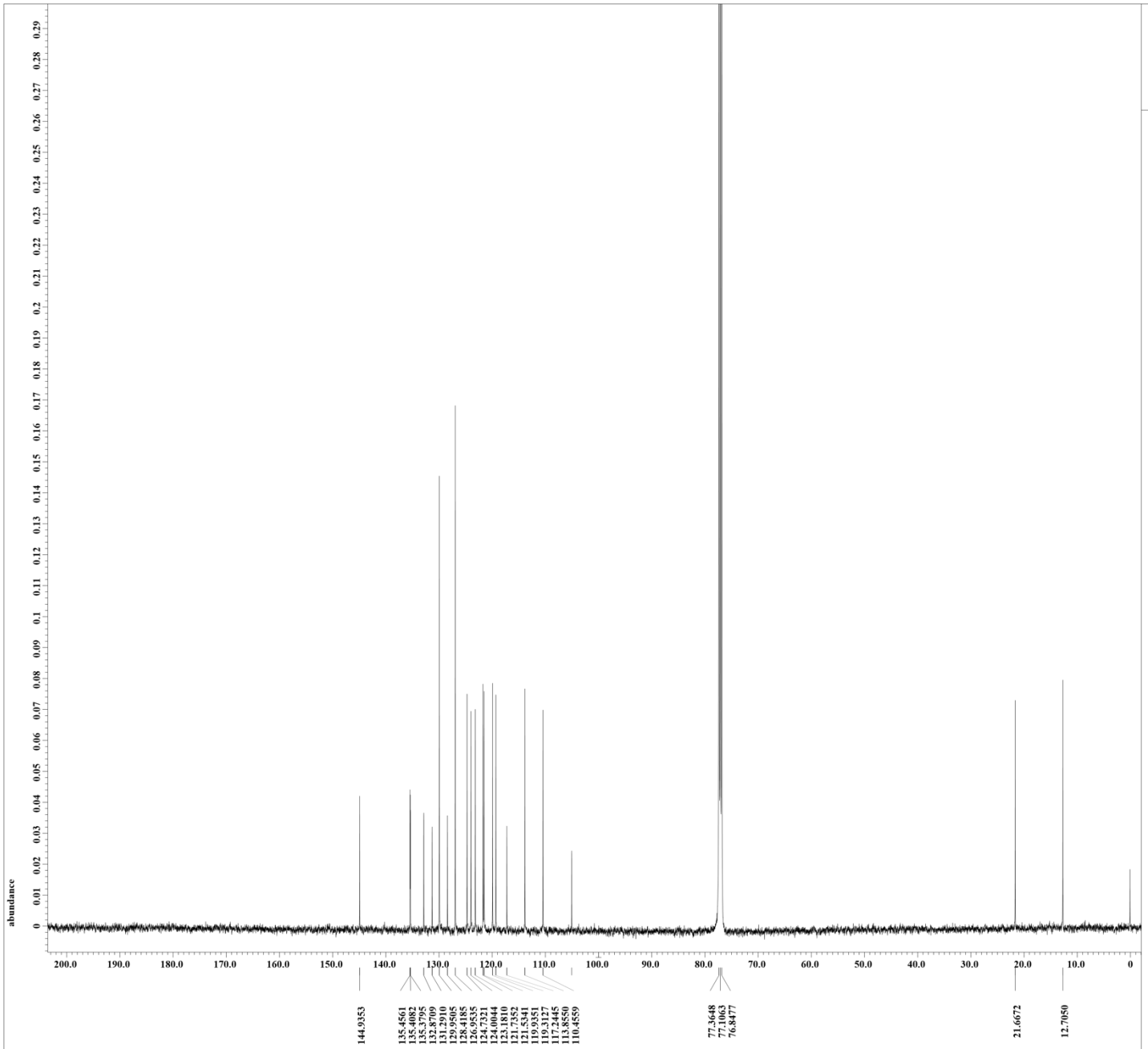
3j




```
Filename          = TA191231-14.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#578338
Solvent           = CHLOROFORM-D
Creation_time     = 31-DEC-2019 15:15:05
Revision_time     =  1-FEB-2020 18:14:11
Current_time      =  1-FEB-2020 18:14:34

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23[dC]
```



6aa

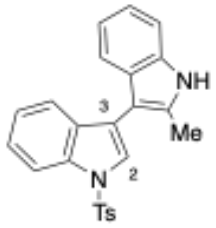


Filename = TA191231-12.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#648086
Solvent = CHLOROFORM-D
Creation_time = 1-JAN-2020 12:35:19
Revision_time = 1-FEB-2020 18:15:45
Current_time = 1-FEB-2020 18:16:13

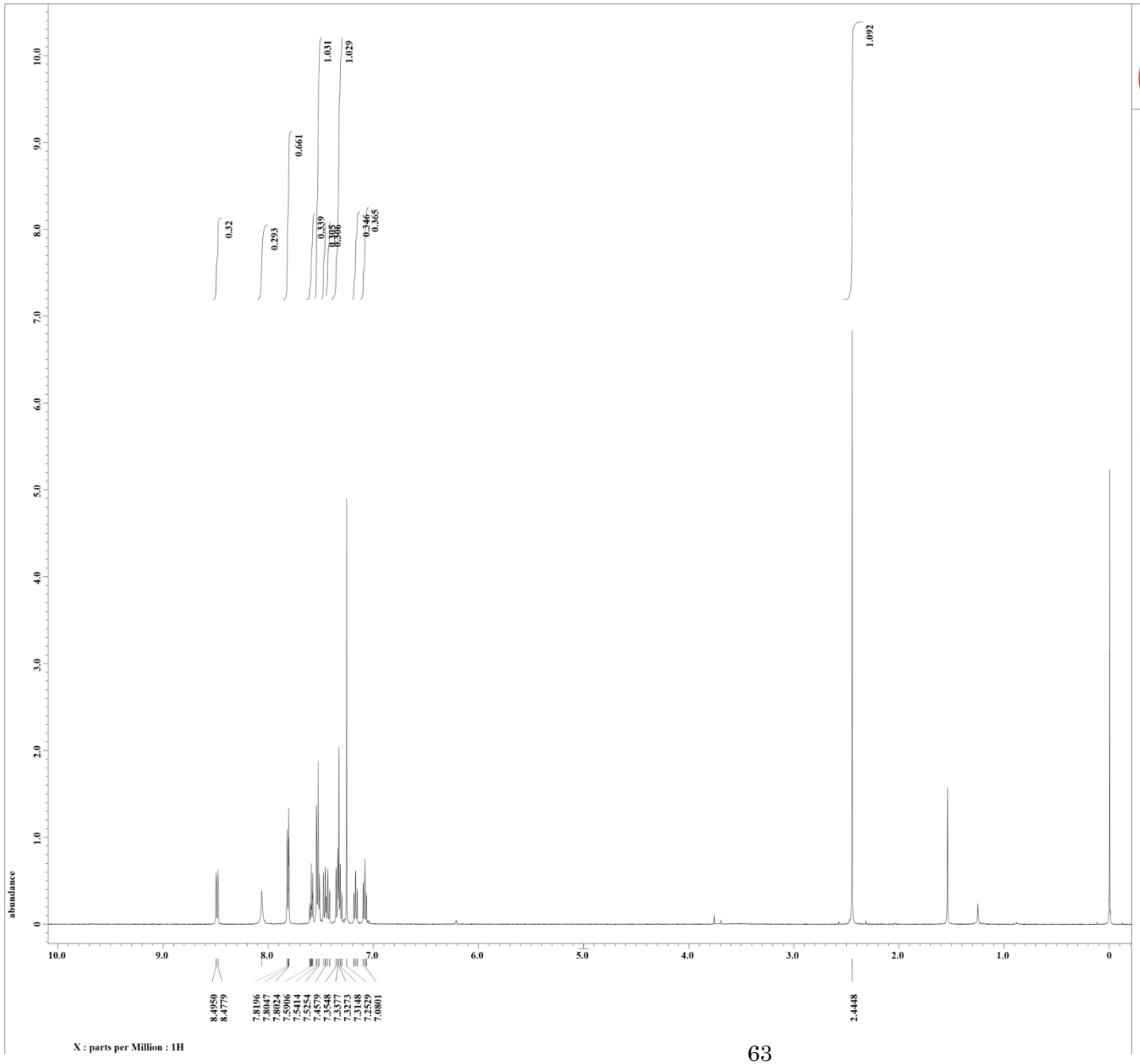
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 24613
Total_scans = 24613

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 23.9[dC]



6aa

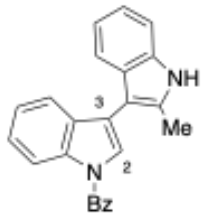


Filename = SH004-1102-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#467375
Solvent = CHLOROFORM-D
Creation_time = 30-JAN-2020 12:05:25
Revision_time = 30-JAN-2020 17:32:57
Current_time = 30-JAN-2020 17:33:18

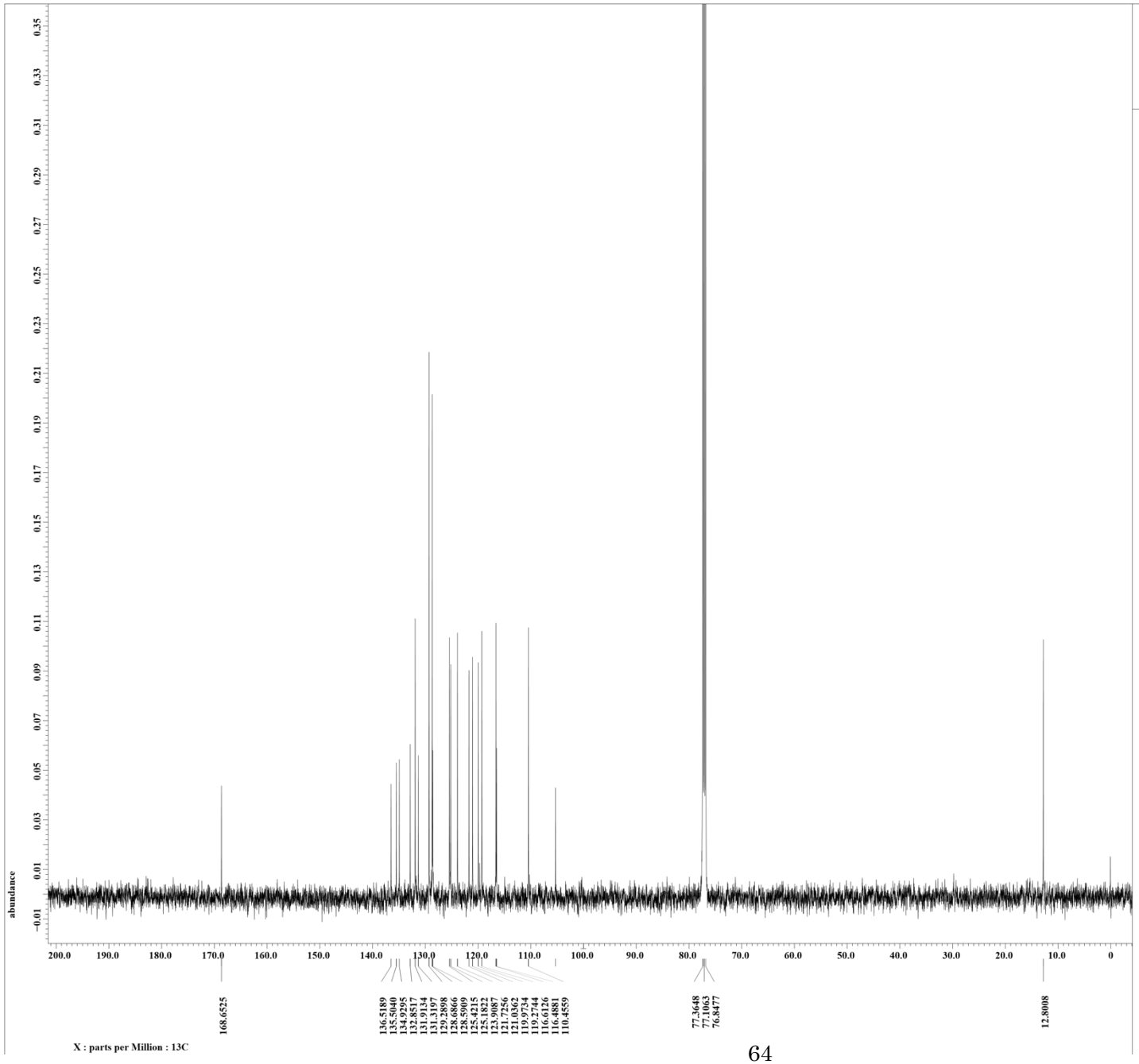
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.6[dC]



6ba

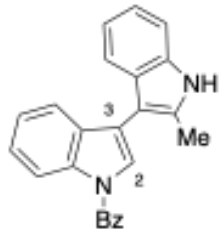


Filename = SH004-1102-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#530593
Solvent = CHLOROFORM-D
Creation_time = 30-JAN-2020 15:13:55
Revision_time = 30-JAN-2020 16:09:33
Current_time = 30-JAN-2020 16:12:13

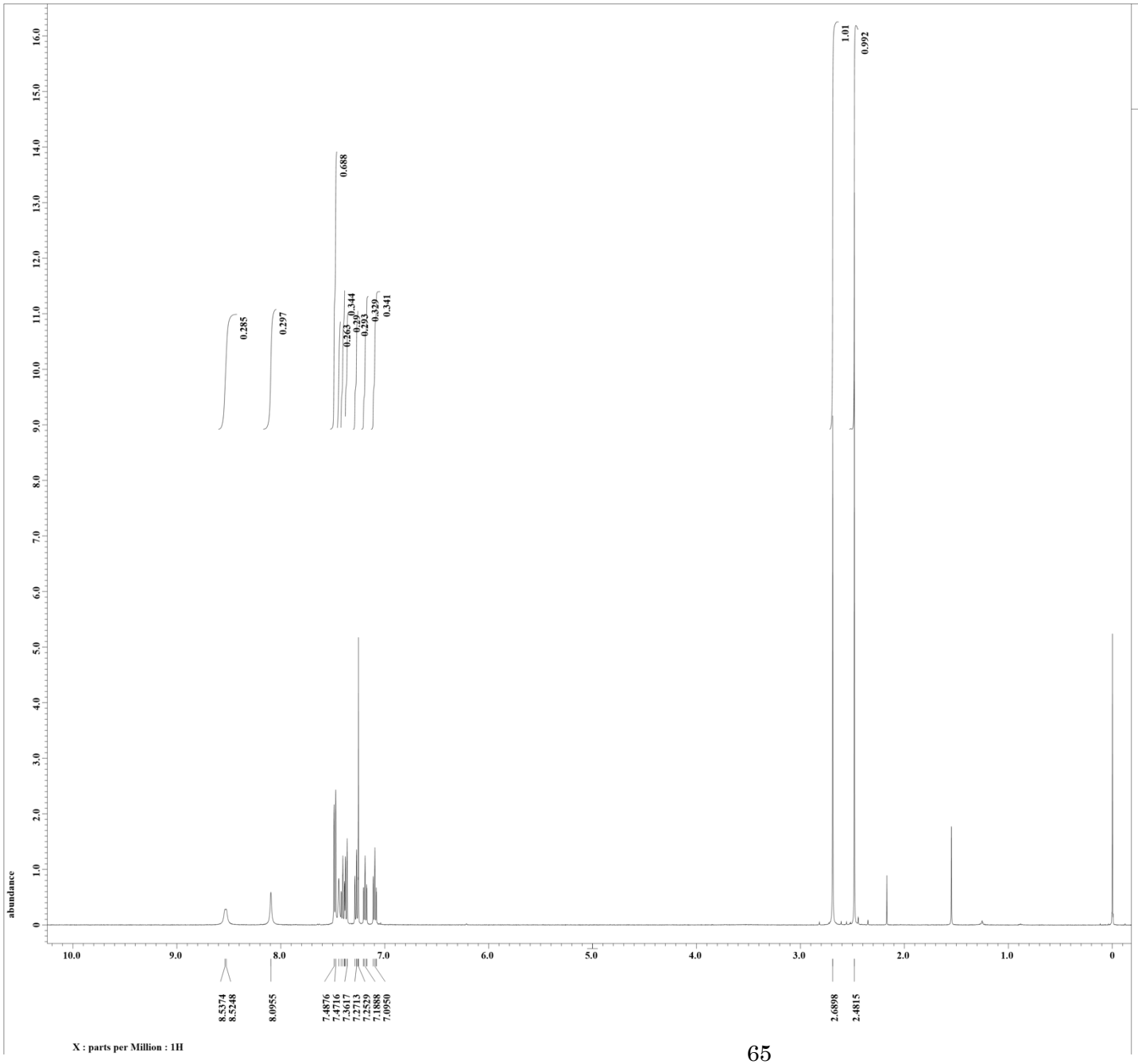
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1766
Total_scans = 1766

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.4[dC]



6ba

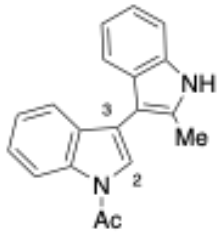


Filename = TA200129-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#633593
Solvent = CHLOROFORM-D
Creation_time = 29-JAN-2020 16:42:32
Revision_time = 29-JAN-2020 17:39:43
Current_time = 29-JAN-2020 17:40:30

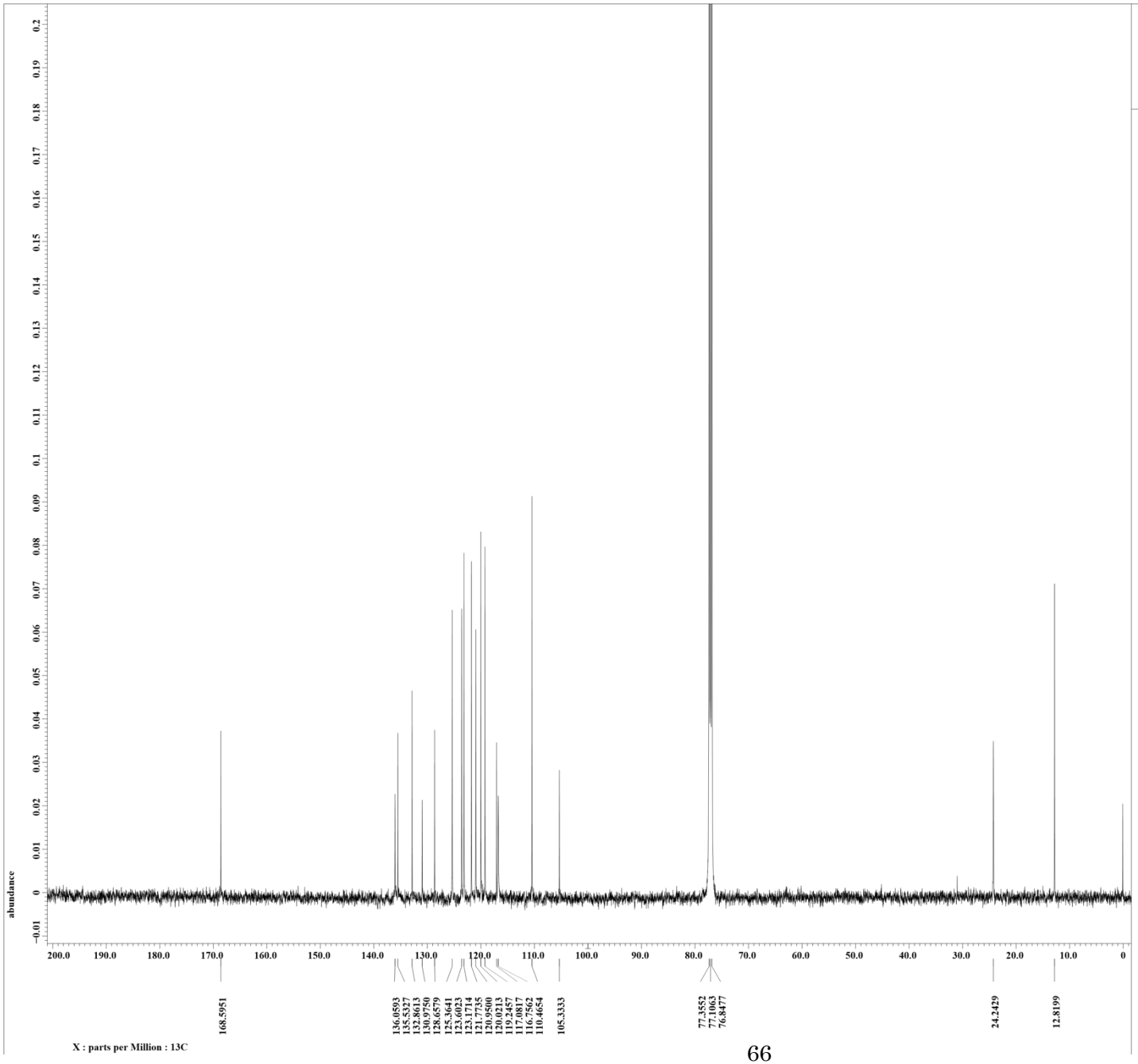
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.7[dC]

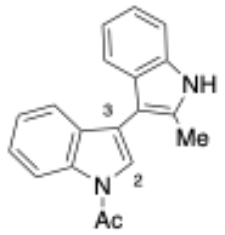


6ca

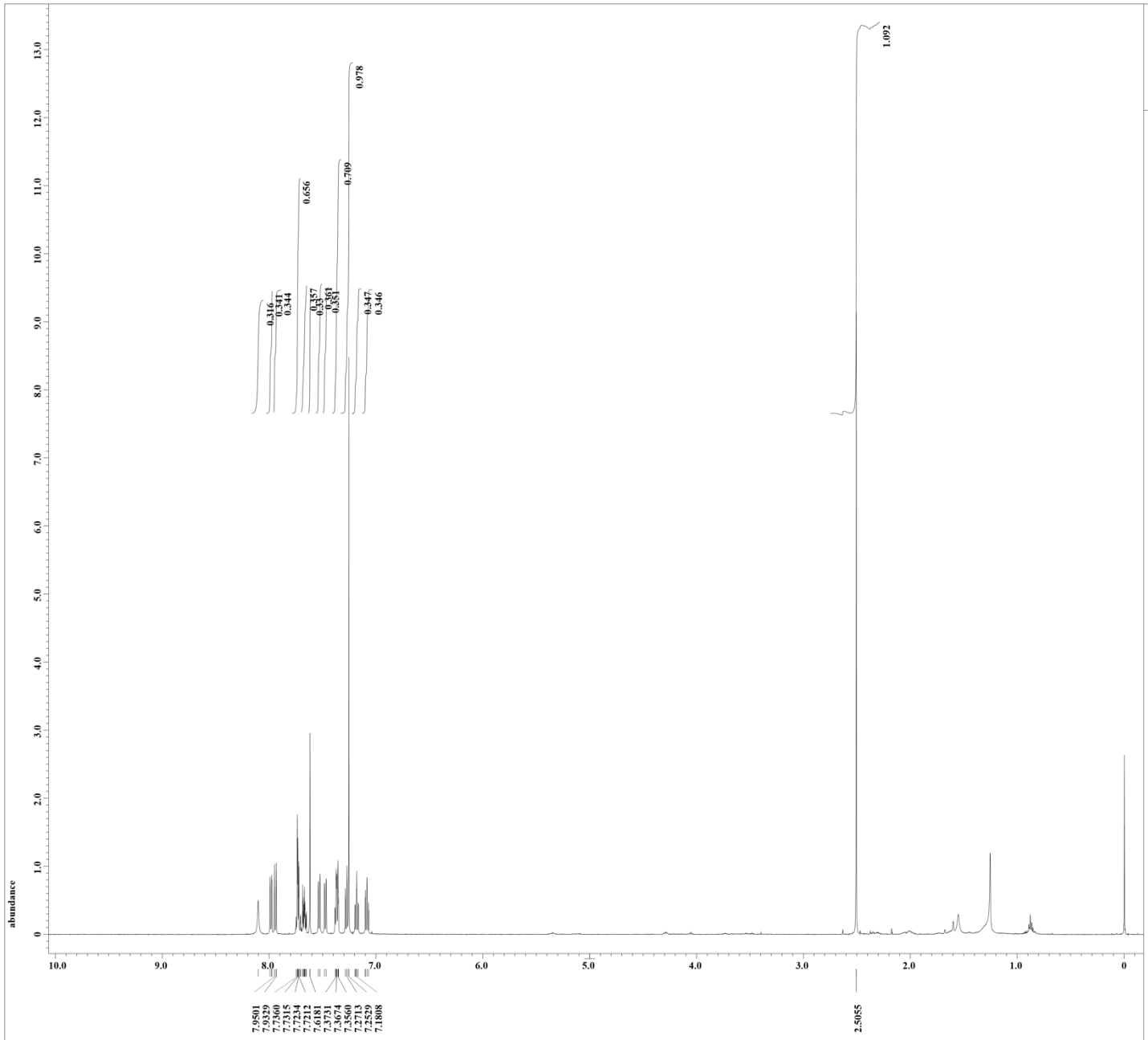


JEOL

```
Filename          = TA200129-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#634679
Solvent           = CHLOROFORM-D
Creation_time     = 30-JAN-2020 07:12:30
Revision_time     = 30-JAN-2020 08:08:24
Current_time      = 30-JAN-2020 08:10:29

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 18357
Total_scans       = 18357

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 24.1[dC]
```



6ca

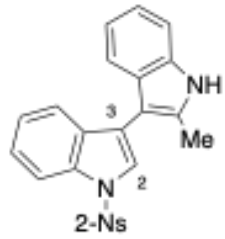


Filename = TA200128-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#741162
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 19:42:13
Revision_time = 28-JAN-2020 20:40:29
Current_time = 28-JAN-2020 20:41:38

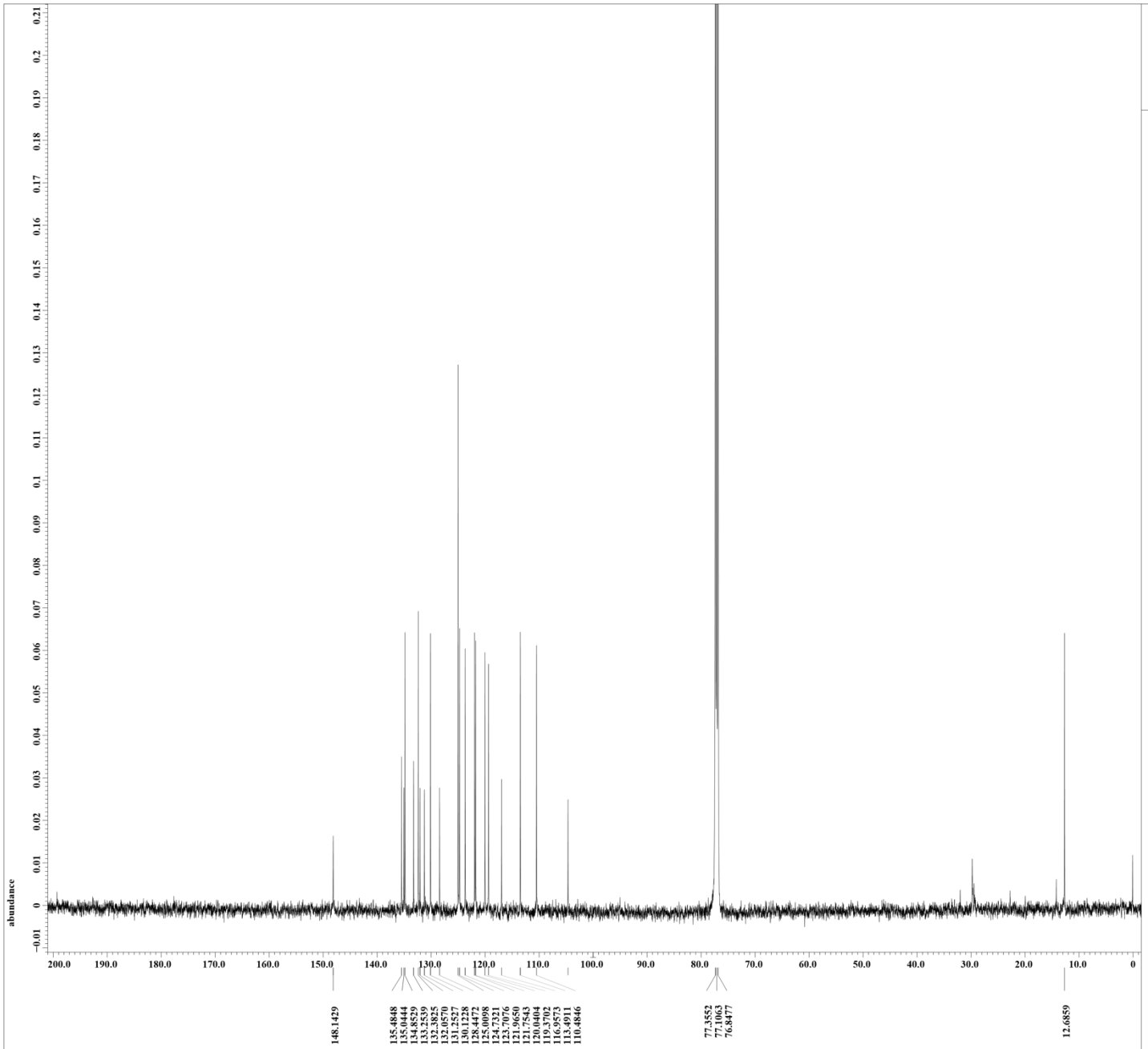
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

