Dehydrative Mannich-type Reaction for the Synthesis of the Azepinobisindole Aklaloid Iheyamine A

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SUPPORTING INFORMATION

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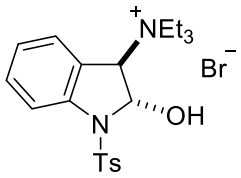
Experimental Section

1. General Experimental

Melting points were recorded with a Yanaco MP3 and are uncorrected. High-resolution MS spectra were recorded with a JEOL JMS-T100LP mass spectrometers. IR spectra were measured with a Shimadzu IRAffinity-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 undeuterated solvent as an internal reference ($CDCl_3$ ^{1}H-NMR δ 7.25, ^{13}C-NMR δ 77.1; DMSO ^{1}H-NMR δ 2.47, ^{13}C-NMR δ 40.0, CD_3OD ^{1}H-NMR δ 3.29, ^{13}C-NMR δ 47.7). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.
Flash column chromatography was performed on silica gel (Silica Gel 60N, Kanto Chemical Co., Ltd.).

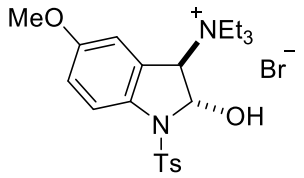
2. Experimental Procedure



2a

***trans*-2-Hydroxy-1-tosylindoline-3-ammonium bromide (2a)**

To a solution of 1-tosylindole (3.00 g, 11 mmol) and H_2O (1.98 mL, 110 mmol) in acetone (110 mL) was added NBS (2.17 g, 12 mmol). The mixture was stirred at room temperature until the complete disappearance of starting material as indicated by TLC. Et_3N (1.69 mL, 12 mmol) was added to the mixture and stirred further 1 h. The resulting precipitate was separated by filtration, washed with acetone, and dried *in vacuo* to give **2a** (3.89 g, 75% yield).
3.89 g, 75% yield. colorless prisms; mp 135—139 °C (MeOH); IR (KBr): 3163, 1601 cm^{-1}; H-NMR (500 MHz, DMSO-d_6): δ 8.02 (d, J = 8.0 Hz, 2H), 7.88 (d, J = 6.9 Hz, 1H), 7.55 (d, J = 8.1 Hz, 1H), 7.48 (t, J = 7.5 Hz, 1H), 7.40 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.0 Hz, 1H), 7.14 (t, J = 7.5 Hz, 1H), 6.35 (d, J = 7.5 Hz, 1H), 4.82 (s, 1H), 3.30—3.46 (m, 6H), 2.33 (s, 3H), 1.01 (t, J = 7.5 Hz, 9H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 145.4, 142.8, 136.1, 132.9, 130.4, 130.4, 128.2, 124.0, 120.4, 113.8, 84.8, 75.4, 53.3, 21.5, 8.8; HRMS (ESI): calcd for $C_{21}H_{29}N_2O_3S$ $[M]^+$ 389.1899, found 389.1926.

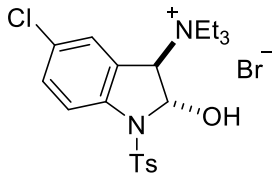


2b

***trans*-2-Hydroxy-5-methoxy1-tosylindoline-3-ammonium bromide (2b)**

To a solution of 5-methoxy-1-tosylindole (2.41 g, 8 mmol) and H_2O (1.44 mL, 80 mmol) in acetone (80 mL) was added NBS (1.56 g, 8.8 mmol). The mixture was stirred at room temperature until the complete disappearance of starting material as indicated by TLC. Et_3N (1.22 mL, 8.8 mmol) was added to the mixture and stirred further 1 h. After addition of Et_2O (80 mL), the resulting precipitate was separated by filtration, washed with acetone (40 mL), and dried *in vacuo* to give **2b** (2.82 g, 71% yield).

2.82 g, 71% yield. white powder; mp 163—167 °C; IR (KBr): 3071, 1595 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 7.99 (d, J = 8.6 Hz, 2H), 7.80 (d, J = 6.9 Hz, 1H), 7.39 (d, J = 8.6 Hz, 2H), 7.30 (d, J = 8.6 Hz, 1H), 7.06—7.09 (m, 2H), 6.30 (d, J = 7.5 Hz, 1H), 4.77 (s, 1H), 3.72 (s, 3H), 3.30—3.46 (m, 6H), 2.33 (s, 3H), 1.00 (t, J = 7.4 Hz, 9H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 156.0, 145.2, 136.4, 136.2, 130.4, 128.1, 121.8, 118.2, 116.1, 114.7, 85.0, 75.5, 56.3, 53.3, 21.5, 8.8; HRMS (ESI): calcd for $C_{22}H_{31}N_2O_4S$ $[M]^+$ 419.2005, found 419.2004.



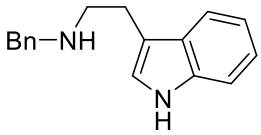
2c

***trans*-2-Hydroxy-5-methoxy1-tosylindoline-3-ammonium bromide (2b)**

To a solution of 5-chloro-1-tosylindole (3.06 g, 10 mmol) and H_2O (1.8 mL, 100 mmol) in acetone (100 mL) was added NBS (1.96 g, 11 mmol). The mixture was stirred at room temperature until the complete disappearance of starting material as indicated by TLC. Et_3N (1.52 mL, 11 mmol) was added to the mixture and stirred further 1 h. After addition of Et_2O (100 mL), the resulting precipitate was separated by filtration, washed with acetone (50 mL), and dried *in vacuo* to give **2c** (3.37 g, 67% yield).

3.37 g, 67% yield. white powder; mp 139—142 °C (decomp.); IR (KBr): 3119, 1597 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 8.01 (d, J = 8.6 Hz, 2H), 7.95 (br d, J = 5.7 Hz, 1H), 7.60 (d, J = 2.3 Hz, 1H), 7.54 (dd, J = 8.6, 2.3 Hz, 1H), 7.41 (d, J = 8.6 Hz, 2H), 7.37 (d, J = 8.6 Hz, 1H), 6.37 (br d, J = 5.2 Hz, 1H), 4.84 (s, 1H), 3.32—3.47 (m, 6H), 2.34 (s, 3H), 1.02 (t, J = 7.5 Hz, 9H); ^{13}C-NMR (125

MHz, DMSO-d_6): δ 145.6, 141.8, 135.8, 132.8, 130.5, 130.3, 128.2, 127.9, 122.6, 115.1, 85.2, 74.9, 53.4, 21.6, 8.9; HRMS (ESI): calcd for $C_{21}H_{28}ClN_2O_3S$ $[M]^+$ 423.1509 and 425.1480, found 423.1515 and 425.1505.

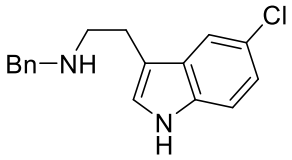


3a

***N*-Benzyltryptamine (3a)**

A mixture of tryptamine (4.80 g, 30 mmol) and benzaldehyde (3.35 mL, 33 mmol) in MeOH (150 mL) was stirred for 3 h at room temperature. To the resultant mixture was added $NaBH_4$ (1.7 g, 45 mmol) and stirred further 1 h. After concentrated *in vacuo* and addition of H_2O (150 mL), the whole was extracted with AcOEt (3 x 100 mL), washed with brine (100 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$, $CHCl_3$:MeOH:28% NH_3 aq. = 46:5:0.5) to give **3b** (6.82 g, 91% yield).

6.82 g, 91% yield. pale brown oil; IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.17 (br s, 1H), 7.63 (d, J = 8.0 Hz, 1H), 7.19—7.38 (m, 7H), 7.13 (t, J = 8.0 Hz, 1H), 6.99 (d, J = 2.3 Hz, 1H), 3.83 (s, 2H), 3.02 (s, 4H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 140.4, 136.5, 128.5, 128.2, 127.6, 127.0, 122.1, 122.1, 119.3, 119.0, 114.0, 111.3, 54.0, 49.5, 25.9; HRMS (ESI): calcd for $C_{17}H_{19}N_2$ $[M + H]^+$ 251.1548, found 251.1553



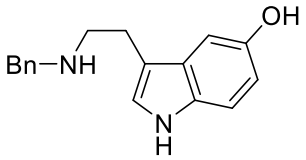
3b

***N*-Benzyl-5-chlorotryptamine (3b)**

A mixture of 5-chlorotryptamine hydrochloride (6.93 g, 30 mmol), benzaldehyde (3.35 mL, 33 mmol) and Et_3N (4.55 mL, 33 mmol) in MeOH (150 mL) was stirred at room temperature for 3 hours. To the resultant mixture was added $NaBH_4$ (1.70 g, 45 mmol) and stirred further 1 hour. After concentrated *in vacuo* and addition of H_2O (150 mL), the whole was extracted with AcOEt (3 x 100 mL), washed with brine (100 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$ and $CHCl_3$:MeOH:28% NH_3 aq. = 46:5:0.5) to give **3b** (6.90 g, 81% yield).

6.90 g, 81% yield. colorless solid; mp 93—94 °C; IR ($CHCl_3$): 3478 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.54 (br s, 1H), 7.54 (d, J = 1.7 Hz, 1H), 7.21—7.30 (m, 6H), 7.11 (dd, J = 8.6, 2.3 Hz,

1H), 7.01 (d, J = 2.3 Hz, 1H), 4.76 (br s, 1H), 3.79 (s, 2H), 2.91—2.96 (m, 4H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 140.2, 134.9, 128.6, 128.5, 128.2, 127.0, 125.0, 123.6, 122.2, 118.5, 113.6, 112.3, 53.9, 49.3, 25.7; HRMS (ESI): calcd for $C_{17}H_{18}ClN_2$ $[M + H]^+$ 285.1159 and 287.1129, found 285.1164 and 287.1142.



3c

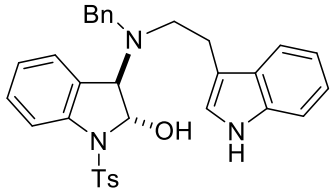
***N*-Benzylserotonin (3c)**

To a solution of serotonin hydrochloride (2.20 g, 10 mmol) and benzaldehyde (3.10 mL, 31 mmol) in MeOH (100 mL) was added 95% $NaBH_3CN$ (1.96 g, 30 mmol) and the mixture was stirred at room temperature for 30 minutes. After addition of H_2O (200 ml), the whole was made acidic (pH = 3) with 7% HCl and washed with $CHCl_3$ (100 mL). The water layer was made alkaline (pH = 9) with 8% NaOH and extracted with AcOEt (3 x 100 mL), washed with brine (100 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$:MeOH:28% NH_3 aq. = 46:3:0.3 and 46:5:0.5) to give **3c** (1.45 g, 53% yield).

1.45 g, 53% yield. colorless solid; mp 54—58 °C IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.07 (br s, 1H), 7.19—7.27 (m, 5H), 7.11 (d, J = 8.6 Hz, 1H), 6.86 (s, 1H), 6.84 (d, J = 2.3 Hz, 1H), 6.74 (dd, J = 8.6, 2.3 Hz, 1H), 4.70 (br s, 2H), 3.78 (s, 2H), 2.87—2.95 (m, 4H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 150.3, 139.3, 131.5, 128.6, 128.5, 128.2, 127.3, 123.1, 112.8, 112.7, 112.0, 103.7, 53.6, 48.9, 25.3; HRMS (ESI): calcd for $C_{17}H_{18}N_2ONa$ $[M + Na]^+$ 289.1317, found 289.1329.

General Procedure for the Reaction of 2a-c with Tryptamines 3a-c

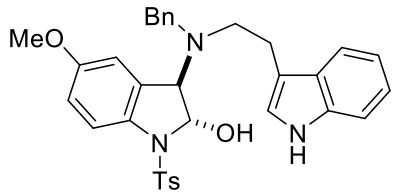
A mixture of **2** (1.0 mmol), **3** (1.1 mmol) and Et_3N (0.7 mL, 5.0 mmol) in AcOEt (20 mL) was heated under reflux with stirring for 2.5 hours. After addition of H_2O, the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt:hexane = 1:5—1:2) to give **5a**—**5i**.



5a

***trans*-*N*-Benzyl-*N*-(2-hydroxy-1-tosylindolin-3-yl)tryptamine (5a)**

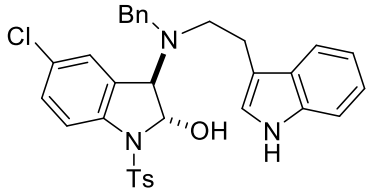
514 g, 96% yield. white powder; mp 190—193 °C (MeOH); IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 10.66 (br s, 1H), 7.69 (d, J = 8.6 Hz, 2H), 7.41 (d, J = 8.0 Hz, 1H), 7.21—7.30 (m, 5H), 7.14—7.17 (m, 3H), 6.93—7.06 (m, 6H), 6.89 (d, J = 2.3 Hz, 1H), 6.79 (t, J = 7.2 Hz, 1H), 5.70 (dd, J = 8.0, 1.7 Hz, 1H), 4.19 (s, 1H), 3.10 (d, J = 14.3 Hz, 1H), 3.05 (d, J = 14.3 Hz, 1H), 2.76—2.82 (m, 1H), 2.54—2.60 (m, 1H), 2.40—2.45 (m, 1H), 2.23—2.30 (m, 1H), 2.10 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.6, 141.5, 140.1, 136.7, 136.0, 130.6, 130.2, 129.8, 128.6, 128.6, 127.6, 127.4, 127.3, 126.5, 124.2, 123.0, 121.3, 118.7, 118.5, 114.0, 112.4, 111.8, 86.6, 70.2, 53.7, 51.1, 24.9, 21.3; HRMS (ESI): calcd for $C_{32}H_{31}N_3O_3SNa$ $[M + Na]^+$ 560.1984, found 560.1980.



5b

***trans*-*N*-Benzyl-*N*-(2-hydroxy-1-tosyl-5-methoxyindolin-3-yl)tryptamine (5b)**

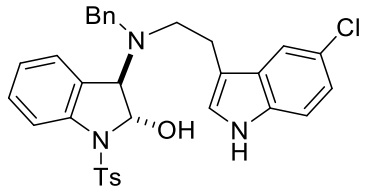
510 mg, 90% yield. white powder; mp 135—136 °C (MeOH); IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.69 (br d, J = 0.9 Hz, 1H), 7.63 (d, J = 8.1 Hz, 2H), 7.38 (d, J = 8.6 Hz, 1H), 7.38 (d, J = 8.6 Hz, 1H), 7.21 (t, J = 7.5 Hz, 2H), 7.14 (t, J = 7.5 Hz, 1H), 7.09 (d, J = 7.5 Hz, 2H), 7.05 (d, J = 8.1 Hz, 2H), 6.90—6.93 (m, 2H), 6.92 (d, J = 8.1 Hz, 1H), 6.91 (d, J = 2.3 Hz, 1H), 6.88 (dd, J = 8.6, 2.3 Hz, 1H), 6.81 (t, J = 7.5 Hz, 1H), 6.75 (d, J = 2.3 Hz, 1H), 5.63 (dd, J = 7.5, 2.3 Hz, 1H), 4.19 (s, 1H), 3.64 (s, 3H), 2.88—2.96 (m, 2H), 2.80 (ddd, J = 14.3, 10.4, 4.5 Hz, 1H), 2.61 (ddd, J = 14.3, 9.8, 6.0 Hz, 1H), 2.39—2.44 (m, 1H), 2.23—2.29 (m, 1H), 2.09 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 156.8, 144.6, 140.0, 136.7, 135.6, 134.8, 132.3, 130.3, 128.6, 128.5, 127.7, 127.4, 127.2, 122.9, 121.3, 118.7, 118.5, 115.5, 115.3, 112.4, 111.8, 111.3, 86.9, 70.5, 55.9, 53.4, 51.2, 24.7, 21.3; HRMS (ESI): calcd for $C_{33}H_{34}N_3O_4S$ $[M + H]^+$ 568.2270, found 568.2262.



5c

***trans*-*N*-Benzyl-*N*-(2-hydroxy-5-chloro-1-tosyl-indolin-3-yl)tryptamine (5c)**

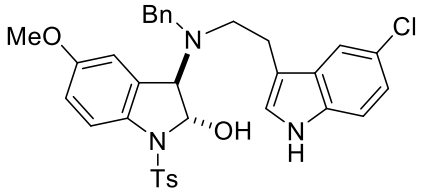
517 mg, 89% yield. white powder; mp 162—164 °C (MeOH); IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.68 (br s, 1H), 7.68 (d, J = 8.1 Hz, 2H), 7.39 (d, J = 9.1 Hz, 1H), 7.33 (dd, J = 8.6, 2.3 Hz, 1H), 7.27 (d, J = 8.0 Hz, 1H), 7.23 (t, J = 7.5 Hz, 2H), 7.19 (d, J = 1.7 Hz, 1H), 7.17 (t, J = 7.5 Hz, 1H), 7.14 (d, J = 8.1 Hz, 2H), 7.07 (d, J = 8.1 Hz, 2H), 7.04 (d, J = 7.4 Hz, 1H), 6.98 (t, J = 7.5 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.90 (d, J = 2.3 Hz, 1H), 6.80 (t, J = 7.5 Hz, 1H), 5.71 (dd, J = 8.0, 2.3 Hz, 1H), 4.16 (s, 1H), 3.13 (d, J = 13.8 Hz, 1H), 3.07 (d, J = 13.8 Hz, 1H), 2.77 (ddd, J = 13.7, 10.1, 5.2 Hz, 1H), 2.58 (ddd, J = 13.7, 9.7, 6.3 Hz, 1H), 2.37—2.43 (m, 1H), 2.17—2.23 (m, 1H), 2.12 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 144.9, 140.4, 139.8, 136.7, 135.6, 133.0, 130.4, 129.8, 128.7, 128.6, 128.2, 127.6, 127.4, 127.4, 126.2, 122.9, 121.3, 118.6, 118.5, 115.4, 112.3, 111.8, 87.0, 69.9, 53.8, 51.0, 24.8, 21.3;HRMS (ESI): calcd for $C_{32}H_{30}ClN_3O_3SNa$ $[M + Na]^+$ 594.1594 and 596.1565, found 594.1596 and 596.1585.



5d

***trans*-*N*-Benzyl-*N*-(2-hydroxy1-1-tosylindolin-3-yl)-5-chlorotryptamine (5d)**

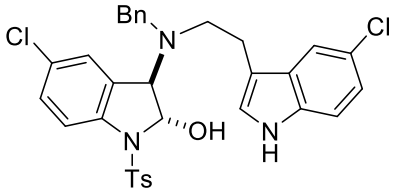
537 mg, 94% yield. white powder; mp 195—196 °C (MeOH); IR ($CHCl_3$): 3478 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.89 (br s, 1H), 7.69 (d, J = 8.6 Hz, 2H), 7.41 (d, J = 8.1 Hz, 1H), 7.26—7.29 (m, 2H), 7.20—7.23 (m, 2H), 7.13—7.17 (m, 4H), 7.06 (d, J = 8.1Hz, 2H), 6.97—7.00 (m, 3H), 6.92 (d, J = 7.5 Hz, 1H), 6.87 (d, J = 2.3 Hz, 1H), 5.68 (dd, J = 7.5, 1.7 Hz, 1H), 4.15 (s, 1H), 3.14 (d, J = 13.8 Hz, 1H), 3.09 (d, J = 14.3 Hz, 1H), 2.71 (ddd, J = 13.8, 10.3, 5.2 Hz, 1H), 2.54 (ddd, J = 14.3, 9.2, 6.3 Hz, 1H), 2.31—2.36 (m, 1H), 2.12—2.18 (m, 1H), 2.09 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 144.6, 141.4, 139.9, 135.9, 135.1, 130.5, 130.2, 130.0, 128.7, 128.6, 128.6, 127.4, 127.3, 126.4, 124.9, 124.1, 123.4, 121.2, 117.9, 113.9, 113.3, 112.4, 86.6, 70.1, 53.7, 50.7, 24.6, 21.3; HRMS (ESI): calcd for $C_{32}H_{30}ClN_3O_3S$ $[M + Na]^+$ 594.1594 and 5961578, found 594.1594 and 594.1581.



5e

***trans*-*N*-Benzyl-*N*-(2-hydroxy-5-methoxy-1-tosylindolin-3-yl)-5-chlorotryptamine (5e)**

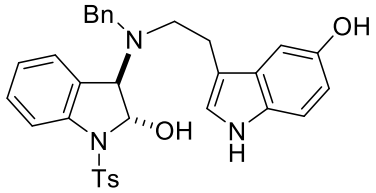
554 mg, 92% yield. white powder; mp 154—156 °C (MeOH); IR ($CHCl_3$): 3479 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.00 (br s, 1H), 7.66 (d, J = 8.6 Hz, 2H), 7.58 (d, J = 9.2 Hz, 1H), 7.19—7.26 (m, 4H), 7.14 (d, J = 6.9 Hz, 2H), 7.07 (dd, J = 8.6, 1.7 Hz, 1H), 6.95 (d, J = 8.0 Hz, 2H), 6.85 (dd, J = 9.2, 2.9 Hz, 1H), 6.82 (br s, 1H), 6.81 (d, J = 2.3 Hz, 1H), 6.68 (br s, 1H), 5.62 (s, 1H), 4.31 (s, 1H), 3.72 (s, 3H), 3.65 (br s, 1H), 3.20 (d, J = 13.2 Hz, 1H), 2.96 (d, J = 13.2 Hz, 1H), 2.59—2.70 (m, 2H), 2.17—2.28 (m, 2H), 2.09 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 157.0, 144.6, 139.2, 134.7, 134.6, 134.3, 131.4, 129.9, 128.6, 128.5, 128.3, 127.2, 126.9, 124.9, 123.3, 122.1, 118.4, 115.3, 115.0, 113.6, 112.1, 111.5, 86.6, 69.1, 55.7, 54.5, 50.4, 24.9, 21.3; HRMS (ESI): calcd for $C_{33}H_{32}ClN_3O_4SNa$ $[M + Na]^+$ 624.1700 and 6261685, found 624.1694 and 626.1719.



5f

***trans*-*N*-Benzyl-*N*-(5-chloro-2-hydroxy-1-tosylindolin-3-yl)-5-chlorotryptamine (5f)**

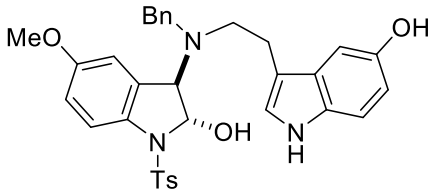
527 mg, 87% yield. white powder; mp 160—162 °C (MeOH); IR ($CHCl_3$): 3477 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.90 (br s, 1H), 7.69 (d, J = 8.6 Hz, 2H), 7.41 (d, J = 8.6 Hz, 1H), 7.32 (dd, J = 8.6, 2.3 Hz, 1H), 7.29 (d, J = 8.6 Hz, 1H), 7.23 (t, J = 8.6 Hz, 2H), 7.16 (t, J = 7.5 Hz, 1H), 7.13 (d, J = 7.5 Hz, 2H), 7.07—7.10 (m, 3H), 7.02 (d, J = 8.0 Hz, 1H), 6.97—6.99 (m, 2H), 6.88 (d, J = 1.7 Hz, 1H), 5.69 (dd, J = 8.0, 1.7 Hz, 1H), 4.12 (s, 1H), 3.16 (d, J = 13.8 Hz, 1H), 3.11 (d, J = 13.8 Hz, 1H), 2.69 (ddd, J = 14.4, 9.7, 5.2 Hz, 1H), 2.55 (ddd, J = 13.8, 9.2, 6.9 Hz, 1H), 2.30—2.35 (m, 1H), 2.06—2.12 (m, 1H), 2.10 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 144.9, 140.3, 139.7, 135.6, 135.1, 132.9, 130.3, 130.0, 128.7, 128.7, 128.6, 128.2, 127.4, 127.4, 126.1, 124.9, 123.4, 121.2, 117.8, 115.4, 113.3, 112.3, 87.0, 69.8, 53.8, 50.7, 24.6, 21.3; HRMS (ESI): calcd for $C_{32}H_{30}Cl_2N_3O_3S$ $[M + H]^+$ 606.1385, 608.1355 and 610.1349, found 606.1374, 608.1372 and 610.1384.



5g

***trans*-*N*-Benzyl-*N*-(2-hydroxy-1-tosylindolin-3-yl)serotonin (5g)**

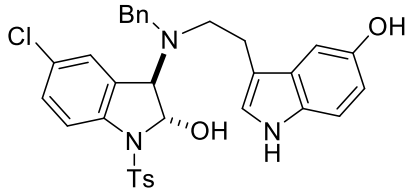
525 mg, 95% yield. white powder; mp 176—178 °C (MeOH); IR ($CHCl_3$): 3481 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.36 (br s, 1H), 8.42 (s, 1H), 7.73 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.26 (t, J = 8.0 Hz, 1H), 7.26 (d, J = 8.0 Hz, 1H), 7.20 (t, J = 7.5 Hz, 2H), 7.09—7.17 (m, 5H), 7.05 (d, J = 8.6 Hz, 1H), 7.02 (t, J = 7.5 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 678 (d, J = 2.3 Hz, 1H), 6.54 (s, 1H), 6.53 (dd, J = 8.6, 2.3 Hz, 1H), 5.72 (dd, J = 7.5, 1.8 Hz, 1H), 4.21 (s, 1H), 3.01 (d, J = 14.3 Hz, 1H), 2.94 (d, J = 14.3 Hz, 1H), 2.69—2.73 (m, 1H), 2.50—2.54 (m, 2H), 2.26—2.33 (m, 1H), 2.15 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 150.5, 144.7, 141.4, 140.1, 136.0, 131.2, 130.5, 130.3, 129.8, 128.6, 128.4, 128.4, 127.5, 127.2, 126.5, 124.2, 123.3, 113.9, 112.0, 111.7, 111.6, 102.8, 86.6, 70.2, 53.5, 51.5, 24.6, 21.3; HRMS (ESI): calcd for $C_{32}H_{31}N_3O_4SNa$ $[M + Na]^+$ 576.1933, found 576.1939.



5h

***trans*-*N*-Benzyl-*N*-(2-hydroxy—5-methoxy-1-tosylindolin-3-yl)serotonin (5h)**

543 mg, 93% yield. white powder; mp 121—123 °C (MeOH); IR ($CHCl_3$): 3481, 3244 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.38 (br d, J = 1.7 Hz, 1H), 8.44 (s, 1H), 7.67 (d, J = 8.6 Hz, 2H), 7.36 (d, J = 8.6 Hz, 1H), 7.16—7.20 (m, 4H), 7.11 (t, J = 8.6 Hz, 1H), 7.05 (d, J = 8.6 Hz, 1H), 7.03 (d, J = 7.5 Hz, 2H), 6.92 (d, J = 7.5 Hz, 1H), 6.85 (dd, J = 8.6, 2.9 Hz, 1H), 6.80 (d, J = 2.3 Hz, 1H), 6.74 (d, J = 2.9 Hz, 1H), 6.57 (d, J = 2.3 Hz, 1H), 6.53 (dd, J = 8.6, 2.3 Hz, 1H), 5.69 (dd, J = 7.4, 2.3 Hz, 1H), 4.20 (s, 1H), 3.64 (s, 3H), 2.84 (d, J = 13.7 Hz, 1H), 2.67—2.77 (m, 2H), 2.51—2.58 (m, 2H), 2.27—2.35 (m, 1H), 2.14 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6, 60 °C): δ 156.7, 150.6, 144.5, 140.2, 136.1, 135.0, 132.1, 131.4, 130.3, 128.5, 128.5, 128.4, 127.5, 127.2, 123.1, 115.7, 115.1, 112.0, 111.8, 111.8, 111.6, 103.0, 87.2, 70.8, 56.0, 53.7, 51.8, 24.6, 21.3; HRMS (ESI): calcd for $C_{33}H_{33}N_3O_5SNa$ $[M + Na]^+$ 606.2039, found 606.2039.



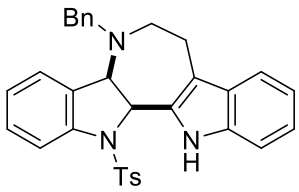
5i

***trans*-*N*-Benzyl-*N*-(5-chloro-2-hydroxy-1-tosylindolin-3-yl)serotonin (5i)**

531 mg, 90% yield. white powder; mp 120—123 °C (MeOH); IR ($CHCl_3$): 3482 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.38 (br d, J = 1.7 Hz, 1H), 8.44 (s, 1H), 7.73 (d, J = 8.6 Hz, 2H), 7.39 (d, J = 8.6 Hz, 1H), 7.31 (dd, J = 8.6, 2.3 Hz, 1H), 7.19—7.22 (m, 3H), 7.18 (d, J = 8.0 Hz, 2H), 7.14 (t, J = 7.5 Hz, 1H), 7.09 (d, J = 6.9 Hz, 2H), 7.06 (d, J = 8.0 Hz, 2H), 6.79 (d, J = 2.3 Hz, 1H), 6.52—6.55 (m, 2H), 5.73 (dd, J = 8.1, 2.3 Hz, 1H), 4.19 (s, 1H), 3.03 (d, J = 14.4 Hz, 1H), 2.93 (d, J = 14.4 Hz, 1H), 2.67—2.73 (m, 1H), 2.49—2.55 (m, 2H), 2.26—2.29 (m, 1H),2.16 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 150.6, 145.0, 140.3, 139.8, 135.7, 133.0, 131.2, 130.5, 129.9, 128.6, 128.4, 128.4, 128.1, 127.5, 127.3, 126.2, 123.3, 115.4, 112.0, 111.7, 111.5, 102.8, 87.0, 69.9, 53.5, 51.4, 24.5, 21.4; HRMS (ESI): calcd for $C_{32}H_{30}ClN_3O_4SNa$ $[M + Na]^+$ 610.1543 and 612.1514, found 610.1541 and 612.1543.

General Procedure for the Cyclization of 5

A mixture of **5** (0.2 mmol) and $InCl_3 \cdot 4H_2O$ (0.03 g, 0.1 mmol) in MeCN (4 mL) was heated under reflux with stirring for 20 hours. After addition of H_2O, the whole was extracted with AcOEt (3 x 10 mL), washed with brine (10 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$–hexane, 1:2—2:3) to give **6a**—**6i**.

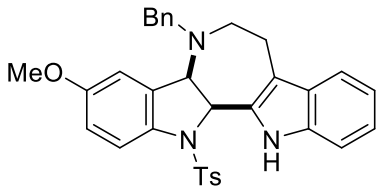


6a

***cis*-5-Benzyl-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6a)**

63.7 mg, 61% yield. white powder; mp 221—223 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.00 (br s, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.37—7.41 (m, 4H), 7.15—7.28 (m, 9H), 7.05 (t, J = 8.0 Hz, 1H), 6.99 (d, J = 8.6 Hz, 2H), 5.57 (d, J = 8.0 Hz, 1H), 4.20 (d, J = 8.1 Hz, 1H), 4.07 (d, J = 13.2 Hz, 1H), 3.81 (d, J = 13.7 Hz, 1H), 3.49—3.55 (m, 1H), 3.15—3.22 (m, 2H), 2.69—2.74 (m, 1H), 2.28 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.3, 139.8, 138.6, 136.3, 135.2, 134.6, 130.0, 129.8, 128.8, 128.5, 128.3, 128.1, 127.2, 126.5, 126.0, 124.8,

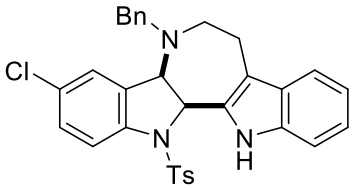
122.8, 119.4, 119.1, 118.7, 112.6, 110.9, 64.9, 64.5, 55.2, 45.9, 23.3, 21.7; HRMS (ESI): calcd for $C_{32}H_{29}N_3O_2SNa$ $[M + Na]^+$ 542.1874, found 542.1878.



6b

***cis*-5-Benzyl-3-methoxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6b)**

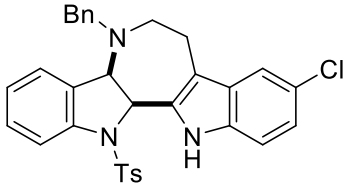
64.0 mg, 58% yield. white powder; mp 192—194 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.94 (br s, 1H), 7.49 (d, J = 8.0 Hz, 1H), 7.40 (d, J = 8.0 Hz, 1H), 7.35—7.38 (m, 3H), 7.24—7.28 (m, 5H), 7.19 (t, J = 8.0 Hz, 1H), 7.05 (t, J = 7.4 Hz, 1H), 6.97 (d, J = 8.0 Hz, 2H), 6.75—6.77 (m, 2H), 5.55 (d, J = 8.6 Hz, 1H), 4.08 (d, J = 8.6 Hz, 1H), 4.05 (d, J = 13.8 Hz, 1H), 3.81 (s, 3H), 3.78 (d, J = 13.8 Hz, 1H),3.48—3.55 (m, 1H), 3.15—3.22 (m, 2H), 2.69—2.74 (m, 1H), 2.28 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 158.5, 144.2, 138.5, 137.2, 136.2, 134.4, 132.9, 130.1, 129.8, 128.5, 128.3, 128.1, 127.2, 126.6, 122.8, 120.4, 119.1, 118.6, 113.3, 112.5, 110.8, 110.7, 64.9, 64.6, 55.8, 55.1, 46.2, 23.3, 21.7; HRMS (ESI): calcd for $C_{33}H_{32}N_3O_3S$ $[M + H]^+$ 550.2164, found 550.2170.



6c

***cis*-5-Benzyl-3-chloro-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6c)**

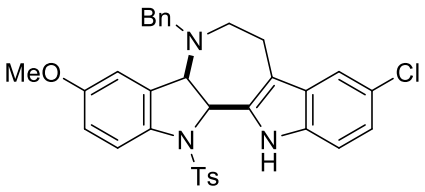
67.6 mg, 61% yield. white powder; mp 255—256 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.96 (br s, 1H), 7.52 (d, J = 8.6 Hz, 1H), 7.41 (d, J = 8.1 Hz, 1H), 7.37—7.40 (m, 3H), 7.24—7.31 (m, 5H), 7.19—7.23 (m, 2H), 7.17—7.18 (m, 1H), 7.07 (t, J = 8.0 Hz, 1H), 7.01 (d, J = 8.0 Hz, 2H), 5.57 (d, J = 8.6 Hz, 1H), 4.15 (d, J = 8.6 Hz, 1H), 4.05 (d, J = 13.7 Hz, 1H), 3.78 (d, J = 13.7 Hz, 1H), 3.46—3.52 (m, 1H), 3.16—3.23 (m, 2H), 2.73—2.77 (m, 1H), 2.30 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.6, 138.4, 138.2, 137.4, 136.3, 134.4, 131.6, 130.0, 129.5, 128.9, 128.6, 128.3, 128.0, 127.4, 126.5, 125.0, 123.0, 120.4, 119.3, 118.7, 112.7, 110.9, 64.8, 64.5, 55.1, 46.3, 23.3, 21.7; HRMS (ESI): calcd for $C_{32}H_{28}ClN_3O_2SNa$ $[M + Na]^+$ 576.1488 and 578.1459, found 576.1492 and 578.1498.



6d

***cis*-5-Benzyl-9-chloro-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6d)**

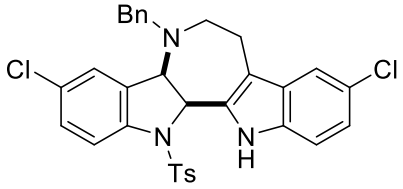
59.1 mg, 53% yield. white prisms; mp 219—221 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3447, 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.04 (br s, 1H), 7.60 (d, J = 8.0 Hz, 1H), 7.38 (d, J = 8.6 Hz, 2H), 7.35 (d, J = 1.8 Hz, 1H), 7.21—7.29 (m, 8H), 7.17 (t, J = 7.5 Hz, 1H), 7.13 (dd, J = 8.6, 2.3 Hz, 1H), 6.99 (d, J = 8.6 Hz, 2H), 5.52 (d, J = 8.6 Hz, 1H), 4.20 (d, J = 8.6 Hz, 1H), 4.05 (d, J = 13.2 Hz, 1H), 3.79 (d, J = 13.8 Hz, 1H), 3.45—3.52 (m, 1H), 3.09—3.17 (m, 2H), 2.60—2.65 (m, 1H), 2.28 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.4, 139.7, 138.4, 134.9, 134.6, 134.5, 131.6, 129.8, 129.3, 128.9, 128.5, 128.3, 127.3, 126.5, 126.0, 124.8, 124.8, 123.0, 119.3, 118.3, 112.4, 111.9, 64.8, 64.3, 55.3, 45.7, 23.2, 21.7; HRMS (ESI): calcd for $C_{32}H_{29}ClN_3O_2S$ $[M + H]^+$ 554.1669 and 556.1653, found 554.1667 and 556.1687.



6e

***cis*-5-Benzyl-9-chloro-3-methoxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6e)**

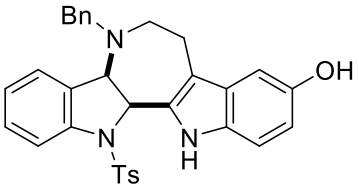
64.3 mg, 55% yield. white powder; mp 221—224 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3417 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.52 (br s, 1H), 7.50 (d, J = 8.6 Hz, 1H), 7.41 (d, J = 8.6 Hz, 2H), 7.38 (d, J = 2.3 Hz, 1H), 7.35 (d, J = 8.6 Hz, 1H), 7.21—7.28 (m, 5H), 7.03—7.06 (m, 3H), 6.81 (dd, J = 8.6, 2.3 Hz, 1H), 6.58 (d, J = 1.7 Hz, 1H), 5.70 (d, J = 8.6 Hz, 1H), 4.13 (d, J = 8.6 Hz, 1H), 3.89 (d, J = 13.8 Hz, 1H), 3.79 (d, J = 14.3 Hz, 1H), 3.68 (s, 3H), 3.15—3.20 (m, 1H), 2.92—3.00 (m, 2H), 2.45—2.49 (m, 1H), 2.21 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 158.1, 144.5, 139.4, 136.6, 134.8, 134.6, 133.4, 133.4, 130.3, 129.4, 128.8, 128.6, 127.5, 127.2, 123.6, 121.9, 119.6, 117.9, 114.2, 113.7, 111.9, 110.8, 64.9, 64.1, 56.3, 56.0, 46.7, 22.6, 21.6; HRMS (ESI): calcd for $C_{33}H_{31}ClN_3O_3S$ $[M + H]^+$ 584.1775 and 586.1760, found 584.1768 and 586.1777.



