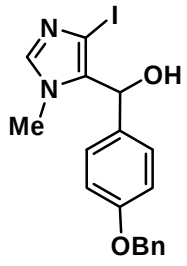
Supplementary Information for

Total Synthesis of (±)-Calcaridine A and (±)-epi-Calcaridine A

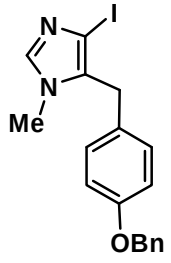
Panduka B. Koswatta, Rasapalli Sivappa, H.V. Rasika Dias and Carl J. Lovely*

Department of Chemistry and Biochemistry, The University of Texas at Arlington, Arlington, Texas 76019

5-(4-Benzyloxy)-phenyl-(4-iodo-1-methyl-1H-imidazol-5-yl)-methanol (10): EtMgBr (3.0 M solution in ether, 2.31 ml, 6.93 mmol) was added to a solution of 4,5-diiodo-1-methyl-1H-imidazole (2.20 g, 6.60 mmol) in dry CH_2Cl_2 (15 ml) at rt over ~5 min. The resulting mixture was stirred at rt for 20 min and 4-benzoyloxybenzaldehyde (1.54 g, 7.25 mmol) in dry CH_2Cl_2 (25 ml) was added and stirred at rt overnight. 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, which was decanted to give **10** (2.80 g, quant) as a white solid; m.p. 195-198 °C ; ^{1}H NMR (DMSO-d_6): δ = 7.58 (s, 1H), 7.43 (d, *J* = 7.3 Hz, 2H), 7.38 (t, *J* = 7.3 Hz, 2 H), 7.32 (t, *J* = 7.3 Hz, 1H), 7.17 (d, *J* = 8.7 Hz, 2H), 6.99 (d, *J* = 8.7 Hz, 2H), 6.22 (d, *J* = 4.1 Hz, 1H), 5.80 (d, *J* = 4.1Hz, 1H), 5.07 (s, 2H), 3.38 (s, 3H); ^{13}C NMR: δ = 157.9, 141.9, 137.6, 135.4, 134.6, 129.0, 128.4, 128.3, 127.0, 115.1, 85.7, 70.0, 66.5, 33.2; IR (KBr, cm^{-1}): 3189 (br), 3034, 2948, 2874, 1607, 1506, 1387, 1236,1166, 1006, 971, 744, 699; HR-ESIMS (*m/z*): Calcd. for $C_{18}H_{18}IN_2O_2$ $[M+H]^+$ 421.0408, found 421.0392; Calcd. for $C_{18}H_{17}IN_2O_2Na$ $[M+Na]^+$ 443.0227, found 443.0194.

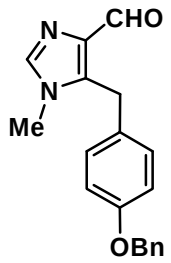


5-(4-Benzoyloxy)-benzyl-4-iodo-1-methyl-1H-imidazole (7): Et_3SiH (5.36 ml, 33.55 mmol) and TFA (2.58 ml, 33.55 mmol) were added to a solution of **10** (2.82 g, 6.71 mmol) in anhydrous $CHCl_3$ (50 ml) at rt, then the resulting mixture was heated at 55-60 °C for 24 h under nitrogen atmosphere. After cooling to rt, the reaction was quenched by the addition of sat. aq. $NaHCO_3$ solution. The resulting mixture was extracted with $CHCl_3$ several times and the combined extracts were dried (Na_2SO_4) and concentrated. The residue was purified by chromatography



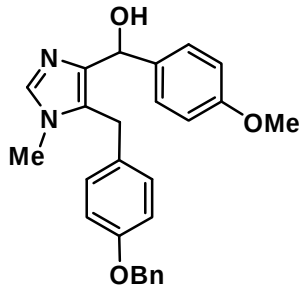
(hexane/EtOAc, 1:1) to give **7** as a thick colorless oil (1.61 g, 60%); ^{1}H NMR ($CDCl_3$): δ = 7.42-7.30 (m, 6H), 7.03 (d, *J*= 8.7 Hz, 2H), 6.89 (d, *J*= 8.7 Hz, 2 H), 5.02 (s, 2H), 3.91 (s, 2H), 3.41 (s, 3H); ^{13}C NMR: δ = 157.8, 139.4, 137.0, 133.4, 129.6, 129.1, 128.7, 128.1, 1127.5, 115.2, 84.8, 70.2, 32.6, 30.0; IR (neat, cm^{-1}): 3031, 2918, 1609, 1509, 1418, 1239, 1175, 1013, 816, 740, 697; HR-ESIMS (*m/z*): Calcd. for $C_{18}H_{18}IN_2O$ $[M+H]^+$ 405.0458, found 405.0470; Calcd. for $C_{18}H_{17}IN_2ONa$ $[M+Na]^+$ 427.0278, found 427.0247.