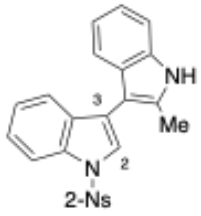
X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.5[dC]



6ea

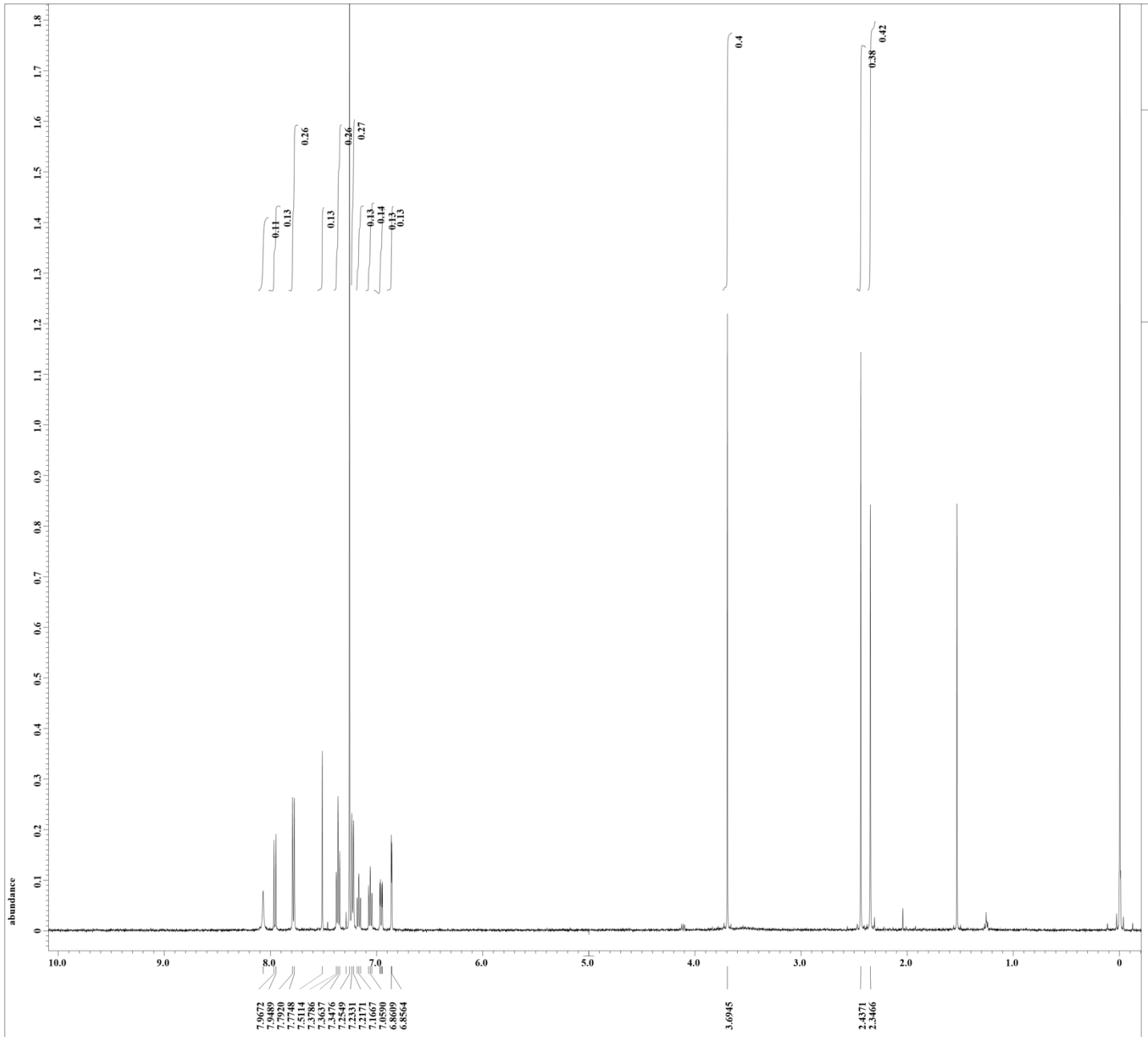


```
Filename          = TA200128-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#742447
Solvent           = CHLOROFORM-D
Creation_time     = 29-JAN-2020 07:19:37
Revision_time     = 29-JAN-2020 08:14:58
Current_time      = 29-JAN-2020 08:18:01

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 14710
Total_scans       = 14710

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 24[dC]
```



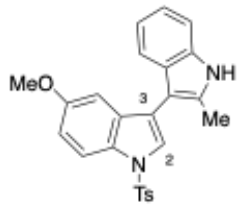
2-Ns

6ea

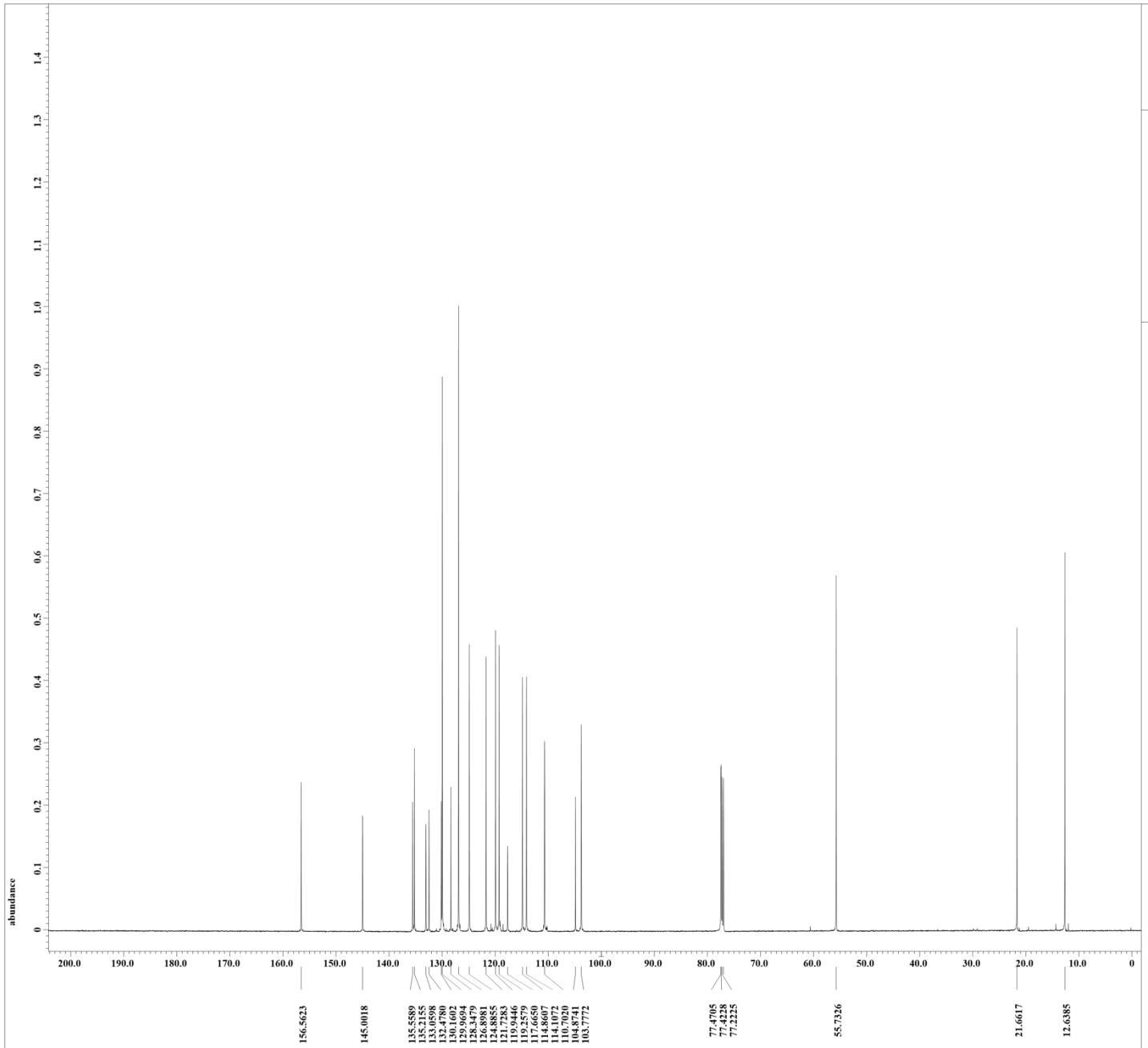



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-0604-1.jdf
```

```
Filename          = SH004-0604-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#751760
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2000 22:41:36
Revision_time     =  2-FEB-2020 11:48:18
Current_time      =  2-FEB-2020 11:48:47

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 23.6[dC]
```

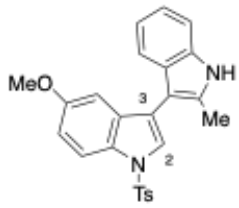


6fa

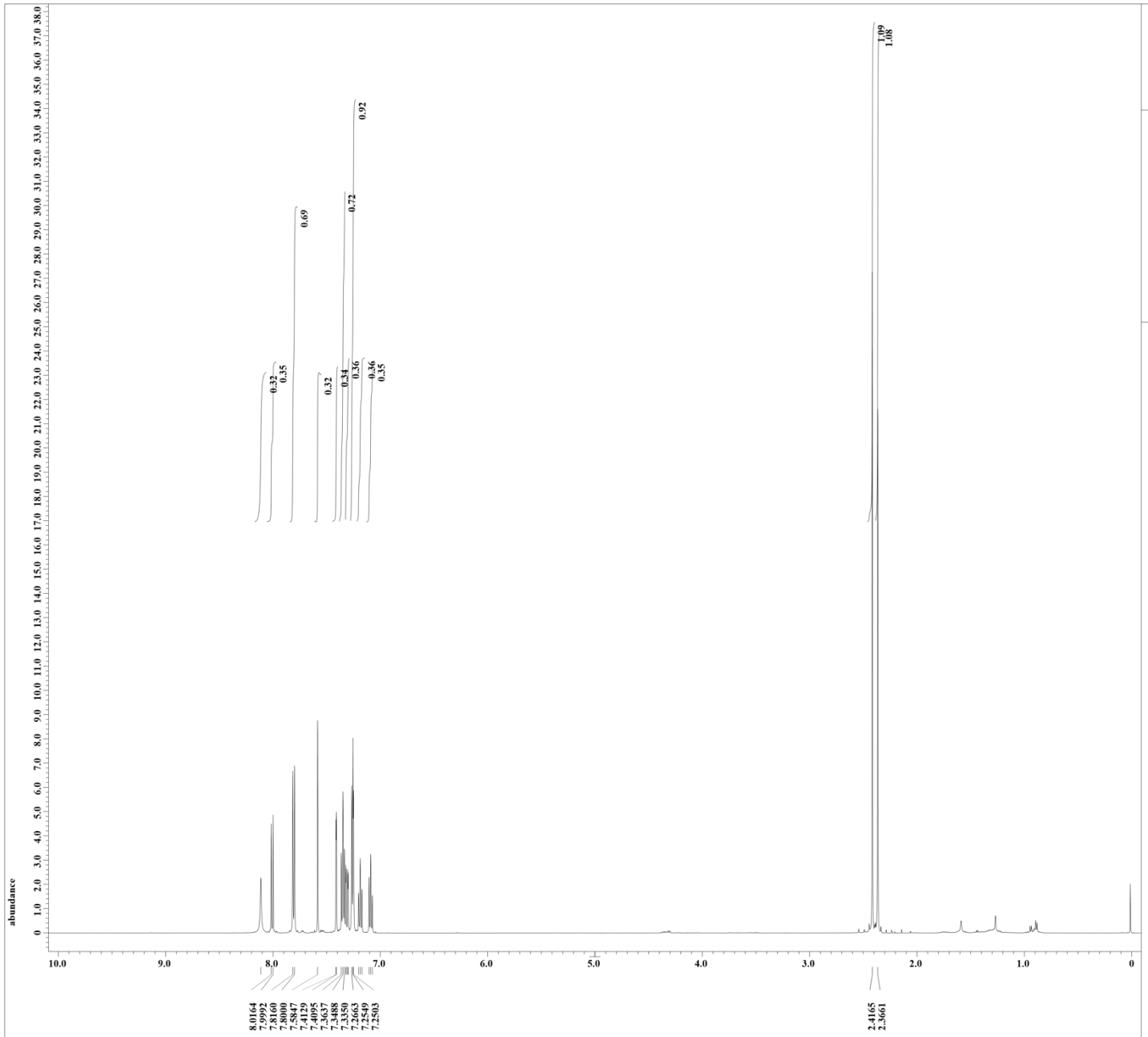



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-0604-2.jdf
```

```
Filename          = SH004-0604-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#765766
Solvent           = CHLOROFORM-D
Creation_time     = 12-APR-2000 15:17:54
Revision_time     =  2-FEB-2020 11:42:59
Current_time      =  2-FEB-2020 11:43:38

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 20611
Total_scans       = 20611

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.6[dC]
```

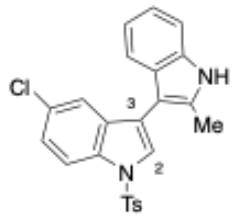


6fa

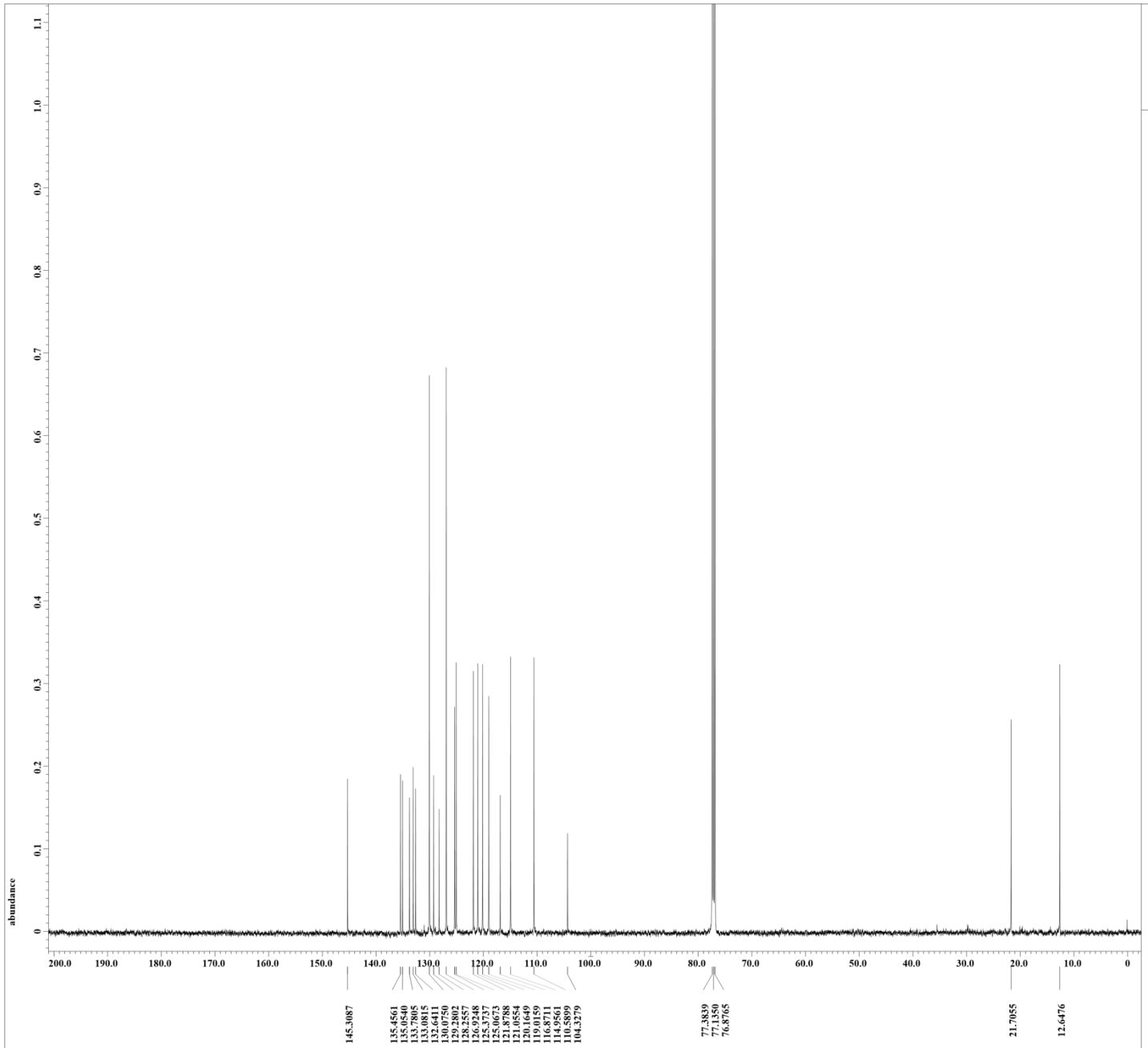


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-1301-5Cl-1.jdf
```

```
Filename          = SH004-1301-5Cl-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#528920
Solvent           = CHLOROFORM-D
Creation_time     = 17-APR-2000 16:30:01
Revision_time     =  1-FEB-2020 19:40:26
Current_time      =  1-FEB-2020 19:40:59

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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           = TRUE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 12[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 22.5[dC]
```



6ga

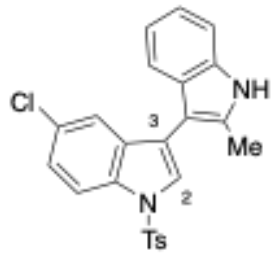


Filename = SH004-1301-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#558165
Solvent = CHLOROFORM-D
Creation_time = 31-JAN-2020 17:39:09
Revision_time = 1-FEB-2020 19:51:08
Current_time = 1-FEB-2020 19:51:57

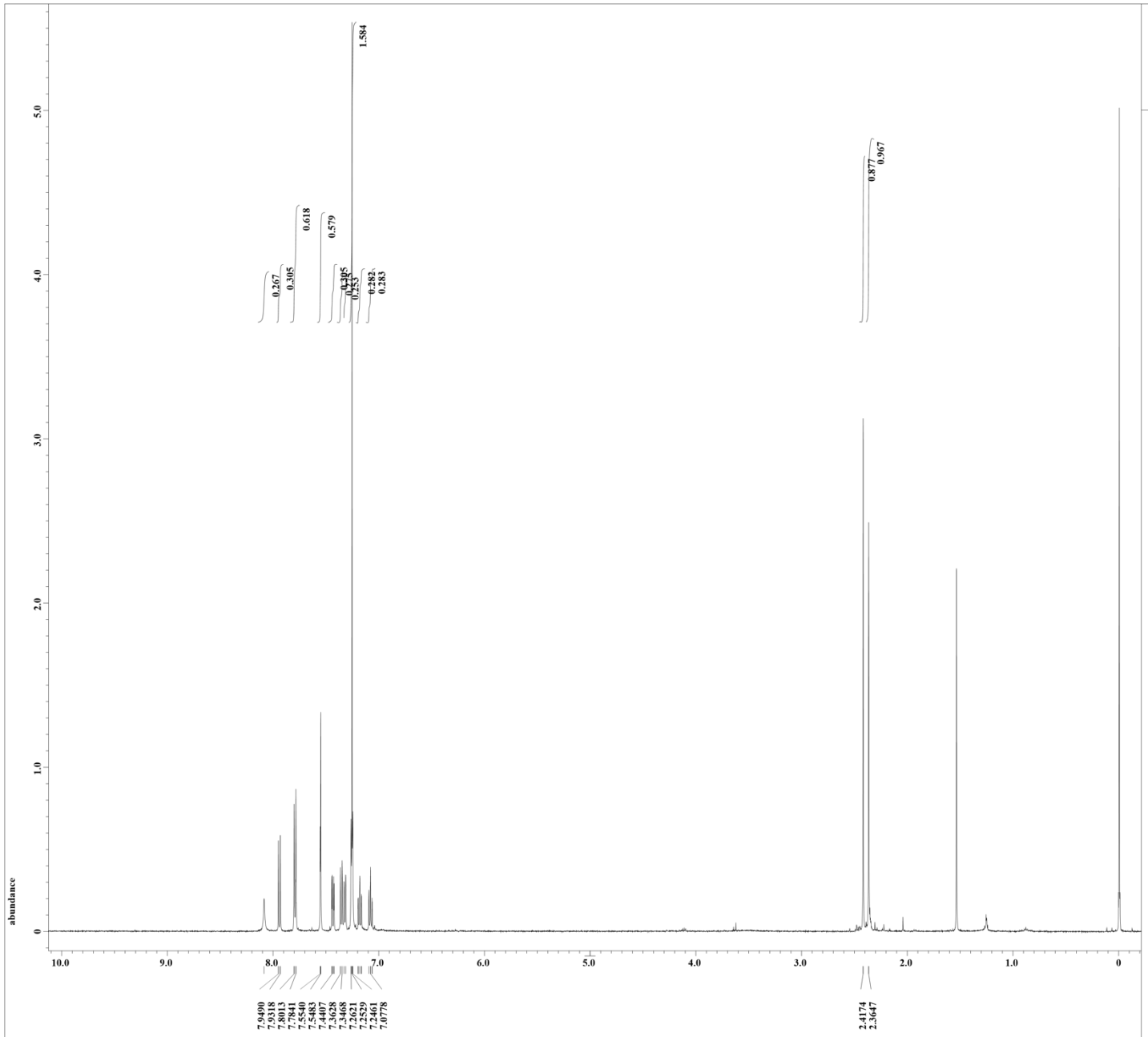
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 3862
Total_scans = 3862

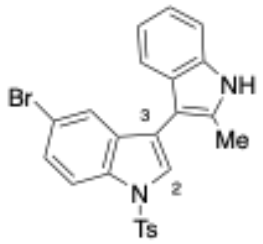
X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.5[dC]



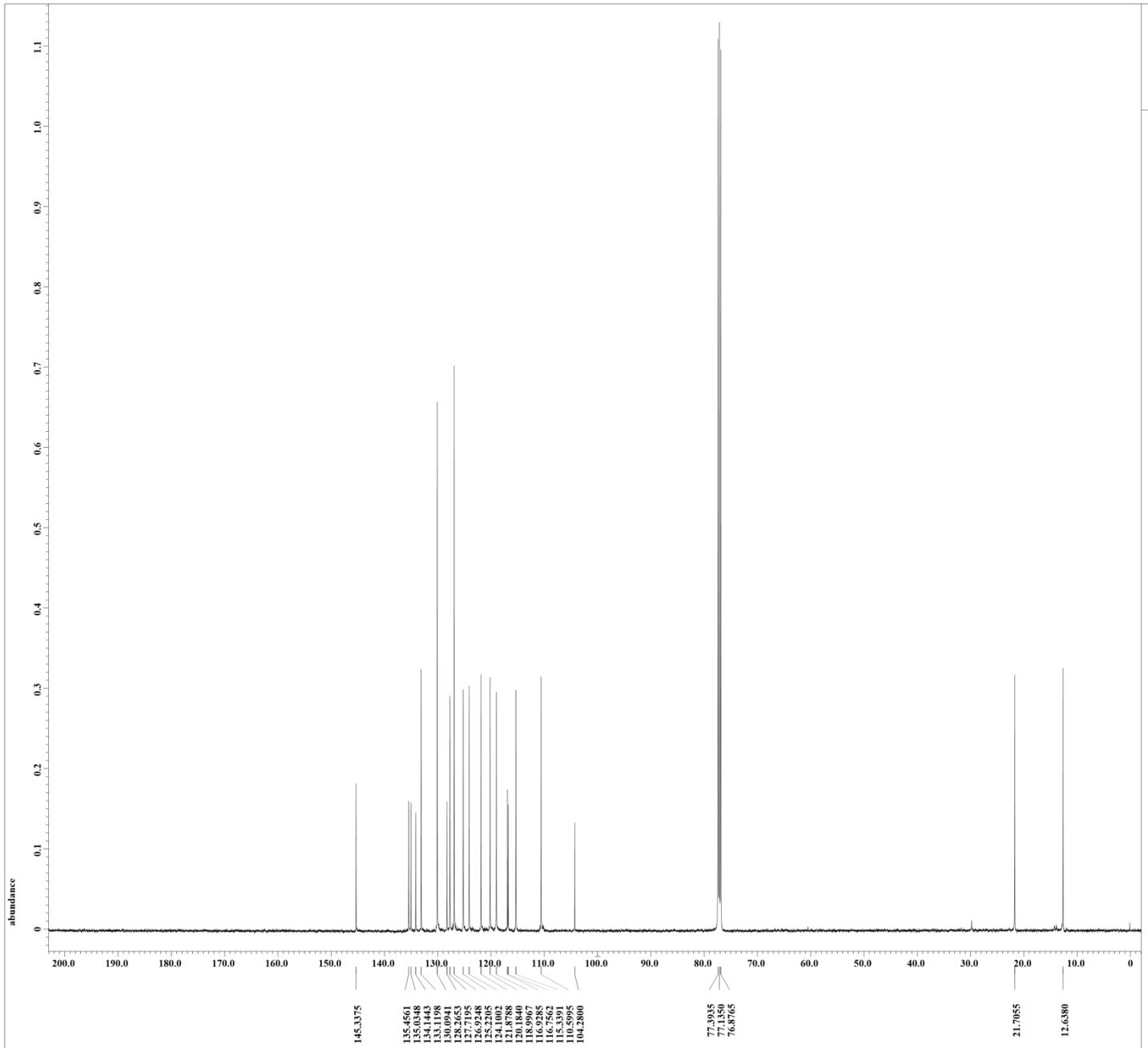
6ga




```
Filename          = TA200130-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#349605
Solvent           = CHLOROFORM-D
Creation_time     = 30-JAN-2020 08:49:11
Revision_time     = 30-JAN-2020 09:47:08
Current_time      = 30-JAN-2020 09:48:04