6f

***cis*-5-Benzyl-3,9-dichloro-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6f)**

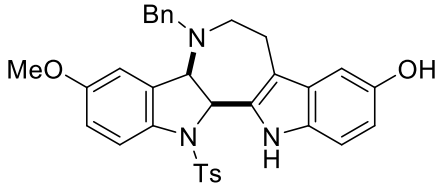
37.6 mg, 32% yield. white powder; mp 191—193 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.99 (br s, 1H), 7.52 (d, J = 8.6 Hz, 1H), 7.38 (d, J = 8.6 Hz, 2H), 7.36 (d, J = 1.8 Hz, 1H), 7.20—7.30 (m, 7H), 7.14—7.16 (m, 2H), 7.01 (d, J = 8.6 Hz, 2H), 5.51 (d, J = 8.0 Hz, 1H), 4.15 (d, J = 8.6 Hz, 1H), 4.04 (d, J = 13.8 Hz, 1H), 3.76 (d, J = 13.8 Hz, 1H), 3.43—3.50 (m, 1H), 3.11—3.21 (m, 2H), 2.64—2.69 (m, 1H), 2.30 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.7, 138.3, 138.0, 137.1, 134.6, 134.2, 131.7, 131.1, 130.0, 129.2, 129.0, 128.6, 128.3, 127.4, 126.5, 125.0, 124.9, 123.2, 120.3, 118.3, 112.5, 111.9, 64.6, 64.4, 55.2, 46.1, 23.2, 21.7; HRMS (ESI): calcd for $C_{32}H_{28}Cl_2N_3O_2S$ $[M + H]^+$ 588.1279, 590.1250 and 592.1243, found 588.1269, 590.1247 and 592.1198.



6g

***cis*-5-Benzyl-9-hydroxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6g)**

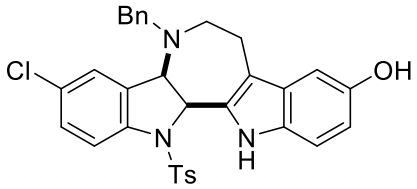
76.1 mg, 71% yield. white powder; mp 240—241 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3447, 3242 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 9.97 (br s, 1H), 8.57 (br s, 1H), 7.42—7.45 (m, 3H), 7.20—7.27 (m, 7H), 7.09—7.14 (m, 2H), 7.06 (d, J = 8.6 Hz, 2H), 6.62 (d, J = 2.3 Hz, 1H), 6.58 (dd, J = 8.6, 2.3 Hz, 1H), 5.62 (d, J = 8.6 Hz, 1H), 4.25 (d, J = 8.6 Hz, 1H), 3.87 (d, J = 14.4 Hz, 1H), 3.78 (d, J = 14.3 Hz, 1H), 3.08—3.14 (m, 1H), 2.82—2.94 (m, 2H), 2.34—2.39 (m, 1H), 2.20 (s, 3H); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 150.9, 144.5, 140.6, 139.6, 134.7, 134.6, 131.5, 130.9, 130.2, 129.1, 128.9, 128.8, 128.6, 127.4, 127.1, 125.9, 125.6, 118.0, 112.4, 111.1, 102.5, 79.7, 64.5, 64.3, 56.4, 46.1, 22.9, 21.5; HRMS (ESI): calcd for $C_{32}H_{30}N_3O_3S$ $[M + H]^+$ 536.2008, found 536.2006.



6h

***cis*-5-Benzyl-9-hydroxy-3-methoxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6h)**

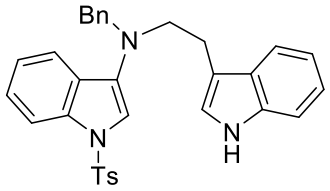
73.8 mg, 65% yield. white powder; mp 227—229 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.80 (br s, 1H), 7.49 (d, J = 8.0 Hz, 1H), 7.35 (d, J = 8.6 Hz, 2H), 7.22—7.27 (m, 5H), 7.19 (d, J = 8.6 Hz, 1H), 6.96 (d, J = 8.0 Hz, 2H), 6.81 (s, 1H), 6.74—6.76 (m, 3H), 5.52 (d, J = 8.6 Hz, 1H), 4.89 (br s, 1H), 4.06 (d, J = 8.1 Hz, 1H), 4.01 (d, J = 13.8 Hz, 1H), 3.80 (s, 3H), 3.74 (d, J = 13.8 Hz, 1H),3.44—3.50 (m, 1H), 3.04—3.15 (m, 2H), 2.54—2.59 (m, 1H), 2.27 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ158.5, 149.2, 144.2, 138.5, 137.2, 134.3, 132.9, 131.5, 131.3, 129.8, 128.8, 128.5, 128.3, 127.2, 126.6, 120.4, 113.4, 112.5, 112.0, 111.5, 110.8, 103.4, 64.9, 64.5, 55.8, 55.1, 46.2, 23.3, 21.7; HRMS (ESI): calcd for $C_{33}H_{31}N_3O_4SNa$ $[M + Na]^+$ 588.1933, found 588.1930.



6i

***cis*-5-Benzyl-3-chloro-9-hydroxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (6i)**

48.9 mg, 43% yield. white powder; mp 225—227 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3420 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 10.04 (br s, 1H), 8.58 (s, 1H), 7.47 (d, J = 8.6 Hz, 2H), 7.44 (d, J = 8.6 Hz, 1H), 7.31 (dd, J = 8.6, 1.7 Hz, 1H), 7.20—7.28 (m, 6H), 7.08—7.11 (m, 3H), 6.62 (d, J = 2.3 Hz, 1H), 6.58 (dd, J = 8.6, 2.3 Hz, 1H), 5.65 (d, J = 8.6 Hz, 1H), 4.27 (d, J = 8.6 Hz, 1H), 3.85 (d, J = 14.3 Hz, 1H), 3.77 (d, J = 14.3 Hz, 1H), 3.03—3.09 (m, 1H), 2.82—2.94 (m, 2H), 2.33—2.38 (m, 1H), 2.22 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 150.9, 144.8, 139.6, 139.4, 137.0, 134.5, 131.0, 130.9, 130.4, 129.9, 129.2, 128.8, 128.8, 128.7, 127.5, 127.2, 125.4, 119.4, 112.5, 112.5, 111.2, 102.4, 64.9, 64.2, 56.5, 46.2, 22.8, 21.6; HRMS (ESI): calcd for $C_{32}H_{29}ClN_3O_3S$ $[M + H]^+$ 570.1618 and 572.1589, found 570.1625 and 572.1615.

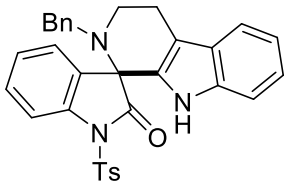


7a

***N*-Benzyl-*N*-(1-tosylindol-3-yl)tryptamine (7a)**

A mixture of **5a** (108 mg, 0.2 mmol) and $InCl_3 \cdot 4H_2O$ (0.018 g, 0.06 mmol) in MeCN (4 mL) was heated under reflux with stirring for 20 hours. After addition of H_2O, the whole was extracted with AcOEt (3 x 10 mL), washed with brine (10 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$:hexane = 2:3 and AcOEt:hexane = 1:5) to give **6a** (53.1 mg, 51%), **8a** (11.1 mg, 10%) and **7a** (12.3 mg, 12%).

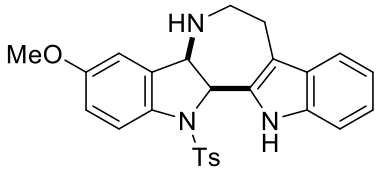
12.3 mg, 12% yield. white powder; mp 160—163 °C ($CHCl_3$-MeOH); IR ($CHCl_3$): 3480 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.01 (d, J = 8.0 Hz, 1H), 7.92 (br s, 1H), 7.50 (d, J = 8.1 Hz, 1H), 7.45 (d, J = 8.6 Hz, 2H), 7.25—7.35 (m, 8H), 7.14—7.18 (m, 2H), 7.00—7.05 (m, 3H), 6.90 (s, 1H), 6.90 (s, 1H), 4.37 (s, 2H), 3.47—3.50 (m, 2H), 2.98—3.01 (m, 2H), 2.26 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 144.4, 138.2, 136.3, 135.4, 135.4, 134.5, 129.6, 128.5, 128.0, 127.5, 127.5, 127.1, 126.7, 125.0, 123.1, 122.1, 121.7, 120.0, 119.4, 118.8, 114.6, 113.8, 112.2, 111.2, 57.0, 53.3, 22.6, 21.6; HRMS (ESI): calcd for $C_{32}H_{30}N_3O_2S$ $[M + H]^+$ 520.2064, found 520.2059.



8a

2'-benzyl-1-tosyl-2',3',4',4a',5',6',9',9a'-octahydrospiro[indoline-3,1'-pyrido[3,4-b]indol]-2-one (8a)

11.1 mg, 10% yield. yellow prisms; mp 229—231 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3455, 1755 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.09 (d, J = 8.6 Hz, 1H), 7.98 (d, J = 8.6 Hz, 2H), 7.44—7.50 (m, 2H), 7.31—7.33 (m, 3H), 7.18—7.24 (m, 4H), 7.01—7.14 (m, 5H), 6.89 (br s, 1H), 3.53—3.59 (m, 1H), 3.14 (d, J = 13.8 Hz, 1H), 2.82—2.91 (m, 3H), 2.74—2.78 (m, 1H), 2.42 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 174.5, 146.1, 140.0, 138.9, 136.7, 135.4, 130.7, 130.0, 129.7, 129.5, 128.4, 128.0, 128.0, 127.3, 126.8, 126.3, 126.0, 122.7, 119.8, 118.9, 114.4, 112.3, 111.1, 66.8, 53.8, 43.5, 21.8, 21.2; HRMS (ESI): calcd for $C_{32}H_{27}N_3O_3SNa$ $[M + Na]^+$ 556.1671, found 556.1672.



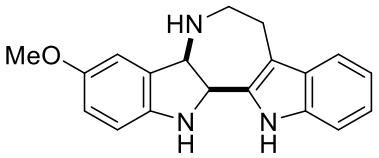
9

***cis*-3-Methoxy-13-tosyl-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (9)**

To a solution of **6b** (550 mg, 1 mmol) in AcOH (20 mL) and AcOEt (20 mL) was added 15% $Pd(OH)_2$ on activated charcoal (469 mg, 0.5 mmol) and the mixture was vigorously stirred for 2.5 hours under hydrogen atmosphere. After filtration, the filtrate was evaporated under reduced pressure. After addition of H_2O and saturated $NaHCO_3$ aq., the whole was extracted with AcOEt (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$, 2% MeOH/$CHCl_3$) to give **9** (449 mg, 98%)

449 mg, 98% yield. colorless powder; mp 195—196 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3458 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.91 (br s, 1H), 7.61 (d, J = 9.2 Hz, 1H), 7.51 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 1H), 7.36 (d, J = 8.6 Hz, 1H), 7.20 (d, J = 8.0 Hz, 2H), 7.16 (t, J = 8.6 Hz, 1H), 7.05 (t, J = 8.0 Hz, 1H), 6.81 (dd, J = 9.7 2.9 Hz, 1H), 6.70—6.71 (m, 1H), 5.60 (d, J = 8.6 Hz, 1H), 4.35 (d, J = 8.6 Hz, 1H), 3.75 (s, 3H), 3.19—3.24 (m, 1H), 2.88—3.04 (m, 3H), 3.75 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 158.6, 144.6, 136.7, 135.5, 134.2, 133.7, 123.0, 129.3, 128.8, 127.3, 122.3, 120.3, 119.3, 118.2, 114.8, 111.0, 110.4, 109.7, 65.8, 63.3, 55.8, 43.0, 27.8, 21.7;

HRMS (ESI): calcd for $C_{26}H_{26}N_3O_3S$ $[M + H]^+$ 460.1695, found 460.1692.



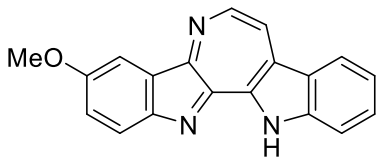
10

***cis*-3-Methoxy-4b,6,7,12,12b,13-hexahydroazepino[3,2-b:4,5-b']bisindole (10)**

To a mixture of **9** (115 mg, 0.25 mmol) and Na_2HPO_4 (710 mg, 5.0 mmol) in THF (1 mL) and MeOH (4 mL) was added 3% Na-Hg (5.75 g, 7.5 mmol) and the mixture was heated under reflux with stirring for 2 hours. After addition of H_2O, the whole was extracted with AcOEt (3 x 10 mL), washed with brine (10 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (2% MeOH/$CHCl_3$) to give **10** (52.7 mg, 69%)

52.7 mg, 69% yield. colorless powder; decomp. 170—173 °C ($CHCl_3$-hexane); IR ($CHCl_3$): 3462, 3445 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.31 (br s, 1H), 7.50 (d, J = 8.0 Hz, 1H), 7.31 (d, J = 8.6

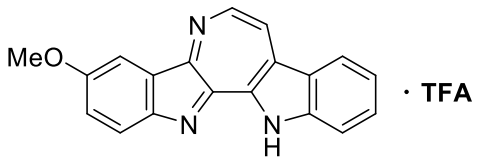
Hz, 1H), 7.16 (t, J = 8.6 Hz, 1H), 7.10 (t, J = 8.1 Hz, 1H), 6.93—6.94 (m, 1H), 6.72—6.75 (m, 2H), 5.00 (d, J = 6.9 Hz, 1H), 4.68 (d, J = 7.5 Hz, 1H), 3.77 (s, 3H), 3.31 (dt, J = 13.8, 5.7 Hz, 1H), 3.14 (ddd, J = 13.8, 8.0, 5.2 Hz, 1H), 2.95—3.07 (m, 2H); ^{13}C-NMR (125 MHz, $CDCl_3$): δ 155.3, 142.9, 135.5, 134.5, 132.2, 129.1, 122.0, 119.5, 118.4, 115.4, 113.7, 110.7, 110.7, 110.5, 65.1, 61.7, 56.0, 45.3, 27.1; HRMS (ESI): calcd for $C_{19}H_{20}N_3O$ $[M + H]^+$ 306.1606, found 306.1599.



Iheyamine A (1)

To a solution of **10** (31.0 mg, 0.1 mmol) in 1,4-dioxane (4 mL) was added DDQ (80.3 mg, 0.35 mmol) and the mixture was stirred for 3 hours at room temperature. After addition of H_2O and saturated $NaHCO_3$ aq., the whole was extracted with AcOEt (3 x 5 mL), washed with brine (5 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$, 1% MeOH/$CHCl_3$) to give **1** (27.1 mg, 89%).

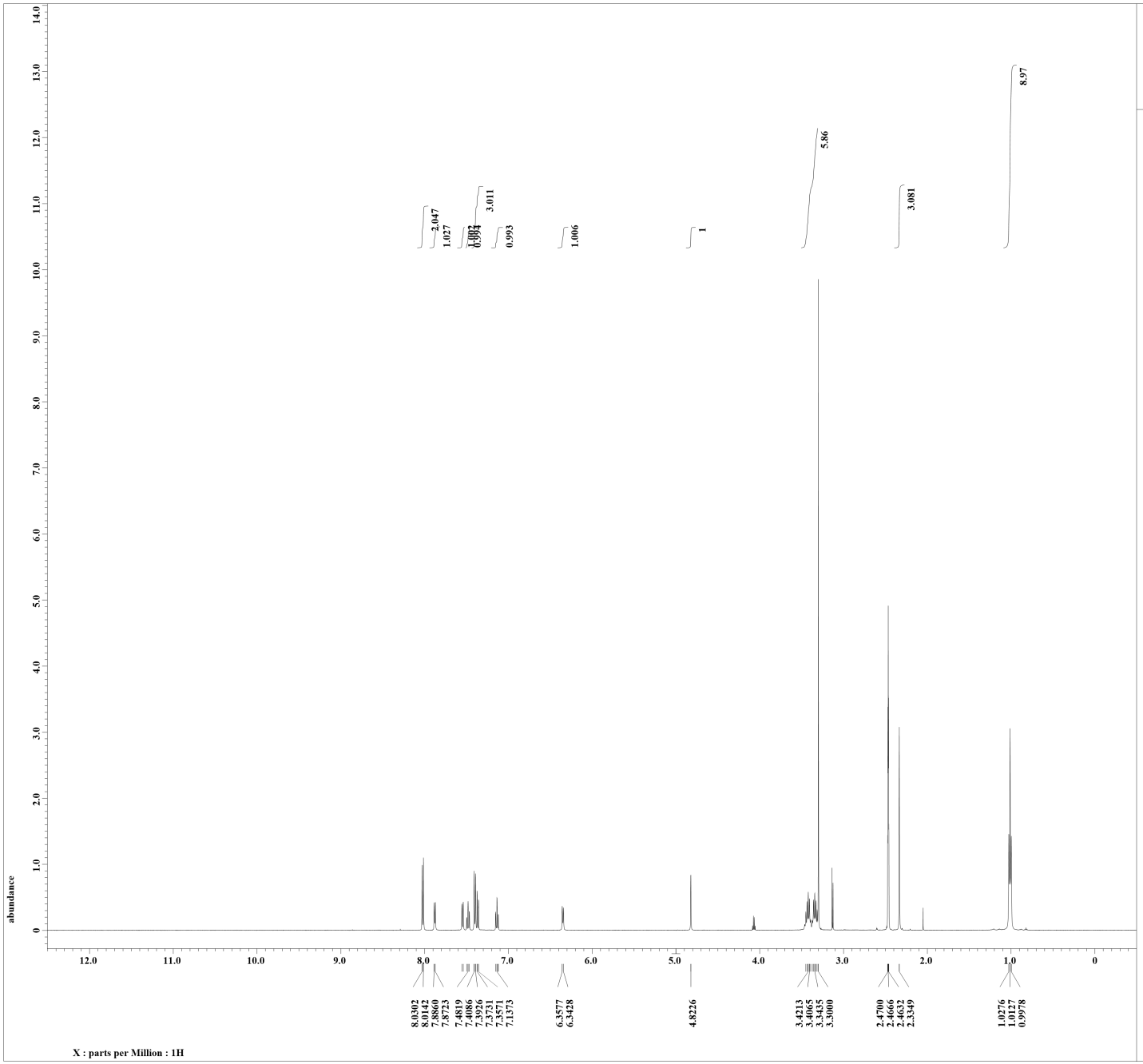
27.1 mg, 89% yield. purple powder; decomp. 289—298 °C ($CHCl_3$); IR (KBr): 1618, 1584, 1562, 1497, 1393, 1383 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.01 (d, J = 6.9 Hz, 1H), 8.53 (d, J = 6.3 Hz, 1H), 8.38 (d, J = 8.0 Hz, 1H), 8.03 (d, J = 2.3 Hz, 1H), 7.75 (d, J = 8.0 Hz, 1H), 7.68 (t, J = 8.0 Hz, 1H), 7.57 (d, J = 8.6 Hz, 1H), 7.51 (t, J = 8.6 Hz, 1H), 7.31 (dd, J = 8.6, 1.7 Hz, 1H), 4.02 (s, 3H); HRMS (ESI): calcd for $C_{19}H_{14}N_3O$ $[M + H]^+$ 300.1137, found 300.1142.



Iheyamine A (1) TFA salt

To a solution of iheyamine A (6.3 mg, 0.02 mmol) in $CDCl_3$ (2 mL) was added TFA (4 drops) and the mixture was stirred for 20 minutes at room temperature. The resulting solution was concentrated *in vacuo* to give the *title compound* (8.3 mg, 0.02 mmol, 99 %).

8.3 mg, 99% yield. purple powder; decomp. 216—222 °C ($CHCl_3$); IR (KBr): 2654, 1661, 1620, 1589, 1506, 1342 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.42 (d, J = 6.3 Hz, 1H), 8.89 (d, J = 6.9 Hz, 1H), 8.44 (d, J = 8.1 Hz, 1H), 8.08 (d, J = 8.6 Hz, 1H), 8.00 (d, J = 3.5 Hz, 1H), 8.00 (d, J = 8.1 Hz, 1H), 7.51 (t, J = 8.1 Hz, 1H), 7.64 (t, J = 8.1 Hz, 1H), 7.55 (d, J = 9.2, 2.3 Hz, 1H), 4.03 (s, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$): 157.7, 149.2, 144.0, 143.4, 138.2, 136.0, 133.2, 128.4, 126.2, 124.7, 124.7, 124.4, 124.0, 121.6, 115.8, 115.2, 102.9, 56.1; HRMS (ESI): calcd for $C_{19}H_{14}N_3O$ $[M + H]^+$ 300.1137, found 300.1134.

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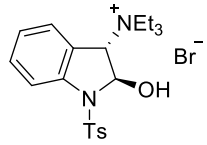
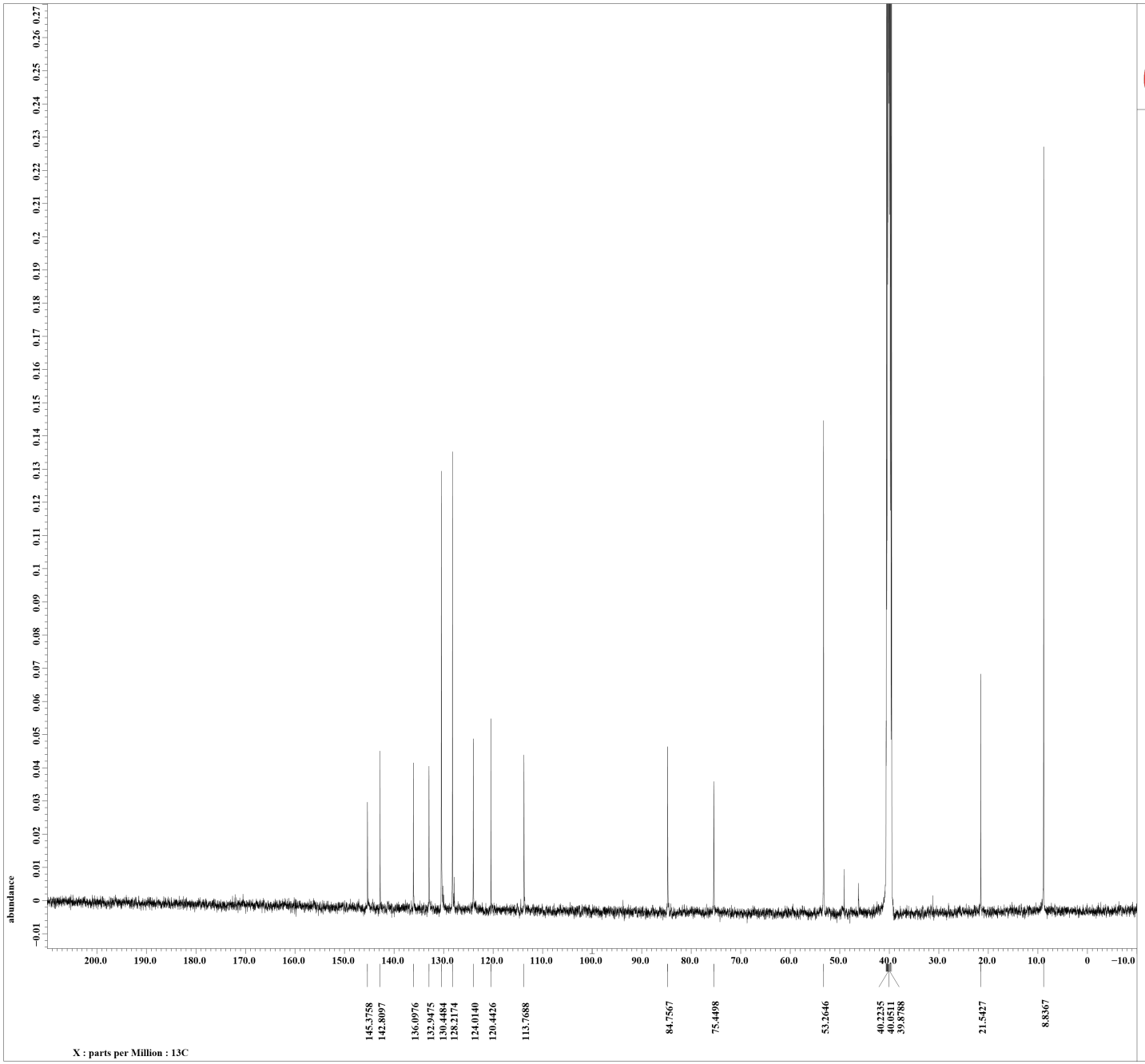


2a

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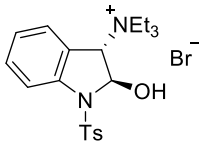
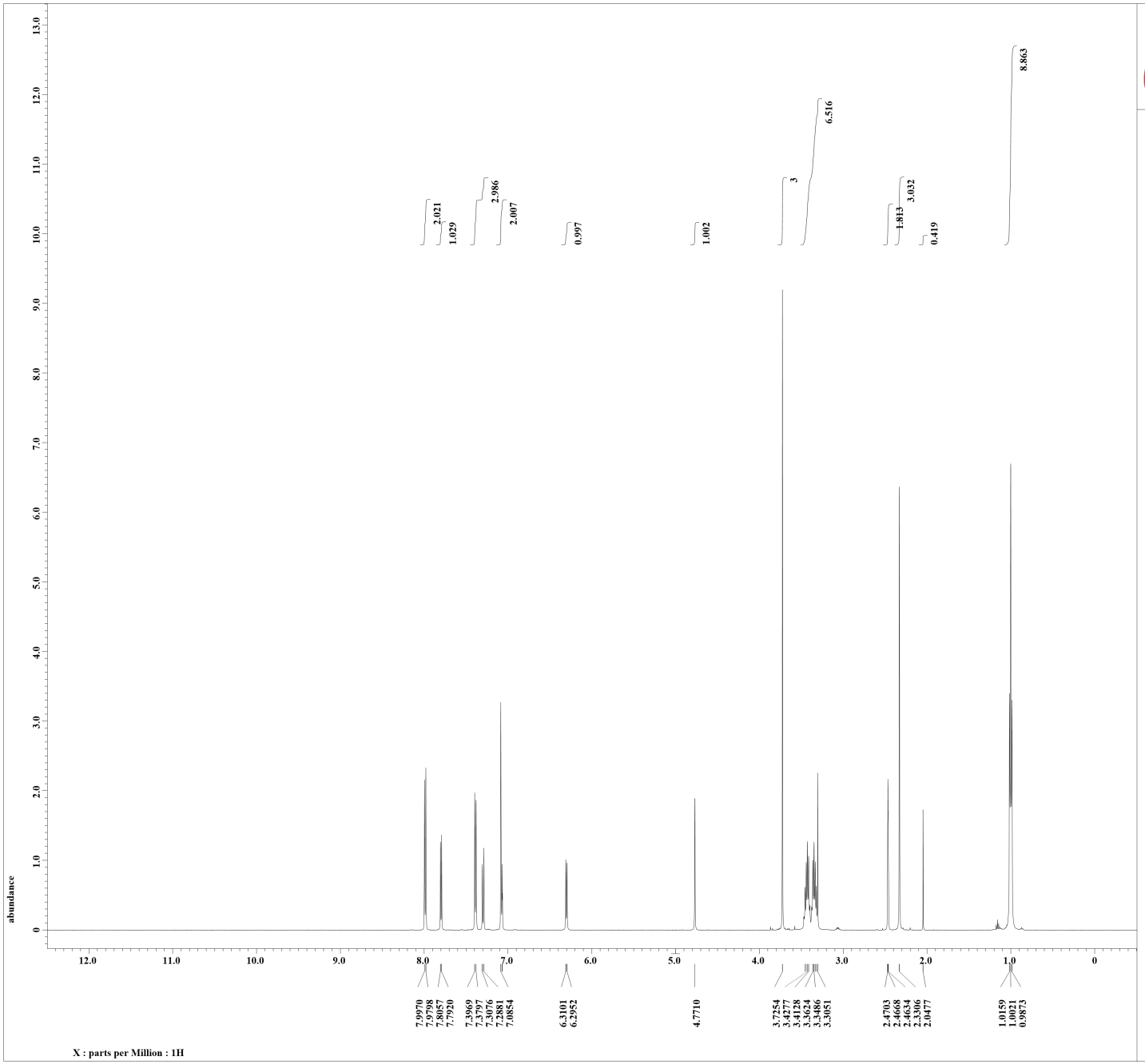


2a

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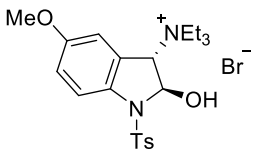
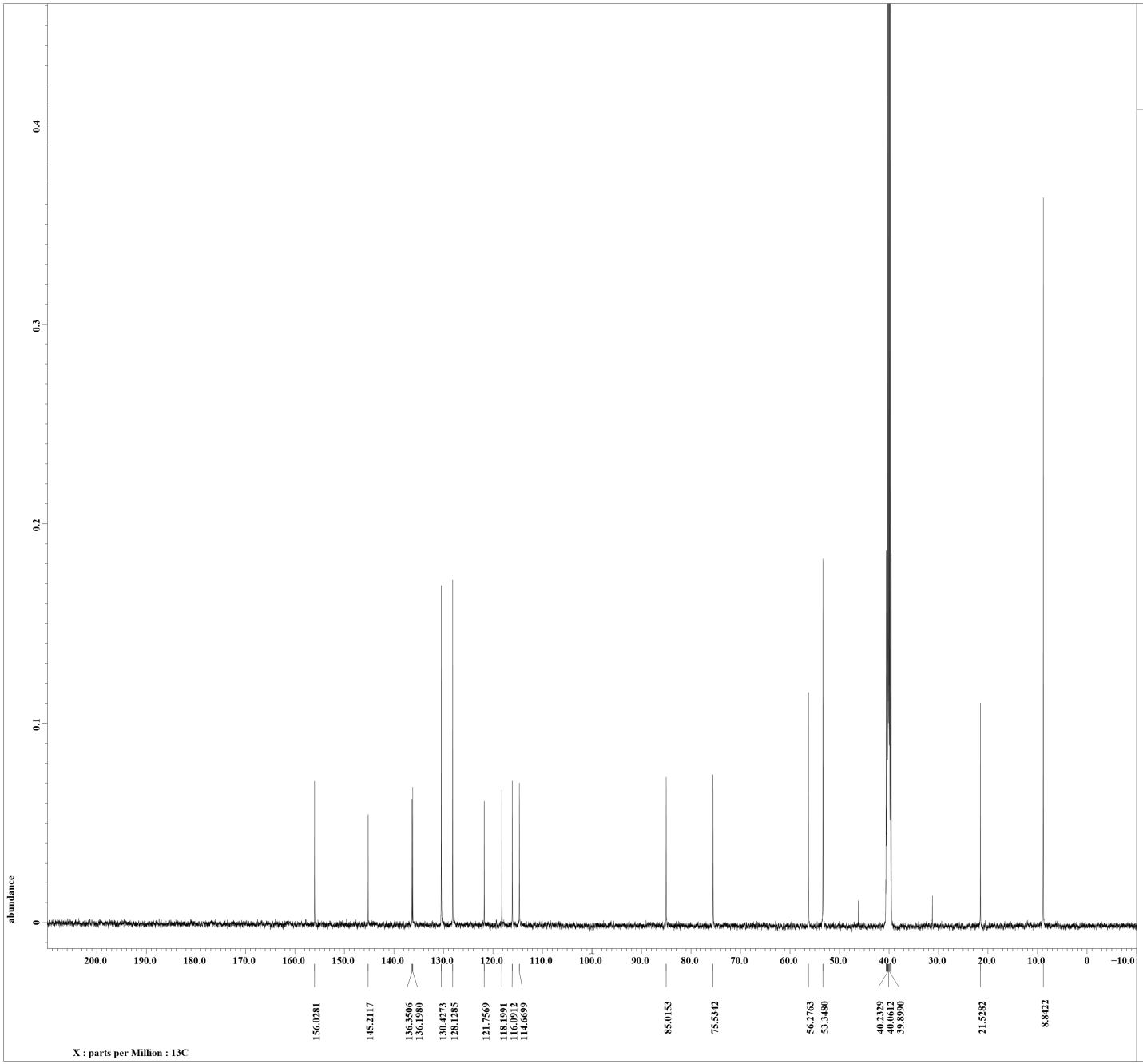
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.3[dC]
```



2b

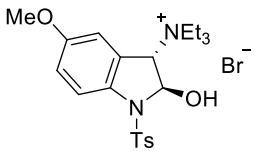



```
Filename          = yama-17-07-24-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#309683
Solvent           = DMSO-D6
Creation_time     = 24-JUL-2017 05:50:36
Revision_time     = 25-DEC-2017 09:39:31
Current_time      = 25-DEC-2017 09:39:55

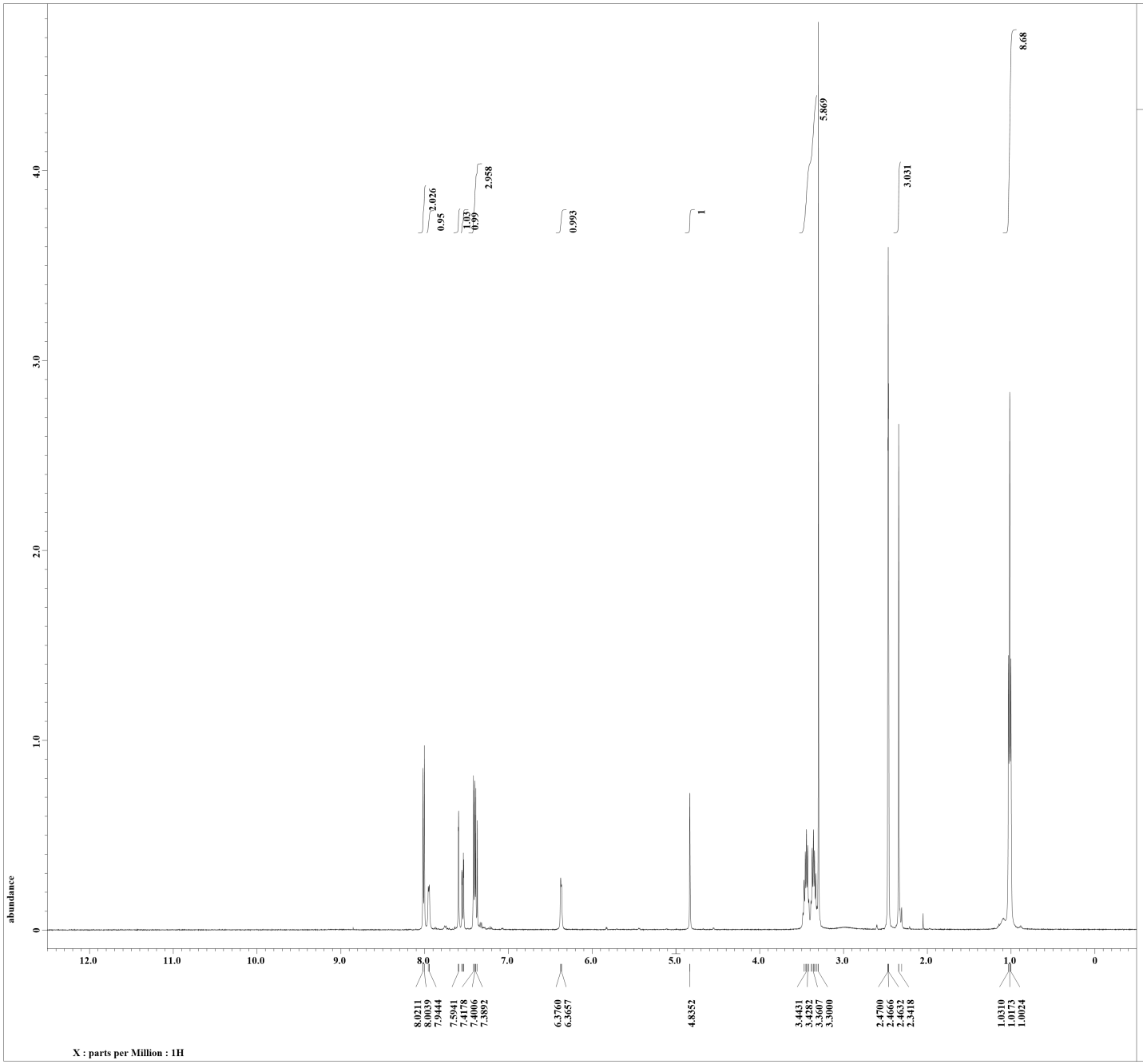
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 2860
Total_scans       = 2860

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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          = 22.7[dC]
```



2b

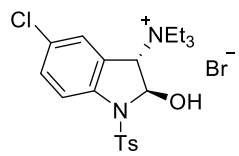



```
Filename          = yama-140.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#470677
Solvent           = DMSO-D6
Creation_time     = 12-JUL-2017 12:20:07
Revision_time     = 12-JUL-2017 18:46:39
Current_time      = 12-JUL-2017 18:48:02

