Supplementary Material (ESI) for Chemical Communications

Supplementary Information for

Concise Total Synthesis of Naamine G and Naamidine H

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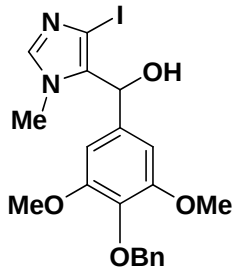
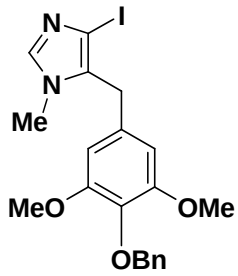
(4-Benzyloxy-3,5-dimethoxyphenyl)-(5-iodo-2-methyl-2H-imidazol-1-yl)-methanol (9):



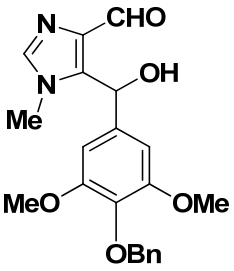
EtMgBr (3.0 M solution in ether, 7.54 ml, 22.6 mmol) was added to a solution of 4,5-diiodo-1-methyl-1H-imidazole (7.19 g, 21.5 mmol) in dry CH_2Cl_2 (100 ml) at rt over ~10 min. The resulting mixture was stirred at rt for 20 min and 4-benzoyloxy-4,5-dimethylbenzaldehyde[1] (6.46 g, 23.7 mmol) was added and stirred at rt for 48 h. Sat. NH_4Cl (10 ml) was added to the reaction and the resulting pale yellow solid was filtered and the filtrate was partitioned with CH_2Cl_2. The organic layer was dried (Na_2SO_4) and concentrated to give a pale yellow solid. The resulting solid was triturated with hexanes, recrystallized with CH_2Cl_2 to give **9** (9.68 g, 95%) as a white solid; m.p = 173-175 ºC; ^{1}H NMR ($CDCl_3$): δ = 7.44 (d, *J* = 6.9 Hz, 2H), 7.31-7.24 (m, 4H), 6.57 (s, 2H), 5.98 (s, 1H), 5.18 (s, 1H), 4.98 (s, 2H), 3.75 (s, 6H), 3.43 (s, 3H); ^{13}C NMR: δ = 153.6, 141.1, 137.8, 136.6, 135.9, 135.2, 128.6, 128.2, 127.9, 102.7, 84.6, 75.0, 67.1,56.3, 33.5; IR (cm^{-1}): 3253 (br), 3104, 1585, 1501, 1411, 1338, 1226, 1140, 1033, 908 ; HR-DART-MS (*m/z*): Calc. for $C_{20}H_{21}IN_2O_4$ $[M]^+$: 480.0546; found: 480.0546; Calc. for $C_{20}H_{22}IN_2O_4$ $[M+H]^+$ 481.0624; found: 481.0624

4-(4-Benzyloxy-3,5-dimethoxy)benzyl-5-iodo-1-methyl-1H-imidazol (6): Et_3SiH (1.00 ml, 6.25 mmol) and TFA (0.40 ml, 5.20 mmol) were added to a solution of **9** (0.50 g, 1.04 mmol) in anhydrous $CHCl_3$ (20 ml) at rt and the resulting mixture was heated at reflux temperature for 24 h under nitrogen atmosphere. Then, the reaction was quenched by the addition of saturated aqueous solution of $NaHCO_3$. The aqueous layer was extracted with $CHCl_3$ several times and the combined extracts were dried (Na_2SO_4) and

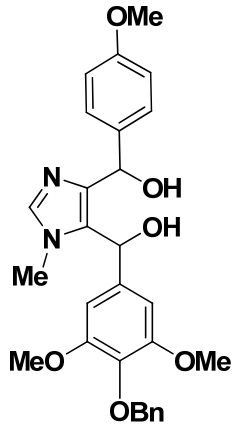


concentrated. The residue was purified by chromatography (EtOAc) to isolate **6** (0.14 g, 28%) as a pale brown semi solid; ^{1}H NMR ($CDCl_3$): δ = 7.44 (d, *J* = 6.9 Hz, 2H), 7.36 (s, 1H), 7.30 (t, *J* = 6.9 Hz, 2H), 7.25 (d, *J* = 6.9 Hz, 1H), 6.31 (s, 2H), 4.95 (s, 2H), 3.87 (s, 2H), 3.73 (s, 6H), 3.40 (s, 3H); ^{13}C NMR: δ = 153.8, 139.6, 137.9, 135.9, 133.1, 128.5, 128.2, 127.9, 105.2, 85.0, 75.1, 56.3, 32.7, 31.0; IR (cm^{-1}): 3107, 2939, 1588, 1494, 1460, 1420, 1237, 1215, 1187, 1100, 978, 758; HR-ESIMS (*m/z*): Calc. for $C_{20}H_{22}IN_2O_3$ $[M+H]^+$ 465.0670; found: 465.0669.

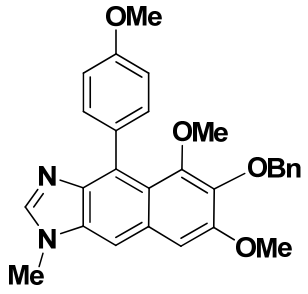
5-[(4-Benzyloxy-3,5-dimethoxyphenyl)-hydroxy-methyl]-1-methyl-1H-imidazole-4-carbaldehyde (12): EtMgBr (3.0 M in ether, 13.8 ml, 41.4 mmol) was added into a solution of **9** (9.03 g, 18.8 mmol) in dry THF (200 ml) at rt, and the resulting mixture was stirred at rt for 20 min. *N*-Methylformanilide (2.78 ml, 22.6 mmol) was added and the resulting mixture was stirred at rt for 33 h. Half saturated NH_4Cl (30 ml) was added to quench the reaction and the organic layer was extracted with EtOAc, dried (Na_2SO_4) and concentrated to give the crude product, which was purified through a short plug of silica gel (EtOAC→Acetone) to give **12** as a pale yellow solid (5.52 g, 77%); m.p = 132-134 ºC; ^{1}H NMR ($CDCl_3$): δ = 9.82 (s, 1H), 7.42 (d, *J* = 7.3 Hz, 2H), 7.38 (s, 1H), 7.29 (t, *J* = 7.3 Hz, 2H), 7.25 (m, 1H), 6.50 (s, 2H), 6.23 (s, 1H), 4.94 (s, 2H), 3.71 (s, 6H), 3.47 (s, 3H); ^{13}C NMR: δ = 188.4, 153.8, 141.7, 139.9, 138.0, 137.7, 136.6, 136.2, 128.5, 128.2, 127.9, 103.4, 75.1, 66.7, 56.3, 33.1; IR (cm^{-1}): 3253 (br), 3106, 2938, 1680, 1587, 1502, 1450, 1415, 1230, 1100, 1056, 824; HR-DART-MS (*m/z*): Calc. for $C_{21}H_{23}N_2O_5$ $[M+H]^+$ 383.1601; found: 383.1597



{5-[(4-Benzyloxy-3,5-dimethoxyphenyl)-hydroxy-methyl]-1-methyl-1H-imidazol-4-yl}-(4-methoxyphenyl)methanol (13): A few drops of *p*-bromoanisole (from 7.23 ml, 56.5 mmol) were added dropwise to a two necked round-bottom flask containing freshly-crushed, oven-dried, magnesium turnings (1.35 g, 56.5 mmol) and a small crystal of iodine in THF (100 ml). This mixture was then heated at 45 °C under nitrogen until the iodine color faded. The remainder of the *p*-bromoanisole was added dropwise while maintaining gentle reflux. After the addition was completed, the mixture was heated to reflux for 1 h, cooled to rt, and then a solution of **12** (5.40 g, 14.1 mmol) in THF (100 ml) was added. The resulting mixture was stirred at reflux overnight. After cooling to 0 ° C, sat. NH_4Cl (50 ml) was added and the organic layer was extracted with EtOAc (3 x 100 ml), washed once with brine, dried (Na_2SO_4), and concentrated to give a thick, brown oil. The crude product was purified through a short plug of silica gel (EtOAc) to give **13** (4.38 g, 63%) as a pale yellow oil, which was used in the next step directly.



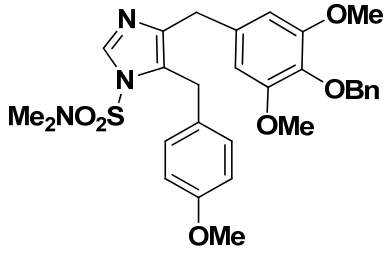
6-Benzyloxy-5,7-dimethoxy-4-(4-methoxyphenyl)-1-methyl-1H-naphtho[2,3-*d*]imidazole (14): Et_3SiH (11.41 ml, 71.44 mmol) and TFA (4.81 ml, 62.5 mmol) were added to a solution of **13** (4.38 g, 8.93 mmol) in anhydrous CH_2Cl_2 (100 ml) at rt and the resulting mixture was stirred for 24 h under nitrogen atmosphere. Then, the reaction was quenched by the addition of saturated aqueous solution of $NaHCO_3$. The aqueous layer was extracted with CH_2Cl_2 several times and the combined extracts were dried (Na_2SO_4) and concentrated. The residue was purified by chromatography (EtOAc→acetone) to provide **14**



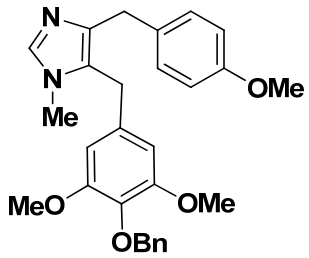
(3.30 g, 81%) as a pale brown solid; m.p = 194- 197 ºC; ^{1}H NMR ($CDCl_3$): δ = 7.87 (s, 1H), 7.61 (s, 1H), 7.53 (d, *J* = 6.9 Hz, 2H), 7.45 (d, *J* = 8,7 Hz, 2H), 7.36 (t, *J* = 6.9 Hz, 2H), 7.30 (m, 1H), 7.08 (s, 1H), 7.05 (d, *J* = 8.7 Hz, 2H), 5.10 (s, 2H), 3.96 (s, 3H), 3.89 (s, 3H), 3.74 (s, 3H), 3.37 (s, 3H); ^{13}C NMR: δ = 158.2, 152.1, 150.6, 146.6, 142.9, 139.8, 138.0, 134.4, 132.4, 131.6, 130.9, 129.7, 128.4, 128.3, 127.9, 119.6, 112.7, 104.0, 102.3, 75.2, 60.9, 55.8, 55.4, 31.0; IR (cm^{-1}): 2929, 2831, 1607, 1514, 1451, 1330, 1274, 1236, 1145, 1075, 1027, 826, 740; HR-DART-MS (*m/z*): Calc. for $C_{28}H_{27}N_2O_4$ $[M+H]^+$ 455.1971; found: 455.1983.

4-Iodo-5-(4-methoxy-benzyl)-1-(*N*,*N*-dimethylsulfonyl)-1H-imidazole (16): EtMgBr (3.0 M solution in ether, 8.60 ml, 25.8 mmol) was added to a solution of 4,5-diiodo-1-(*N*,*N*-dimethylsulfonyl)-1H-imidazole **15** (7.19 g, 21.5 mmol) in dry CH_2Cl_2 (150 ml) at rt. The resulting mixture was stirred at rt for 20 min and 1.0 M solution of CuCN.2LiCl in dry THF (26 ml, 26 mmol) was added followed by 4-methoxybenzyl bromide (3.80 ml, 25.8 mmol) was added. The orange reaction solution was stirred at rt for 48 h and poured into half Sat. NH_4Cl containing 2% concentrated NH_3 (50 ml). After stirring for 20 min the resulting solid was filtered off and the filtrate was partitioned with CH_2Cl_2 (3x50 ml). The organic layer was dried (Na_2SO_4) and concentrated and purified by chromatography (EtOAc/hexane, 3:7) to afford **16** (6.41 g, 65%) as a pale yellow solid; m.p. = 76-78 ºC; ^{1}H NMR ($CDCl_3$): δ = 7.87 (s. 1H), 6.99 (d, *J* = 8.7 Hz, 2H), 6.77 (d, *J* = 8.7 Hz, 2H), 6.37 (s, 2H), 4.09 (s, 2H), 3.71 (s, 3H), 2.49 (s, 6H); ^{13}C NMR: δ = 158.5, 139.7, 137.9, 132.5, 129.1, 114.0, 90.6, 55.4, 37.6, 29.9; IR (cm^{-1}): 3111, 2919, 1514, 1459, 1415, 1240, 1173, 1174, 1095, 960, 802; HR-DART-MS (*m/z*): Calc. for $C_{13}H_{17}IN_3O_3S$ $[M+H]^+$ 422.0030; found: 422.0047.

4-(4-Benzyloxy-3,5-dimethoxybenzyl)-5-(4-methoxybenzyl)-1-(*N*,*N*-dimethylsulfonyl)-1H-imidazole (17): Following the above procedure, EtMgBr (3.0 M solution in ether, 4.98 ml, 14.95 mmol), **16** (5.72 g, 13.6 mmol) in dry CH_2Cl_2 (150 ml), 1.0 M solution of CuCN.2LiCl in dry THF (16.3 ml, 16.3 mmol) and 4-benzyloxy-3,5-dimethoxybenzyl bromide (**10**)[2] (6.87 g, 20.4 mmol) were used to produce **17** (5.18 g, 70%) as a pale yellow oil after the purification by chromatography (EtOAc:hexane, 1:1); ^{1}H NMR ($CDCl_3$): δ = 7.94 (s. 1H), 7.48 (d, *J* = 7.4 Hz, 2H), 7.35-7.26 (m, 3H), 6.94 (d, *J* = 8.7 Hz, 2H), 6.77 (d, *J* = 8.7 Hz, 2H), 6.37 (s, 2H), 4.94 (s, 2H), 4.14 (s, 2H), 3.78 (s, 2H), 3.75 (s, 3H), 3.71 (s, 6H), 2.57 (s, 6H); ^{13}C NMR: δ = 158.4, 153.4, 141.3, 138.1, 138.0, 135.6, 134.8, 130.3, 129.9, 128.9, 128.5, 128.2, 127.8, 114.0, 106.0, 75.1, 56.1, 55.4, 37.5, 34.0, 28.1; IR (cm^{-1}): 2929, 2857, 1691, 1507, 1393, 1252, 1124, 909, 836, 779; HR-DART-MS (*m/z*): Calc. for $C_{29}H_{34}N_3O_6S$ $[M+H]^+$ 552.2163; found: 552.2180.