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.5[dC]
```



6ha

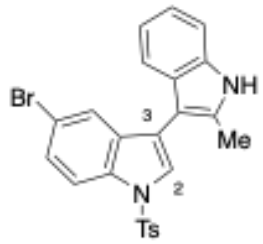


Filename = TA200130-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#630137
Solvent = CHLOROFORM-D
Creation_time = 31-JAN-2020 07:13:17
Revision_time = 31-JAN-2020 08:09:47
Current_time = 31-JAN-2020 08:10:39

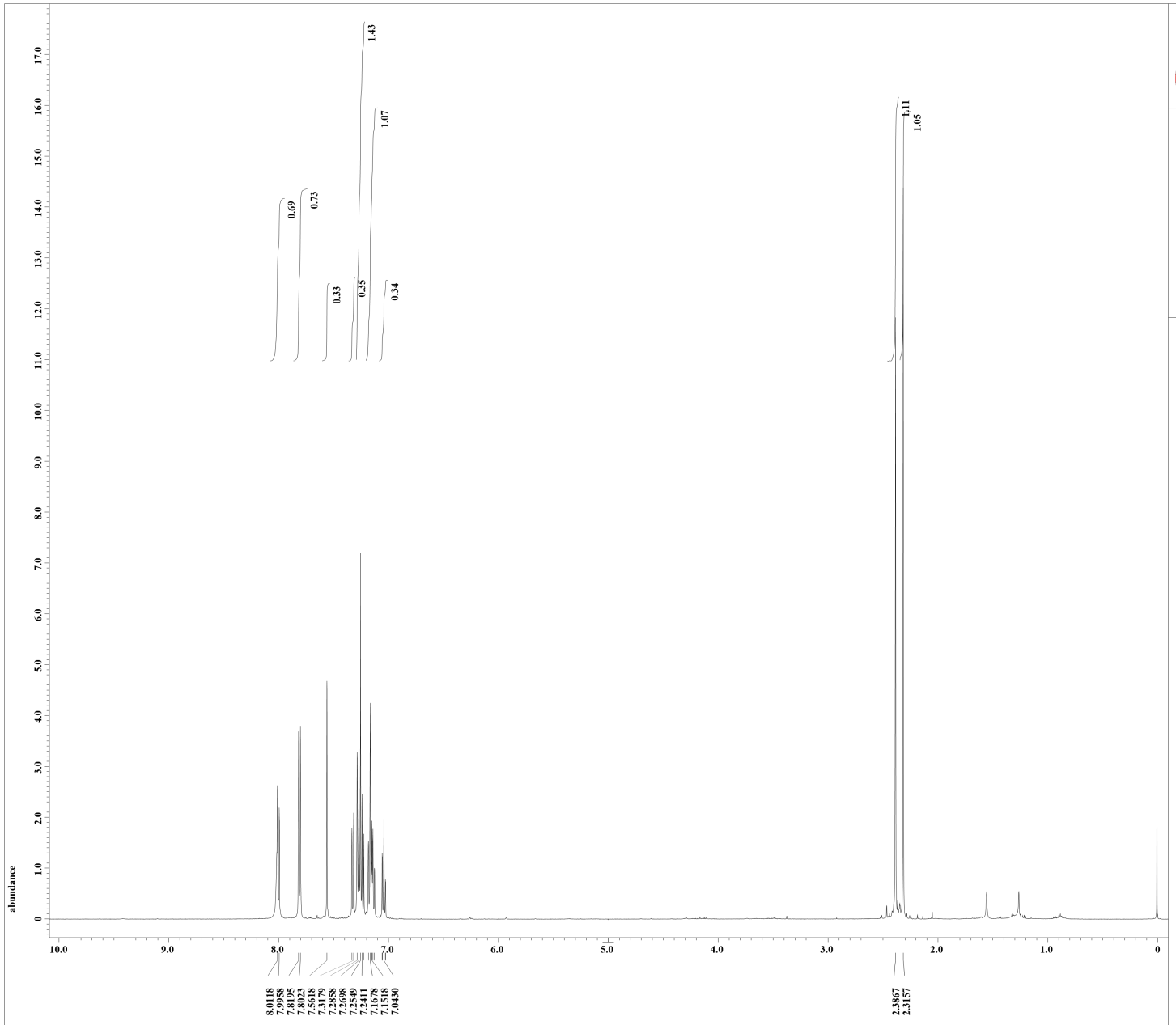
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 18528
Total_scans = 18528

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.1[dC]

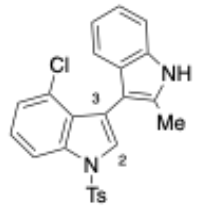


6ha

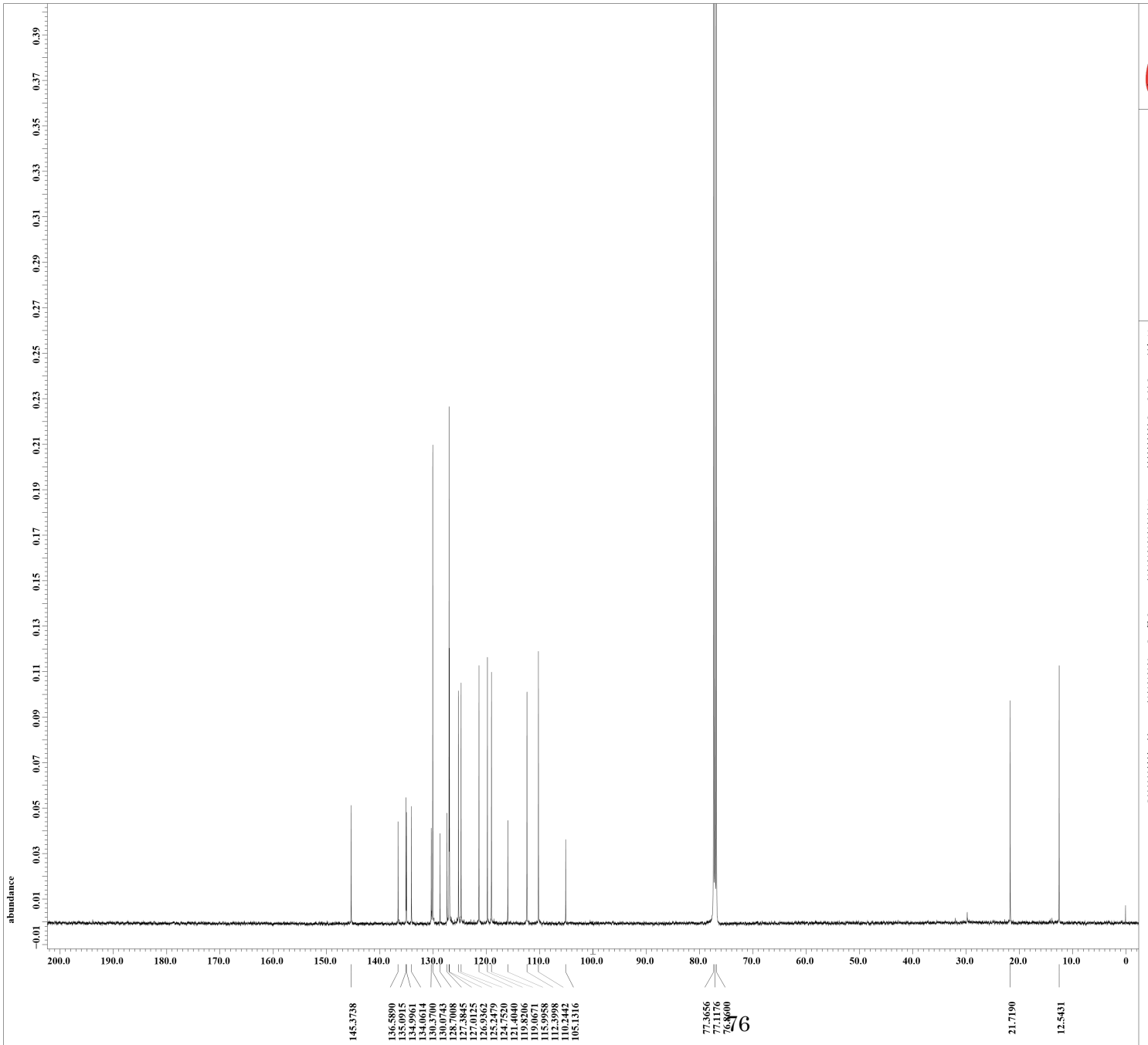


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-1603-4Cl-1.jdf
```

```
Filename          = SH004-1603-4Cl-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#618278
Solvent           = CHLOROFORM-D
Creation_time     = 19-APR-2000 18:58:52
Revision_time     =  2-FEB-2020 18:11:13
Current_time      =  2-FEB-2020 18:11:34

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 23.7[dC]
```

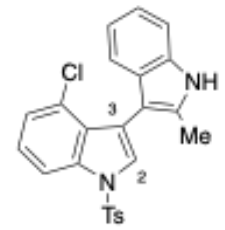


6ia

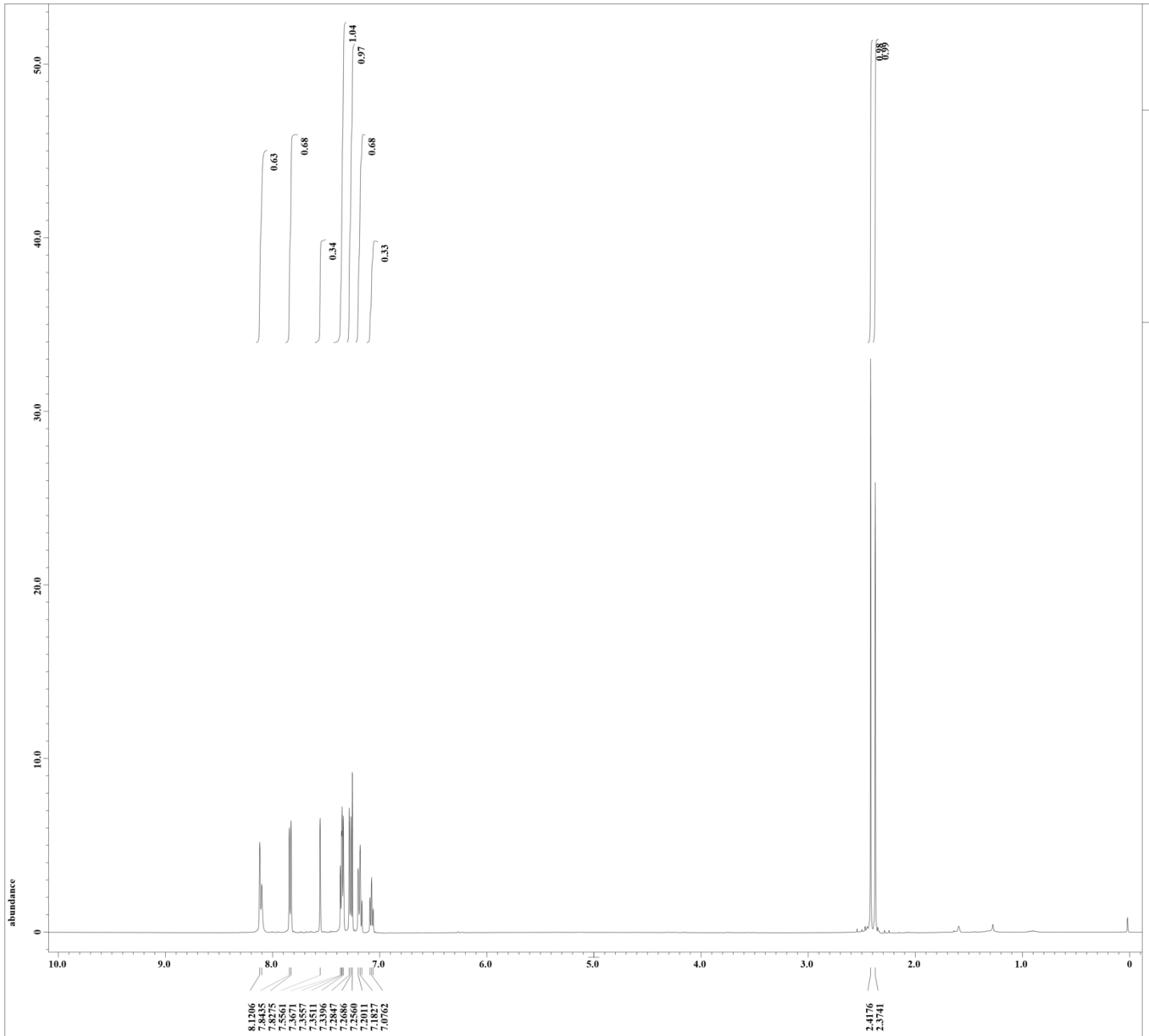



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-1603-4Cl-2.jdf
```

Filename	= SH004-1603-4Cl-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#619146
Solvent	= CHLOROFORM-D
Creation_time	= 20-APR-2000 09:43:48
Revision_time	= 3-FEB-2020 07:56:42
Current_time	= 3-FEB-2020 07:58:53
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
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	= 1
Scans	= 18715
Total_scans	= 18715
X_90_width	= 12.8[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 5.3[dB]
X_pulse	= 4.26666667[us]
Irr_atn_dec	= 21.09[dB]
Irr_atn_noe	= 21.09[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 56
Relaxation_delay	= 2[s]
Repetition_time	= 2.83361792[s]
Temp_get	= 25.1[dC]



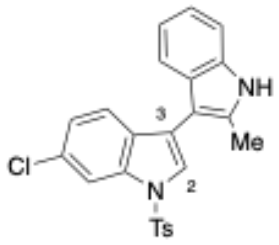
6ia



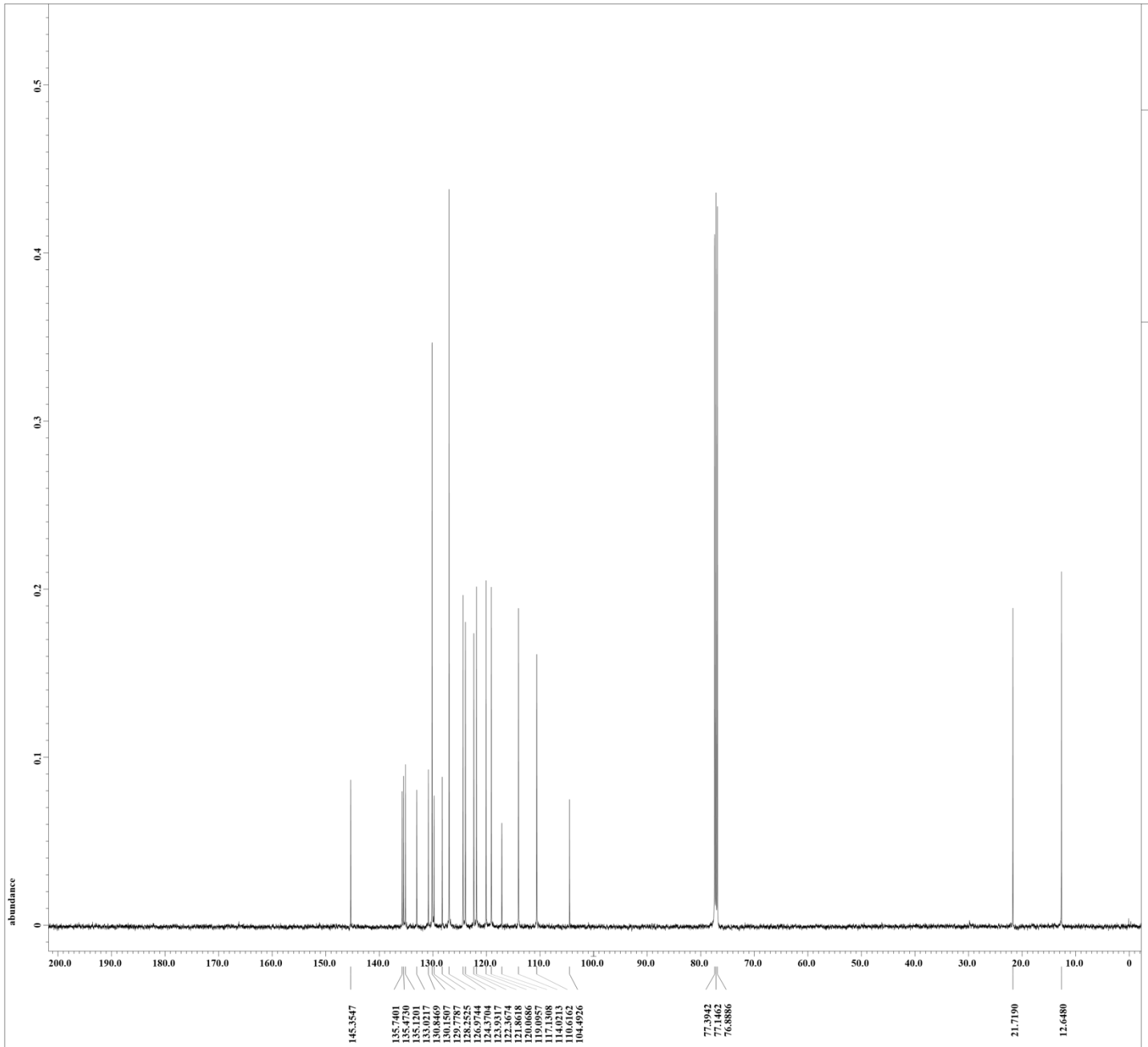
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: SH004-1504-6Cl-1.jdf

Filename	= SH004-1504-6Cl-6.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#323329
Solvent	= CHLOROFORM-D
Creation_time	= 19-APR-2000 10:47:18
Revision_time	= 2-FEB-2020 11:39:07
Current_time	= 2-FEB-2020 11:39:28
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA500
Spectrometer	= DELTA2_NMR
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	= 1
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	= TRUE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 12[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 3.4[dB]
X_pulse	= 6[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	= 23.7[dC]



6ja



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

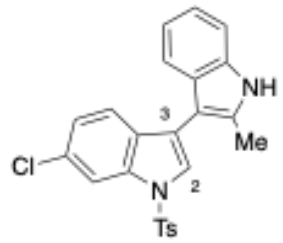
Derived from: SH004-1504-6Cl-2.jdf

Filename = SH004-1504-6Cl-4.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#325162
Solvent = CHLOROFORM-D
Creation_time = 19-APR-2000 13:12:47
Revision_time = 2-FEB-2020 11:39:40
Current_time = 2-FEB-2020 11:40:15

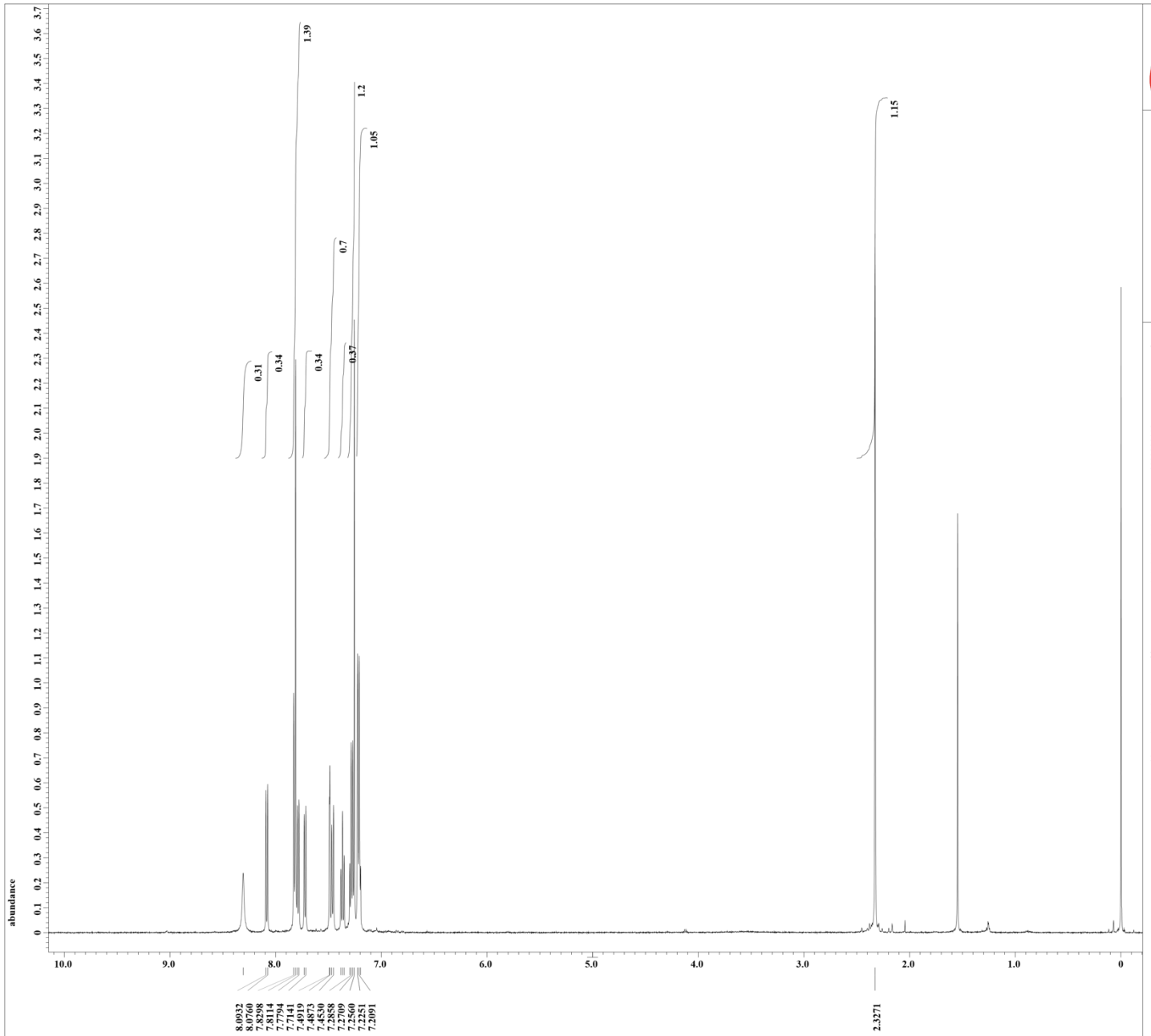
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
Scans = 3025
Total_scans = 3025

X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.9[dC]



6ja



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

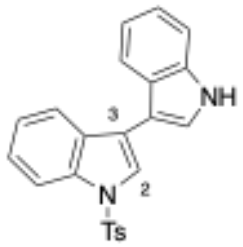
Derived from: TA2019-1231-8.jdf

Filename = TA2019-1231-18.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#540954
Solvent = CHLOROFORM-D
Creation_time = 17-MAR-2000 16:51:07
Revision_time = 1-FEB-2020 23:41:42
Current_time = 1-FEB-2020 23:41:59

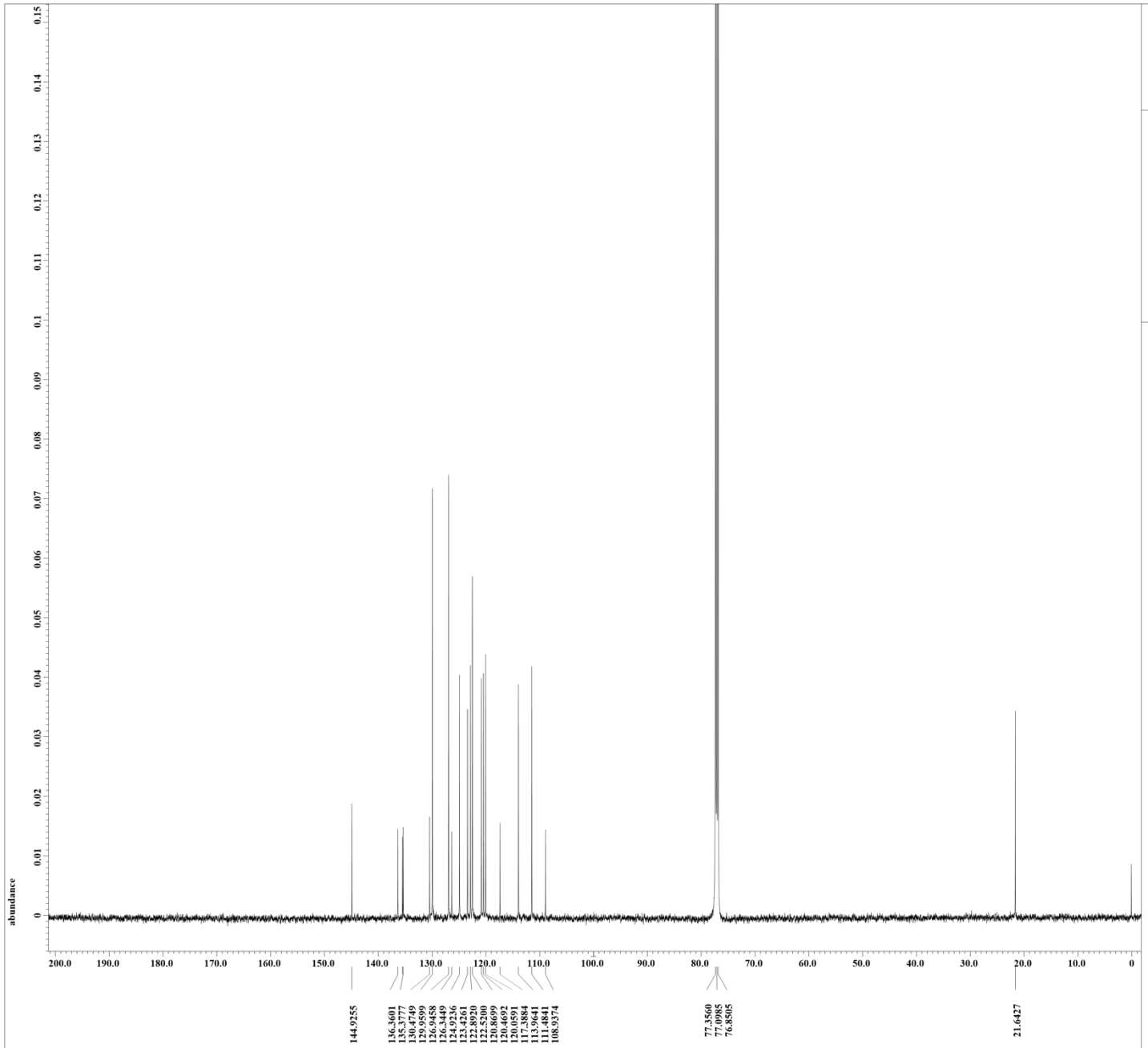
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 22.5[dC]



6ab



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

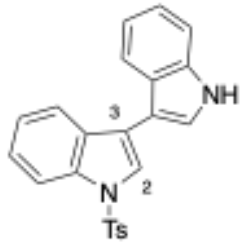
Derived from: TA2019-1231-14.jdf

Filename = TA2019-1231-17.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#635226
Solvent = CHLOROFORM-D
Creation_time = 18-MAR-2000 14:56:56
Revision_time = 1-FEB-2020 23:42:13
Current_time = 1-FEB-2020 23:42:45

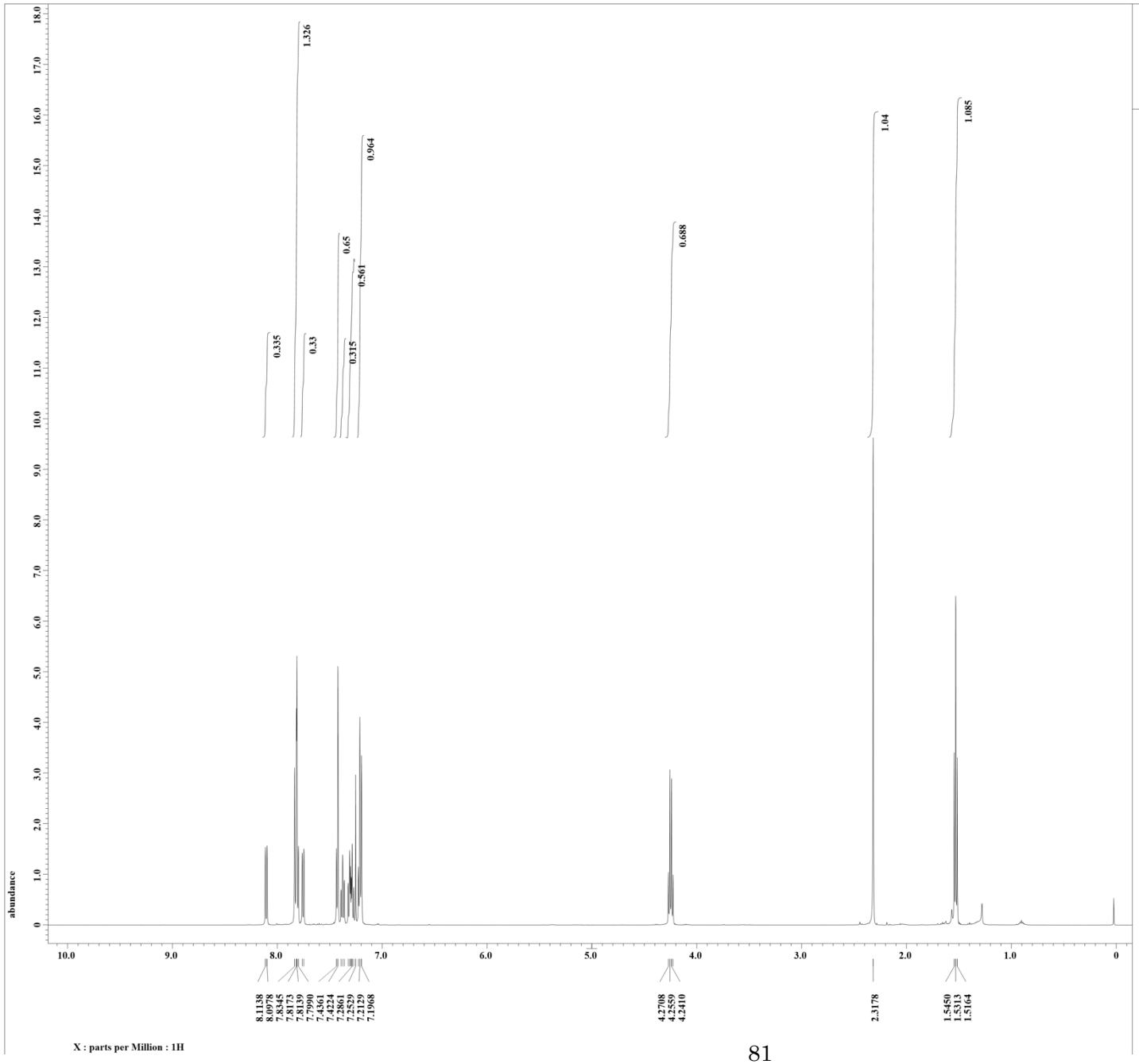
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
Scans = 24757.0
Total_scans = 24757.0

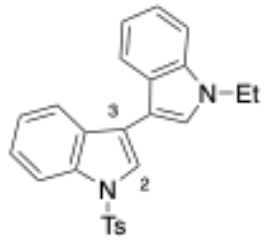
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 56
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 24.8[dC]



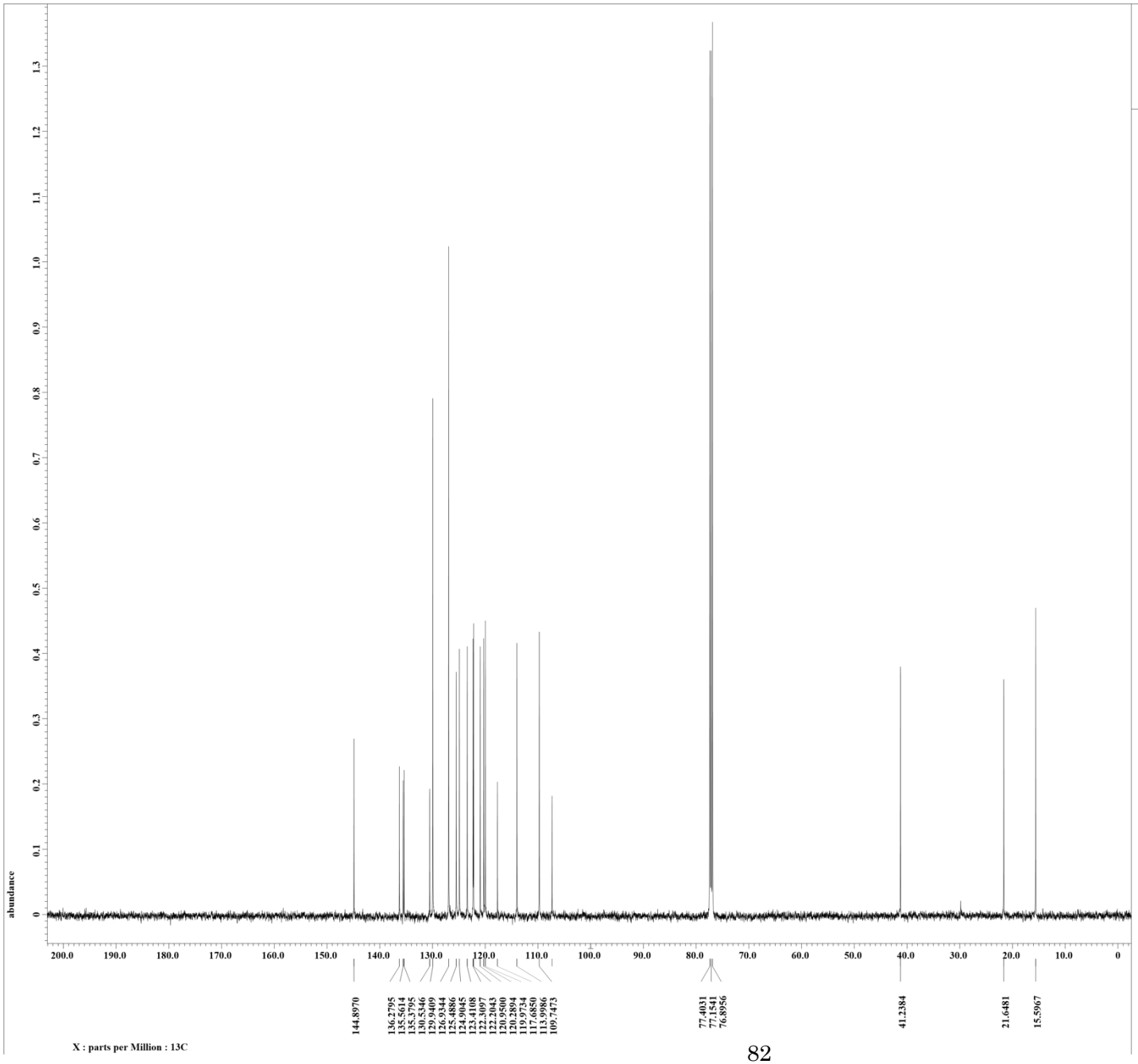
6ab



