Electronic Supplementary Material (ESI) for RSC Advances.

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

Tomoki Itoh, Yuusuke Chiba, Shunsuke Kawaguchi, Yuki Koitaya, Yuuma Yoneta, Koji Yamada and Takumi Abe*

Faculty of Pharmaceutical Sciences, Health Sciences University of Hokkaido, Ishikari-tobetsu, Hokkaido 0610293, Japan

E-mail: abe-t@hoku-iryo-u.ac.jp

SUPPORTING INFORMATION

Contents

Experimental Section

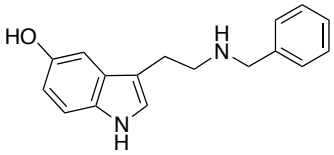
1. General Experimental

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. 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

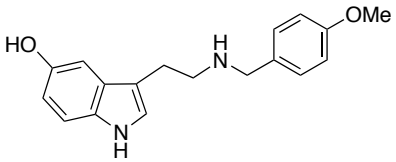
General Procedure for the Synthesis of 4

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 $NaHCO_3$ (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 $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography ($CHCl_3$/MeOH/Et_3N = 100/10/1) to give **4**.



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

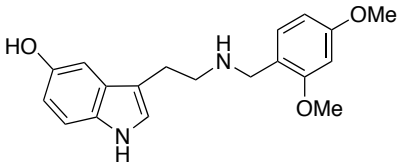
1.52 g, 57%: Pale yellow oil: IR ($CHCl_3$): 3296 cm^{-1}; ^{1}H-NMR (500 MHz, CD_3OD): δ 9.91 (br s, 1H), 7.20-7.23 (m, 2H), 7.13-7,18 (m, 4H), 6.92 (s, 1H), 6.91 (d, *J* = 2.3 Hz, 1H), 6.67 (dd, *J* = 2.3, 8.6 Hz, 1H), 3.66 (s, 2H), 3.33 (br s, 1H), 2.79-2.87 (m, 4H); ^{13}C-NMR (125 MHz, CD_3OD): 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 $C_{17}H_{19}N_2O$ $[M+H]^+$ 267.1497, found 267.1497.



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

1.54 g, 52%: Colorless powder: mp 138—139 °C; IR ($CHCl_3$): 3481 cm^{-1}; ^{1}H-NMR (500 MHz, CD_3OD): 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, CD_3OD): 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 $C_{18}H_{21}N_2O_2$ $[M+H]^+$ 297.1603, found 297.1604.

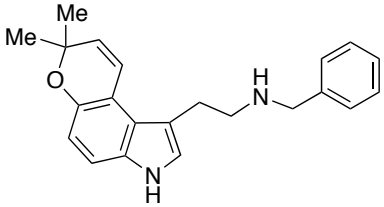


3-[2-(2,4-Dimethoxybenzylamino)ethyl]-1*H*-indol-5-ol (4c).

1.50 g, 46%: Colorless powder: mp 139—140 °C;: IR ($CHCl_3$): 3479 cm^{-1}; ^{1}H-NMR (500 MHz, CD_3OD): 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, CD_3OD): 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 $C_{18}H_{21}N_2O_2$ $[M+H]^+$ 313.1552, found 313.1553.

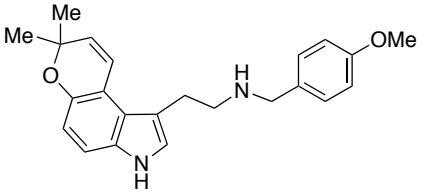
General Procedure for the C4 Pictet–Spengler/Allylic Transposition

A mixture of **4** (5 mmol) and 3-methyl-2-butenal (0.72 mL, 7.5 mmol) in 2-propanol/Et_3N (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 H_2O (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 $MgSO_4$ and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane/Et_3N = 50/10/1) to give **8**.



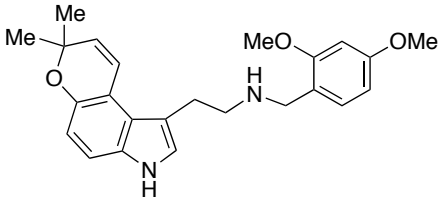
***N*-Benzyl-2-(7,7-dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)ethan-1-amine (8a).**

1.3 g, 78%: Colorless powder: mp 110—112 °C; IR ($CHCl_3$): 3481 cm^{-1}; ^{1}H-NMR (500 MHz, CD_3OD): 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, CD_3OD): 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 $C_{22}H_{25}N_2O$ $[M+H]^+$ 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 ($CHCl_3$): 3481 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): 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); ^{13}C-NMR (125 MHz, $CDCl_3$): 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 $C_{23}H_{27}N_2O_2$ $[M+H]^+$ 363.2073, found 363.2070.

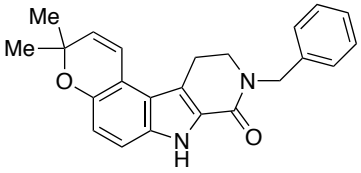


***N*-(2,4-Dimethoxybenzyl)-2-(7,7-dimethyl-3,7-dihydropyrano[3,2-*e*]indol-1-yl)ethan-1-amine (8c).**

911 mg, 46%: Colorless powder: mp 54—56 °C; IR ($CHCl_3$): 3479, 3309, 1732, 1614, 1589 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): 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); ^{13}C-NMR (125 MHz, $CDCl_3$): 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 $C_{24}H_{29}N_2O_3$ $[M+H]^+$ 393.2178, found 393.2178.

General Procedure for the Carbonylative Cyclization of 8 using Triphosgene, Et_3N and HBr

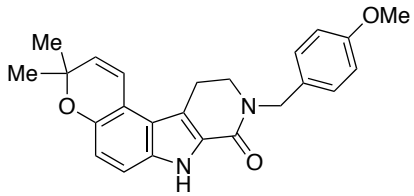
Triphosgene (149 mg, 0.5 mmol) was added to a mixture of **8** (1 mmol) and Et_3N (0.35 mL, 2.5 mmol) in toluene (100 mL) at room temperature and the mixture was heated at 70 °C with stirring for 0.5 h. After cooling to room temperature, HBr (30% in acetic acid, 0.3 mL) was added to the mixture and heated under reflux for 0.5 h. After cooling to room temperature and addition of H_2O (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 $MgSO_4$ 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).

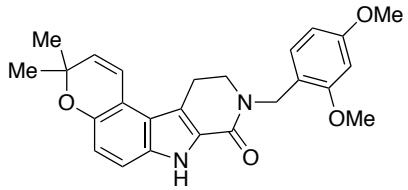
18 mg, 5%: Colorless powder: mp 254—257 °C; IR ($CHCl_3$): 3460, 1641 cm^{-1}; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 11.50 (br s, 1H), 7.29-7.34 (m, 4H), 7.24 (t, *J* = 6.9 Hz, 1H), 7.12 (d, *J* = 8.6 Hz, 1H), 6.75 (d, *J* = 9.8 Hz, 1H), 6.68

(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}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 $C_{23}H_{23}N_2O_2$ $[M+H]^+$ 359.1760, found 359.1760.



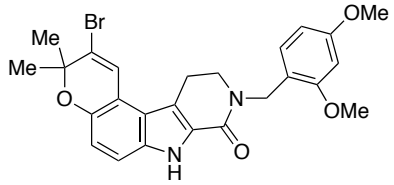
9-(4-Methoxybenzyl)-3,3-dimethyl-7,9,10,11-tetrahydropyrano[3,2-*e*]pyrido[3,4-*b*]indol-8(3*H*)-one (9b).

24 mg, 6%: Colorless powder: mp 186—187 °C; IR ($CHCl_3$): 3462, 1638 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): 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}C-NMR (125 MHz, $CDCl_3$): 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 $C_{24}H_{25}N_2O_3$ $[M+H]^+$ 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).

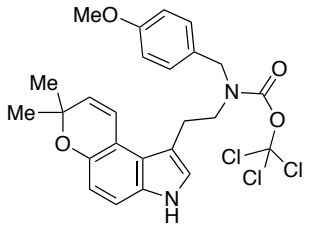
8 mg, 2%: Colorless powder: mp 209—211 °C; IR ($CHCl_3$): 3460, 1719, 1687, 1638, 1614 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): δ 9.86 (br s, 1H), 7.27 (d, *J* = 8.0 Hz, 1H), 7.15 (d, *J* = 9.2 Hz, 1H), 6.76 (t, *J* = 9.5 Hz, 2H), 6.47 (d, *J* = 2.3 Hz, 1H), 6.44 (dd, *J* = 2.3, 8.6 Hz, 1H), 5.60 (d, *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}C-NMR (125 MHz, $CDCl_3$): 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 $C_{25}H_{27}N_2O_4$ $[M+H]^+$ 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 ($CHCl_3$): 3207, 1636 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): 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}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 $C_{25}H_{25}BrN_2NaO_4$ $[M+Na]^+$ 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 (AcOEt/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}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}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 $C_{25}H_{26}Cl_3N_2O_4$ $[M+H]^+$ 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 (AcOEt/hexane = 3/1—5:1) to give **9b** (33 mg, 86%) as a colorless powder.

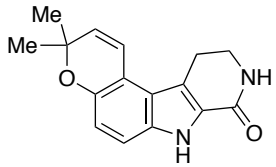
General Procedure for the the Interrupted Phosgene Cyclization/Bischler–Napieralski-type Cyclization of 8

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 (AcOEt/hexane = 3/1—5:1) to give **9**.

9a: 280 mg, 78%

9b: 253 mg, 65%

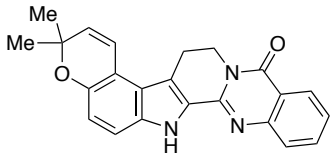
9c: 242 mg, 58%



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/$CHCl_3$/MeOH/28% NH_3 = 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 ($CHCl_3$): 3460, 3421, 3229, 1719, 1663 cm^{-1}; ^{1}H-NMR (500 MHz, $CDCl_3$): 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); ^{13}C-NMR (125 MHz, $CDCl_3$): 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 $C_{16}H_{17}N_2O_2$ $[M+H]^+$ 269.1290, found 269.1289.