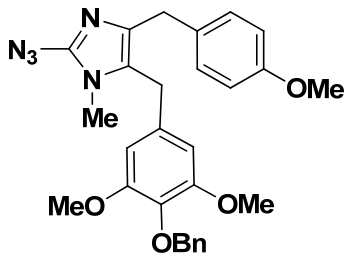


5-(4-Benzyloxy-3,5-dimethoxybenzyl)-4-(4-methoxybenzyl)-1-methyl-1H-imidazole (5): Methyl trifluoromethanesulfonate (0.95 ml, 8.66 mmol) was added dropwise to a solution of **17** (3.96 g, 7.18 mmol) in CH_2Cl_2 (50 ml), at 0 ºC under N_2 and stirred for 4 h at the same temperature. The solvent was evaporated under reduced pressure and the crude pale yellow oil was dissolved in dry acetonitrile (30 ml) and benzylamine (0.95 ml, 8.67 mmol) was added to it. Then the resulting solution was heated at 80



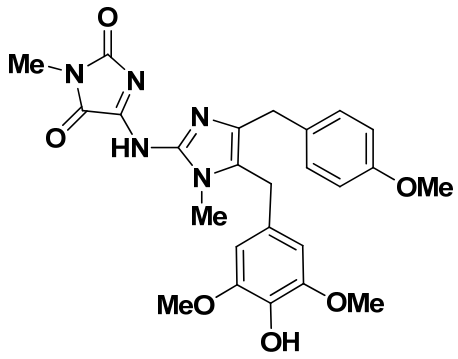
ºC for 10 h. The solvent was evaporated to give a crude oil, which was purified with a gradient column (EtOAc:hexanes, 3:1→EtOAc:acetone; 1:1) to get **5** (2.96 g, 90%) as a pale brown oil; ^{1}H NMR ($CDCl_3$): δ = 7.45 (d, *J* = 7.4 Hz, 2H), 7.40 (s, 1H), 7.38-7.26 (m, 3H), 7.19 (d, *J* = 8.7 Hz, 2H), 6.78 (d, *J* = 8.7 Hz, 2H), 6.15 (s, 2H), 4.96 (s, 2H), 3.88 (s, 2H), 3.87 (s, 2H), 3.75 (s, 3H), 3.64 (s, 6H), 3.34 (s, 3H); ^{13}C NMR: δ = 157.9, 153.8, 138.8, 137.9, 136.8, 135.5, 134.1, 133.1, 129.5, 128.5, 128.2, 127.9, 125.5, 113.9, 105.1, 75.1, 56.1, 55.3, 32.8, 31.9, 29.5; IR (cm^{-1}): 2929, 2857, 1691, 1507, 1391, 1251, 1176, 1150, 910; HR-ESIMS (*m/z*): Calc. for $C_{28}H_{31}N_2O_4$ $[M+H]^+$ 459.2278; found: 459.2278.

2-Azido-5-(4-benzyloxy-3,5-dimethoxybenzyl)-4-(4-methoxybenzyl)-1-methyl-1H-imidazole (20): *n*-Butyl lithium (1.6 M solution in hexanes, 1.31 ml, 2.09 mmol) was added dropwise to a stirred solution of **5** (870 mg, 1.90 mmol) in dry THF (20 ml) at -78 °C and the reaction was stirred for 1 h at the same temperature. The cooling bath was removed for 10 min, then the reaction mixture was re-cooled to -78 °C, and then $TrisN_3$ (706 mg, 2.28 mmol) was added. After stirring for an additional 45 min at -78 °C, the reaction mixture was quenched by the addition of sat. NH_4Cl (5 ml). The aqueous layer was extracted with EtOAc (3x15 ml), and the combined organic extracts were dried (Na_2SO_4) and concentrated to give a pale brown oil, which was purified through a short column of silica gel (hexane/EtOAc, 7:3) to give azide **20** (600 mg, 63%) as a pale brown oil; ^{1}H NMR ($CDCl_3$): δ = 7.46 (d, *J* = 7.7 Hz, 2H), 7.38-7.26 (m, 3H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 6.16 (s, 2H), 4.95(s, 2H), 3.85 (s, 2H), 3.80 (s, 2H), 3.76 (s, 3H), 3.64 (s, 6H), 3.08 (s, 3H); ^{13}C NMR: δ = 158.0, 153.7, 139.2, 137.9, 136.5, 135.5, 134.0, 132.9, 129.5,



128.6, 128.2, 127.9, 124.6, 113.9, 105.0, 75.0, 56.1, 55.3, 32.7, 30.0, 29.6; IR (cm^{-1}): 2929, 2857, 2129, 1691, 1507, 1391, 1252, 1150, 1124, 909, 836, 779; HR-ESIMS (*m/z*): Calc. for $C_{28}H_{30}N_5O_4$ $[M+H]^+$ 500.2292; found: 500.2290.

Naamine G (2): Azide **20** (600 mg, 1.20 mmol) was dissolved in EtOH (15 ml) and stirred overnight under a hydrogen atmosphere (55 psi) in the presence of 20% $Pd(OH)_2$ on charcoal (100 mg) at rt. The catalyst was filtered through a pad of Celite and the filtrate was concentrated to give naamine G, **2** (430 mg, 95%) as a greenish-yellow solid; m.p. = 218-220 °C; ^{1}H NMR (CD_3OD): δ = 7.17 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.34 (s, 2H), 3.92 (s, 2H), 3.84 (s, 2H), 3.74 (s, 3H), 3.69 (s, 6H), 3.23 (s, 3H); ^{13}C NMR: δ = 158.8, 148.3, 146.5, 134.3, 129.8, 129.2, 127.3, 122.8, 122.4, 114.0, 105.2, 55.5, 54.5, 28.8, 28.3, 27.9; IR (cm^{-1}): 3244 (br), 3004, 2836, 1667, 1654, 1609, 1500, 1461, 1429, 1245, 1216, 1110, 1022; HR-DART-MS (*m/z*): Calc. for $C_{21}H_{26}N_3O_4$ $[M+H]^+$ 384.1918; found: 384.1907.

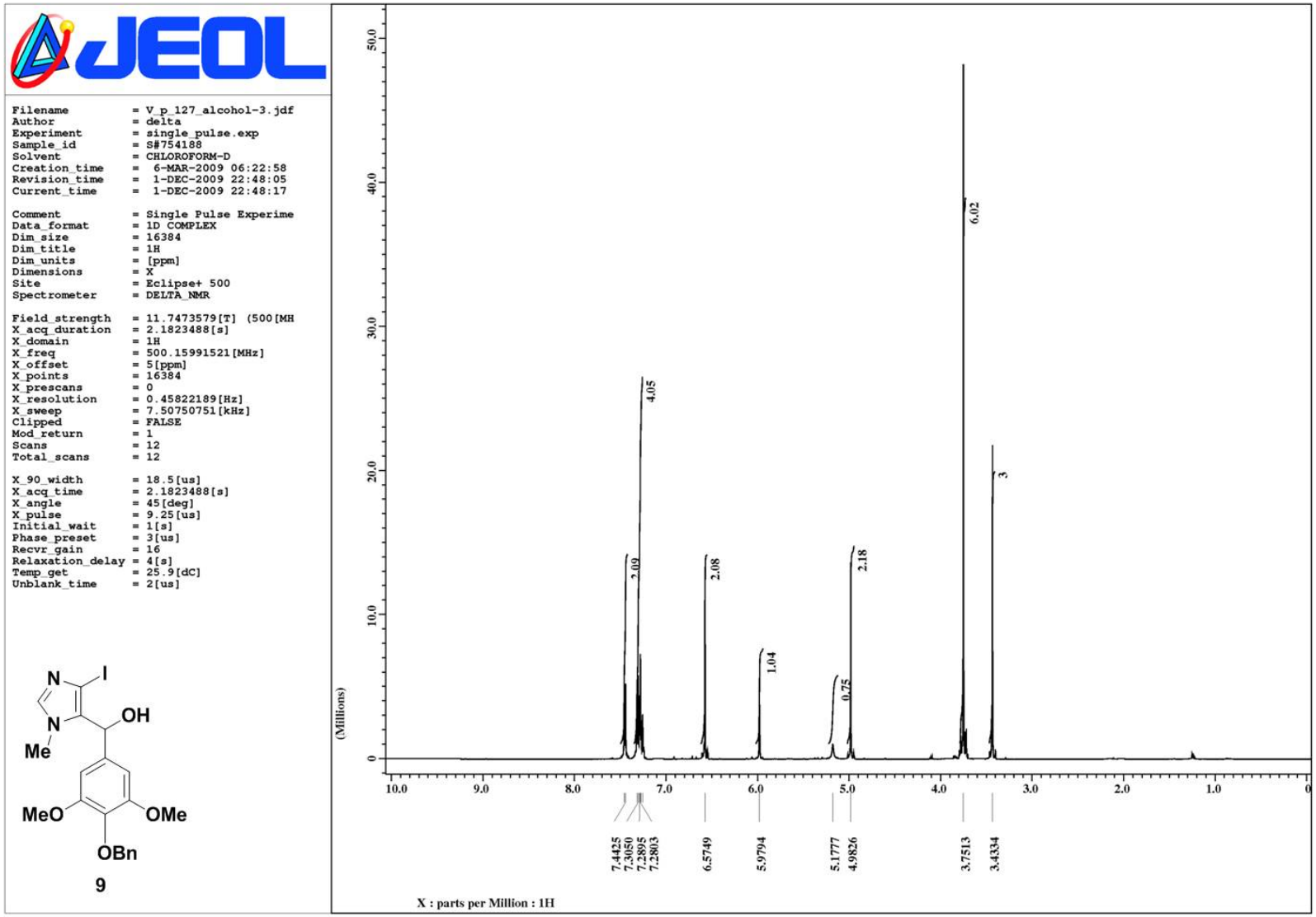


Naamidine H (3): *N,O*-Bis(trimethysilyl)acetamide (0.63 ml, 2.58 mmol) was added to a solution of 1-Methyparabanic acid (331 mg, 4.11 mmol) in dry CH_3CN (10 ml) under an N_2 atmosphere and the resulting mixture was heated at reflux temperature for 1.5 h. Then, the solvent was removed by distillation and

to the resulting yellow residue was added naamine G (198 mg, 0.52 mmol) under N_2. After, this mixture was heated at 80 ºC overnight in dry toluene (5 ml), water (5 ml) was added and the organic layer was extracted in to EtOAc. The dried organic layer (Na_2SO_4) was concentrated and the yellow residue was purified over silica gel (EtOAc/hexanes, 4/6) to provide naamidine H (**3**) as a yellow amorphous solid (205 mg, 80%); m.p. = 204-205 ºC; ^{1}H NMR ($CDCl_3$): δ = 7.13 (d, *J* = 8.7 Hz, 2H), 6.98 (br, 1H), 6.78 (d, *J* = 8.7 Hz, 2H), 6.14 (s, 2H), 3.89 (s, 2H), 3.88 (s, 2H), 3.75 (s, 3H), 3.69 (s, 6H), 3.49 (s, 3H), 3.47 (s, 3H), 3.16 (s, 3H); ^{13}C NMR δ = 162.3, 158.3, 155.5, 147.4, 146.6, 144.7, 136.1, 133.7, 131.7, 129.4, 128.1, 126.7, 114.1, 104.7, 56.3, 55.4, 32.3, 30.0, 29.7, 24.8; IR (cm^{-1}): 3501, 3212, 2929, 2837, 1784, 1718, 1652, 1511, 1392, 1113, 1039, 1020, 918; HR-DART-MS (*m/z*): Calc. for $C_{25}H_{28}N_5O_6$ $[M+H]^+$ 494.2034; found: 494.2049.

References:

1. Deveau, A. M.; Costa, N. E.; Joshi, E. M.; Macdonald, T. L. *Bioorg. Med. Chem. Lett.* **2008**, *18*, 3522-3525.
2. Brown, E.; Daugan, A. *Heterocycles* **1987**, *26*, 1169-1172.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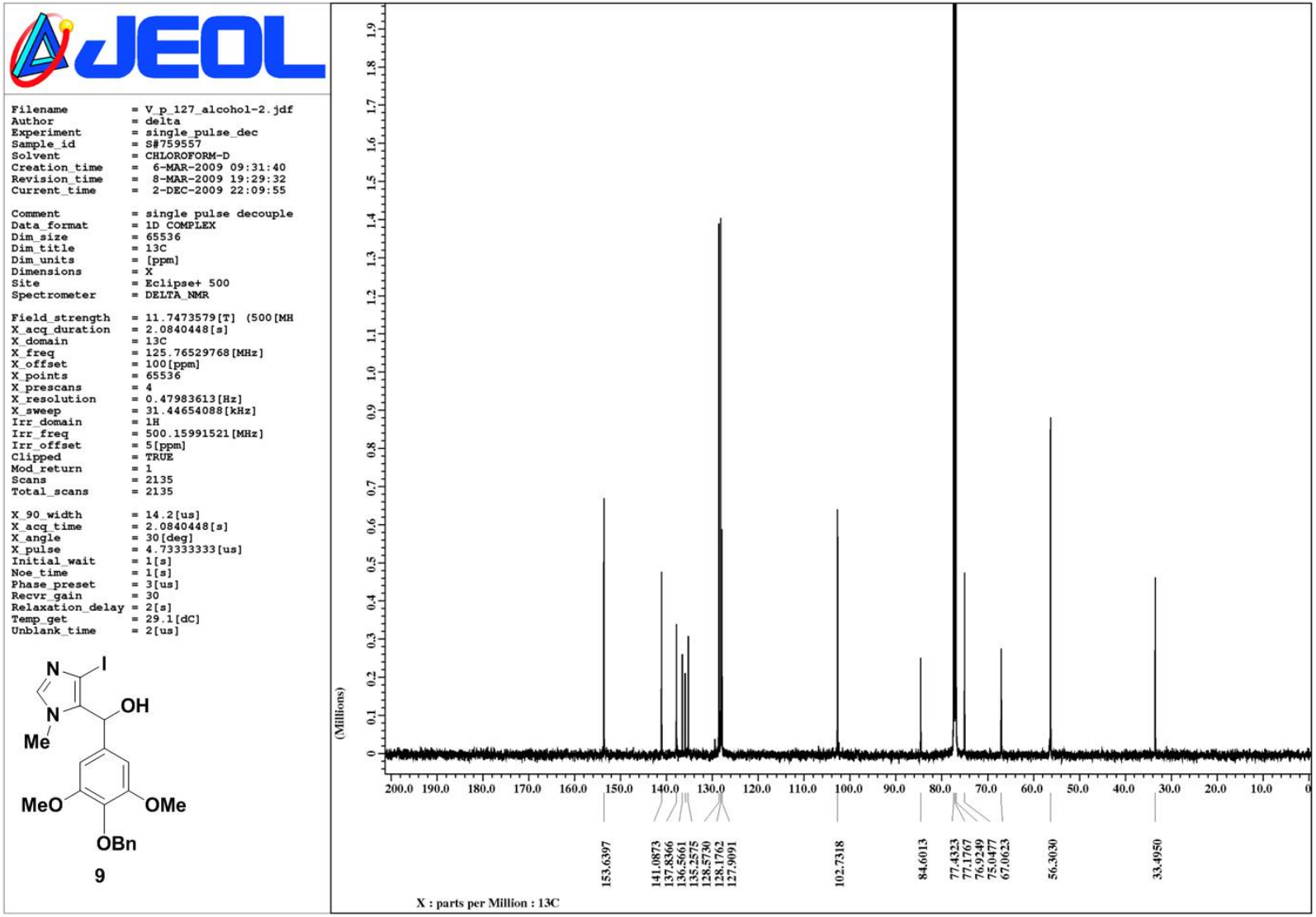
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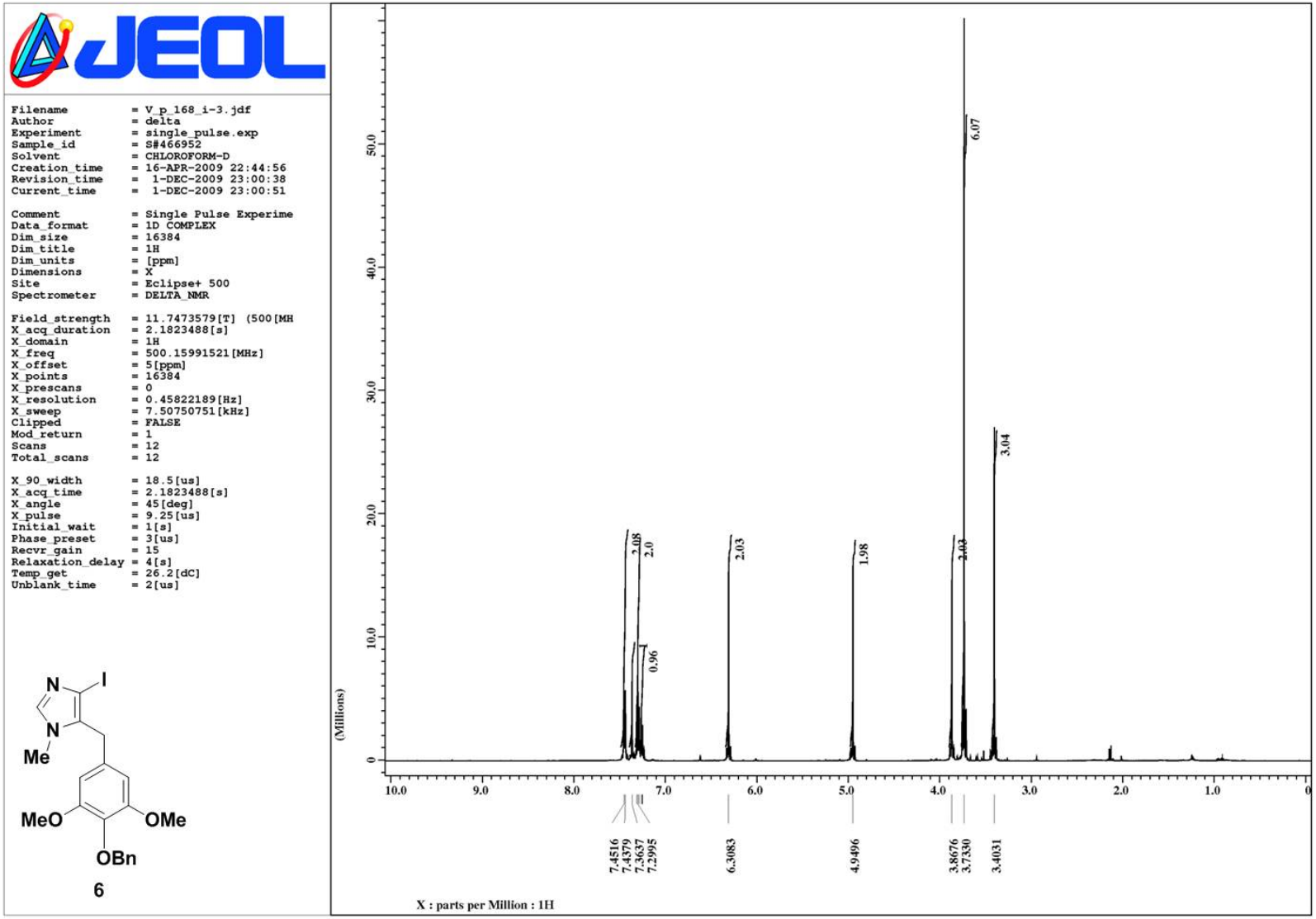
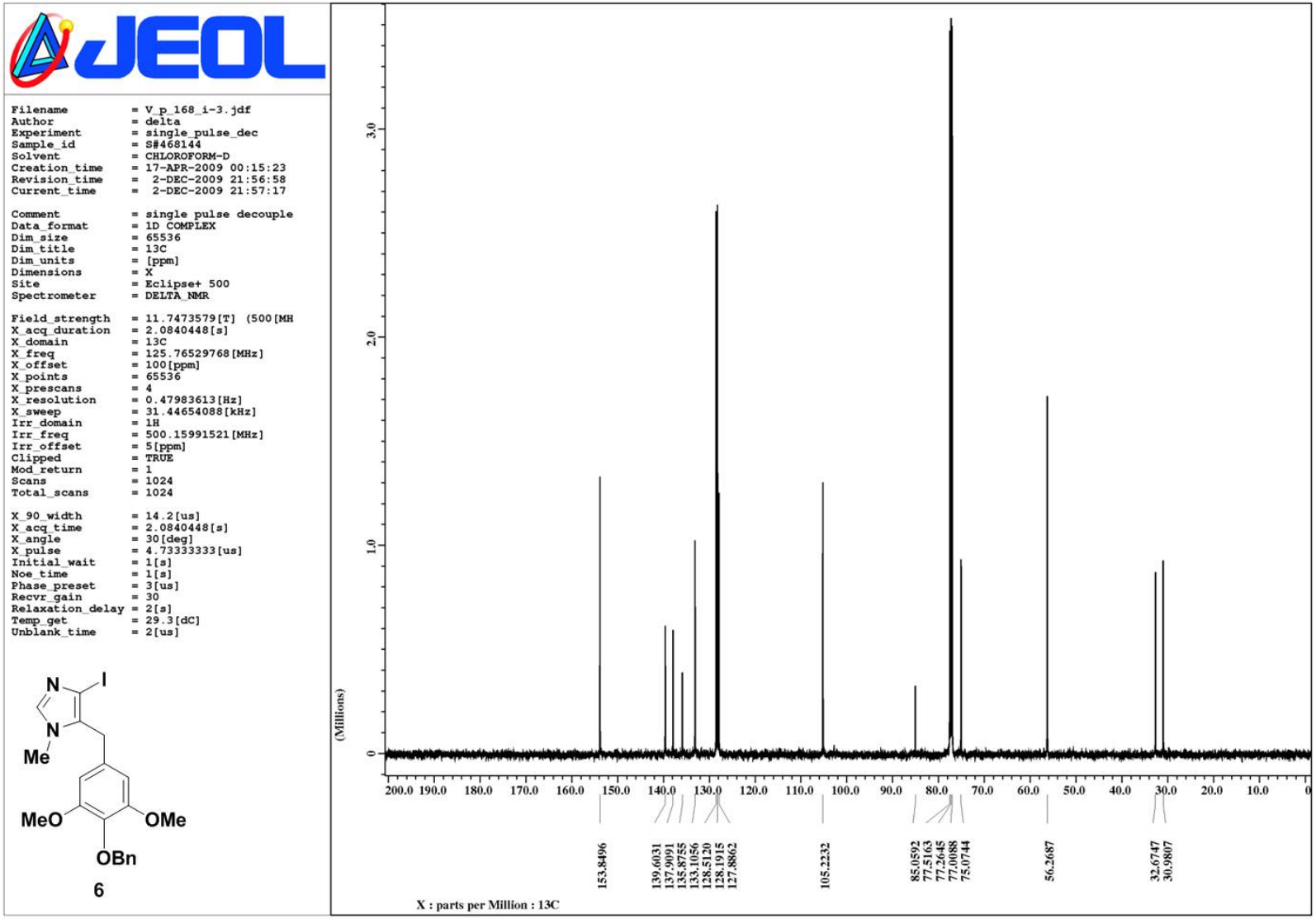
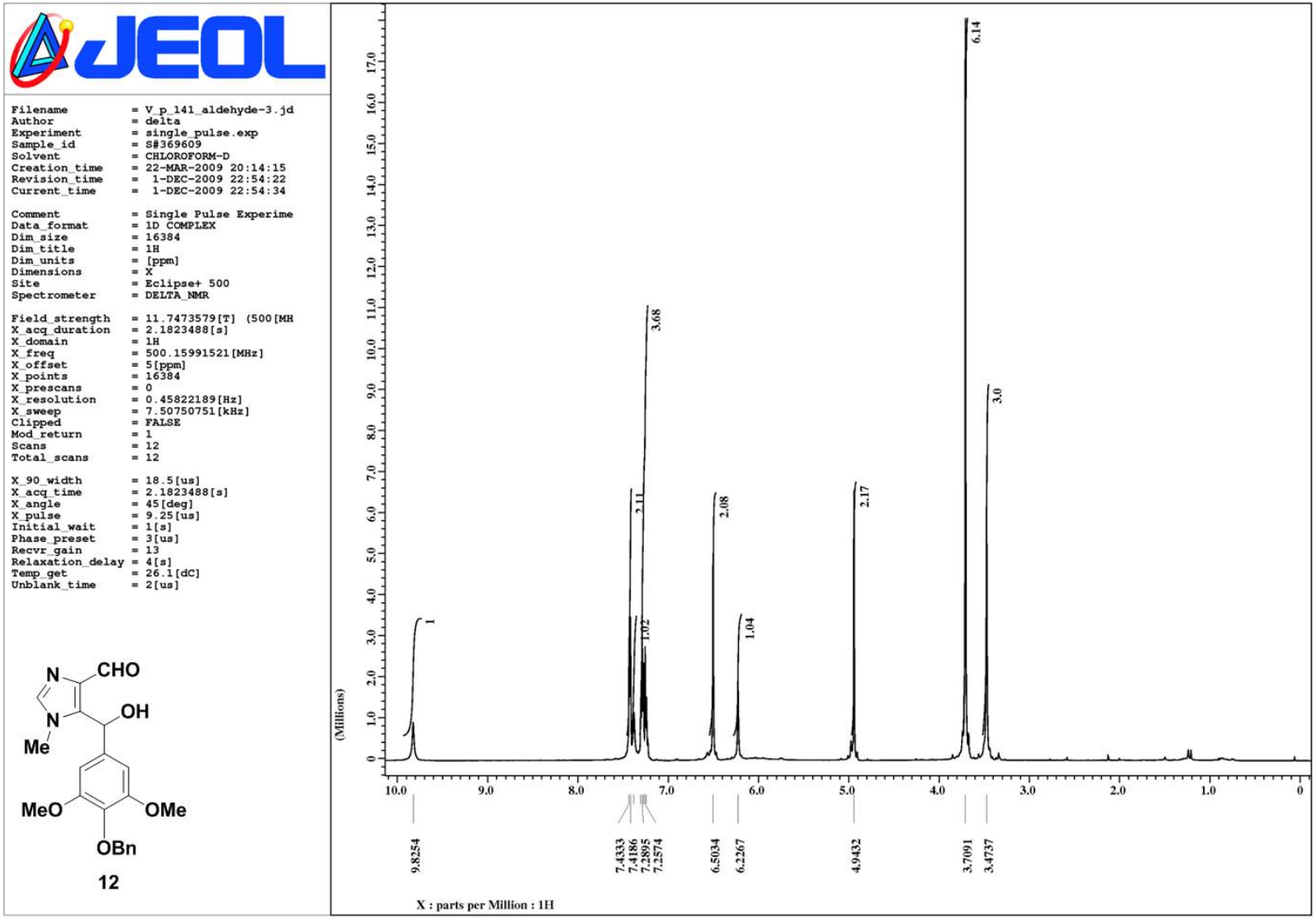
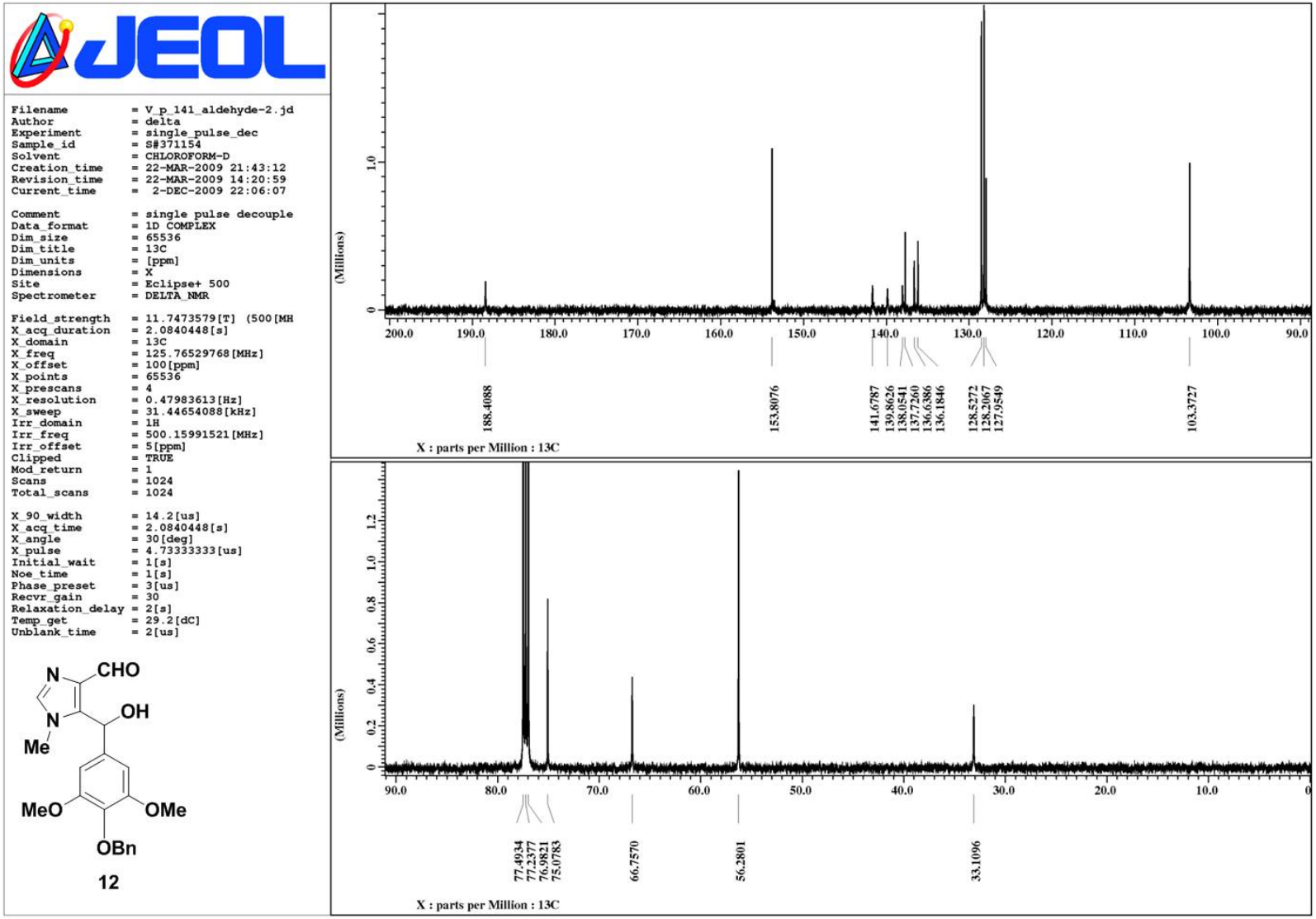
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 2135
Total_scans = 2135
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 29.1[dC]
Unblank_time = 2[us]
N
I
N
OH
Me
MeO
OMe
OBn
9
(Millions)
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
153.6397
141.0873
137.8366
136.5661
135.2575
128.5730
128.1762
127.9091
102.7318
84.6013
77.4323
77.1767
76.9249
75.0477
67.0623
56.3030
33.4950
X : parts per Million : 13C