5-(4-Benzoyloxy)-benzyl-1-methyl-1H-imidazole-4-carboxaldehyde (11): EtMgBr (3.0 M in ether, 1.90 ml, 5.69 mmol) was added into a solution of **7** (2.19 g, 5.42 mmol) in dry THF (30 ml) at rt, and the resulting mixture was stirred at rt for 20 min. *N*-Methylformanilide (0.74 ml, 5.96 mmol) was added and the resulting mixture was stirred at rt overnight. Half saturated NH_4Cl 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. This residue was purified through a short plug of silica gel (hexane/EtOAc, 3:2) to give **11** as an off-white solid (1.09 g, 66%); m.p. 148-150 °C; ^{1}H NMR ($CDCl_3$): δ = 10.01 (s, 1H), 7.51 (s, 1H), 7.41-7.30 (m, 5H), 7.05 (d, *J*= 8.7 Hz, 2H), 6.88 (d, *J*= 8.7 Hz, 2H), 5.02 (s, 2H), 4.34 (s, 2H), 3.47 (s, 3H); ^{13}C NMR: δ = 187.5, 157.9, 138.7, 138.0, 137.8, 136.9, 129.4, 128.7, 128.6, 128.1, 127.5, 115.3, 70.1, 31.7, 28.5; IR (KBr, cm^{-1}): 3107, 3032, 2859, 1674, 1510, 1244, 1175, 799, 780, 740, 698; HR-ESIMS (*m/z*): Calcd. for $C_{19}H_{19}N_2O_2$ $[M+H]^+$ 307.1441, found 307.1462; Calcd. for $C_{19}H_{18}N_2O_2Na$ $[M+Na]^+$ 329.1260, found 329.1258.

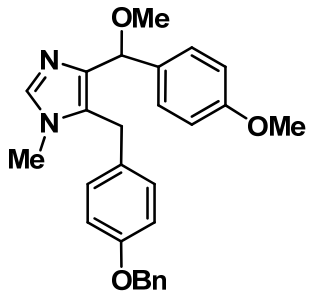


{5-[4-(Benzoyloxy)-benzyl]-1-methyl-1H-imidazol-4-yl}-(4-methoxyphenyl)-methanol (12): A few drops of *p*-bromoanisole (from 2.21 ml, 17.6 mmol) was added dropwise to a two necked round-bottom flask containing freshly-crushed oven-dried, magnesium turnings (0.42 g, 17.6

mmol) and a small crystal of iodine in THF (20 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 over 10 min while heating at the same temperature. After the addition was complete, the mixture was heated to reflux for 1 h and then cooled to rt, then, a solution of **3** (1.08 g, 3.52 mmol) in THF (10 ml) was added and the resulting mixture was stirred at reflux overnight. After cooling to 0 °C, sat, NH_4Cl (20 ml) was added and the organic layer was extracted with EtOAc (x3), 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 **12** as a pale yellow solid (1.47 g, 100%); m.p. 124-127 °C; ^{1}H NMR ($CDCl_3$): δ = 7.42-7.29 (m, 8H), 6.89-6.76 (m, 6H), 5.78 (s, 1H), 5.00 (s, 2H), 4.38 (br, 1H), 3.78 (s, 2H), 3.73 (s, 3H), 3.28 (s, 3H); ^{13}C NMR δ = 158.8, 157.5, 140.9, 137.1, 137.0, 136.2, 130.2, 129.1, 128.7, 128.1, 127.6, 125.9, 115.1, 113.7, 70.1, 69.5, 55.3, 31.8, 28.2; IR (KBr, cm^{-1}): 3198 (br), 3031, 2932, 2835, 1611, 1584, 1509, 1454, 1302, 1244, 1175, 1035, 801, 752, 698; HR-ESIMS (*m/z*): Calcd. for $C_{26}H_{27}N_2O_3$ $[M+H]^+$ 415.2016, found 415.2016; Calcd. for $C_{26}H_{26}N_2O_3Na$ $[M+Na]^+$ 437.1856, found 437.1817.

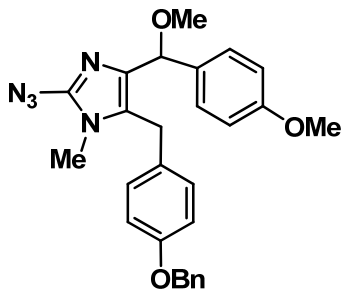


5-[4-(Benzoyloxy)-benzyl]-4-[methoxy-(4-methoxy-phenyl)-methyl]-1-methyl-1H-imidazole (13): TFA (0.53 ml, 6.81 mmol) was added to a solution of **4** (1.42 g, 3.41 mmol) in anhyd MeOH (20 ml) at rt and the mixture was then heated at 55 °C for overnight. Sat. aq. $NaHCO_3$ (20 ml) was added to the above reaction mixture and the aqueous layer was extracted with EtOAc (x3) and the organic layer was washed with aq. $NaHCO_3$ (x2), washed with water, brine and dried (Na_2SO_4), concentrated to give **13** (1.58 g,



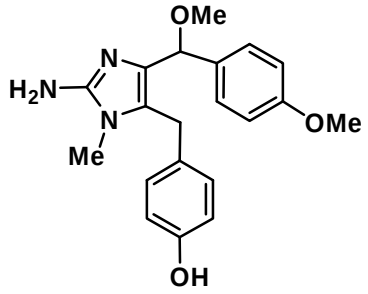
quant) as a pale yellow oil; ^{1}H NMR ($CDCl_3$): δ = 7.51 (s, 1H), 7.42-7.29 (m, 7H), 6.92-6.76 (m, 6H), 5.35 (s, 1 H), 5.02 (s, 2H), 3.95 (s, 2H), 3.77 (s, 3H), 3.35 (s, 3H), 3.33 (s, 3H); ^{13}C NMR: δ = 158.9, 157.5, 139.5, 137.2, 137.1, 133.7, 130.5, 129.2, 128.7, 128.2, 128.1,1127.6, 127.1, 115.0, 113.7, 79.5, 70.1, 56.8, 55.3, 31.7, 28.3; IR (neat, cm^{-1}) = 3032, 2971, 2916, 1610, 1509, 1244, 1174, 1011, 804, 742; HR-ESIMS (*m/z*): Calcd. for $C_{27}H_{29}N_2O_3$ $[M+H]^+$ 429.2117, found 429.2176; Calcd. for $C_{27}H_{28}N_2O_3Na$ $[M+Na]^+$ 451.1992, found 451.1955.