```
Filename          = TA200128-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#701193
Solvent           = CHLOROFORM-D
Creation_time     = 28-JAN-2020 18:35:51
Revision_time     = 28-JAN-2020 19:36:00
Current_time      = 28-JAN-2020 19:36:48

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.4[dC]
```



6ac

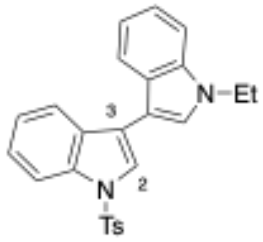


Filename = TA200128-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#703009
Solvent = CHLOROFORM-D
Creation_time = 28-JAN-2020 19:30:31
Revision_time = 28-JAN-2020 20:26:17
Current_time = 28-JAN-2020 20:28:37

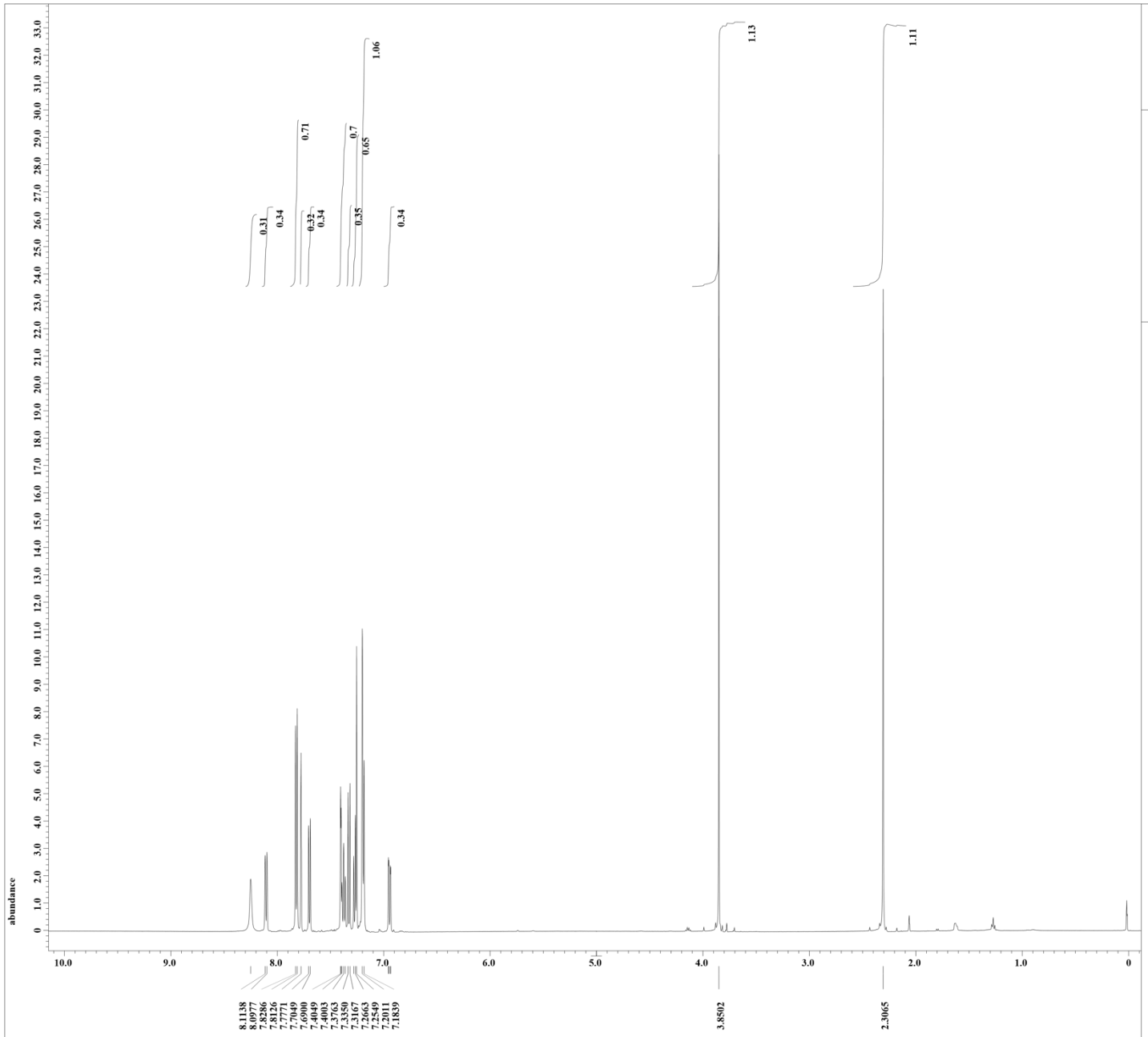
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1108
Total_scans = 1108

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24[dC]



6ac



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

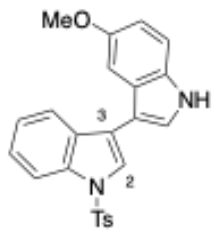
Derived from: TA2020-0106-6.jdf

Filename = TA2020-0106-17.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#603947
Solvent = CHLOROFORM-D
Creation_time = 23-MAR-2000 18:35:59
Revision_time = 1-FEB-2020 18:55:43
Current_time = 1-FEB-2020 18:56:05

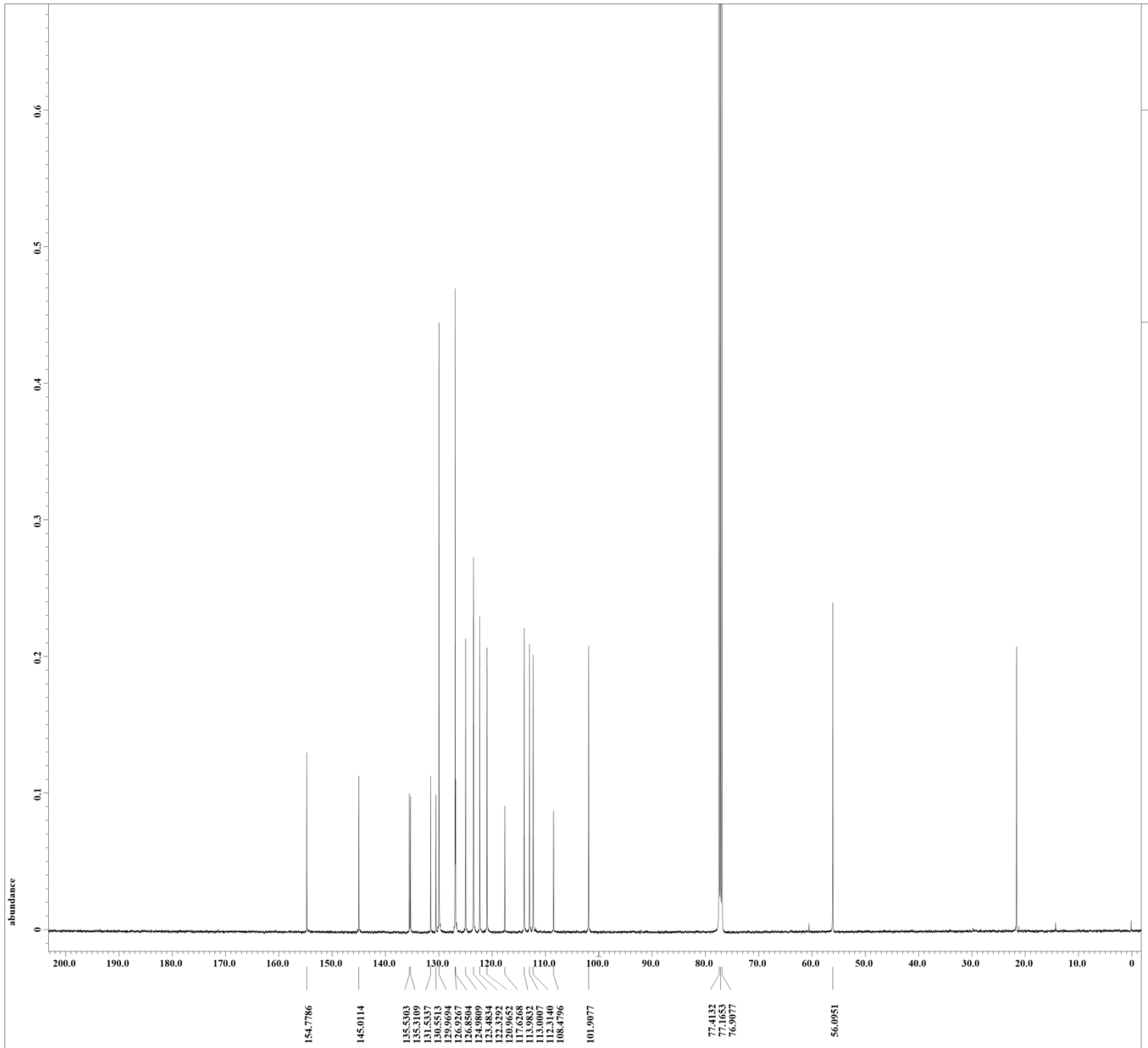
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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 = TRUE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 12[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 23.5[dC]



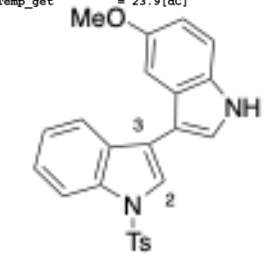
6ae



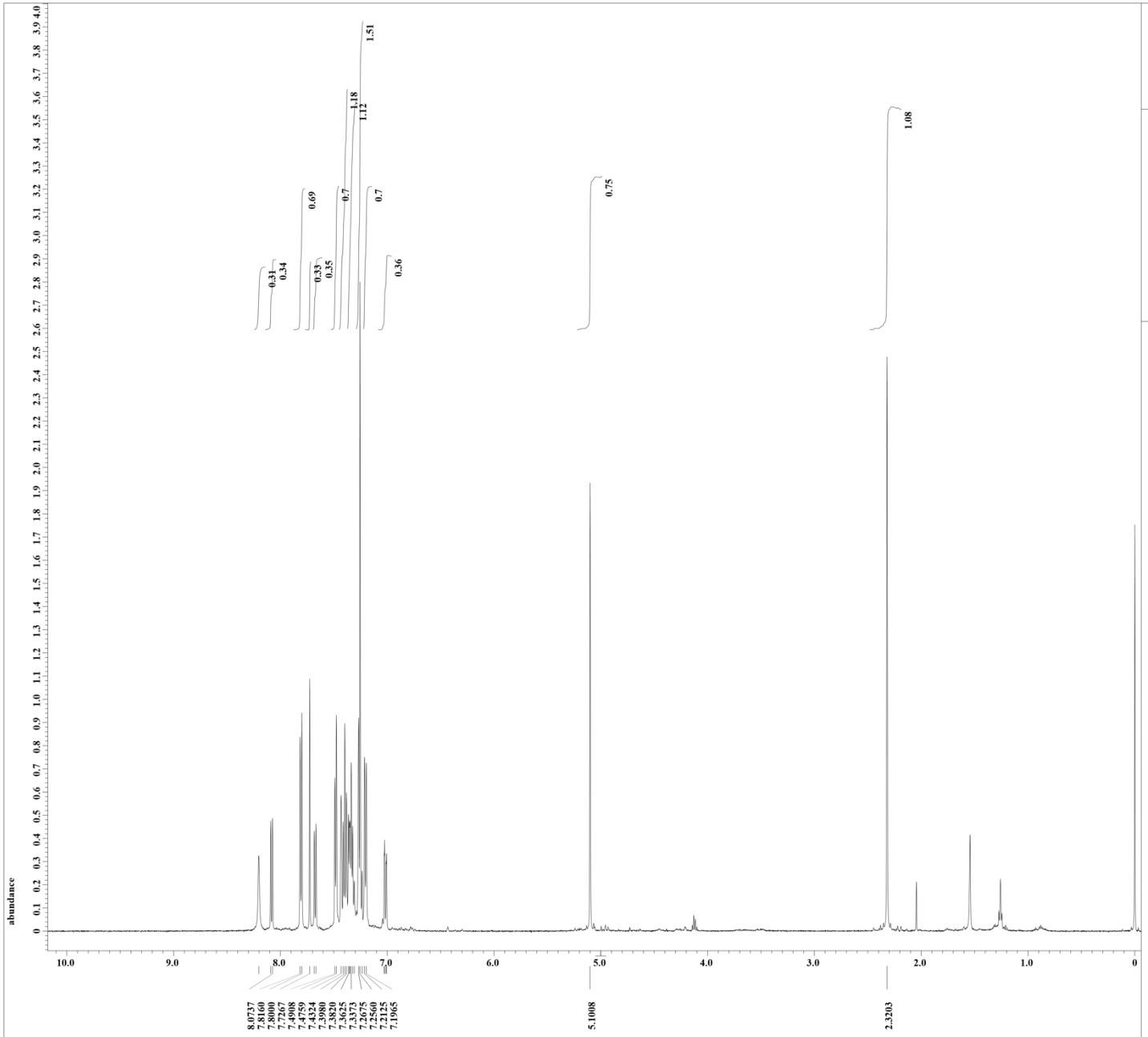

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0106-14.jdf
```



```
Filename          = TA2020-0106-18.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#731689
Solvent           = CHLOROFORM-D
Creation_time     = 24-MAR-2000 09:54:49
Revision_time     =  1-FEB-2020 18:51:11
Current_time      =  1-FEB-2020 18:51:48

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 14959
Total_scans       = 14959

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 23.9[dC]
```

6ae



---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

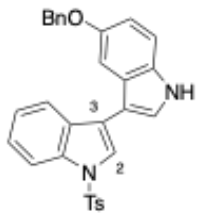
Derived from: TA2020-0102-1.jdf

Filename = TA2020-0102-9.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#721749
Solvent = CHLOROFORM-D
Creation_time = 18-MAR-2000 21:52:23
Revision_time = 1-FEB-2020 23:36:07
Current_time = 1-FEB-2020 23:36:31

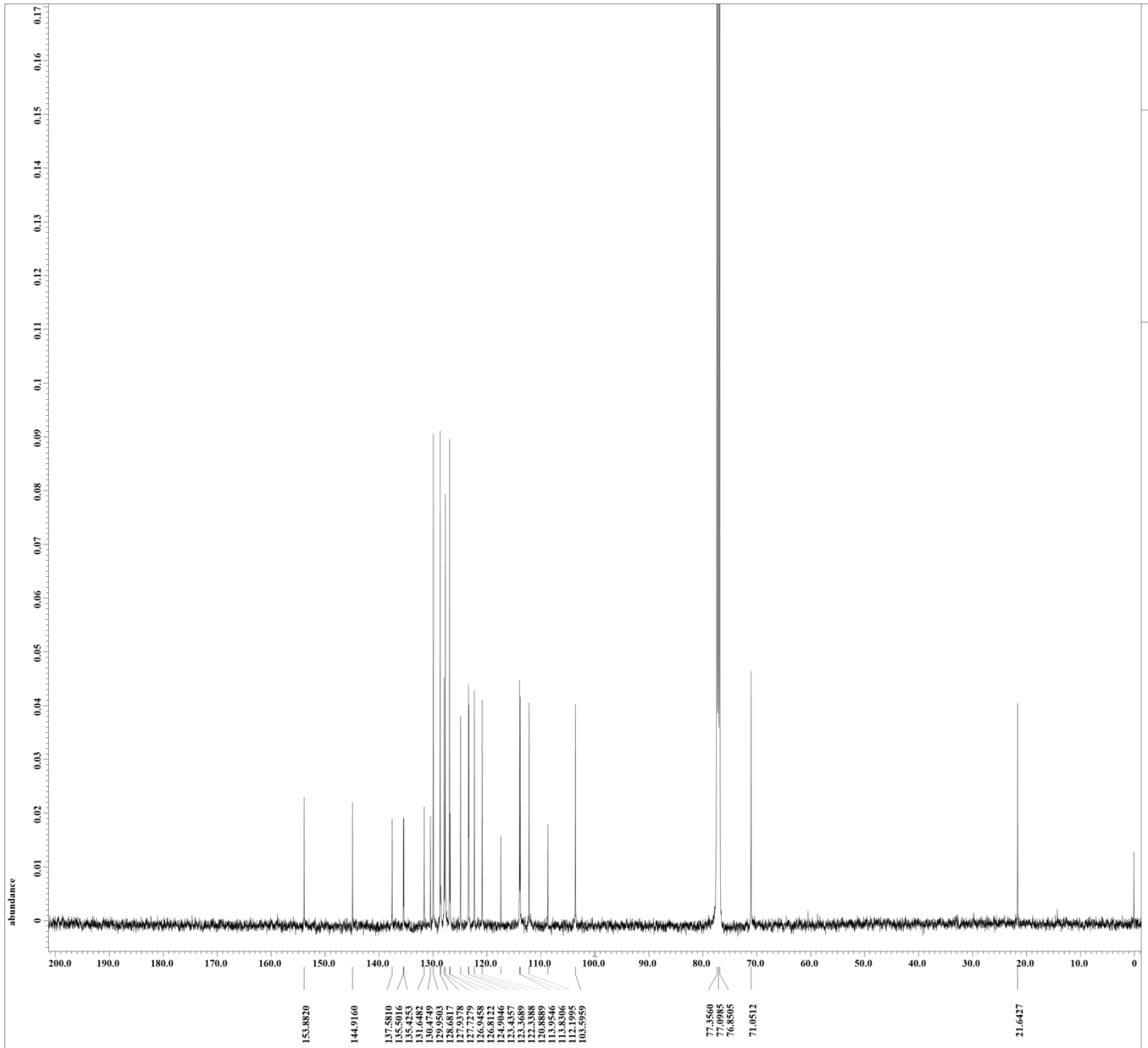
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 1
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[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 6[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 = 23.9[dC]



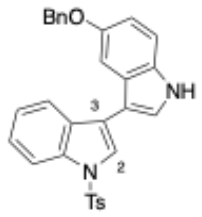
6af



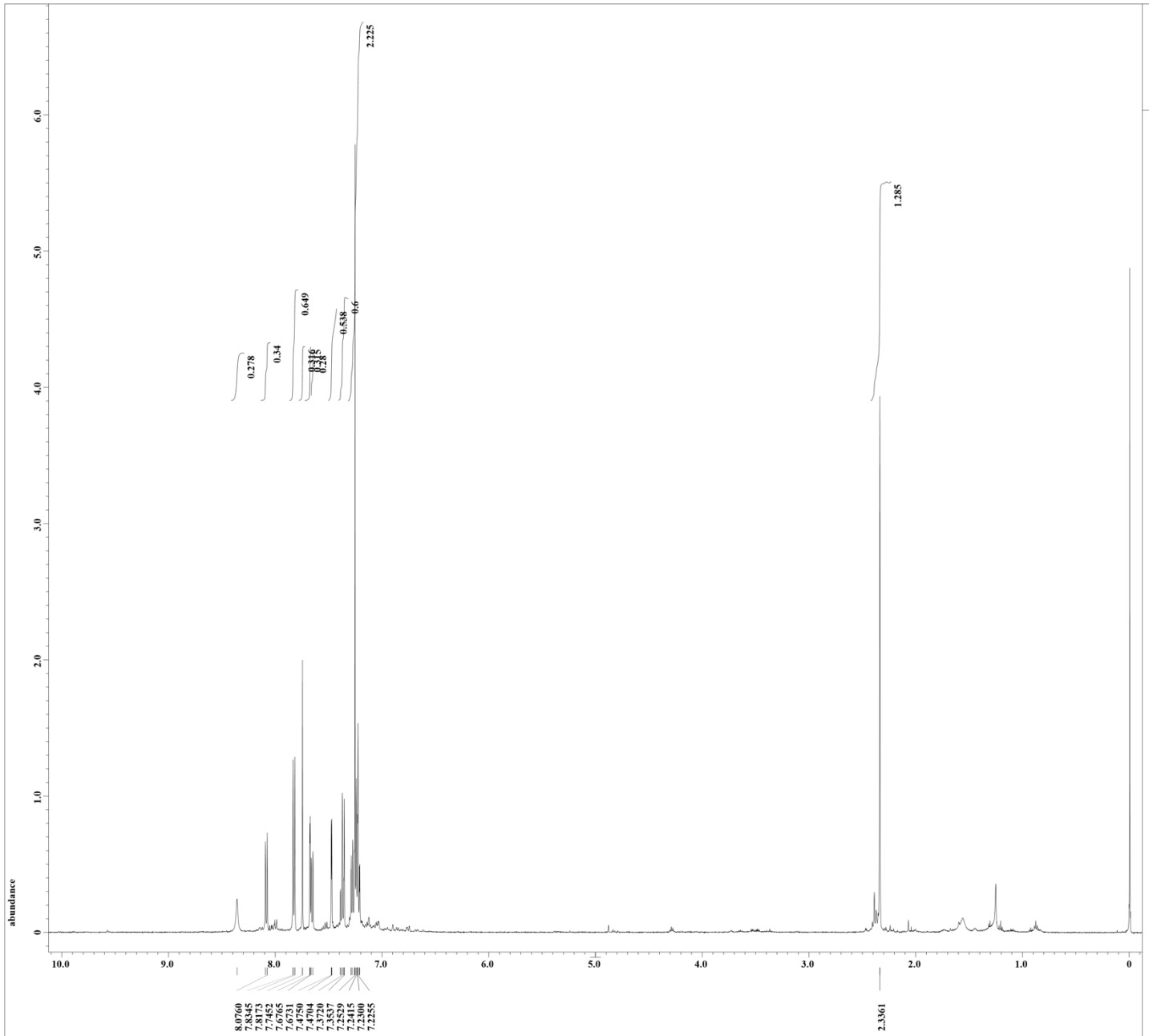

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0102-2.jdf
```



```
Filename          = TA2020-0102-8.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#722581
Solvent           = CHLOROFORM-D
Creation_time     = 19-MAR-2000 13:02:20
Revision_time     =  1-FEB-2020 23:31:22
Current_time      =  1-FEB-2020 23:32:10