Filename = V_p_168_i-3.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#466952
Solvent = CHLOROFORM-D
Creation_time = 16-APR-2009 22:44:56
Revision_time = 1-DEC-2009 23:00:38
Current_time = 1-DEC-2009 23:00:51
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.1823488[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45822189[Hz]
X_sweep = 7.50750751[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1823488[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 15
Relaxation_delay = 4[s]
Temp_get = 26.2[dC]
Unblank_time = 2[us]
N
I
N
Me
MeO
OMe
OBn
6
(Millions)
50.0
40.0
30.0
20.0
10.0
0
6.07
3.04
2.08
2.0
2.03
1.98
3.03
0.96
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.4516
7.4379
7.3637
7.2995
6.3083
4.9496
3.8676
3.7330
3.4031
X : parts per Million : 1H

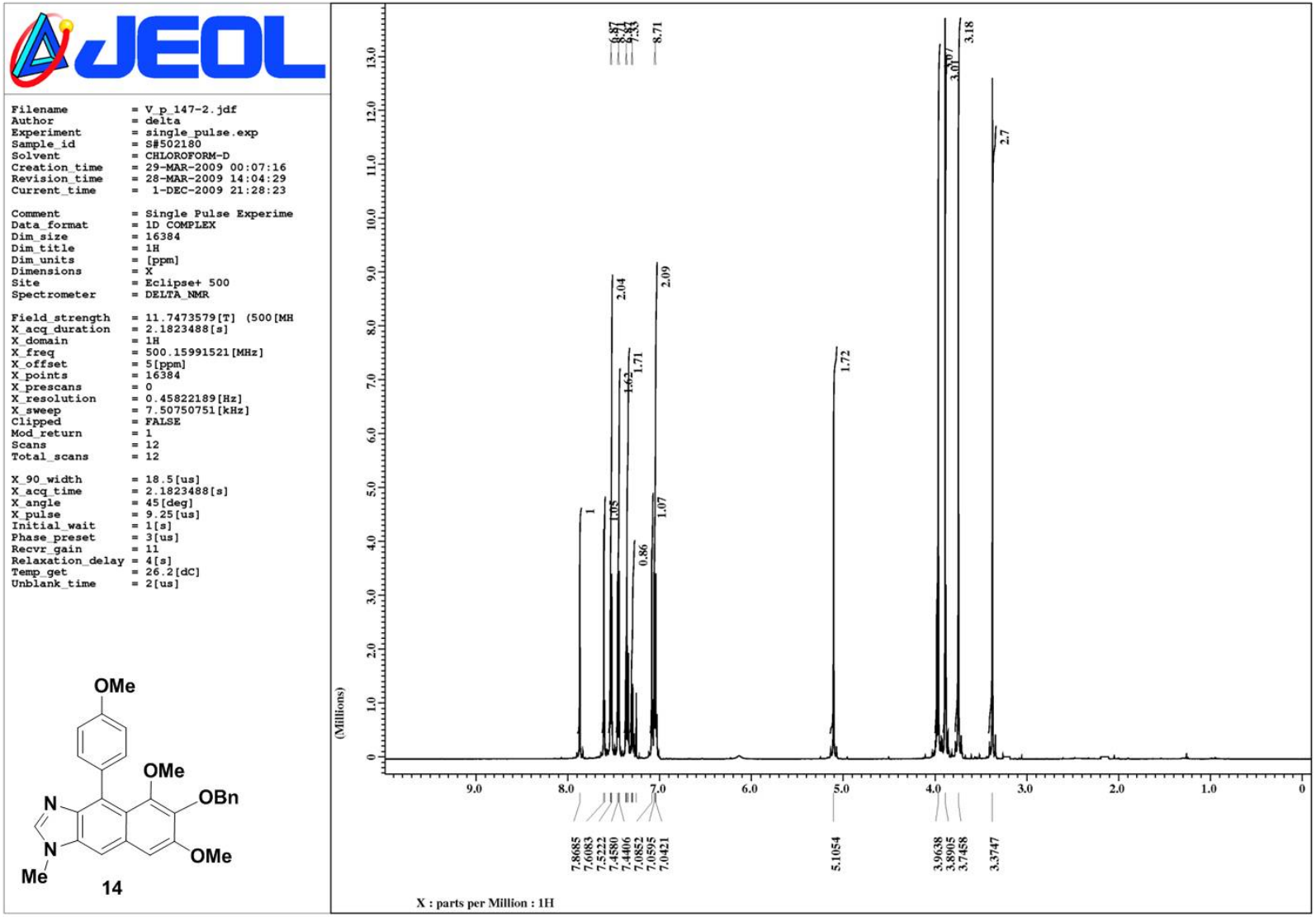

Filename = V_p_168_i-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#468144
Solvent = CHLOROFORM-D
Creation_time = 17-APR-2009 00:15:23
Revision_time = 2-DEC-2009 21:56:58
Current_time = 2-DEC-2009 21:57:17
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 1024
Total_scans = 1024
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 29.3[dC]
Unblank_time = 2[us]
N
I
N
Me
MeO
OMe
OBn
6
(Millions)
3.0
2.0
1.0
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
153.8496
139.6031
137.9091
135.8755
133.1056
128.5120
128.1915
127.8862
105.2232
85.0592
77.5163
77.2645
77.0088
75.0744
56.2687
32.6747
30.9807
X : parts per Million : 13C

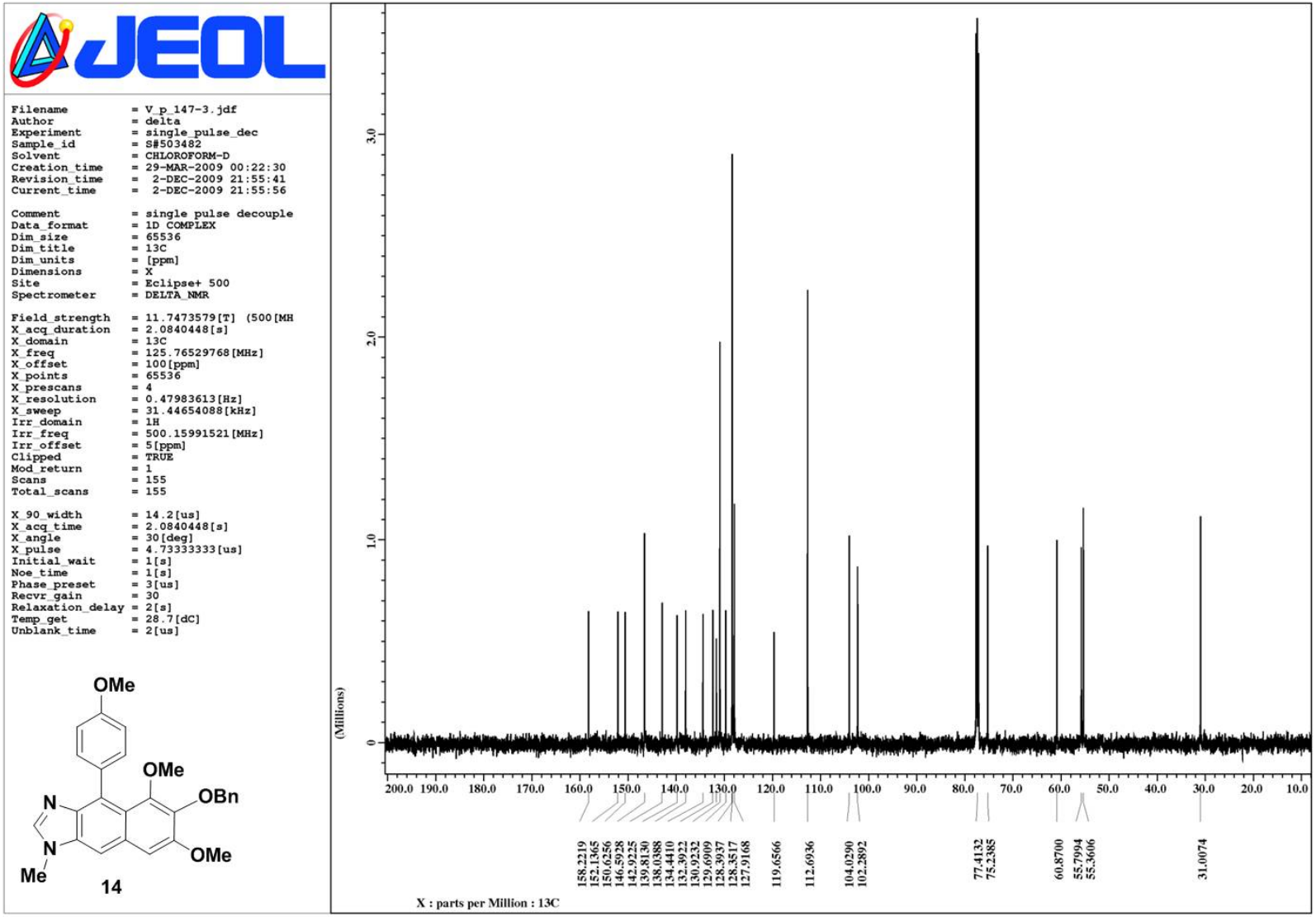

JEOL
Filename = V_p_141_aldehyde-3.jd
Author = delta
Experiment = single_pulse.exp
Sample_id = S#369609
Solvent = CHLOROFORM-D
Creation_time = 22-MAR-2009 20:14:15
Revision_time = 1-DEC-2009 22:54:22
Current_time = 1-DEC-2009 22:54:34
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.1823488[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45822189[Hz]
X_sweep = 7.50750751[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1823488[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 13
Relaxation_delay = 4[s]
Temp_get = 26.1[dC]
Unblank_time = 2[us]
N
CHO
N
OH
Me
MeO
OMe
OBn
12
(Millions)
6.14
3.68
3.0
2.11
2.08
2.17
1.04
1.02
1
9.8254
7.4333
7.4186
7.2895
7.2574
6.5034
6.2267
4.9432
3.7091
3.4737
X : parts per Million : 1H

