

Total Synthesis of Pyrano[3,2-*e*]indole Alkaloid Fontanesine B by Double Cyclization Strategy

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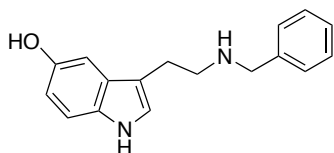
1. General Experimental

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. The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Flash column chromatography was performed on silica gel (Silica Gel 60N, Kanto Chemical Co., Ltd.).

2. Experimental Procedure

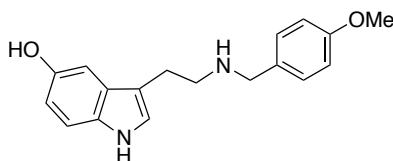
General Procedure for the Synthesis of 4

A mixture of serotonin hydrochloride (2.55 g, 12 mmol) and aldehyde (10.0 mmol) in MeOH (150 mL) was stirred at room temperature for 0.5 h. Then to the mixture was added sodium cyanoborohydride (943 mg, 15 mmol) and stirred at room temperature for 1.5 h until the complete disappearance of starting material as indicated by TLC. After MeOH was removed by evaporation, saturated NaHCO₃ (150 mL) and AcOEt (150 mL) were added the residue. The whole was extracted with AcOEt (3 x 200 mL), washed with brine (2 x 150 mL). The organic layer was dried over MgSO₄ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (CHCl₃/MeOH/Et₃N = 100/10/1) to give **4**.



3-(2-Benzylaminoethyl)-1H-indol-5-ol (**4a**).

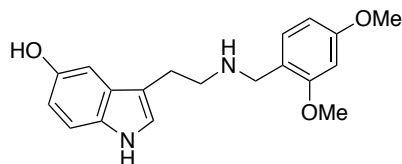
1.52 g, 57%: Pale yellow oil: IR (CHCl₃): 3296 cm⁻¹; ¹H-NMR (500 MHz, CD₃OD): δ 9.91 (br s, 1H), 7.20-7.23 (m, 2H), 7.13-7.18 (m, 4H), 6.92 (s, 1H), 6.91 (d, $J = 2.3$ Hz, 1H), 6.67 (dd, $J = 2.3, 8.6$ Hz, 1H), 3.66 (s, 2H), 3.33 (br s, 1H), 2.79-2.87 (m, 4H); ¹³C-NMR (125 MHz, CD₃OD): δ 149.9, 139.0, 131.9, 128.2, 128.0, 126.9, 123.0, 111.5, 111.3, 111.2, 102.3, 52.9, 48.8, 24.7 (three carbons are overlapped); HRMS (ESI): calcd for C₁₇H₁₉N₂O [M+H]⁺ 267.1497, found 267.1497.



3-[2-(4-methoxybenzylaminoethyl)]-1H-indol-5-ol (**4b**).

1.54 g, 52%: Colorless powder: mp 138–139 °C; IR (CHCl₃): 3481 cm⁻¹; ¹H-NMR (500 MHz, CD₃OD): δ 7.13-7.15 (m, 3H), 6.96 (s, 1H), 6.87 (d, $J = 2.3$ Hz, 1H), 6.80-6.82 (m, 2H), 6.65 (dd, $J = 2.3, 8.6$ Hz, 1H), 3.72 (s, 2H), 3.70

(br s, 1H), 3.33 (s, 3H), 2.88 (s, 4H); ^{13}C -NMR (125 MHz, CD₃OD): δ 159.3, 149.9, 131.9, 129.9, 129.6, 127.9, 123.0, 113.6, 111.4, 111.2, 110.8, 102.2, 54.3, 52.1, 48.4, 24.4 (two carbons are overlapped); HRMS (ESI): calcd for C₁₈H₂₁N₂O₂ [M+H]⁺ 297.1603, found 297.1604.

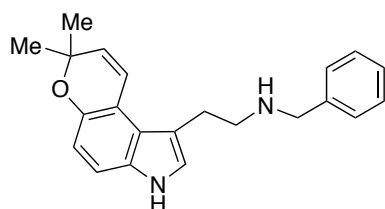


3-[2-(2,4-Dimethoxybenzylamino)ethyl]-1H-indol-5-ol (4c).

1.50 g, 46%: Colorless powder: mp 139—140 °C; IR (CHCl₃): 3479 cm⁻¹; ^1H -NMR (500 MHz, CD₃OD): δ 7.19 (t, J = 8.6 Hz, 2H), 7.09 (s, 1H), 6.85 (s, 1H), 6.70 (dd, J = 2.3, 8.6 Hz, 1H), 6.48 (dd, J = 2.3, 8.6 Hz, 1H), 6.44 (s, 1H), 4.05 (s, 2H), 3.75 (s, 3H), 3.61 (s, 3H), 3.18 (t, J = 6.9 Hz, 2H), 3.06 (t, J = 7.4 Hz, 2H); ^{13}C -NMR (125 MHz, CD₃OD): δ 162.7, 158.9, 150.3, 132.2, 131.9, 127.4, 124.0, 111.8, 111.6, 110.9, 107.8, 104.9, 101.9, 97.9, 54.7, 54.6, 46.7, 46.5, 21.9; HRMS (ESI): calcd for C₁₈H₂₁N₂O₂ [M+H]⁺ 313.1552, found 313.1553.

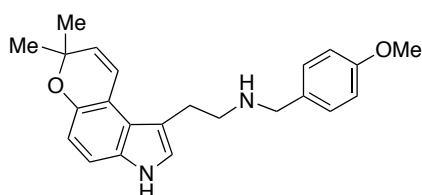
General Procedure for the C4 Pictet–Spengler/Allylic Transposition

A mixture of **4** (5 mmol) and 3-methyl-2-butenal (0.72 mL, 7.5 mmol) in 2-propanol/Et₃N (40 mL, v/v = 1/1) was heated under reflux with stirring until the complete disappearance of starting material as indicated by TLC. The mixture was cooled to rt. After addition of H₂O (80 mL), the whole was extracted with AcOEt (3 x 150 mL), washed with brine (2 x 100 mL). The organic layer was dried over MgSO₄ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane/Et₃N = 50/10/1) to give **8**.



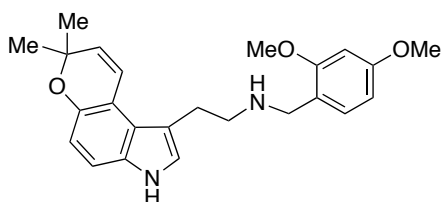
N-Benzyl-2-(7,7-dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)ethan-1-amine (8a).

1.3 g, 78%: Colorless powder: mp 110—112 °C; IR (CHCl₃): 3481 cm⁻¹; ^1H -NMR (500 MHz, CD₃OD): δ 7.17-7.24 (m, 5H), 7.05 (d, J = 8.6 Hz, 1H), 6.94 (s, 1H), 6.86 (d, J = 10.3 Hz, 1H), 6.56 (d, J = 8.6 Hz, 1H), 5.56 (d, J = 8.6 Hz, 1H), 3.75 (s, 3H), 3.69 (s, 2H), 2.98 (t, J = 7.5 Hz, 2H), 2.81 (t, J = 6.9 Hz, 2H), 1.34 (s, 6H); ^{13}C -NMR (125 MHz, CD₃OD): δ 146.1, 139.1, 133.1, 129.0, 128.2, 126.9, 124.1, 122.5, 120.2, 112.5, 112.1, 111.8, 111.2, 74.5, 52.9, 49.3, 26.8, 26.1 (four carbons are overlapped); HRMS (ESI): calcd for C₂₂H₂₅N₂O [M+H]⁺ 333.1967, found 333.1967.



2-(7,7-Dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)-*N*-(4-methoxybenzyl)ethan-1-amine (8b).

890 mg, 60%: Colorless powder: mp 97—100 °C; IR (CHCl₃): 3481 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 7.92 (br s, 1H), 7.20 (d, *J* = 9.2 Hz, 2H), 7.07 (d, *J* = 9.2 Hz, 1H), 6.94 (d, *J* = 2.3 Hz, 1H), 6.90 (d, *J* = 10.3 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 2H), 6.71 (d, *J* = 8.6 Hz, 1H), 5.60 (d, *J* = 9.8 Hz, 1H), 3.78 (s, 3H), 3.76 (s, 2H), 3.05 (t, *J* = 6.9 Hz, 2H), 2.95 (t, *J* = 6.9 Hz, 2H), 1.43 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃): δ 158.7, 146.8, 132.6, 129.7, 129.4, 123.7, 122.9, 120.3, 114.0, 113.9, 113.2, 112.9, 111.3, 74.9, 55.4, 53.4, 49.7, 27.9, 27.3 (four carbons are overlapped); HRMS (ESI): calcd for C₂₃H₂₇N₂O₂ [M+H]⁺ 363.2073, found 363.2070.

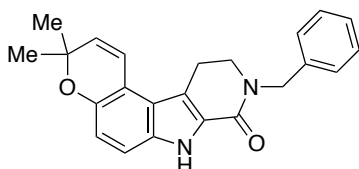


***N*-(2,4-Dimethoxybenzyl)-2-(7,7-dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)ethan-1-amine (8c).**

911 mg, 46%: Colorless powder: mp 54—56 °C; IR (CHCl₃): 3479, 3309, 1732, 1614, 1589 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 8.24 (br s, 1H), 7.08 (d, *J* = 9.2 Hz, 1H), 7.04 (d, *J* = 9.2 Hz, 1H), 6.90 (s, 1H), 6.88 (d, *J* = 10.3 Hz, 1H), 6.69 (d, *J* = 8.6 Hz, 1H), 6.37-6.39 (m, 2H), 5.57 (d, *J* = 9.7 Hz, 1H), 3.77 (s, 3H), 3.76 (s, 3H), 3.65 (s, 2H), 3.05 (t, *J* = 7.2 Hz, 2H), 2.92 (t, *J* = 6.6 Hz, 2H), 2.10 (br s, 1H), 1.43 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃): δ 160.2, 158.7, 146.7, 132.7, 130.5, 129.6, 123.9, 122.9, 120.6, 120.3, 113.8, 113.2, 112.8, 11.3, 103.7, 98.6, 74.9, 55.4, 55.2, 27.8, 27.3 (one carbon is overlapped); HRMS (ESI): calcd for C₂₄H₂₉N₂O₃ [M+H]⁺ 393.2178, found 393.2178.

General Procedure for the Carbonylative Cyclization of **8 using Triphosgene, Et₃N and HBr**

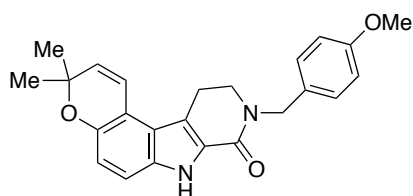
Triphosgene (149 mg, 0.5 mmol) was added to a mixture of **8** (1 mmol) and Et₃N (0.35 mL, 2.5 mmol) in toluene (100 mL) at room temperature and the mixture was heated at 70 °C with stirring for 0.5 h. After cooling to room temperature, HBr (30% in acetic acid, 0.3 mL) was added to the mixture and heated under reflux for 0.5 h. After cooling to room temperature and addition of H₂O (30 mL), the whole was extracted with AcOEt (3 x 70 mL), washed with brine (2 x 50 mL). The organic layer was dried over MgSO₄ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 3/1) to give **9** and **12**.



9-Benzyl-3,3-dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (9a).