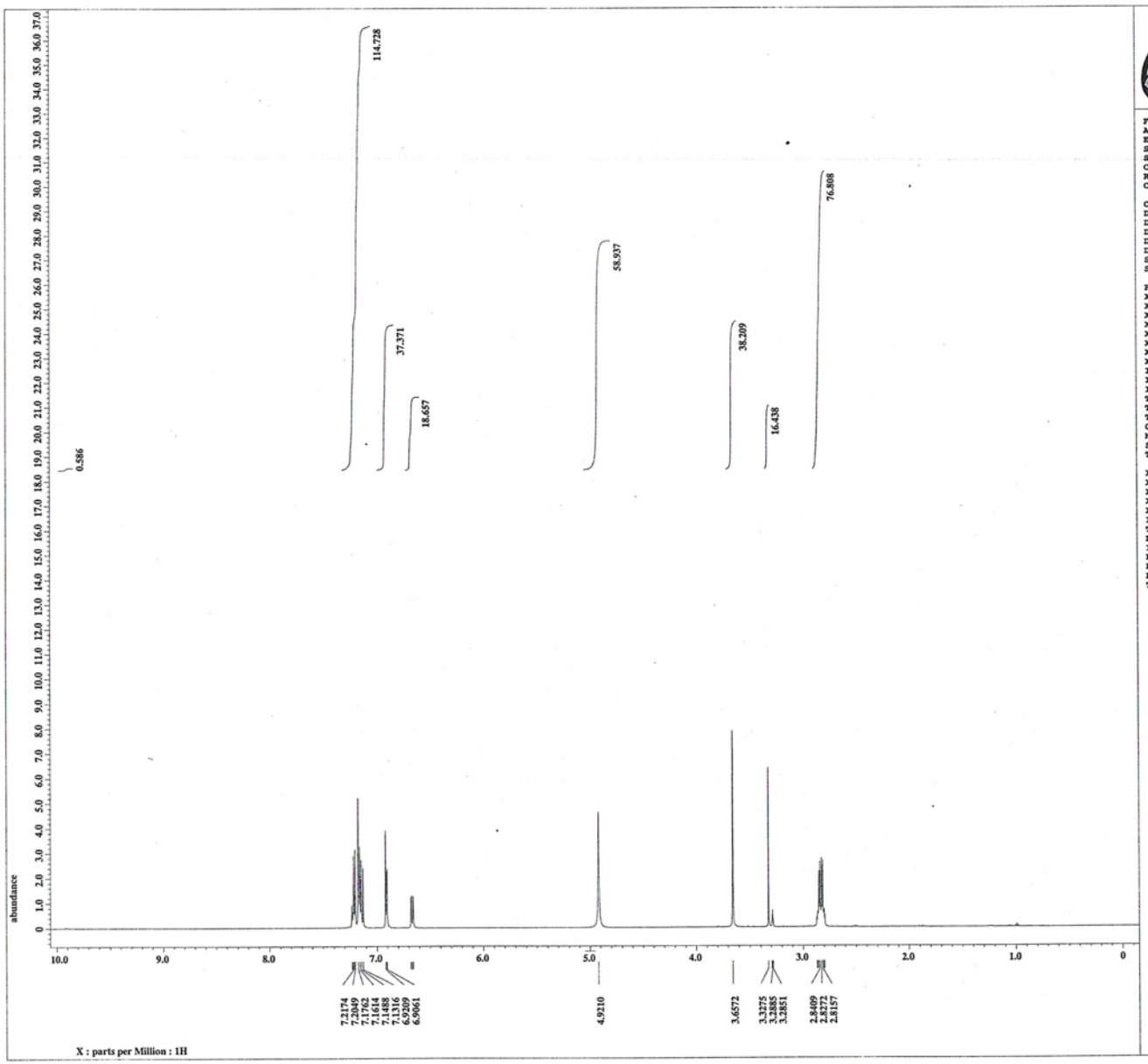


fontanesine B

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).

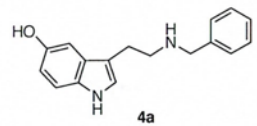
To a solution of compound **10** (13 mg, 0.05 mmol) and anthranilic acid (8 mg, 0.06 mmol) in toluene was added $POCl_3$ (18 mg, 0.12 mmol) at room temperature and the mixture was heated at 100 °C for 2 h. After removal of toluene by evaporation, the resultant mixture was purified by silica gel column chromatography (AcOEt/$CHCl_3$ = 5/1) to give **2** (14 mg, 73%) as a pale yellow powder.

pale yellow powder; mp 261—263 °C; IR ($CHCl_3$): 3396, 1668, 1660 cm^{-1}; ^{1}H-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); ^{13}C-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 $C_{23}H_{20}N_3O_2$ $[M+H]^+$ 370.1556, found 370.1557.

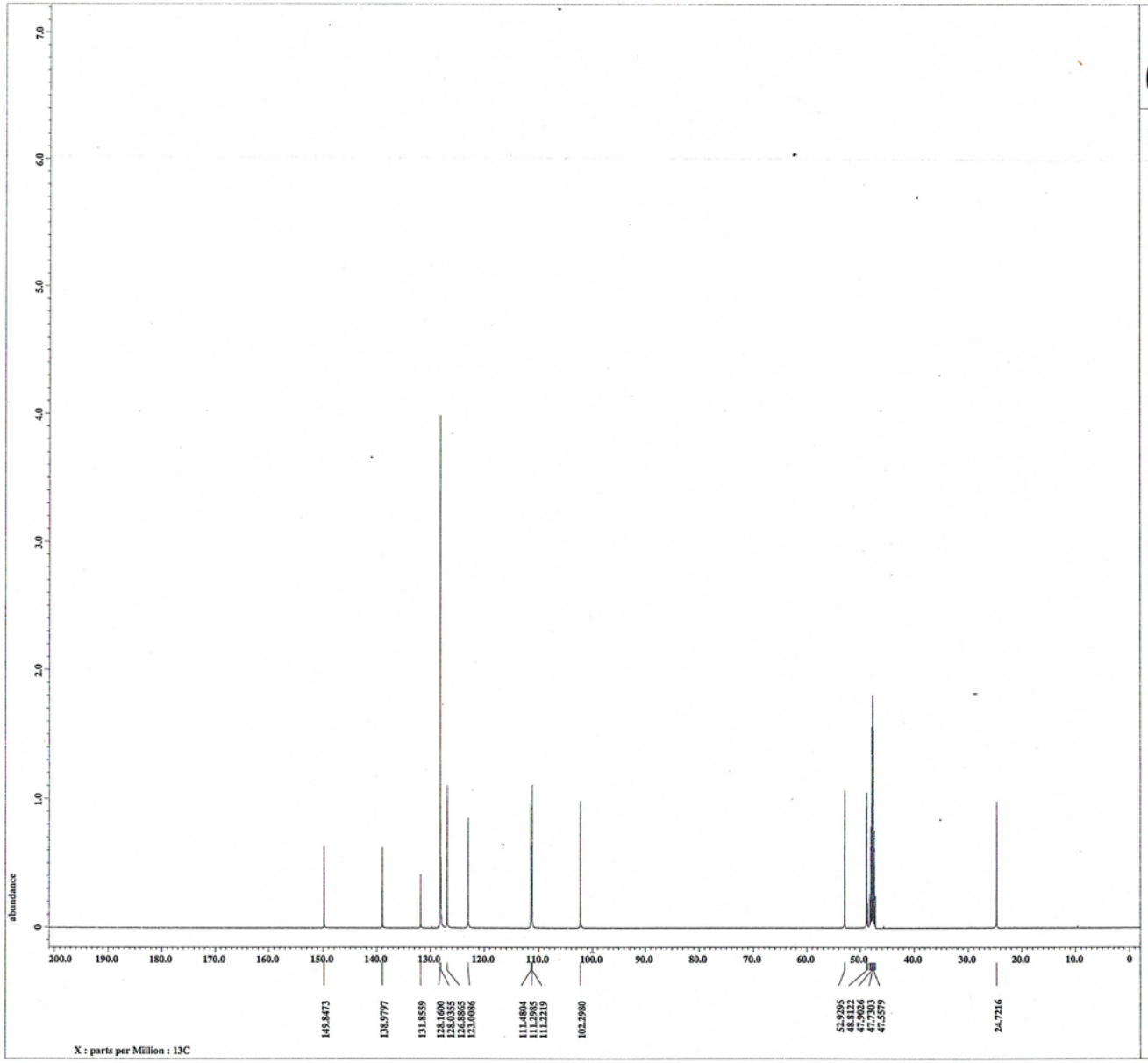


JEOL

Filename	= 20181105O1-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#639004
Solvent	= METHANOL-D3
Creation_time	= 5-NOV-2018 17:18:31
Revision_time	= 5-NOV-2018 17:53:31
Current_time	= 5-NOV-2018 17:53:36
Content	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_strength	= 11.62926421[T] (500[M
X_acq_duration	= 1.76422912[s]
X_domain	= 1H
X_freq	= 495.13191398[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.5668198[Hz]
X_sweep	= 9.28677563[kHz]
Irr_domain	= 1H
Irr_freq	= 495.13191398[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 495.13191398[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12.7[us]
X_acq_time	= 1.76422912[s]
X_angle	= 45[deg]
X_atn	= 3.3[dB]
X_pulse	= 6.35[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 6.76422912[s]
Temp_get	= 22.5[dC]