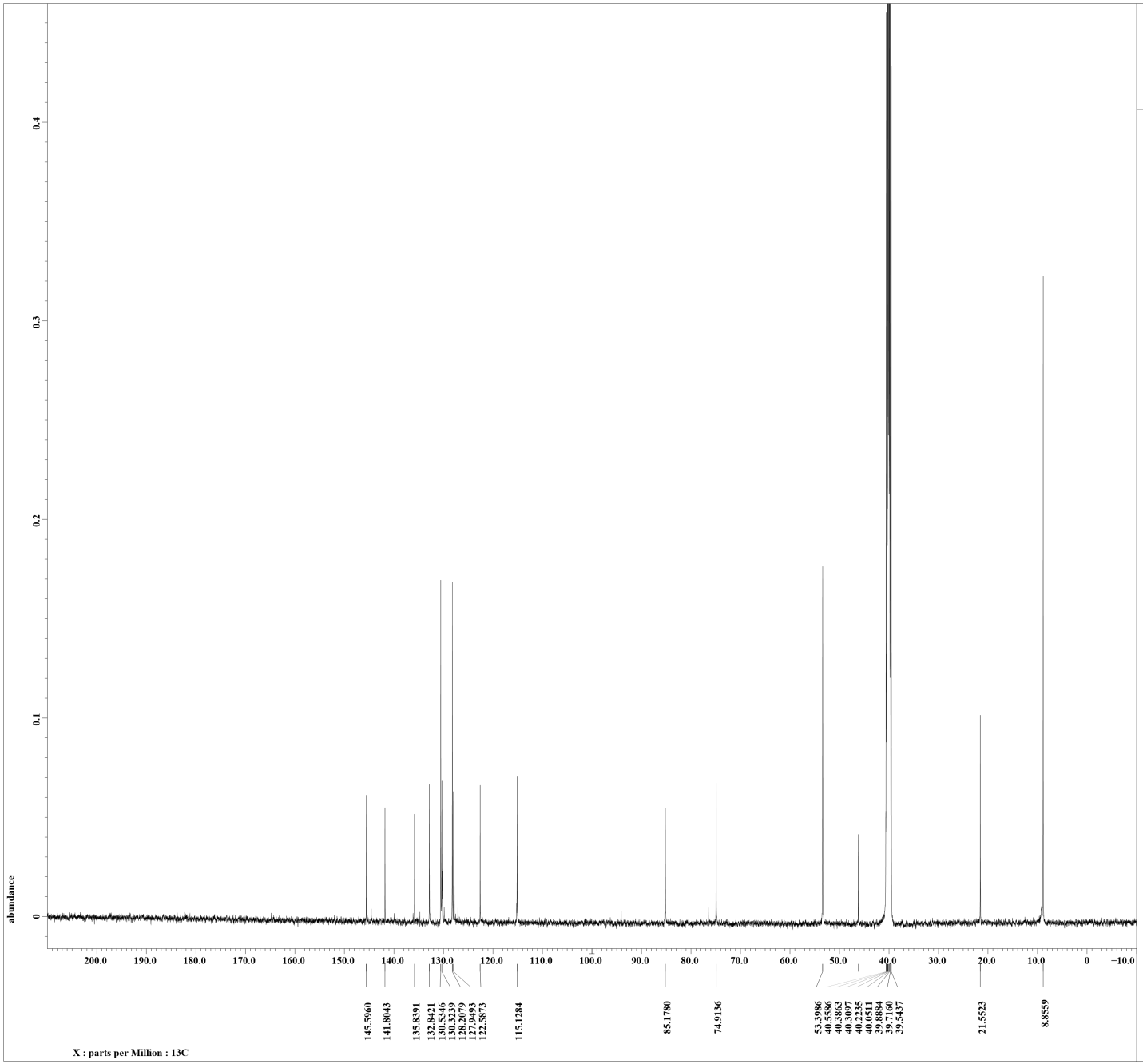
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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          = 21.2[dC]
```



2c

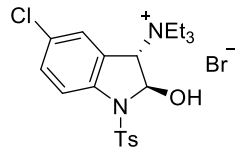



```
Filename          = yama-17-07-12-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#674438
Solvent           = DMSO-D6
Creation_time     = 13-JUL-2017 07:00:40
Revision_time     = 13-JUL-2017 07:50:48
Current_time      = 13-JUL-2017 07:52:55

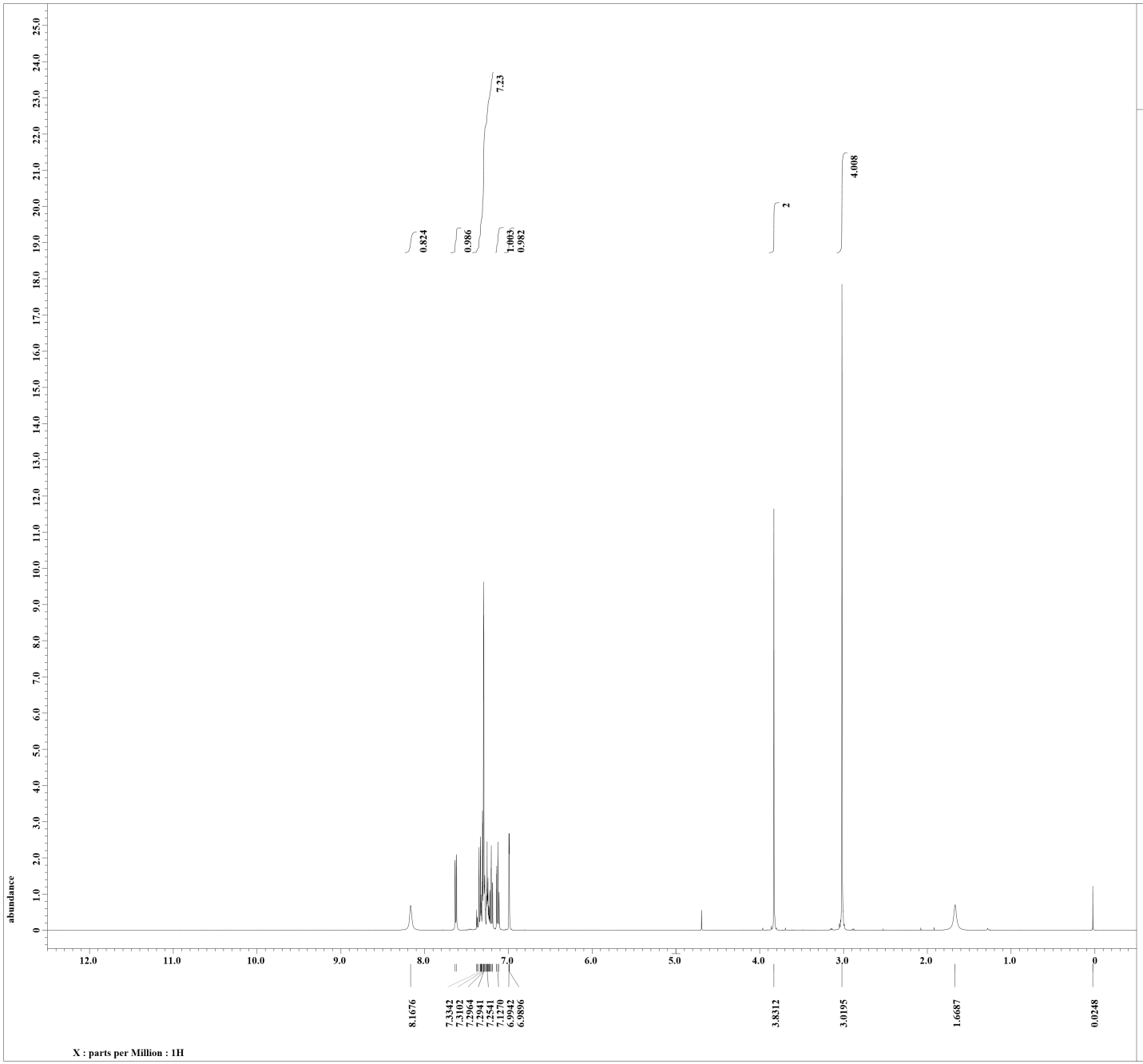
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16523
Total_scans       = 16523

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22.2[dC]
```



2c

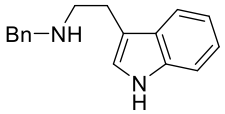



```
Filename          = yama-18-01-11-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#308785
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2018 08:17:40
Revision_time     = 11-JAN-2018 08:40:28
Current_time      = 11-JAN-2018 08:42:48

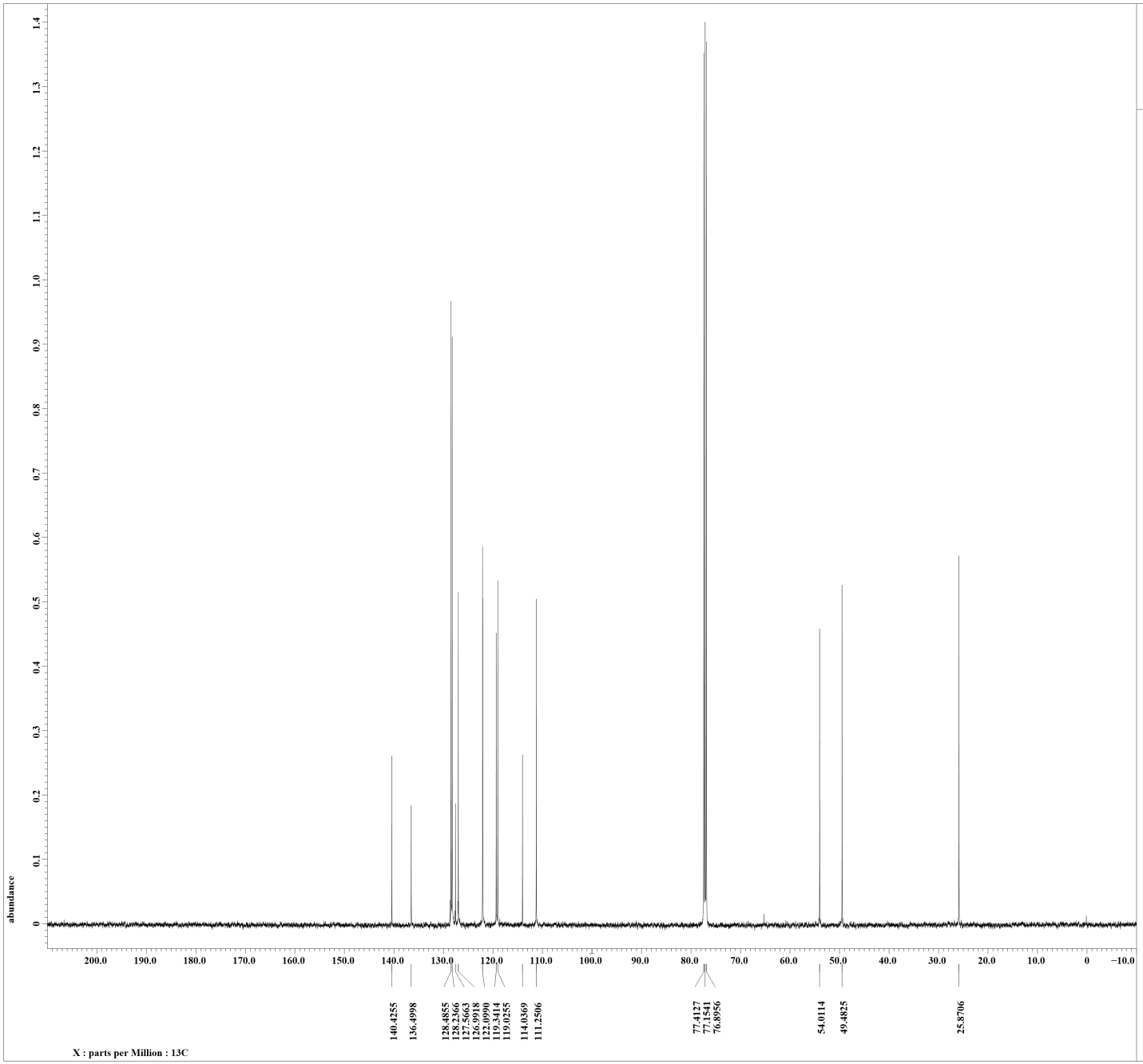
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.3[dC]
```



3a

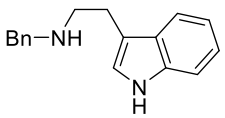



```
Filename          = yama-18-01-11-8.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#310945
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2018 10:26:28
Revision_time     = 11-JAN-2018 10:47:54
Current_time      = 11-JAN-2018 10:51:19

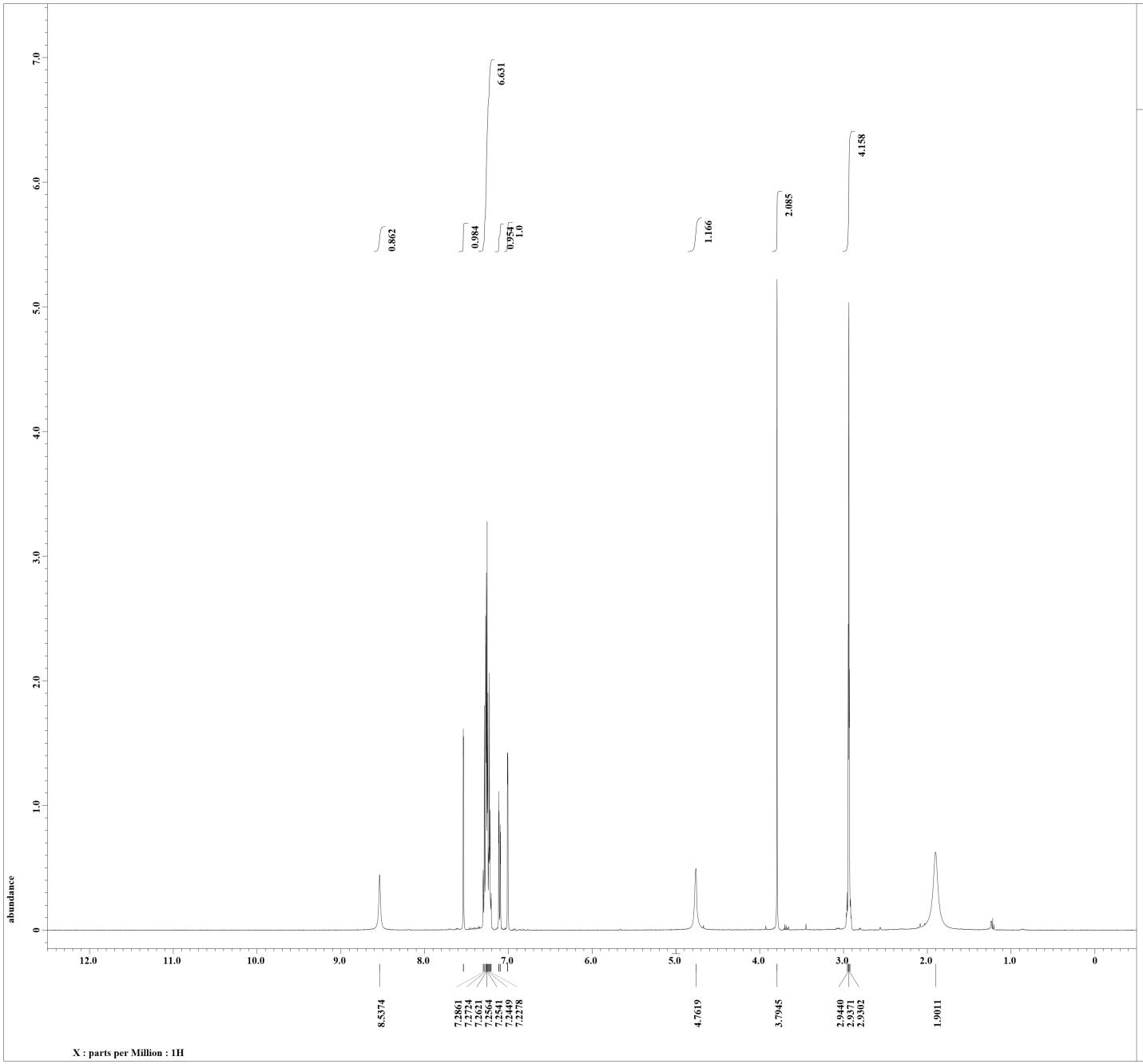
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 2670
Total_scans       = 2670

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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]
```



3a

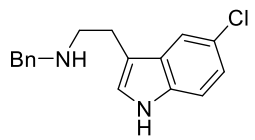



```
Filename          = yama-17-07-25-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#387542
Solvent           = CHLOROFORM-D
Creation_time     = 25-JUL-2017 09:55:32
Revision_time     = 25-JUL-2017 10:50:48
Current_time      = 25-JUL-2017 10:51:11

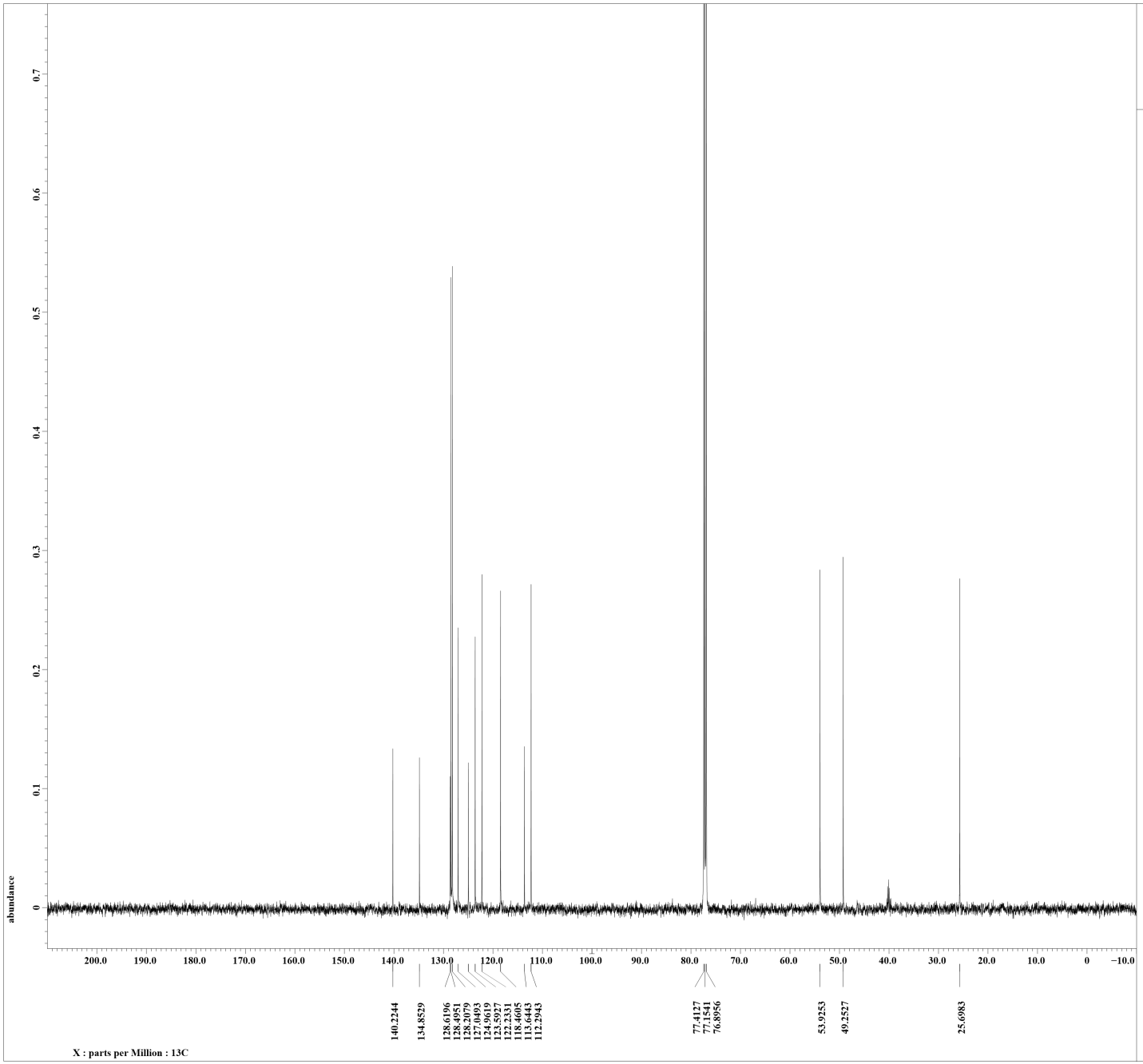
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 21.6[dC]
```



3b

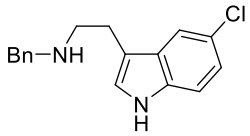



```
Filename          = yama-17-07-25-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#389270
Solvent           = CHLOROFORM-D
Creation_time     = 25-JUL-2017 11:33:48
Revision_time     = 25-JUL-2017 12:27:43
Current_time      = 25-JUL-2017 12:28:00

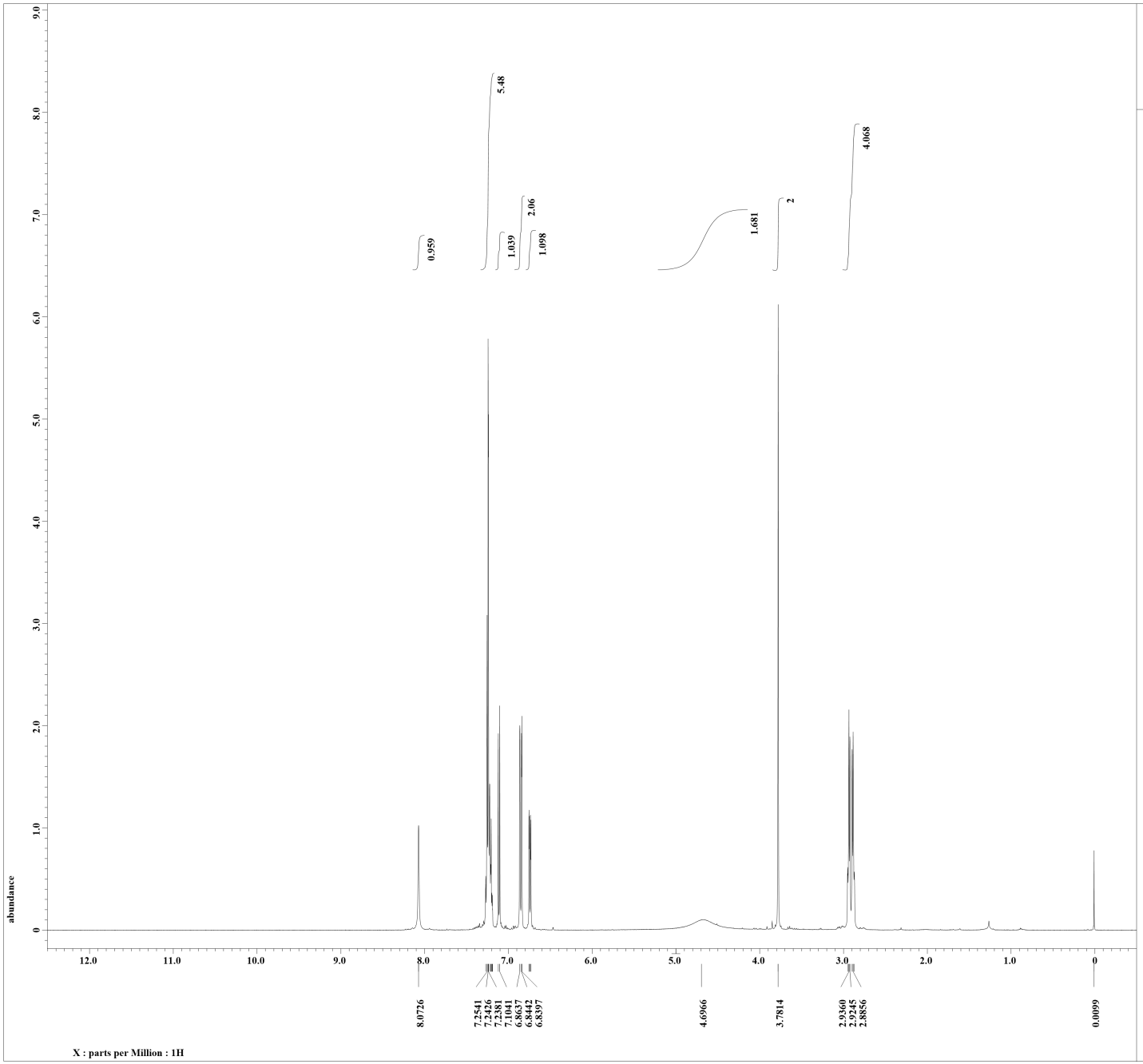
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 2038.0
Total_scans       = 2038.0

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22[dC]
```



3b

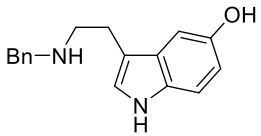



```
Filename          = yama-18-01-11-2-9.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#442394
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2018 12:00:07
Revision_time     = 11-JAN-2018 12:21:21
Current_time      = 11-JAN-2018 12:22:31

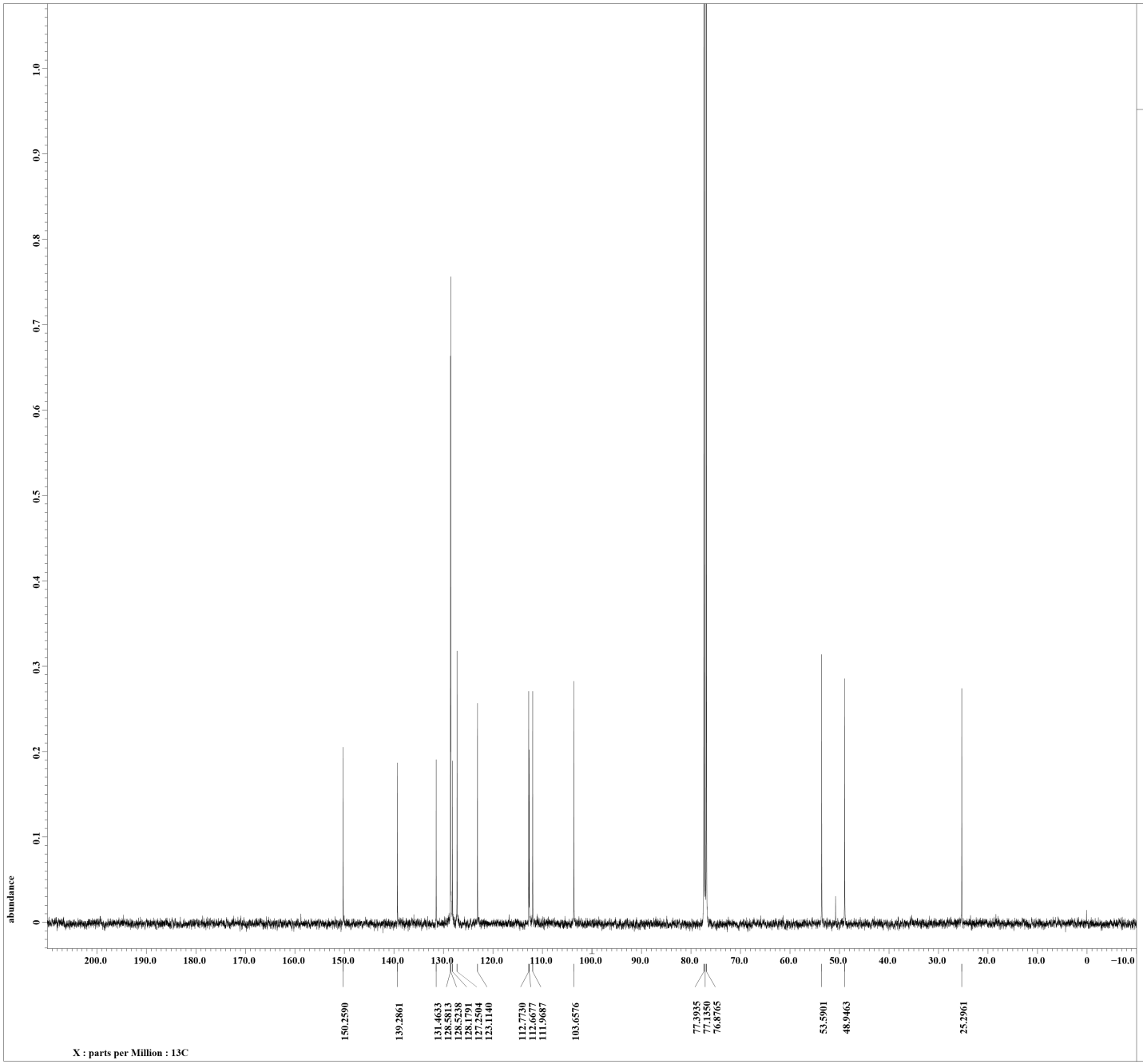
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.9[dC]
```



3c

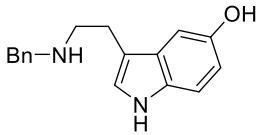



```
Filename          = yama-18-01-11-2-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#399659
Solvent           = CHLOROFORM-D
Creation_time     = 11-JAN-2018 11:54:28
Revision_time     = 11-JAN-2018 12:14:45
Current_time      = 11-JAN-2018 12:15:37

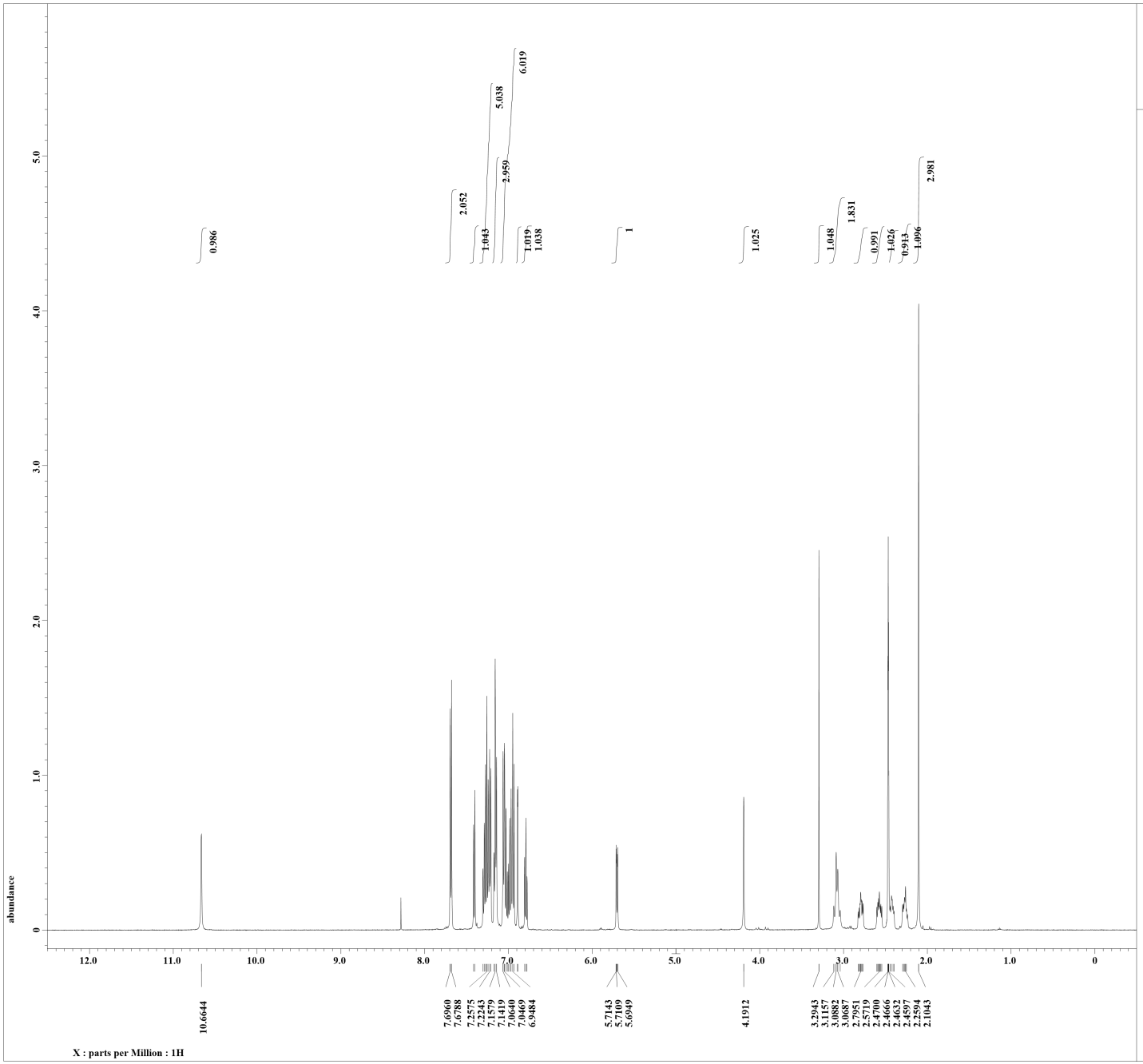
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 1398
Total_scans       = 1398

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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]
```



3c

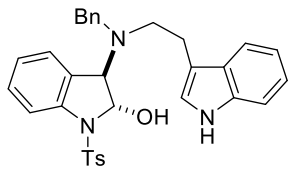



```
Filename          = yama-161.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#527567
Solvent           = DMSO-D6
Creation_time     = 27-JUL-2017 13:48:26
Revision_time     = 27-JUL-2017 14:47:00
Current_time      = 27-JUL-2017 14:47:44

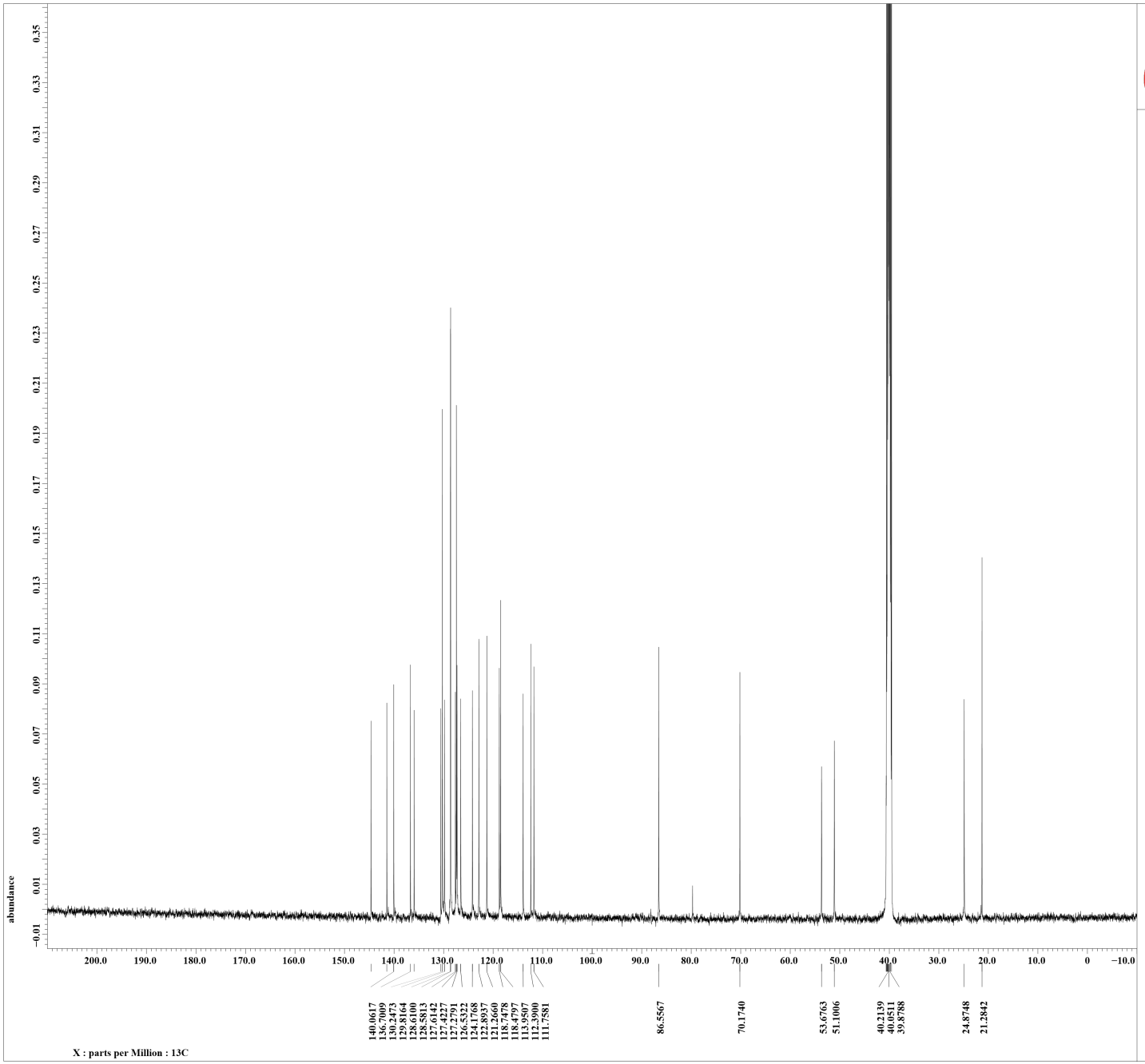
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 24.7[dC]
```



5a

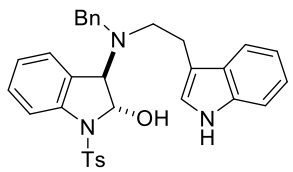



```
Filename          = yama-17-07-27-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#673164
Solvent           = DMSO-D6
Creation_time     = 28-JUL-2017 06:54:51
Revision_time     = 28-JUL-2017 07:48:20
Current_time      = 28-JUL-2017 07:48:54

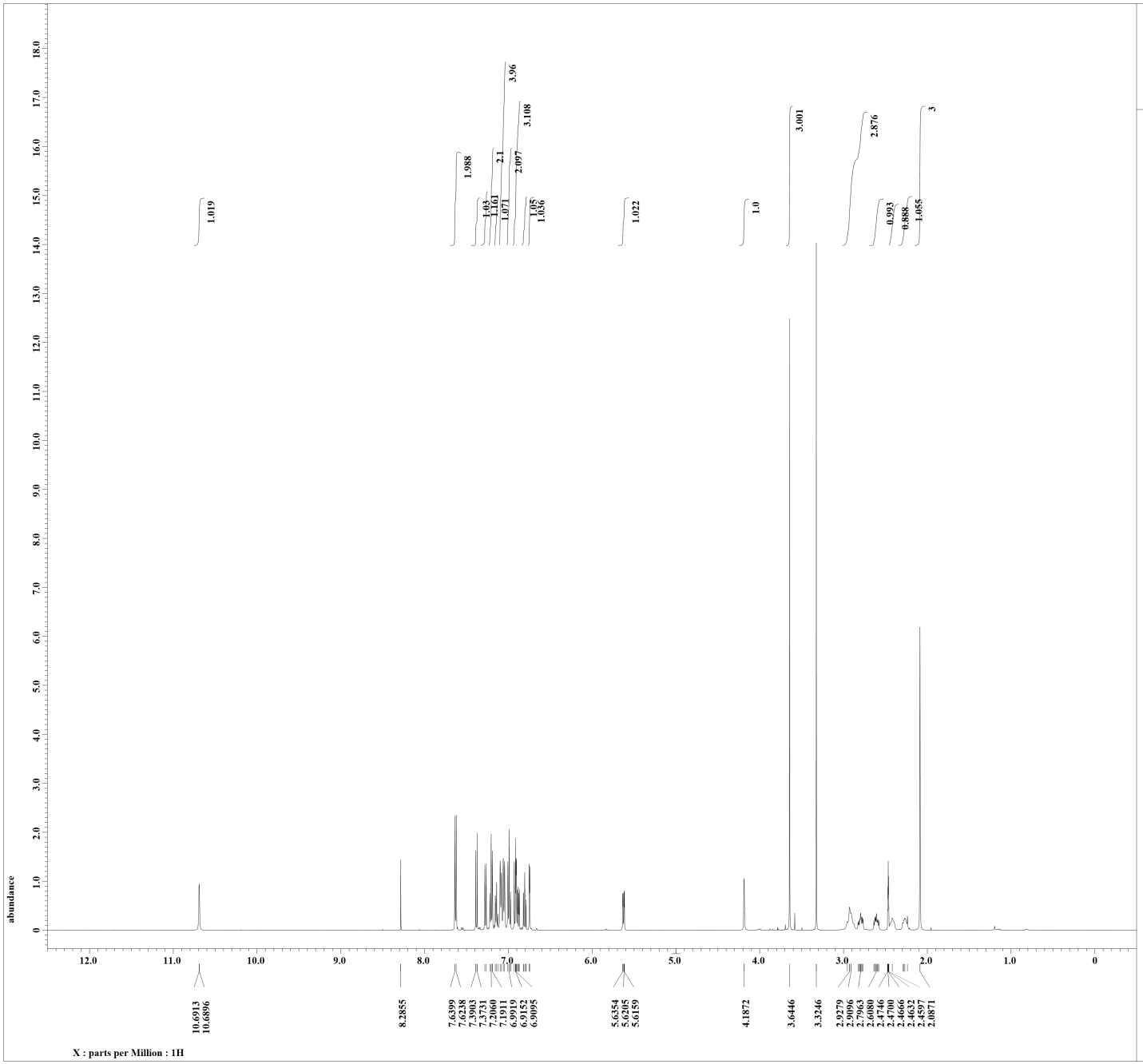
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16584
Total_scans       = 16584