2-Azido-5-[4-(benzoyloxy)-benzyl]-4-[methoxy-(4-methoxyphenyl)-methyl]-1-methyl-1H-imidazole (14): *n*-Butyl lithium (1.6 M solution in hexanes, 0.72 ml, 1.14 mmol) was added dropwise to a stirred solution of **5** (445 mg, 1.04 mmol) in dry THF (8 ml) at -78 °C. The reaction was stirred for 30 min at the same temperature. The cooling bath was removed for 10 min, then the reaction mixture was re-cooled to -78 °C, and then TsN_3 (246 mg, 1.25 mmol) in THF (1 ml) was added dropwise. After stirring for an additional 10 min at -78 °C, the reaction mixture was allowed to come to rt and stirred for 40 min. The reaction was quenched by the careful addition of sat. NH_4Cl (3 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. The crude product was purified through a short column of silica gel (hexane/EtOAc, 4:1) to give unreacted starting material and **6** (283 mg, 58%) as a reddish brown oil; ^{1}H NMR ($CDCl_3$): δ = 7.49-7.34 (m, 7H), 6.98-6.88 (m, 6H), 5.23 (s, 1 H), 5.02 (s, 2H), 3.85 (s, 2H), 3.78 (s, 3H), 3.35 (s, 3H), 3.04 (s, 3H); ^{13}C NMR δ = 159.1, 157.6, 139.8, 137.1, 136.9, 133.4, 130.3, 129.2, 128.7, 128.5, 128.1, 127.6, 126.2, 115.1, 113.8, 79.2, 70.1, 56.9, 55.3, 29.5, 28.7; IR (neat, cm^{-1}): 3032, 2933, 2835, 2137, 1610, 1509, 1244, 1173, 1088, 1033, 832, 744, 697; HR-ESIMS (*m/z*): Calcd. for $C_{26}H_{24}N_5O_2$

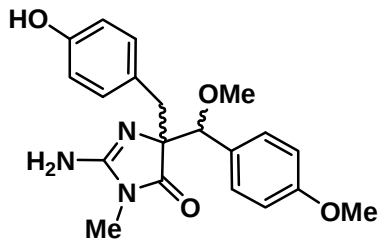


[M+H-MeOH]$^+$ 438.1925, found 438.1898 ; Calcd. for $C_{27}H_{27}N_5O_3Na$ [M+Na]$^+$ 492.2006, found 492.1977.

2-Amino-5-[4-(hydroxy)-benzyl]-4-[methoxy-(4-methoxyphenyl)-methyl]-1-methyl-1H-imidazole (6): Azide **6** (258 mg, 0.55 mmol) was dissolved in EtOH (3 ml) and stirred under a hydrogen atmosphere (55 psi) in the presence of 20% $Pd(OH)_2$ on charcoal (77 mg) at rt for overnight. The catalyst was filtered through a pad of celite and the filtrate was concentrated to give amine **6** (196 mg, quant) as a pale yellow solid; m.p 91-95 °C; ^{1}H NMR (500 MHz, CD_3OD): δ = 7.28 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.2 Hz 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 6.67 (d, *J* = 8.2 Hz, 2H), 5.25 (s, 1H), 3.83 (s, 2H), 3.74 (s, 3H), 3.31 (s, 3H), 3.11 (s, 3H); ^{13}C NMR δ = 160.0, 156.3, 147.0, 130.8, 128.9, 127.8, 126.9, 124.3, 123.3, 115.4, 113.8, 75.1, 55.7, 54.5, 28.7, 27.0; IR (KBr, cm^{-1}) = 3548, 3475, 3417, 2996, 2934, 1614, 1512, 1247, 1174, 1114, 823, 618.; HR-ESIMS (*m/z*): Calcd. for $C_{20}H_{24}N_3O_3$ [M+H]$^+$ 354.1827, found 354.1812; Calcd. for $C_{20}H_{23}N_3O_3Na$ [M+Na]$^+$ 376.1645, found 376.1632.



2-Amino-5-(4-hydroxy-benzyl)-5-[methoxy-(4-methoxy-phenyl)-methyl]-3-methyl-3,5-dihydro-imidazol-4-one (Calcaridine A): Amine **6** (175 mg, 0.51 mmol) and 2-benzenesulfonyl-3-(4-nitrophenyl)oxaziridine (311 mg, 1.02 mmol) were dissolved in methanol (5 ml) at rt. Then, the mixture was heated at 40 °C for 4 h. After checking TLC the reaction was stopped, concentrated and purified by flash column chromatography using 10% methanol in EtOAc to



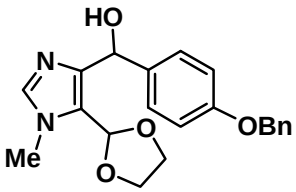
give a pale yellow solid (100 mg, 54%), of a 1:2 mixture of diastereomers calcaridine A and *epi*-calcaridine A.