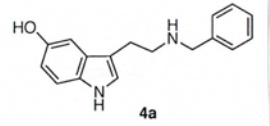


4a

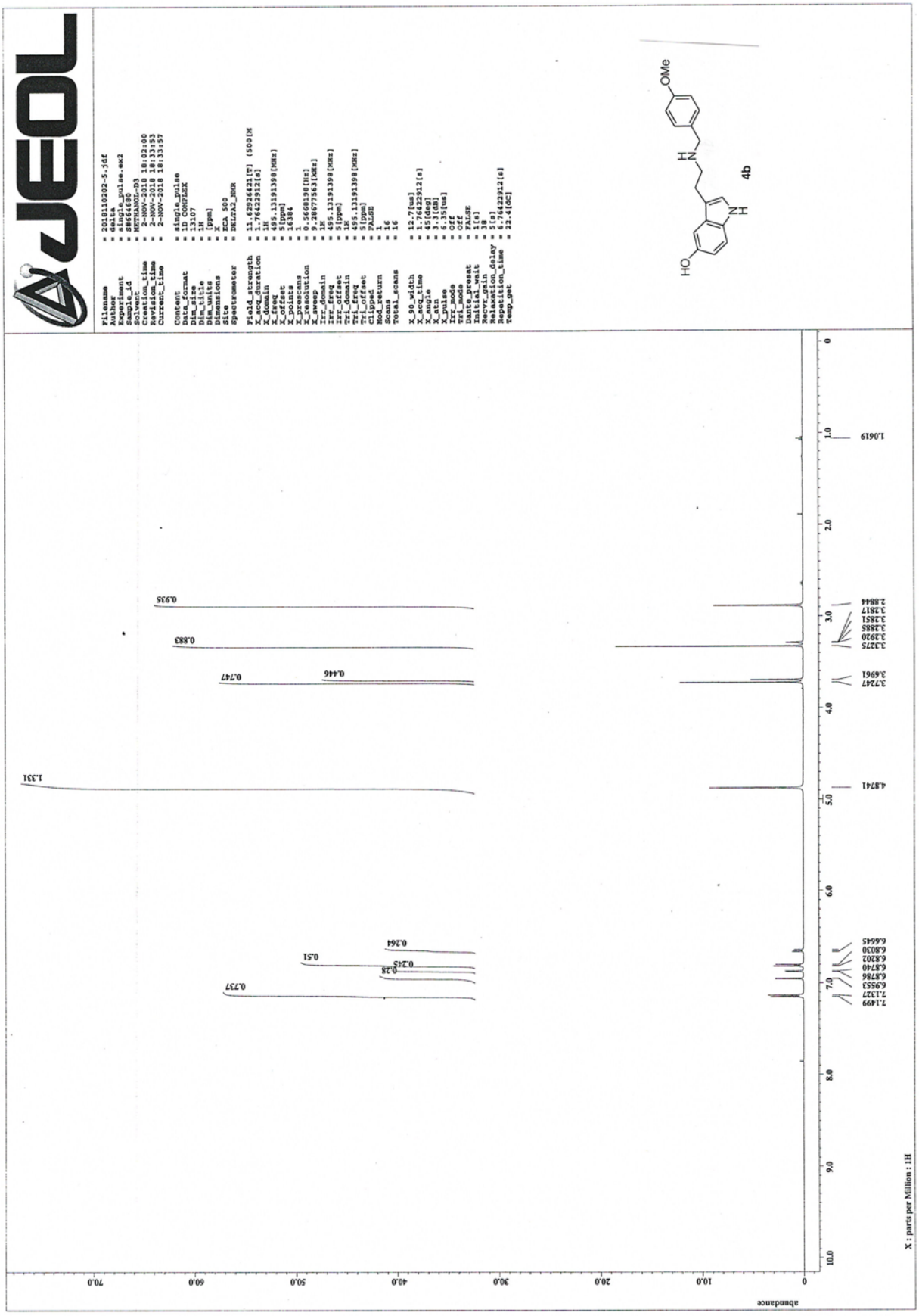


JEOL

```
Filename          = 20181105O1-10.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#718688
Solvent           = METHANOL-D3
Creation_time     =  6-NOV-2018 08:56:47
Revision_time     =  6-NOV-2018 09:26:23
Current_time      =  6-NOV-2018 09:26:37

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             = 17048
Total_scans       = 17048

X_90_width        = 10.6[us]
X_acq_time        = 0.8388608[s]
X_angle           = 30[deg]
X_atn             = 9.5[dB]
X_pulse           = 3.53333333[us]
Irr_atn_dec       = 20.54[dB]
Irr_atn_noe       = 20.54[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          = 22.8[dC]
```



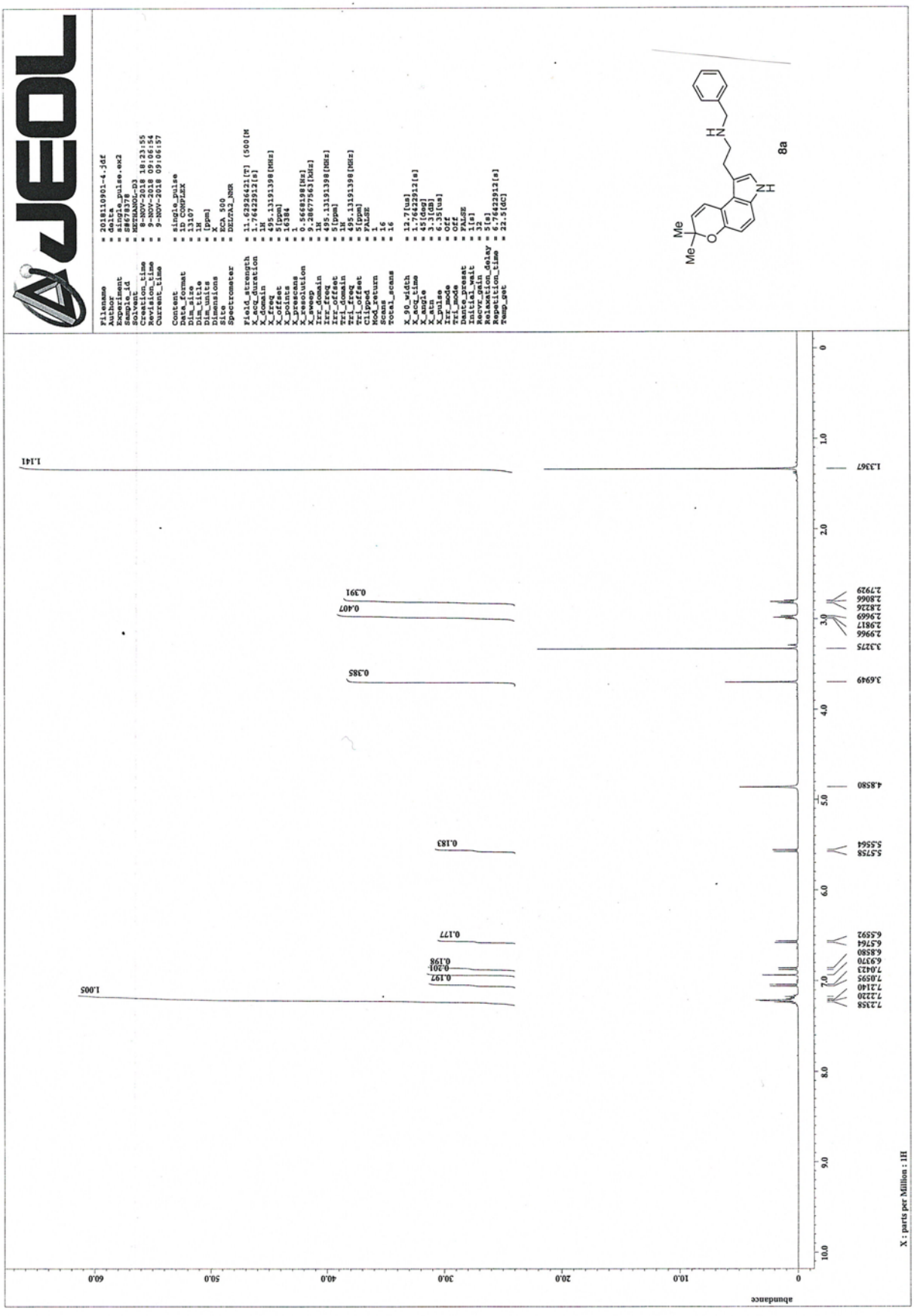
4a


JEOL
Filename = 20181010202-5.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SB664680
Solvent = MSTHOC-D3
Creation_time = 2-NOV-2018 18:02:00
Revision_time = 2-NOV-2018 18:33:53
Current_time = 2-NOV-2018 18:33:57
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 = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.566819[Hz]
X_sweep = 9.28697545[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Irr_origin = Jeol
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.7[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Repetition = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 6.764229121[s]
Temp_get = 22.4[dC]
OMe
HO
4b
abundance
X : parts per Million : 1H
1.331
0.737
0.747
0.446
0.935
0.883
0.264
0.51
0.245
0.28
4.8741
3.7247
3.6961
3.3275
3.2920
3.2885
3.2851
3.2817
2.8844
1.0619
7.1499
7.1327
6.9553
6.8786
6.8740
6.8202
6.8030
6.6645

JEOL
Filename = 20181102O2_copy-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SM333285
Solvent = DMSO-d3
Creation_time = 5-NOV-2018 09:55:33
Revision_time = 5-NOV-2018 10:25:25
Current_time = 5-NOV-2018 10:25:40
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 125.5010059[MHz]
X_offset = 100.[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.1920929[Hz]
X_sweep = 31.2625[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Irr_points = 65536
Incomplete_copy = TRUE
Mod_return = 1
Scans = 1431
Total_scans = 1431
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30.[deg]
X_pulse = 3.533333333[us]
Irr_atn_dec = 20.548[dB]
Irr_atn_noe = 20.548[dB]
Irr_mode = WALTZ16
Decoupling = TRUE
Initial_wait = 1[s]
NOE = TRUE
Noe_time = 2[s]
Recyc_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 21[DC]
OMe
HO
4b
24.3482
47.3281
47.5005
47.6728
47.8452
48.0175
48.1899
102.1544
111.5773
111.4125
111.1836
110.8293
123.0374
127.9206
129.6249
129.9314
131.8463
149.8664
159.2595
abundance
X : parts per Million : 13C

JEOL
Filename = 20181023502-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SM606577
Solvent = DMSO-D3
Creation_time = 24-OCT-2018 16:26:22
Revision_time = 24-OCT-2018 17:23:46
Current_time = 24-OCT-2018 17:23:50
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 = 1.764229312[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.566919[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 = 12.7[us]
X_acq_time = 1.764229312[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Data_format = FALSE
Irr_level = 110[]
Recvr_gain = 30
Relaxation_delay = 5[s]
Repetition_time = 6.764229312[s]
Temp_get = 21.0[DC]
OMe
OMe
H
N
H
HO
4c
abundance
X : parts per Million : 1H
0.749
0.76
0.769
1.092
1.086
0.719
0.731
0.386
0.391
0.37
0.742
7.2083
7.1911
7.1762
7.0927
6.4823
6.7115
6.7069
6.4825
6.4665
6.4459
4.9313
4.0533
3.7499
3.6045
3.3275
3.2885
3.2851
3.1958
3.1809
3.1672
3.0733
3.0584
3.0436