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22.4[dC]
```



5a

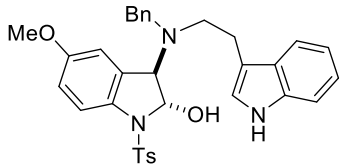



```
Filename          = yama-17-07-05-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = yama
Solvent           = DMSO-D6
Creation_time     =  5-JUL-2017 18:13:58
Revision_time     =  6-JUL-2017 07:54:57
Current_time      =  6-JUL-2017 07:55:30

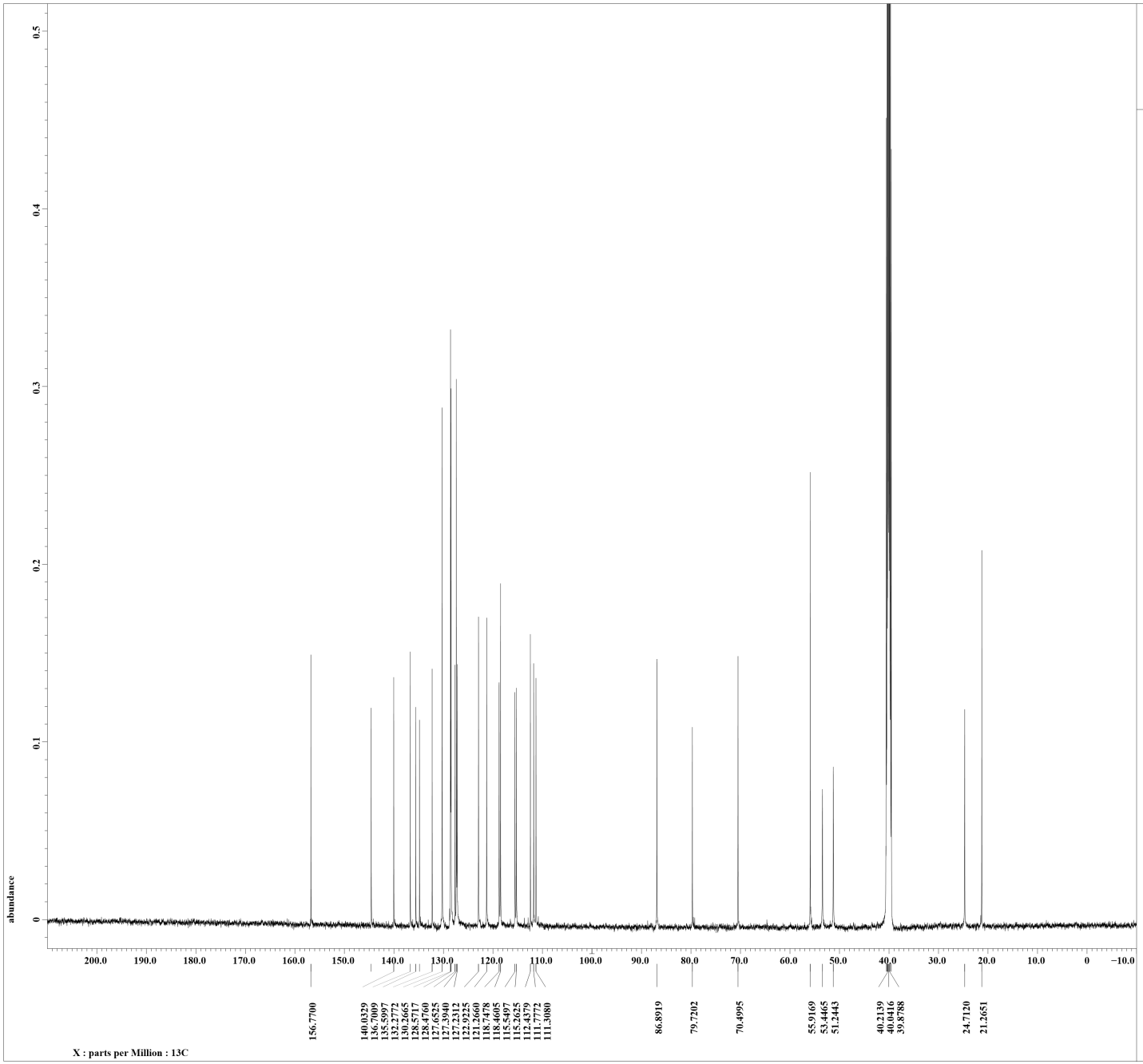
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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          = 21.7[dC]
```



5b

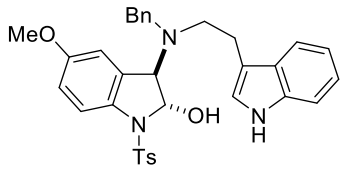



```
Filename          = yama-17-07-05-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = yama
Solvent           = DMSO-D6
Creation_time     =  6-JUL-2017 07:00:14
Revision_time     =  6-JUL-2017 07:47:55
Current_time      =  6-JUL-2017 07:48:23

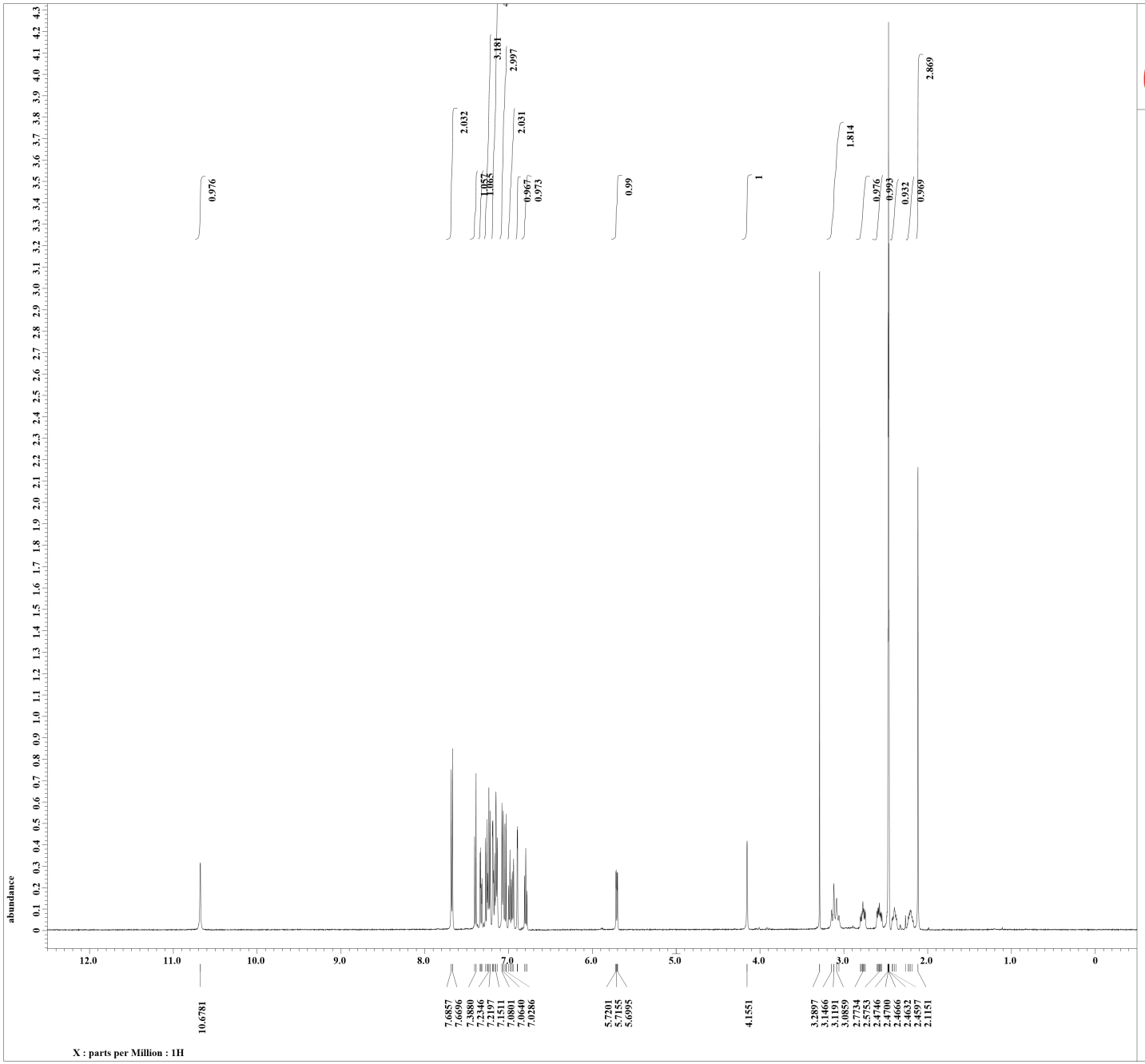
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16167
Total_scans       = 16167

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22.4[dC]
```



5b

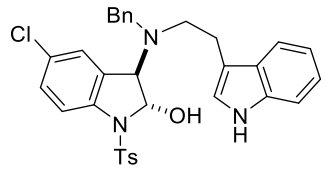



```
Filename          = yama-17-07-18-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#673415
Solvent           = DMSO-D6
Creation_time     = 18-JUL-2017 17:53:00
Revision_time     = 18-JUL-2017 18:47:37
Current_time      = 18-JUL-2017 18:48:00

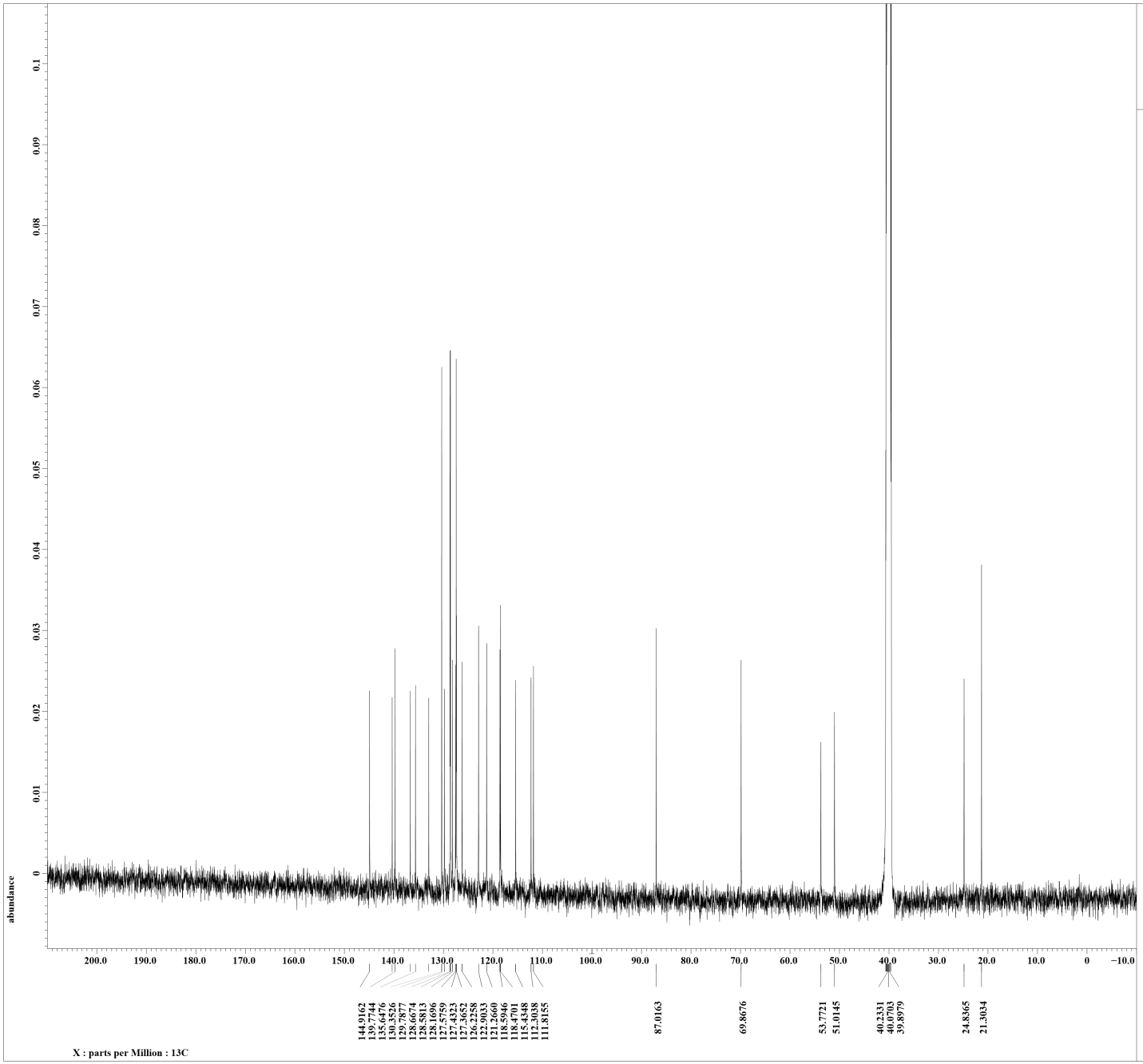
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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          = 23.5[dC]
```



5c

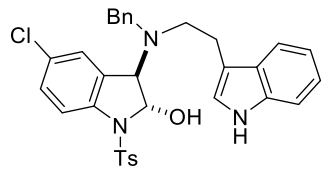



```
Filename          = yama-17-07-18-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#673823
Solvent           = DMSO-D6
Creation_time     = 19-JUL-2017 06:55:27
Revision_time     = 19-JUL-2017 07:48:54
Current_time      = 19-JUL-2017 07:49:36

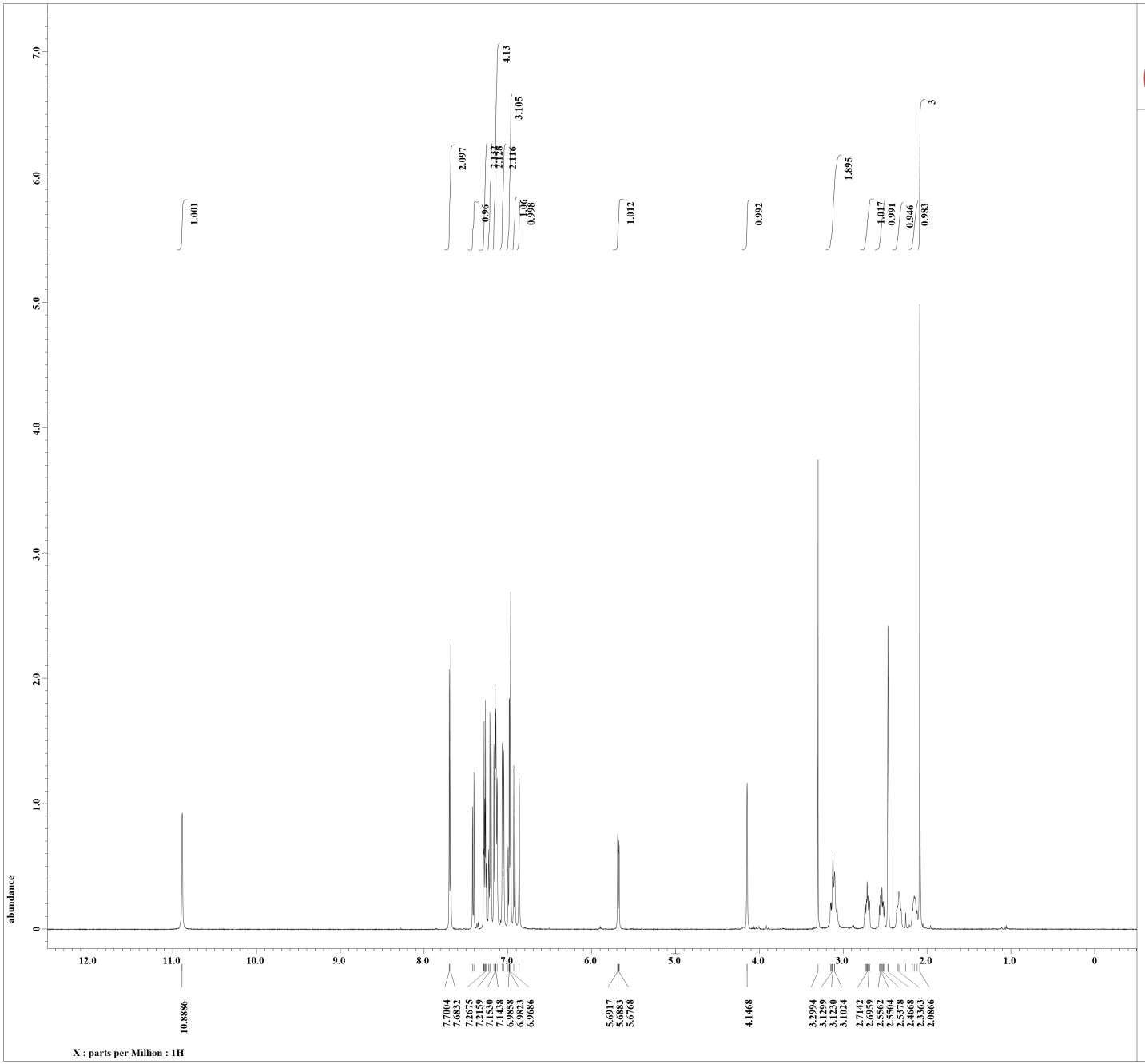
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16508
Total_scans       = 16508

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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]
```



5c

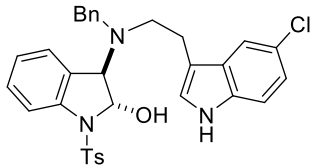



```
Filename          = HITAB-Bn-5Cl-serotoni
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#678872
Solvent           = DMSO-D6
Creation_time     = 26-JUL-2017 13:50:47
Revision_time     = 25-DEC-2017 15:23:30
Current_time      = 25-DEC-2017 15:23:58

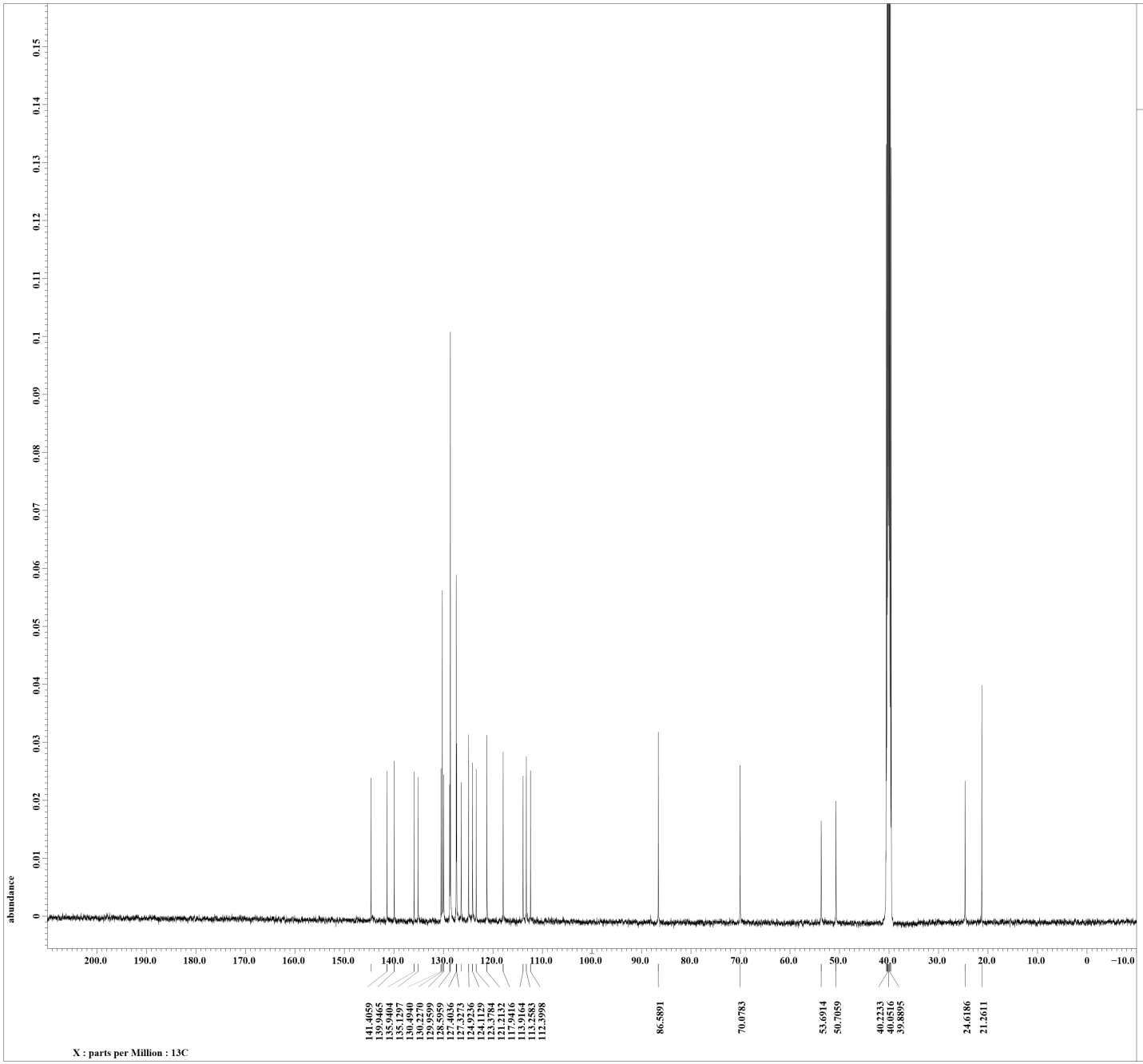
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 52
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22[dC]
```



5d

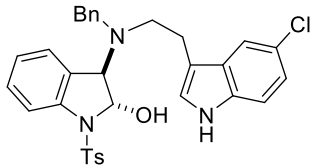



```
Filename          = HITAB-Bn-5Cl-serotoni
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#680238
Solvent           = DMSO-D6
Creation_time     = 27-JUL-2017 02:43:56
Revision_time     = 25-DEC-2017 15:26:52
Current_time      = 25-DEC-2017 15:27:34

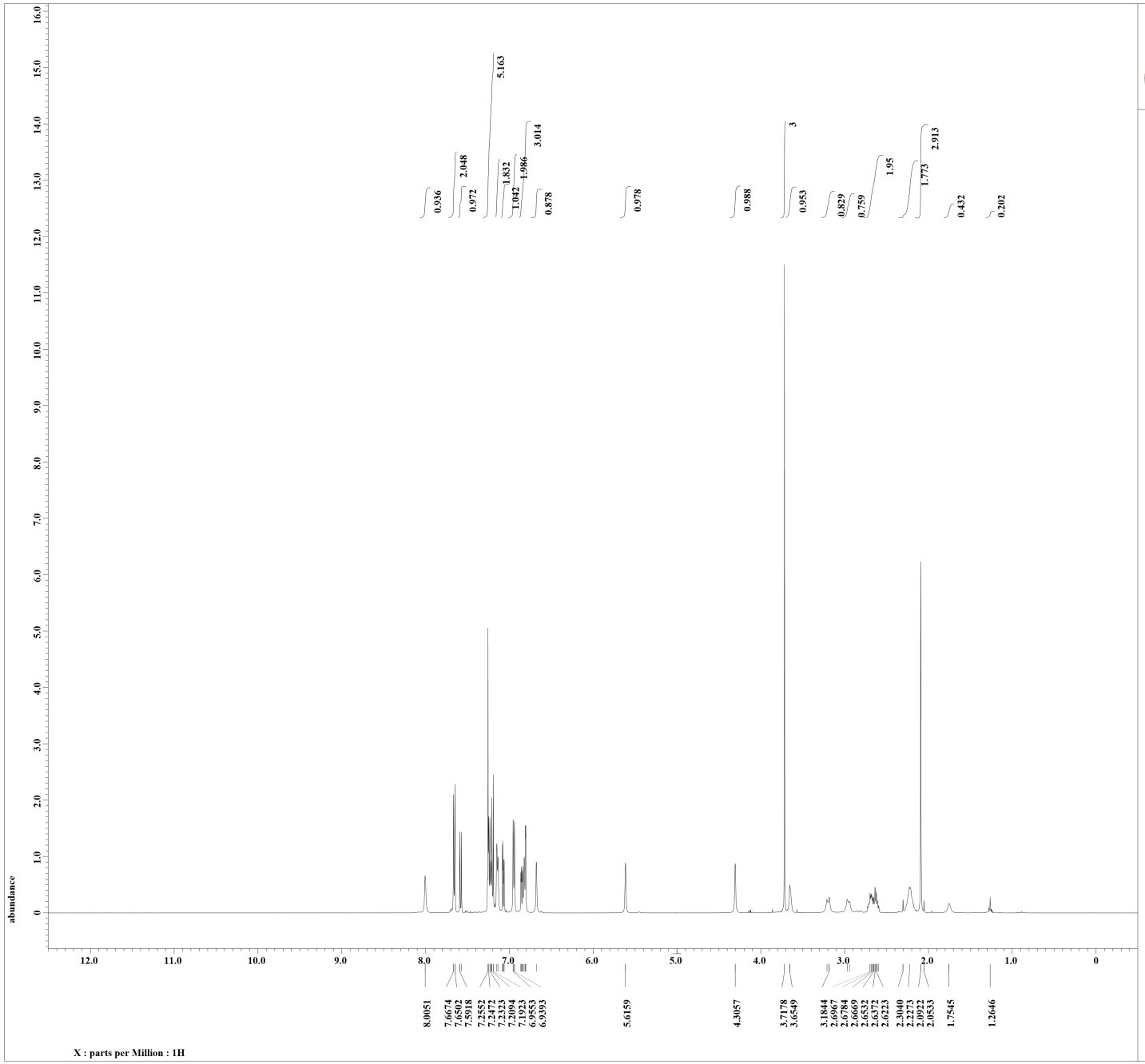
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 16341
Total_scans       = 16341

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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          = 22.5[dC]
```



5d

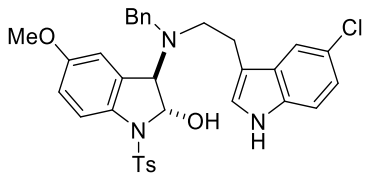



```
Filename          = 5-MeO-HITAB-5-Cl-sero
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#806963
Solvent           = CHLOROFORM-D
Creation_time     = 26-JUL-2017 21:34:05
Revision_time     = 25-DEC-2017 15:36:58
Current_time      = 25-DEC-2017 15:37:21

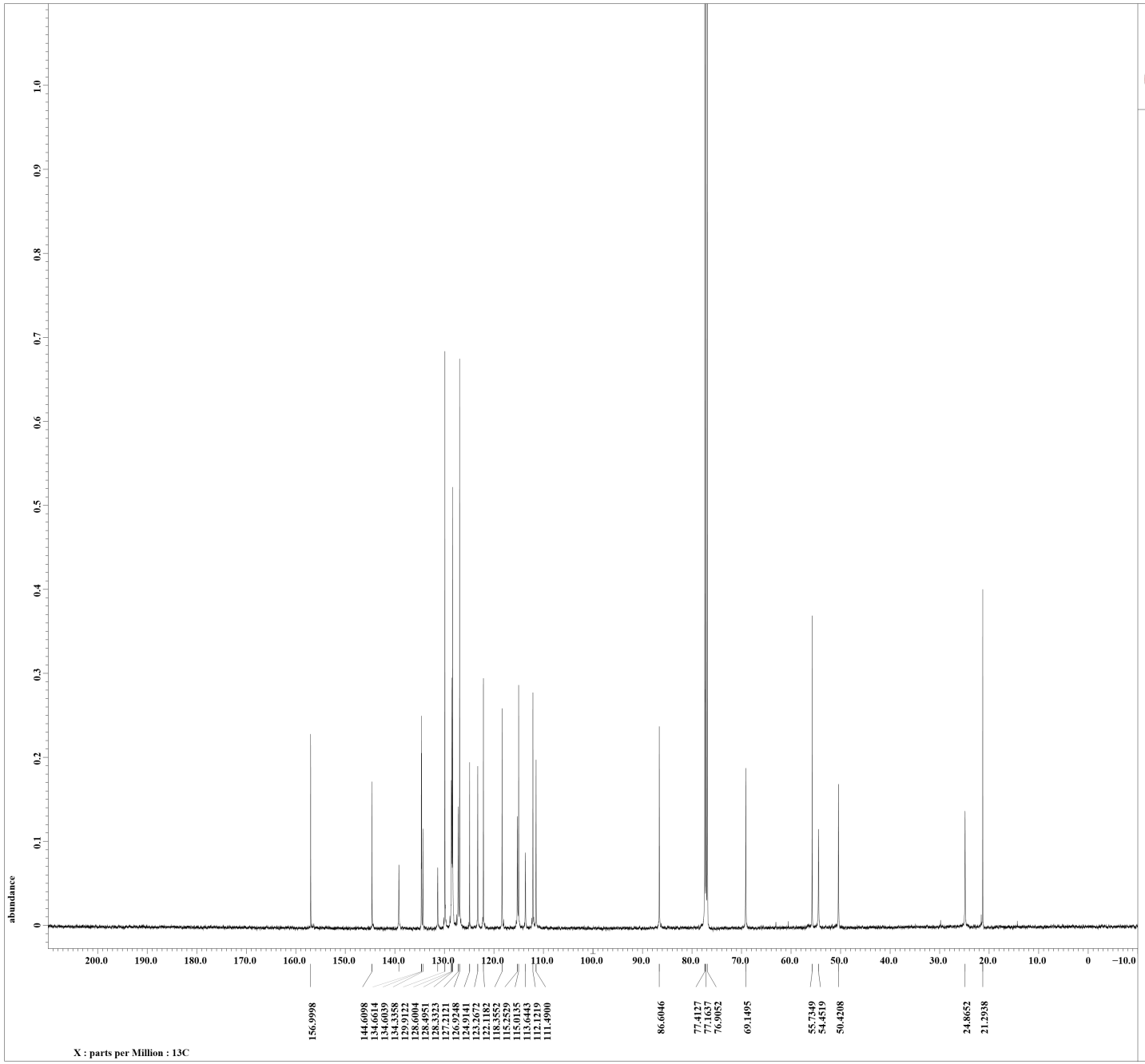
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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          = 20.8[dC]
```



5e

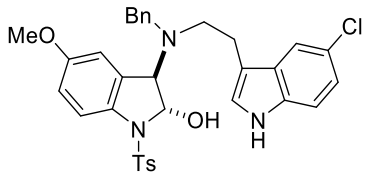



```
Filename          = 5-MeO-HITAB-5-Cl-sero
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#808598
Solvent           = CHLOROFORM-D
Creation_time     = 27-JUL-2017 07:36:35
Revision_time     = 26-DEC-2017 09:36:51
Current_time      = 26-DEC-2017 09:38:17

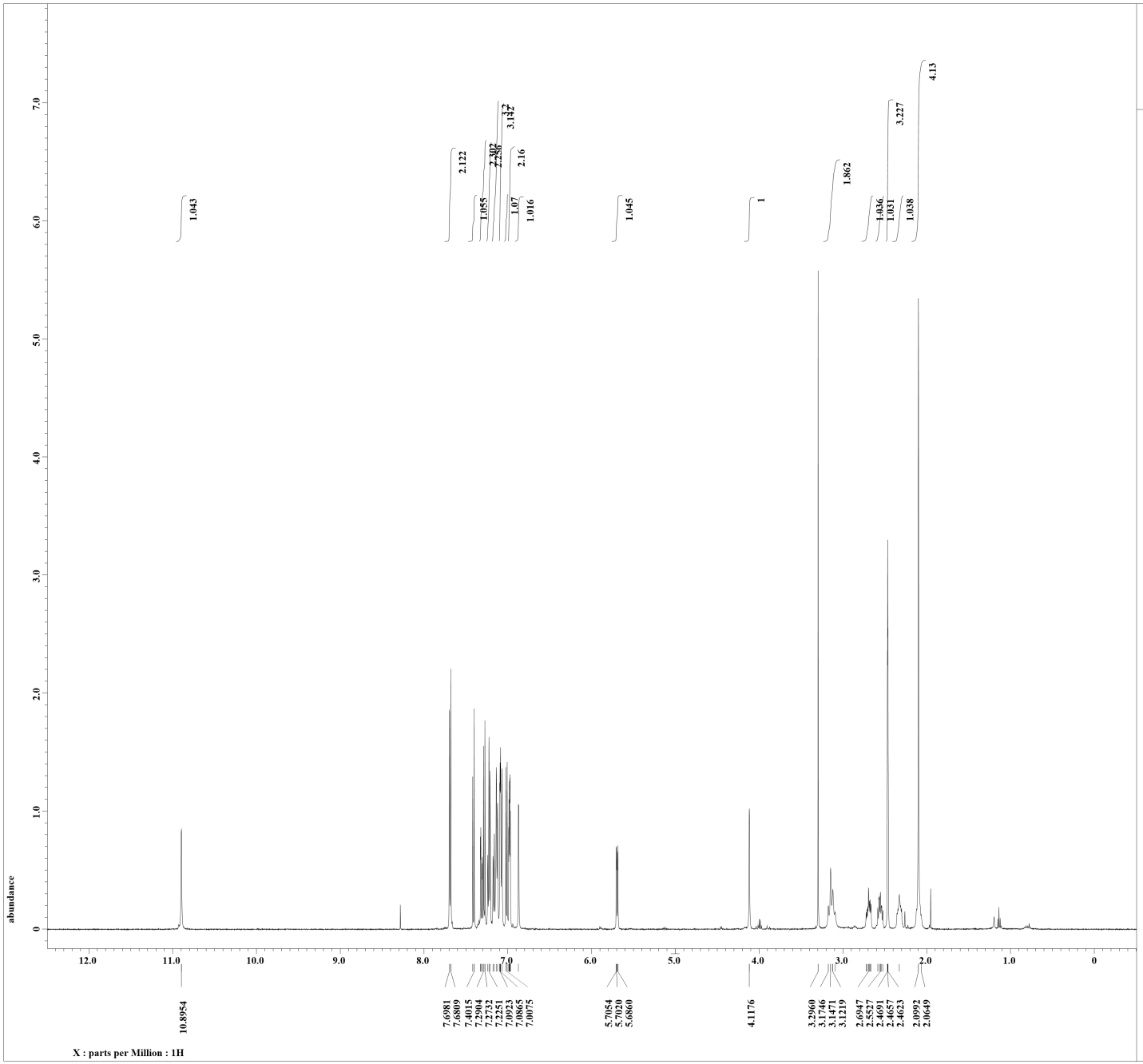
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 12692
Total_scans       = 12692

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22.5[dC]
```



5e

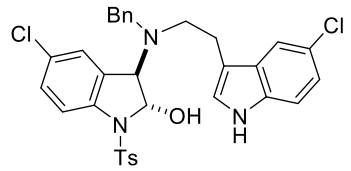


Filename = 5-Cl-HITAB-Bn-5-Cl-se
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#580911
Solvent = DMSO-D6
Creation_time = 5-AUG-2017 11:07:13
Revision_time = 25-DEC-2017 15:42:42
Current_time = 25-DEC-2017 15:43:18

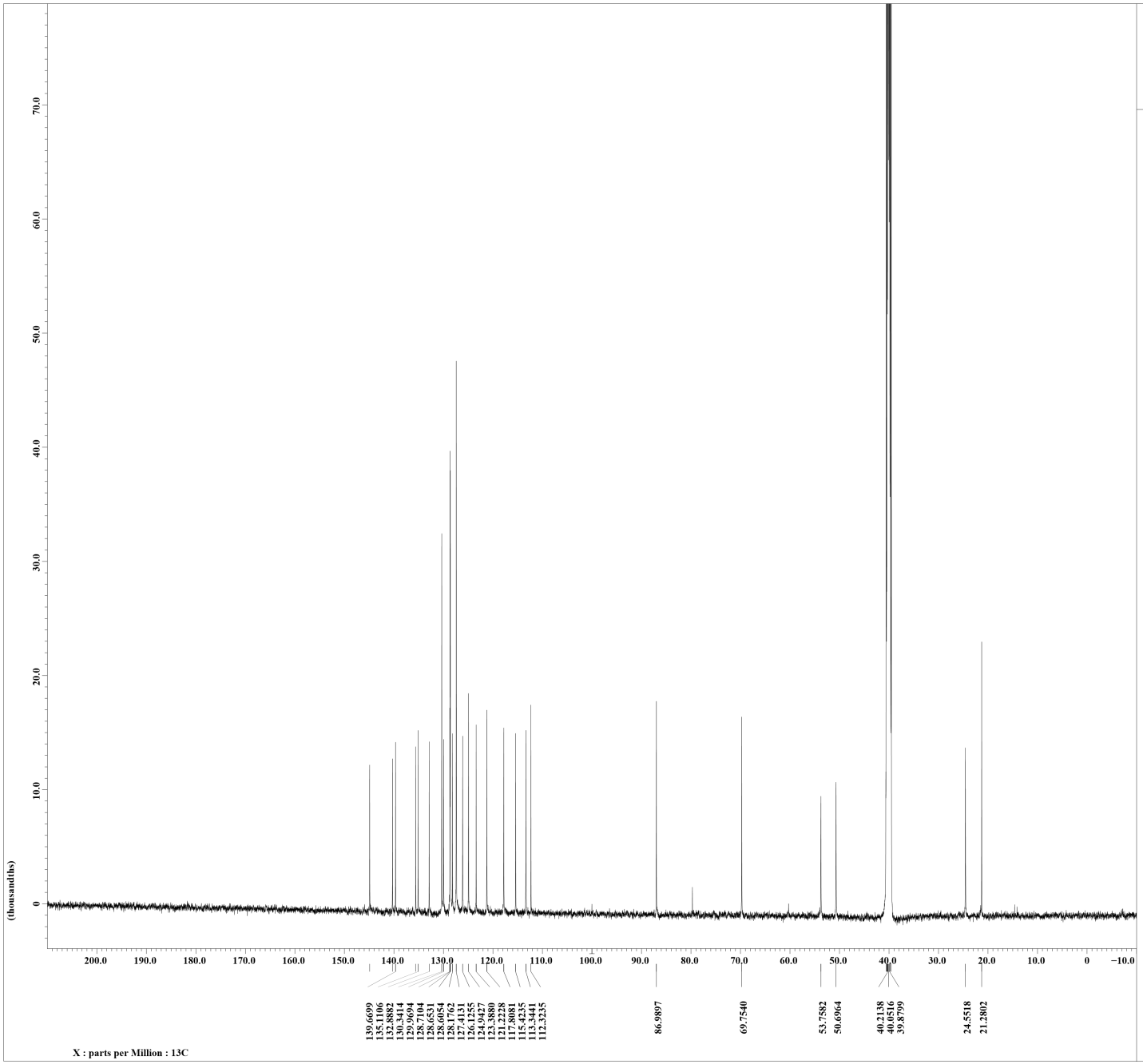
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 11.8[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3.4[dB]
X_pulse = 5.9[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 54
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 22.1[dC]



5f

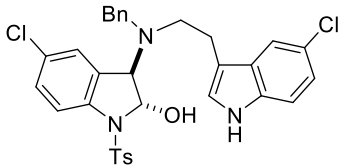