1-Methyl-4-iodoimidazole-5-carboxaldehyde (16): A solution of EtMgBr (3.0 M in ether, 2.62 ml, 7.86 mmol) was added into a solution of 4,5-diiodo-1-methyl-1H-imidazole (**8**) (2.50 g, 7.50 mmol) in dry CH_2Cl_2 (20 ml) at rt over 10 min. The resulting mixture was stirred at rt under nitrogen atmosphere with monitoring by TLC to ensure all starting material reacted with the Grignard reagent. Then, *N*-methylformanilide (1.01 ml, 8.23 mmol) was added dropwise to above mixture and stirred at rt for a further 16 h. Half saturated NH_4Cl (10 ml) was added to it and the resulting suspension was extracted with dichloromethane. The organic layer was dried (Na_2SO_4), concentrated and the resulting residue was purified by flash chromatography (EtOAc/hexanes, 1:4) to give a white solid,**1** (1.09 g, 61%); m.p. 69-72 °C; ^{1}H NMR ($CDCl_3$): δ = 9.62 (d, *J* = 0.5 Hz, 1H), 7.55 (s, 1H), 3.91 (s, 3H); ^{13}C NMR: δ = 181.3, 145.0, 130.0, 100.3, 34.5; IR (KBr, cm^{-1}): = 2811, 1666, 1504, 1338, 1243, 964, 782, 707; HR-ESIMS (*m/z*): Calcd. for $C_5H_6IN_2O$ $[M+H]^+$ 236.9519, found 236.9527; Calcd. for $C_5H_5IN_2ONa$ $[M+Na]^+$ 258.9339, found 258.9362.

5-[1,3]Dioxolan-2-yl-4-iodo-1-methyl-1H-imidazole (17): *p*-Toluenesulfonic acid monohydrate (140 mg, 0.74 mmol) and ethylene glycol (95%, 4.10 ml, 73.7 mmol) were added to a solution of **1** (3.48 g, 14.7 mmol) in toluene (75 ml). The reaction mixture was heated to reflux for 22 h with a Dean-Stark condenser fitted. The mixture was cooled to rt, and then the reaction mixture was washed with sat. $NaHCO_3$ (3x25 ml) and water. The resulting toluene solution was dried (Na_2SO_4), concentrated and purified by chromatography (hexane/EtOAc, 65:35) to give **17** (4.02 g, 97%) as an off-white solid; m.p. 115 - 118 °C; ^{1}H NMR ($CDCl_3$): δ = 7.39 (s, 1H), 5.79 (s, 1H), 4.15 (m, 2H), 4.04 (m, 2H), 3.69 (s,

3H); ^{13}C NMR: δ = 141.9, 127.0, 98.8, 87.8, 65.3, 33.2; IR (KBr, cm^{-1}): = 2951, 2887, 1578, 1494, 1473, 1370, 1245, 1217, 1085, 952, 815; HR-ESIMS (*m/z*): Calcd. for $C_7H_{10}IN_2O_2$ $[M+H]^+$ 280.9782, found 280.9791; Calcd. for $C_7H_9IN_2O_2Na$ $[M+Na]^+$ 302.9610, found 302.9612.

(4-Benzyloxyphenyl)-(5-[1,3]dioxolan-2-yl-1-methyl-1H-imidazol-4-yl)-methanol (18): A



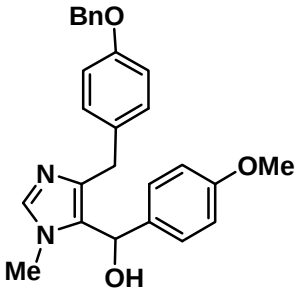
solution of EtMgBr (3.0 M in ether, 2.67 ml, 8.02 mmol) was added to a solution of **2** (2.14 g, 7.64 mmol) in dry THF (20 ml) at rt over 10 min. The resulting mixture was stirred at rt until all the starting material reacted (TLC analysis, ca. 30 min) and then 4-benzyloxybenzaldehyde (1.78 g, 8.40 mmol) in dry THF (10 ml) was added at rt followed by stirring for 38 h. Saturated aq. NH_4Cl (10 ml) was added to quench the reaction and the organic layer was extracted with EtOAc and washed once with brine. The EtOAc solution was dried (anhyd. Na_2SO_4) and concentrated to give the crude product which was purified by column chromatography (EtOAc→EtOH/EtOAc, 1:9) to give **18** as a white solid (2.46 g, 87%); m.p. 160-162 °C; ^{1}H NMR ($CDCl_3$): δ = 7.40-7.27 (m, 8H), 6.92 (d, *J* = 8.3 Hz, 2H), 5.90 (s, 1H), 5.84 (s, 1H), 5.03 (s, 2H), 4.00 (m, 2H), 3.92 (m, 2H), 3.66 (s, 3H); ^{13}C NMR: δ = 158.1, 145.0, 138.8, 137.2, 136.2, 128.6, 128.0, 127.9, 127.5, 121.8, 114.7, 97.3, 70.1, 69.5, 65.1, 33.0; IR (KBr, cm^{-1}): = 3176 (br), 3120, 2918, 1606, 1511, 1419, 1226, 1076, 1035, 951, 843, 698; HR-ESIMS (*m/z*): Calcd. for$C_{21}H_{23}N_2O_4$ $[M+H]^+$ 367.1652, found 367.1662; Calcd. for $C_{21}H_{22}N_2O_4Na$ $[M+Na]^+$ 389.1472, found 389.1469.