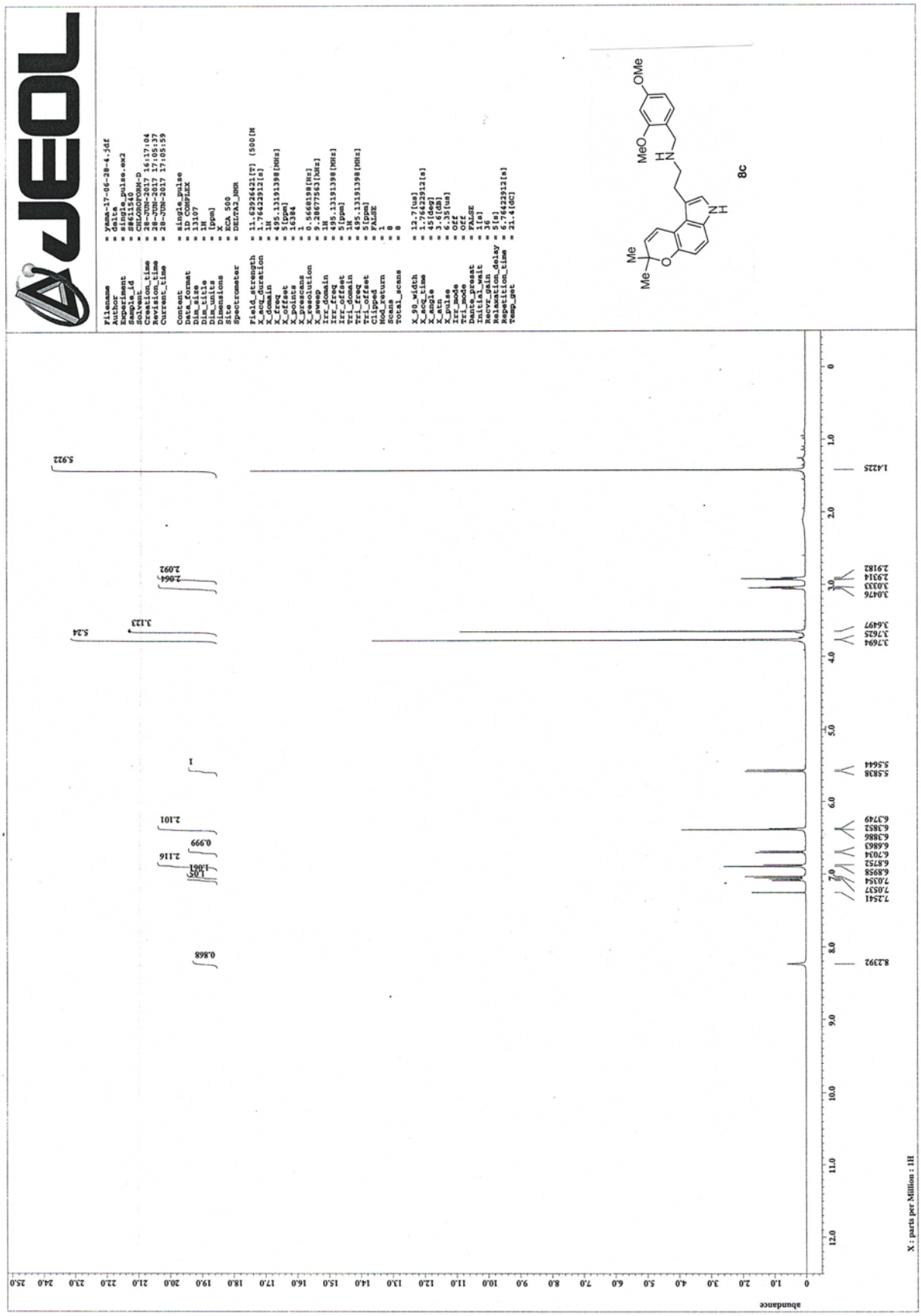
JEOL
Filename = 20181025O2-9.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SB663352
Solvent = MeOD-D3
Creation_time = 25-OCT-2018 08:57:10
Revision_time = 25-OCT-2018 09:25:01
Current_time = 25-OCT-2018 09:25:18
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
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 = 499.1311991398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 18974
Total_scans = 18974
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30.0[deg]
X_atn = 9.5[dB]
X_pulse = 3.533333333[us]
Irr_atn_dec = 20.54[dB]
Irr_atn_noe = 20.54[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repeat_time = 0.8388608[s]
Temp_get = 22.2[dC]
OMe
OMe
H
N
H
HO
4c
21.8970
47.3856
47.5579
47.7303
47.9026
48.0750
54.4638
54.6338
97.9893
101.8767
104.8641
107.7461
110.8963
111.8347
111.8412
124.0427
127.4227
132.2198
131.8751
150.1260
158.9435
162.6969
abundance
X : parts per Million : 13C


JEOL
Filename = 20181109O1-4.jdf
Author = delta
Experiment = single_pulse.exc2
Sample_id = SH678378
Solvent = CHLOROFORM-D3
Creation_time = 8-NOV-2018 18:23:55
Revision_time = 9-NOV-2018 09:06:54
Current_time = 9-NOV-2018 09:06:57
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.56668198[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 = 12.7[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 3.00[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 5[s]
Recvr_gain = 32
Relaxation_delay = 5[s]
Repetition_time = 6.764229121[s]
Temp_get = 22.5[deg]
8a
Me
Me
N
H
abundance
X : parts per Million : 1H
1.141
0.391
0.407
0.385
0.183
0.177
0.198
0.201
0.197
1.005
1.3367
2.7929
2.8066
2.8326
2.9669
2.9817
2.9966
3.3275
3.6949
4.8580
5.5564
5.5758
6.5592
6.5764
6.8580
6.9370
7.0423
7.0595
7.2140
7.2320
7.2358

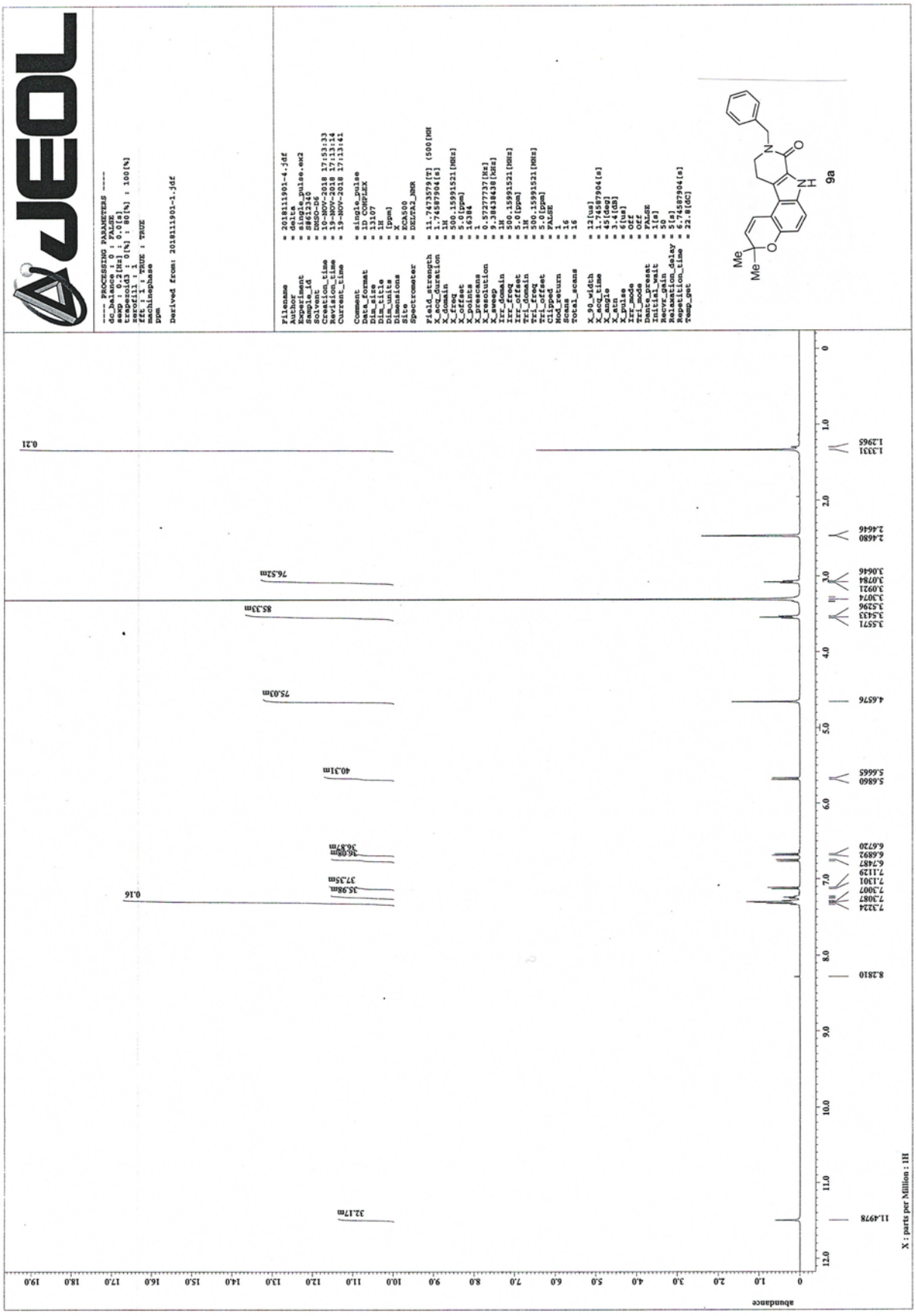
JEOL
Filename = 20181109O1-8.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 8a
Solvent = METHANOL-D3
Creation_time = 9-NOV-2018 08:32:01
Revision_time = 9-NOV-2018 09:02:07
Current_time = 9-NOV-2018 09:08:27
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 32768
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.3988608[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 = 495.1131913398[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 17150
Total_scans = 17150
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 9.5[dB]
X_pulse = 3.533333333[us]
Irr_atn_dec = 20.54[dB]
Irr_atn_noe = 20.54[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Mode = TRUE
Mod_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
X_wait_time = 22.4[dC]
Temp_get
Me
Me
O
N
H
N
H
8a
abundance
X : parts per Million : 13C
146.1226
139.1137
133.1390
129.0121
128.1600
126.8578
124.0906
122.5203
120.1936
112.0453
111.8059
111.2314
74.4540
48.0558
47.8835
47.7111
47.5388
47.3760
26.7802
26.1004