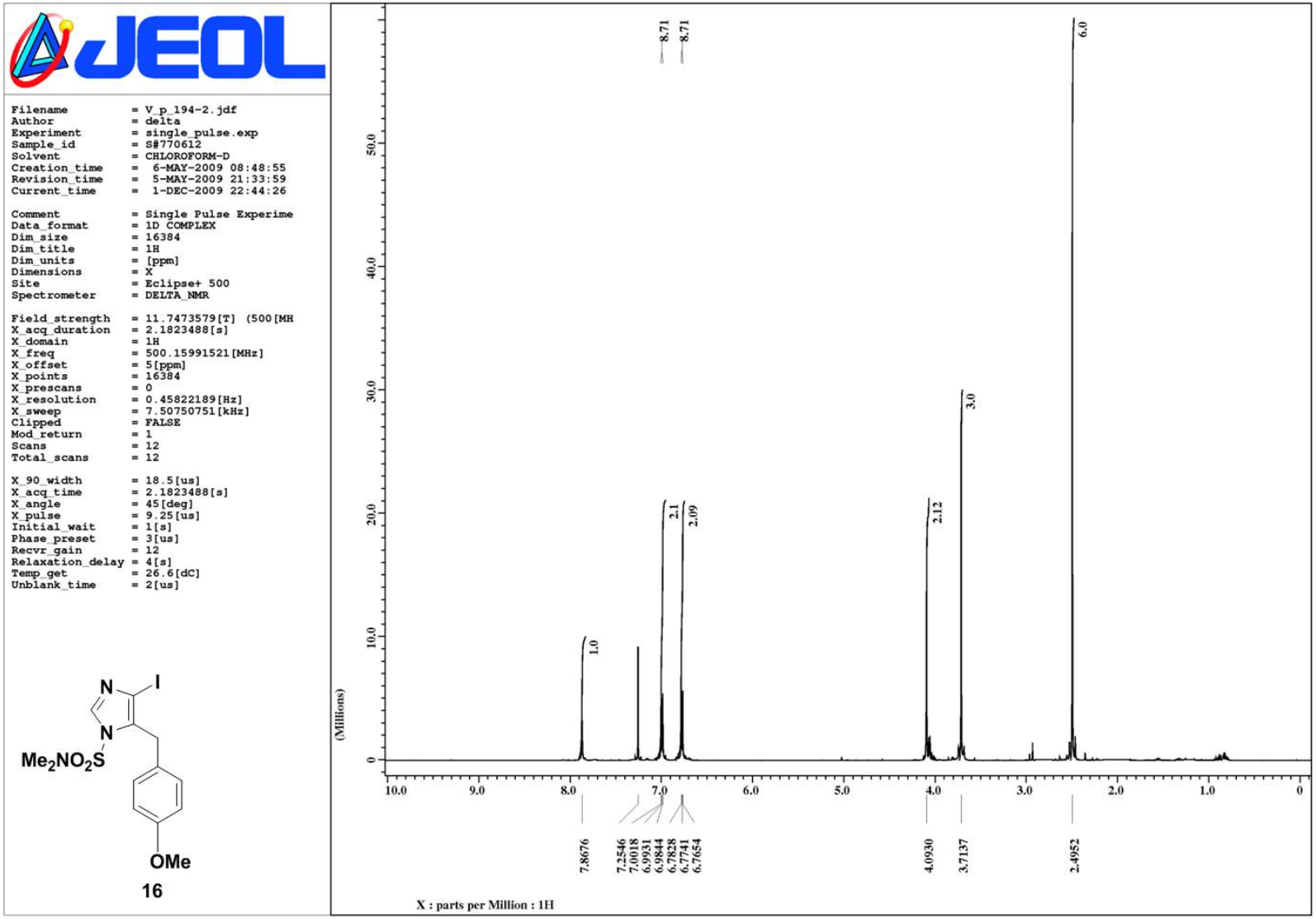


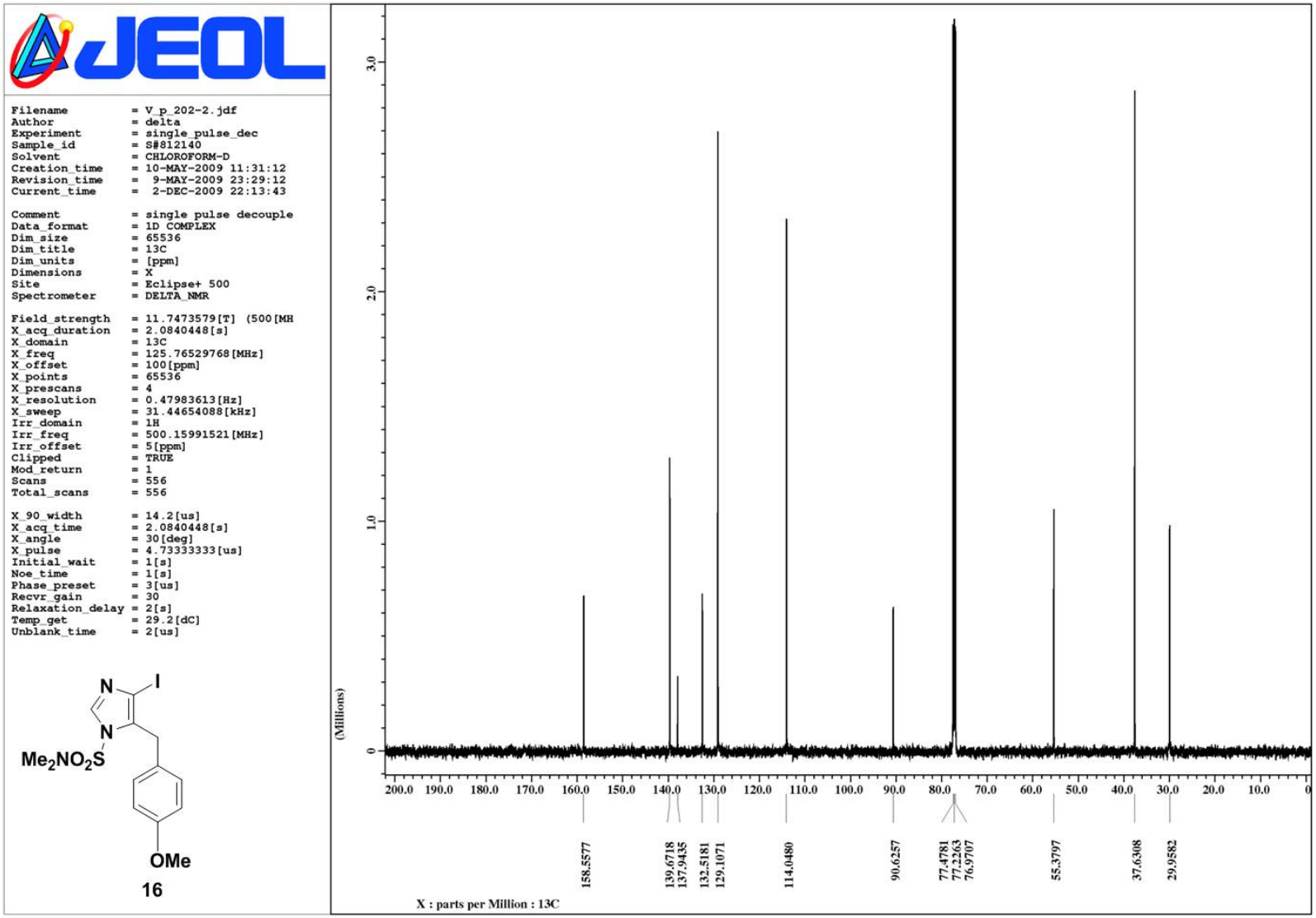
```
Filename          = V_p_141_aldehyde-2.jd
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#3711154
Solvent           = CHLOROFORM-D
Creation_time     = 22-MAR-2009 21:43:12
Revision_time     = 22-MAR-2009 14:20:59
Current_time      =  2-DEC-2009 22:06:07

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 65536
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 500
Spectrometer      = DELTA_NMR

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 2.0840448[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 65536
X_prescans        = 4
X_resolution      = 0.47983613[Hz]
X_sweep           = 31.44654088[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 1024
Total_scans       = 1024

X_90_width        = 14.2[us]
X_acq_time        = 2.0840448[s]
X_angle           = 30[deg]
X_pulse           = 4.73333333[us]
Initial_wait      = 1[s]
Noe_time          = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 2[s]
Temp_get          = 29.2[dC]
Unblank_time      = 2[us]
```

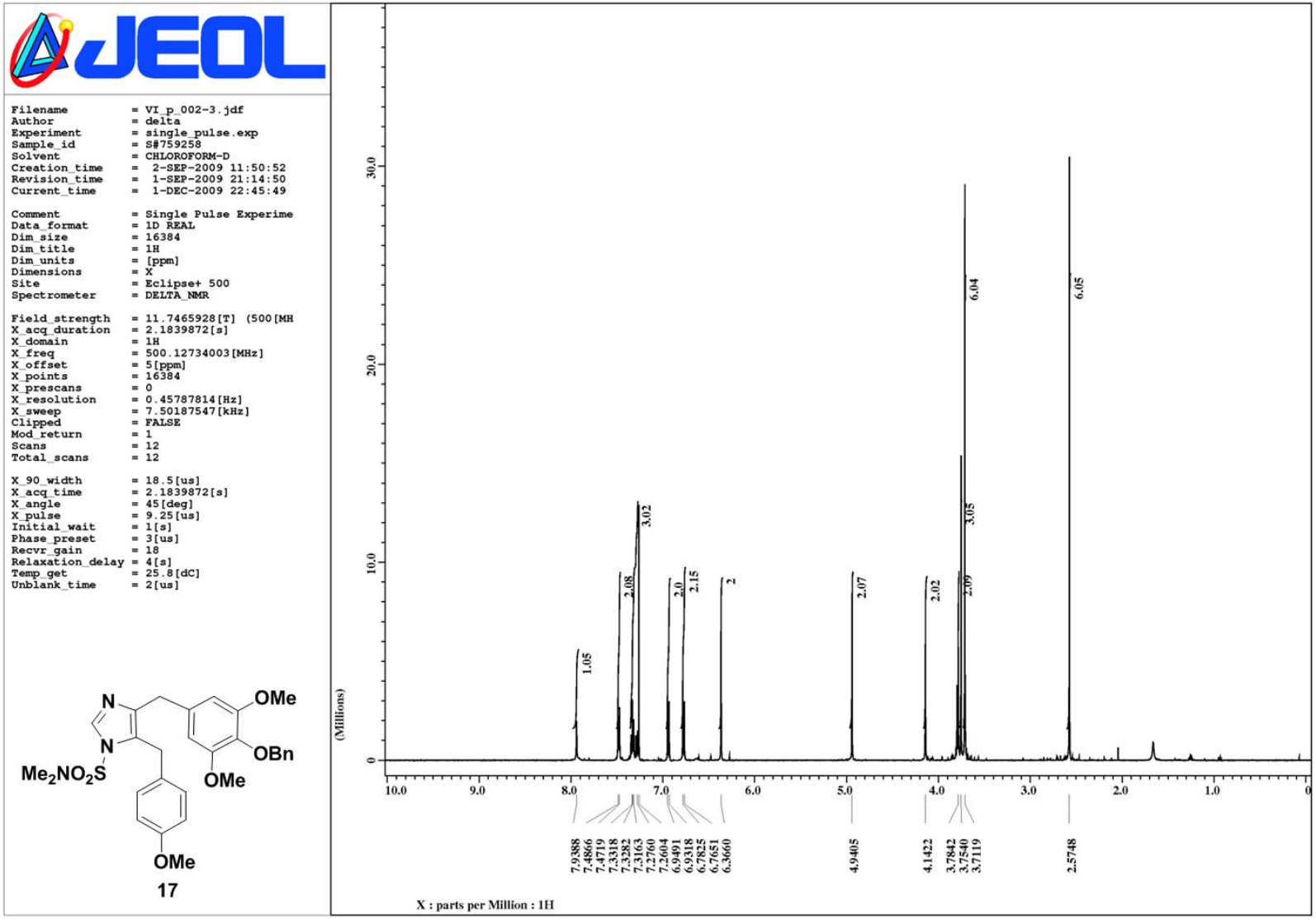

12

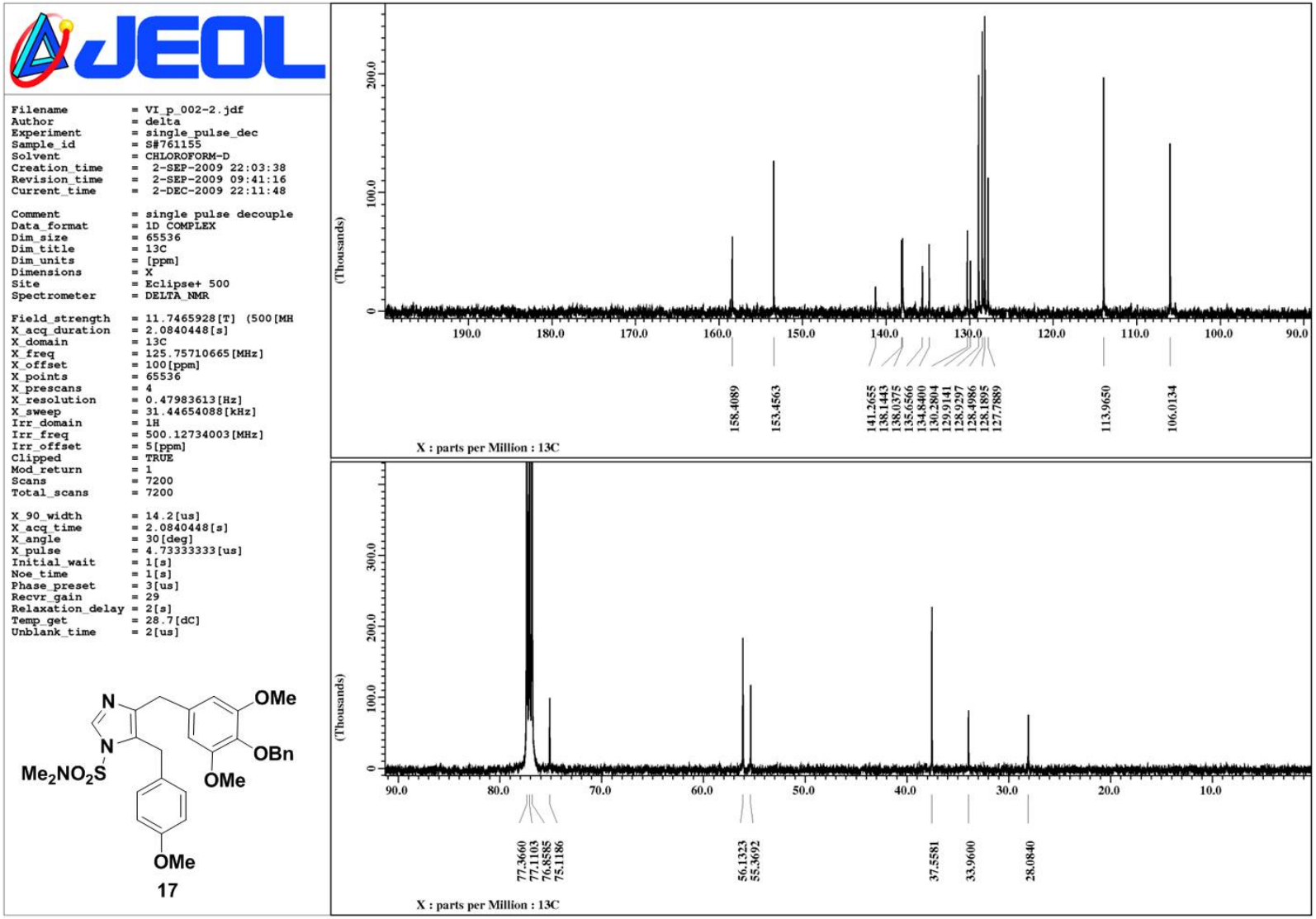

JEOL
Filename = V_p_147-2.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#502180
Solvent = CHLOROFORM-D
Creation_time = 29-MAR-2009 00:07:16
Revision_time = 28-MAR-2009 14:04:29
Current_time = 1-DEC-2009 21:28:23
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.1823488[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45822189[Hz]
X_sweep = 7.50750751[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1823488[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 11
Relaxation_delay = 4[s]
Temp_get = 26.2[dC]
Unblank_time = 2[us]
OMe
OMe
OBn
N
N
Me
OMe
14
(Millions)
8.71
3.18
3.07
3.01
2.7
2.09
2.04
1.72
1.71
1.63
1.07
1.05
1
0.86
7.8685
7.6083
7.5222
7.4580
7.4406
7.0852
7.0595
7.0421
5.1054
3.9638
3.8905
3.7458
3.3747
X : parts per Million : 1H