4-[(4-benzyloxyphenyl)-hydroxymethyl)-1-methyl-1H-imidazole-5-carboxaldehyde: 10% HCl (10 ml) was added to a solution of acetal **3** (2.23 g, 6.09 mmol) in THF (100 ml) and the resulting cloudy reaction was heated at 55 °C (The reaction became clear while all solid dissolving after 10 min) while the reaction progress was monitored by taking 0.5 ml aliquots and neutralizing with sat'd $NaHCO_3$. The aqueous layer was extracted with EtOAc, and the organic layer was dried over anhyd. Na_2SO_4 and concentrated to give the crude product which was evaluated by H^1 NMR. After all starting material was consumed (2 h), the reaction was worked-up following the above procedure giving the pure aldehyde (1.95 g, quant), as a cream colored solid, was isolated; m.p. 135 °C; ^{1}H NMR ($CDCl_3$): δ = 9.90 (s, 1H), 7.46 (s, 1H), 7.41-7.31 (m, 7H), 6.94 (d, *J* = 8.7 Hz, 2 H), 6.01 (s, 1H), 5.03 (s, 2H), 3.87 (s, 3H); ^{13}C NMR: δ = 180.4, 158.7, 156.8, 142.1, 137.0, 135.1, 128.7, 128.1, 128.0, 127.5, 126.6, 115.1, 70.9, 70.1, 34.5; IR (KBr, cm^{-1}) = 3327 (br), 3088, 3009, 2862, 1655, 1513, 1352, 1297, 1245, 1045, 1014, 807, 786, 741, 715; HR-ESIMS (*m/z*): Calcd. for $C_{19}H_{19}N_2O_3$ $[M+H]^+$ 323.1390, found 323.1382; Calcd. for $C_{19}H_{18}N_2O_3Na$ $[M+Na]^+$ 345.1210, found 345.1198.

4-(4-Benzyloxy)benzyl-1-methyl-1H-imidazole-5-carbaldehyde (19): Et_3SiH (3.86 ml, 24.20 mmol) and TFA (2.80 ml, 36.29 mmol) were added to a solution of the aldehyde **4** (1.95 g, 6.05 mmol) in anhydrous $CHCl_3$ (100 ml) under nitrogen at r.t. Then the resulting mixture was stirred for 24 h while monitoring the reaction progress by TLC. Then, the reaction was quenched with saturated aqueous $NaHCO_3$ solution. The organic layer was extracted with $CHCl_3$ (3x50 ml). Combined organic extracts were dried over anhyd. Na_2SO_4 and concentrated to give a yellowish white solid, which was purified over silica gel with (EtOAc/hexanes, 3:1) to give **19** as a pale

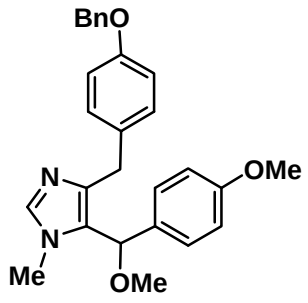
yellow solid, (1.21 g, 65%); m.p. 85 - 86 °C; ^{1}H NMR ($CDCl_3$): δ = 9.85 (s, 1H), 7.47 (s, 1H), 7.40-7.30 (m, 5H), 7.18 (d, *J* = 8.7 Hz, 2 H), 6.90 (d, *J* = 8.7 Hz, 2 H), 5.01 (s, 2H), 4.12 (s, 2H), 3.85 (s, 3H); ^{13}C NMR: δ = 179.1, 157.6, 155.8, 142.9, 137.1, 131.3, 129.7, 128.6, 128.0, 127.5, 127.0, 115.2, 70.1, 34.4, 33.2; IR (KBr, cm^{-1}) = 3121, 3058, 3028, 2915, 2826, 2746, 1763, 1665, 1520, 1332, 1247, 1171, 1009, 845, 744, 699, 633; HR-ESIMS (*m/z*): Calcd. for $C_{19}H_{19}N_2O_2$ $[M+H]^+$ 307.1441, found 307.1444; Calcd. for $C_{19}H_{18}N_2O_2Na$ $[M+Na]^+$ 329.1266, found 329.1207.

4-(4-Benzyloxybenzyl)-5-[hydroxyl-(4-methoxyphenyl)-methyl]-1-methyl-1H-imidazole (20): A few drops of *p*-bromoanisole (from 1.98 ml, 15.8 mmol) was added dropwise to a two neck round-bottom flask containing freshly-crushed, oven-dried magnesium turnings (0.38 g, 15.8 mmol) and a small crystal of iodine in THF (25 ml). This mixture was then heated at 45 °C under nitrogen until the iodine color faded. The rest of the *p*-bromoanisole was added dropwise over 10 min while heating at the same temperature. After the addition was completed, the mixture was heated at reflux for 1 h and cooled to rt. A solution of **5** (1.21 g, 3.95 mmol) in THF (10 ml) was added. The resulting mixture was stirred at reflux for overnight and cooled to 0 ° C; sat. NH_4Cl (20 ml) was added and the organic layer was extracted with EtOAc (x3), washed once with brine, and dried over anhyd. Na_2SO_4, concentrated to give thick brown oil, which was purified by a short plug of silica gel with 100% EtOAc to give a white solid, **20** (1.64 g, 84%); m.p. 148-149 °C; ^{1}H NMR ($CDCl_3$): δ = 7.41-7.25 (m, 6H), 7.16 (d, *J* = 8.5 Hz, 2 H), 7.10 (d, *J* = 8.5 Hz, 2 H), 6.85 (d, *J* = 8.5 Hz, 2 H), 6.81 (d, *J* = 8.8 Hz, 2 H), 6.05 (s, 1H), 4.99 (s, 2H), 3.88 (s, 2 H), 3.78 (s, 3H), 3.34 (s, 3H); ^{13}C NMR: δ = 159.0, 157.3, 138.4, 137.2, 132.6_1, 132.5_5, 129.7, 129.0, 128.6, 128.0,