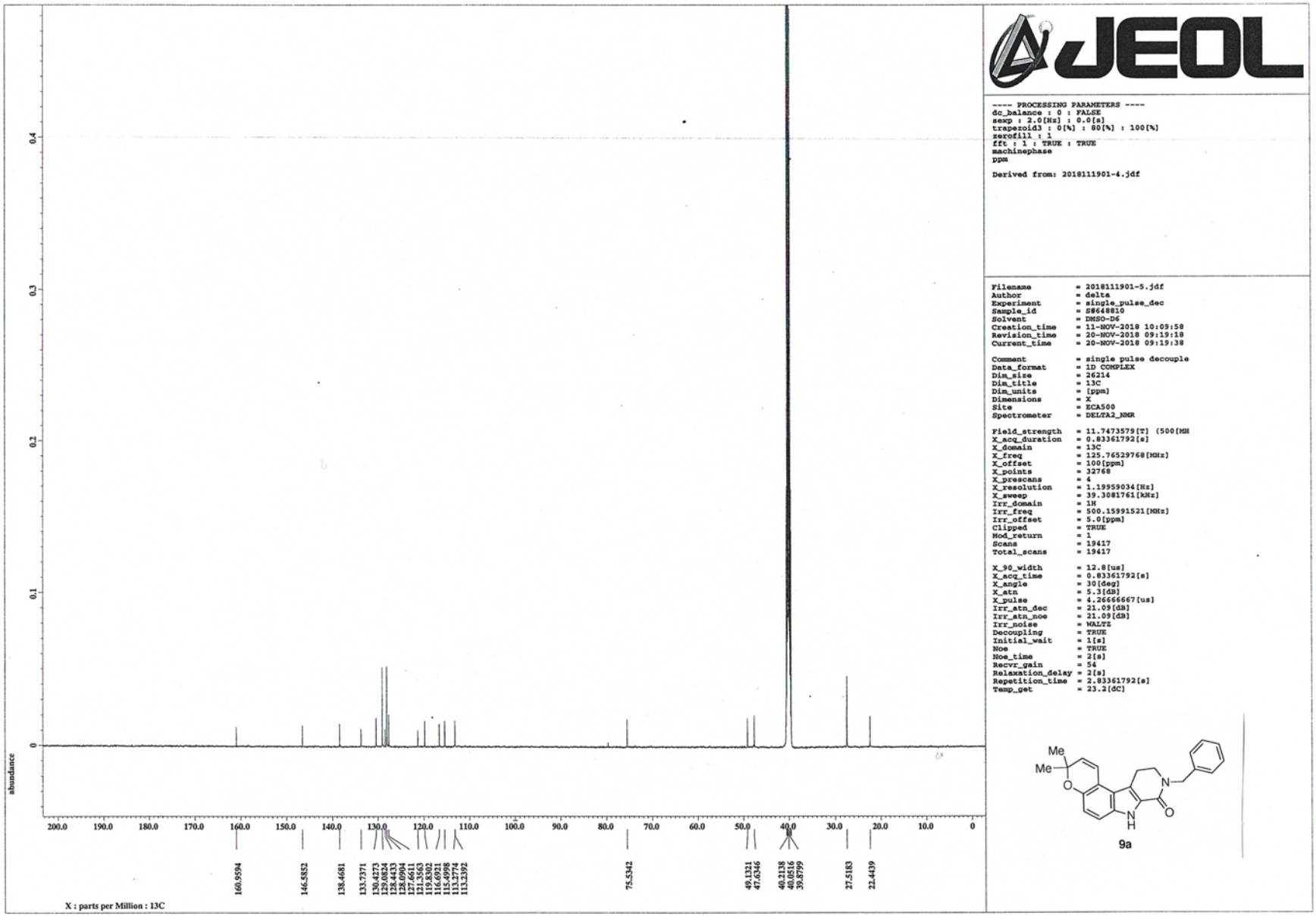
JEOL
Filename = 20181122203-5.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = SH553913
Solvent = CHLOROFORM-D
Creation_time = 22-NOV-2018 14:54:27
Revision_time = 22-NOV-2018 15:32:27
Current_time = 22-NOV-2018 15:32:29
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 495.13191390[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.56682981[Hz]
X_sweep = 9.28677565[kHz]
Irr_domain = 1H
Irr_freq = 495.13191390[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 495.13191390[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Sod_latch = 0
Sweep = 16
Total_scans = 16
X_90_width = 12.7[us]
X_acq_time = 1.76422912[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.35[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_get = 22.4[dC]
OMe
Me
Me
O
N
H
N
H
8b
1.12
0.561
0.39
0.384
0.393
0.203
0.186
0.403
0.188
0.187
0.368
0.178
1.4306
1.5645
2.9508
2.9646
3.0378
3.0516
3.7568
3.7819
5.5907
5.6102
6.7183
6.8225
6.8397
7.0595
7.0778
7.1946
7.2129
7.2529
7.9203
abundance
X : parts per Million : 1H

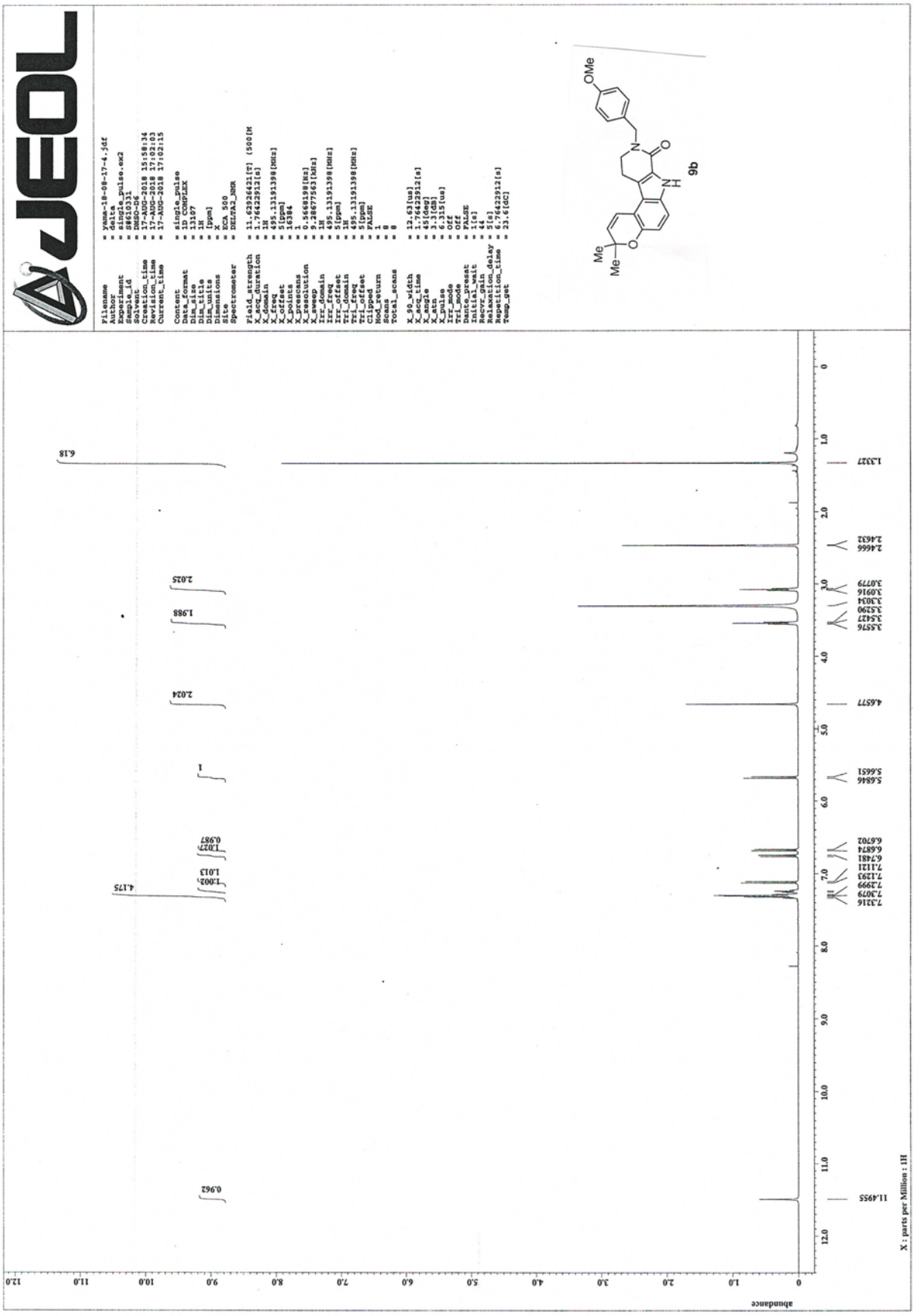
JEOL
Filename = 20181122202-10.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SM701128
Solvent = CHLOROFORM-D
Creation_time = 23-NOV-2018 09:52:58
Revision_time = 23-NOV-2018 10:25:32
Current_time = 23-NOV-2018 10:25:50
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 0.8388608[s]
X_domain = 13C
X_freq = 100[ppm]
X_offset = 32768
X_points = 4
X_prescans = 1.1920929[Hz]
X_resolution = 39.0625[Hz]
X_sweep = 495.11191398[MHz]
Irr_domain = 1H
Irr_freq = 5.0[ppm]
Irr_offset = TRUE
Clipped = 1
Mod_return = 18904
Scans = 18904
Total_scans = 10.6[us]
X_90_width = 0.8388608[s]
X_acq_time = 90.0[deg]
X_angle = 3.5[dB]
X_atn = 3.533333333[us]
X_pulse = 20.548[dB]
Irr_atn_dec = 20.548[dB]
Irr_atn_noe = 32.768[dB]
Irr_mode = noise
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[DEG]
OMe
Me
Me
O
H
N
H
N
8b
abundance
X : parts per Million : 13C
158.6467
146.8024
132.5836
129.6920
129.3664
123.6502
122.8842
120.2798
114.0082
113.1847
113.8454
112.9262
111.2793
77.3744
77.1158
76.8573
74.9232
55.3519
53.4465
49.6548
27.9005
27.2590