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 19250
Total_scans       = 19250

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.9[dC]
```

6af

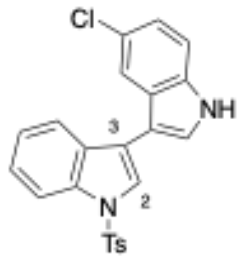


Filename = TA200103-6.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#421477
Solvent = CHLOROFORM-D
Creation_time = 3-JAN-2020 10:53:25
Revision_time = 1-FEB-2020 18:21:46
Current_time = 1-FEB-2020 18:22:26

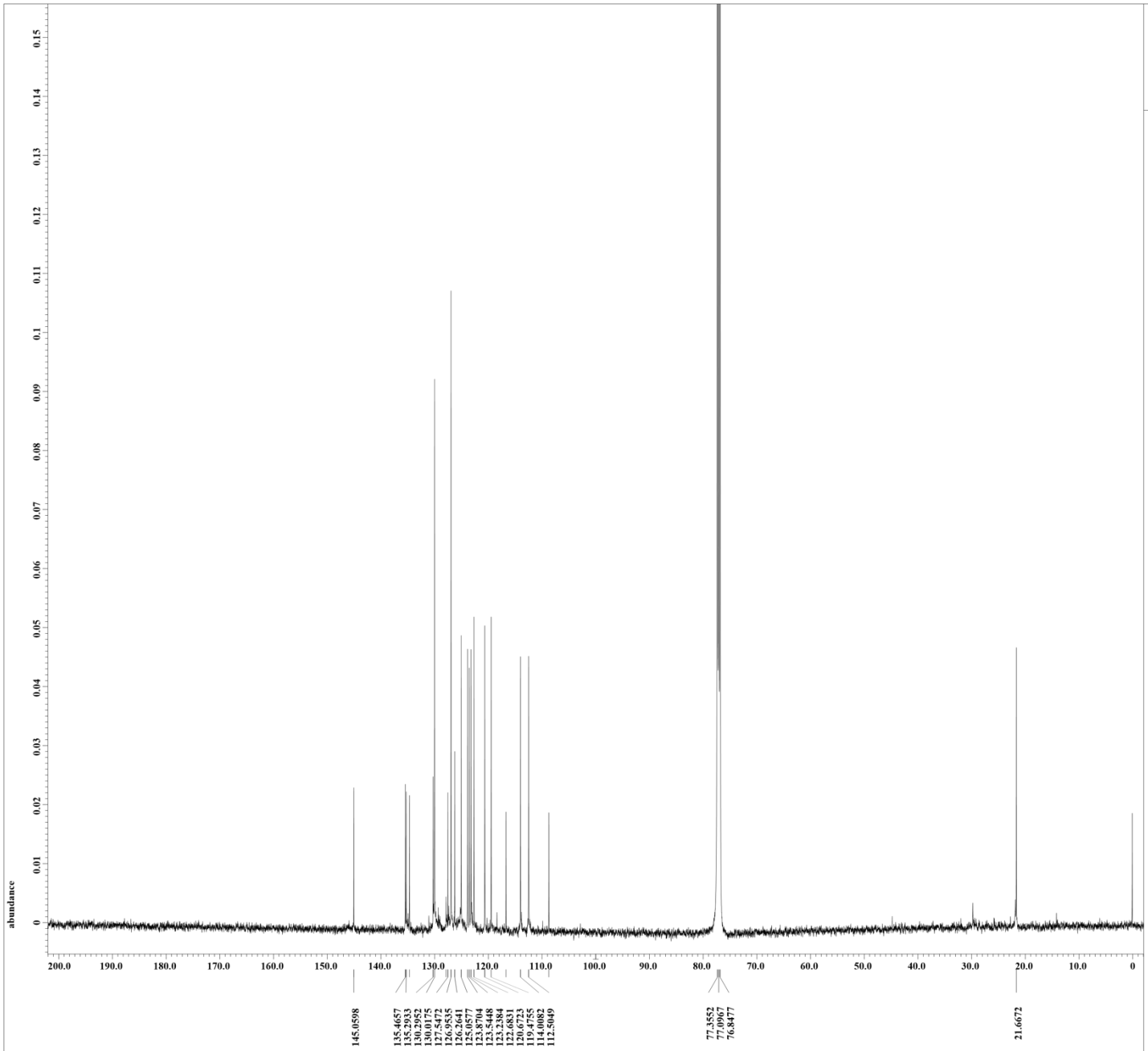
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 22.8[dC]

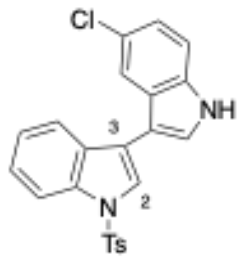


6ag

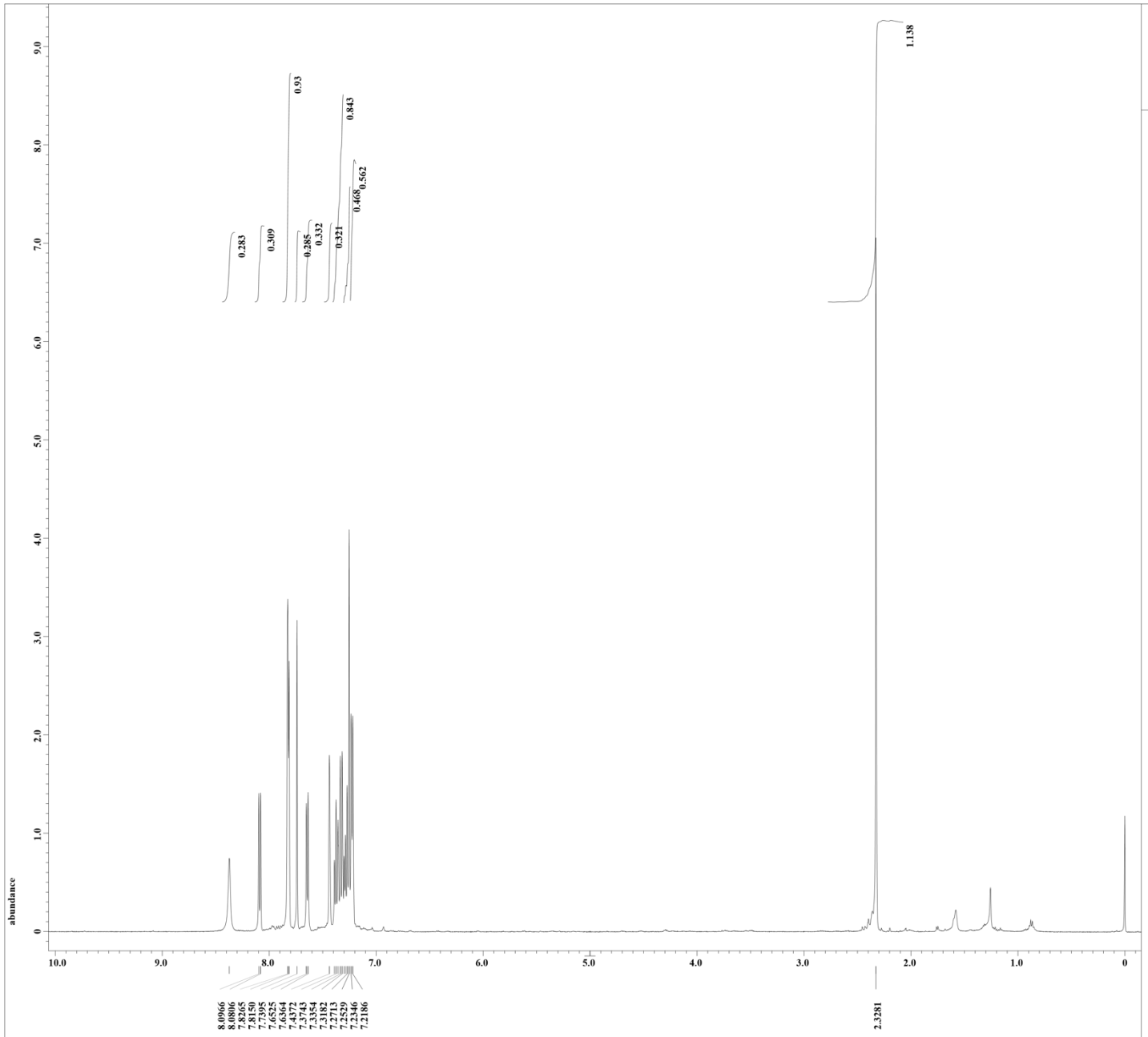


JEOL

```
Filename          = TA200103-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#422774
Solvent           = CHLOROFORM-D
Creation_time     =  6-JAN-2020 07:22:18
Revision_time     =  1-FEB-2020 18:23:43
Current_time      =  1-FEB-2020 18:24:12

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 86811
Total_scans       = 86811

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 23.5[dC]
```



6ag

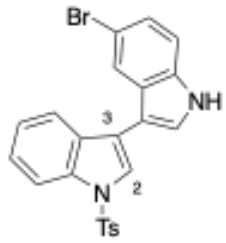


Filename = TA200109-21.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#489122
Solvent = CHLOROFORM-D
Creation_time = 9-JAN-2020 12:45:35
Revision_time = 1-FEB-2020 18:31:14
Current_time = 1-FEB-2020 18:31:38

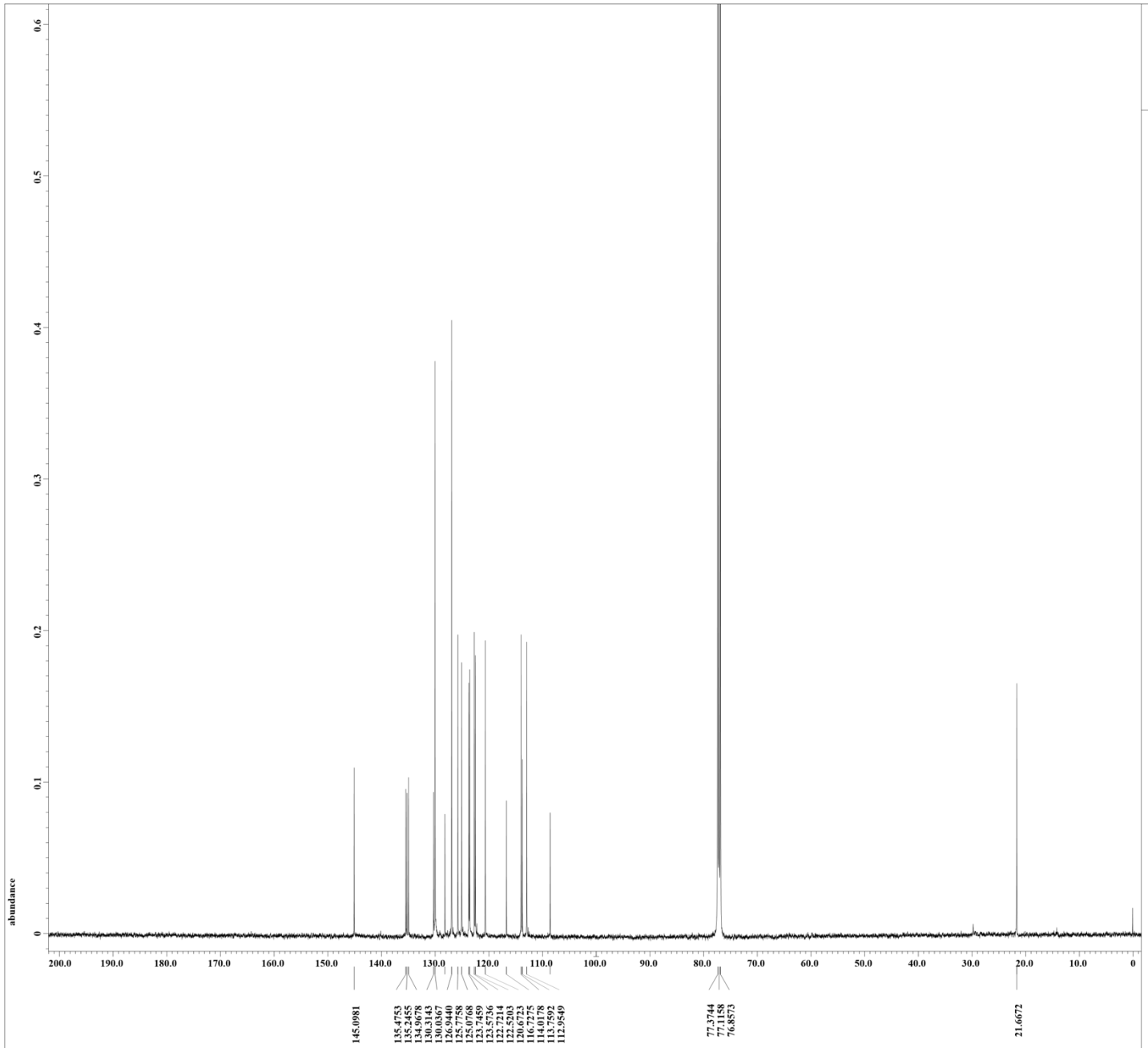
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.6[dC]

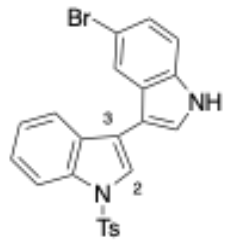


6ah

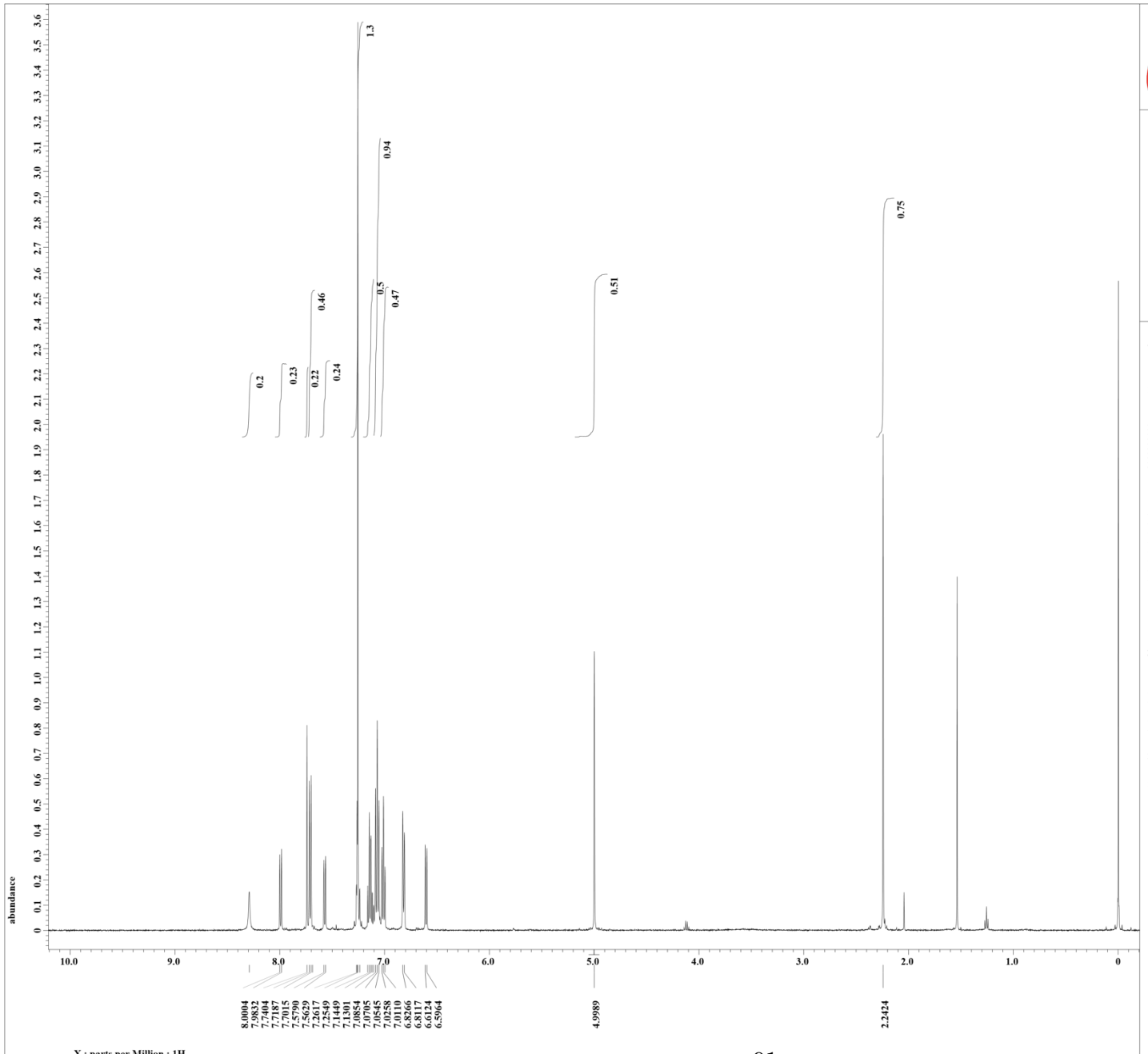


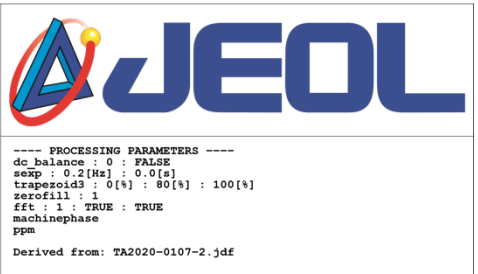
JEOL

```
Filename          = TA200109-19.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#653477
Solvent           = CHLOROFORM-D
Creation_time     = 10-JAN-2020 08:12:59
Revision_time     =  1-FEB-2020 18:31:57
Current_time      =  1-FEB-2020 18:32:26

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 18907
Total_scans       = 18907

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 23.8[dC]
```



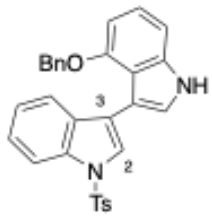
6ah



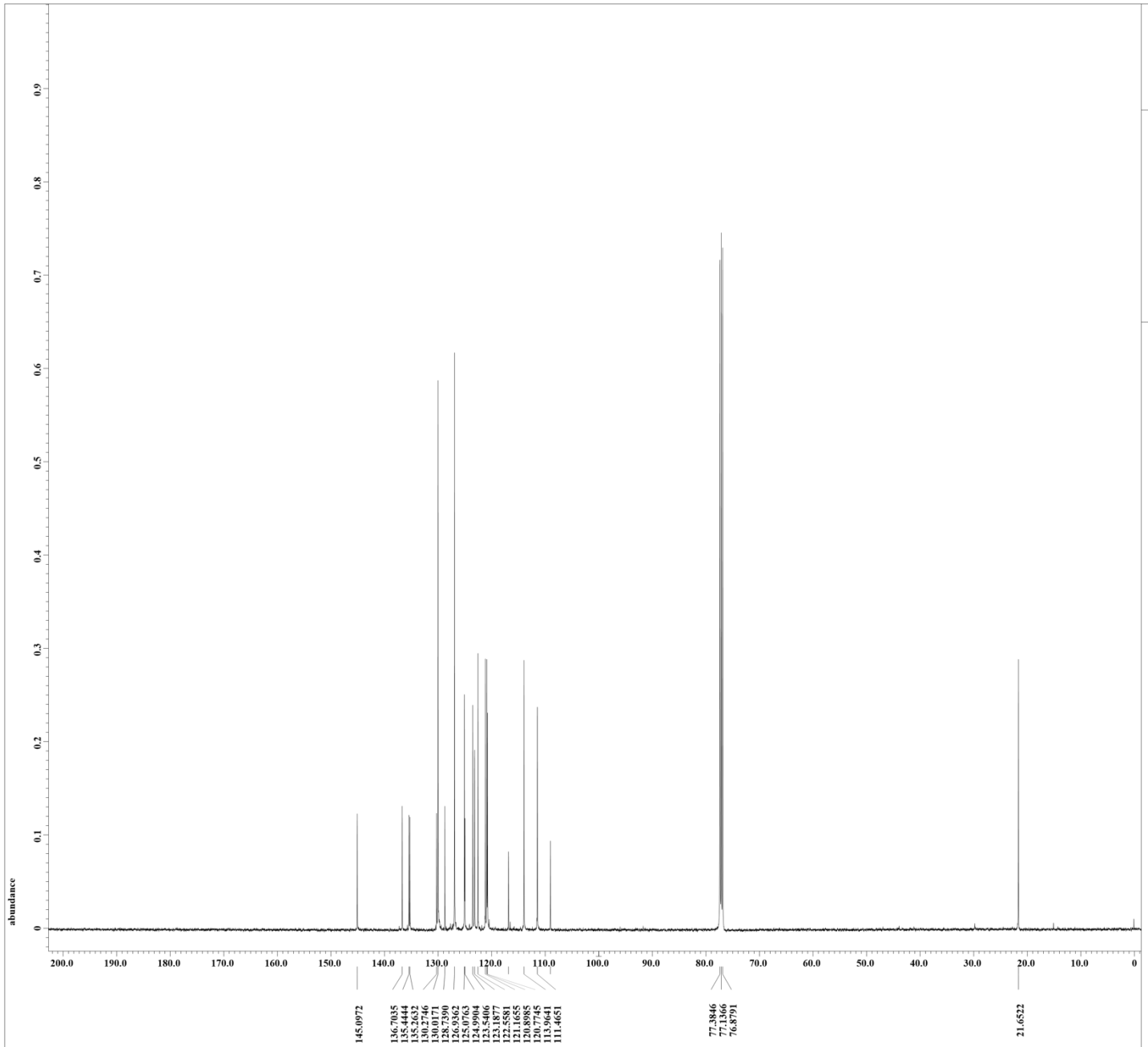


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0107-2.jdf
```

```
Filename          = TA2020-0107-21.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#442818
Solvent           = CHLOROFORM-D
Creation_time     = 25-MAR-2000 14:07:15
Revision_time     =  1-FEB-2020 19:09:59
Current_time      =  1-FEB-2020 19:12:22

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 23.2[dC]
```

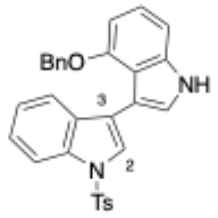


6ai

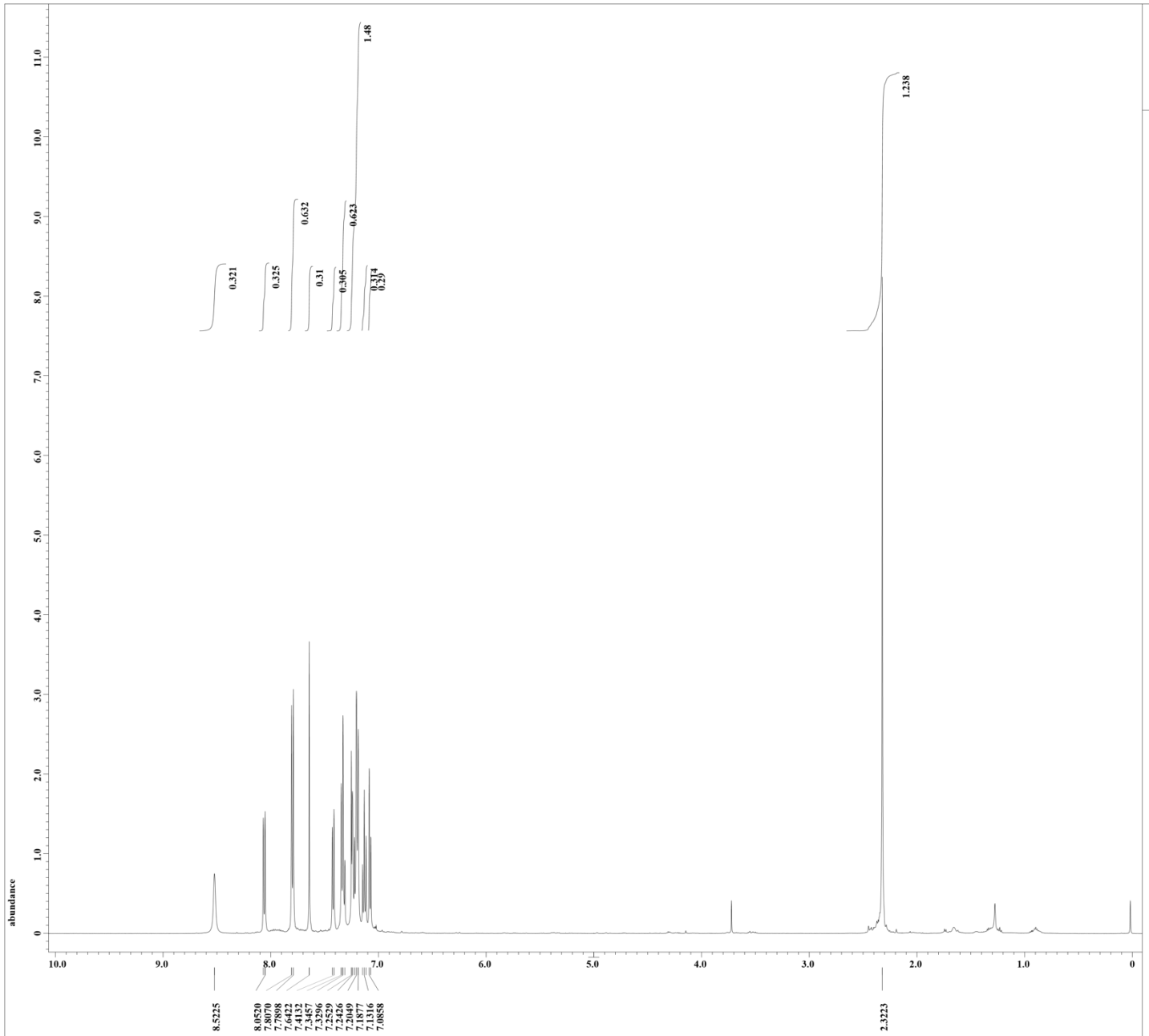



```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0107-11.jdf
```

```
Filename          = TA2020-0107-21.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#676370
Solvent           = CHLOROFORM-D
Creation_time     = 26-MAR-2000 10:13:38
Revision_time     =  1-FEB-2020 19:00:20
Current_time      =  1-FEB-2020 19:00:54

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 17313
Total_scans       = 17313

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.6[dC]
```



6ai

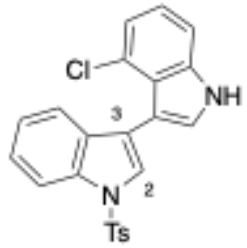


Filename = TA200102-12.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#462431
Solvent = CHLOROFORM-D
Creation_time = 2-JAN-2020 12:02:06
Revision_time = 1-FEB-2020 18:46:17
Current_time = 1-FEB-2020 18:46:40