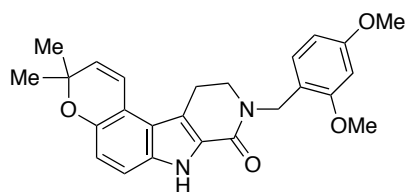
18 mg, 5%: Colorless powder: mp 254—257 °C; IR (CHCl₃): 3460, 1641 cm⁻¹; ¹H-NMR (500 MHz, DMSO-*d*₆): δ 11.50 (br s, 1H), 7.29-7.34 (m, 4H), 7.24 (t, *J* = 6.9 Hz, 1H), 7.12 (d, *J* = 8.6 Hz, 1H), 6.75 (d, *J* = 9.8 Hz, 1H), 6.68

(d, $J = 8.6$ Hz, 1H), 5.67 (d, $J = 9.8$ Hz, 1H), 4.66 (s, 2H), 3.54 (t, $J = 6.9$ Hz, 2H), 3.08 (t, $J = 6.9$ Hz, 2H), 1.33 (s, 6H); $^{13}\text{C-NMR}$ (125 MHz, DMSO- d_6): δ 161.0, 146.6, 138.5, 133.7, 130.4, 129.1, 128.4, 128.1, 127.7, 121.4, 119.8, 116.7, 115.5, 113.3, 113.2, 75.5, 49.1, 47.6, 27.5, 22.5 (three carbons are overlapped); HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 359.1760, found 359.1760.



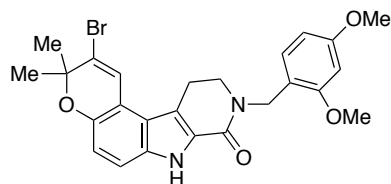
9-(4-Methoxybenzyl)-3,3-dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (9b).

24 mg, 6%: Colorless powder: mp 186—187 °C; IR (CHCl₃): 3462, 1638 cm⁻¹; $^1\text{H-NMR}$ (500 MHz, CDCl₃): δ 9.42 (br s, 1H), 7.28 (d, $J = 8.6$ Hz, 2H), 7.17 (d, $J = 9.2$ Hz, 1H), 6.86 (d, $J = 9.2$ Hz, 2H), 6.80 (d, $J = 9.2$ Hz, 1H), 6.73 (d, $J = 9.7$ Hz, 1H), 5.60 (d, $J = 9.7$ Hz, 1H), 4.72 (s, 2H), 3.80 (s, 3H), 3.60 (t, $J = 7.2$ Hz, 2H), 3.14 (t, $J = 7.5$ Hz, 2H), 1.43 (s, 6H); $^{13}\text{C-NMR}$ (125 MHz, CDCl₃): δ 161.3, 159.1, 147.1, 133.2, 130.1, 129.6, 129.4, 128.0, 121.6, 119.6, 116.9, 116.1, 114.1, 113.6, 112.5, 75.5, 55.4, 48.9, 47.1, 27.3, 22.8 (three carbons are overlapped); HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 389.1865, found 389.1868.



9-(2,4-Dimethoxybenzyl)-3,3-dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (9c).

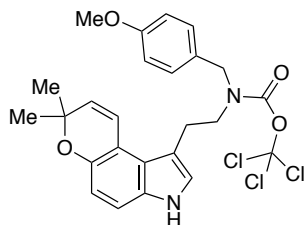
8 mg, 2%: Colorless powder: mp 209—211 °C; IR (CHCl₃): 3460, 1719, 1687, 1638, 1614 cm⁻¹; $^1\text{H-NMR}$ (500 MHz, CDCl₃): δ 9.86 (br s, 1H), 7.27 (d, $J = 8.0$ Hz, 1H), 7.15 (d, $J = 9.2$ Hz, 1H), 6.76 (t, $J = 9.5$ Hz, 2H), 6.47 (d, $J = 2.3$ Hz, 1H), 6.44 (dd, $J = 2.3, 8.6$ Hz, 1H), 5.60 (d, $J = 9.7$ Hz, 1H), 4.75 (s, 2H), 3.82 (s, 3H), 3.79 (s, 3H), 3.67 (t, $J = 7.5$ Hz, 2H), 3.14 (t, $J = 6.9$ Hz, 2H), 1.43 (s, 6H); $^{13}\text{C-NMR}$ (125 MHz, CDCl₃): δ 161.6, 160.4, 158.7, 147.0, 133.4, 130.4, 129.9, 128.3, 121.6, 119.7, 118.1, 116.8, 115.8, 113.4, 112.7, 104.3, 98.6, 75.2, 55.5, 47.5, 44.0, 27.4, 22.9 (two carbons are overlapped); HRMS (ESI): calcd for $\text{C}_{23}\text{H}_{23}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$ 419.1971, found 419.1973.



2-Bromo-9-(2,4-dimethoxybenzyl)-3,3-dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (12)

58 mg, 12%: Colorless powder: mp 189—191 °C; IR (CHCl₃): 3207, 1636 cm⁻¹; $^1\text{H-NMR}$ (500 MHz, CDCl₃): δ 9.98 (br s, 1H), 7.27 (d, $J = 8.6$ Hz, 1H), 7.19 (d, $J = 8.6$ Hz, 1H), 7.09 (s, 1H), 6.77 (d, $J = 9.2$ Hz, 1H), 6.48 (d, $J = 2.3$ Hz, 1H), 6.45 (dd, $J = 2.6, 8.1$ Hz, 1H), 4.75 (s, 2H), 3.82 (s, 3H), 3.80 (s, 3H), 3.69 (t, $J = 7.6$ Hz, 2H), 3.14 (t, $J =$

7.4 Hz, 2H), 1.54 (s, 6H); $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 161.4, 160.4, 158.7, 145.7, 133.6, 130.5, 128.6, 124.2, 123.2, 120.7, 118.0, 116.5, 115.6, 114.1, 113.2, 104.3, 98.6, 79.4, 55.5, 47.5, 44.1, 26.0, 22.7 (two carbons are overlapped); HRMS (ESI): calcd for $\text{C}_{25}\text{H}_{25}\text{BrN}_2\text{NaO}_4$ $[\text{M}+\text{Na}]^+$ 519.0895, 521.0875, found 519.0896, 521.0876.



Trichloromethyl (2-(7,7-dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)ethyl)(4-methoxybenzyl)carbamate (13b)

Triphosgene (312 mg, 1.05 mmol) was added to a mixture of **8b** (1 mmol) and Et_3N (0.35 mL, 2.5 mmol) in THF (40 mL) at room temperature and the mixture was stirring for 0.5 h. After addition of H_2O (20 mL), the whole was extracted with AcOEt (3 x 50 mL), washed with brine (2 x 30 mL). The organic layer was dried over MgSO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($\text{AcOEt}/\text{hexane} = 1/10$) to give **13b** (460 mg, 88%) as a colorless powder.

460 mg, 88%: Colorless powder: mp 83–84 °C; IR (CHCl_3): 3477, 1722 cm^{-1} ; $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 7.87, 7.91 (2 br s, 1H), 7.08–7.14 (m, 3H), 6.92 (s, 1H), 6.82–6.89 (m, 3H), 6.73 (dd, $J = 6.9, 8.6$ Hz, 1H), 5.64 (d, $J = 10.3$ Hz, 1H), 4.49 (s, 1H), 4.36 (s, 1H), 3.77, 3.78 (2s, 3H), 3.64 (t, $J = 8.0$ Hz, 1H), 3.57 (t, $J = 7.5$ Hz, 1H), 3.13 (t, $J = 8.0$ Hz, 1H), 3.10 (t, $J = 7.5$ Hz, 1H), 1.45 (s, 3H), 1.43 (s, 3H); $^{13}\text{C-NMR}$ (125 MHz, CDCl_3): δ 159.5, 159.5, 150.2, 149.3, 147.0, 146.9, 132.4, 130.2, 129.9, 128.8, 127.9, 127.5, 124.1, 124.0, 122.6, 122.5, 119.8, 119.9, 114.3, 114.2, 113.3, 113.2, 112.2, 112.0, 111.6, 111.4, 75.0, 55.4, 54.8, 52.6, 50.8, 50.5, 27.3, 26.4, 25.0 (rotamers were observed); HRMS (ESI): calcd for $\text{C}_{25}\text{H}_{25}\text{Cl}_3\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$ 523.0958, 525.0929, found 523.0953, 525.0925.

Procedure for the C2 Pictet–Spengler Reaction of 13b

A solution of compound **13b** (52 mg, 0.1 mmol) in DMSO was heated at 100 °C for 0.5 h. After removal of MeCN by evaporation, the resultant mixture was purified by silica gel column chromatography ($\text{AcOEt}/\text{hexane} = 3/1–5/1$) to give **9b** (33 mg, 86%) as a colorless powder.

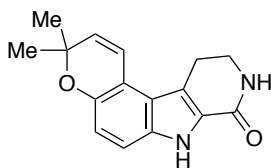
General Procedure for the the Interrupted Phosgene Cyclization/Bischler–Napieralski-type Cyclization of 8

Triphosgene (312 mg, 1.05 mmol) was added to a mixture of **8** (1 mmol) and Et_3N (0.35 mL, 2.5 mmol) in THF (40 mL) was added at room temperature and the mixture was stirring for 0.5 h. After addition of H_2O (20 mL), the whole was extracted with AcOEt (3 x 50 mL), washed with brine (2 x 30 mL). The organic layer was dried over MgSO_4 and concentrated *in vacuo*. The crude residue was used without further purification. A solution of the crude in DMSO was heated at 100 °C for 0.5 h. After removal of MeCN by evaporation, the resultant mixture was purified by silica gel column chromatography ($\text{AcOEt}/\text{hexane} = 3/1–5/1$) to give **9**.

9a: 280 mg, 78%

9b: 253 mg, 65%

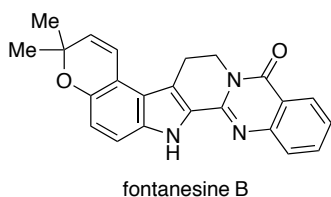
9c: 242 mg, 58%



3,3-Dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (10).

To a solution of compound **9c** (42 mg, 0.1 mmol) in toluene was *p*-toluenesulfonic acid (69 mg, 0.4 mmol) and heated at 100 °C for 16 h. After removal of toluene by evaporation, the resultant mixture was purified by silica gel column chromatography (AcOEt/hexane/CHCl₃/MeOH/28% NH₃ = 50/100/50/50/1) to give **10** (18 mg, 67%) as a colorless powder.

18 mg, 67%: Colorless powder: mp 149–150 °C; IR (CHCl₃): 3460, 3421, 3229, 1719, 1663 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ 9.09 (br s, 1H), 7.17 (d, *J* = 8.6 Hz, 1H), 6.84 (d, *J* = 9.2 Hz, 1H), 6.80 (d, *J* = 8.6 Hz, 1H), 5.73 (br s, 1H), 5.64 (d, *J* = 10.3 Hz, 1H), 3.69–3.71 (m, 2H), 3.21 (t, *J* = 6.9 Hz, 2H), 1.45 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃): δ 163.0, 147.3, 133.0, 130.3, 127.2, 121.7, 119.5, 118.7, 116.6, 113.7, 112.5, 75.5, 42.2, 27.4, 23.0 (one carbon is overlapped); HRMS (ESI): calcd for C₁₆H₁₇N₃O₂ [M+H]⁺ 269.1290, found 269.1289.



4,4-Dimethyl-4,7,8,16-tetrahydro-10*H*-pyrano[3'',2'':4',5']indolo[2',3':3,4]pyrido[2,1-*b*]quinazolin-10-one (Fontanesine B, 2).