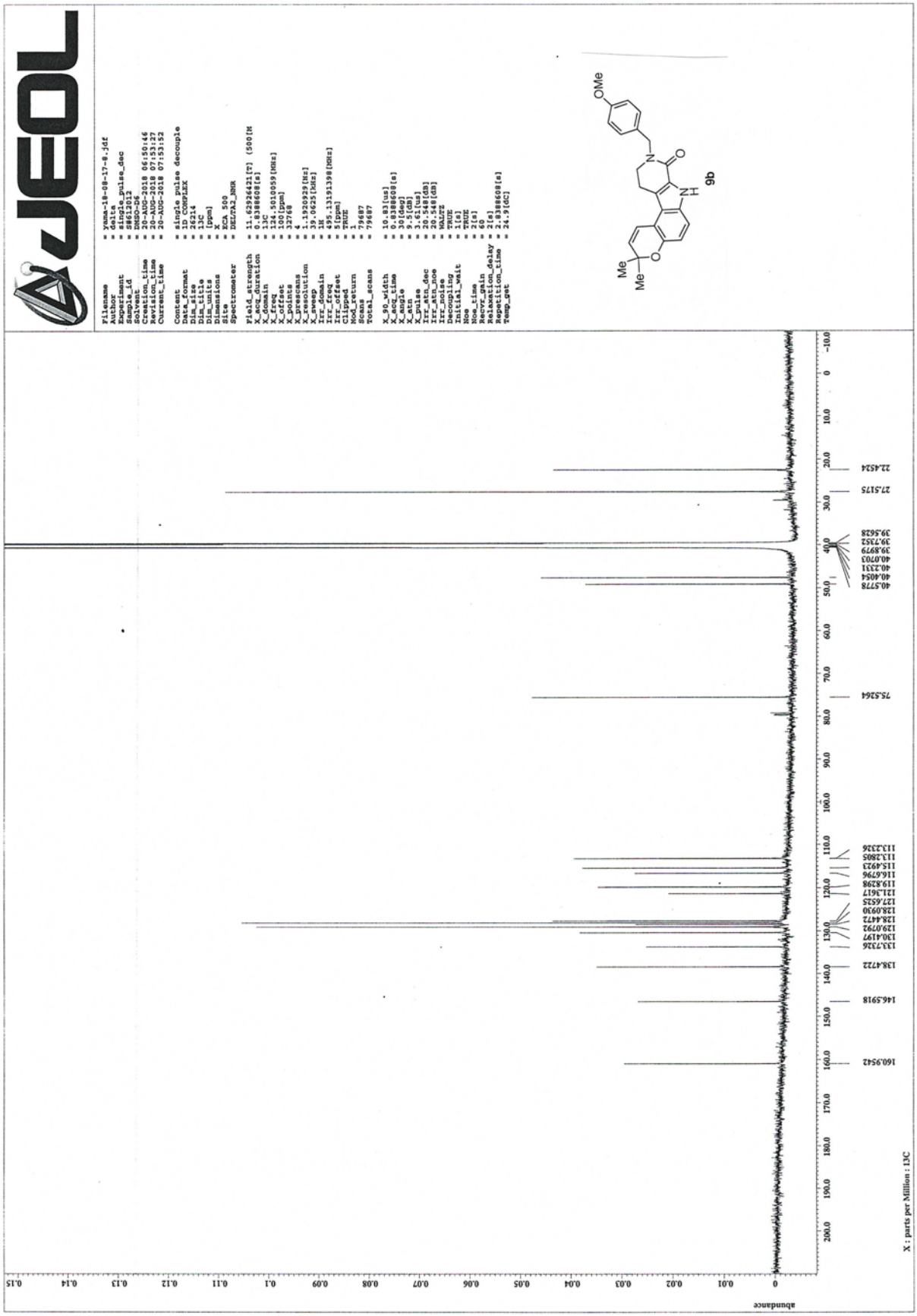

JEOL
Filename = yama-17-06-28-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = #SAMPLE_ID
Solvent = CHLOROFORM-D
Creation_time = 28-JUN-2017 16:17:04
Revision_time = 28-JUN-2017 17:05:37
Current_time = 28-JUN-2017 17:05:59
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = H1 (1H)
Dim_units = [ppm]
Dimensions = X
Site = RCA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.56681918[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]
Comment = PULSE
Mod_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12.7[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 3.5[dB]
X_pulse = 2.35[us]
Irr_mode = Off
Tri_mode = Off
Date_present = PULSE
Irr_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.764229121[s]
Temp_get = 21.4[dC]
MeO
OMe
Me
Me
8c
5.922
5.24
3.123
2.092
2.064
1.00
1.021
2.116
0.999
2.101
1
0.868
1.4225
2.9182
2.9314
3.0133
3.0476
3.6497
3.7625
3.7694
5.5644
5.5838
6.3749
6.3852
6.3886
6.4863
6.7034
6.8752
6.8958
7.0354
7.0537
7.2541
8.2392
abundance
X : parts per Million : 1H

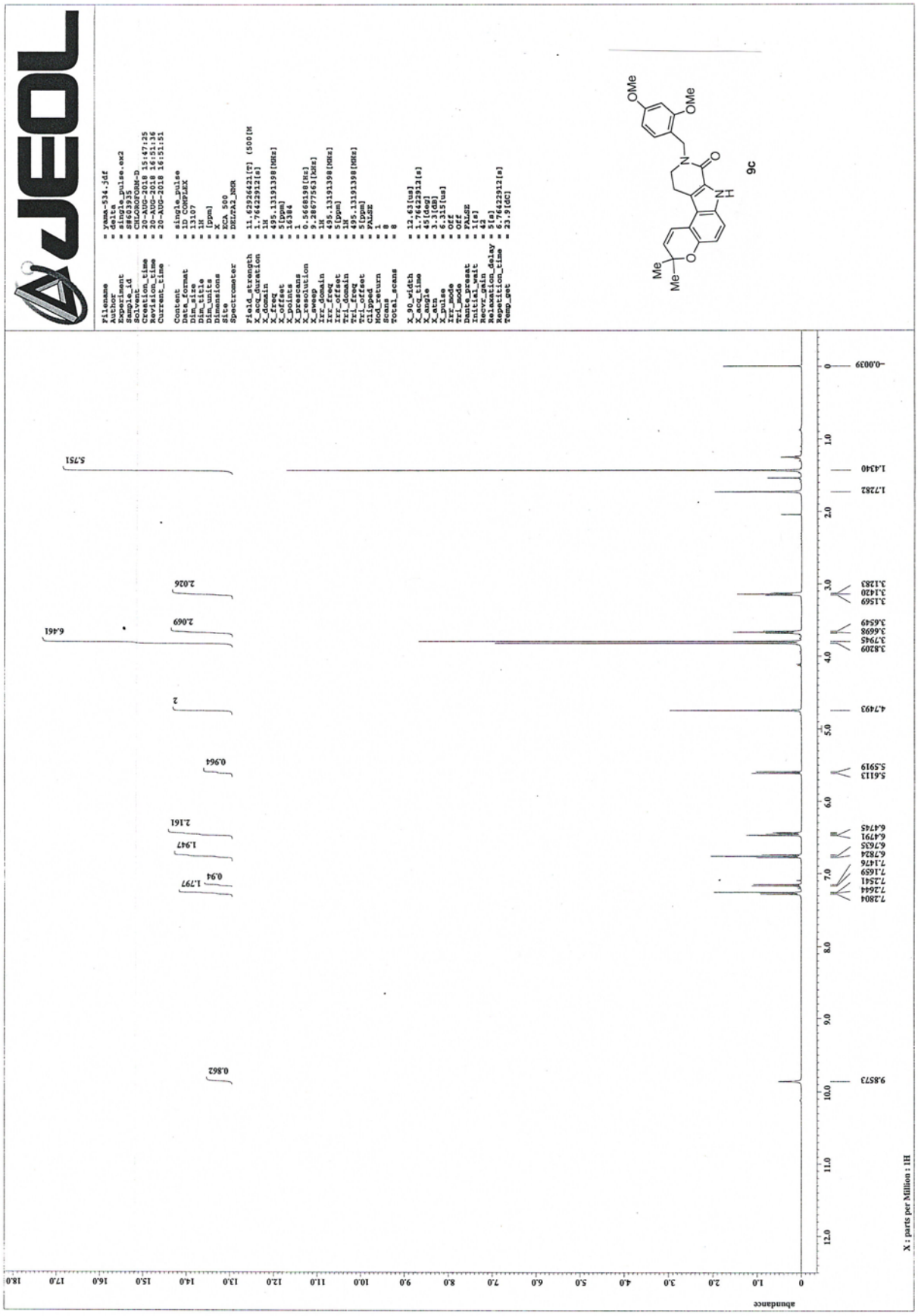
JEOL
Filename = yasa-17-06-28-8.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SM613160
Solvent = CHLOROFORM-D
Creation_time = 29-JUN-2017 07:00:31
Revision_time = 29-JUN-2017 07:46:41
Current_time = 29-JUN-2017 07:46:50
Context = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 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 = 124.5010959[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 = 495.1319398[MHz]
Irr_offset = 5[ppm]
Irr_mode = PULSE
Mod_return = 1
Scans = 18632
Total_scans = 18632
X_90_width = 10.6[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 3.5[dB]
X_pulse = 3.533333333[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]
Relaxation = T1
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 22.6[dC]
OMe
MeO
H
N
Me
Me
O
H
8c
abundance
X : parts per Million : 13C
27.2494
27.8143
49.0037
49.2814
55.1796
55.4285
74.9040
76.8956
77.1541
77.4031
98.5733
103.7342
111.3080
111.7539
111.7688
113.1560
120.3372
120.5479
122.8937
123.8608
129.5866
130.2346
132.6506
146.6971
158.6467
160.1595


JEOL
9a
Me
Me
abundance
X : parts per Million : 1H

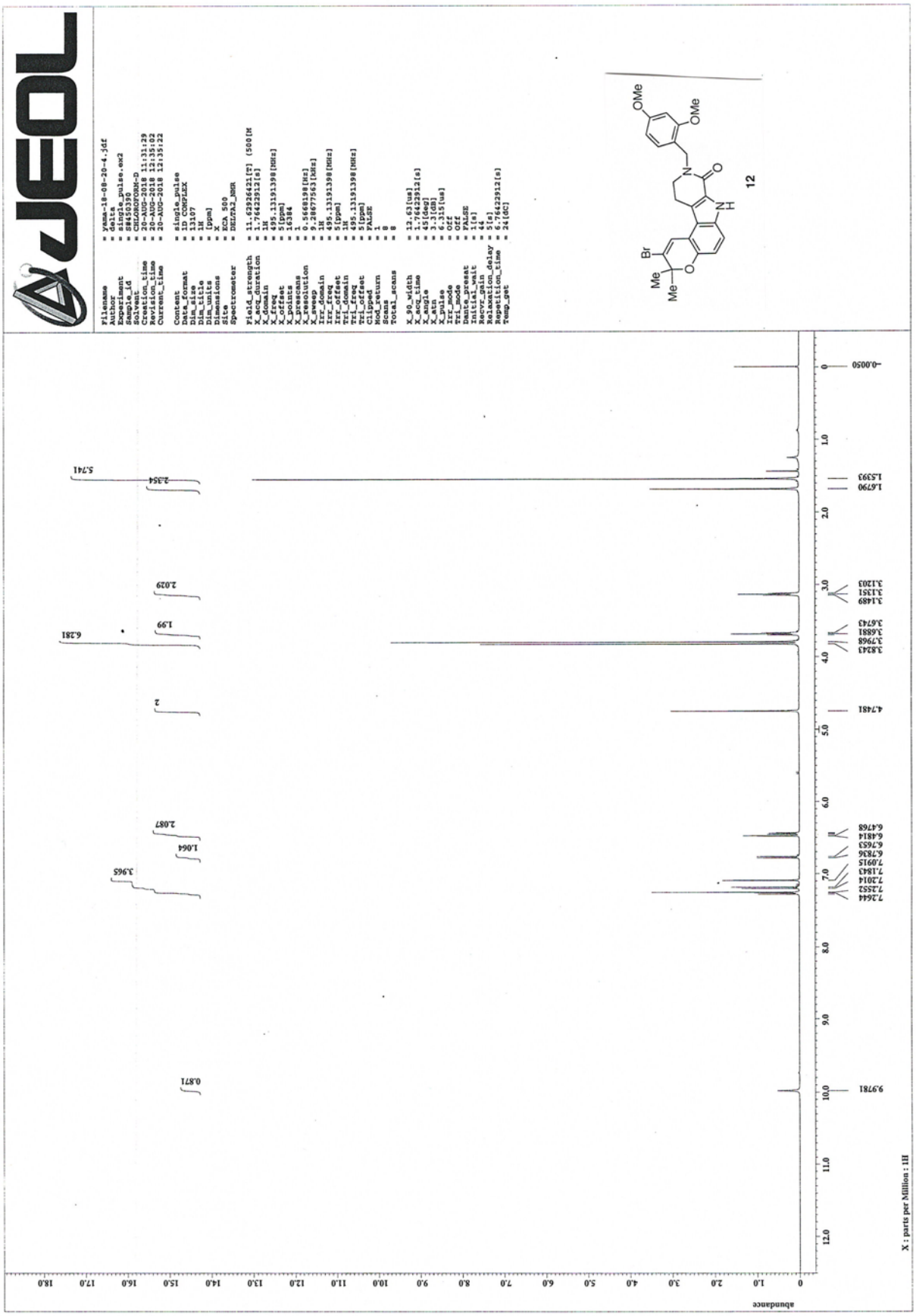

JEOL
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp : 2.0[Hz] : 0.0[s]
trapezoid3 : 0[%] : 80[%] : 100[%]
zerofill : 1
fft : 1 : TRUE : TRUE
machinephase
ppm
Derived from: 2018111901-4.jdf
Filename = 2018111901-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SH648810
Solvent = DMSO-D6
Creation_time = 11-NOV-2018 10:09:58
Revision_time = 20-NOV-2018 09:19:18
Current_time = 20-NOV-2018 09:19:38
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 19417
Total_scans = 19417
X_90_width = 12.8[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.26666667[us]
Irr_atn_dec = 21.09[dB]
Irr_atn_noe = 21.09[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Repetition_time = 2.83361792[s]
Temp_get = 23.2[dC]
Me
Me
O
N
H
O
9a
abundance
0.4
0.3
0.2
0.1
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
160.9594
146.5852
138.4681
133.7371
130.4273
129.0824
128.4433
128.0904
127.6611
121.1455
119.8302
116.6921
115.4998
113.2774
113.2392
75.5342
49.1331
47.6346
40.3138
40.0516
39.8799
27.5183
22.4439
X : parts per Million : 13C