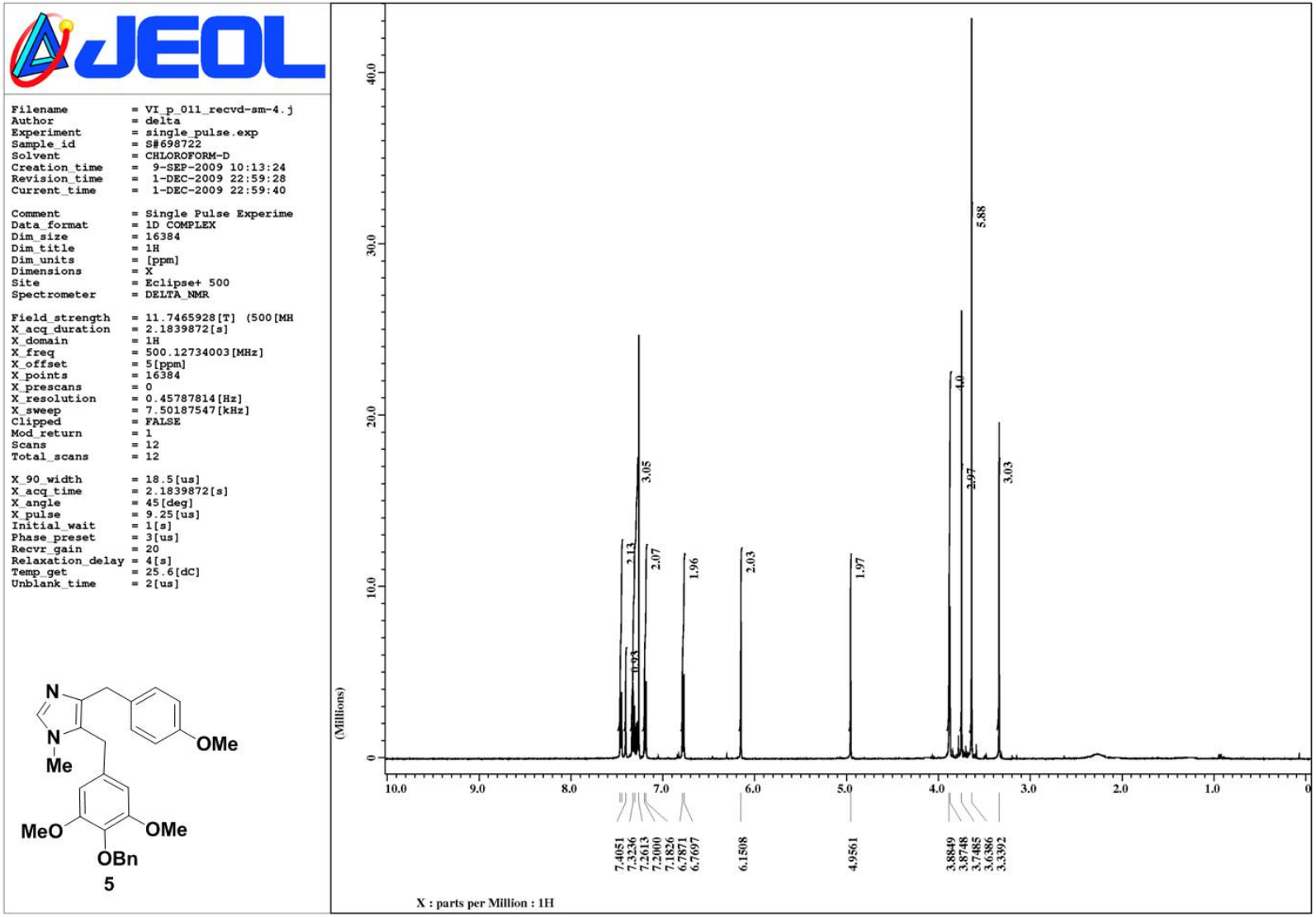

Filename = V_p_147-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#503482
Solvent = CHLOROFORM-D
Creation_time = 29-MAR-2009 00:22:30
Revision_time = 2-DEC-2009 21:55:41
Current_time = 2-DEC-2009 21:55:56
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 155
Total_scans = 155
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 28.7[dC]
Unblank_time = 2[us]
OMe
OMe
OBn
N
N
Me
OMe
14
(Millions)
3.0
2.0
1.0
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
158.2219
152.1365
150.6256
146.5928
142.9225
139.8130
138.0388
134.4410
132.3922
130.9232
129.6909
128.3937
128.3517
127.9168
119.6566
112.6936
104.0290
102.2892
77.4132
75.2385
60.8700
55.7994
55.3606
31.0074
X : parts per Million : 13C

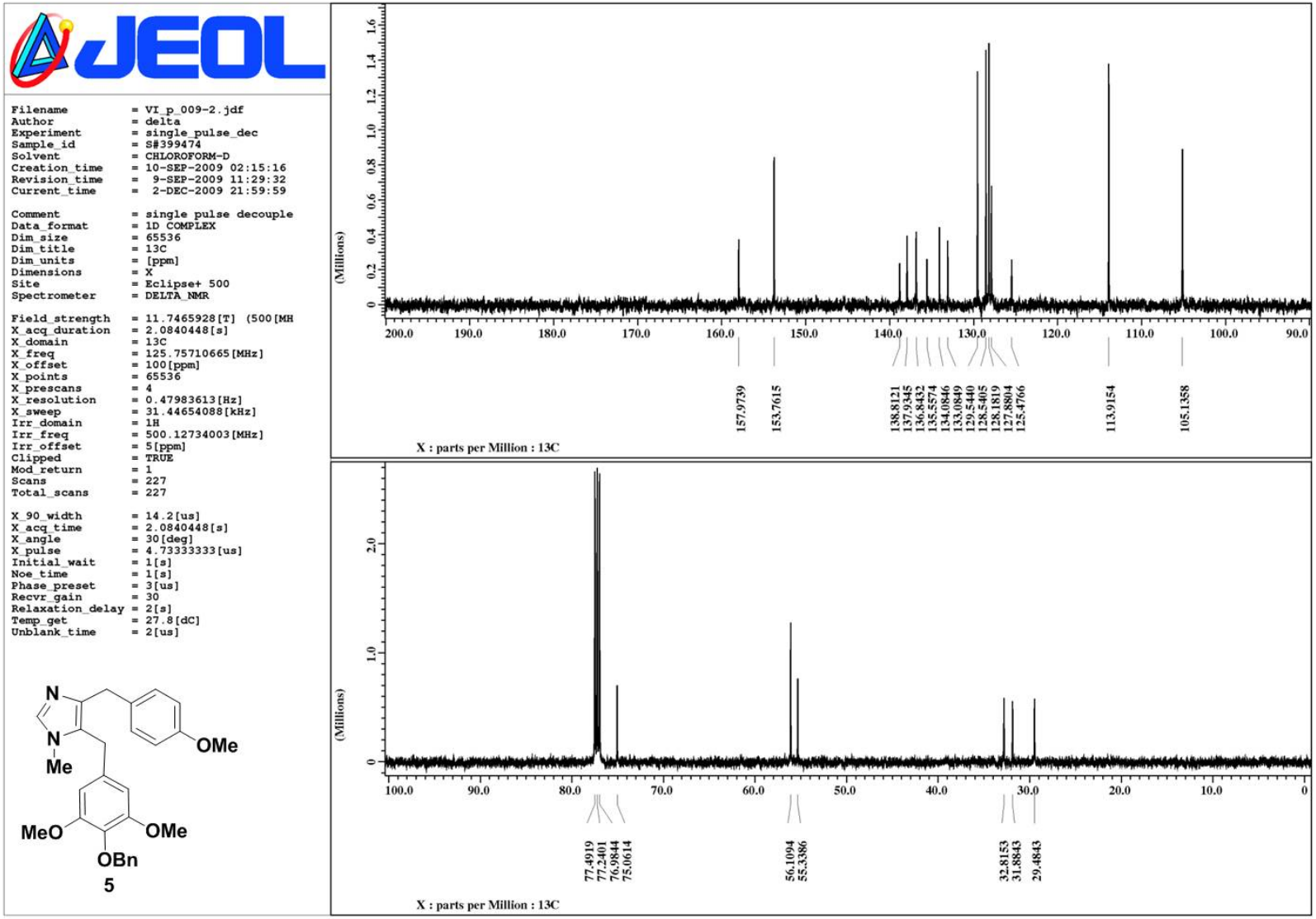

Filename = V_p_194-2.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#770612
Solvent = CHLOROFORM-D
Creation_time = 6-MAY-2009 08:48:55
Revision_time = 5-MAY-2009 21:33:59
Current_time = 1-DEC-2009 22:44:26
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.1823488[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45822189[Hz]
X_sweep = 7.50750751[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1823488[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 12
Relaxation_delay = 4[s]
Temp_get = 26.6[dC]
Unblank_time = 2[us]
N
I
N
Me2NO2S
OMe
16
8.71
8.71
6.0
3.0
2.1
2.09
2.12
1.0
(Millions)
50.0
40.0
30.0
20.0
10.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.8676
7.2546
7.0018
6.9931
6.9844
6.7828
6.7741
6.7654
4.0930
3.7137
2.4952
X : parts per Million : 1H

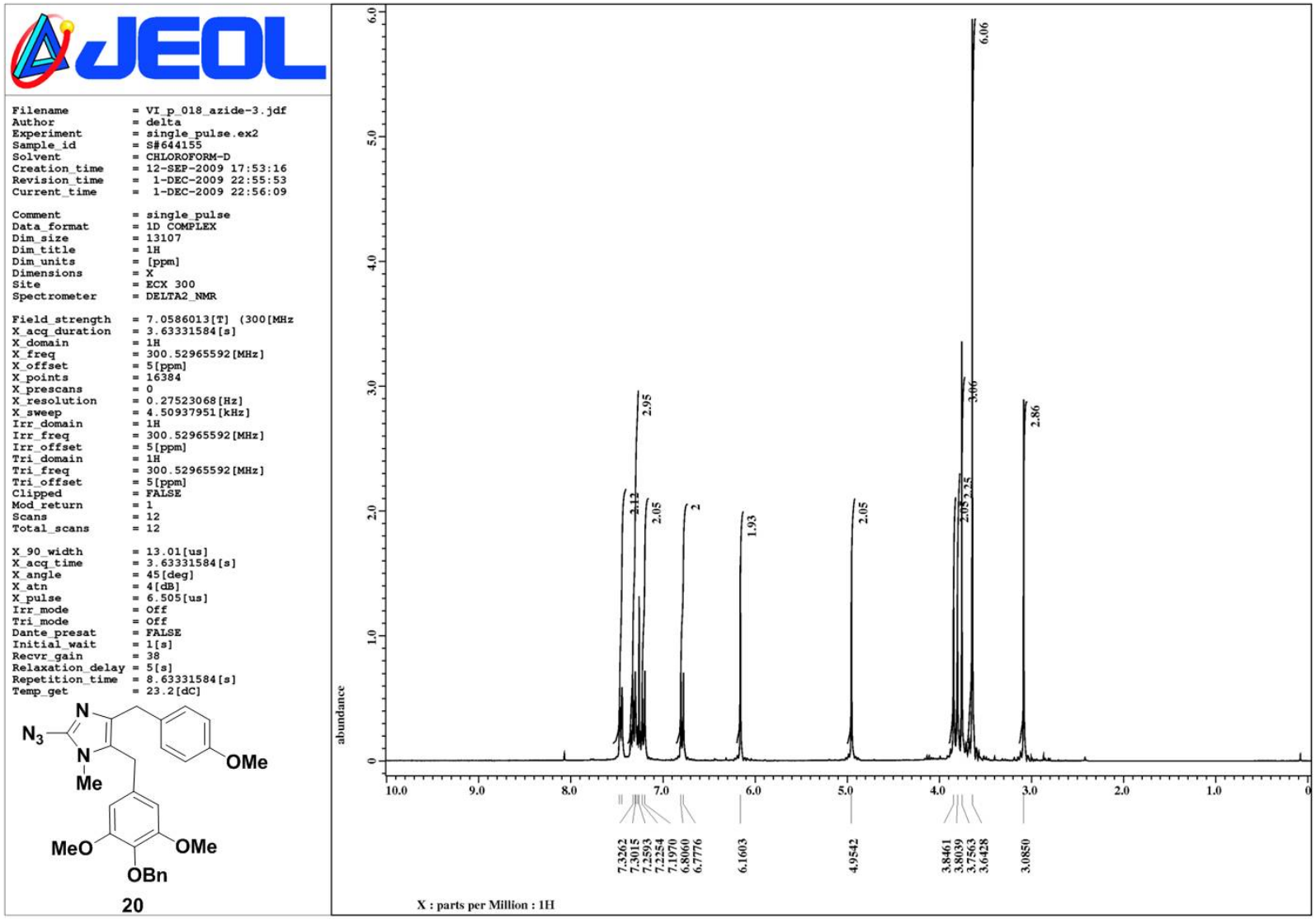

Filename = V_p_202-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#812140
Solvent = CHLOROFORM-D
Creation_time = 10-MAY-2009 11:31:12
Revision_time = 9-MAY-2009 23:29:12
Current_time = 2-DEC-2009 22:13:43
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 556
Total_scans = 556
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 29.2[dC]
Unblank_time = 2[us]
N
I
N
Me2NO2S
OMe
16
(Millions)
3.0
2.0
1.0
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
158.5577
139.6718
137.9435
132.5181
129.1071
114.0480
90.6257
77.4781
77.2263
76.9707
55.3797
37.6308
29.9582
X : parts per Million : 13C