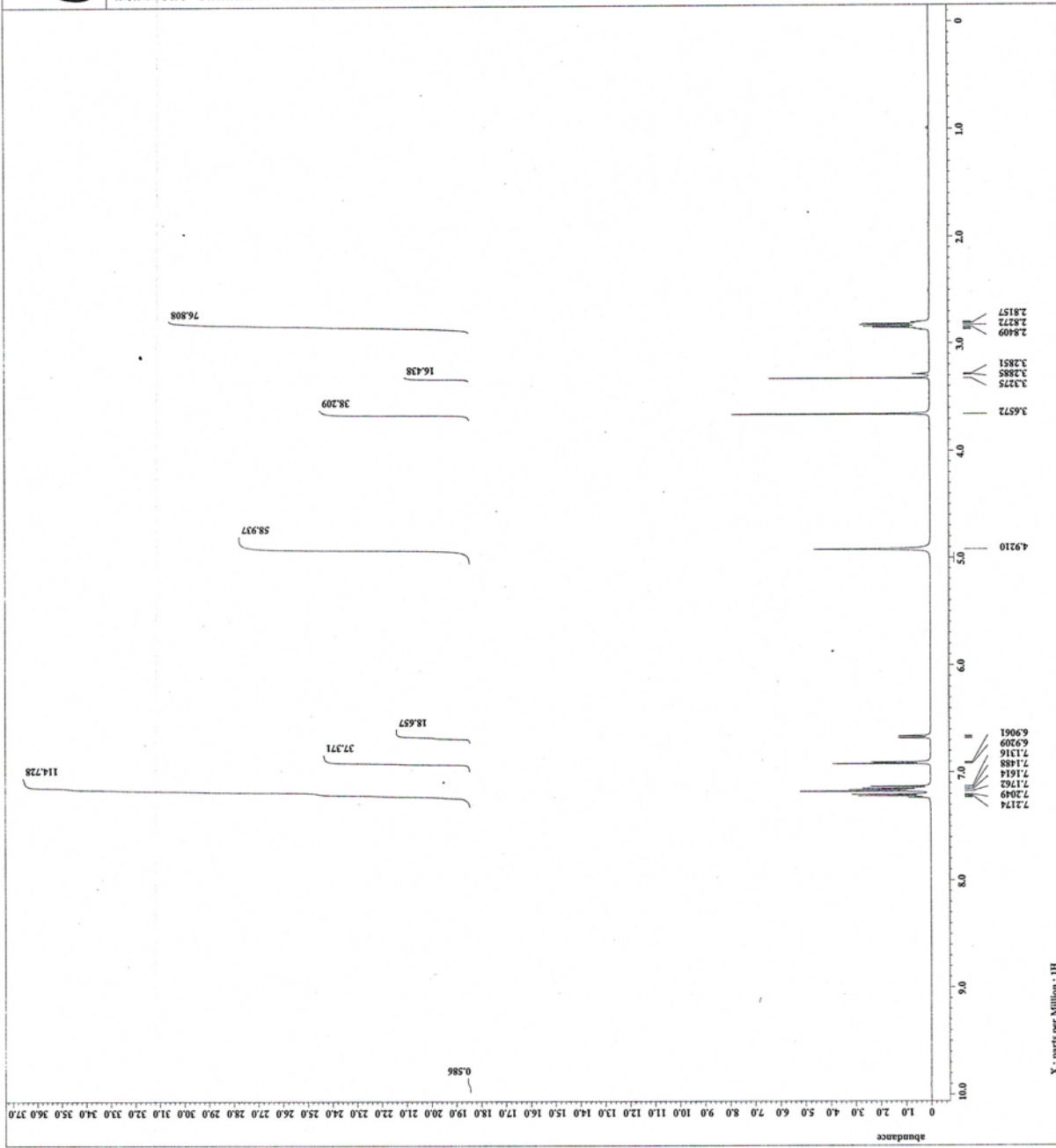
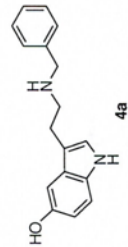
To a solution of compound **10** (13 mg, 0.05 mmol) and anthranilic acid (8 mg, 0.06 mmol) in toluene was added POCl₃ (18 mg, 0.12 mmol) at room temperature and the mixture was heated at 100 °C for 2 h. After removal of toluene by evaporation, the resultant mixture was purified by silica gel column chromatography (AcOEt/CHCl₃ = 5/1) to give **2** (14 mg, 73%) as a pale yellow powder.

pale yellow powder; mp 261–263 °C; IR (CHCl₃): 3396, 1668, 1660 cm⁻¹; ¹H-NMR (500 MHz, DMSO-*d*₆): δ 11.71 (br s, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.77 (td, *J* = 1.2, 6.5 Hz, 1H), 7.64 (d, *J* = 8.1 Hz, 1H), 7.43 (td, *J* = 1.2, 7.4 Hz, 1H), 7.12 (s, 1H), 6.93 (s, 1H), 6.53 (d, *J* = 9.7 Hz, 1H), 5.81 (d, *J* = 9.8 Hz, 1H), 4.38 (t, *J* = 6.9 Hz, 2H), 3.06 (t, *J* = 7.5 Hz, 2H), 1.34 (s, 6H); ¹³C-NMR (125 MHz, DMSO-*d*₆): δ 161.1, 148.0, 146.8, 145.8, 135.0, 130.7, 128.5, 127.1, 127.0, 126.5, 121.5, 121.2, 119.8, 117.1, 116.1, 113.3, 113.2, 75.6, 41.1, 27.5, 21.3; HRMS (ESI): calcd for C₂₃H₂₀N₄O₂ [M+H]⁺ 370.1556, found 370.1557.



```

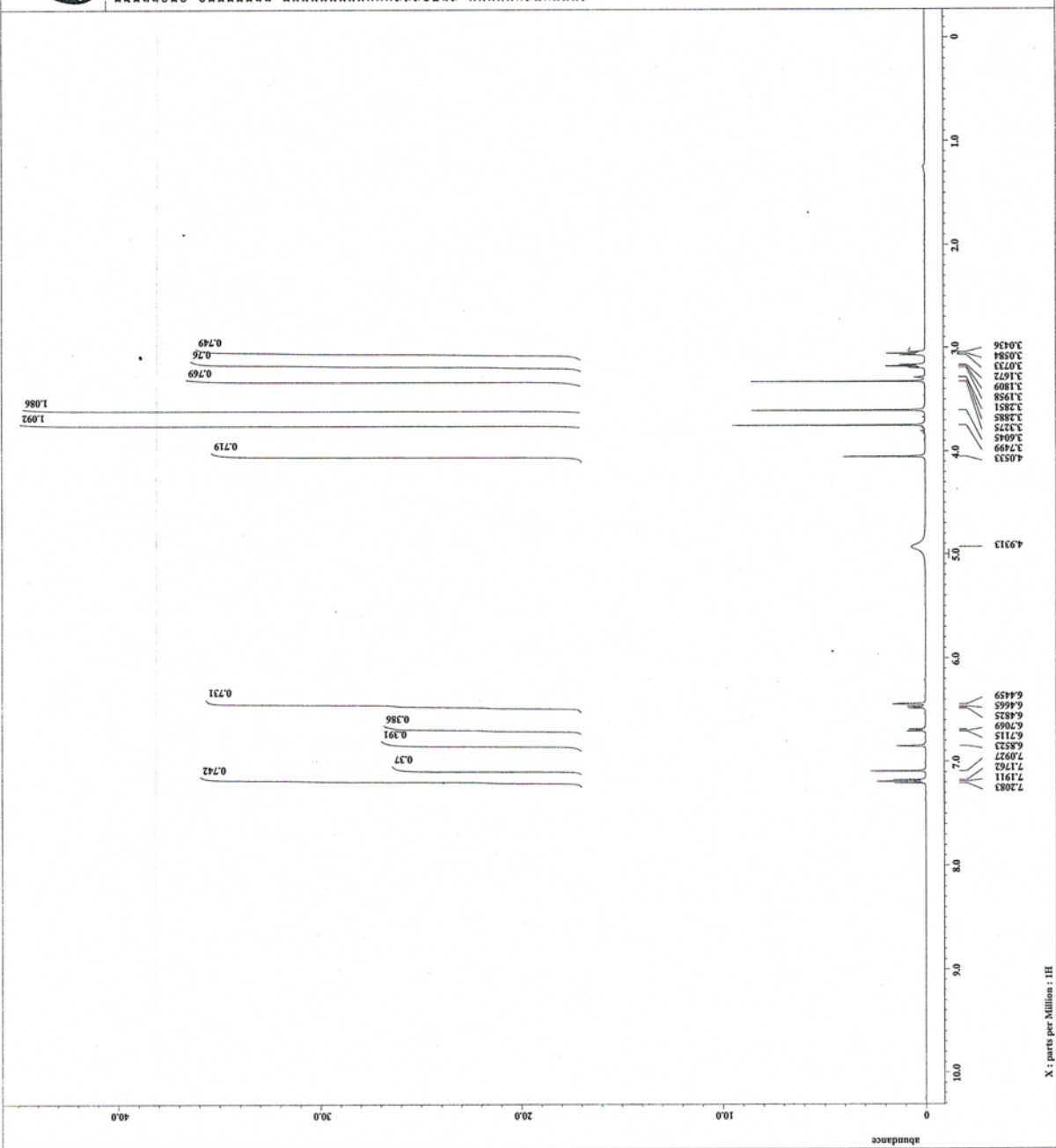
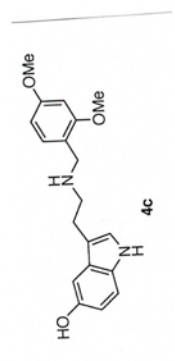
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Dimensions = X (ppm)
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Spectrometer = MZRHA_NMR
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X_domain = 11.62526421(T)
X_freq = 99.13191398(MHz)
X_points = 45384
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Irr_domain = IR 13191398(MHz)
Irr_offset = 5(ppm)
Tri_domain = IR 13191398(MHz)
Tri_offset = 5(ppm)
Clipped = FALSE
Spectrum = 16
Sols = 16
Total_scans = 16
X_90_width = 12.7(us)
X_acc_time = 1.76422912(s)
X_acq = 3.31(DS)
X_pulse = 6.35(us)
X_mode = OFF
X_waveform = OFF
Dance_preset = FALSE
Dance_preset = 10
Mhz = 500
Relaxation_delay = 5(s)
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Temperature = 22.5(C)
  
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X : parts per Million : 1H



Filename = 2016102503-4_2of
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Date_time = 11:07
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Disk_size = 1107
Dimensions = X ppm
Site = ECA 500
Spectrometer = DELTA1_00R
Field_strength = 11.62926421[G] (500[M
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X_prescans = 1
X_resolution = 5.66848[Hz]
X_sweep = 9.28677563[MHz]
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Irr_freq = 133.191398[MHz]
Irr_offset = 5[Dppm]
Tri_domain = 1H
Tri_freq = 133.191398[MHz]
Tri_offset = 5[Dppm]
Clipped = FALSE
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Total_scans = 16
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X_pulse = 6.32[us]
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Tri_mode = Off
Tri_wave = Off
Dance_preset = FALSE
Dance_preset_time = 1.00[us]
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Acquisition_time = 21.81627[us]
Temp_set = 21.81627

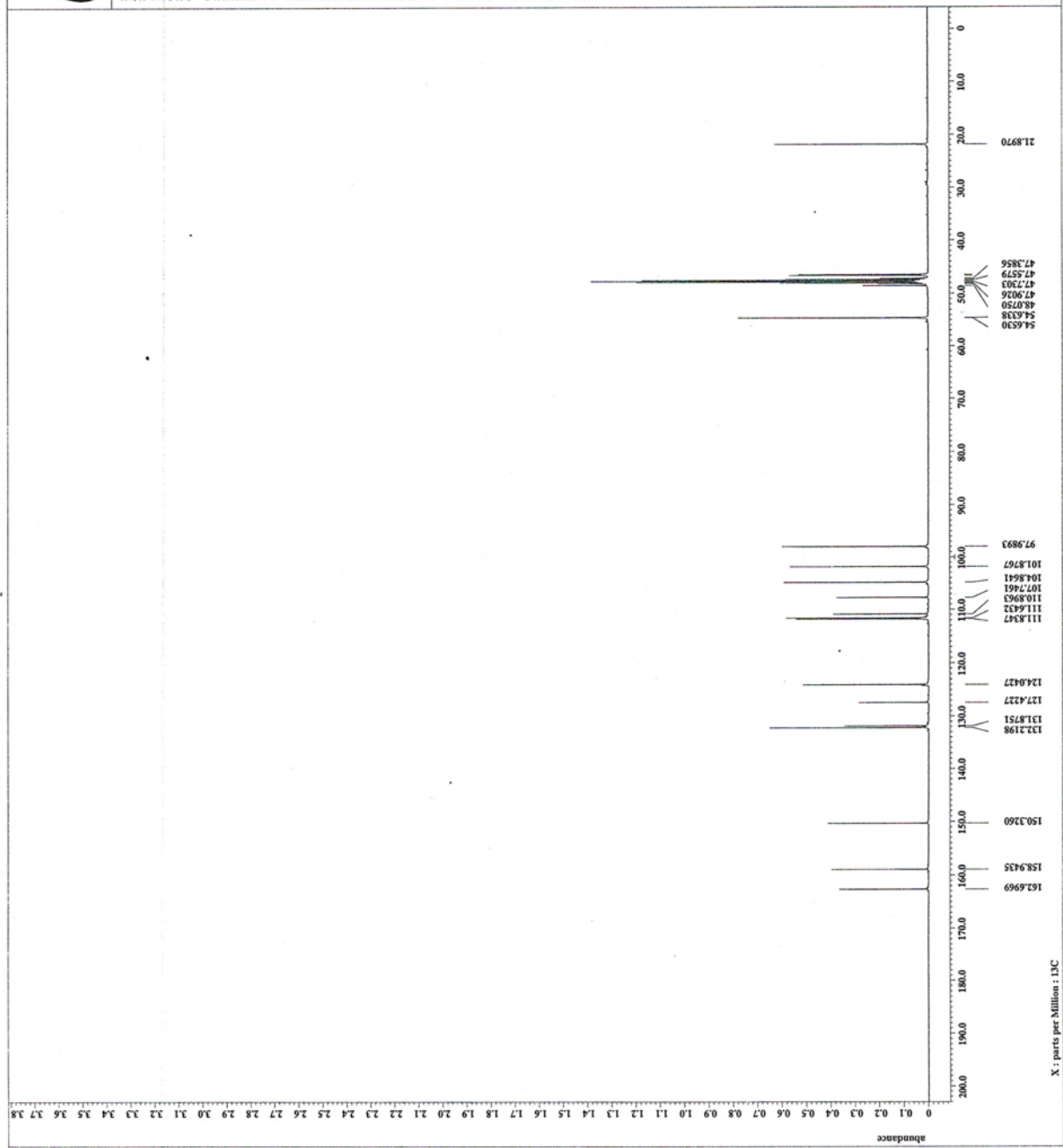
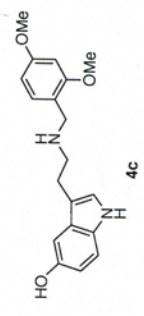




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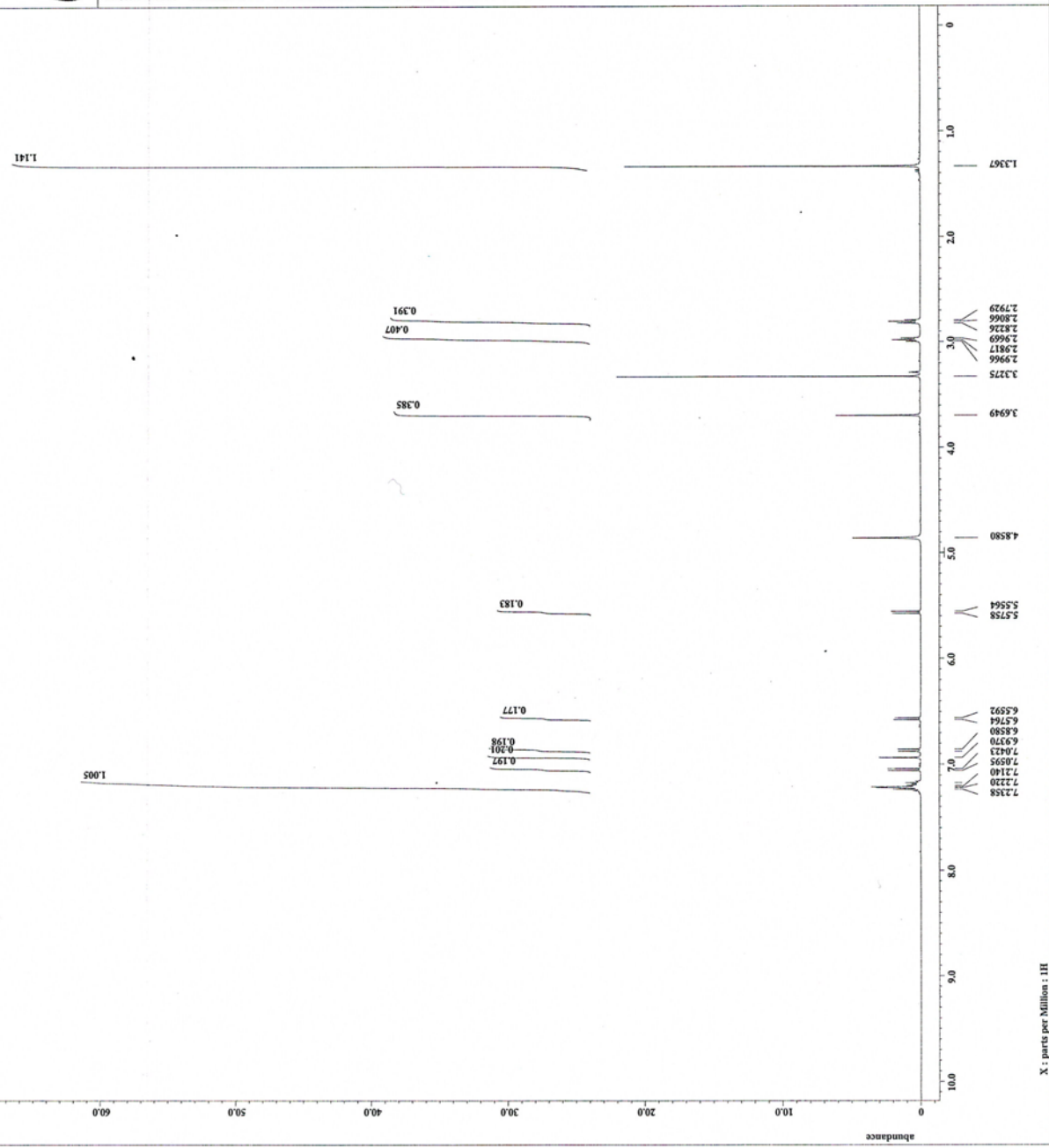
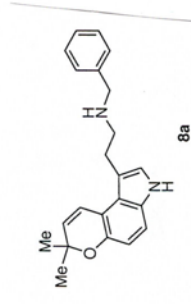
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Creation_Time = 25-OCT-2018 09:25:01
Revision_Time = 25-OCT-2018 09:25:01
Current_Time  = 25-OCT-2018 09:25:14

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X994        = 13C
X995        = 13C
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X997        = 13C
X998        = 13C
X999        = 13C
X1000       = 13C
  