JEOL
Filename = yuan-18-08-17-4.jdf
Author = default
Experiment = single_pulse.ex2
Sample_id = SH6210331
Solvent = DMSO-D6
Creation_time = 17-AUG-2018 15:58:34
Revision_time = 17-AUG-2018 17:02:03
Current_time = 17-AUG-2018 17:02:15
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 = 11.62326421[T] (500[M
X_acq_duration = 1.76422912[s]
X_domain = 1H
X_freq = 499.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.56692198[Hz]
X_sweep = 9.28077961[kHz]
Irr_domain = 1H
Irr_freq = 499.13191398[MHz]
Irr_offset = 5[ppm]
Irr_points = 16
Tri_freq = 499.13191398[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Co_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_preset = FALSE
Initial_wait = 1[s]
Repetition = 1[s]
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_set = 23.6[DC]
OMe
Me
Me
9b
6.18
2.025
1.988
2.024
1
0.987
1.027
1.002
1.013
4.175
0.962
11.4955
1.3327
2.4666
2.4632
3.0779
3.0916
3.3034
3.5290
3.5427
3.5576
4.6577
5.6846
5.6651
6.6702
6.6874
6.7481
7.1121
7.1293
7.2999
7.3079
7.3216
abundance
X : parts per Million : 1H

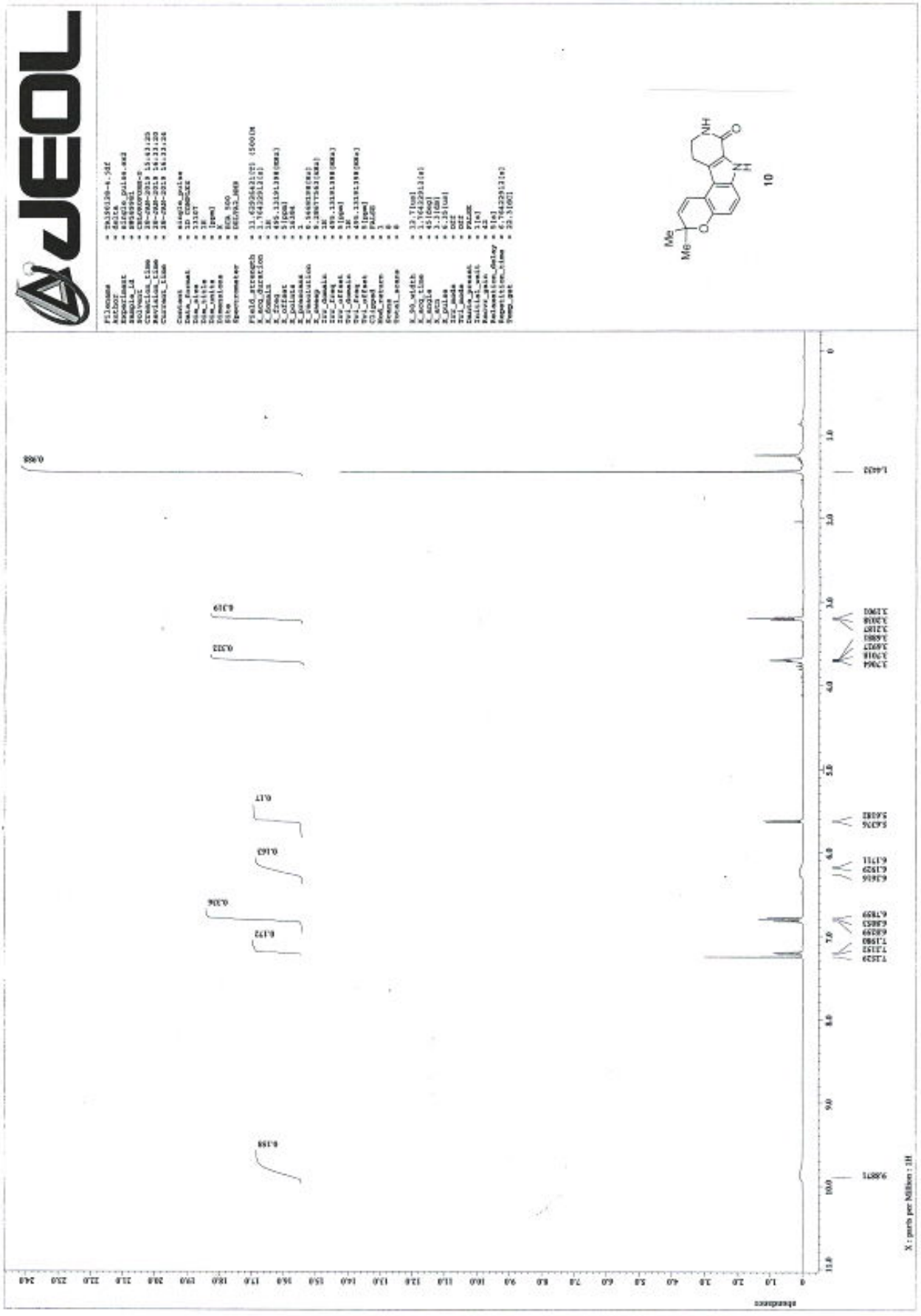

JEOL
Filename = yana-18-08-17-8.jdf
Author = default
Experiment = single_pulse_dec
Sample_id = SH6132012
Solvent = DMSO-D6
Creation_time = 20-AUG-2018 06:50:46
Revision_time = 20-AUG-2018 07:53:27
Current_time = 20-AUG-2018 07:53:52
Context = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 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 = 125.5010959[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 = 495.1319398[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 79687
Total_scans = 79687
X_90_width = 10.83[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 3.5[dB]
X_pulse = 3.61[us]
Irr_atn_dec = 20.548[dB]
Irr_atn_noe = 20.548[dB]
Irr_noise = NONE
Decoupling = TRUE
Initial_wait = 1[s]
NOe = TRUE
Relax_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.9[dC]
OMe
Me
Me
O
N
O
N
H
9b
22.4524
27.5175
39.5628
39.7352
39.8979
40.0703
40.2331
40.4054
40.5778
75.5264
113.3236
113.2805
115.4923
116.6796
119.8298
121.3617
127.6535
128.0930
128.4472
129.0792
130.4197
133.7236
138.4722
146.5918
160.9542
abundance
X : parts per Million : 13C


JEOL
Filename = yasu-534.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SH603935
Solvent = CHLOROFORM-D
Creation_time = 20-AUG-2018 15:47:25
Revision_time = 20-AUG-2018 16:51:36
Current_time = 20-AUG-2018 16:51:51
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 = 0.56869[Hz]
X_sweep = 9.28877563[kHz]
Irr_domain = 1H
Irr_freq = 495.13191398[MHz]
Irr_offset = 5[ppm]
Irr_points = 16
Tri_freq = 495.13191398[MHz]
Tri_offset = 5[ppm]
Tri_pulsed = FALSE
Wait_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 = 6.32[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Recvr_delay = 16[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.76422912[s]
Temp_set = 23.9[dC]
OMe
OMe
N
O
NH
O
Me
Me
9c
5.751
2.026
2.069
6.461
2
0.964
2.161
1.947
0.94
1.797
0.862
-0.0039
1.4340
1.7282
3.1283
3.1420
3.1569
3.6549
3.6698
3.7945
3.8209
4.7493
5.5919
5.6113
6.4745
6.4791
6.7624
6.7635
7.1476
7.1659
7.2541
7.2644
7.2804
9.8573
abundance
X : parts per Million : 1H

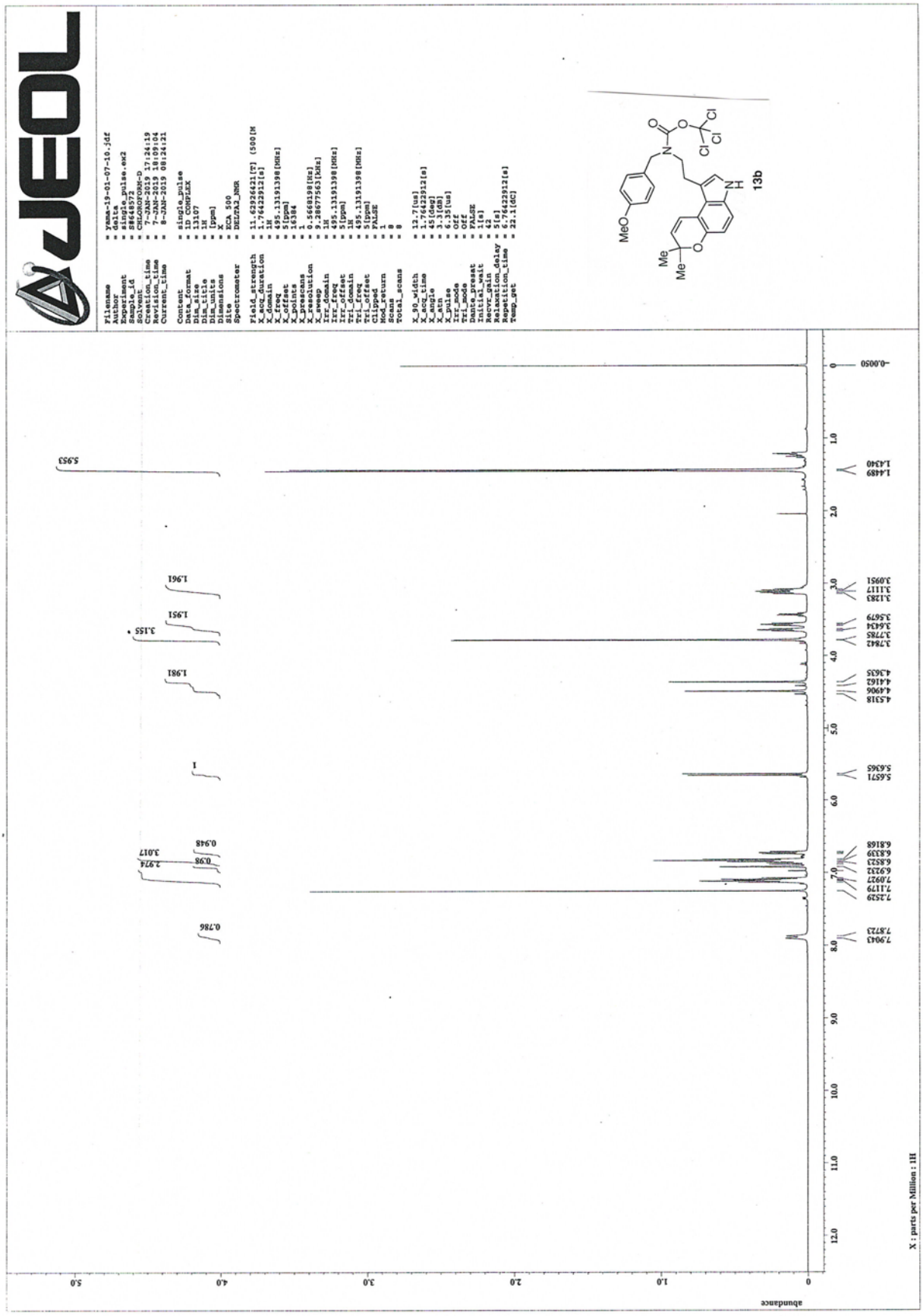
JEOL
Filename = yuan-18-08-21-11.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 88656044
Solvent = CHLOROFORM-D
Creation_time = 21-AUG-2018 06:51:35
Revision_time = 21-AUG-2018 07:53:17
Current_time = 21-AUG-2018 07:55:02
Content = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 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 = 125.5010059[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 = 495.1319139[MHz]
Irr_offset = 5[ppm]
Lock = TRUE
Mod_return = 1
Scans = 17290
Total_scans = 17290
X_90_width = 10.83[us]
X_acq_time = 0.8388608[s]
X_angle = 30[deg]
X_atn = 3.51[dB]
X_pulse = 3.61[us]
Irr_atn_dec = 20.54[dB]
Irr_atn_noe = 20.54[dB]
Irr_coupling = TRUE
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_get = 24.4[dC]
OMe
OMe
Me
Me
O
N
H
O
9c
x2
x2
22.8736
27.3452
43.9769
47.5196
55.4956
76.8477
77.1063
77.3648
98.5829
104.3087
112.7156
113.4337
115.8370
116.7753
118.1158
119.4861
121.5522
128.2940
129.9026
130.4101
133.3592
146.9748
158.7233
160.3893
161.5479
abundance
X : parts per Million : 13C