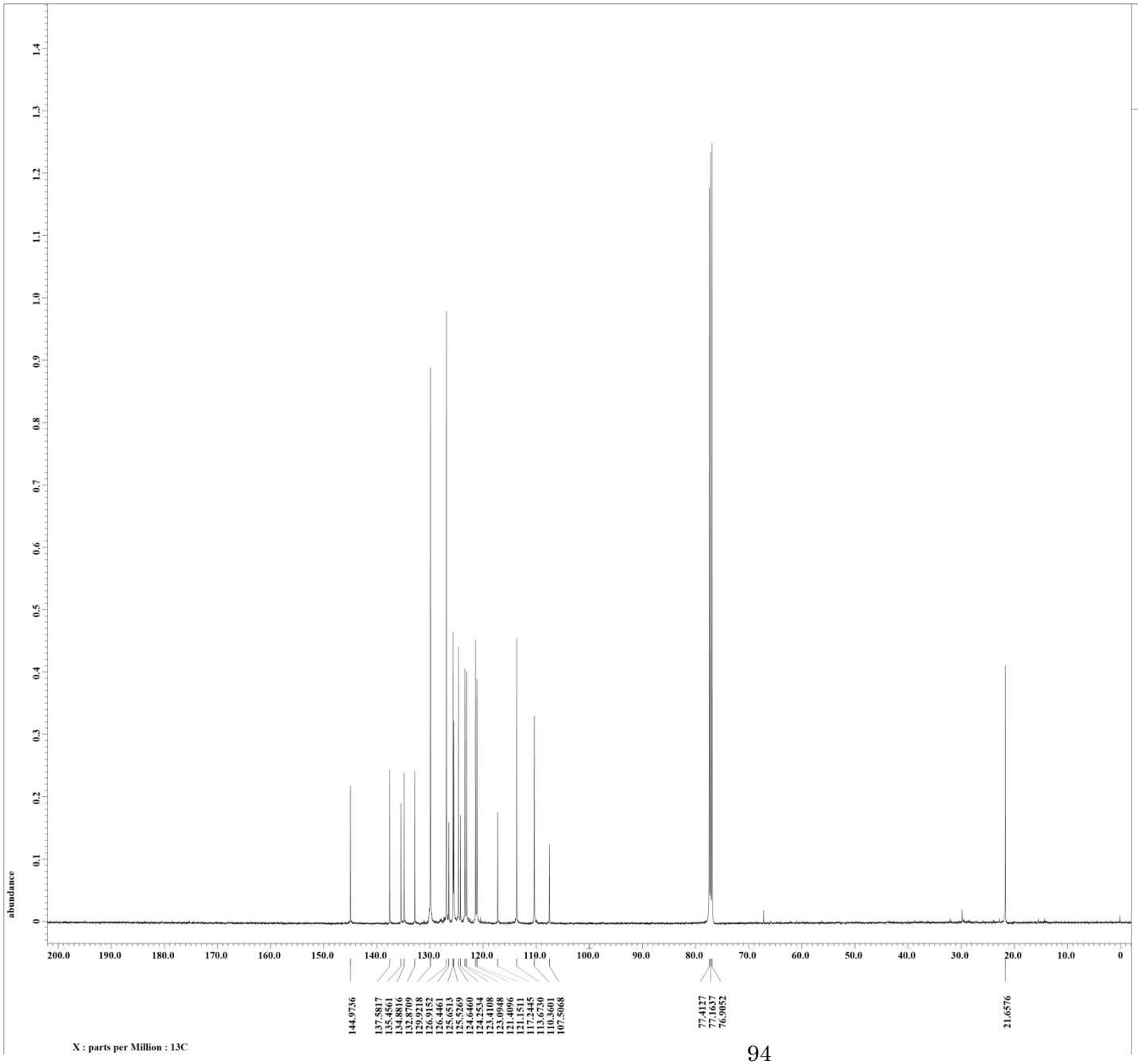
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.2[dC]



6aj

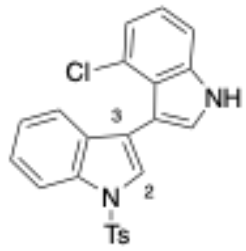


Filename = TA200102-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#499169
Solvent = CHLOROFORM-D
Creation_time = 3-JAN-2020 09:44:28
Revision_time = 1-FEB-2020 18:48:00
Current_time = 1-FEB-2020 18:48:39

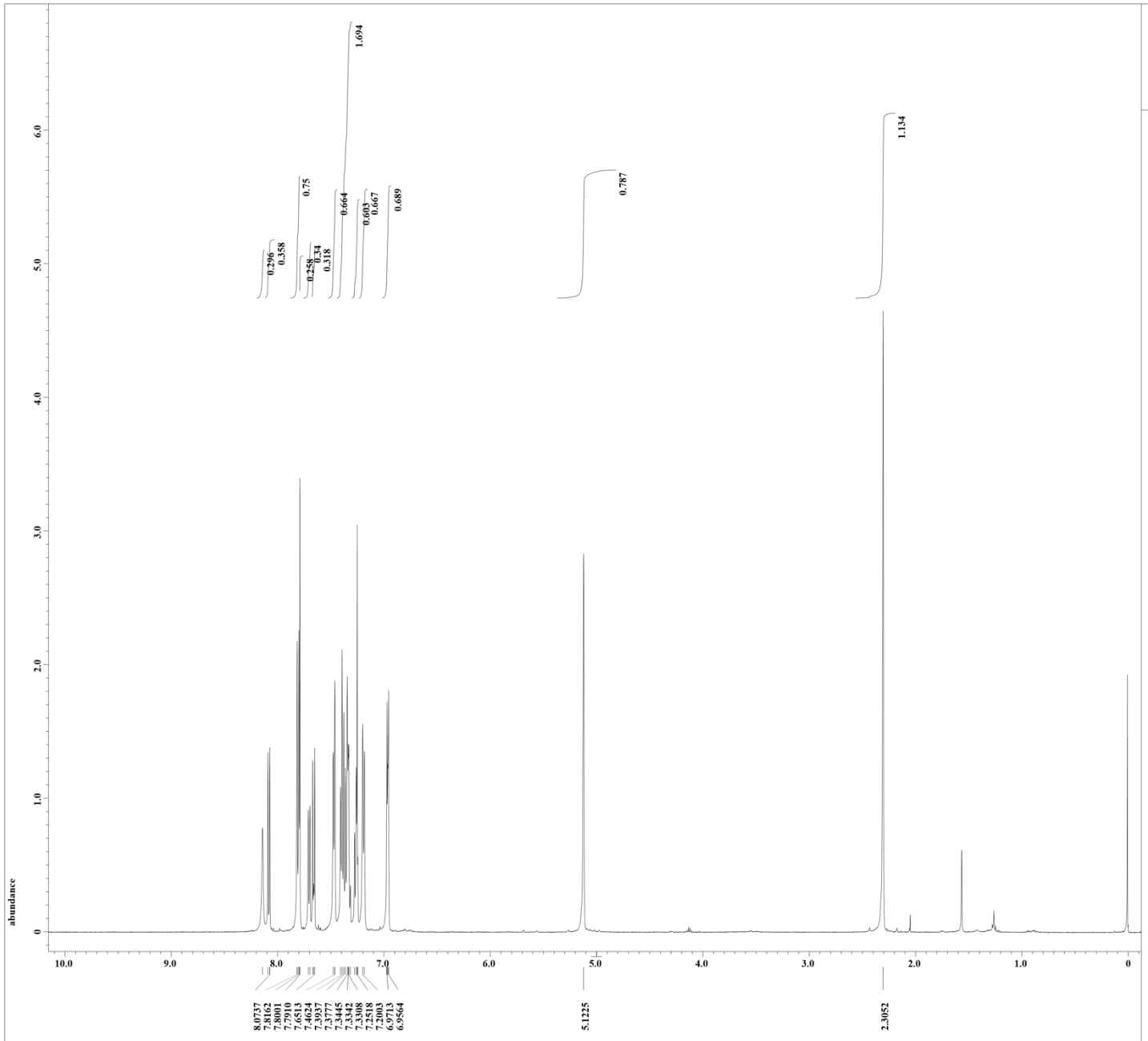
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 26249.0
Total_scans = 26249.0

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 23.6[dC]



6aj

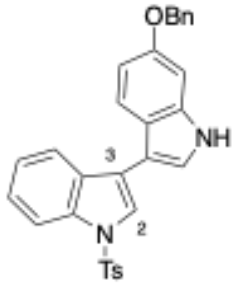


Filename = TA200106-10.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#495301
Solvent = CHLOROFORM-D
Creation_time = 6-JAN-2020 12:56:29
Revision_time = 1-FEB-2020 18:39:39
Current_time = 1-FEB-2020 18:40:06

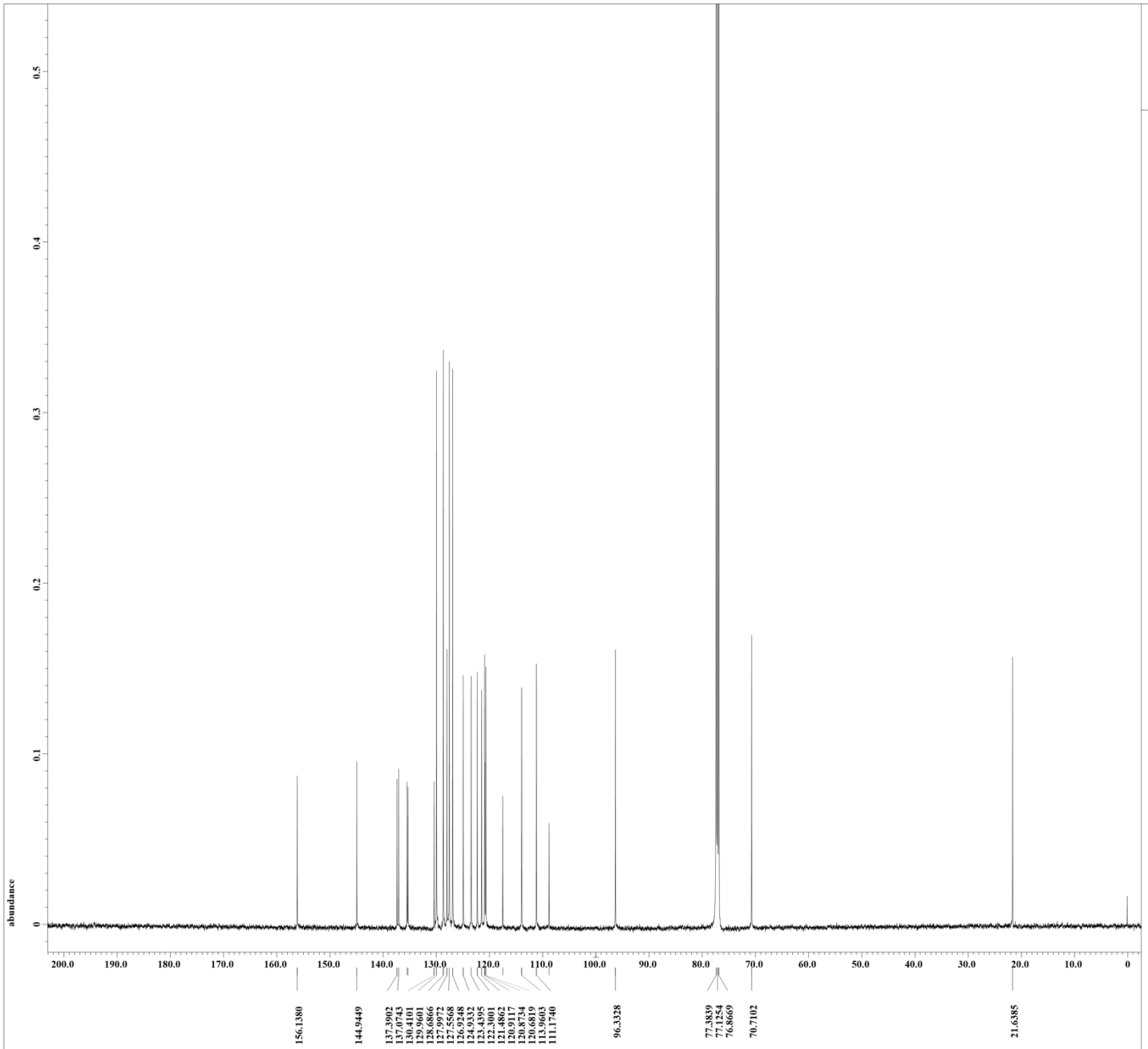
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 79.4[dC]



6ak

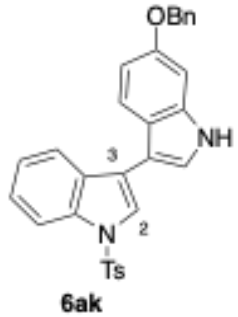


Filename = TA200106-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#565702
Solvent = CHLOROFORM-D
Creation_time = 7-JAN-2020 07:35:25
Revision_time = 1-FEB-2020 18:35:34
Current_time = 1-FEB-2020 18:36:11

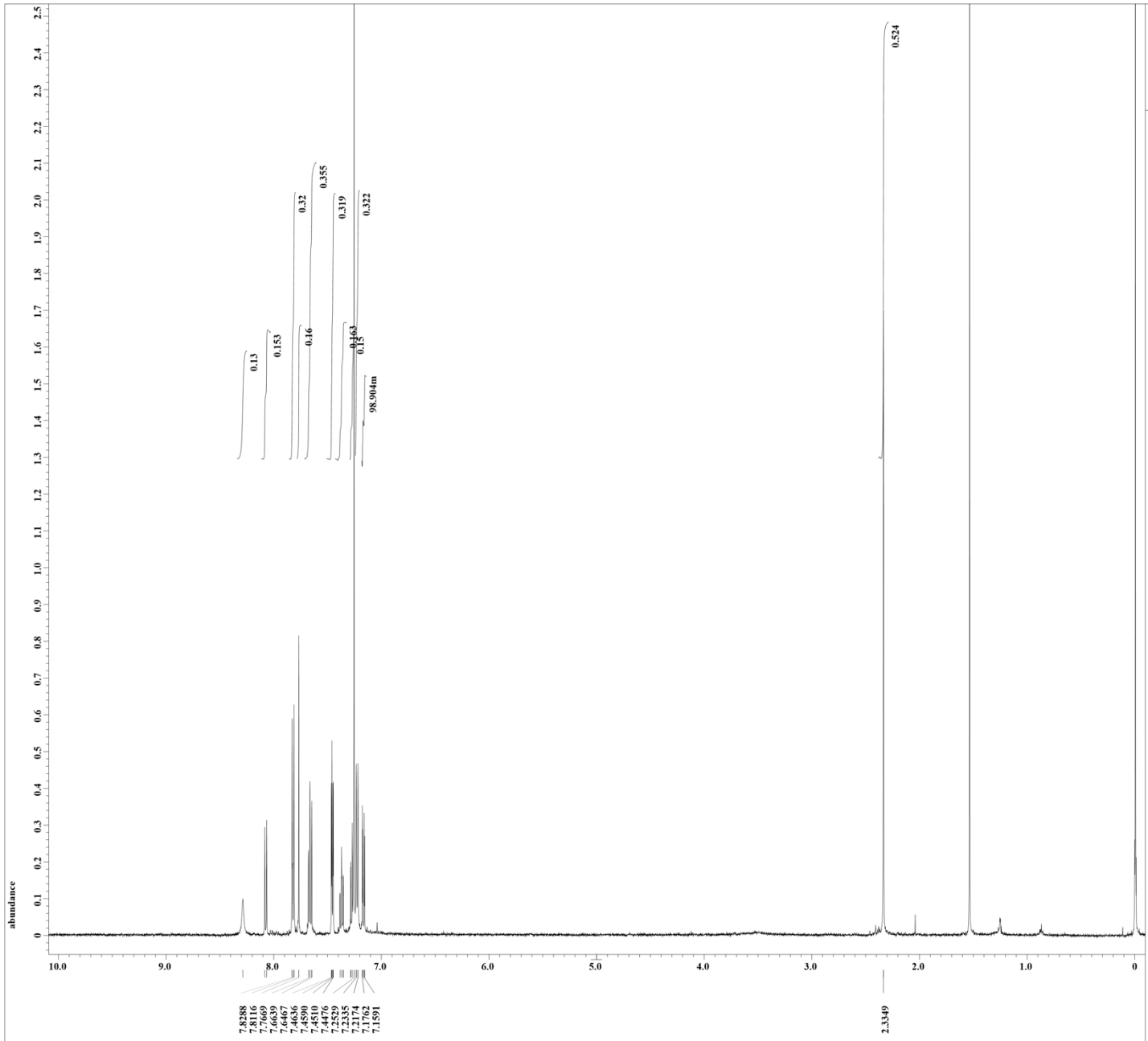
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 21195
Total_scans = 21195

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 101.3[dC]



6ak

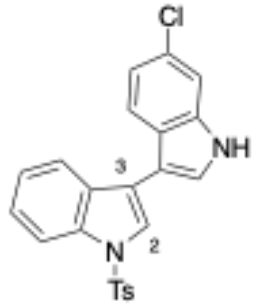


Filename = TA200108-9.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#632215
Solvent = CHLOROFORM-D
Creation_time = 8-JAN-2020 16:43:38
Revision_time = 1-FEB-2020 18:59:32
Current_time = 1-FEB-2020 19:09:53

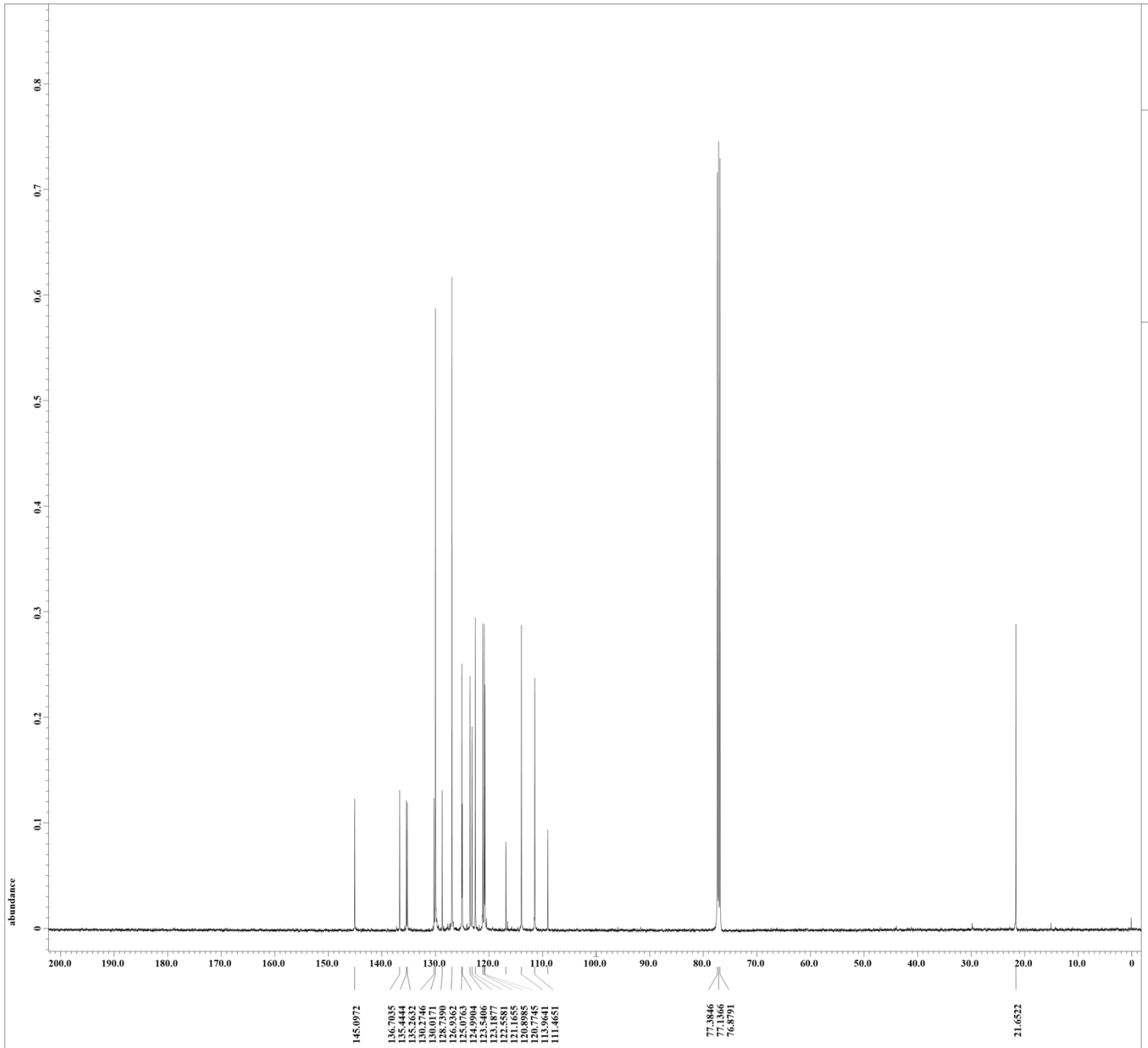
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 114.1[dC]

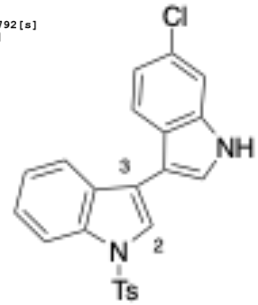


6al

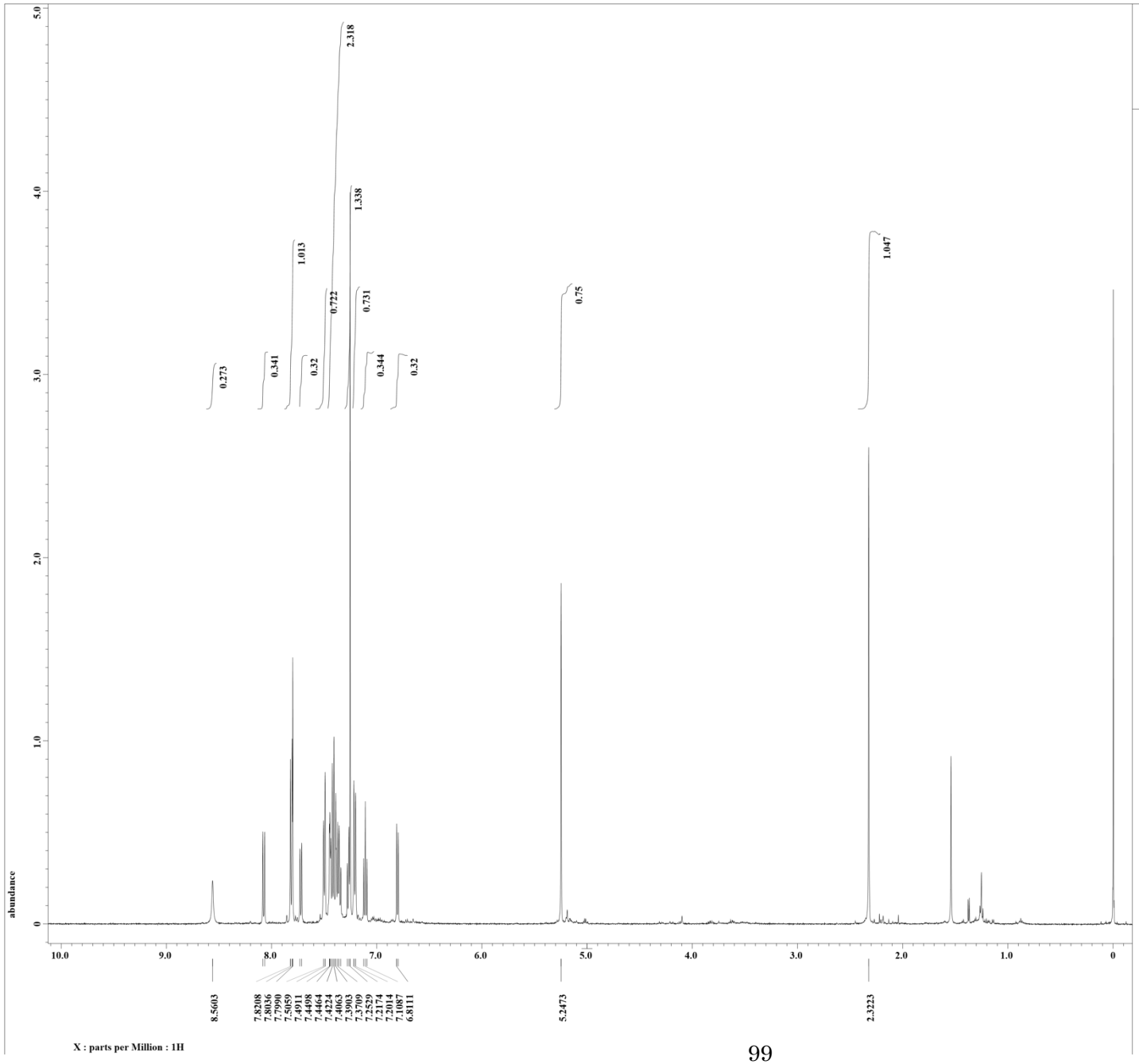


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0107-11.jdf
```

```
Filename          = TA2020-0107-17.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#676370
Solvent           = CHLOROFORM-D
Creation_time     = 26-MAR-2000 10:13:38
Revision_time     =  1-FEB-2020 18:44:43
Current_time      =  1-FEB-2020 18:45:22

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 17313
Total_scans       = 17313