```





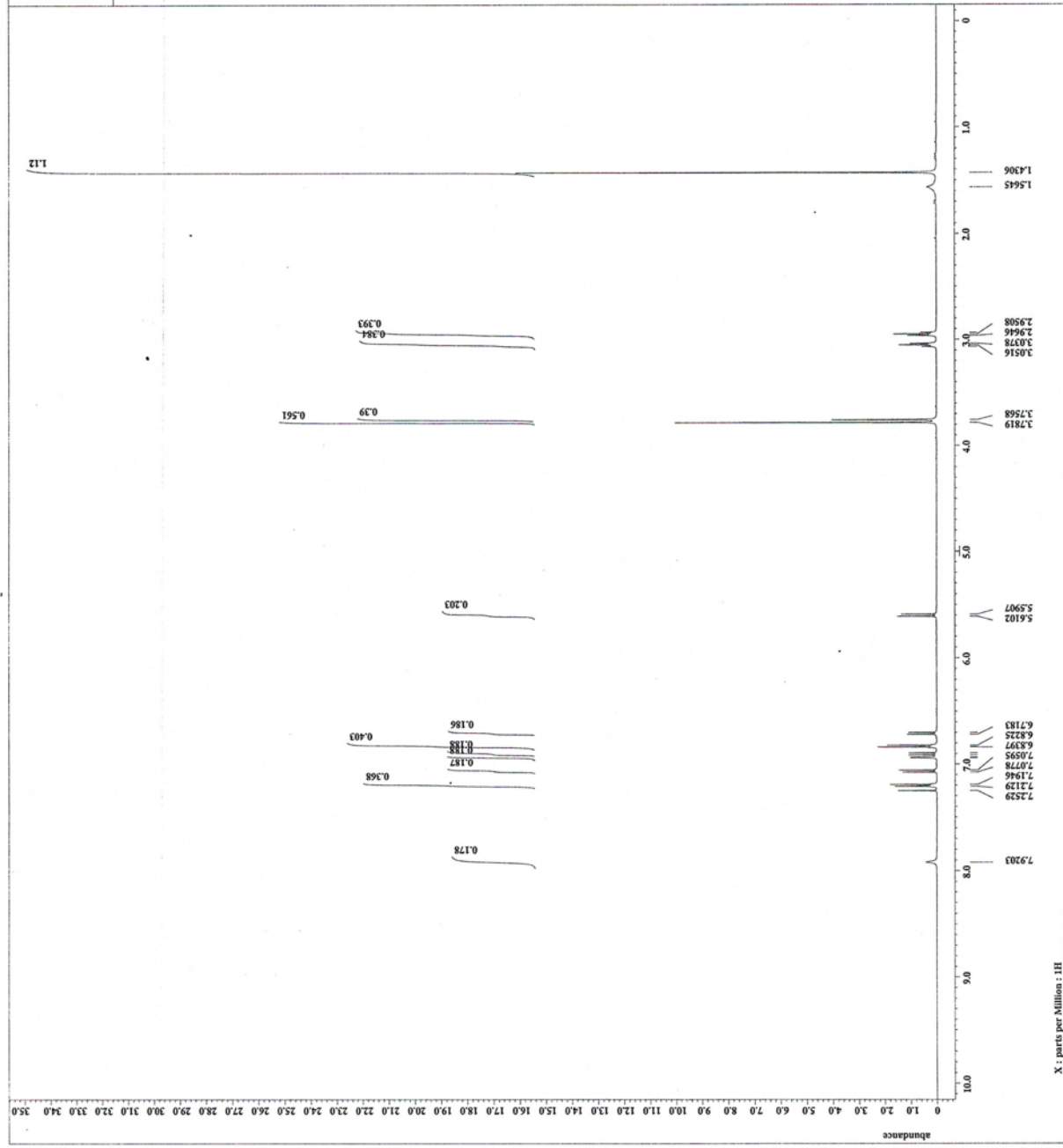
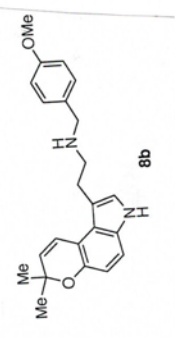
Filename = 20110901-4_3d2
Experiment = single_pulse.exe2
Sample_id = 8878178
Sample = 8-NOV-2018 18:23:55
Creation_Time = 9-NOV-2018 09:08:52
Revision_Time = 9-NOV-2018 09:08:52
Current_Time = 9-NOV-2018 09:08:52
Content = single_pulse
Dir_name = 13187
Dir_size = 131872828
Dir_title = 1H
Dimensions = X 1
Site = MCA_500
Spectrometer = MCA500_NMR
F1ld_strength = 11.62526212 [T] (500M)
X_domain = 1H 64829212 [G]
X_freq = 500.1391398 [MHz]
X_points = 6584
X_resolution = 0.2668198 [Hz]
X_swept = 5.24877563 [kHz]
ir_domain = 1H 11391398 [MHz]
ir_freq = 5 [ppm]
ir_offset = 1H 11391398 [MHz]
ir_points = 5 [ppm]
Clipped = FALSE
Scans = 16
Total_scans = 16
X_f0_width = 12.7 [us]
X_acc_time = 1.76422912 [s]
X_acq_time = 3.3 [s]
X_atq = 3.3 [s]
X_pulse = 6.35 [us]
X_rf_mode = OFF
X_rf_mode = OFF
Dance_preset = FALSE
Dance_preset = FALSE
Relaxation_delay = 5 [s]
Relaxation_delay = 22.5 [s]
Relaxation_time = 22.5 [s]
Temp_0pt = 300