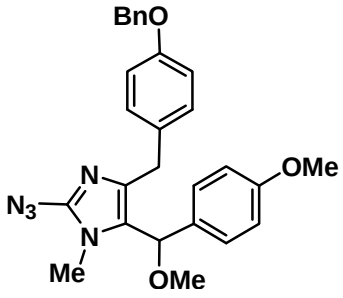
127.5, 127.0, 115.1, 113.9, 70.1, 65.5, 55.4, 33.4, 32.1; IR (KBr, cm^{-1}) = 3200 (br), 3115, 2998, 2908, 2834, 1611, 1510, 1459, 1238, 1173, 1032, 804, 697; HR-ESIMS (*m/z*): Calcd. for $C_{26}H_{27}N_2O_3$ $[M+H]^+$ 415.2016, found 415.2034; Calcd. for $C_{26}H_{26}N_2O_3Na$ $[M+Na]^+$ 437.1836, found 437.1819.

4-(4-Benzyloxybenzyl)-5-[methoxy-(4-methoxyphenyl)-methyl]-1-methyl-1H-imidazole (21)



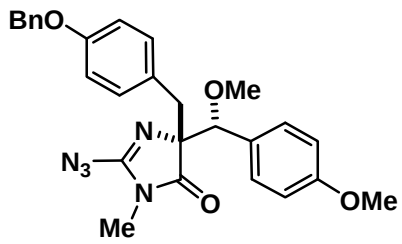
NaH (60%, 162 mg, 4.05 mmol) was added portionwise to a stirred mixture of alcohol **20** (1.12 g, 2.70 mmol) in anhydrous THF (25 ml) at 0 °C. After completion of the addition the resulting mixture was stirred for 10 min at the same temperature. The reaction was warmed to rt and stirred for 1.5 h and then re-cooled (0 °C) followed by the dropwise addition of MeI (0.20 ml). After 10 min the reaction was allowed to come to rt and stirred for 36 h. Water (20 ml) was added to the reaction mixture and the aqueous layer was extracted with EtOAc (3x30 ml). The organic solution was dried (Na_2SO_4), concentrated and the residue was purified through short plug of silica gel with (EtOAc/hexanes, 3:1) to give the **21** as a pale yellow oil (0.97 g, 84%); ^1H NMR ($CDCl_3$): δ = 7.41-7.25 (m, 6H), 7.20 (d, *J* = 8.3 Hz, 2 H), 7.09 (d, *J* = 8.7 Hz, 2 H), 6.88 (d, *J* = 8.3 Hz, 2 H), 6.81 (d, *J* = 8.7 Hz, 2 H), 5.48 (s, 1H), 5.01 (s, 2H), 3.94 (s, 2 H), 3.76 (s, 3H), 3.27 (s, 3H), 3.23 (s, 3H); ^{13}C NMR: δ = 158.9, 157.2, 142.1, 138.4, 137.3, 133.3, 132.0, 129.7, 128.6, 127.9, 127.5, 127.3, 125.2, 114.9, 113.8, 75.0, 70.1, 56.6, 55.3, 33.0, 32.7; IR (neat, cm^{-1}): 3032, 2931 1609, 1509, 1246, 1174, 1086, 1031, 805, 741, 698; HR-ESIMS (*m/z*): Calcd. for $C_{27}H_{29}N_2O_3$ $[M+H]^+$ 429.2173, found 429.2181; Calcd. for $C_{27}H_{28}N_2O_3Na$ $[M+Na]^+$ 451.1992, found 451.1951.

2-Azido-4-(4-benzyloxybenzyl)-5-[methoxy-(4-methoxyphenyl)-methyl]-1-methyl-1H-imidazole (22): *n*-Butyl lithium (1.33 M solution in hexane, 1.87 ml, 2.49 mmol) was added dropwise to a stirred solution of **7** (888 mg, 2.07 mmol) in dry THF (10 ml) at -78 °C. The reaction mixture was stirred for 45 min at the same temperature. Then, the ice/acetone bath was removed for 5 min followed by re-cooling to -78 °C and dropwise addition of TsN_3 (491 mg, 2.49 mmol). After 1 h stirring at -78 °C, the reaction was quenched by the careful addition of sat. NH_4Cl solution (3 ml). The aqueous layer was extracted with EtOAc (3x25 ml), and then the combined organics 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, 4:1) to give **8** (972 mg, 76%) as a thick, pale yellow oil; ^{1}H NMR ($CDCl_3$): δ = 7.42-7.31 (m, 5H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.07 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 6.79 (d, *J*= 8.7 Hz, 2 H), 5.37 (s, 1H), 5.03 (s, 2H), 3.90 (s, 2 H), 3.78 (s, 3H), 3.21 (s, 3H), 3.02 (s, 3H); ^{13}C NMR: δ = 158.9, 157.3, 140.8, 139.6, 137.3, 133.0, 131.8, 129.7, 128.6, 128.0, 127.5, 127.3, 124.3, 114.9, 113.8, 75.0, 70.1, 56.5, 55.3, 32.8, 30.4; IR (neat cm^{-1}): 2932, 2835, 2136, 1610, 1509, 1248, 1172, 1085, 1033, 833, 738, 697; HR-ESIMS (*m/z*): Calcd. for $C_{27}H_{28}N_5O_3$ $[M+H]^+$ 470.2187, found 470.2191; Calcd. for $C_{27}H_{27}N_5O_3Na$ $[M+Na]^+$ 492.2006, found 492.1969.