```
Filename          = 5-Cl-HITAB-Bn-5-Cl-se
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#693285
Solvent           = DMSO-D6
Creation_time     =  7-AUG-2017 04:04:31
Revision_time     = 26-DEC-2017 09:41:49
Current_time      = 26-DEC-2017 09:42:28

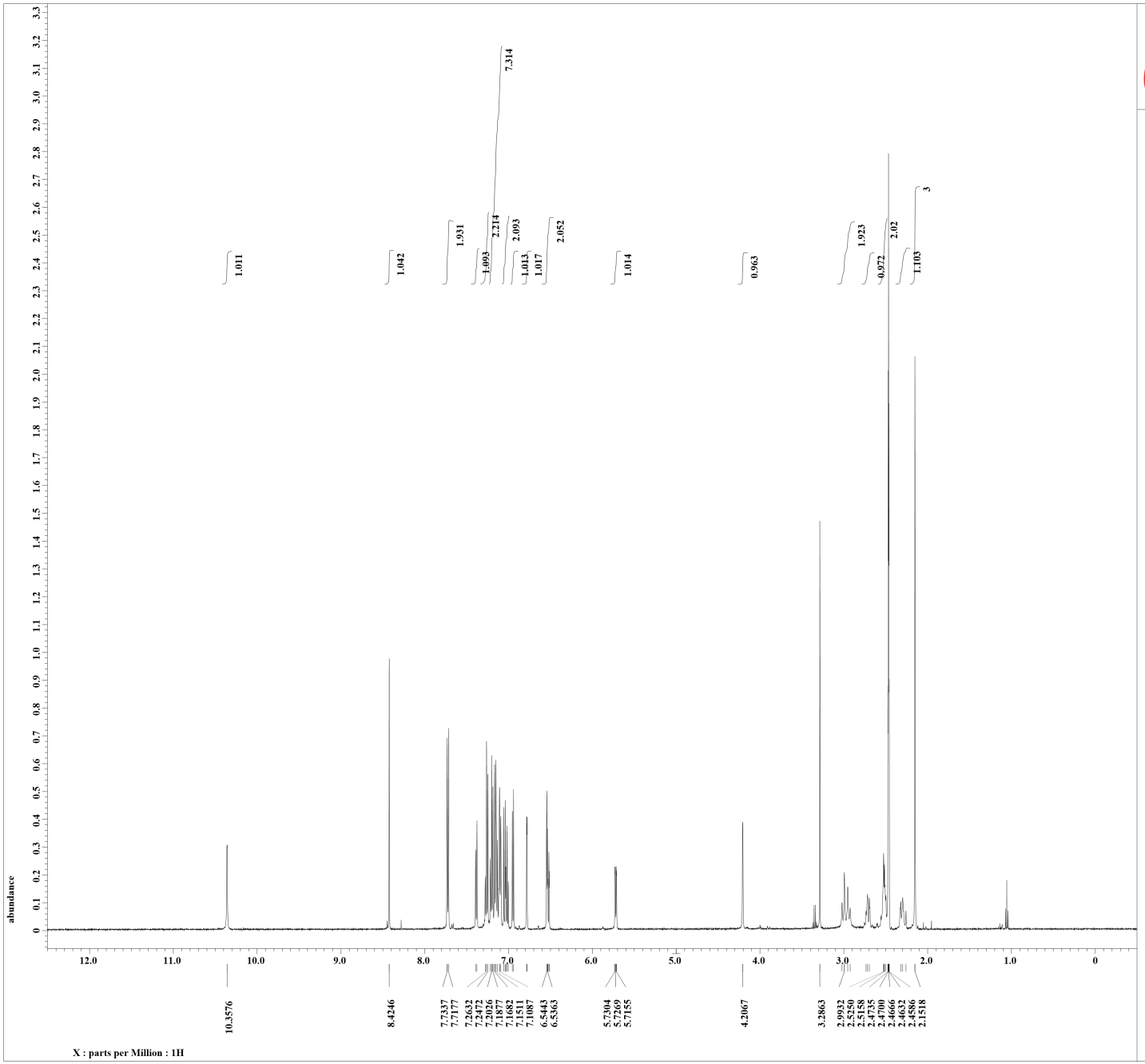
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 48087
Total_scans       = 48087

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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          = 22.8[dC]
```



5f

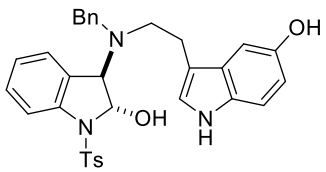


Filename = yama-18-01-10-10.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#319579
Solvent = DMSO-D6
Creation_time = 10-JAN-2018 08:35:38
Revision_time = 10-JAN-2018 10:41:30
Current_time = 10-JAN-2018 10:42:41

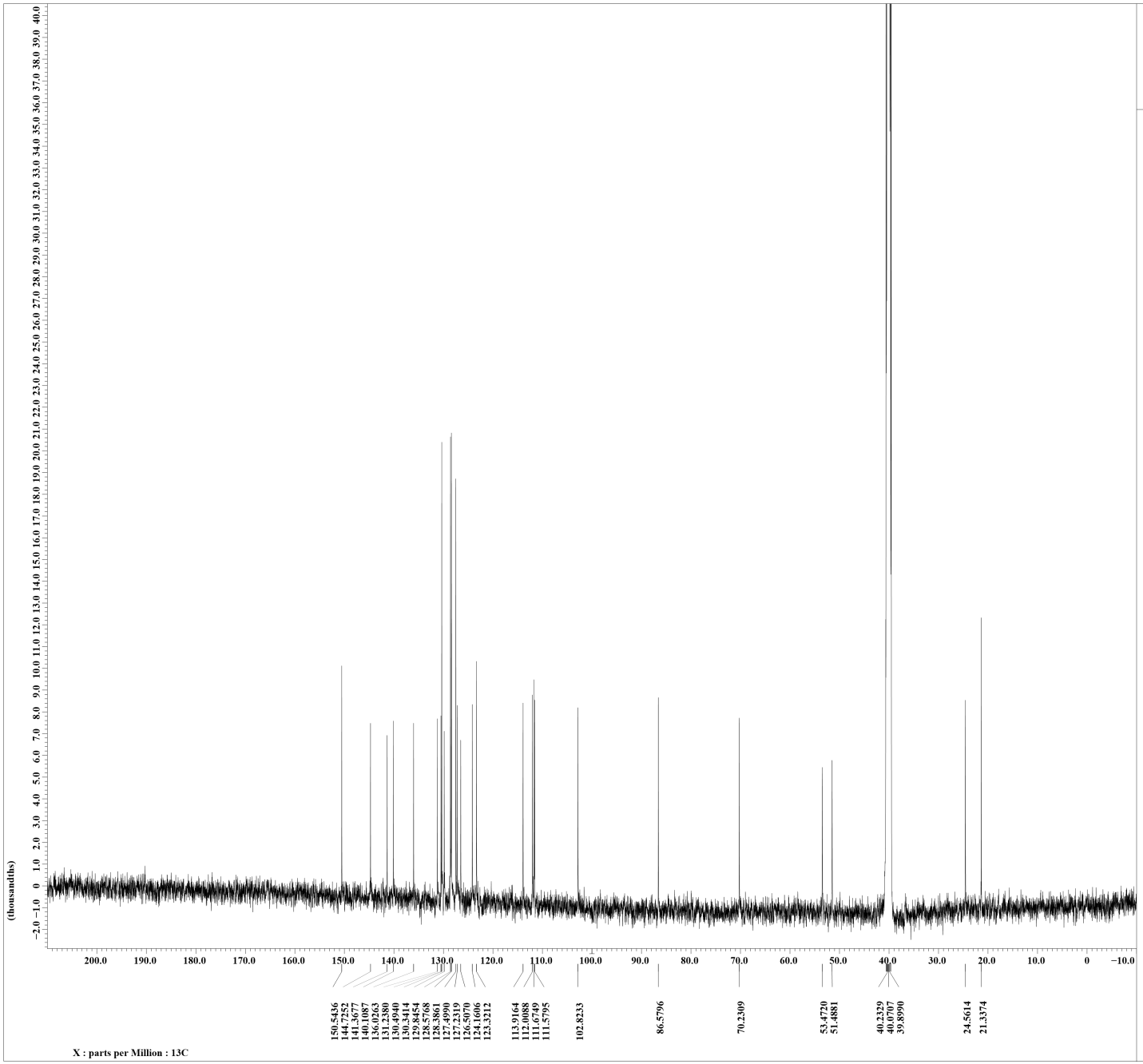
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

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 = 12.63[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.315[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 = 23.8[dC]



5g

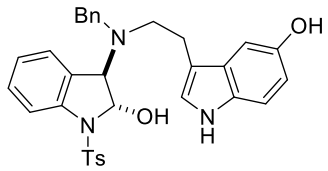



```
Filename          = yama-18-01-10-8.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#666751
Solvent           = DMSO-D6
Creation_time     =  3-JAN-2018 21:20:07
Revision_time     = 11-JAN-2018 07:37:15
Current_time      = 11-JAN-2018 07:38:25

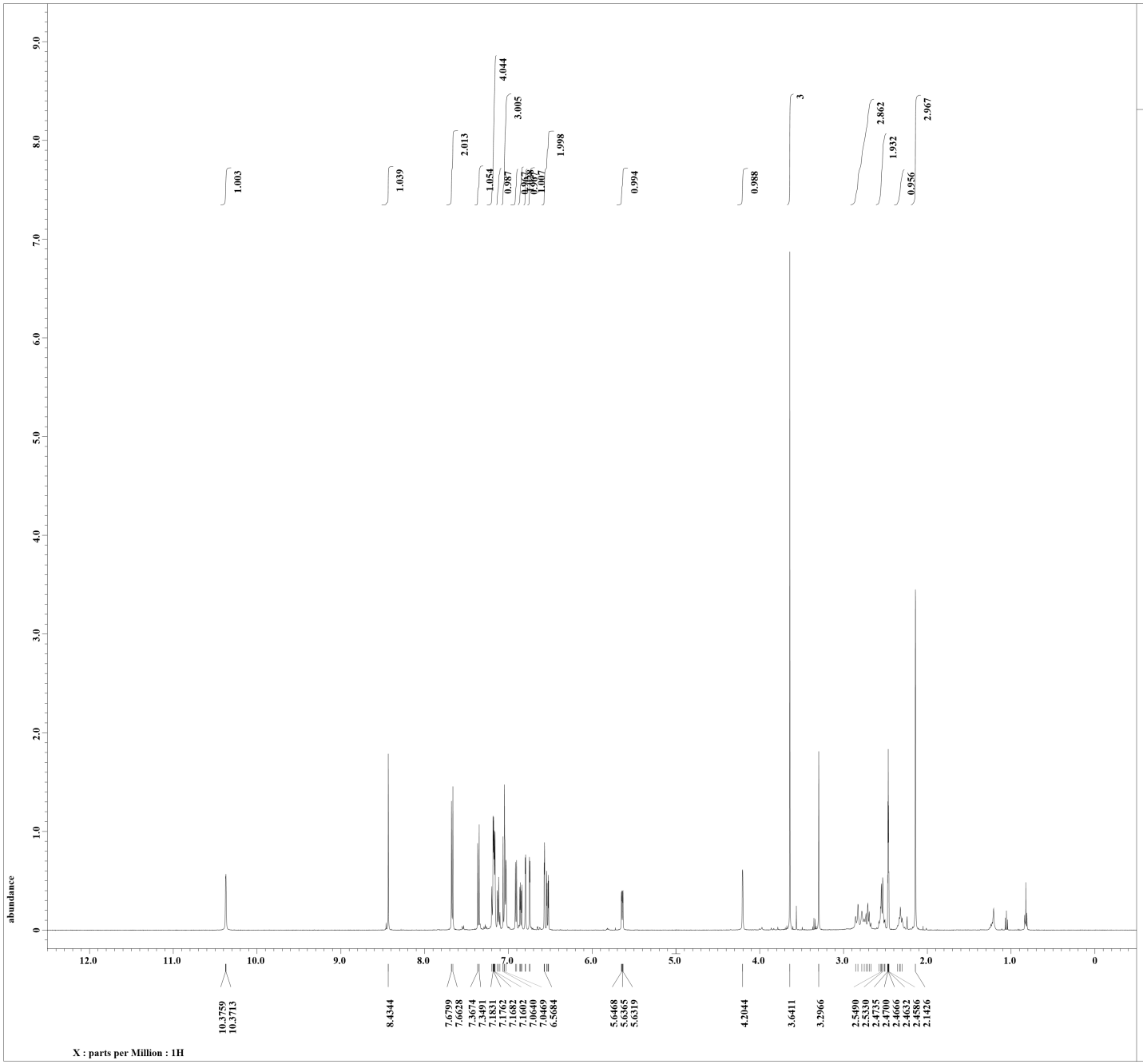
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 16231
Total_scans       = 16231

X_90_width        = 11.43[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 3.81[us]
Irr_atn_dec       = 20.81[dB]
Irr_atn_noe       = 20.81[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]
```



5g

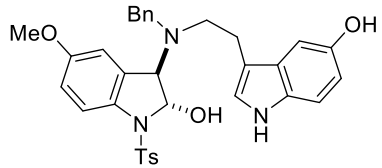


Filename = yama-18-01-10-2-9.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#531282
Solvent = DMSO-D6
Creation_time = 10-JAN-2018 14:28:22
Revision_time = 10-JAN-2018 14:57:52
Current_time = 10-JAN-2018 14:58:46

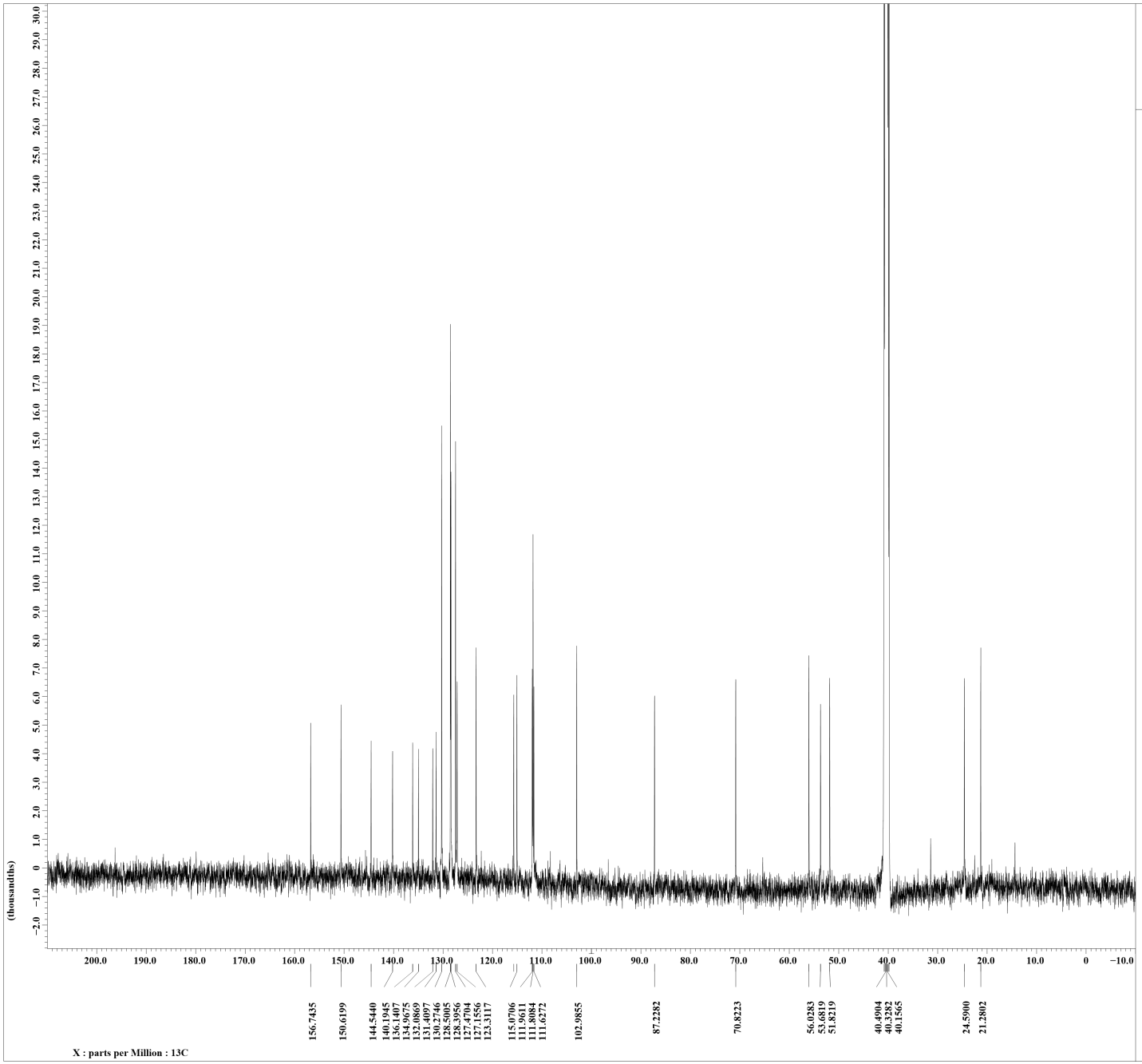
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

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 = 12.63[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.315[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.8[dC]



5h

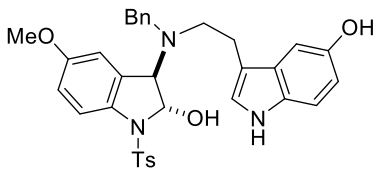



```
Filename          = 5-MeO-HITAB-Bn-seroto
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#619743
Solvent           = DMSO-D6
Creation_time     = 25-JUL-2017 03:32:52
Revision_time     = 25-DEC-2017 15:57:19
Current_time      = 25-DEC-2017 15:58:15

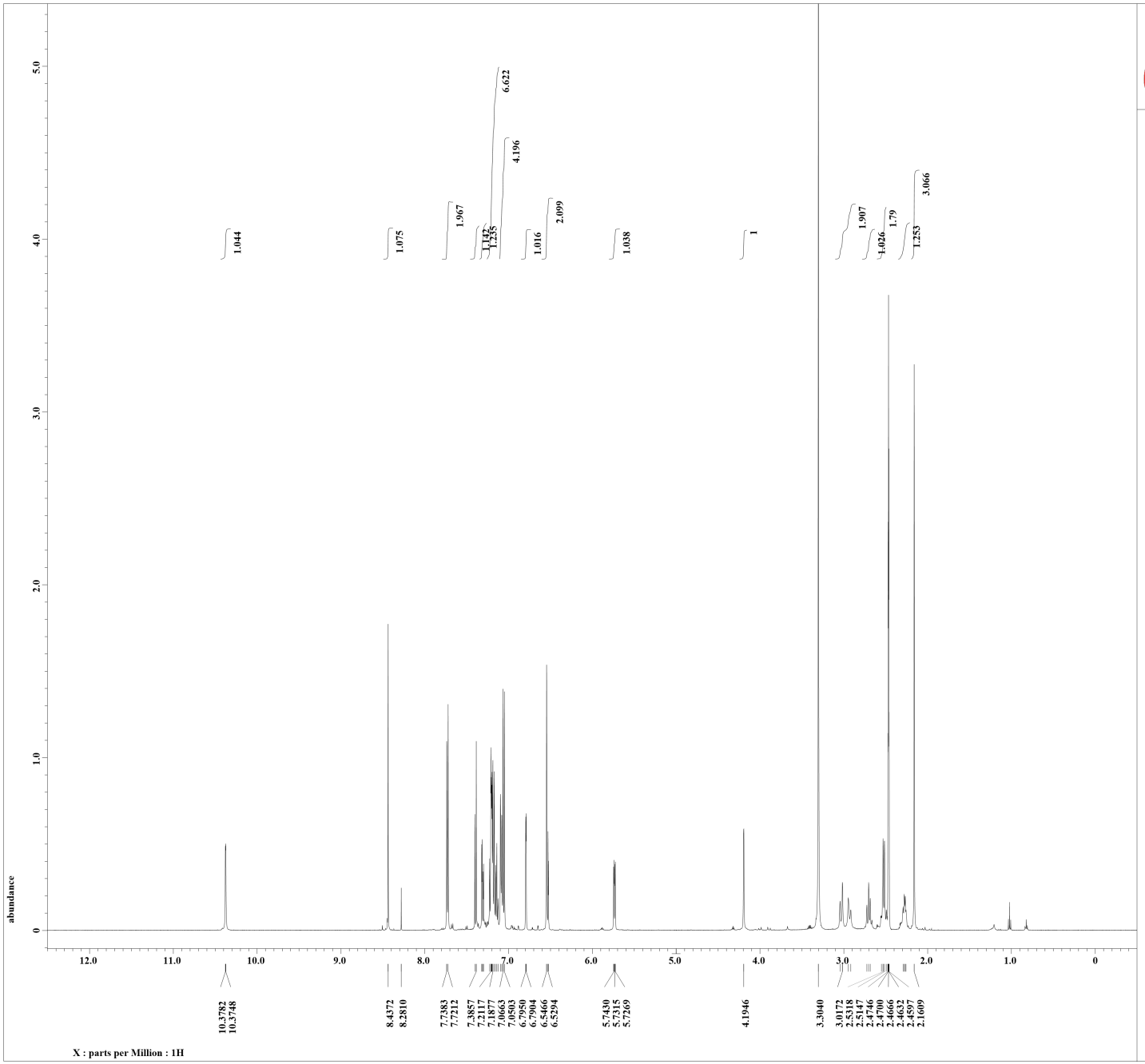
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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
Incomplete_copy   = TRUE
Mod_return        = 1
Scans             = 19510
Total_scans       = 19510

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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          = 60.2[dC]
```



5h

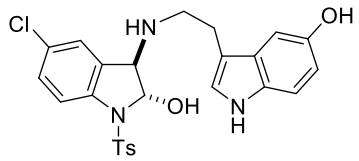


Filename = yama-17-07-19-7.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = yama
Solvent = DMSO-D6
Creation_time = 19-JUL-2017 17:46:28
Revision_time = 20-JUL-2017 07:51:22
Current_time = 20-JUL-2017 07:52:21

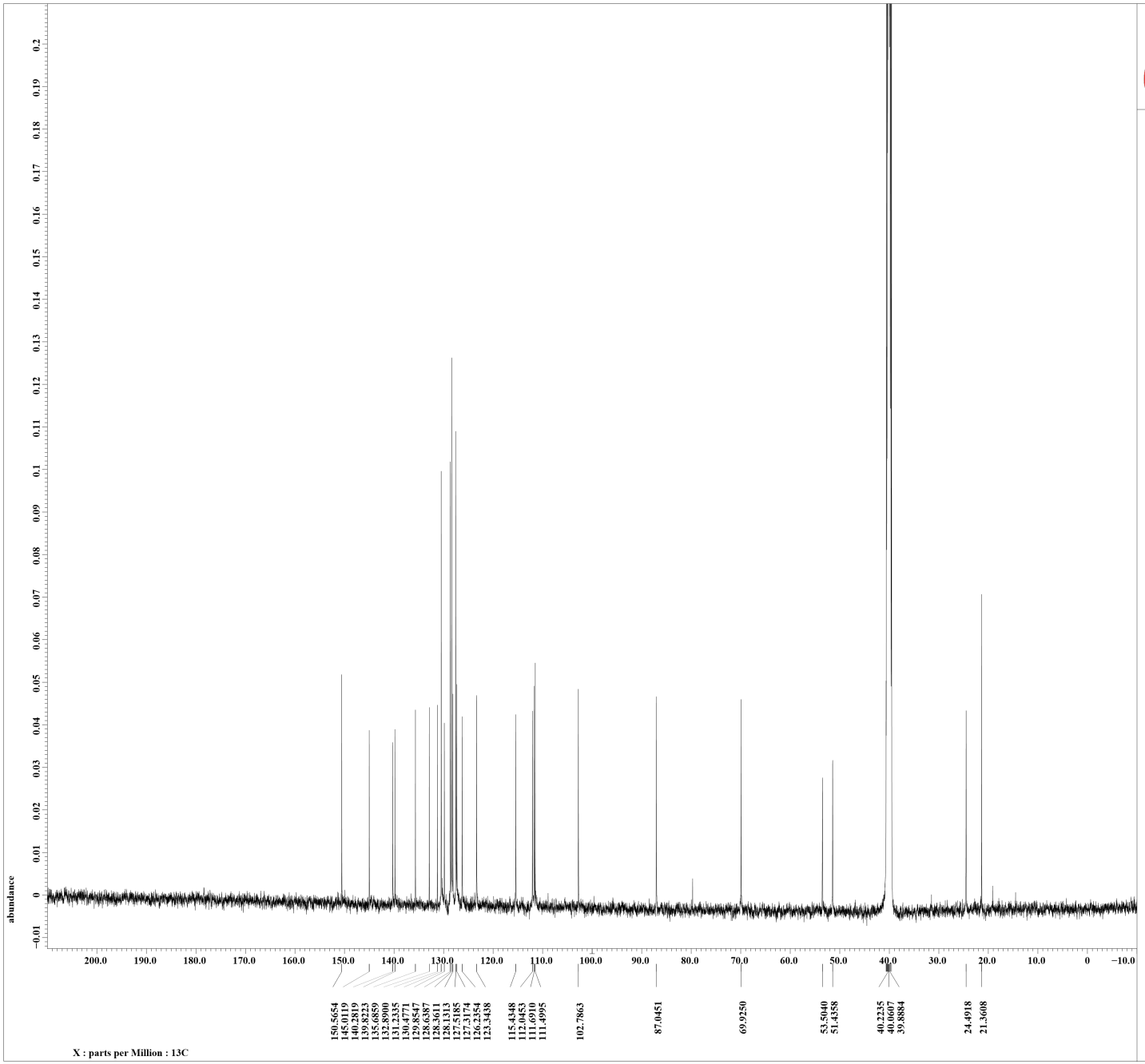
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

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 = 32
Total_scans = 32

X_90_width = 12.7[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.6[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 23.3[dC]



5i

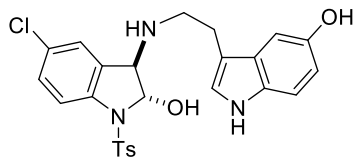



```
Filename          = yama-17-07-19-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = yama
Solvent           = DMSO-D6
Creation_time     = 20-JUL-2017 06:54:43
Revision_time     = 20-JUL-2017 07:48:11
Current_time      = 20-JUL-2017 07:48:38

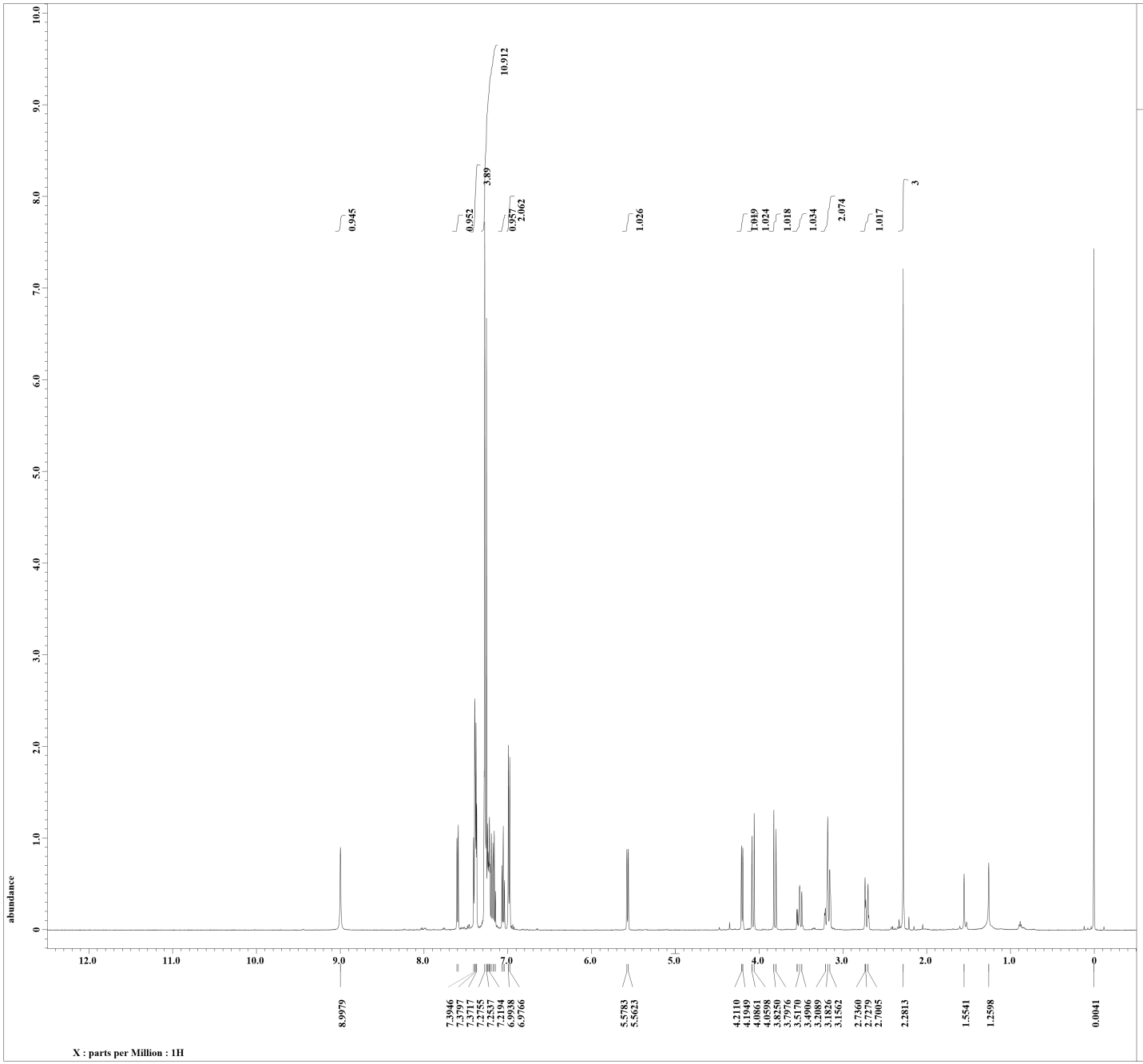
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16632
Total_scans       = 16632

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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]
```



5i

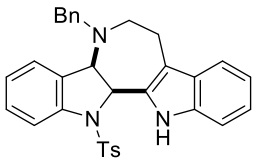



```
Filename          = yama131209-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#616522
Solvent           = CHLOROFORM-D
Creation_time     =  9-DEC-2013 17:09:10
Revision_time     = 25-DEC-2017 10:05:48
Current_time      = 25-DEC-2017 10:06:27

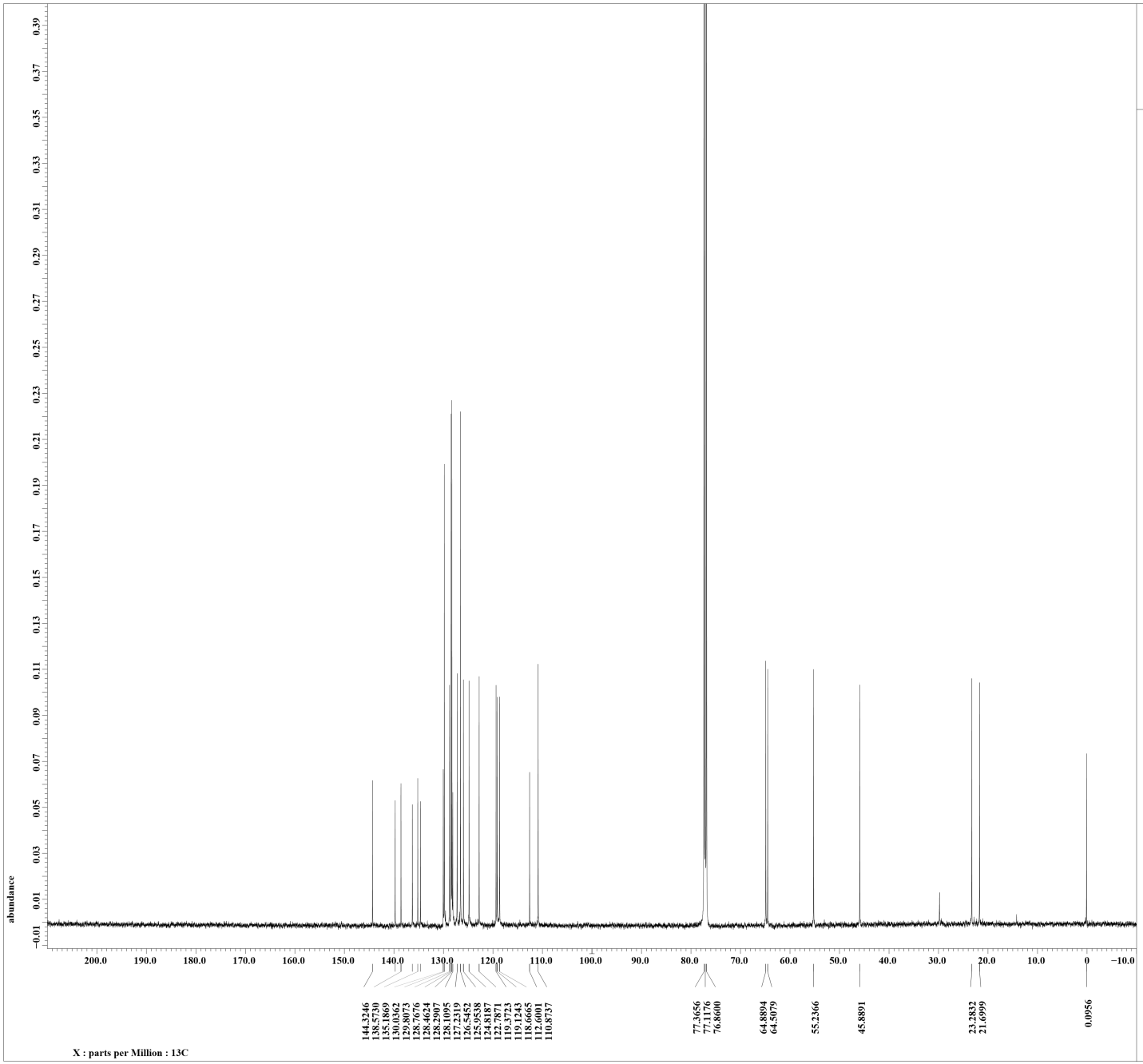
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 12.5[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6.25[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.6[dC]
```



6a

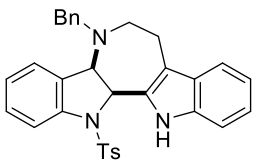



```
Filename          = yama131209-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#619788
Solvent           = CHLOROFORM-D
Creation_time     = 10-DEC-2013 07:06:56
Revision_time     = 25-DEC-2017 10:08:18
Current_time      = 25-DEC-2017 10:08:51