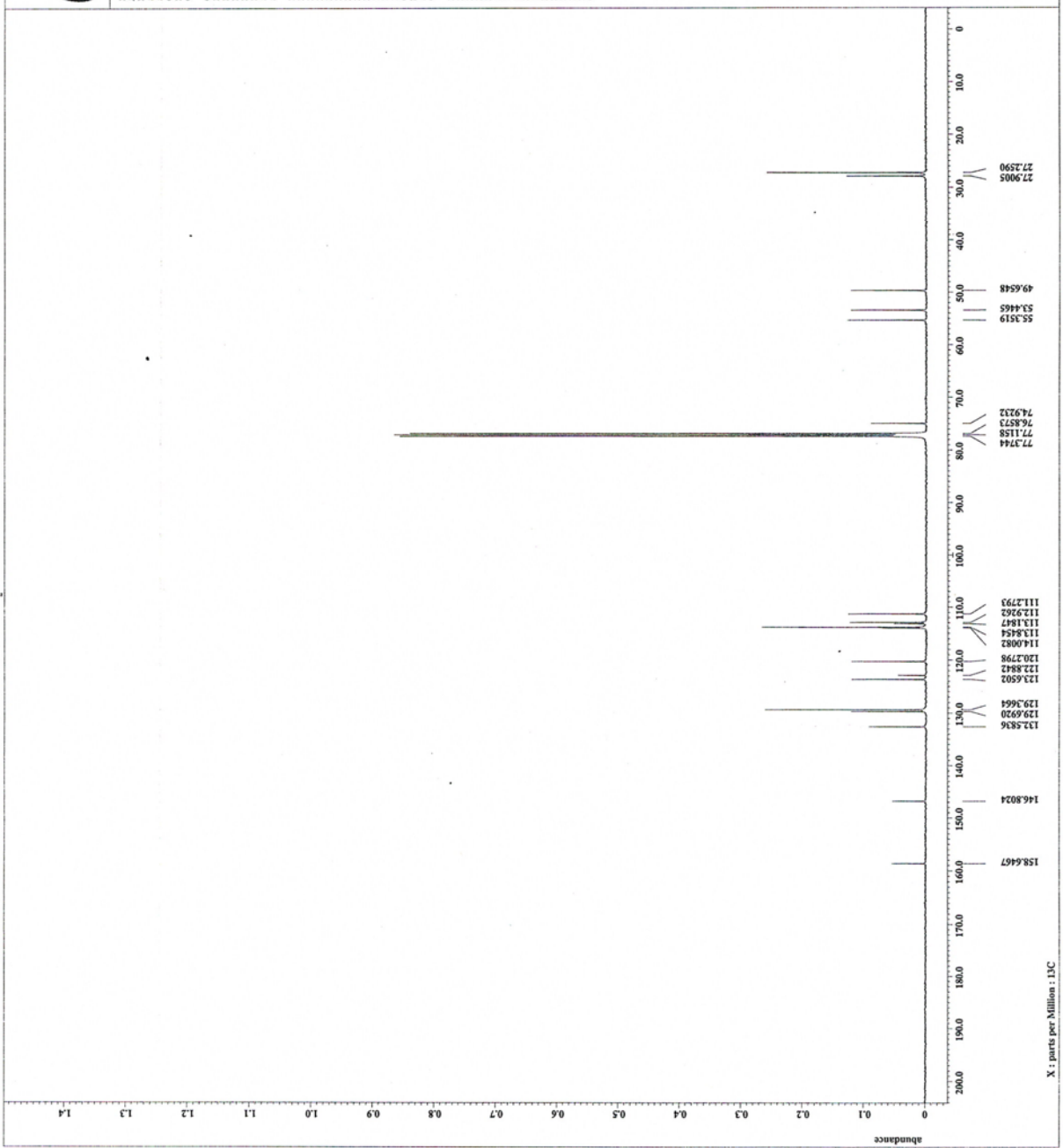
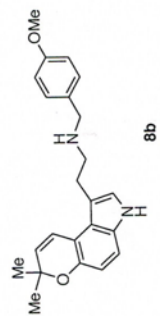
```

Filename = 201812202-5.jdf
Experiment = single_pulse.exe2
Sample_id = 8855313
Sample_name = 22-NOV-2018
Creation_time = 22-NOV-2018 24:54:27
Revision_time = 22-NOV-2018 15:32:27
Current_time = 22-NOV-2018 15:32:29
Content = single_pulse
Pulse_program = 13302PRFLX
Dir_name = IN
Dir_title = IN
Dimensions = X(1)
Site = ECA 500
Spectrometer = DELTA2_HHR
Field_strength = 11.62926421[T] (500 MHz)
X_acq_duration = IN (6.622512[s])
X_freq = 498.13131398[MHz]
X_offset = 0.00000000
X_prescans = 1
X_resolution = 0.25681616[Hz]
X_rf_domain = IN (48677453)
X_rf_freq = 498.13131398[MHz]
X_rf_offset = 0.00000000
X_rf_domain = IN (48677453)
X_rf_freq = 498.13131398[MHz]
X_rf_offset = 0.00000000
Clipped_at = FALSE
Mod_return = 1
Total_scans = 16
X_80_width = 12.7[us]
X_acq_time = 1.76422512[s]
X_angle = 45.0[deg]
X_p1 = 9.00[us]
X_pulse = 6.35[us]
X_rf_mode = off
X_rf_offset = 0.00000000
X_rf_domain = FALSE
X_rf_freq = 498.13131398[MHz]
X_rf_offset = 0.00000000
Initial_wait = 1[s]
X_rf_offset = 0.00000000
Repetition_delay = 5[s]
Repetition_time = 6.76422512[s]
Temp_set = 22.4[degC]
  
```





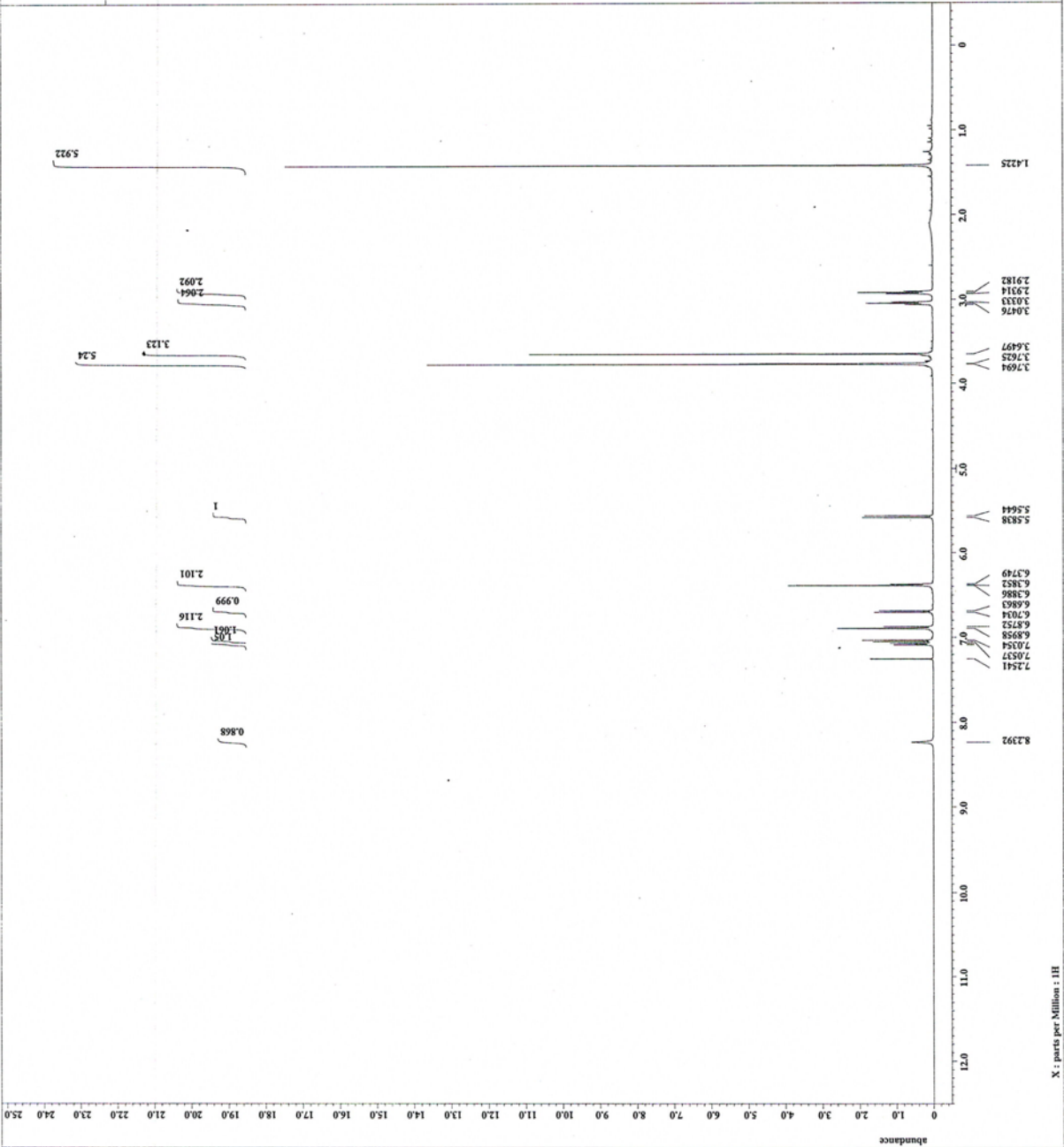
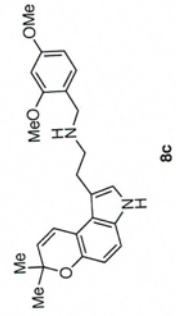
File Name = 201112102-10_jeol
Autob =
Experiment = single_pulse_dec
Sample = CML203028M-D
Creation_Time = 23-NOV-2018 09:12:58
Revision_Time = 23-NOV-2018 09:12:58
Current_Time = 23-NOV-2018 10:25:20
Concept = single pulse decouple
Dir_title =
Dir_size = 26214
Dimensions = 13C
Site = X
Spectrometer = MCA 520
Field_strength = 11.6226421[T] (500[M
X_domain = 13C
X_freq = 124.5610055[MHz]
X_points = 32748
X_prescans = 4
X_resolution = 1.828228[Hz]
X_sweep = 39.66251[MHz]
Irr_domain = 1H
Irr_offset = 11.13191398[MHz]
Clipped = TRUE
Spectrum = 18994
Stops = 18994
Total_scans = 10.6[us]
X_90_width = 0.838608[us]
X_acq_time = 5.5[us]
X_gain = 2.5333333[us]
X_pulse_dec = 20.544[us]
Irr_sun_pos = 20.544[us]
Irr_noise = 10.6[us]
Initial_wait = 10
Roc_Las = TRUE
Meovr_Gain = 60
Relaxation_delay = 2[s]
Acquisition_time = 22.31[us]
Temp_284



```

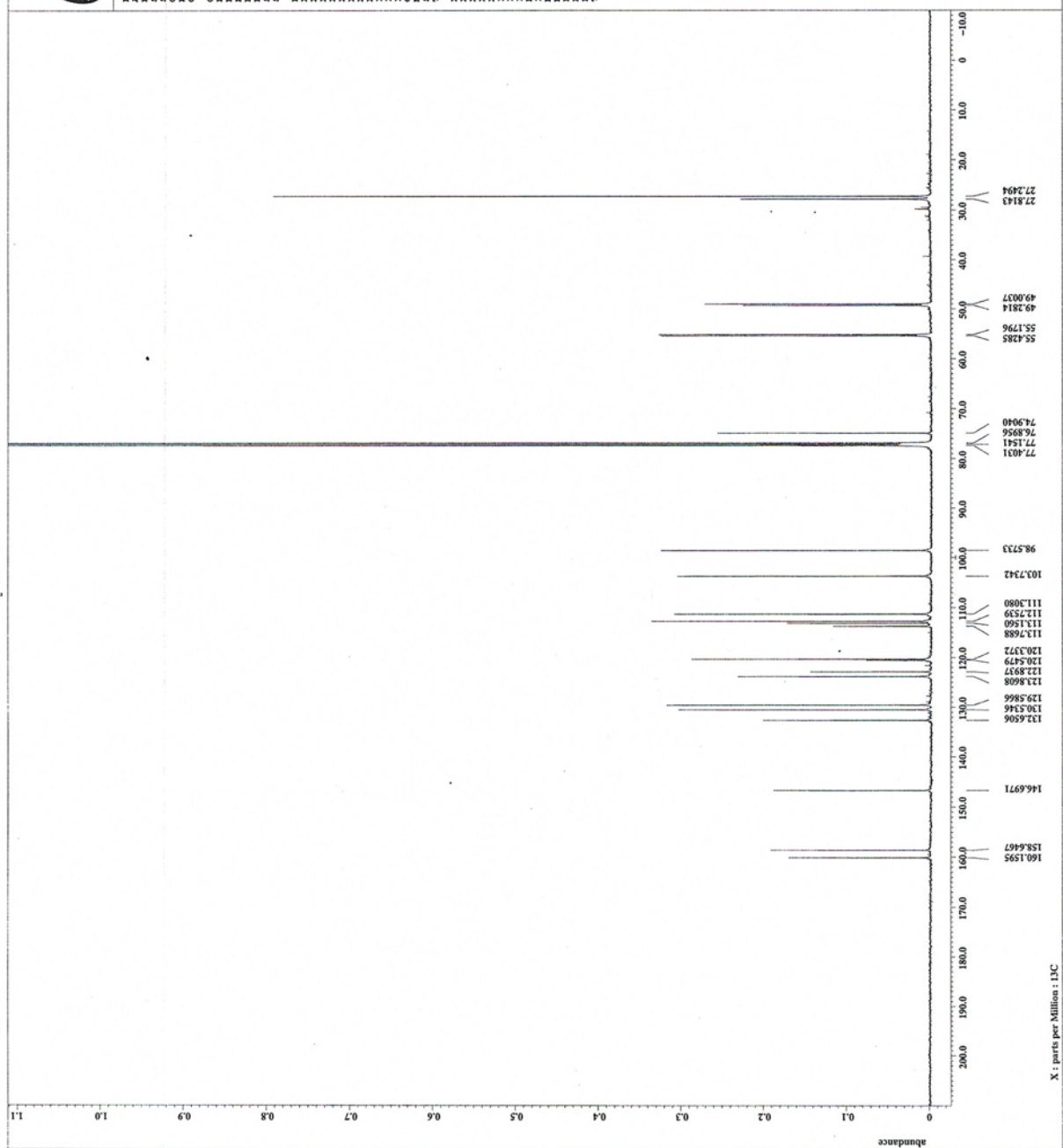
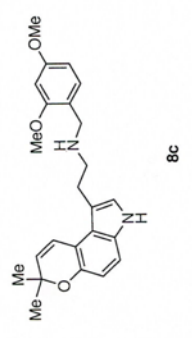
Name: 17-06-28-4_jdf
Date:
Author:
Experiment:
Pulse_program:
Solvent:
Creation_time:
Acquisition_time:
Current_time:
Operator:
Date_collected:
Date_format:
Date_size:
Date_unit:
Dimensions:
Site:
Spectrometer:
Field_strength:
X_domain:
X_center:
X_freq:
X_points:
X_resolution:
X_sweep:
Xr_domain:
Xr_offset:
Xr_offset:
Xr_domain:
Xr_offset:
Clipped:
Spectrum:
Total_scans:
X_f0_width:
X_acq_time:
X_acq:
X_gain:
X_pulse:
X_pulse:
Xr_mode:
Xr_mode:
Data_presat:
Data_presat:
Relaxation_delay:
Relaxation_delay:
Sweep_rate:

```



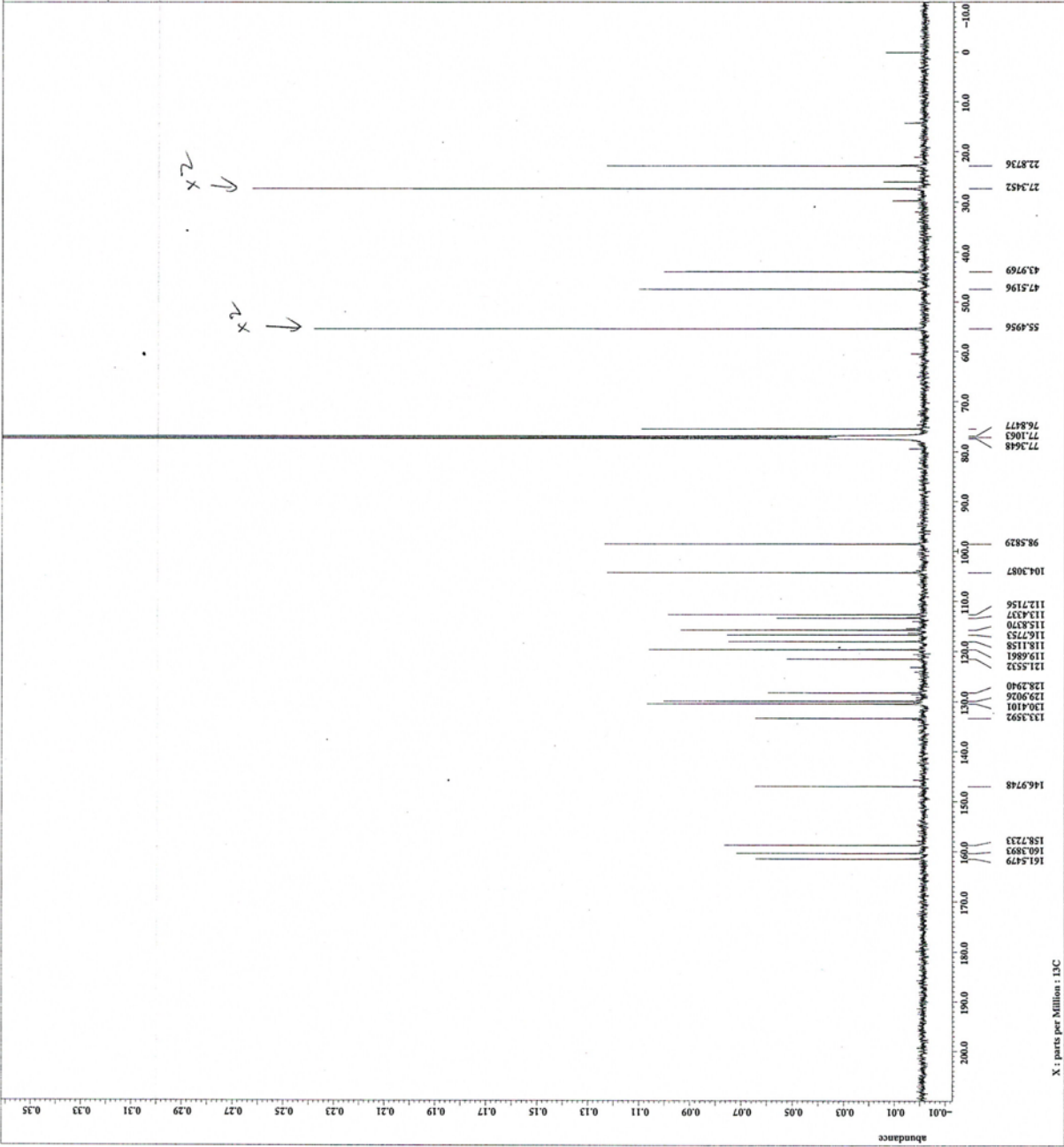
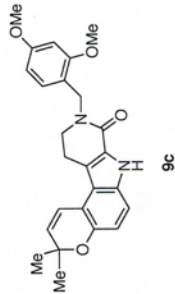


Filename = Yema-17-06-28-8.jdf
Experiment = single_pulse_dec
Sample_id = S8613160
Sample_name = 28-JUN-2017 07:00:31
Creation_time = 28-JUN-2017 07:46:41
Revision_time = 28-JUN-2017 07:46:50
Current_time = 28-JUN-2017 07:46:50
Contest = single pulse decouple
Date_acq = 20170628
Date_time = 13C
Name_of_experiment = X(ppm)
Name_of_site = ECA 500
Spectrometer = DELTAI_NMR
Field_strength = 11.629262171 (500[M]
X_coordination = 11.629262171 (500[M]
X_freq = 124.5010659 [MHz]
X_offset = 30 [ppm]
X_prescans = 4
X_resolution = 1.15755 [Hz]
X_resolution_ppm = 18.642 [ppm]
Irr_domain = 99.1191398 [MHz]
Irr_freq = 18.642 [MHz]
Irr_power = FALSE
Mod_return = 18632
Total_scans = 18632
X_90_width = 10.4 [us]
X_acq_time = 0.8388606 [s]
X_acq_time_min = 30 [sec]
X_pulse = 3.5333333 [us]
Irr_en_dec = 20.8 [dB]
Irr_noise = WALZ
Decoupling = TRUE
Irr_en_dec_wait = 2 [s]
Irr_noise_wait = 2 [s]
Irr_en_dec_wait = 2 [s]
Relaxation_delay = 2 [s]
Repetition_time = 2.8388606 [s]
Temp_unit = 21.0 [C]





Filename = Xme-18-08-21-11.jdf
Experiment = single_pulse_dec
Sample_id = 88656644
Date_acq = 21-AUG-2018 06:15:13
Creation_time = 21-AUG-2018 07:53:17
Revision_time = 21-AUG-2018 07:53:17
Current_time = 21-AUG-2018 07:53:02
Content = single pulse decouple
Dir_name = 88656644
Dir_title = 13C
Dimensions = 26214
X_name = X
Site = MCA 550
Spectrometer = MZRAC_NMR
Field_strength = 11.6282621(7) (500)M
X_domain = 13C
X_resolution = 0.398608(e)
X_freq = 124.5610095 (MHz)
X_gamma = 137.48
X_points = 4
X_prescans = 4
X_resolution = 4.82628 (Hz)
X_sweep = 39.8625 (MHz)
IR_domain = IN
IR_resolution = 11.1191398 (MHz)
IR_offset = 5 (cm)
Clipped = TRUE
Spectrum = 17290
Scan = 17290
Total_scans = 17290
X_90_width = 10.83(fus)
X_acq_time = 0.938608(e)
X_delay = 1.00(e)
X_wait = 9.5(d)
X_pulse = 1.61(us)
IR_resolution = 20.548 (cm)
IR_sweep = 20.548 (cm)
IR_noise = HALT
Decoupling = TRUE
Sib_wait = 2(e)
Sib_time = 2(e)
Relaxation_delay = 2.038608(e)
Repetition_time = 24.4(d)