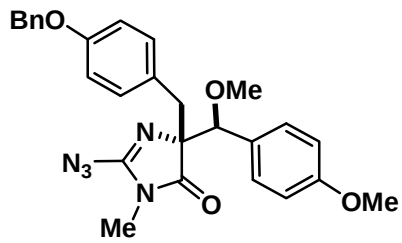


(*4R*, 8S) and (*4R*, 8R**)-2-Azido-4-(4-benzyloxybenzyl)-4-[methoxy-(4-methoxyphenyl)-methyl]-1-methyl-1,5-dihydroimidazol-5-one (24) and (*epi*-24):** 3-(4-Nitrophenyl)-2-(phenylsulfonyl)oxaziridine (254 mg, 0.83 mmol) was added to a stirred solution of azide **22** (255 mg, 0.54 mmol) in $CHCl_3$ (3 ml) at rt and stirred overnight. On completion of the reaction, the solvent was removed and the yellow residue was purified by gravity column chromatography

(CH_2Cl_2/toluene, 1:1) to give *epi*-**24** (123 mg, 47%) as a pale yellow semi-solid; ^{1}H NMR

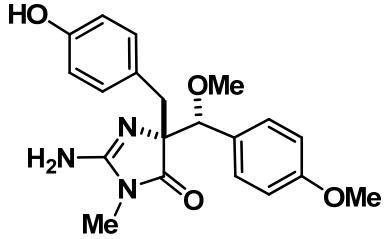


($CDCl_3$) : δ = 7.36-7.26 (m, 5H), 6.95 (d, *J* = 8.7 Hz, 2H), 6.78 (d, *J* = 8.7 Hz, 2H), 6.72 (d, *J* = 8.7 Hz, 2H), 6.68 (d, *J* = 8.7 Hz, 2H), 4.90 (s, 2H), 4.78 (s, 1H), 3.92 (d, *J* = 14.2 Hz, 1 H), 3.72 (s, 3H), 3.35 (d, *J* = 14.2 Hz, 1 H), 3.33 (s, 3H), 2.76 (s, 3H); ^{13}C NMR: δ = 173.7, 160.2, 158.4, 158.3, 136.7, 130.7, 128.9, 128.6, 128.1, 127.6, 125.4, 124.5, 115.0, 114.0, 84.1, 79.0, 70.0, 57.7, 55.3, 38.3, 27.1 ; IR (neat, cm^{-1})= 2931, 1764, 1599, 1512, 1455, 1250, 1177, 1098, 1029, 834, 797, 738, 698; Calcd. for $C_{27}H_{28}N_5O_4$ $[M+H]^+$ 486.2136, found 486.2141.



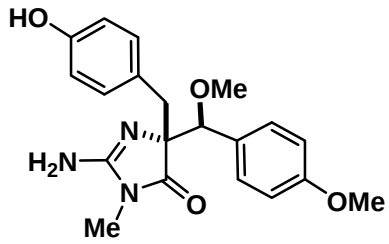
From the above reaction **24** (121 mg, 46%) as a pale yellow solid; m.p. 54-56 °C; ^{1}H NMR ($CDCl_3$): δ = 7.35-7.26 (m, 7H), 6.99 (d, *J* = 8.7 Hz, 2H), 6.65 (m, 4 H), 4.90 (s, 2H), 4.77 (s, 1H), 3.85 (s, 3H), 3.25 (d, *J* = 14.2 Hz, 1 H), 3.13 (s, 3H), 3.05 (s, 3H), 2.94 (d, *J* = 14.2 Hz, 1 H); ^{13}C NMR δ = 175.4, 160.7, 159.1, 158.3, 136.7, 130.5, 130.1, 128.6, 128.1, 127.6, 125.3, 123.6, 114.9, 114.3, 84.2, 79.0, 69.9, 57.1, 55.4, 38.4, 27.4 ; IR (neat, cm^{-1}) = 2931, 1764, 1599, 1512, 1455, 1250, 1177, 1098, 1029,834, 797, 738, 698; Calcd. for $C_{27}H_{28}N_5O_4$ $[M+H]^+$ 486.2136, found 486.2138.

(*4R*, 8R)-epi-Calcaridine *epi*-(1):** Azide *epi*-**24** (94 mg, 0.20 mmol) was dissolved in EtOH (3 ml) and stirred under a hydrogen atmosphere (55 psi) in the presence of 20% $Pd(OH)_2$ on charcoal (40 mg) at rt overnight. The catalyst was filtered through a pad of Celite and the filtrate was concentrated to give *epi*-calcaridine A, *epi*-**1** (73 mg, quant) as an off-white solid; m.p. 218-220 °C ; ^{1}H NMR (CD_3OD): δ = 7.20 (d, *J* = 8.7 Hz,



2H), 6.94 (d, *J* = 8.7 Hz, 2H), 6.89 (d, *J* = 8.7 Hz, 2H), 6.66 (d, *J* = 8.7 Hz, 2H), 4.59 (s, 1 H), 3.77 (s, 3H), 3.43 (d, *J* = 14.2 Hz, 1 H), 3.31 (s, 3H), 3.18 (d, *J* = 14.2 Hz, 1 H), 2.51 (s, 3H); ^{13}C NMR: δ = 172.2, 160.5, 157.8, 156.8, 130.9, 129.0, 126.2, 124.40 114.9, 113.5, 84.1, 73.5, 56.3, 54.4, 38.3, 24.1; ; IR (KBr, cm^{-1}) = 3311 (br), 3001, 2830, 1770, 1693, 1613, 1560, 1513, 1440, 1309, 1256, 1089, 1032, 832, 793, 718; HR-ESIMS (*m/z*): Calcd. for $C_{20}H_{24}N_3O_4$ $[M+H]^+$ 370.1761, found 370.1761; Calcd. for $C_{20}H_{23}N_3O_4Na$ $[M+Na]^+$ 392.1586, found 392.1512.