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.6[dC]
```



6al

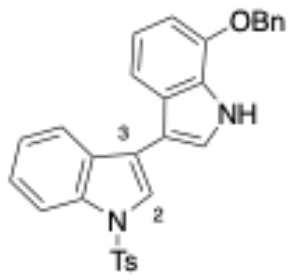


Filename = TA200101-15.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#485945
Solvent = CHLOROFORM-D
Creation_time = 1-JAN-2020 12:41:10
Revision_time = 1-FEB-2020 19:47:38
Current_time = 1-FEB-2020 19:48:09

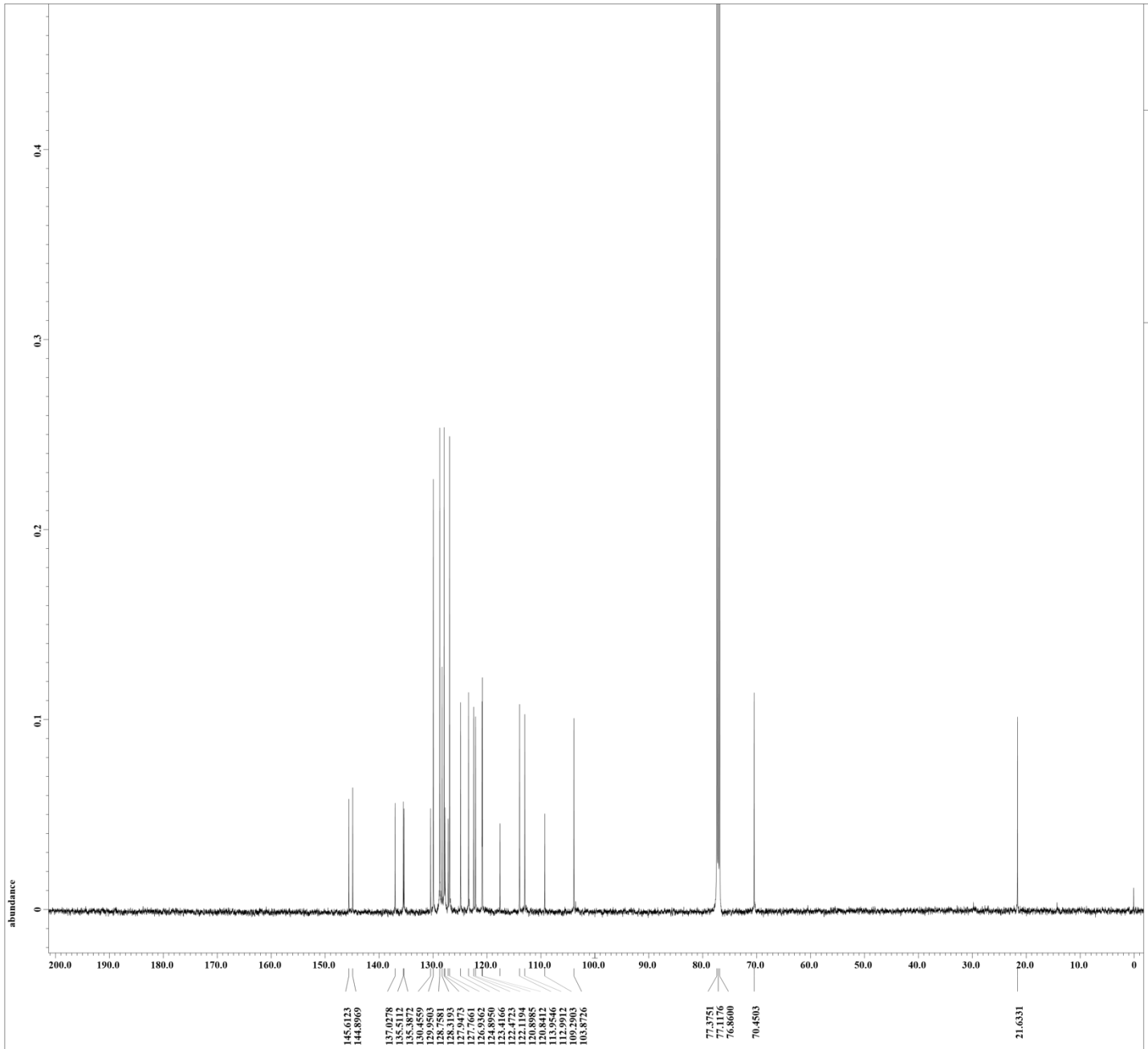
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.4[dC]

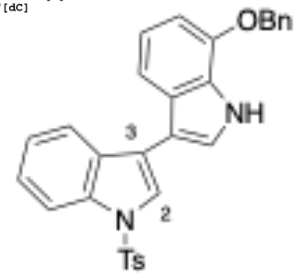


6am

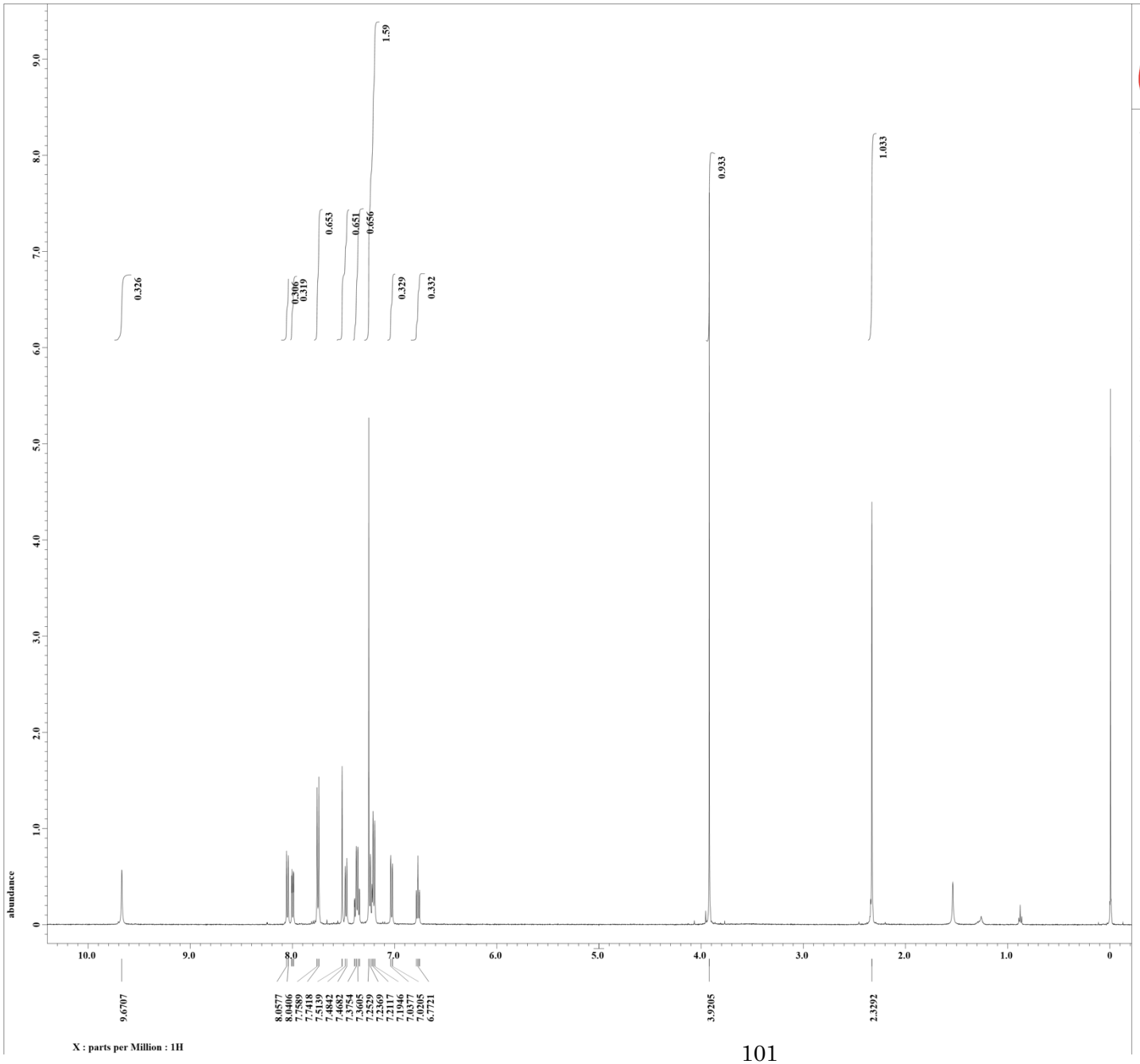


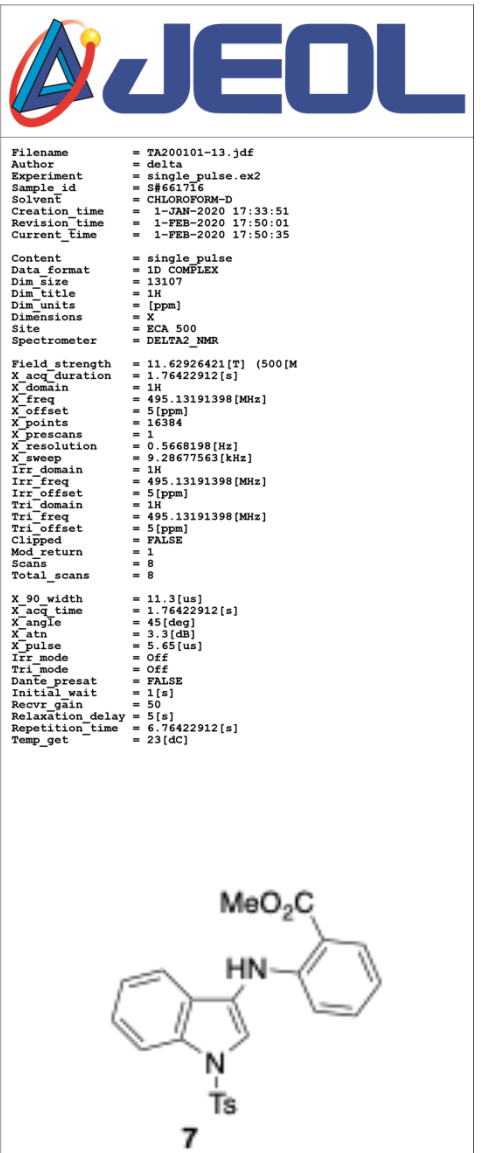

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0101-1.jdf
```

```
Filename          = TA2020-0101-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#486029
Solvent           = CHLOROFORM-D
Creation_time     = 18-MAR-2000 21:38:55
Revision_time     =  1-FEB-2020 19:14:57
Current_time      =  1-FEB-2020 19:15:44

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 8045
Total_scans       = 8045

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.7[dC]
```



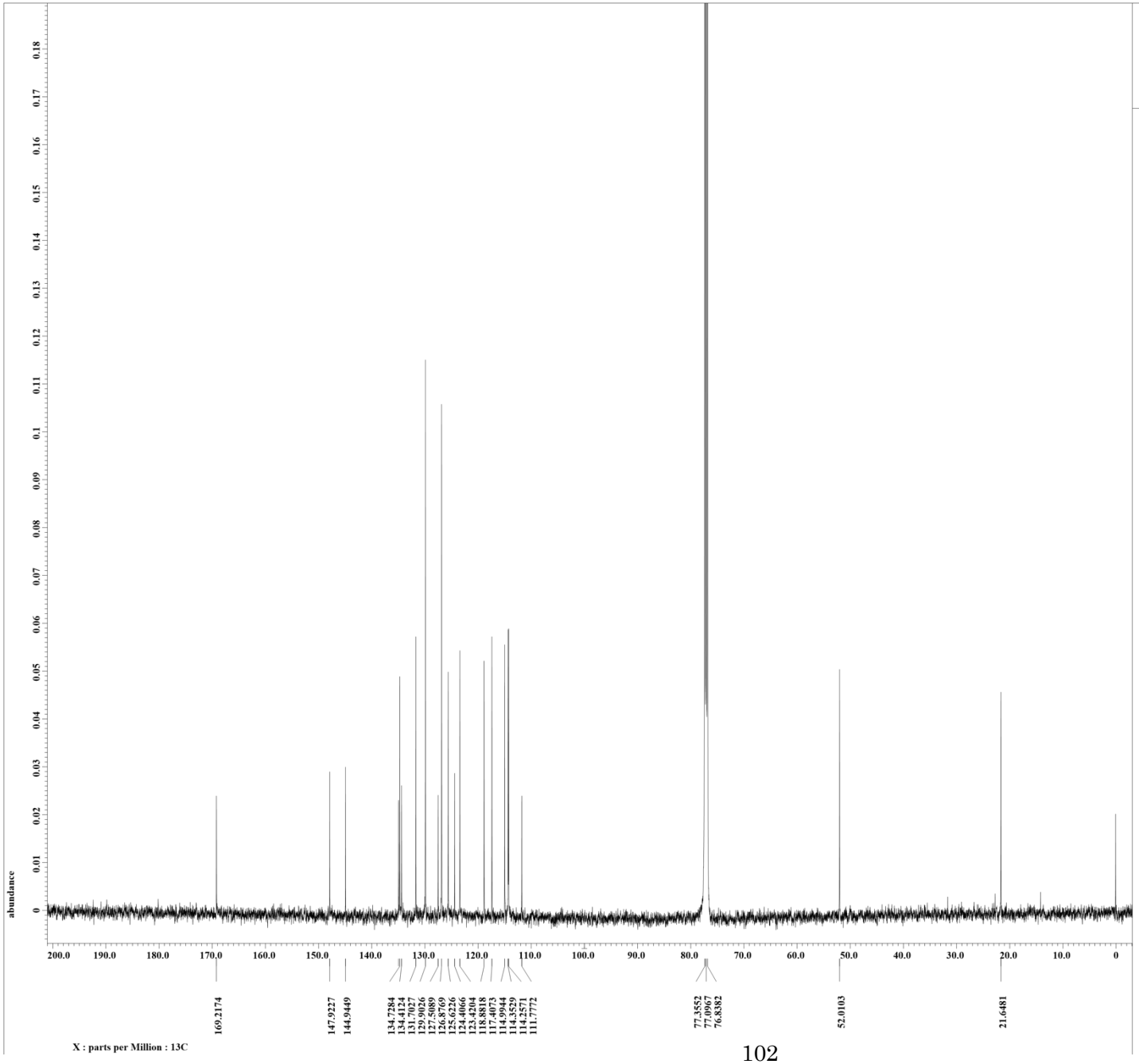
6am





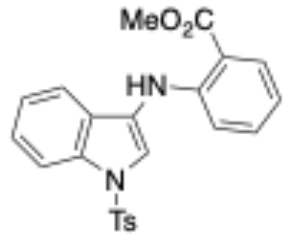
```
Filename          = TA200101-13.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#661716
Solvent           = CHLOROFORM-D
Creation_time     =  1-JAN-2020 17:33:51
Revision_time     =  1-FEB-2020 17:50:01
Current_time      =  1-FEB-2020 17:50:35

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 50
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23[dC]
```


7

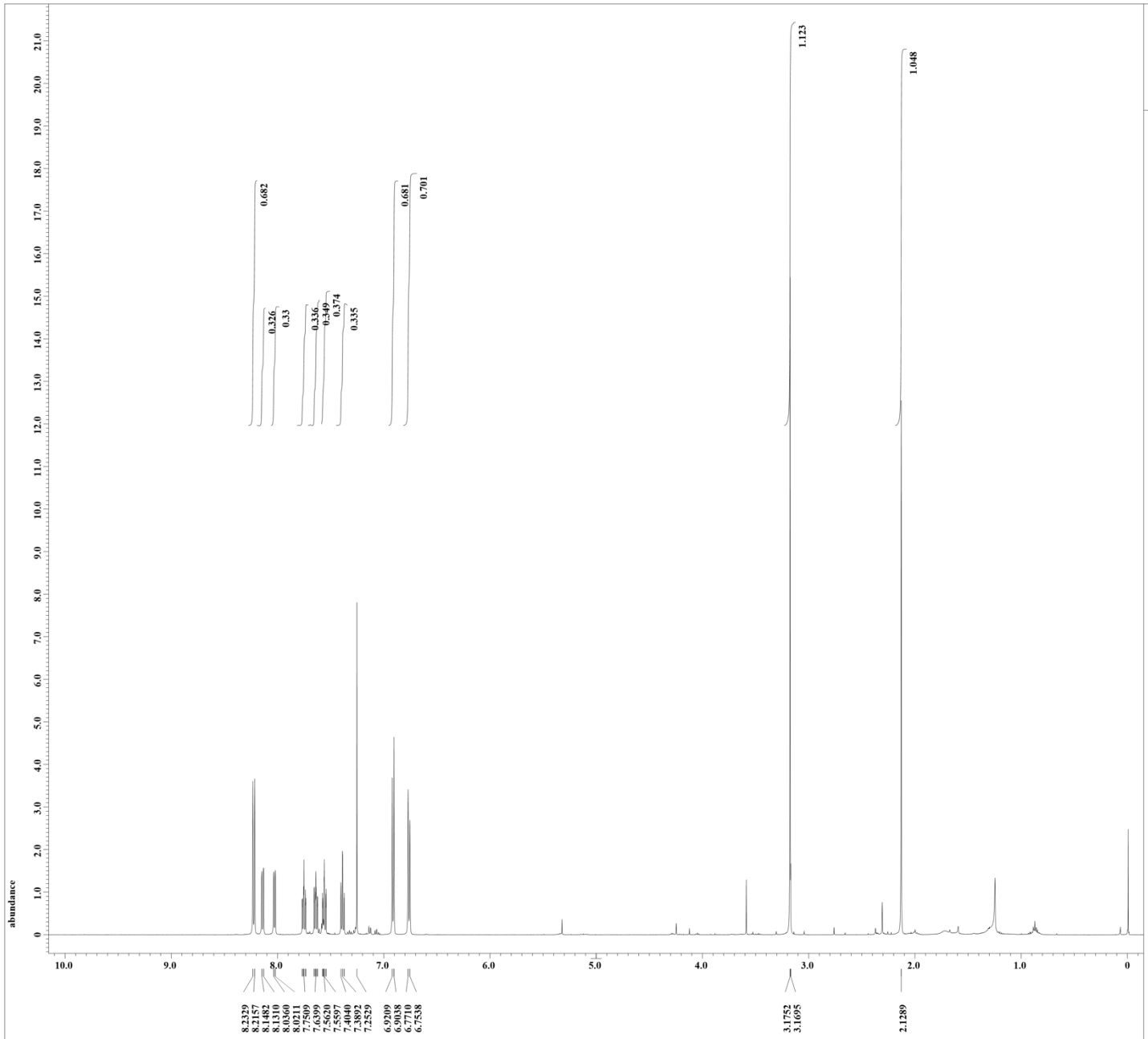


JEOL

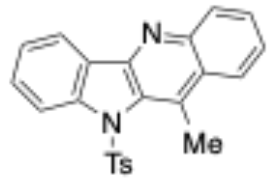
```
Filename          = TA200101-13.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#736458
Solvent           = CHLOROFORM-D
Creation_time     =  2-JAN-2020 10:42:35
Revision_time     =  1-FEB-2020 17:46:34
Current_time      =  1-FEB-2020 17:47:10

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 19119
Total_scans       = 19119

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 23.8[dC]
```



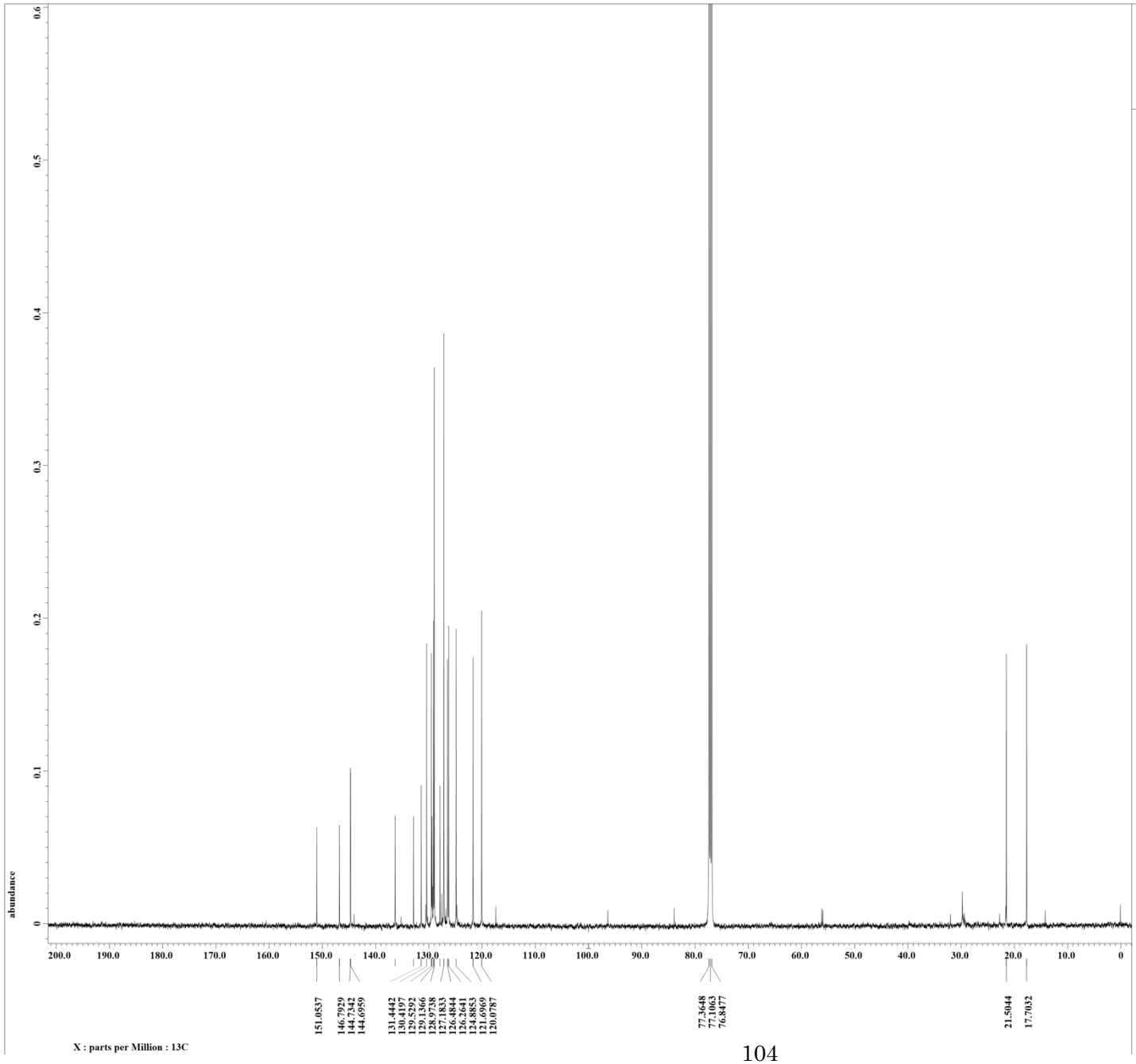
7




```
Filename          = TA200121-8.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#667909
Solvent           = CHLOROFORM-D
Creation_time     = 21-JAN-2020 17:41:41
Revision_time     =  1-FEB-2020 17:56:26
Current_time      =  1-FEB-2020 17:56:42

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.5[dC]
```



8

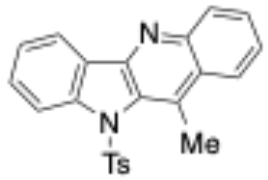


Filename = TA200121-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#669662
Solvent = CHLOROFORM-D
Creation_time = 22-JAN-2020 06:36:34
Revision_time = 1-FEB-2020 17:56:59
Current_time = 1-FEB-2020 17:57:39

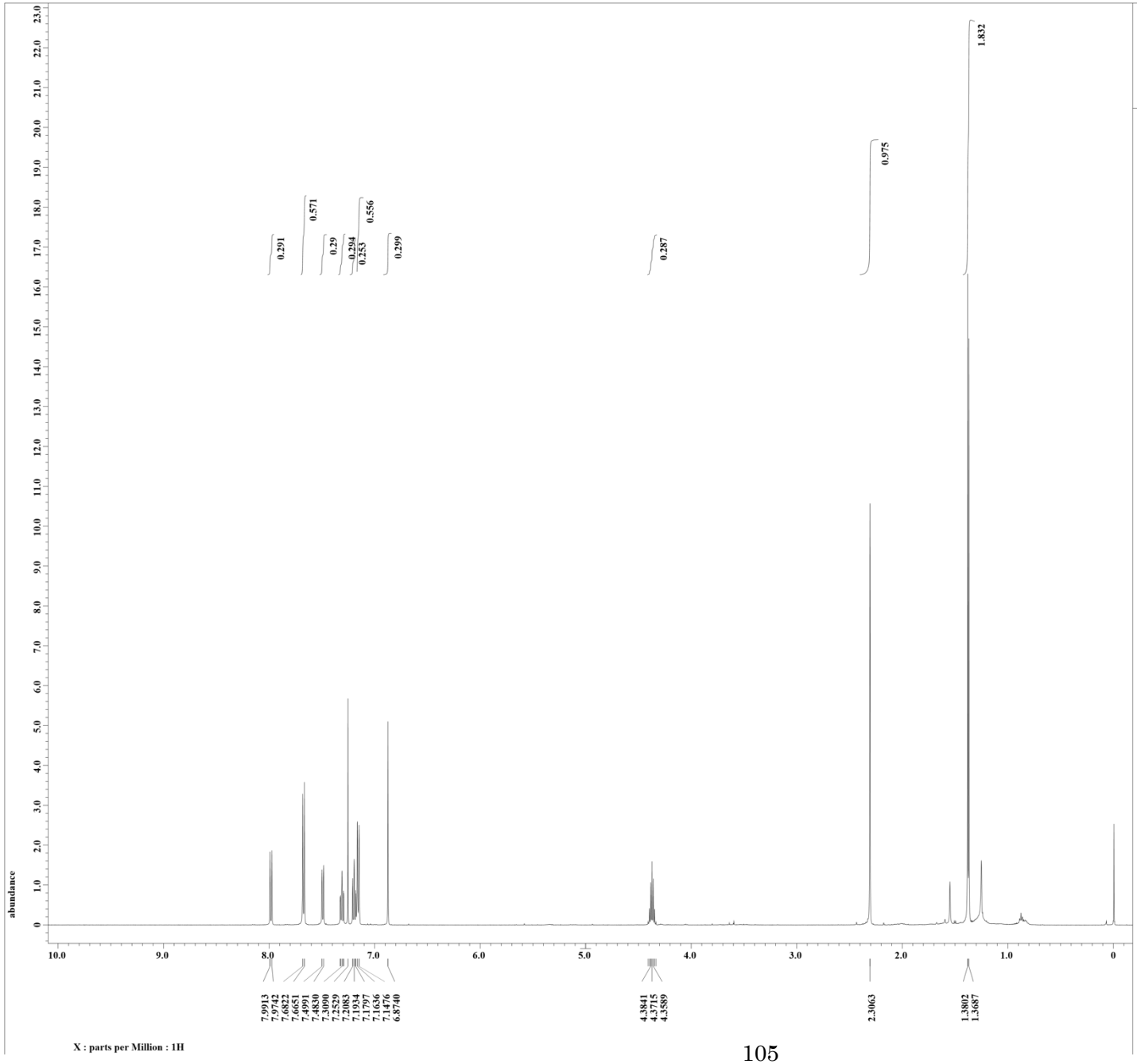
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16337
Total_scans = 16337

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.1[dC]

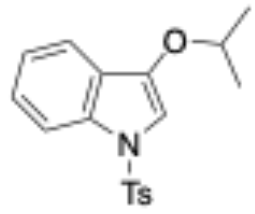


8



JEOL

```
Filename          = TA200122-10.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#325349
Solvent           = CHLOROFORM-D
Creation_time     = 22-JAN-2020 08:10:13
Revision_time     =  1-FEB-2020 17:42:25
Current_time      =  1-FEB-2020 17:42:55

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.6[dC]
```



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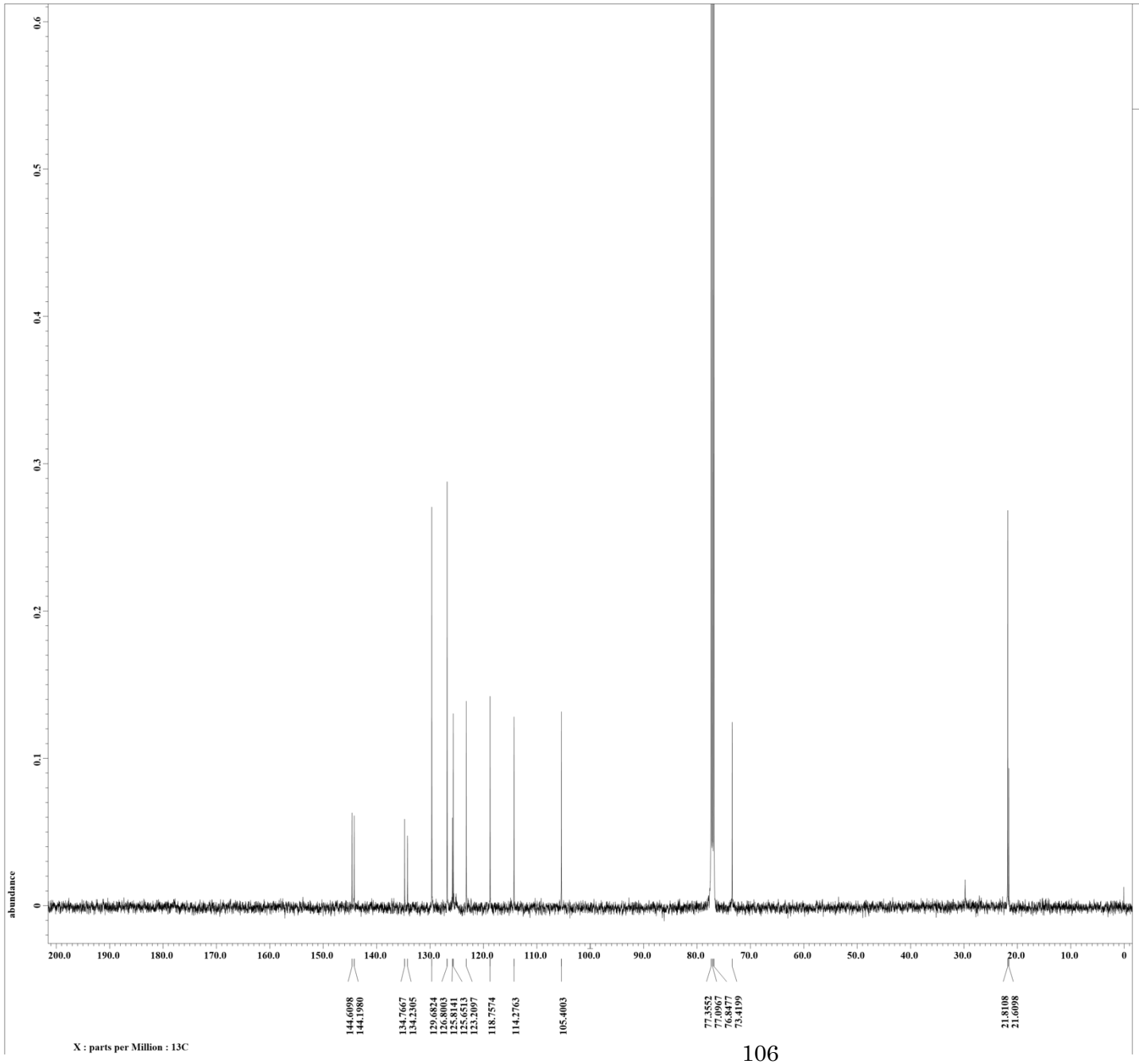
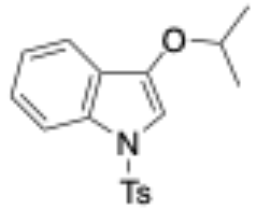