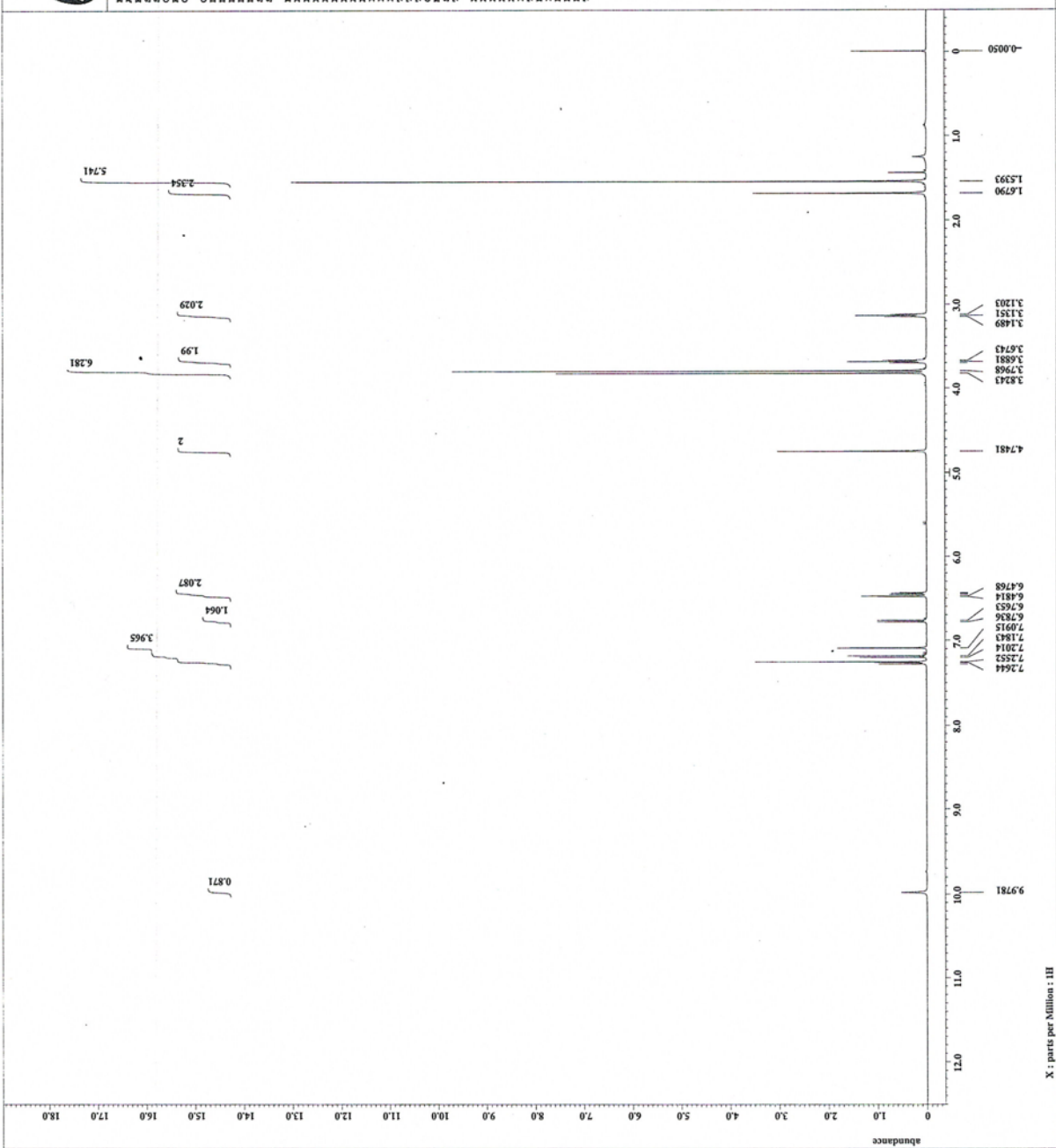
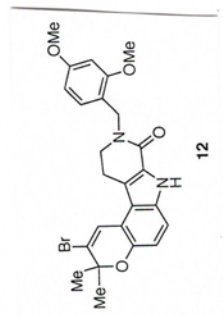


```

=====
File Name      = Yema-18-08-20-4.jdz
Sample Name    = 
Experiment     = single_pulse_002
Sample_ID      = 8845030
Date_Exp      = 20-AUG-2018 11:31:29
Creation_Time  = 20-AUG-2018 11:31:29
Revision_Time  = 20-AUG-2018 12:35:02
Current_Time   = 20-AUG-2018 12:35:22

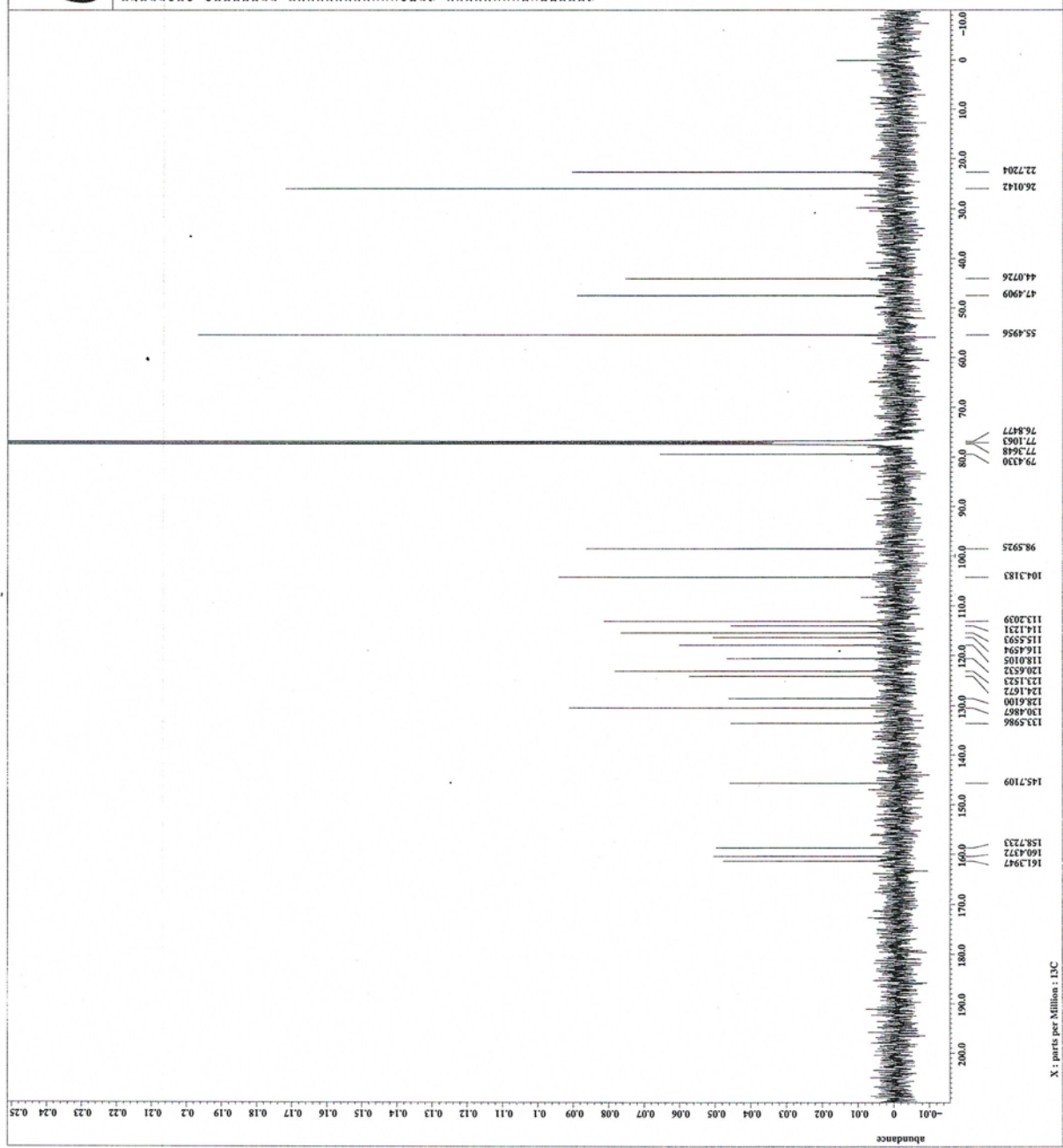
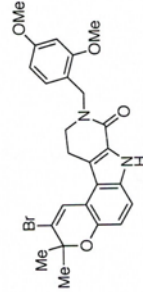
=====
Contest       = single_pulse
Date_Exp      = 20-AUG-2018 11:31:29
Date_Rev      = 20-AUG-2018 12:35:02
Date_Contest  = 20-AUG-2018 12:35:22
Diag_File     = 
Diag_Title    = 
Diag_Unit     = 
Diag_Min      = 
Diag_Max      = 
Diag_Min2     = 
Diag_Max2     = 
Diag_Min3     = 
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Diag_Max98   = 
Diag_Min99    = 
Diag_Max99   = 
Diag_Min100   = 
Diag_Max100  = 
Diag_Min101   = 
Diag_Max101  = 
Diag_Min102   = 
Diag_Max102  = 
Diag_Min103   = 
Diag_Max103  = 
Diag_Min104   = 
Diag_Max104  = 
Diag_Min105   = 
Diag_Max105  = 
Diag_Min106   = 
Diag_Max106  = 
Diag_Min107   = 
Diag_Max107  = 
Diag_Min108   = 
Diag_Max108  = 
Diag_Min109   = 
Diag_Max109  = 
Diag_Min110   = 
Diag_Max110  = 
Diag_Min111   = 
Diag_Max111  = 
Diag_Min112   = 
Diag_Max112  = 
Diag_Min113   = 
Diag_Max113  = 
Diag_Min114   = 
Diag_Max114  = 
Diag_Min115   = 
Diag_Max115  = 
Diag_Min116   = 
Diag_Max116  = 
Diag_Min117   = 
Diag_Max117  = 
Diag_Min118   = 
Diag_Max118  = 
Diag_Min119   = 
Diag_Max119  = 
Diag_Min120   = 
Diag_Max120  = 
Diag_Min121   = 
Diag_Max121  = 
Diag_Min122   = 
Diag_Max122  = 
Diag_Min123   = 
Diag_Max123  = 
Diag_Min124   = 
Diag_Max124  = 
Diag_Min125   = 
Diag_Max125  = 
Diag_Min126   = 
Diag_Max126  = 
Diag_Min127   = 
Diag_Max127  = 
Diag_Min128   = 
Diag_Max128  = 
Diag_Min129   = 
Diag_Max129  = 
Diag_Min130   = 
Diag_Max130  = 
Diag_Min131   = 
Diag_Max131  = 
Diag_Min132   = 
Diag_Max132  = 
Diag_Min133   = 
Diag_Max133  = 
Diag_Min134   = 
Diag_Max134  = 
Diag_Min135   = 
Diag_Max135  = 
Diag_Min136   = 
Diag_Max136  = 
Diag_Min137   = 
Diag_Max137  = 
Diag_Min138   = 
Diag_Max138  = 
Diag_Min139   = 
Diag_Max139  = 
Diag_Min140   = 
Diag_Max140  = 
Diag_Min141   = 
Diag_Max141  = 
Diag_Min142   = 
Diag_Max142  = 
Diag_Min143   = 
Diag_Max143  = 
Diag_Min144   = 
Diag_Max144  = 
Diag_Min145   = 
Diag_Max145  = 
Diag_Min146   = 
Diag_Max146  = 
Diag_Min147   = 
Diag_Max147  = 
Diag_Min148   = 
Diag_Max148  = 
Diag_Min149   = 
Diag_Max149  = 
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Diag_Max150  = 
Diag_Min151   = 
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Diag_Min152   = 
Diag_Max152  = 
Diag_Min153   = 
Diag_Max153  = 
Diag_Min154   = 
Diag_Max154  = 
Diag_Min155   = 
Diag_Max155  = 
Diag_Min156   = 
Diag_Max156  = 
Diag_Min157   = 
Diag_Max157  = 
Diag_Min158   = 
Diag_Max158  = 
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Diag_Max159  = 
Diag_Min160   = 
Diag_Max160  = 
Diag_Min161   = 
Diag_Max161  = 
Diag_Min162   = 
Diag_Max162  = 
Diag_Min163   = 
Diag_Max163  = 
Diag_Min164   = 
Diag_Max164  = 
Diag_Min165   = 
Diag_Max165  = 
Diag_Min166   = 
Diag_Max166  = 
Diag_Min167   = 
Diag_Max167  = 
Diag_Min168   = 
Diag_Max168  = 
Diag_Min169   = 
Diag_Max169  = 
Diag_Min170   = 
Diag_Max170  = 
Diag_Min171   = 
Diag_Max171  = 
Diag_Min172   = 
Diag_Max172  = 
Diag_Min173   = 
Diag_Max173  = 
Diag_Min174   = 
Diag_Max174  = 
Diag_Min175   = 
Diag_Max175  = 
Diag_Min176   = 
Diag_Max176  = 
Diag_Min177   = 
Diag_Max177  = 
Diag_Min178   = 
Diag_Max178  = 
Diag_Min179   = 
Diag_Max179  = 
Diag_Min180   = 
Diag_Max180  = 
=====
Spectrometer   = ECA 500
Site           = DELTA1_BOR

=====
Field_strength = 11.62926621(7) (500[M]
X_coord        = 11.76422921(6)
X_duration     = 11.76422921(6)
X_freq         = 495.13191398(MHz)
X_fwhm         = 13.994
X_offset       = 13.994
X_resolution   = 1.066445(6)
X_sweep        = 9.28677563(MHz)
X_sub          = 1.066445(6)
Irr_domain    = 1H
Irr_freq      = 595.13191398(MHz)
Irr_gate      = 1H
Tri_domain    = 1H
Tri_freq      = 595.13191398(MHz)
Tri_gate      = 1H
Clipped       = FALSE
Soc_return    = 8
Total_scans   = 8
X_90_width    = 12.63(us)
X_90_time     = 1.76422921(6)
X_acq_time    = 3.1080
X_gate        = 3.1080
X_pulse       = 6.3215(us)
Irr_mode      = Off
Tri_mode      = Off
Date_Preset   = FALSE
Date_Acquire  = 20180820
Date_Rev      = 20180820
Date_Contest  = 20180820
Relaxation_delay = 5(s)
Relaxation_time = 24.661(s)
=====
  