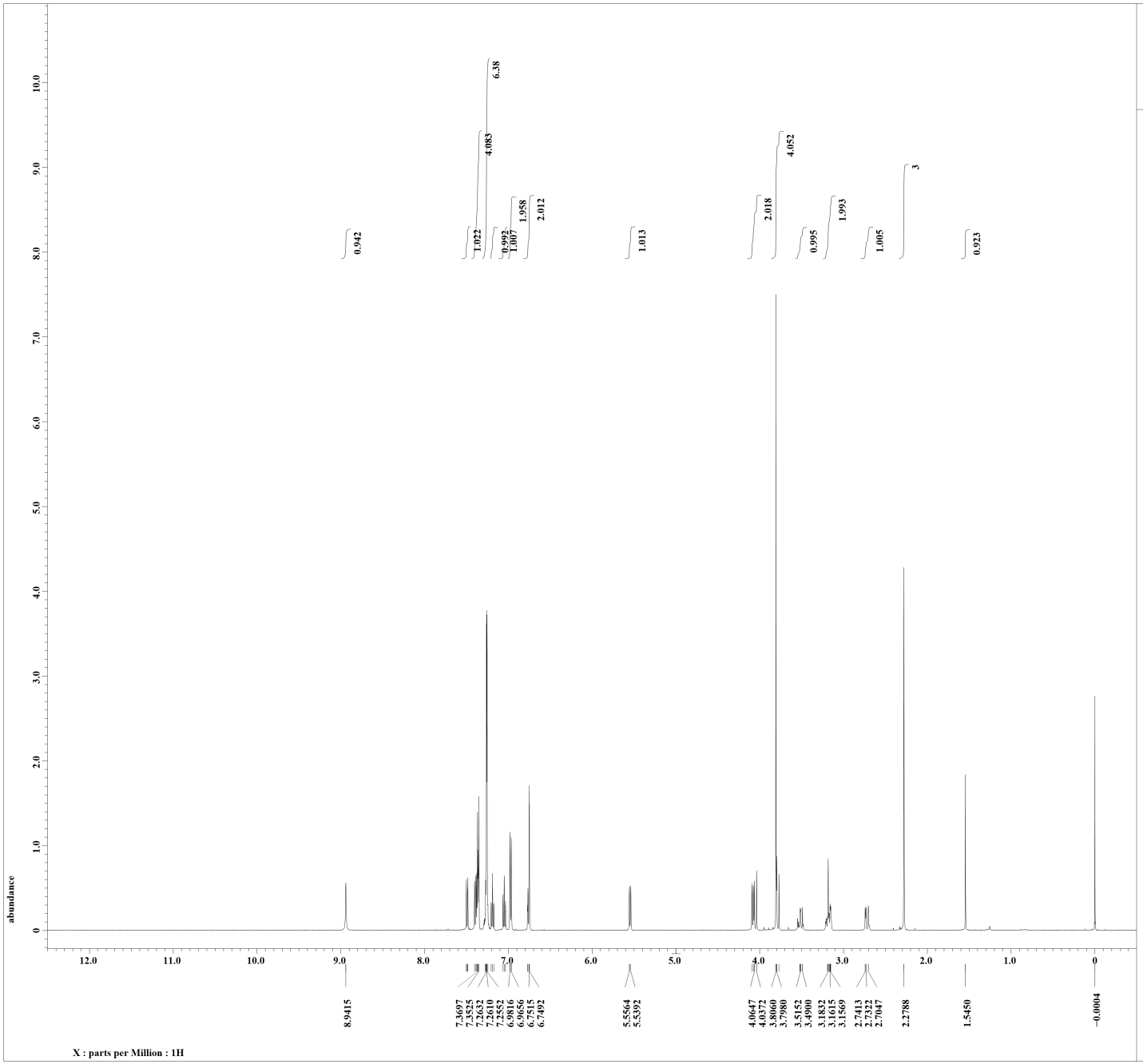
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 17646
Total_scans       = 17646

X_90_width        = 11.2[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.73333333[us]
Irr_atn_dec       = 20.965[dB]
Irr_atn_noe       = 20.965[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          = 23.5[dC]
```



6a

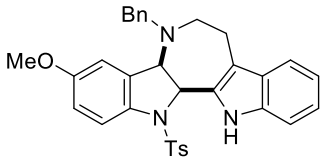


```
Filename          = yama-17-10-04-6.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = yama
Solvent           = CHLOROFORM-D
Creation_time     =  4-OCT-2017 09:11:12
Revision_time     = 25-DEC-2017 09:58:51
Current_time      = 25-DEC-2017 09:59:14

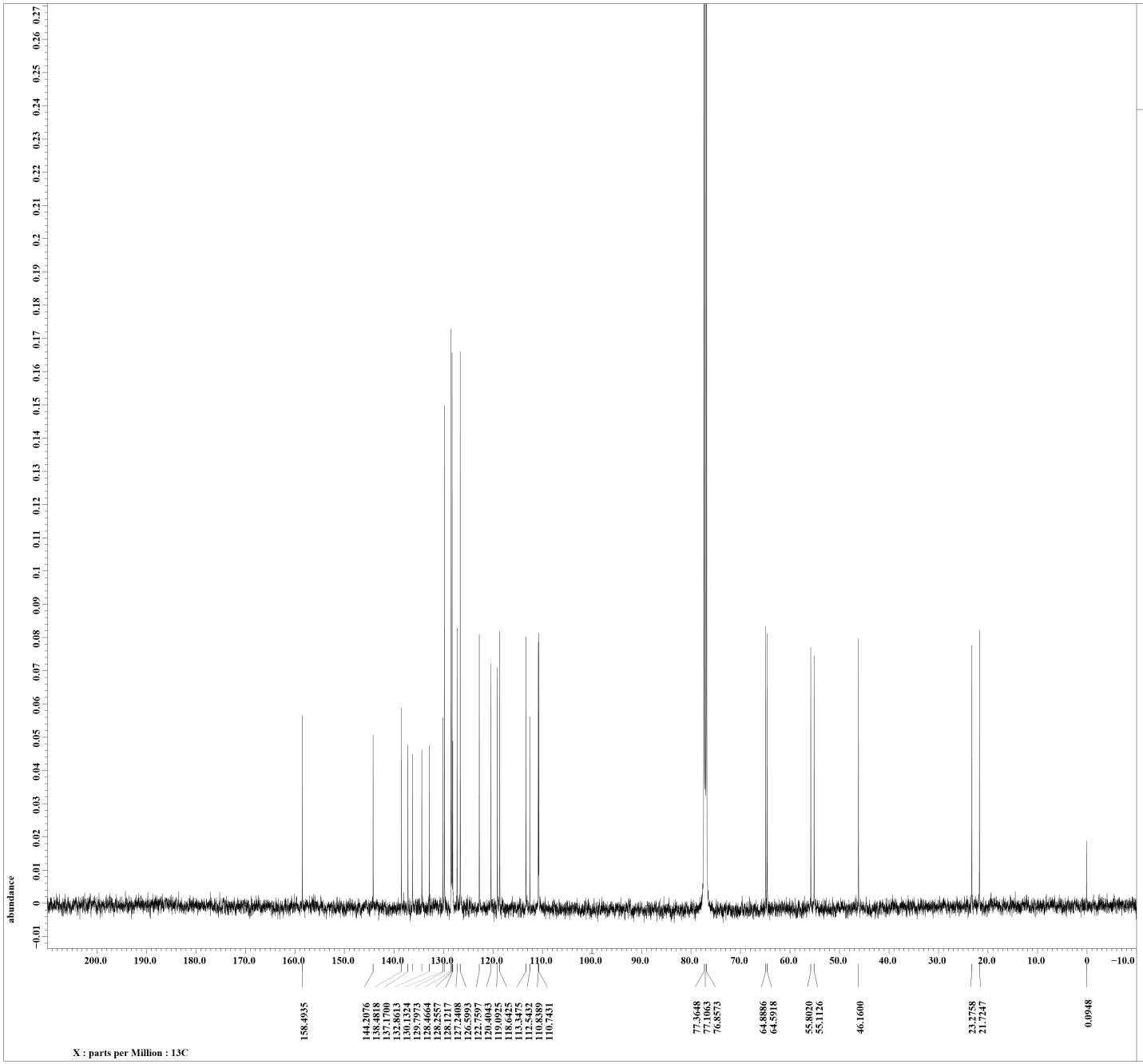
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16
Total_scans       = 16

X_90_width        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[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[dC]
```



6b

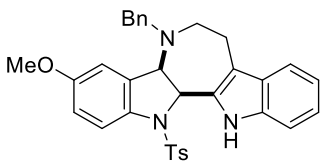



```
Filename          = yama-17-10-04-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = yama
Solvent           = CHLOROFORM-D
Creation_time     =  4-OCT-2017 14:29:49
Revision_time     =  4-OCT-2017 14:37:52
Current_time      =  4-OCT-2017 14:39:54

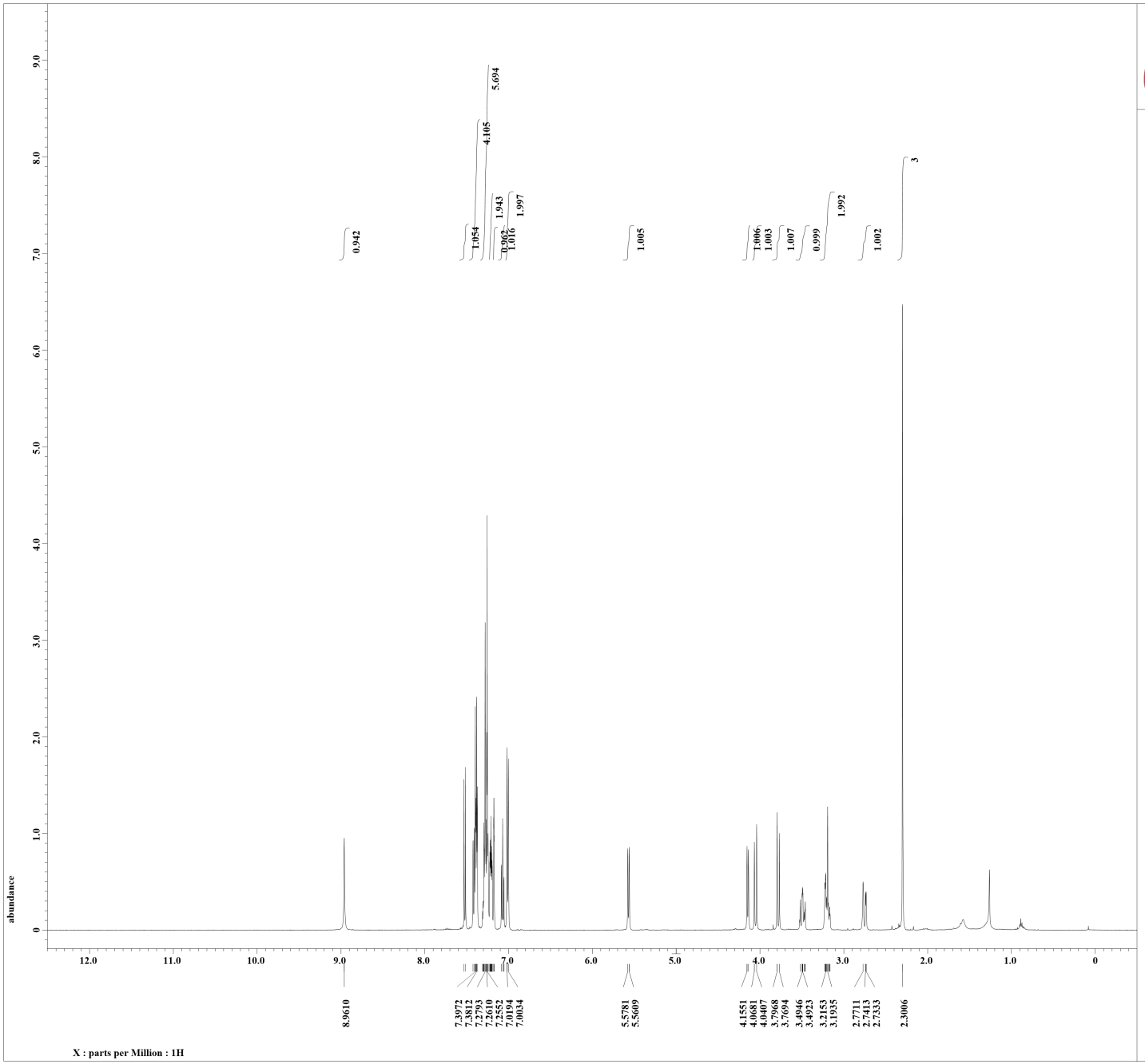
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 6698
Total_scans       = 6698

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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          = 22.6[dC]
```



6b

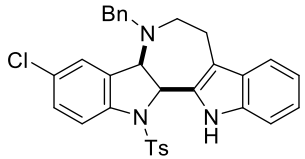



```
Filename          = yama-17-07-28-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#332747
Solvent           = CHLOROFORM-D
Creation_time     = 28-JUL-2017 08:23:29
Revision_time     = 28-JUL-2017 09:18:36
Current_time      = 28-JUL-2017 09:20:42

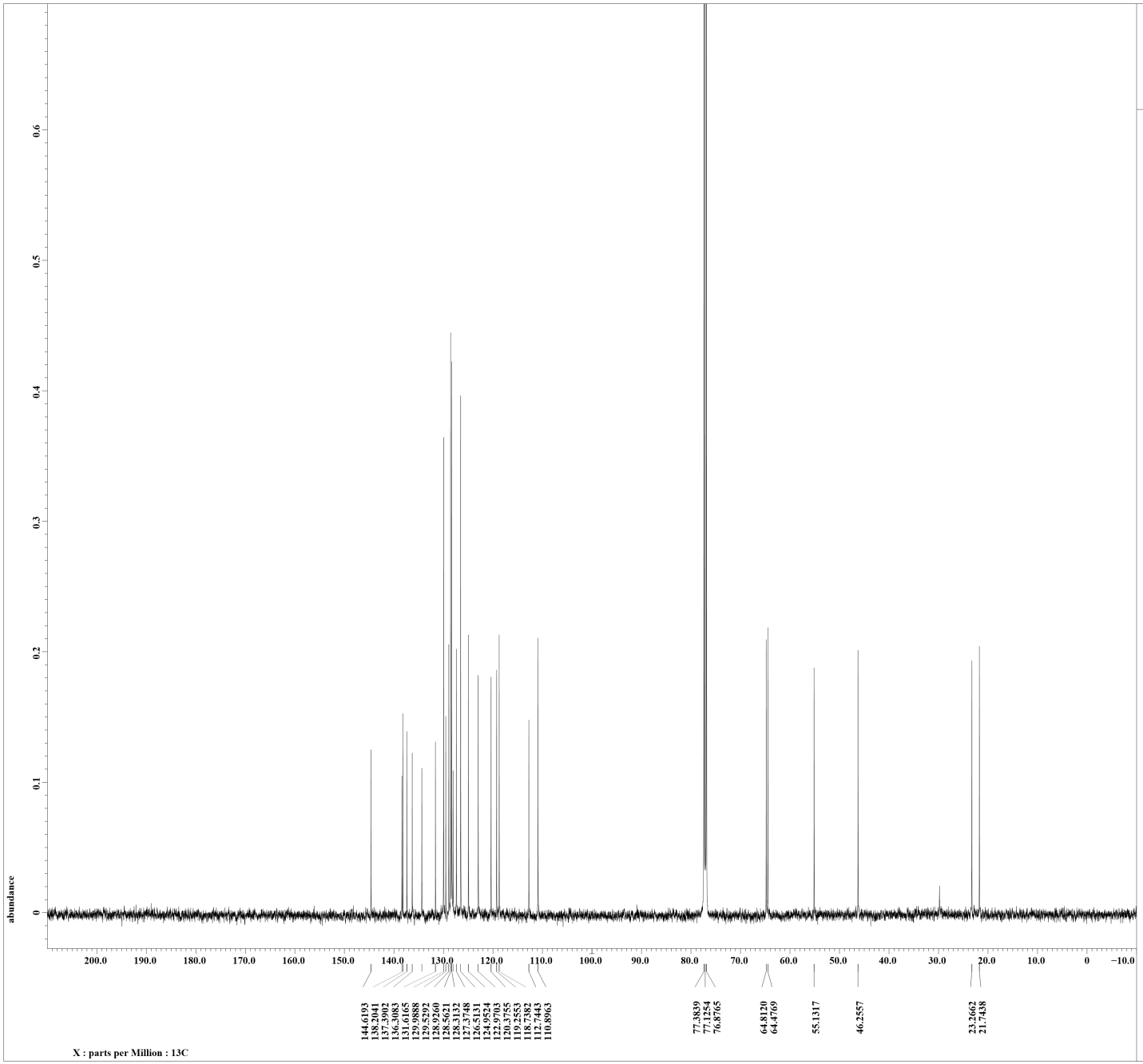
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 21[dC]
```



6c

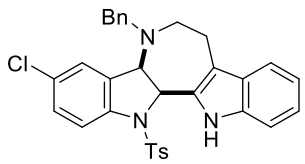



```
Filename          = yama-17-07-28-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#552307
Solvent           = CHLOROFORM-D
Creation_time     = 28-JUL-2017 16:11:29
Revision_time     = 28-JUL-2017 17:06:07
Current_time      = 28-JUL-2017 17:06:43

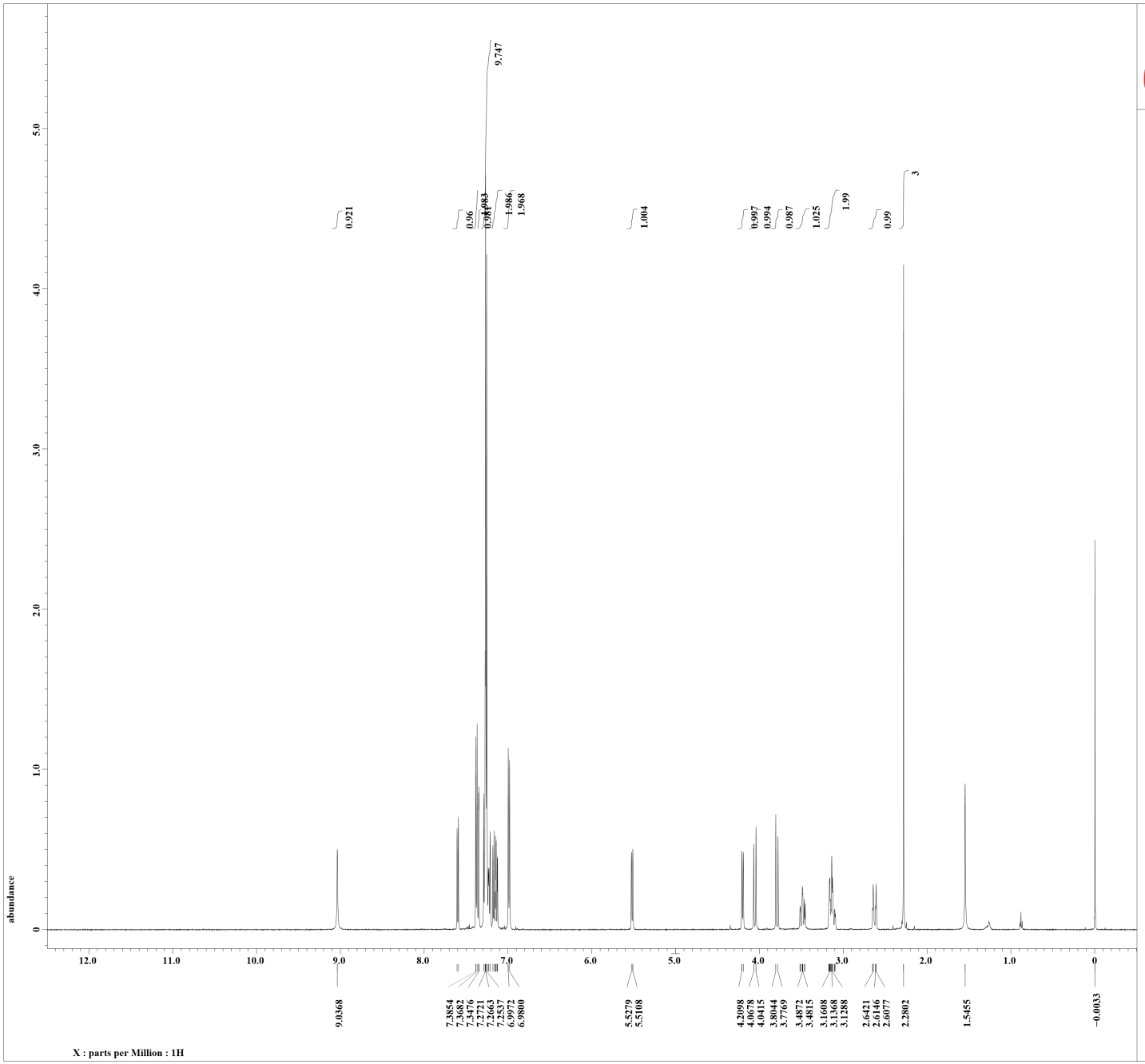
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 2175
Total_scans       = 2175

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22.6[dC]
```



6c

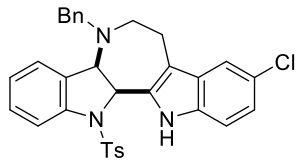


```
Filename          = yama-18-01-09-2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#575792
Solvent           = CHLOROFORM-D
Creation_time     =  2-JAN-2018 06:02:29
Revision_time     =  9-JAN-2018 16:22:27
Current_time      =  9-JAN-2018 16:23:21

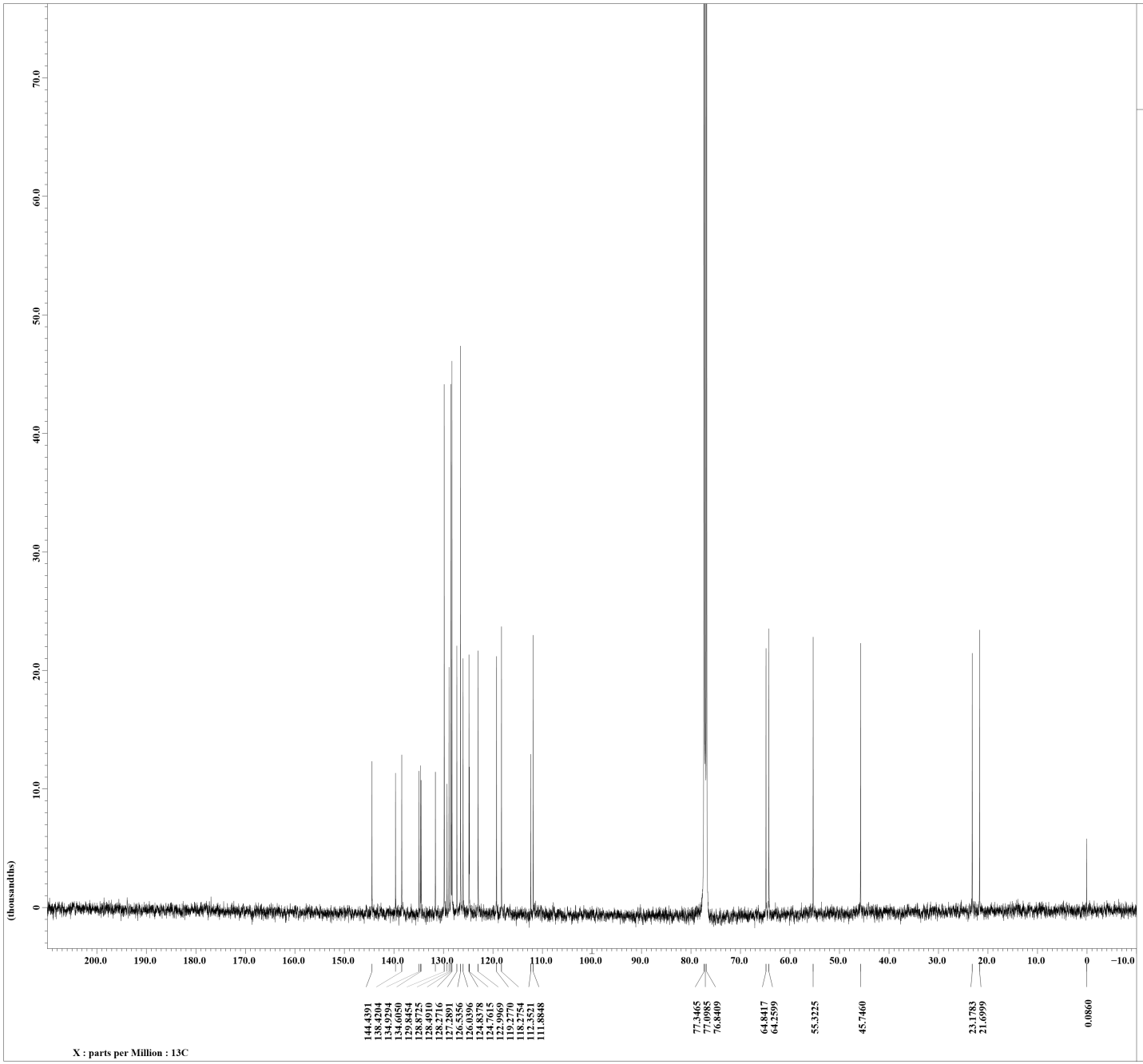
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 12.39[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6.195[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          = 23.1[dC]
```



6d

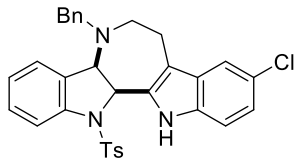



```
Filename          = yama-18-01-09-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#577276
Solvent           = CHLOROFORM-D
Creation_time     =  2-JAN-2018 21:24:21
Revision_time     = 10-JAN-2018 07:44:19
Current_time      = 10-JAN-2018 07:44:54

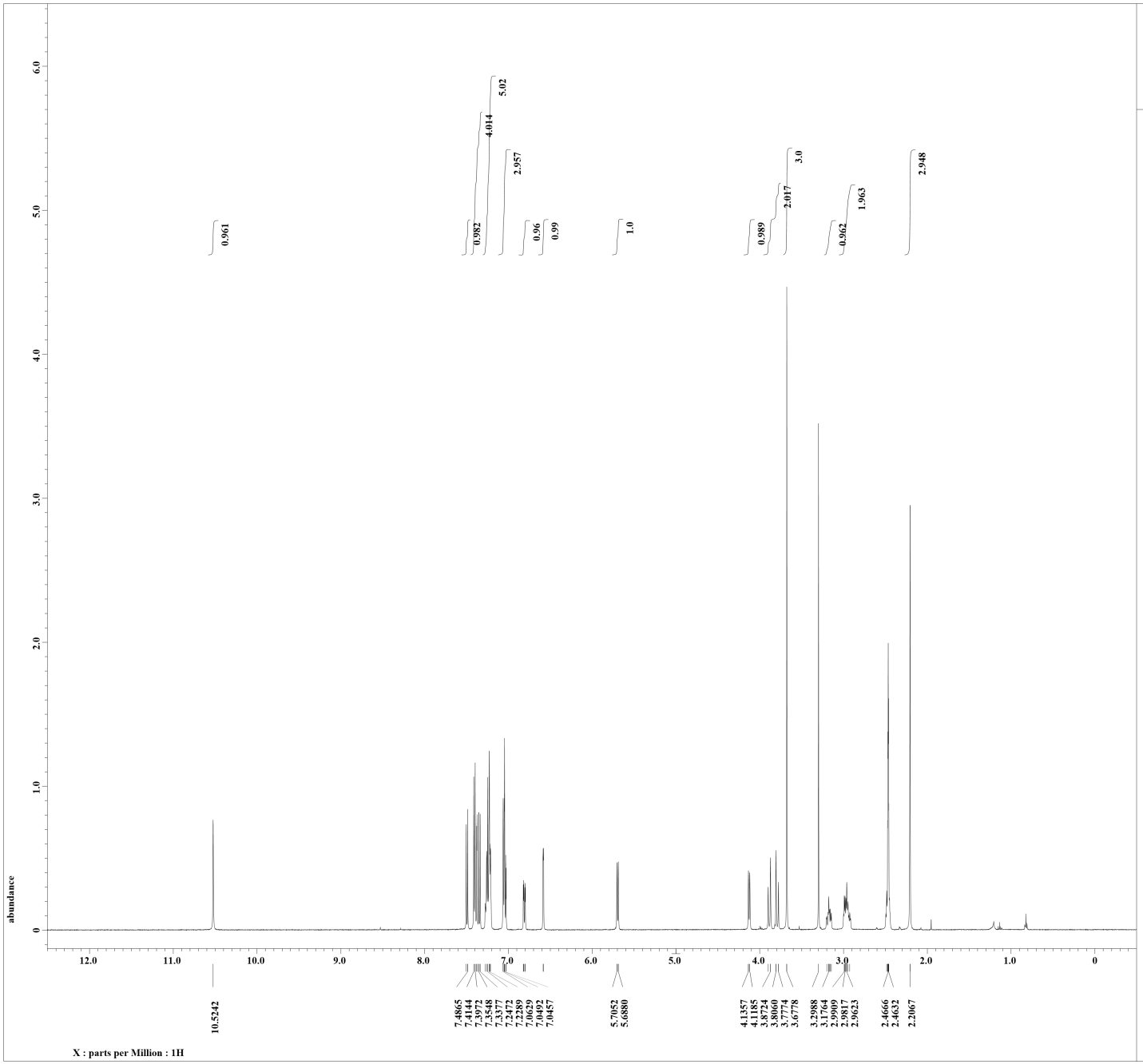
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 19477
Total_scans       = 19477

X_90_width        = 11.43[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 3.81[us]
Irr_atn_dec       = 20.81[dB]
Irr_atn_noe       = 20.81[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          = 24.7[dC]
```



6d

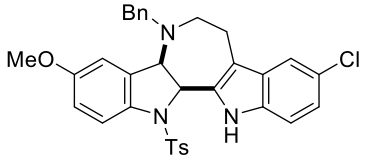



```
Filename          = In-cyclization-5-MeO-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#570984
Solvent           = DMSO-D6
Creation_time     =  8-AUG-2017 14:58:47
Revision_time     = 26-DEC-2017 10:08:21
Current_time      = 26-DEC-2017 10:08:46

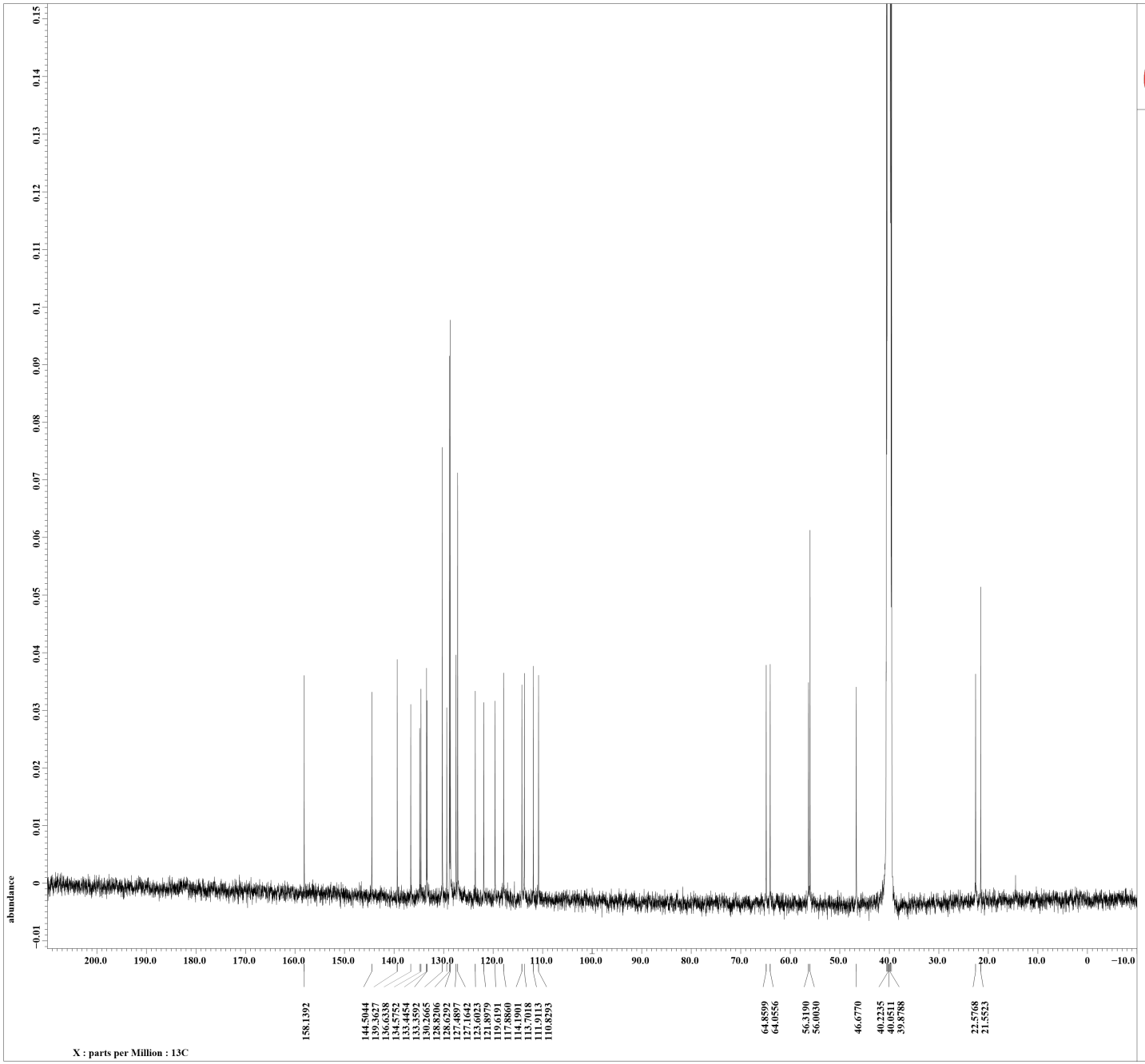
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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          = 21.8[dC]
```



6e

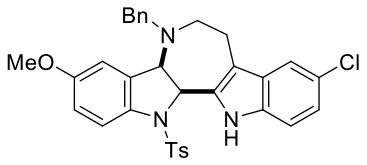



```
Filename          = In-cyclization-5-MeO-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#635370
Solvent           = DMSO-D6
Creation_time     =  9-AUG-2017 06:56:58
Revision_time     = 26-DEC-2017 10:10:36
Current_time      = 26-DEC-2017 10:11:25

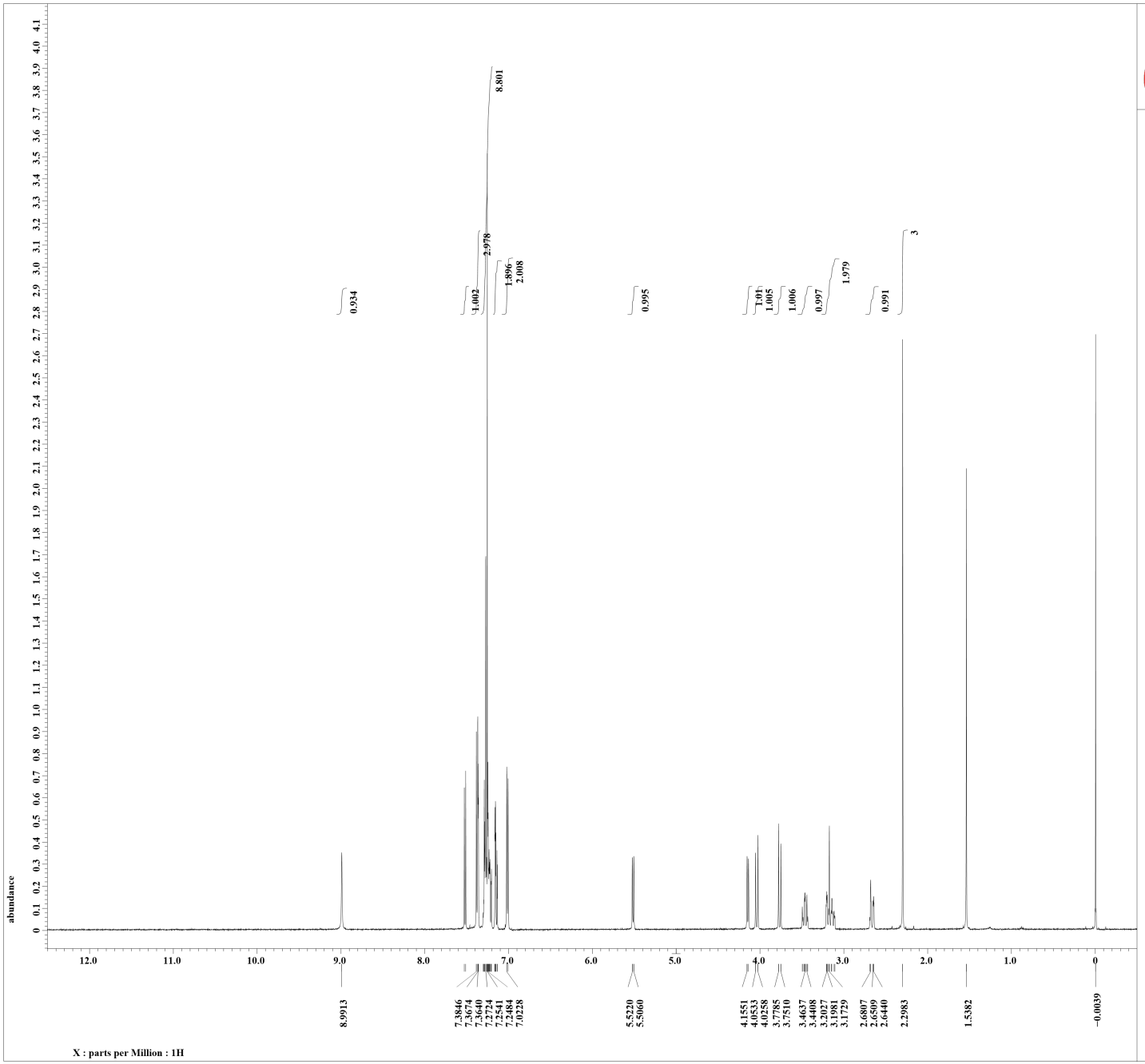
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 18001
Total_scans       = 18001

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22[dC]
```



6e

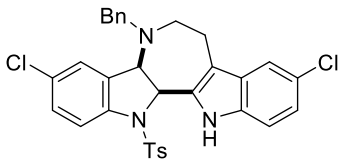



```
Filename          = yama-18-01-09-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#495976
Solvent           = CHLOROFORM-D
Creation_time     =  9-JAN-2018 13:29:41
Revision_time     =  9-JAN-2018 13:54:28
Current_time      =  9-JAN-2018 16:05:56