JEOL
Filename = yana-18-08-20-4.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SH450390
Solvent = CHLOROFORM-D
Creation_time = 20-AUG-2018 11:31:29
Revision_time = 20-AUG-2018 12:35:02
Current_time = 20-AUG-2018 12:35:22
Context = 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 = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.566918[Hz]
X_sweep = 9.26775913[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
Wood_return = 1
Scans = 8
Total_scans = 8
X_90_width = 12.63[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 3.31[dB]
X_pulse = 6.31[us]
Irr_mode = Off
Tri_mode = Off
Data_preset = FALSE
Fill_factor = 44
Recv_gain = 5[s]
Relaxation_delay = 6.764229121[s]
Repetition_time = 24[dC]
Temp_get
OMe
OMe
N
O
H
N
O
Br
Me
Me
12
5.741
2.354
2.029
1.99
6.281
2
2.087
1.064
3.965
0.871
-0.0050
1.5790
1.5393
3.1489
3.1351
3.1203
3.8243
3.7968
3.6881
3.6743
4.7481
6.4768
6.4814
6.7653
6.7836
7.0915
7.1843
7.2014
7.2352
7.2644
9.9781
abundance
X : parts per Million : 1H

JEOL
Filename = yuma-18-08-20-8.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = 8M452106
Solvent = CDCl3-TMS-D
Creation_time = 20-AUG-2018 12:58:45
Revision_time = 20-AUG-2018 14:02:15
Current_time = 20-AUG-2018 14:02:19
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 = 0.9388608[s]
X_domain = 13C
X_freq = 100[ppm]
X_offset = 32768
X_points = 4
X_prescans = 1.19209929[Hz]
X_resolution = 39.0625[kHz]
X_sweep = 1H
Irr_domain = 495.1319398[MHz]
Irr_freq = 4.5[ppm]
Irr_offset = TRUE
Clipped = 1
Mod_return = 1800
Scans = 1800
Total_scans = 10.83[us]
X_90_width = 0.8388608[s]
X_acq_time = 3.5[dB]
X_angle = 9.5[dB]
X_pulse = 3.6[us]
Irr_atn_dec = 20.548[dB]
Irr_atn_one = 20.548[dB]
Irr_mode = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
NOE = TRUE
Num_time = 256
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 2.8388608[s]
Temp_set = 24.3[dC]
OMe
OMe
Br
Me
Me
O
N
H
O
12
22.7204
26.0142
44.0726
47.4909
55.4956
76.8477
77.1063
77.3648
79.4330
98.5925
104.3183
113.2039
114.1231
115.5593
116.4594
118.0105
120.4572
123.1523
124.1672
128.6100
130.4867
133.5986
145.7109
158.7233
160.4372
161.3947
abundance
X : parts per Million : 13C


JEOL
NH
O
N
H
Me
Me
O
1D
X : parts per Million : 1H

JEOL
Me
Me
NH
O
N
H
10


JEOL
Filename = yama-19-01-07-10.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = SM648572
Solvent = CDCl3+TMS+BHT-D
Creation_time = 7-JAN-2019 17:24:19
Revision_time = 7-JAN-2019 18:09:04
Current_time = 8-JAN-2019 08:24:21
Content = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR
Field_strength = 11.62926421[T] (500[M
X_acq_duration = 1.764229121[s]
X_domain = 1H
X_freq = 495.13191398[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.56691981[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]
dfrapped = FALSE
MCAL_return = 1
Scans = 8
Total_scans = 8
X_90_width = 13.7[us]
X_acq_time = 1.764229121[s]
X_angle = 45[deg]
X_atn = 3.3[dB]
X_pulse = 6.85[us]
Irr_mode = Off
Tri_mode = Off
Dante_preset = FALSE
Dummy_wait = 1[s]
Recvr_Gain = 41
Relaxation_delay = 5[s]
Repetition_time = 6.764229121[s]
Temp_get = 22.1[dC]
MeO
Me
Me
13b
abundance
X : parts per Million : 1H
5.953
1.961
1.951
3.155
1.981
1
0.948
0.98
3.017
7.974
0.786
-0.0050
1.4489
1.4340
3.1283
3.1117
3.0951
3.7842
3.7785
3.6434
3.5679
4.5318
4.4906
4.4162
4.3635
5.6571
5.6365
7.9043
7.8723
7.3529
7.1179
7.0927
6.9232
6.8523
6.8339
6.8168

JEOL
Filename = yman-19-01-07-15.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = SH651245
Solvent = CHLOROFORM-D
Creation_time = 8-JAN-2019 07:41:01
Revision_time = 8-JAN-2019 08:22:59
Current_time = 8-JAN-2019 08:23:25
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 = 11.629264217[T] (500[M
X_acq_duration = 0.83886608[s]
X_domain = 13C
X_freq = 100[ppm]
X_offset = 32768
X_points = 4
X_prescans = 4.1920929[Hz]
X_resolution = 19.0625[kHz]
X_sweep = 1H
Irr_domain = 495.13191398[MHz]
Irr_freq = 5[ppm]
Irr_offset = TRUE
Clipped = 1
Mod_return = 10028
Scans = 10028
Total_scans = 10.6[us]
X_90_width = 0.83886608[s]
X_acq_time = 30[deg]
X_angle = 3.1533333333[us]
X_delay = 20.540[dB]
X_pulse = 20.540[dB]
Irr_atn_dec = 20.540[dB]
Irr_atn_noe = WALTZ
Irr_noise = TRUE
Irr_spoffset = 1[s]
Decoupling = TRUE
Initial_wait = 2[s]
Nos = 1
Noe_time = 2[s]
Relaxation_delay = 2.83886608[s]
Repetition_time = 23.6[dC]
Temp_get
MeO
Me
Me
O
N
O
O
Cl
Cl
Cl
H
13b
abundance
X : parts per Million : 13C
0.0852
13.5764
25.0089
26.4164
27.3686
50.4496
50.8230
54.7774
55.3807
74.9806
76.8477
77.1063
77.3618
113.2135
113.3996
114.2188
114.2763
119.8106
119.8585
124.0236
124.1098
128.8398
129.8547
130.1516
132.4304
146.9556
147.0227
149.3398
150.2399
159.5084
159.5371

JEOL
fontanesine B (2)
Me
Me
O
N
N
H
O
abundance
X : parts per Million : 1H
11.7108
8.1321
8.1161
7.7736
7.7576
7.6594
7.6454
7.4518
7.4381
7.4231
7.3182
7.3011
6.8564
6.8369
6.7441
6.7258
5.7421
5.7236
4.4091
4.3953
4.3816
3.3028
3.2948
3.2753
2.4657
1.3915
1.3594
1.3377
54.87
50.99
18.91
8.91
8.66
8.48
8.91
9.72
8.99
9.54
9.5
7.83

JEOL
---- PROCESSING PARAMETERS ----
dc_bias : FALSE
exp : 2.0[Hz] ; 0.0[s]
trapezoid3 : 0[%] ; 80[%] ; 100[%]
zerofill : 3
ffts : 1 ; TRUE ; TRUE
machinephase
ppm
Derived from: 20181016O2-7.jdf
Filename = 20181016O2-8.jdf
Author = default
Experiment = single_pulse_dec
Sample_id = ##$SOG49
Solvent = DMSO-D6
Creation_time = 9-OCT-2018 10:10:18
Revision_time = 17-OCT-2018 09:18:44
Current_time = 17-OCT-2018 09:19:10
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
X_domain = 13C
Site = ECA500
Spectrometer = DELTA2_NMR
Field_strength = 11.7473579[T] (500[MH
X_acquisition = 0.83363792[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 = 39.30817614[kHz]
X_window = EM
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 22884
Total_scans = 22884
X_90_width = 12.2[us]
X_acq_time = 0.83363792[s]
X_angle = 30[deg]
X_atn = 5.3[dB]
X_pulse = 4.06666667[us]
Irr_atn_dec = 30.07[dB]
Irr_atn_noe = 20.07[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 54
Relaxation_delay = 2[s]
Revision_time = 23.5[dc]
Temp_get
Me
Me
O
N
N
H
O
fontanesine B (2)
21.2993
27.5183
39.8895
40.0612
40.2233
75.6200
113.1915
113.3964
116.1838
117.1022
119.7654
121.3037
121.4803
126.4689
126.9819
128.1175
128.4910
130.6752
134.9770
145.7458
146.7664
147.9969
161.0644
abundance
X : parts per Million : 13C