```



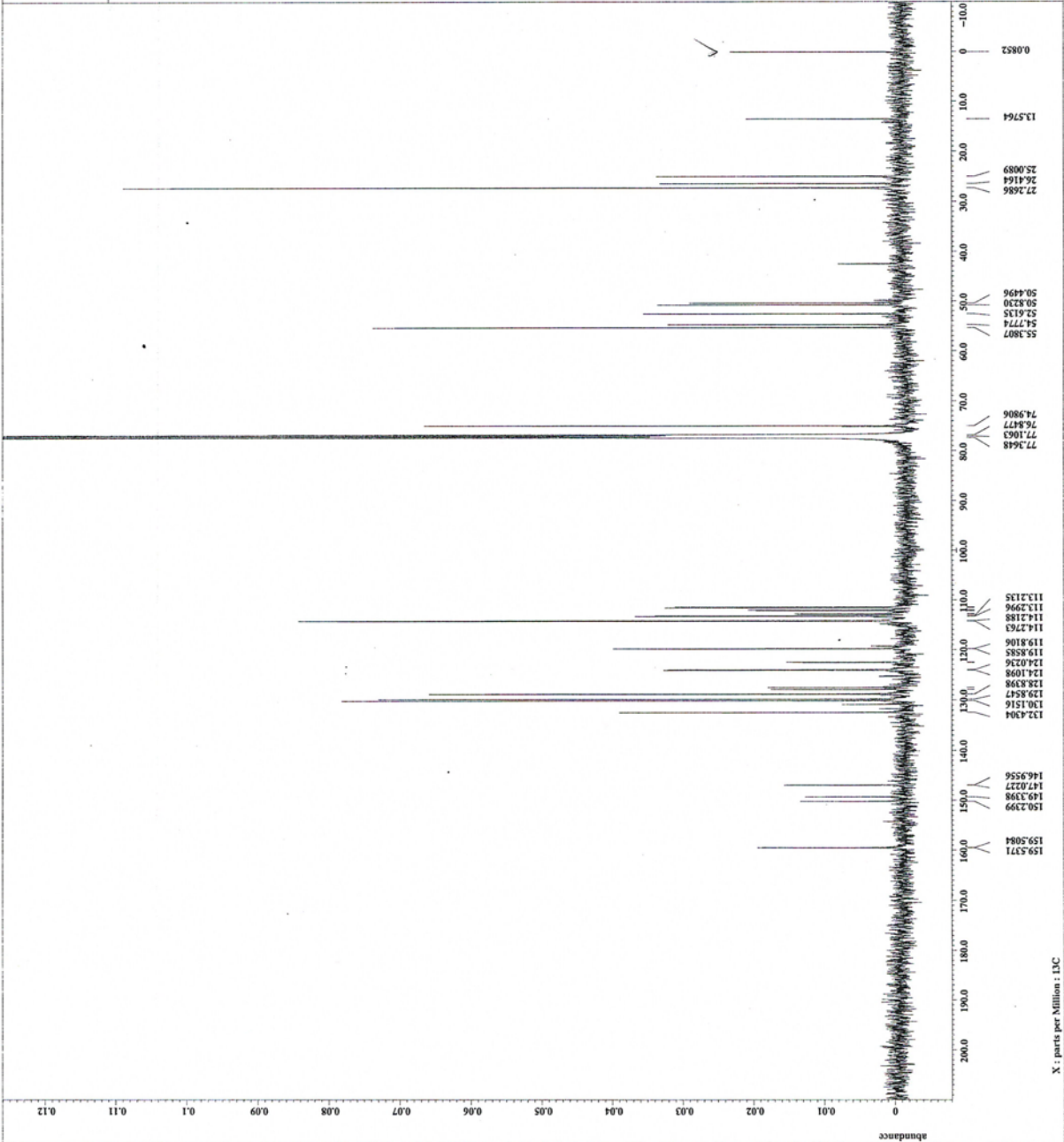
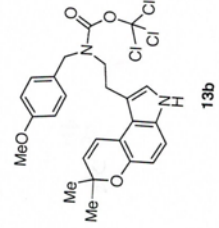


Filename * yane-18-08-20-8.dzf
Experiment * single_pulse_dec
Sample_id * 8842106
Sample_name * 18-08-20-8
Creation_time * 20-AUG-2018 11:58:45
Revision_time * 20-AUG-2018 14:02:15
Current_time * 20-AUG-2018 14:02:15
Content * single pulse decouple
Date_acq * 20180820
Date_time * 20180820
Dimensions * X (ppm)
Site * ECA 500
Spectrometer * JEOLJNMJ
Field_strength * 11.62926421[T] (500[M
X_acq * 11.62926421[T]
X_domain * 13C
X_freq * 124.9010059 [MHz]
X_gamma * 13
X_joints * 32748
X_prescans * 4
X_resolution * 4.152025 [Hz]
X_sweep * 39.0625 [MHz]
Irr_domain * 1H
Irr_freq * 16.13151398 [MHz]
Irr_offset * 5 [ppm]
Irr_pulse * TRUE
Clipped * 1800
Sofware * 1800
Total_scans * 1800
X_90_width * 10.83 [us]
X_acq_time * 0.8388606 [s]
X_delay * 9.0 [us]
X_min * 9.5 [dB]
X_max * 9.5 [dB]
X_pulse_dec * 3.6 [us]
Irr_min_pwr * 20.548 [dB]
Irr_max_pwr * 20.548 [dB]
Irr_pulse * WALTZ
Irr_phase * 1153
Initial_wait * 1 [s]
Now_alm * TRUE
Recor_gain * 60
Relaxation_delay * 2 [s]
Spectrum_time * 24.3 [sec]
Temp_get * 24.3 [C]





File Name = Yme-19-01-07-15_1d1
Experiment = single_pulse_dec
Sample_ID = 8851245
Sample_Name = 13b
Creation_Time = 8-JUN-2019 07:41:01
Revision_Time = 8-JUN-2019 08:21:59
Current_Time = 8-JUN-2019 08:21:25
Content = single pulse decouple
Pulse_Program = zgpg30
Dim_title = 13C
Dim_x1 = 13C
Dimensions = X (ppm)
Site = EUCAL 500
Spectrometer = EUCAL_500
Field_strength = 11.6226421[T] (500M)
X_coordination = 13C
X_freq = 125.5019099 [MHz]
X_gain = 32746
X_offset = 32746
X_prescan = 4
X_prescan_delay = 10.000 [us]
X_pulseprog = 1Hc_3131398 [MHz]
X_ref = 13C
X_ref_delay = 5 [ppm]
X_ref_offset = TRUE
X_ref_return = 18028
X_ref_return_delay = 18028
Total_scans = 18028
X_90_width = 19.6 [us]
X_acq_time = 0.838606 [s]
X_angle = 30 [deg]
X_delay = 30 [us]
X_pulse = 3.5333333 [us]
X_pulse_delay = 4
X_ref_delay = 20.58 [us]
X_ref_offset = 20.58 [us]
X_ref_return = 18028 [us]
X_ref_return_delay = 18028 [us]
Decoupling = TRUE
Nucleus = 13C
Nuc1 = 13C
Nuc2 = 13C
Nuc3 = 13C
Relaxation_delay = 2 [s]
Repetition_time = 2.838606 [s]
Temp_set = 29.6 [deg]



X : parts per Million : 13C



```

----- PROCESSING PARAMETERS -----
4d -> b
prog : 2.0 [Hz] : 0.0 [s]
freq : 400.131000 [MHz] : 80 [N] : 100 [N]
xpr : 0.000000 [s] : 0.000000 [s]
zfc : 1 : TRUE : TRUE
acqphase
pnm
Derived from: 201801602-7.jcf

```

```

-----
Filename      = 201801602-8.jcf
Subst         = 
Manipulation  = single_pulse_dec
Sample_id     = 88550049
Solvent       = 
Date_1        = 8-OCT-2018 10:10:18
Date_2        = 17-OCT-2018 09:18:44
Revision_time = 17-OCT-2018 09:18:44
Current_time  = 17-OCT-2018 09:19:10
Comment       = single pulse decouple
Data_format   = 2D F2
Proc_params   = 2D F2
Dim1          = 13C
Dim2          = 13C
Dim3          = 13C
Dim4          = 13C
Dim5          = 13C
Dim6          = 13C
Dim7          = 13C
Dim8          = 13C
Dim9          = 13C
Dim10         = 13C
Dim11         = 13C
Dim12         = 13C
Dim13         = 13C
Dim14         = 13C
Dim15         = 13C
Spectrometer = ECA500
Site          = DEUTZ_JMR
Field_strength = 11.7473578 [T] (500 [MH]
X_acq_duration = 0.83361792 [s]
X_resolution   = 0.83361792 [Hz]
X_freq        = 125.76529768 [MHz]
X_offset      = 100 [ppm]
X_cp         = 4
X_procs      = 4
X_res        = 1.1995004 [Hz]
X_resol      = 18.3051741 [MHz]
X_domain      = 500.1591521 [MHz]
X_start       = 500.1591521 [MHz]
X_end         = 500.1591521 [MHz]
X_clip_start  = FALSE
X_clip_end    = FALSE
Mod_return    = 1
Mod          = 1
Total_scans   = 22884
X_sb_width   = 12.2 [kHz]
X_sb         = 0.83361792 [s]
X_acq_time   = 30 [sec]
X_angle      = 90
X_pulse      = 4.06666667 [ns]
X_pulse      = 4.06666667 [ns]
Irr_atn_dec  = 20.07 [dB]
Irr_atn_dec  = 20.07 [dB]
Irr_atn_dec  = 20.07 [dB]
Irr_atn_dec  = 20.07 [dB]
Irr_atn_dec  = 20.07 [dB]
Decoupling   = TRUE
Decoupling   = TRUE
Initial_wait = 7 [s]
Initial_wait = 7 [s]
Nox_time     = 2 [s]
Nox_time     = 2 [s]
Rever_gain   = 2 [s]
Rever_gain   = 2 [s]
Repetition_time = 2.83361792 [s]
Repetition_time = 2.83361792 [s]
Temp_get     = 23.3 [C]

```