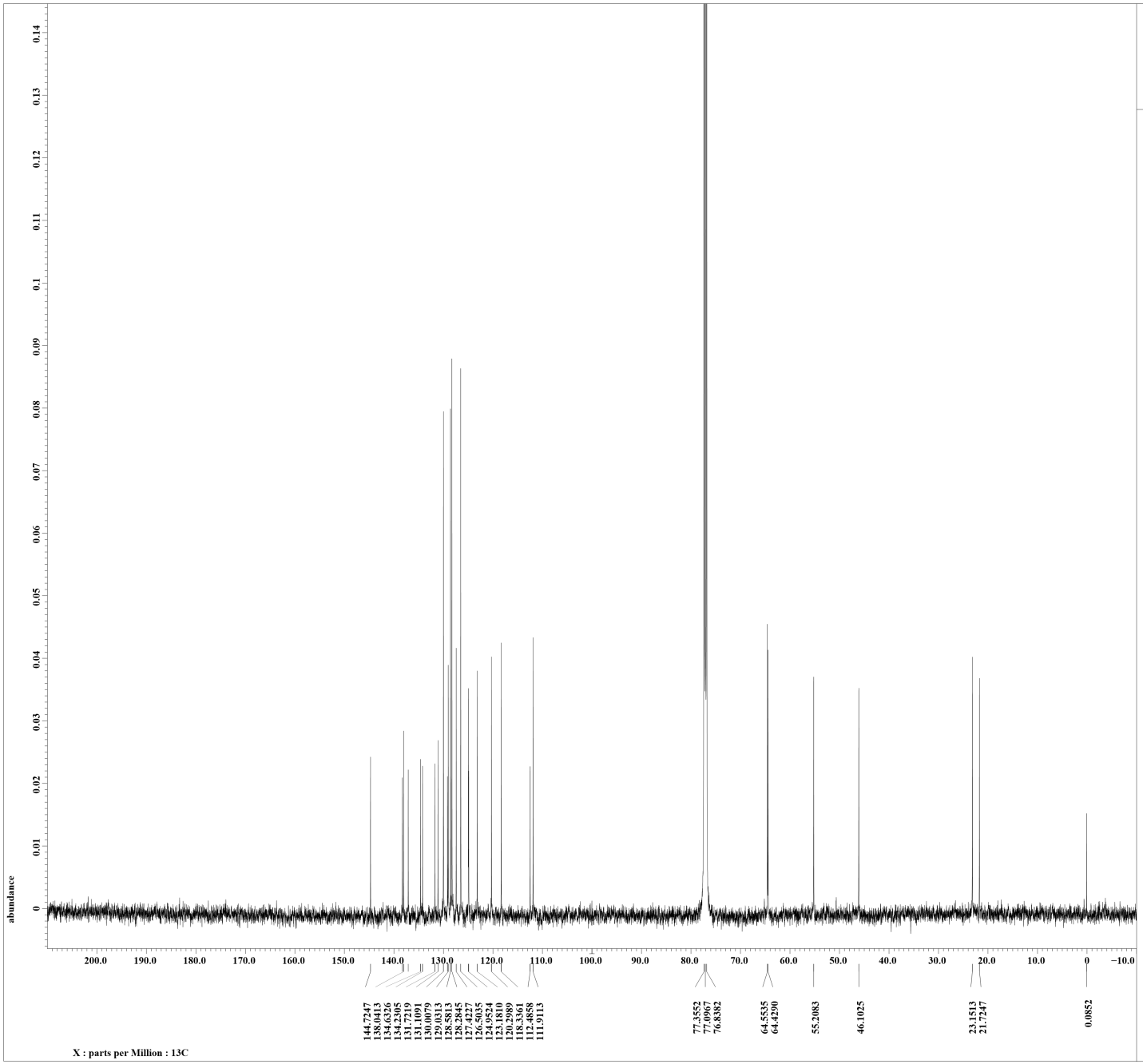
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[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          = 23.7[dC]
```



6f

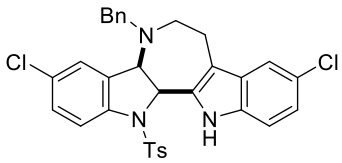



```
Filename          = yama-18-01-09-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#499689
Solvent           = CHLOROFORM-D
Creation_time     = 10-JAN-2018 07:09:20
Revision_time     = 10-JAN-2018 07:31:43
Current_time      = 10-JAN-2018 07:33:08

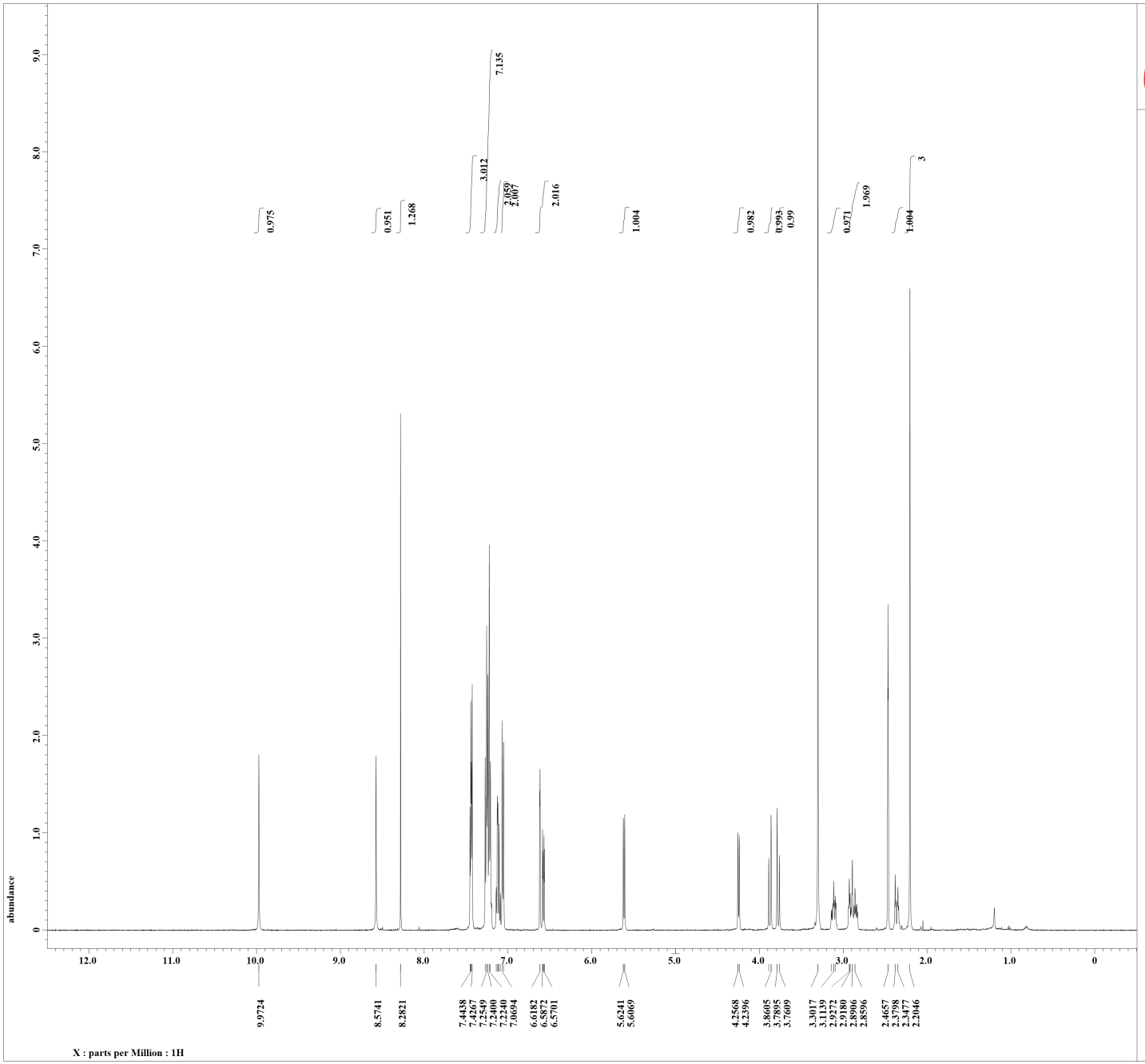
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 22283
Total_scans       = 22283

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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]
```



6f

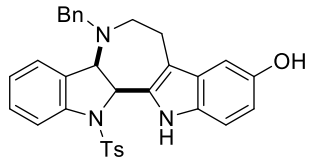



```
Filename          = In-cyclization-Bn-ser
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#638628
Solvent           = DMSO-D6
Creation_time     =  6-JUL-2017 12:37:10
Revision_time     = 26-DEC-2017 10:31:32
Current_time      = 26-DEC-2017 10:32:08

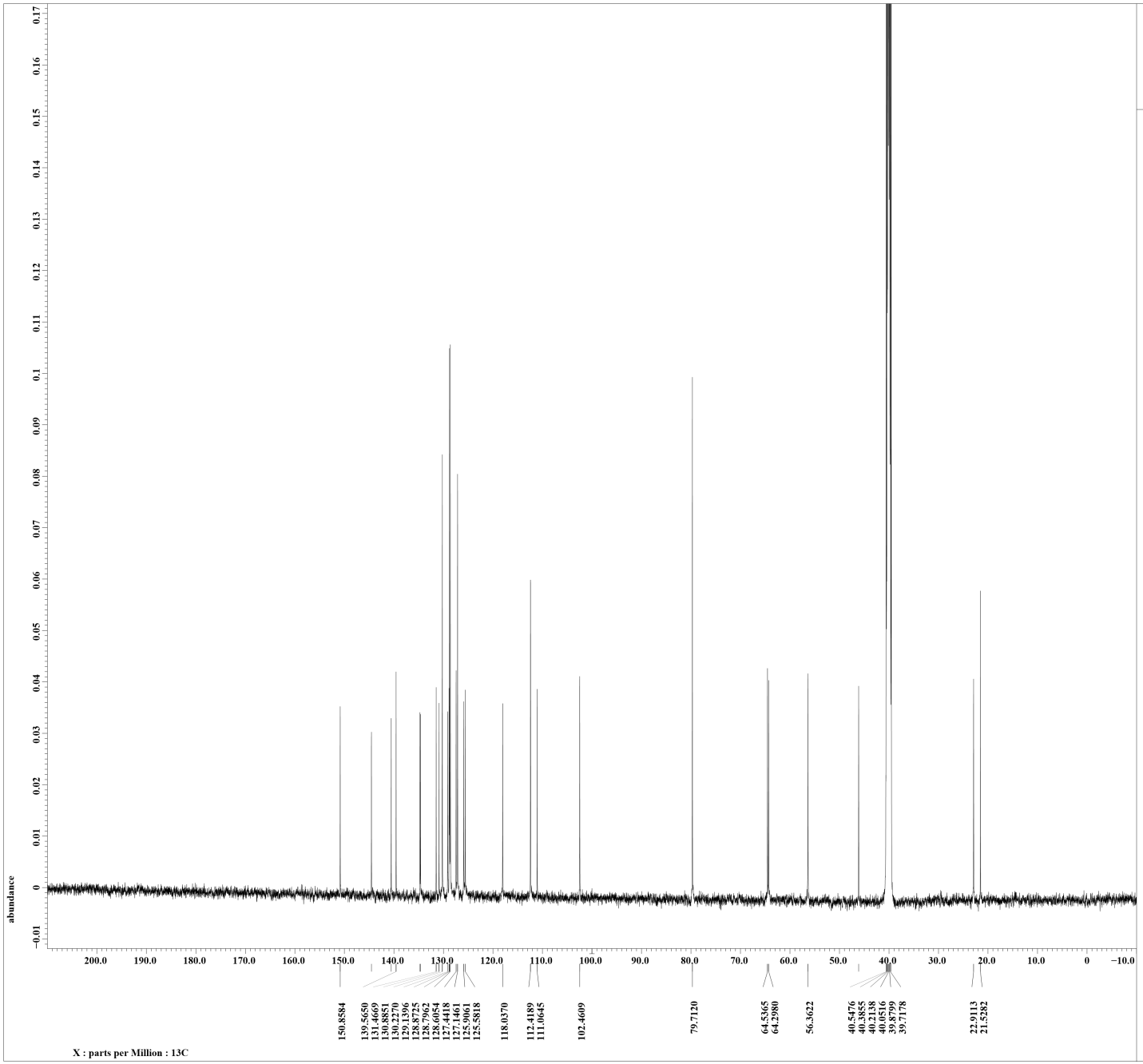
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 54
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22.5[dC]
```



6g

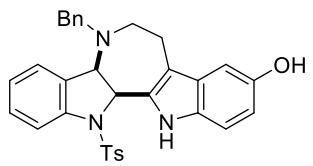


Filename = In-cyclization-Bn-ser
Author = delta
Experiment = single_pulse_dec
Sample_id = S#639772
Solvent = DMSO-D6
Creation_time = 7-JUL-2017 02:01:31
Revision_time = 26-DEC-2017 10:35:16
Current_time = 26-DEC-2017 10:36:20

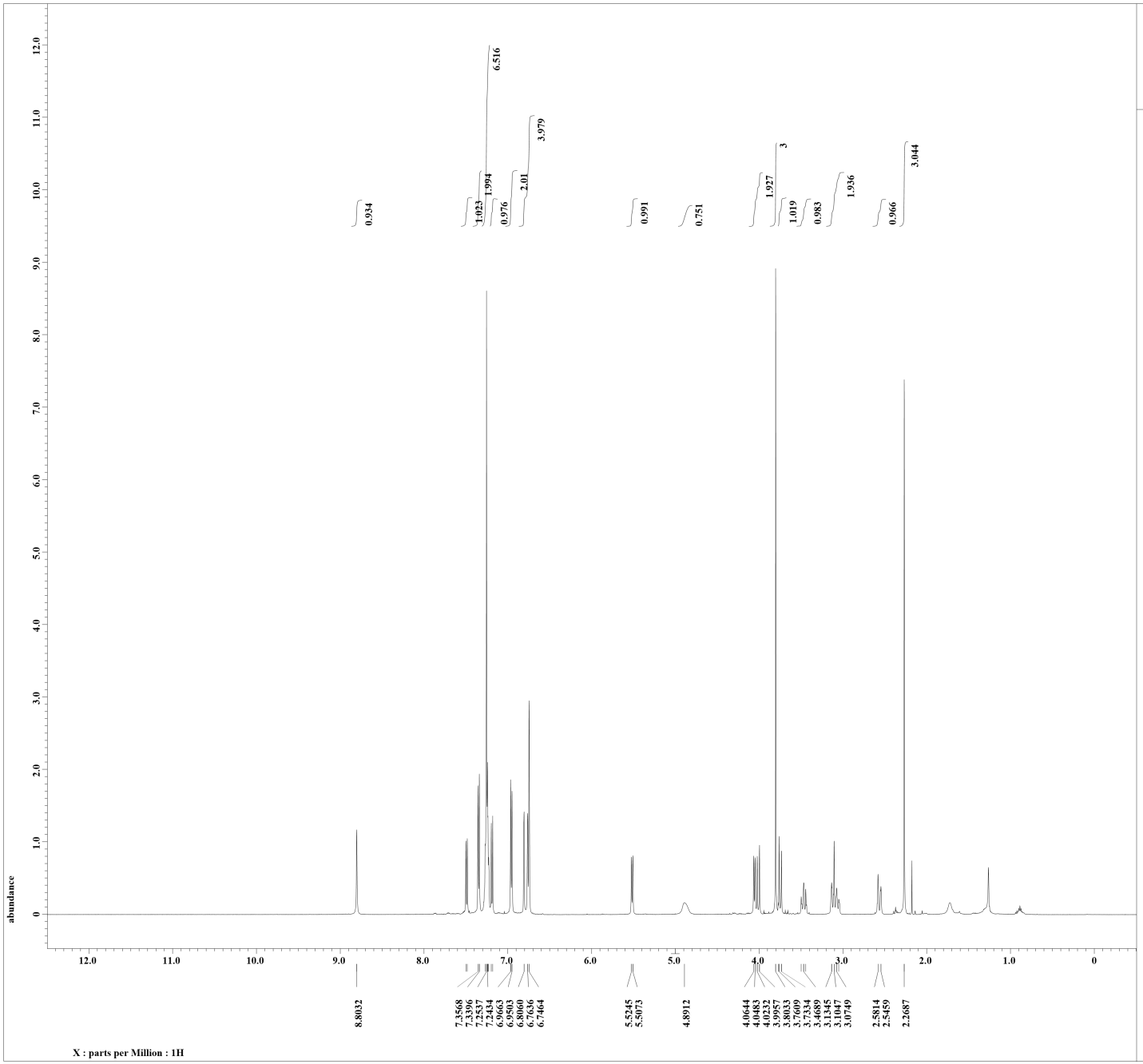
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR

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 = 17013
Total_scans = 17013

X_90_width = 11.3[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.5[dB]
X_pulse = 3.76666667[us]
Irr_atn_dec = 21.238[dB]
Irr_atn_noe = 21.238[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 = 22.3[dC]



6g

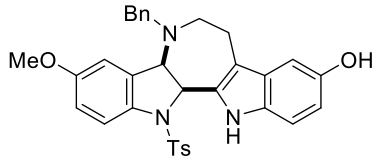



```
Filename          = In-cyclization-5-MeO-
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#315342
Solvent           = CHLOROFORM-D
Creation_time     = 19-JUL-2017 03:45:36
Revision_time     = 26-DEC-2017 10:41:06
Current_time      = 26-DEC-2017 10:41:45

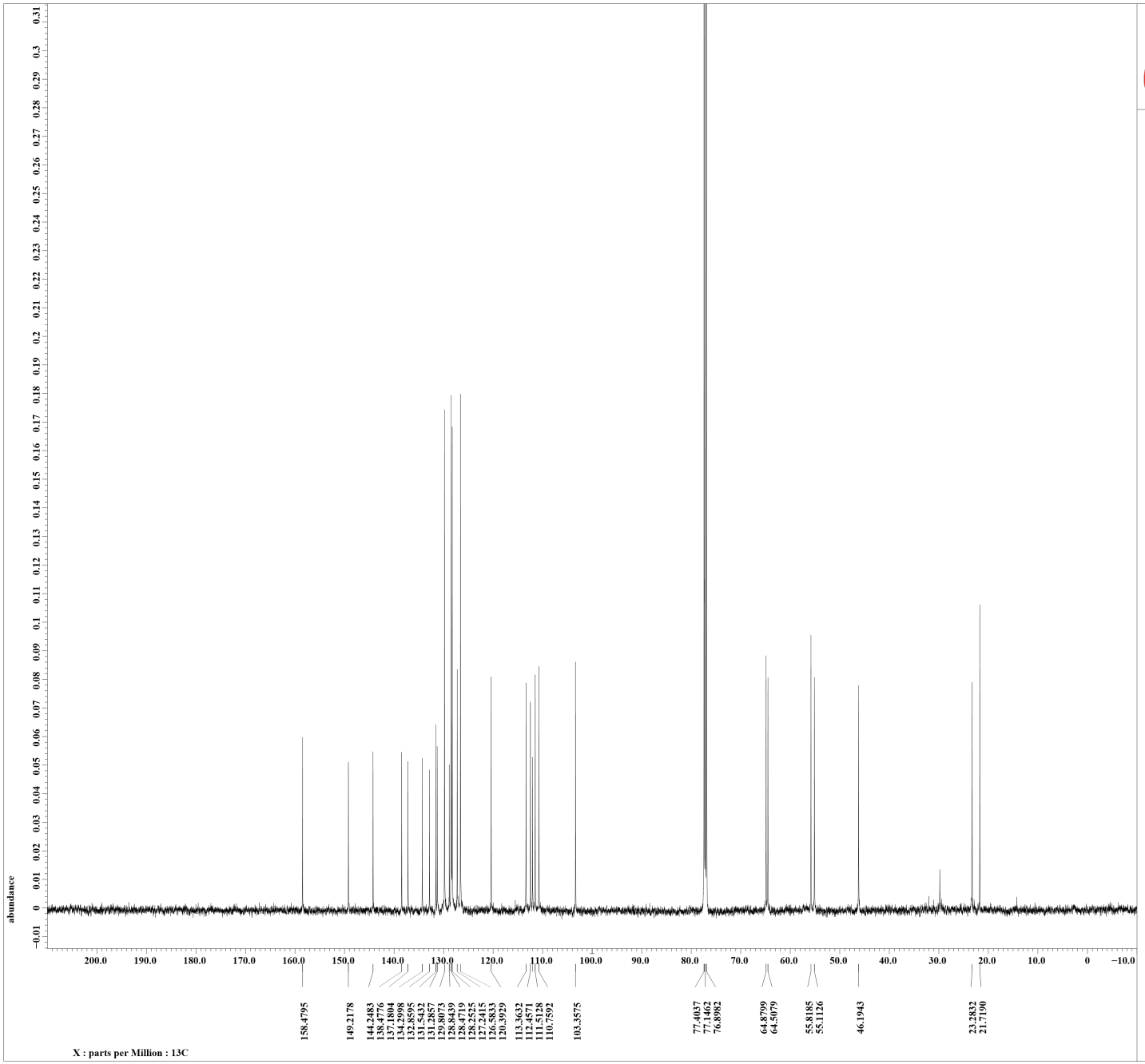
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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        = 11.8[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 5.9[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 21.8[dC]
```



6h

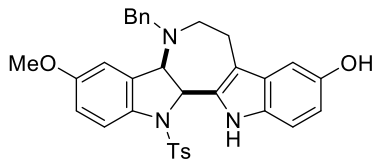



```
Filename          = In-cyclization-5-MeO-
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#388453
Solvent           = CHLOROFORM-D
Creation_time     = 19-JUL-2017 07:37:34
Revision_time     = 26-DEC-2017 10:46:20
Current_time      = 26-DEC-2017 10:47:00

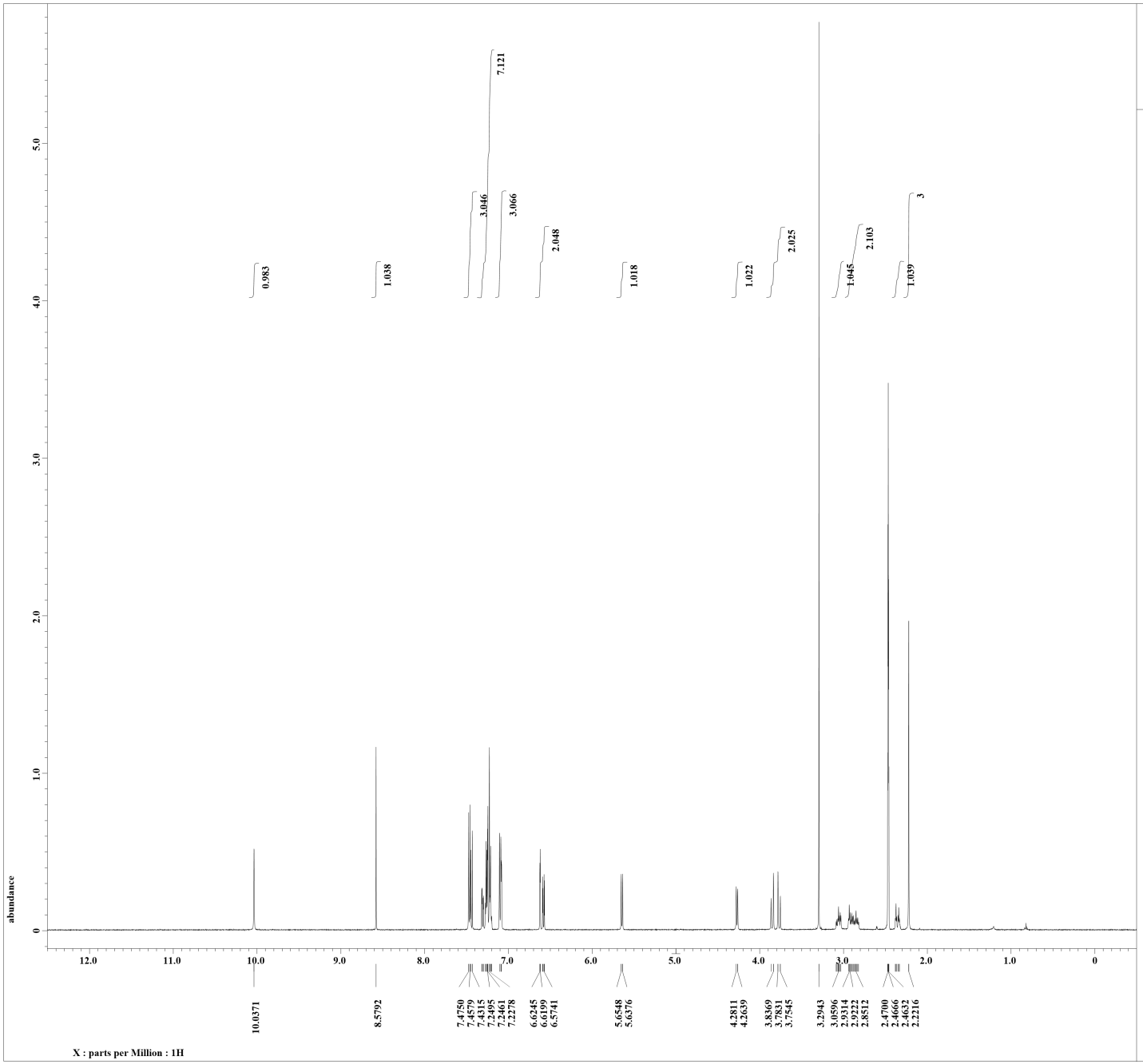
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 2361
Total_scans       = 2361

X_90_width        = 11.3[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.5[dB]
X_pulse           = 3.76666667[us]
Irr_atn_dec       = 21.238[dB]
Irr_atn_noe       = 21.238[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          = 22.2[dC]
```



6h

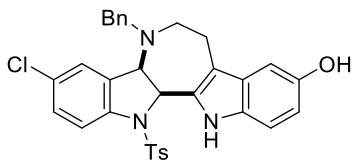



```
Filename          = yama-17-08-09-2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#631202
Solvent           = DMSO-D6
Creation_time     =  9-AUG-2017 16:38:27
Revision_time     =  9-AUG-2017 17:36:55
Current_time      =  9-AUG-2017 17:37:22

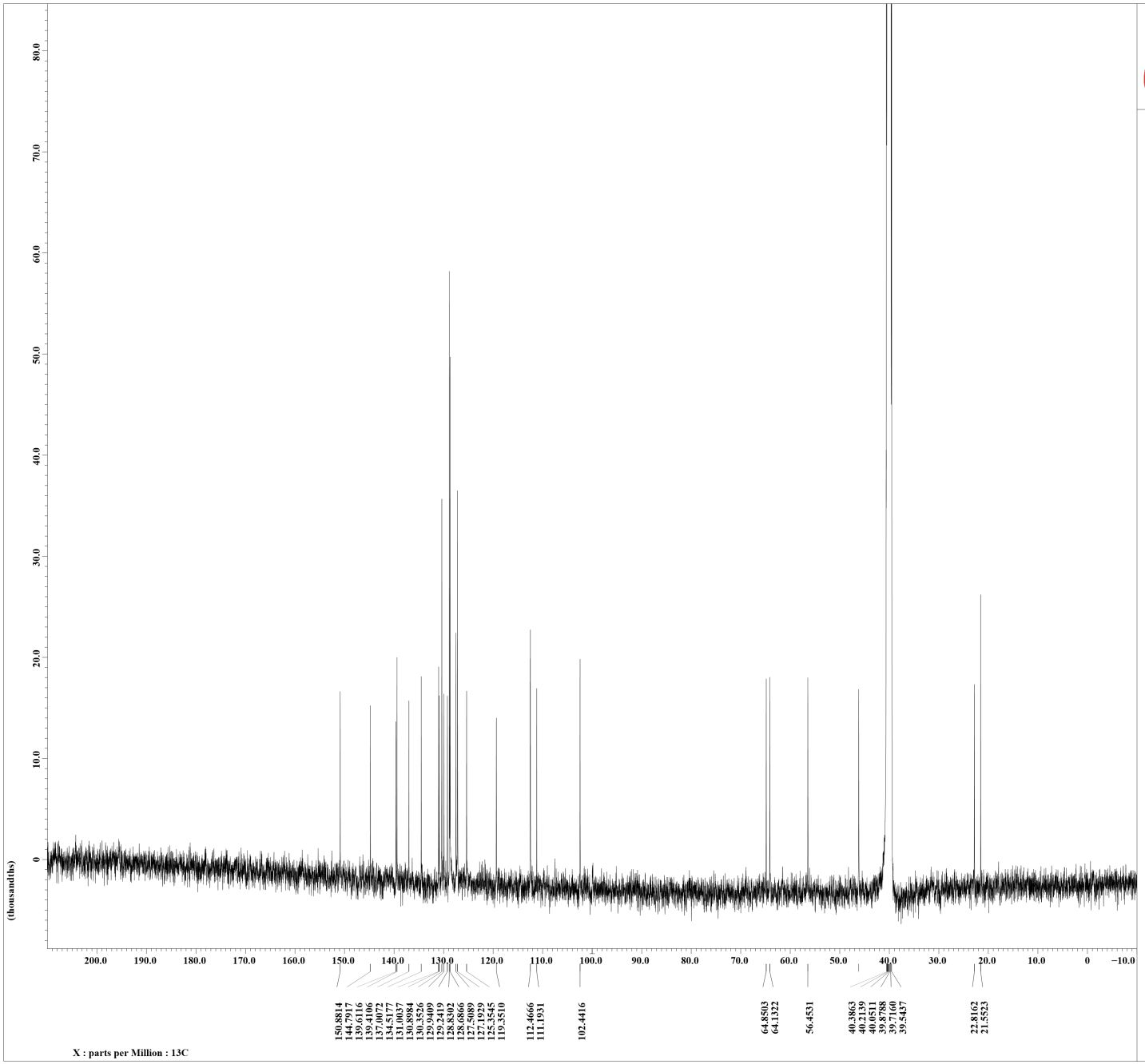
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[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          = 21.6[dC]
```



6i

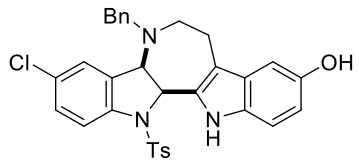



```
Filename          = yama-17-08-09-2-8.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#633041
Solvent           = DMSO-D6
Creation_time     = 10-AUG-2017 06:52:26
Revision_time     = 10-AUG-2017 07:50:44
Current_time      = 10-AUG-2017 07:51:18

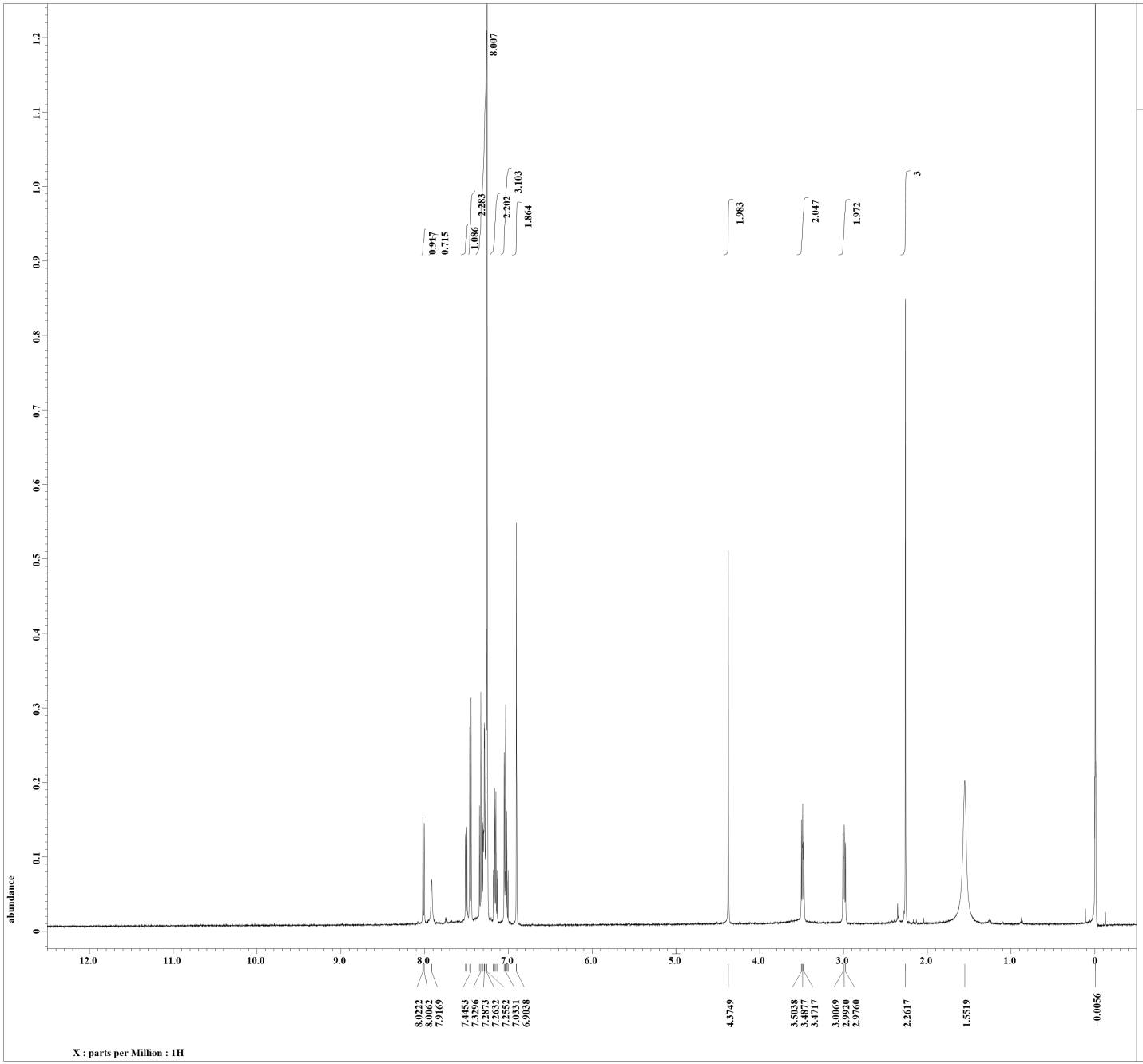
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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           = TRUE
Mod_return        = 1
Scans             = 17995
Total_scans       = 17995

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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          = 21.5[dC]
```



6i

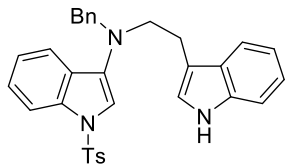



```
Filename          = yama-16-11-27-11.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#295813
Solvent           = CHLOROFORM-D
Creation_time     = 27-NOV-2016 08:11:31
Revision_time     = 25-DEC-2017 09:52:22
Current_time      = 25-DEC-2017 09:53:12

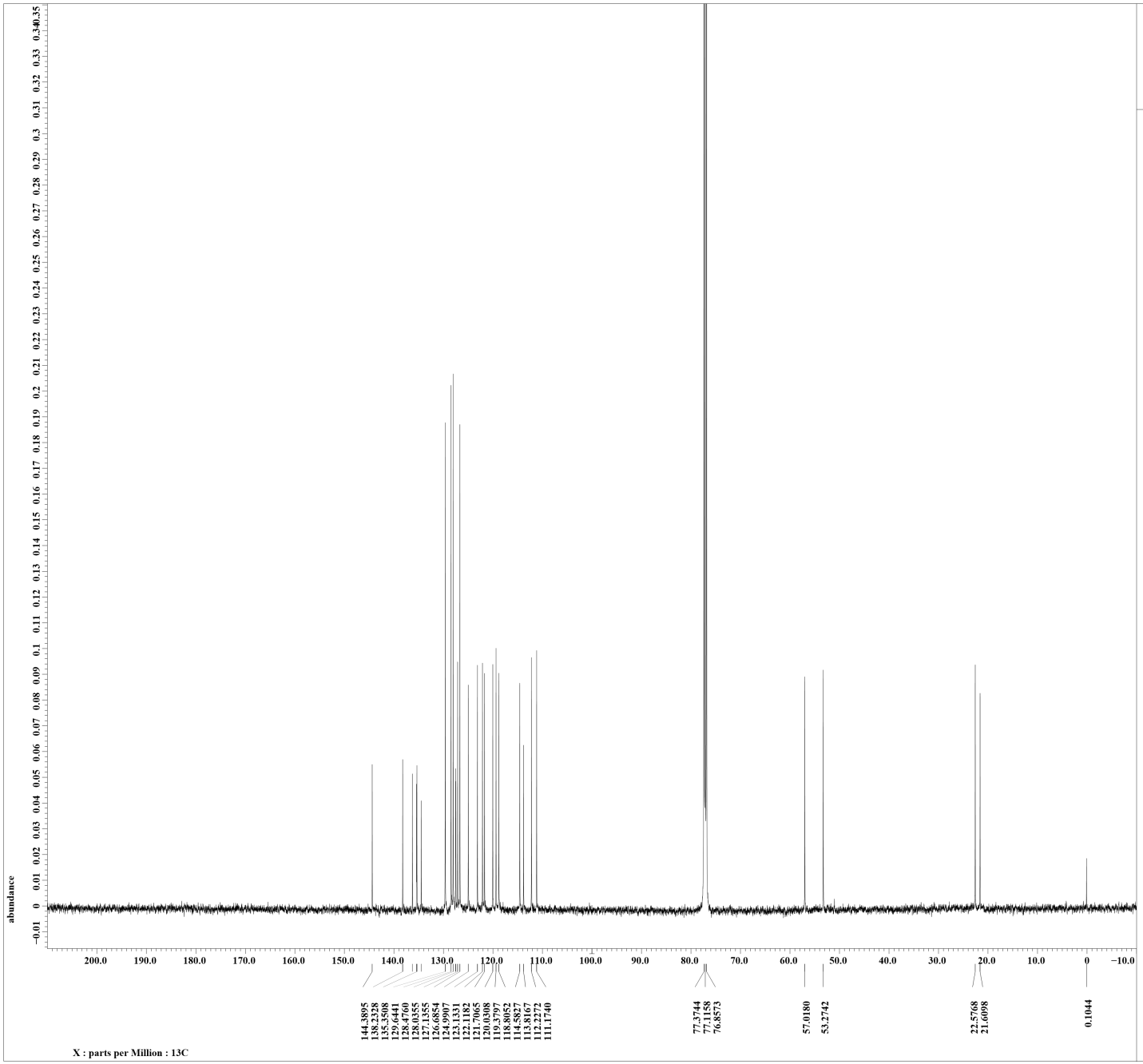
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 64
Total_scans       = 64

X_90_width        = 12.7[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.6[dB]
X_pulse           = 6.35[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[dC]
```



7a

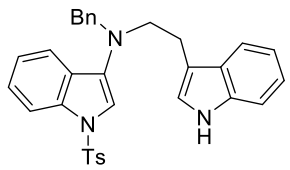



```
Filename          = yama-16-11-27-12.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#657546
Solvent           = CHLOROFORM-D
Creation_time     = 30-NOV-2016 08:01:30
Revision_time     = 25-DEC-2017 09:48:49
Current_time      = 25-DEC-2017 09:49:41