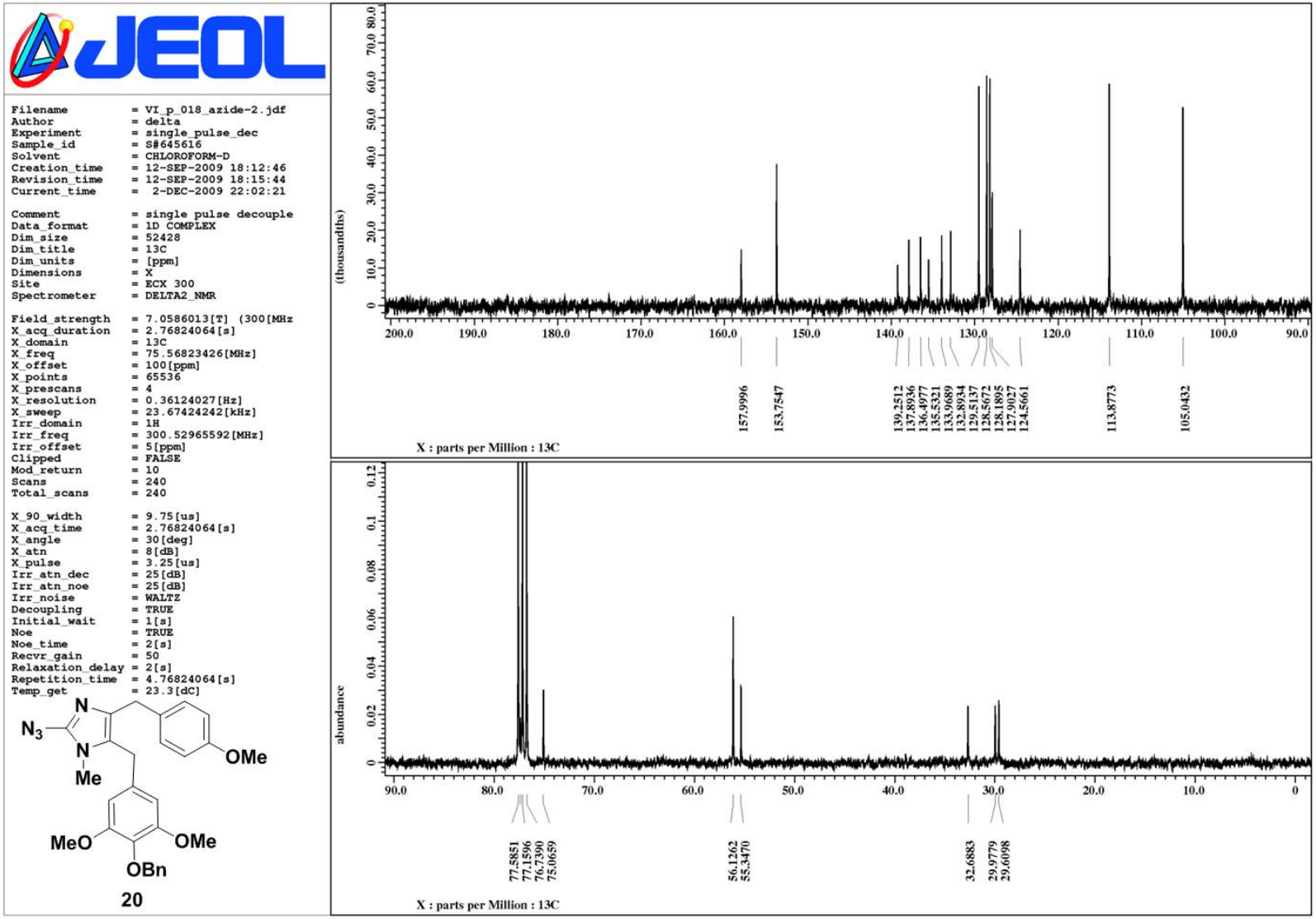

Filename = VI_p_002-3.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#759258
Solvent = CHLOROFORM-D
Creation_time = 2-SEP-2009 11:50:52
Revision_time = 1-SEP-2009 21:14:50
Current_time = 1-DEC-2009 22:45:49
Comment = Single Pulse Experime
Data_format = 1D REAL
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.1839872[s]
X_domain = 1H
X_freq = 500.12734003[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45787814[Hz]
X_sweep = 7.50187547[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1839872[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 18
Relaxation_delay = 4[s]
Temp_get = 25.8[dC]
Unblank_time = 2[us]
N
N
Me2NO2S
OMe
OBn
OMe
OMe
17
(Millions)
30.0
20.0
10.0
0
1.05
2.08
3.02
2.0
2.15
2
2.07
2.02
2.09
3.05
6.04
6.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.9388
7.4866
7.4719
7.3318
7.3282
7.3163
7.2760
7.2604
6.9491
6.9318
6.7825
6.7651
6.3660
4.9405
4.1422
3.7842
3.7540
3.7119
2.5748
X : parts per Million : 1H

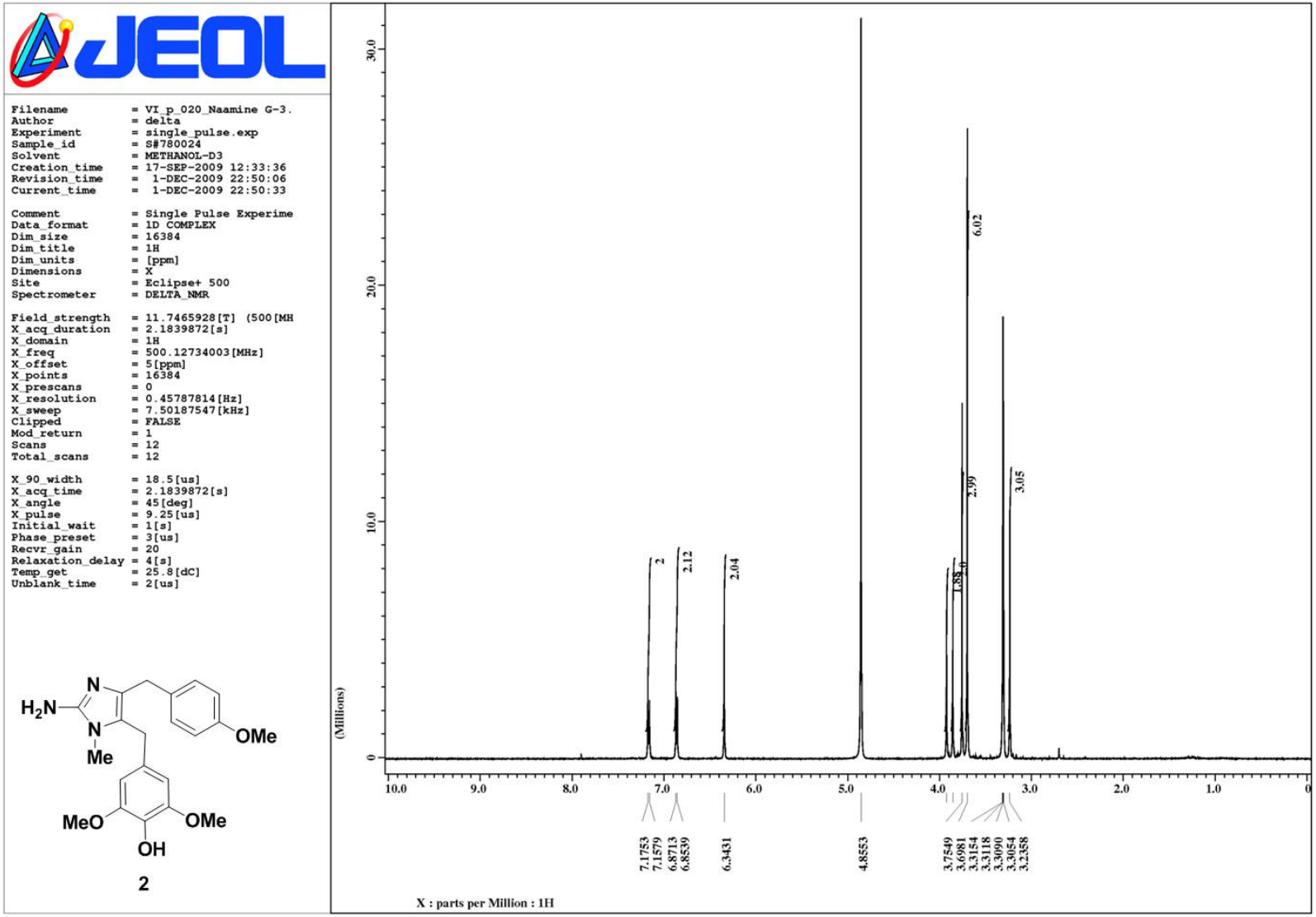

JEOL
Filename = VI_p_002-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#761155
Solvent = CHLOROFORM-D
Creation_time = 2-SEP-2009 22:03:38
Revision_time = 2-SEP-2009 09:41:16
Current_time = 2-DEC-2009 22:11:48
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.75710665[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.12734003[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 7200
Total_scans = 7200
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 29
Relaxation_delay = 2[s]
Temp_get = 28.7[dC]
Unblank_time = 2[us]
N
N
Me2NO2S
OMe
OBn
OMe
OMe
17
(Thousands)
200.0
100.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
158.4089
153.4563
141.2655
138.1443
138.0375
135.6566
134.8400
130.2804
129.9141
128.9297
128.4986
128.1895
127.7889
113.9650
106.0134
X : parts per Million : 13C
(Thousands)
300.0
200.0
100.0
0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
77.3660
77.1103
76.8585
75.1186
56.1323
55.3692
37.5581
33.9600
28.0840
X : parts per Million : 13C

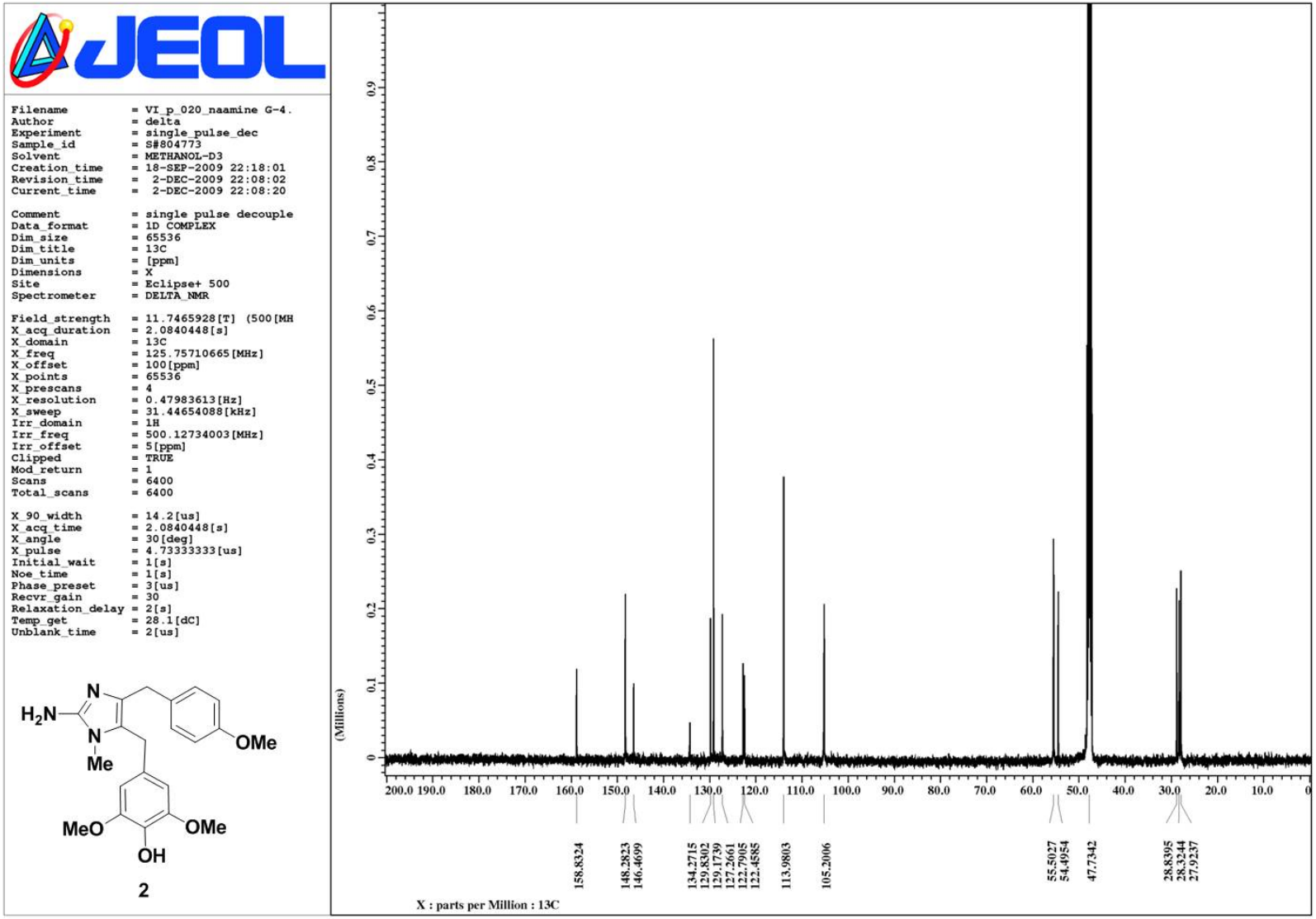

Filename = VI_p_011_recvd-sm-4.j
Author = delta
Experiment = single_pulse.exp
Sample_id = S#698722
Solvent = CHLOROFORM-D
Creation_time = 9-SEP-2009 10:13:24
Revision_time = 1-DEC-2009 22:59:28
Current_time = 1-DEC-2009 22:59:40
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.1839872[s]
X_domain = 1H
X_freq = 500.12734003[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45787814[Hz]
X_sweep = 7.50187547[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1839872[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 20
Relaxation_delay = 4[s]
Temp_get = 25.6[dC]
Unblank_time = 2[us]
N
N
Me
OMe
MeO
OMe
OBn
5
(Millions)
40.0
30.0
20.0
10.0
0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
3.05
2.13
0.93
2.07
1.96
2.03
1.97
5.88
4.0
2.97
3.03
7.4051
7.3236
7.2613
7.2000
7.1826
6.7871
6.7697
6.1508
4.9561
3.8849
3.8748
3.7485
3.6386
3.3392
X : parts per Million : 1H