Filename = TA200122-8.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#326587
Solvent = CHLOROFORM-D
Creation_time = 22-JAN-2020 10:10:35
Revision_time = 1-FEB-2020 17:35:43
Current_time = 1-FEB-2020 17:36:50

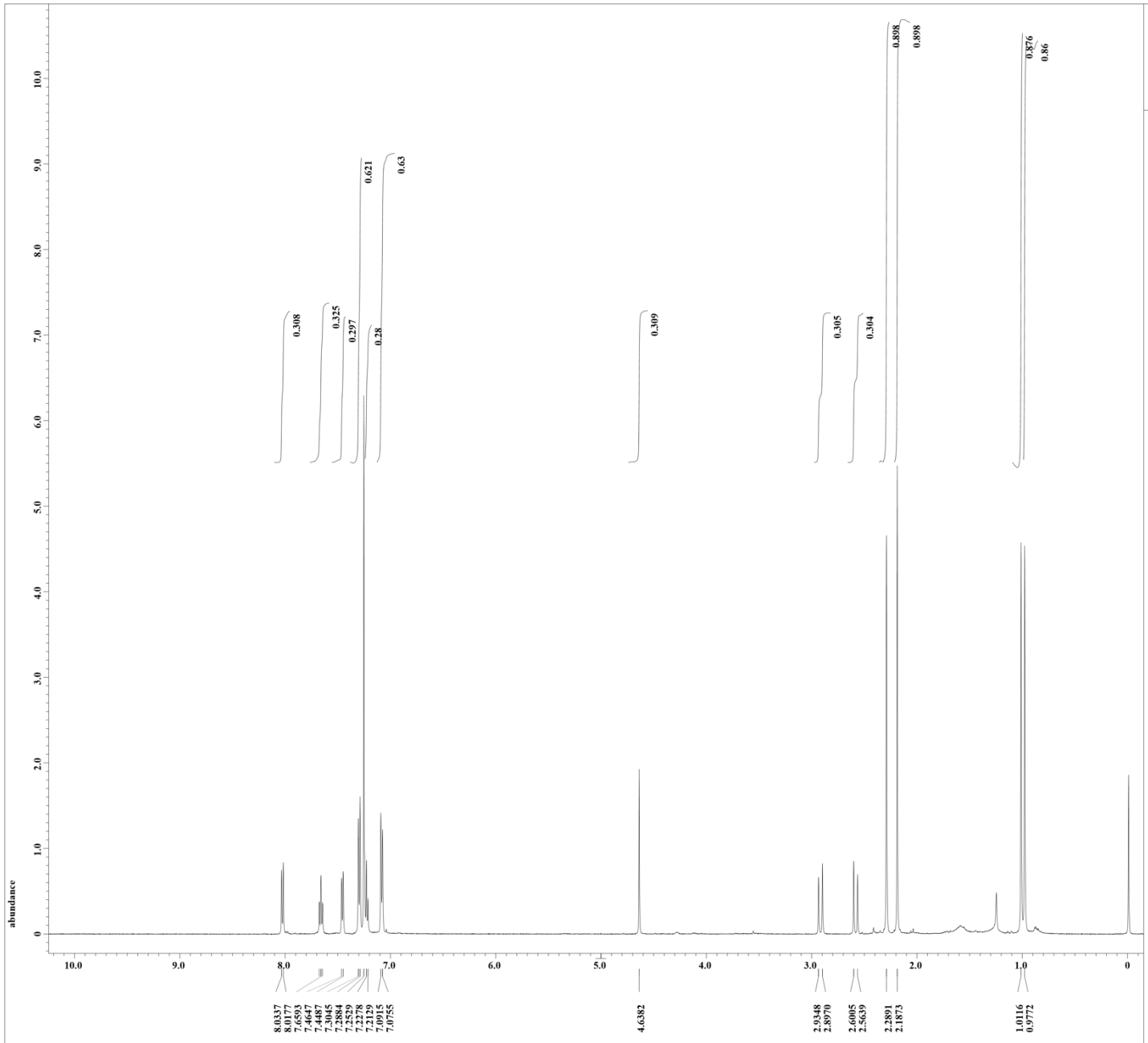
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 2513
Total_scans = 2513

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.4[dC]



9

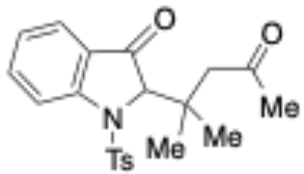


Filename = TA191126-12.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#395871
Solvent = CHLOROFORM-D
Creation_time = 26-NOV-2019 10:15:49
Revision_time = 1-FEB-2020 17:20:04
Current_time = 1-FEB-2020 17:20:37

Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.5668198[Hz]
X_sweep = 9.28677563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 11.3[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 5.65[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.4[dC]



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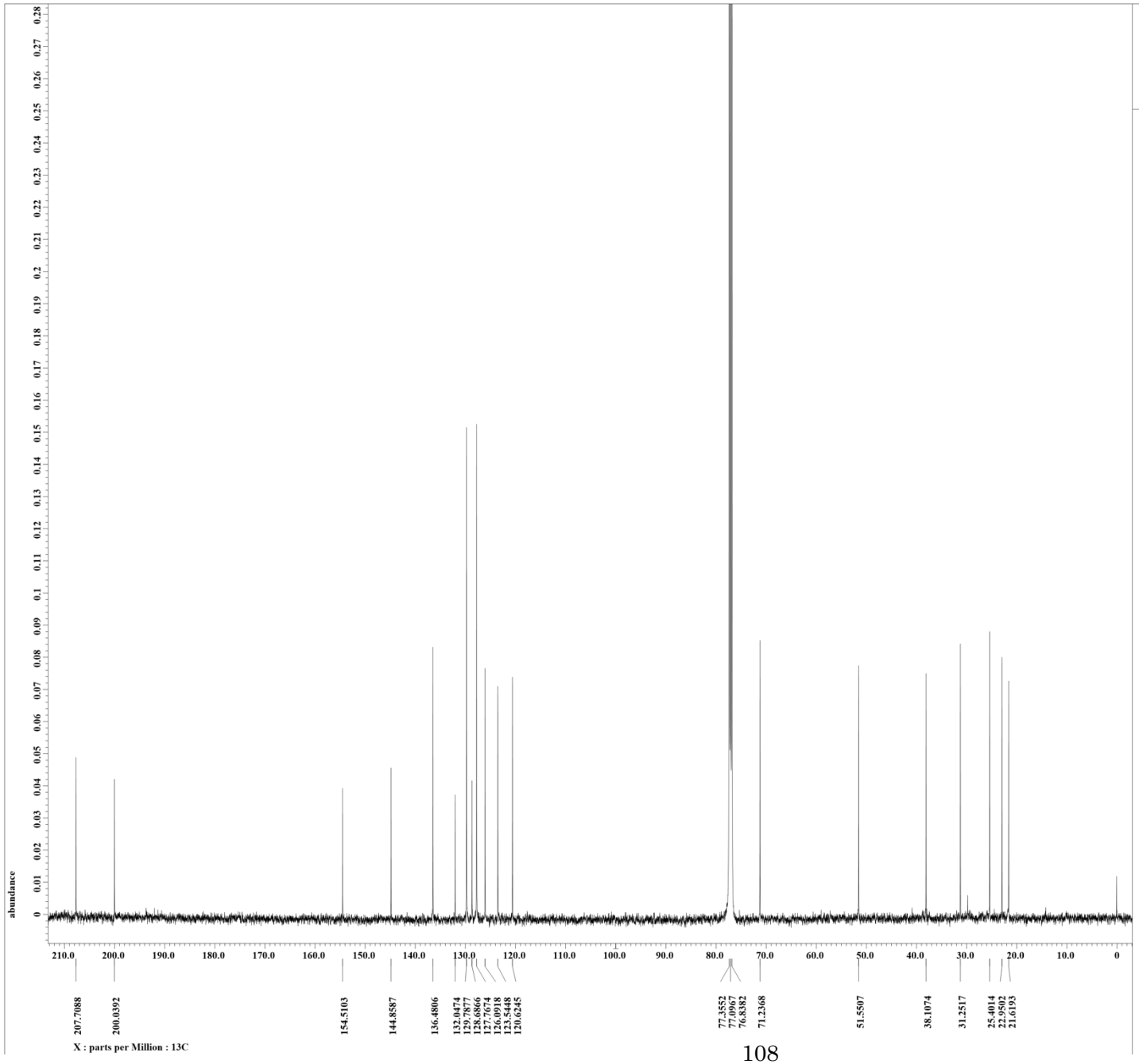
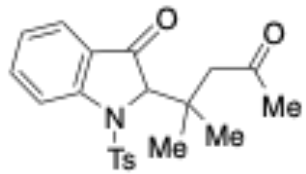
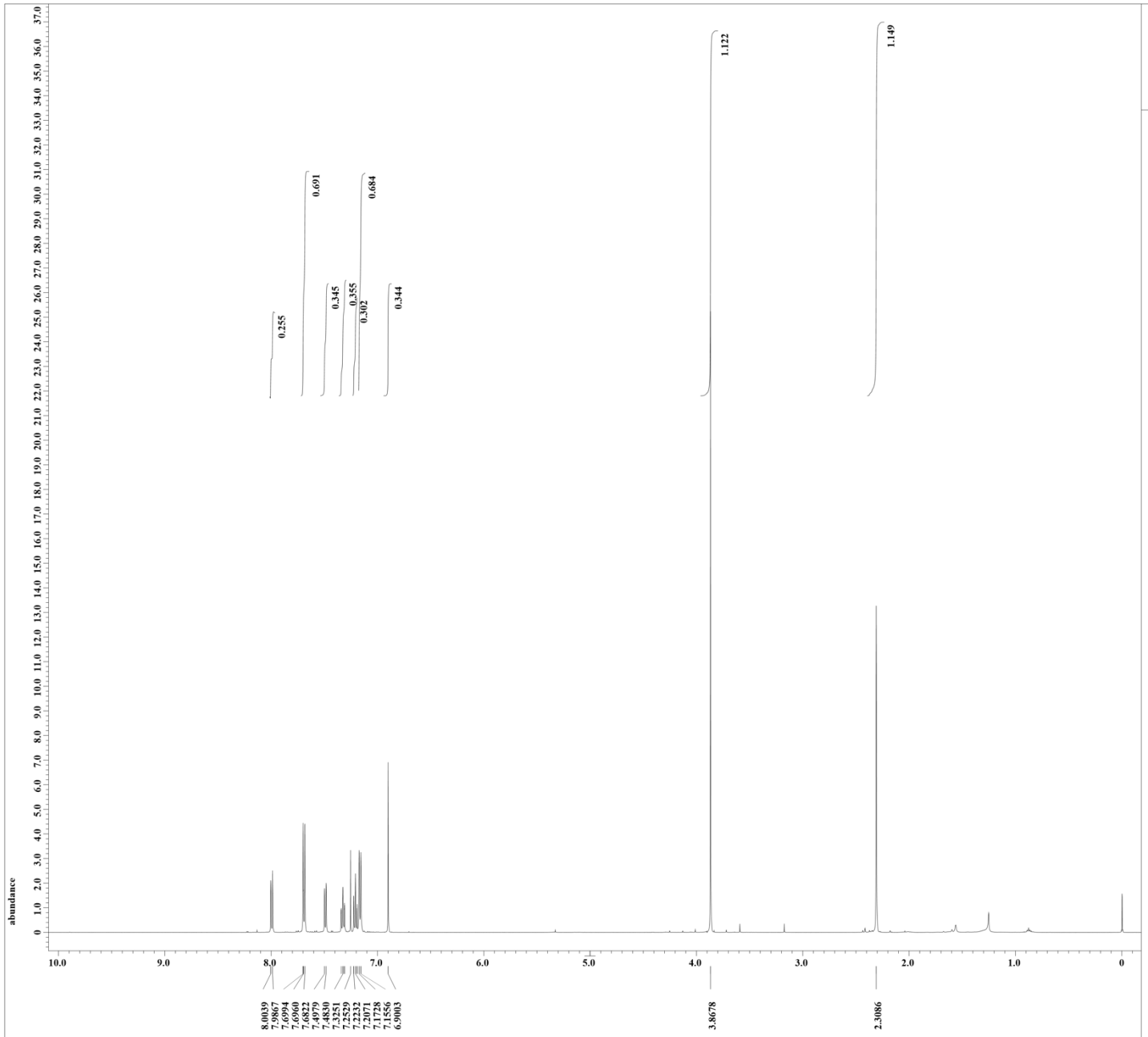


```
Filename          = TA191126-11.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#633866
Solvent           = CHLOROFORM-D
Creation_time     = 27-NOV-2019 07:20:44
Revision_time     =  1-FEB-2020 17:16:02
Current_time      =  1-FEB-2020 17:17:18

Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 0.8388608[s]
X_domain          = 13C
X_freq            = 124.5010059[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.1920929[Hz]
X_sweep           = 39.0625[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 18355
Total_scans       = 18355

X_90_width        = 10.1[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.36666667[us]
Irr_atn_dec       = 21.51[dB]
Irr_atn_noe       = 21.51[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 2.8388608[s]
Temp_get          = 24.5[dC]
```

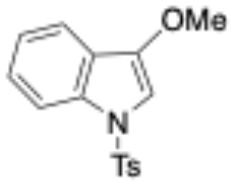


10



JEOL

```
Filename          = TA200114-12.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#562957
Solvent           = CHLOROFORM-D
Creation_time     = 14-JAN-2020 14:47:54
Revision_time     =  1-FEB-2020 18:11:13
Current_time      =  1-FEB-2020 18:11:33

Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_strength    = 11.62926421[T] (500[M
X_acq_duration    = 1.76422912[s]
X_domain          = 1H
X_freq            = 495.13191398[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.5668198[Hz]
X_sweep           = 9.28677563[kHz]
Irr_domain        = 1H
Irr_freq          = 495.13191398[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 495.13191398[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.3[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 5.65[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.4[dC]
```



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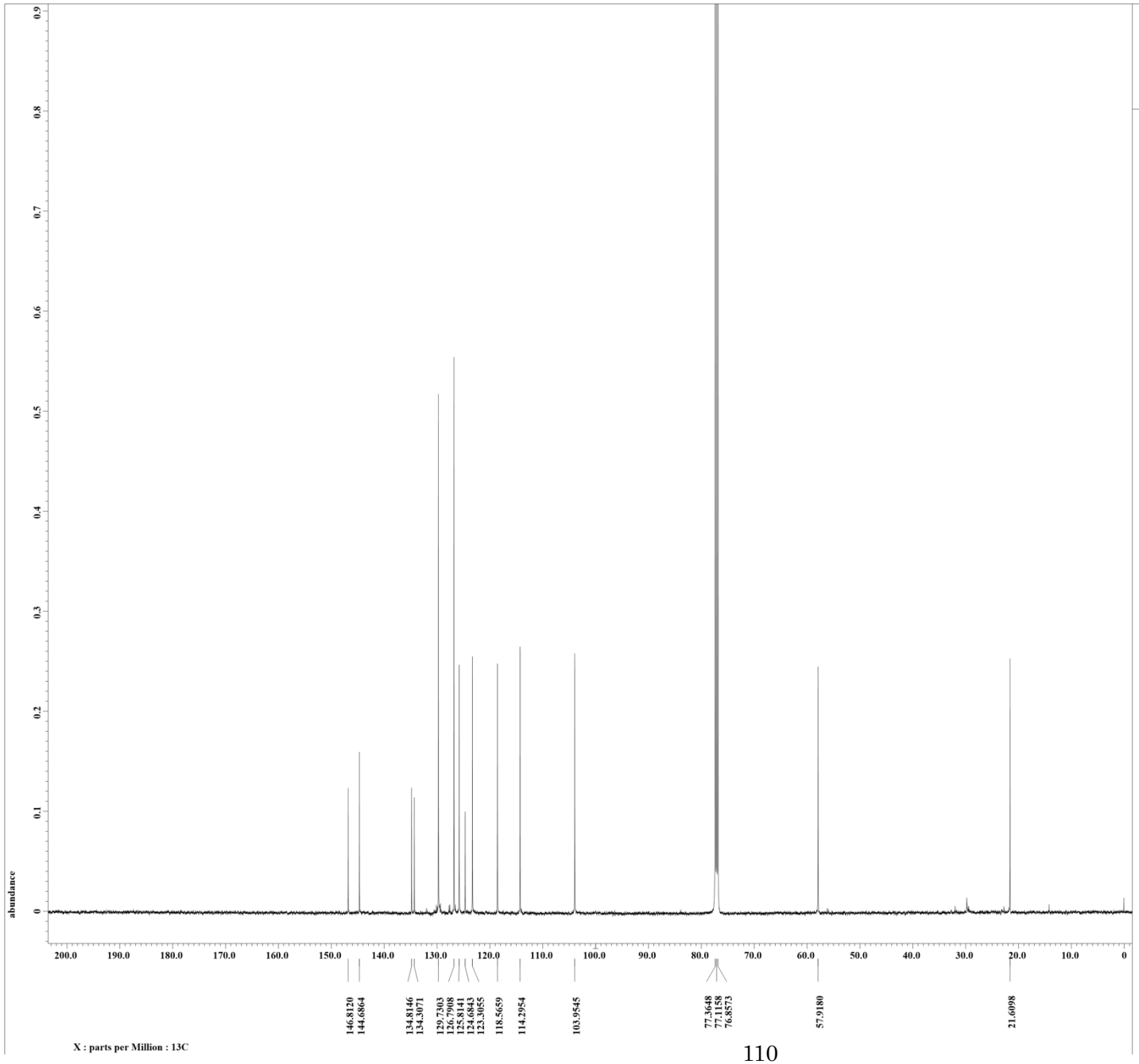
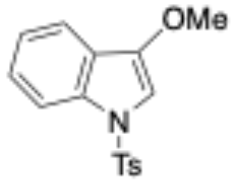


Filename = TA200114-12.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#626862
Solvent = CHLOROFORM-D
Creation_time = 15-JAN-2020 07:53:21
Revision_time = 1-FEB-2020 18:07:32
Current_time = 1-FEB-2020 18:08:09

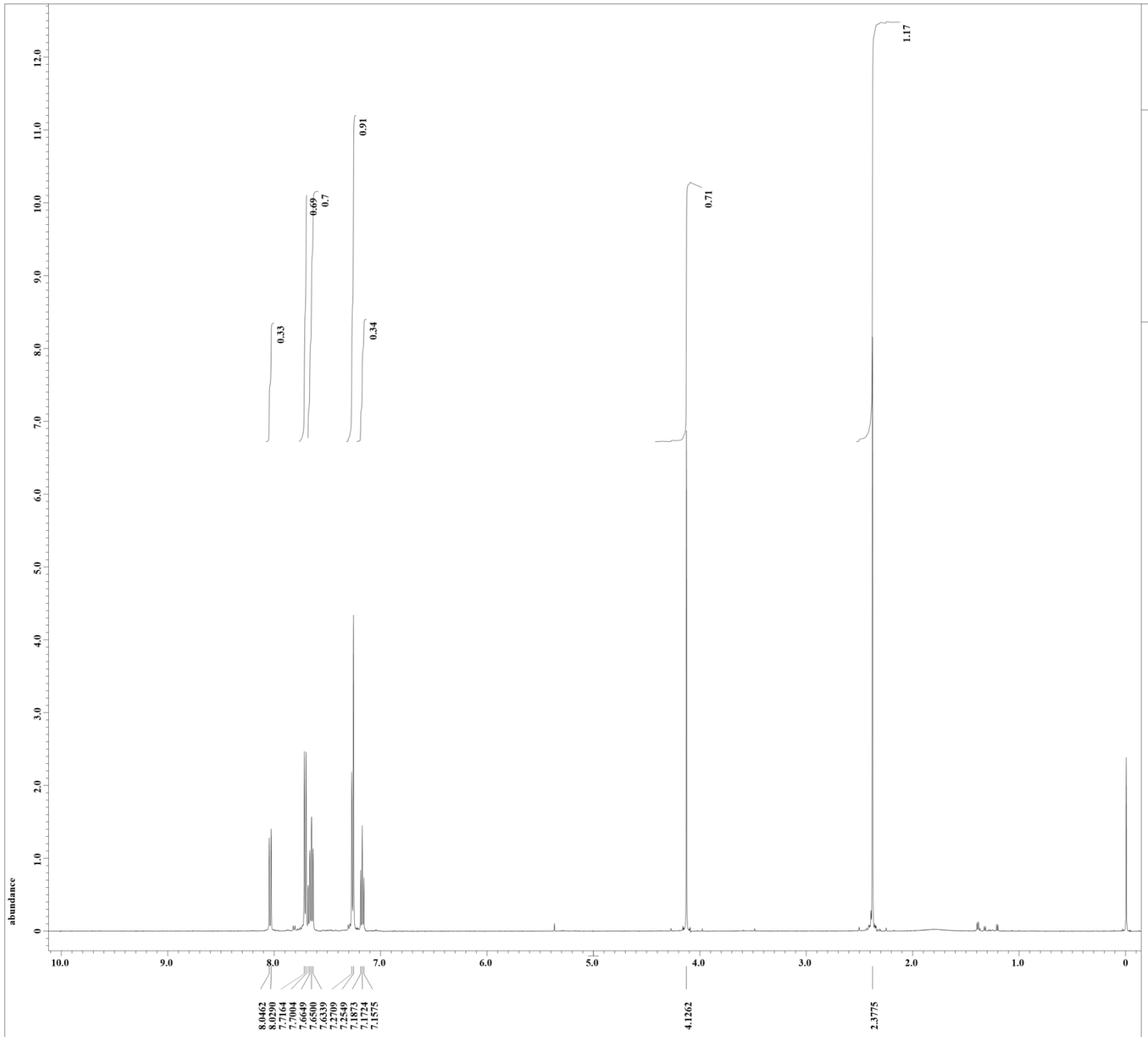
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 124.5010059[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 39.0625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 19444
Total_scans = 19444

X_90_width = 10.1[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.36666667[us]
Irr_atn_dec = 21.51[dB]
Irr_atn_noe = 21.51[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.1[dC]

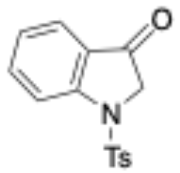


11




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 0.2[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0118-1.jdf
```

```
Filename          = TA2020-0118-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#589067
Solvent           = CHLOROFORM-D
Creation_time     =  4-APR-2000 18:10:42
Revision_time     =  1-FEB-2020 17:35:56
Current_time      =  1-FEB-2020 17:36:14

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
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[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6[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          = 24.1[dC]
```



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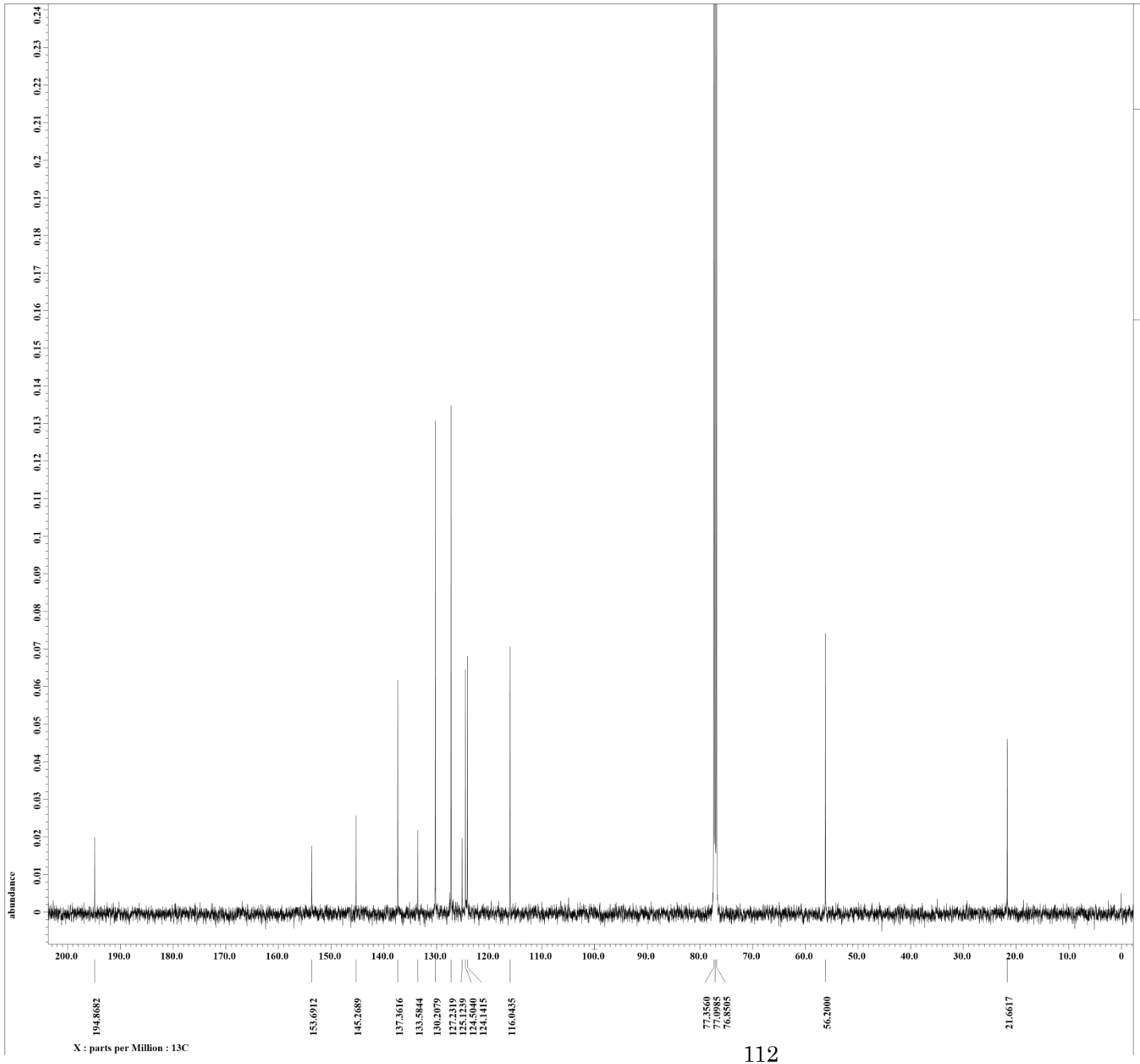
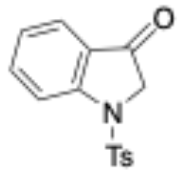


```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm

Derived from: TA2020-0118-3.jdf
```

```
Filename          = TA2020-0118-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#607389
Solvent           = CHLOROFORM-D
Creation_time     =  4-APR-2000 20:21:09
Revision_time     =  1-FEB-2020 17:32:36
Current_time      =  1-FEB-2020 17:33:53

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 1
Scans             = 2125
Total_scans       = 2125

X_90_width        = 12.8[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 4.26666667[us]
Irr_atn_dec       = 21.09[dB]
Irr_atn_noe       = 21.09[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 56
Relaxation_delay  = 2[s]
Repetition_time   = 2.83361792[s]
Temp_get          = 24.6[dC]
```



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