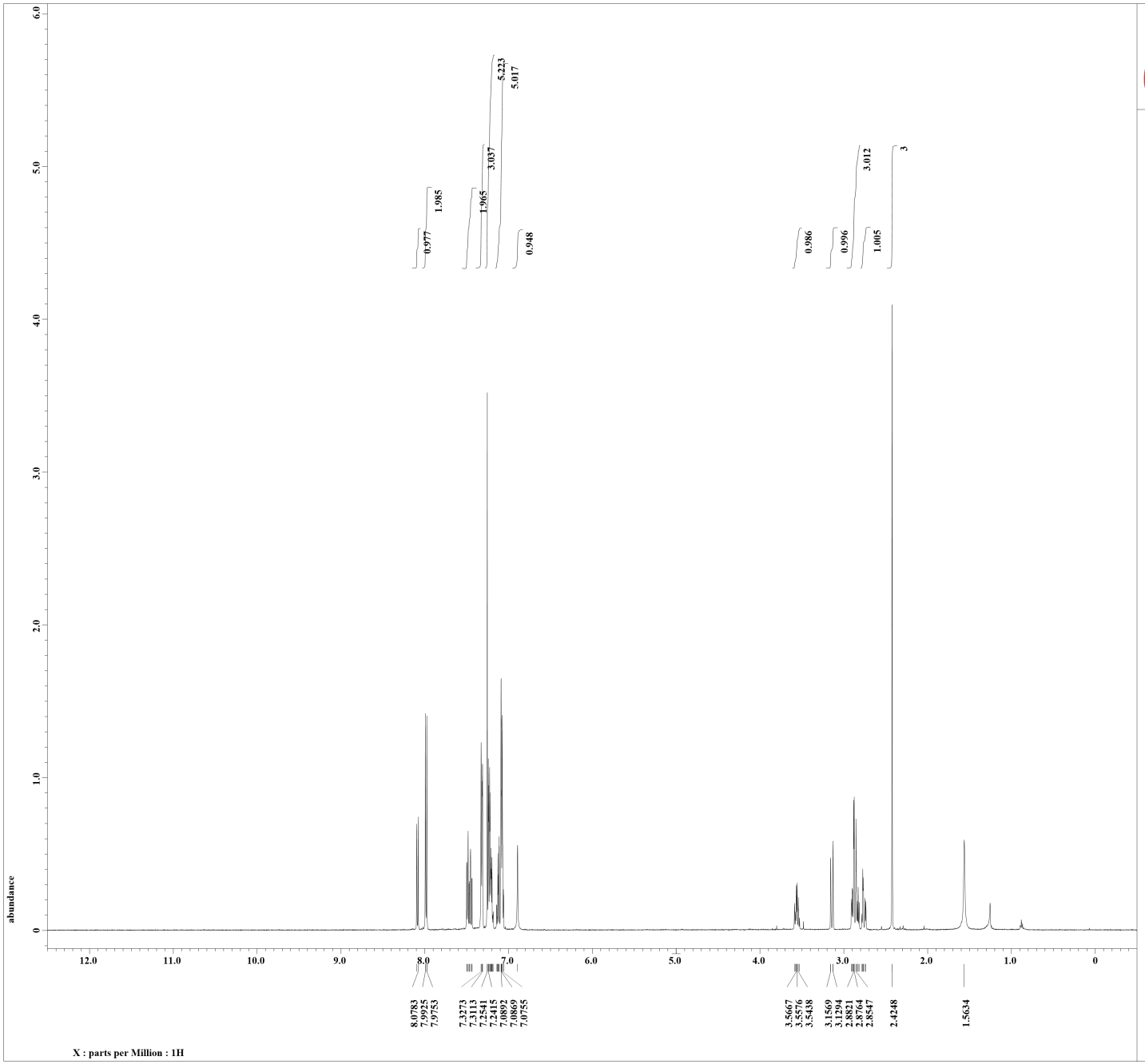
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 17639
Total_scans       = 17639

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 21.7[dC]
```



7a

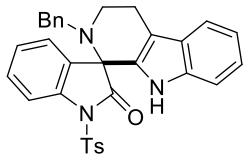


Filename = yama-17-07-20-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#381310
Solvent = CHLOROFORM-D
Creation_time = 20-JUL-2017 09:46:00
Revision_time = 20-JUL-2017 10:40:33
Current_time = 20-JUL-2017 10:40:57

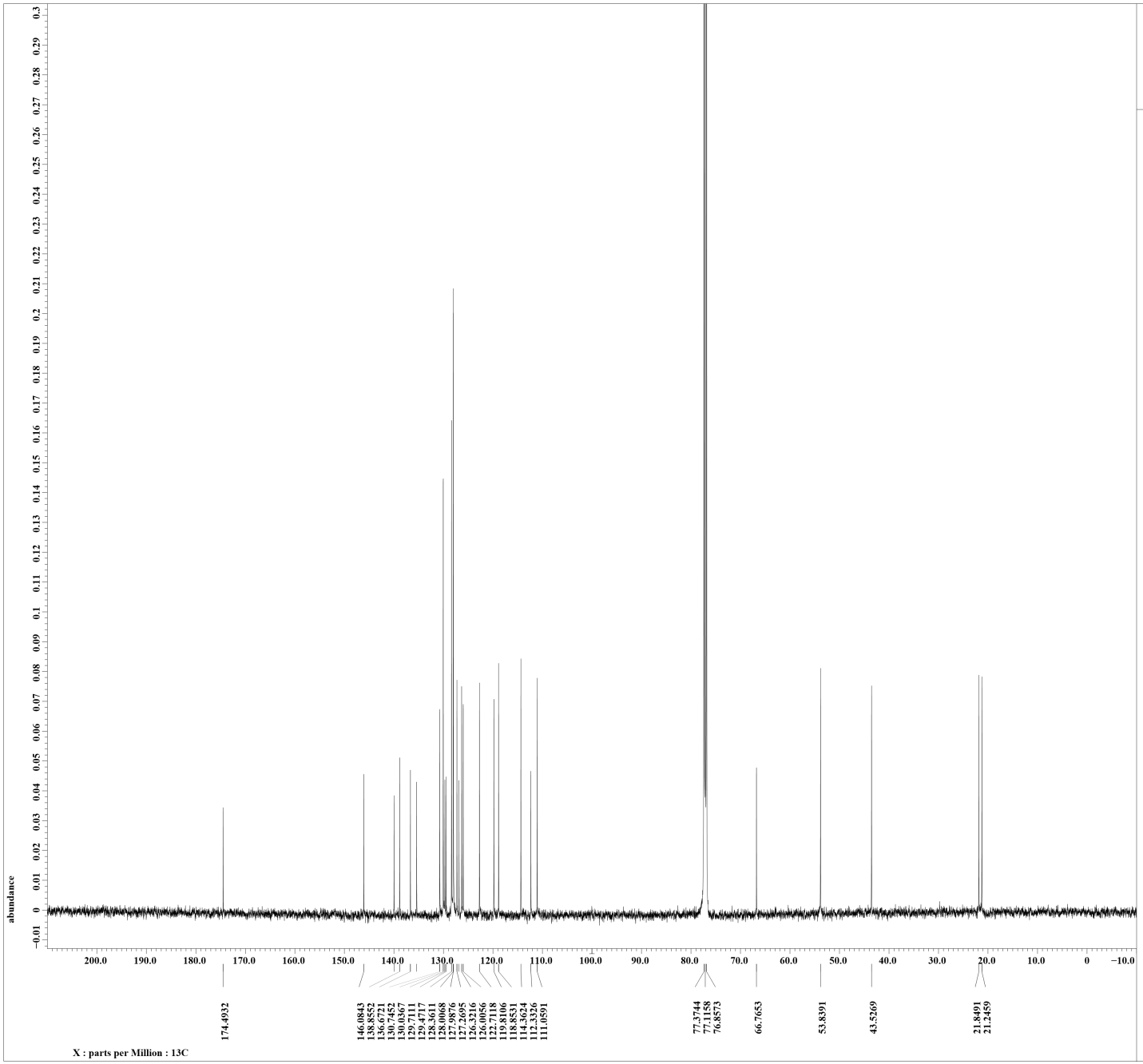
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

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 = 12.7[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.6[dB]
X_pulse = 6.35[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]



8a

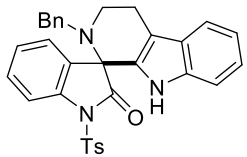



```
Filename          = yama-17-07-20-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = yama
Solvent           = CHLOROFORM-D
Creation_time     = 21-JUL-2017 06:54:23
Revision_time     = 21-JUL-2017 07:48:12
Current_time      = 21-JUL-2017 07:49:06

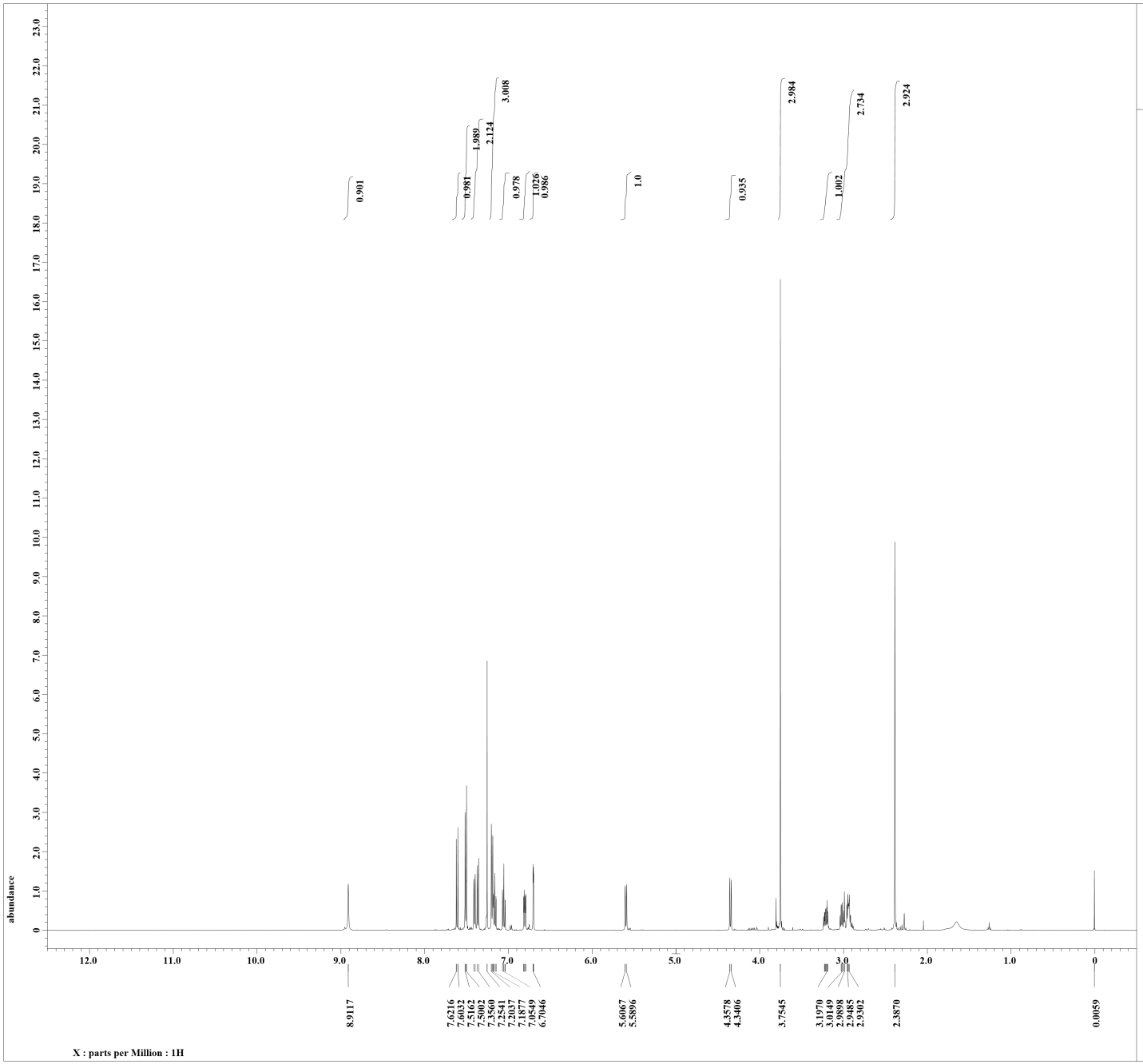
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16336
Total_scans       = 16336

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.8[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.8[dB]
Irr_atn_noe       = 20.8[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          = 22[dC]
```



8a

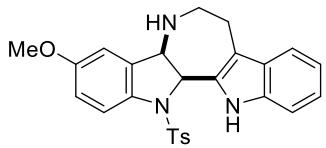



```
Filename          = yama-17-11-28-2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#445275
Solvent           = CHLOROFORM-D
Creation_time     = 28-NOV-2017 12:11:57
Revision_time     = 28-NOV-2017 12:25:59
Current_time      = 28-NOV-2017 12:26:31

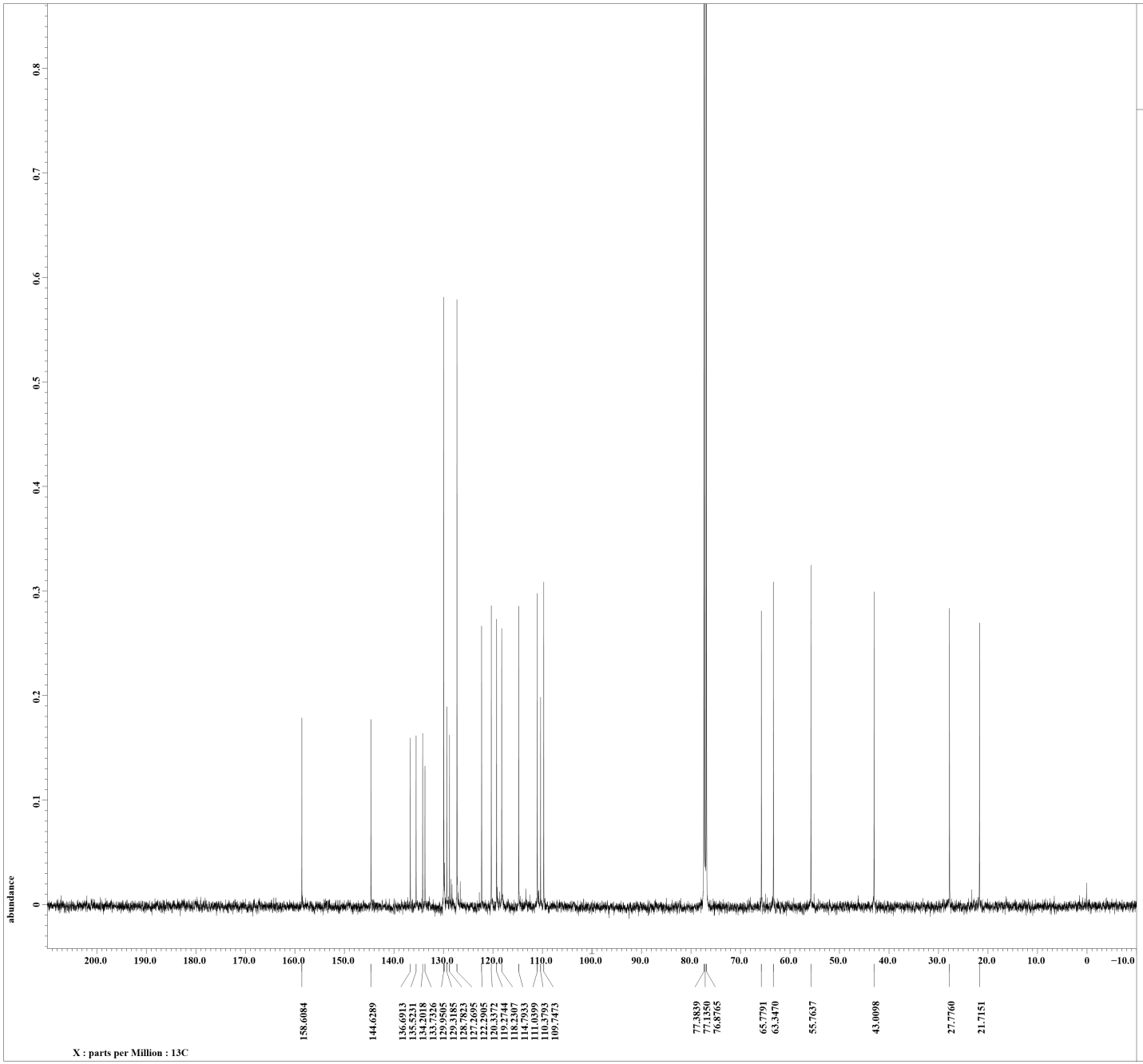
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 6.76422912[s]
Temp_get          = 23.7[dC]
```



9

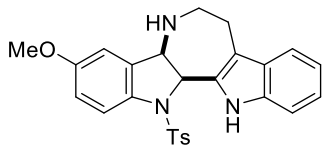



```
Filename          = yama-17-11-28-2-7.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#445568
Solvent           = CHLOROFORM-D
Creation_time     = 28-NOV-2017 13:24:36
Revision_time     = 28-NOV-2017 13:37:05
Current_time      = 28-NOV-2017 13:38:15

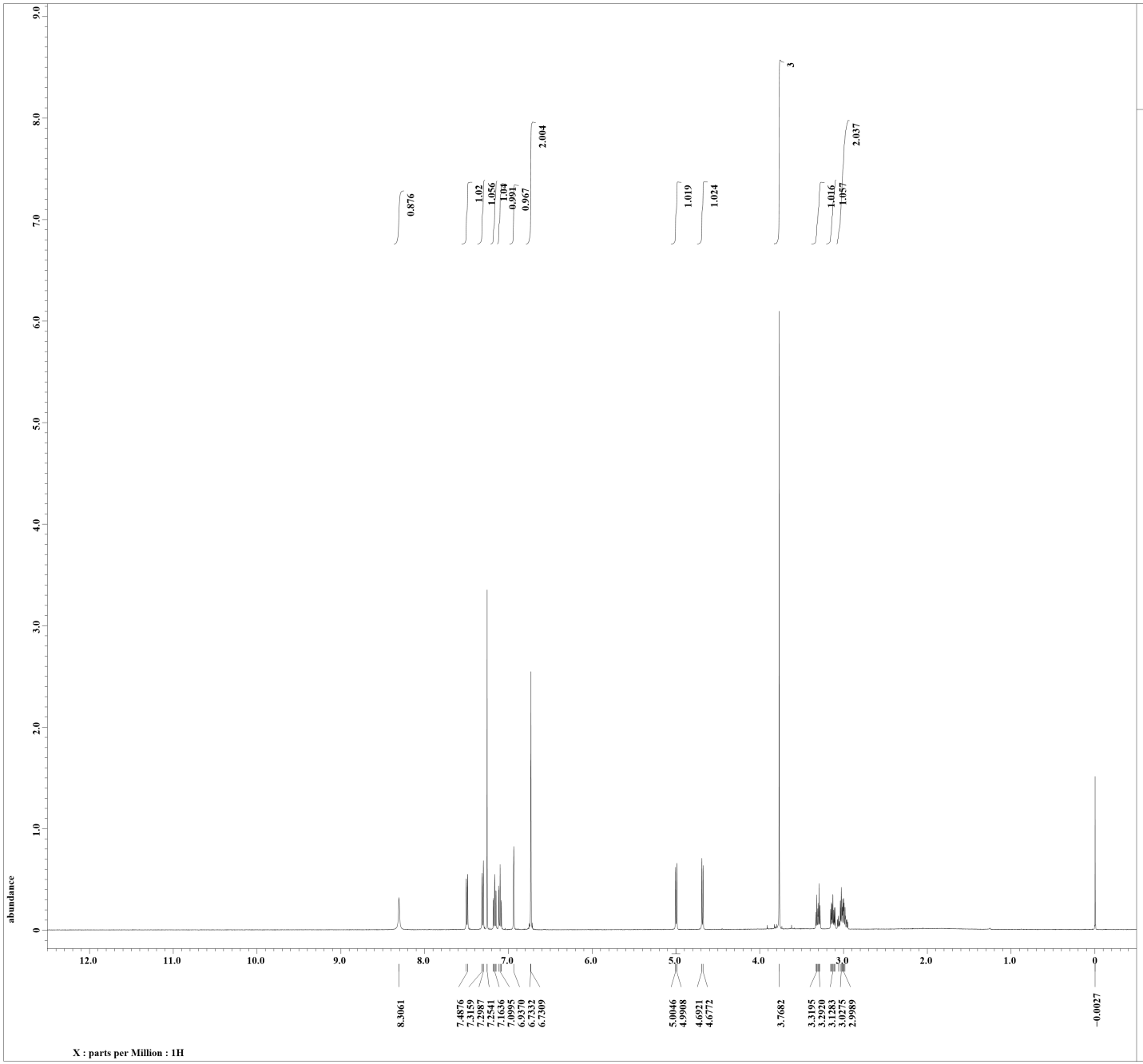
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 1505
Total_scans       = 1505

X_90_width        = 10.83[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.61[us]
Irr_atn_dec       = 20.548[dB]
Irr_atn_noe       = 20.548[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

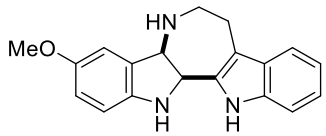



```
Filename          = yama-17-12-19-18.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#421409
Solvent           = CHLOROFORM-D
Creation_time     = 20-DEC-2017 11:29:31
Revision_time     = 20-DEC-2017 11:49:44
Current_time      = 20-DEC-2017 11:49:50

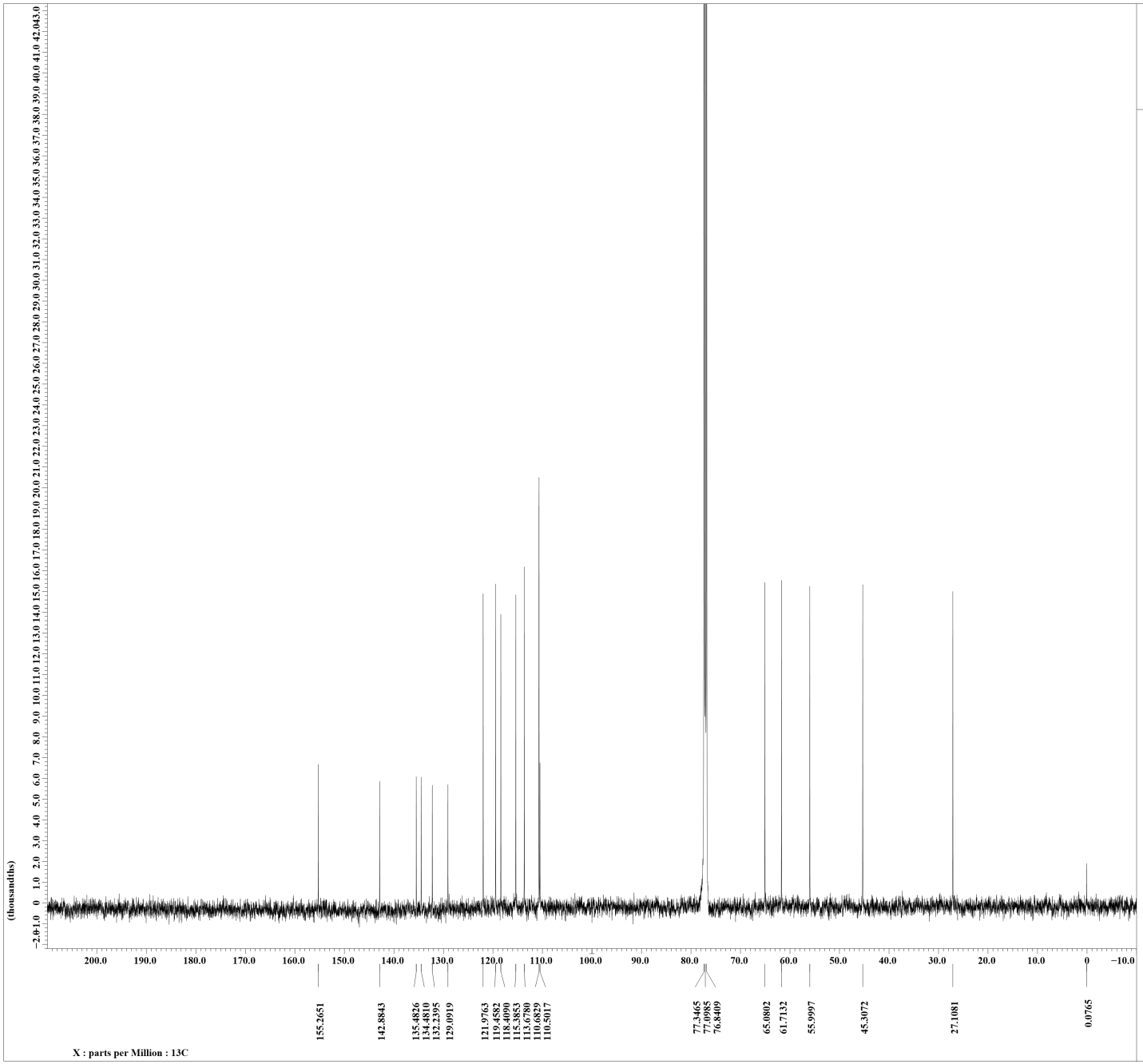
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 16
Total_scans       = 16

X_90_width        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[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          = 23.7[dC]
```



10

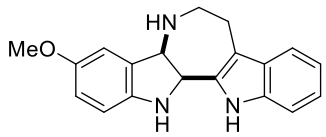



```
Filename          = yama-17-12-21-3.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#654655
Solvent           = CHLOROFORM-D
Creation_time     = 14-DEC-2017 23:26:06
Revision_time     = 25-DEC-2017 10:00:12
Current_time      = 25-DEC-2017 10:00:47

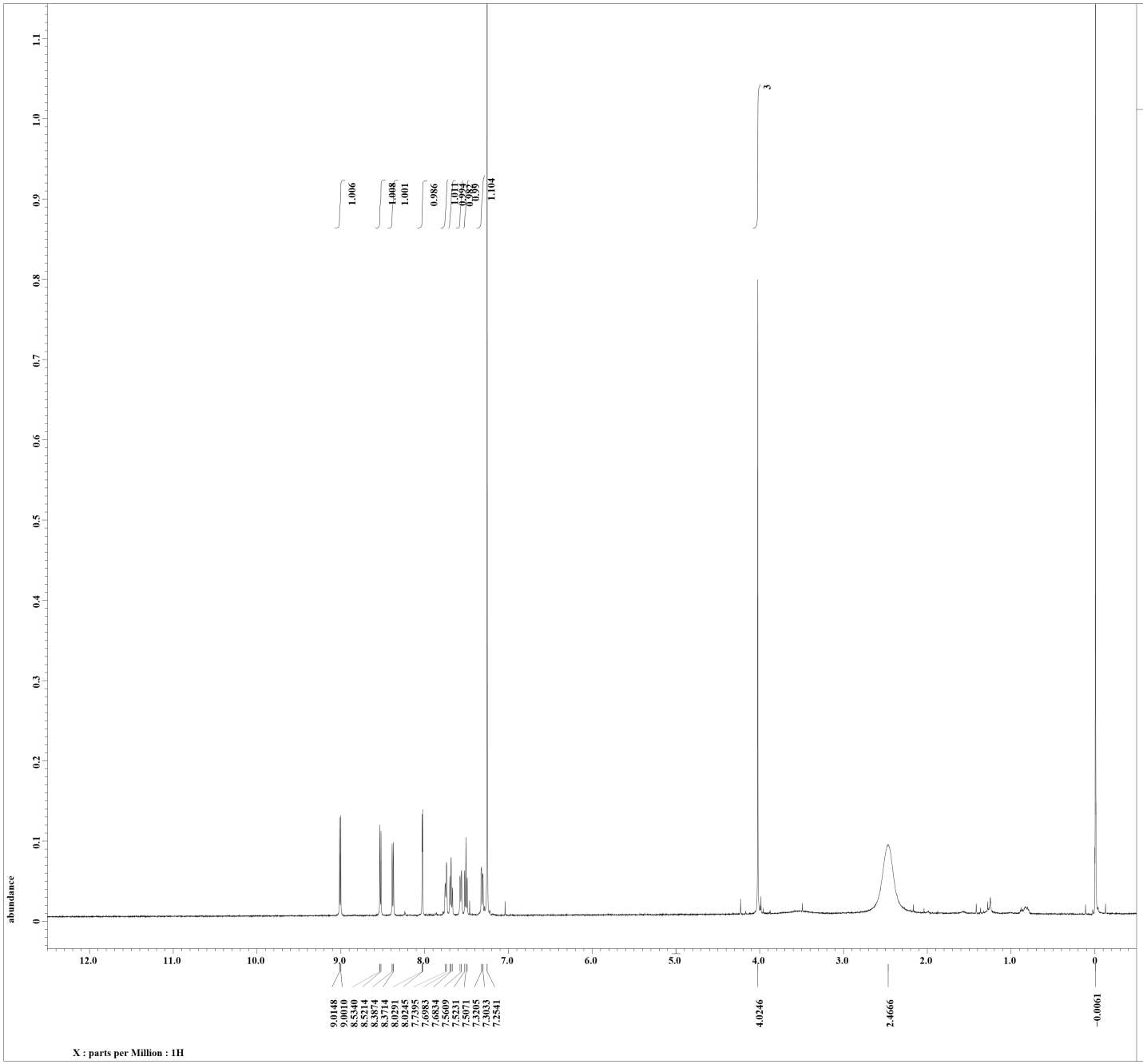
Content           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 19311
Total_scans       = 19311

X_90_width        = 11.43[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 3.81[us]
Irr_atn_dec       = 20.81[dB]
Irr_atn_noe       = 20.81[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]
```



10

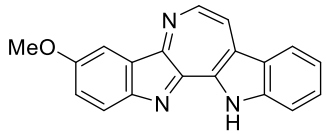



```
Filename          = iheyamine A-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#435552
Solvent           = CHLOROFORM-D
Creation_time     = 13-DEC-2017 12:06:39
Revision_time     = 13-DEC-2017 12:25:39
Current_time      = 13-DEC-2017 12:26:40

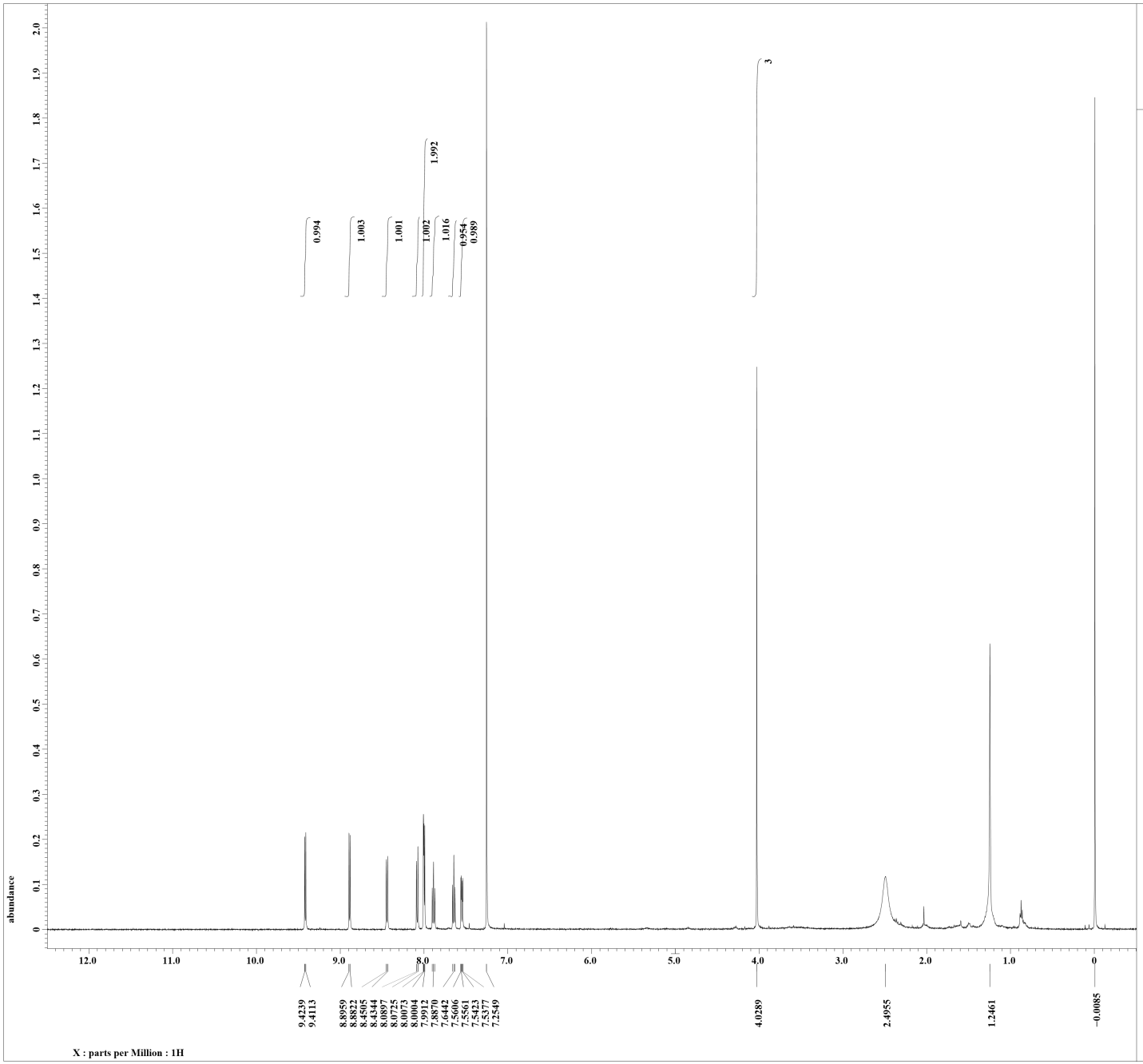
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

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             = 128
Total_scans       = 128

X_90_width        = 12.63[us]
X_acq_time        = 1.76422912[s]
X_angle           = 45[deg]
X_atn             = 3.3[dB]
X_pulse           = 6.315[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          = 23.9[dC]
```



iheyamine A

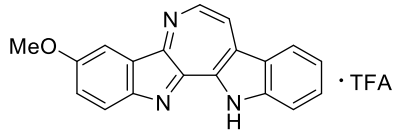



```
Filename          = Iheyamine A TFA salt
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#332718
Solvent           = CHLOROFORM-D
Creation_time     =  4-JAN-2018 23:19:58
Revision_time     = 14-JAN-2018 10:15:04
Current_time      = 14-JAN-2018 10:15:28

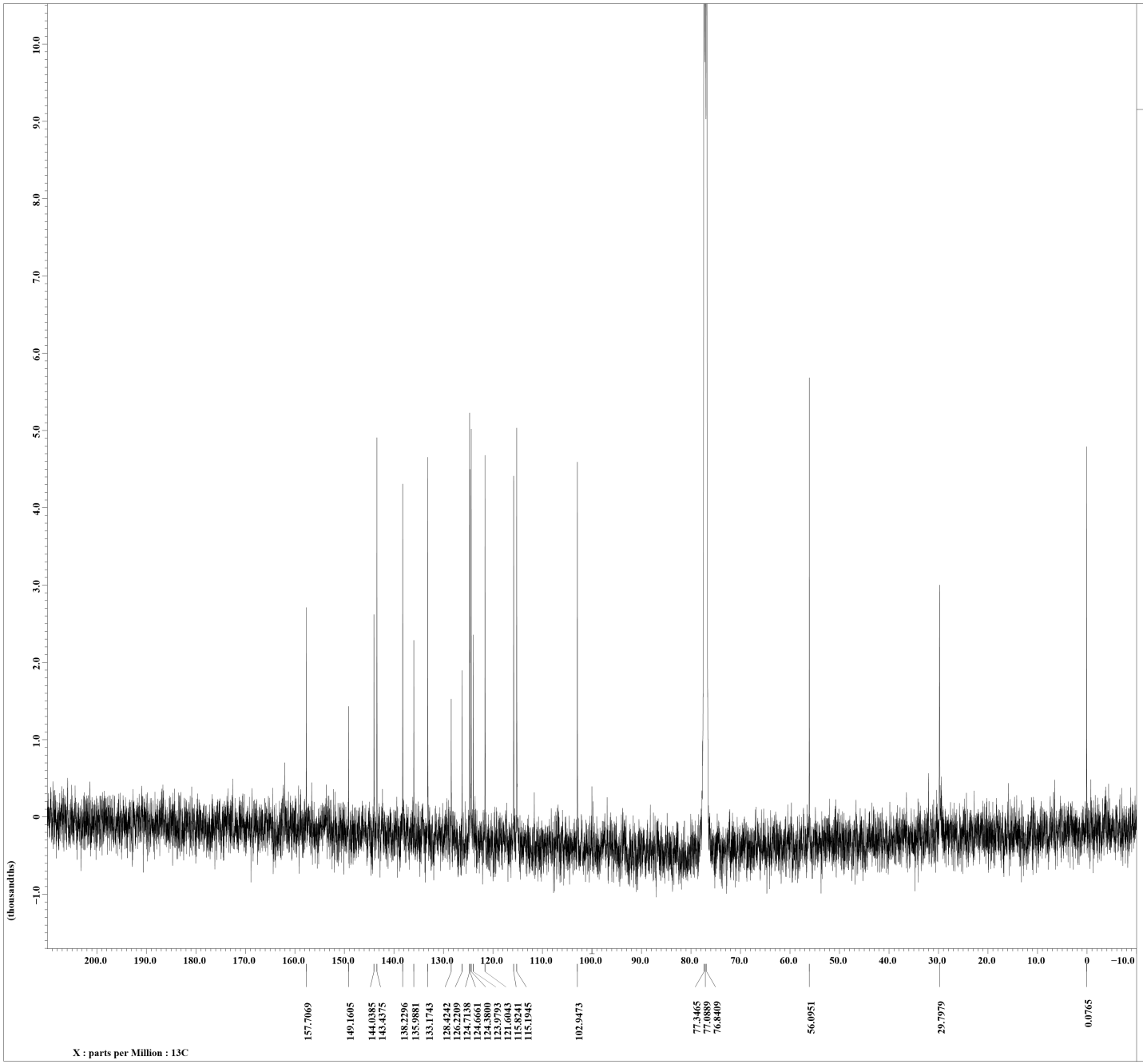
Content           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA500
Spectrometer      = DELTA2_NMR

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             = 32
Total_scans       = 32

X_90_width        = 12.39[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 3.4[dB]
X_pulse           = 6.195[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]
```



iheyamine A TFA salt

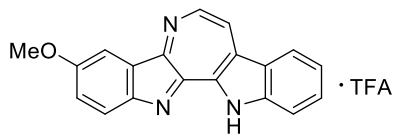



```
Filename          = Iheyamine A TFA salt
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#688955
Solvent           = CHLOROFORM-D
Creation_time     =  6-JAN-2018 05:14:17
Revision_time     = 14-JAN-2018 10:18:53
Current_time      = 14-JAN-2018 10:19:37

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
Incomplete_copy   = TRUE
Mod_return        = 1
Scans             = 25487
Total_scans       = 25487

X_90_width        = 11.43[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 5.3[dB]
X_pulse           = 3.81[us]
Irr_atn_dec       = 20.81[dB]
Irr_atn_noe       = 20.81[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          = 24.2[dC]
```



iheyamine A TFA salt