JEOL
Filename = VI_p_009-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#399474
Solvent = CHLOROFORM-D
Creation_time = 10-SEP-2009 02:15:16
Revision_time = 9-SEP-2009 11:29:32
Current_time = 2-DEC-2009 21:59:59
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.75710665[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.12734003[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 227
Total_scans = 227
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 27.8[dC]
Unblank_time = 2[us]
N
N
Me
OMe
MeO
OMe
OBn
5
(Millions)
157.9739
153.7615
138.8121
137.9345
136.8432
135.5574
134.0846
133.0849
129.5440
128.5405
128.1819
127.8804
125.4766
113.9154
105.1358
X : parts per Million : 13C
(Millions)
77.4919
77.2401
76.9844
75.0614
56.1094
55.3386
32.8153
31.8843
29.4843
X : parts per Million : 13C


JEOL
Filename = VI_p_018_azide-3.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#644155
Solvent = CHLOROFORM-D
Creation_time = 12-SEP-2009 17:53:16
Revision_time = 1-DEC-2009 22:55:53
Current_time = 1-DEC-2009 22:56:09
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 3.63331584[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.27523068[Hz]
X_sweep = 4.50937951[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965592[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 13.01[us]
X_acq_time = 3.63331584[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.505[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 23.2[dC]
N3
N
N
Me
OMe
MeO
OMe
OBn
20
abundance
6.06
2.95
3.06
2.86
2.13
2.05
2
1.93
2.05
2.05
2.25
7.3262
7.3015
7.2593
7.2254
7.1970
6.8060
6.7776
6.1603
4.9542
3.8461
3.8039
3.7563
3.6428
3.0850
X : parts per Million : 1H

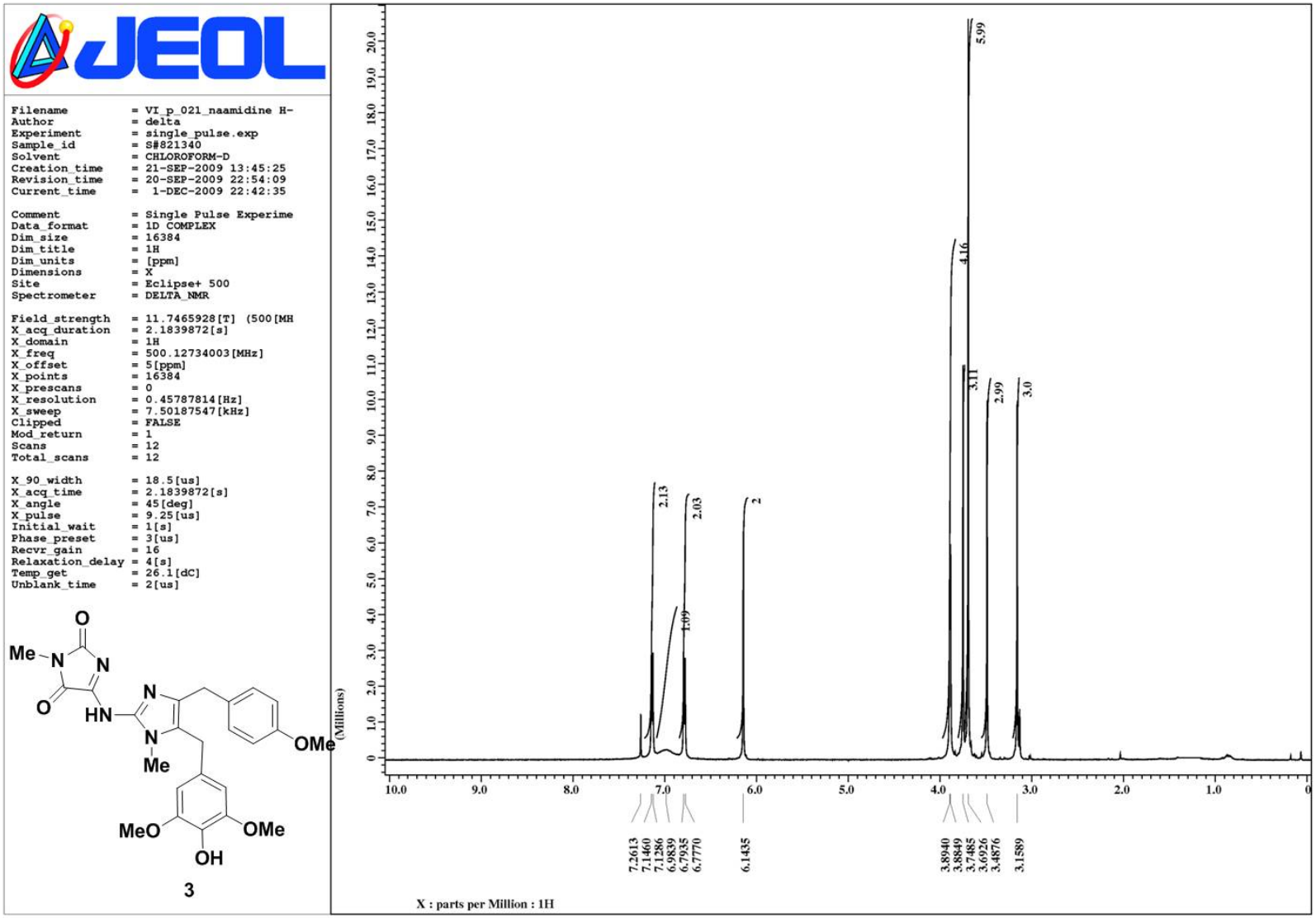

Filename = VI_p_018_azide-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#645616
Solvent = CHLOROFORM-D
Creation_time = 12-SEP-2009 18:12:46
Revision_time = 12-SEP-2009 18:15:44
Current_time = 2-DEC-2009 22:02:21
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.36124027[Hz]
X_sweep = 23.67424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 240
Total_scans = 240
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_noe = 25[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 50
Relaxation_delay = 2[s]
Repetition_time = 4.76824064[s]
Temp_get = 23.3[dC]
N
N3
N
Me
OMe
MeO
OMe
OBn
20
(thousandths)
157.9996
153.7547
139.2512
137.8936
136.4977
135.5321
133.9689
132.8934
129.5137
128.5672
128.1895
127.9027
124.5661
113.8773
105.0432
X : parts per Million : 13C
abundance
77.5851
77.1596
76.7390
75.0659
56.1262
55.3470
32.6883
29.9779
29.6098
X : parts per Million : 13C

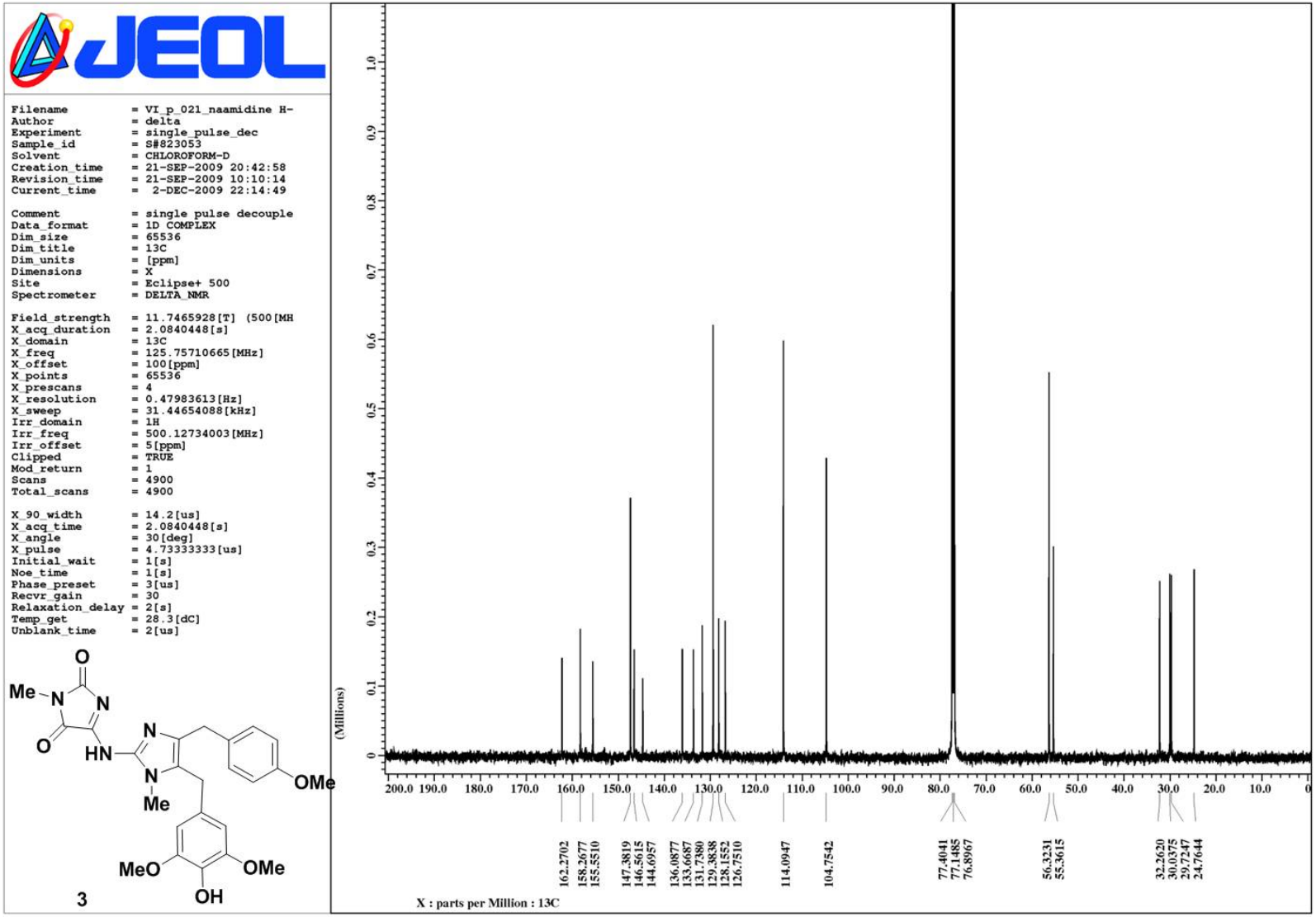

JEOL
Filename = VI_p_020_Naamine G-3.
Author = delta
Experiment = single_pulse.exp
Sample_id = S#780024
Solvent = METHANOL-D3
Creation_time = 17-SEP-2009 12:33:36
Revision_time = 1-DEC-2009 22:50:06
Current_time = 1-DEC-2009 22:50:33
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.1839872[s]
X_domain = 1H
X_freq = 500.12734003[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45787814[Hz]
X_sweep = 7.50187547[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1839872[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 20
Relaxation_delay = 4[s]
Temp_get = 25.8[dC]
Unblank_time = 2[us]
H2N
N
N
Me
OMe
MeO
OMe
OH
2
(Millions)
30.0
20.0
10.0
0
2
2.12
2.04
6.02
2.99
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.1753
7.1579
6.8713
6.8539
6.3431
4.8553
3.7549
3.6981
3.3154
3.3118
3.3090
3.3054
3.2358
X : parts per Million : 1H



```
Filename          = VI_p_020_naamine G-4.
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#804773
Solvent           = METHANOL-D3
Creation_time     = 18-SEP-2009 22:18:01
Revision_time     =  2-DEC-2009 22:08:02
Current_time      =  2-DEC-2009 22:08:20

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 65536
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 500
Spectrometer      = DELTA_NMR

Field_strength    = 11.7465928[T] (500[MH
X_acq_duration    = 2.0840448[s]
X_domain          = 13C
X_freq            = 125.75710665[MHz]
X_offset          = 100[ppm]
X_points          = 65536
X_prescans        = 4
X_resolution      = 0.47983613[Hz]
X_sweep           = 31.44654088[kHz]
Irr_domain        = 1H
Irr_freq          = 500.12734003[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 6400
Total_scans       = 6400

X_90_width        = 14.2[us]
X_acq_time        = 2.0840448[s]
X_angle           = 30[deg]
X_pulse           = 4.73333333[us]
Initial_wait      = 1[s]
Noe_time          = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 2[s]
Temp_get          = 28.1[dC]
Unblank_time      = 2[us]
```


2


Filename = VI_p_021_naamidine H-
Author = delta
Experiment = single_pulse.exp
Sample_id = S#821340
Solvent = CHLOROFORM-D
Creation_time = 21-SEP-2009 13:45:25
Revision_time = 20-SEP-2009 22:54:09
Current_time = 1-DEC-2009 22:42:35
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.1839872[s]
X_domain = 1H
X_freq = 500.12734003[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45787814[Hz]
X_sweep = 7.50187547[kHz]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 18.5[us]
X_acq_time = 2.1839872[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 16
Relaxation_delay = 4[s]
Temp_get = 26.1[dC]
Unblank_time = 2[us]
Me
O
N
N
O
HN
N
N
Me
OMe
MeO
OMe
OH
3
(Millions)
5.99
4.16
3.11
2.99
3.0
2.13
2.03
2
1.09
7.2613
7.1460
7.1286
6.9839
6.7935
6.7770
6.1435
3.8940
3.8849
3.7485
3.6926
3.4876
3.1589
X : parts per Million : 1H


JEOL
Filename = VI_p_021_naamidine H-
Author = delta
Experiment = single_pulse_dec
Sample_id = S#823053
Solvent = CHLOROFORM-D
Creation_time = 21-SEP-2009 20:42:58
Revision_time = 21-SEP-2009 10:10:14
Current_time = 2-DEC-2009 22:14:49
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7465928[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.75710665[MHz]
X_offset = 100[ppm]
X_points = 65536
X_prescans = 4
X_resolution = 0.47983613[Hz]
X_sweep = 31.44654088[kHz]
Irr_domain = 1H
Irr_freq = 500.12734003[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 4900
Total_scans = 4900
X_90_width = 14.2[us]
X_acq_time = 2.0840448[s]
X_angle = 30[deg]
X_pulse = 4.73333333[us]
Initial_wait = 1[s]
Noe_time = 1[s]
Phase_preset = 3[us]
Recvr_gain = 30
Relaxation_delay = 2[s]
Temp_get = 28.3[dC]
Unblank_time = 2[us]
Me
O
N
N
O
HN
N
N
Me
OMe
MeO
OMe
OH
3
(Millions)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
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
162.2702
158.3677
155.5510
147.3819
146.5615
144.6957
136.0877
133.6687
131.7380
129.3838
128.1552
126.7510
114.0947
104.7542
77.4041
77.1485
76.8967
56.3231
55.5615
32.2620
30.0375
29.7247
24.7644
X : parts per Million : 13C