(*4R*, 8S)-Calcaridine A (1):** Following the procedure above, azide **24** (102 mg, 0.21 mmol) and 20% $Pd(OH)_2$ on charcoal (40 mg) in EtOH (3 ml) gave calcaridine A, (**1**) (78 mg, quant) as a pale yellow solid; m.p. 163-165 °C; ^{1}H NMR (CD_3OD): δ = 7.37 (d, *J* = 8.3 Hz, 2H), 7.03 (d, *J* = 8.3 Hz, 2H), 6.86 (d, *J* = 8.3 Hz, 2H), 6.64 (d, *J* = 8.3 Hz, 2H), 4.58 (s, 1H), 3.82 (s, 3H), 3.16 (s, 3H), 3.16 (d, *J* = 14.2 Hz, 1H), 2.83 (s, 3H), 2.50 (d, *J* = 14.2 Hz, 1H); ^{13}C NMR δ = 173.3, 160.7, 158.7, 156.9, 130.8, 129.4, 126.1, 123.0, 114.9, 114.1, 84.2, 73.1, 56.1, 54.6, 37.9, 24.6; IR (KBr, cm^{-1}) = 3265 (br), 2833, 1781, 1692, 1612, 1560, 1513, 1449, 1346, 1250, 1093, 1023, 836, 799; HR-ESIMS (*m/z*): Calcd. for $C_{20}H_{24}N_3O_4$ $[M+H]^+$ 370.1761, found 370.1761.



X-ray crystallography. A suitable crystal of compound *epi*-**1** covered with a layer of hydrocarbon oil was selected and mounted with paratone-N oil in a cryo-loop, and immediately placed in the low-temperature nitrogen stream. The X-ray intensity data were measured at 100(2) K on a Bruker SMART APEX CCD area detector system equipped with a Oxford Cryosystems 700 Series cooler, a graphite monochromator, and a Mo Kα fine-focus sealed tube (λ = 0.710 73 Å). The data frames were integrated with the Bruker SAINT-Plus software package. Data were corrected for absorption effects using the multi-scan technique (SADABS). Structures were solved and refined using Bruker SHELXTL (Version 6.14) software package. Further details are in the cif file (deposited at the Cambridge Crystallographic Data Centre, CCDC 696635).

Table S1. Crystal data and structure refinement for *epi*-**1•MeOH**.

Identification code	dias530s	
Empirical formula	C21 H27 N3 O5	
Formula weight	401.46	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 12.3916(6) Å	α= 90°.
	b = 8.4464(4) Å	β= 101.228(1)°.
	c = 19.6714(10) Å	γ = 90°.
Volume	2019.49(17) $Å^3$	
Z	4	
Density (calculated)	1.320 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	

F(000)	856
Crystal size	0.36 x 0.15 x 0.08 mm^3
Theta range for data collection	1.80 to 26.00°.
Index ranges	-15<=h<=15, -10<=k<=10, -24<=l<=24
Reflections collected	16278
Independent reflections	3969 [R(int) = 0.0248]
Completeness to theta = 26.00°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9929 and 0.9665
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3969 / 0 / 274
Goodness-of-fit on F^2	1.043
Final R indices [I>2sigma(I)]	R1 = 0.0477, wR2 = 0.1291
R indices (all data)	R1 = 0.0544, wR2 = 0.1372
Largest diff. peak and hole	0.777 and -0.600 e.$Å^{-3}$

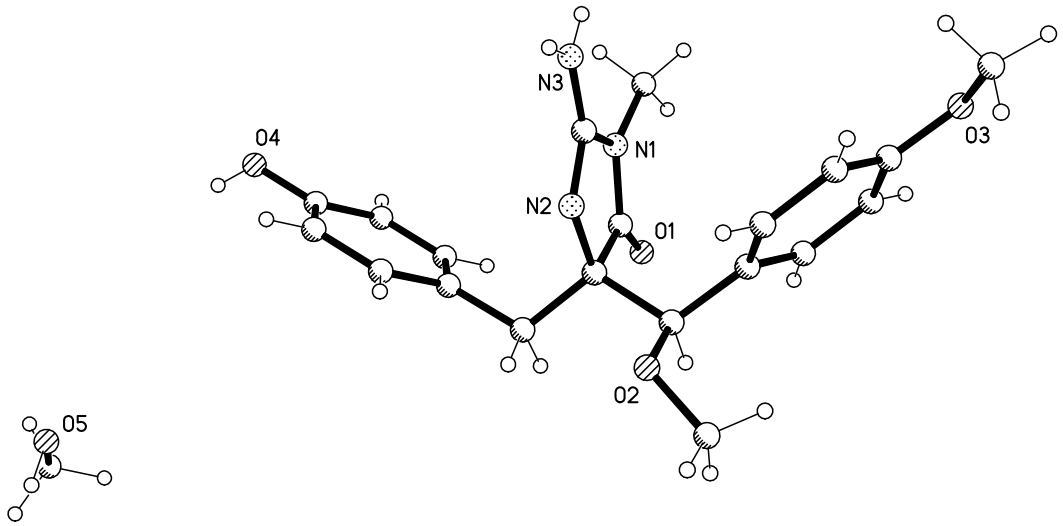


Figure S1. Crystal structure of *epi*-**1**•MeOH (*epi*-**1** crystallizes with a molecule of MeOH)

Table S2. Bond lengths [Å] and angles [°] for *epi*-**1•MeOH**.

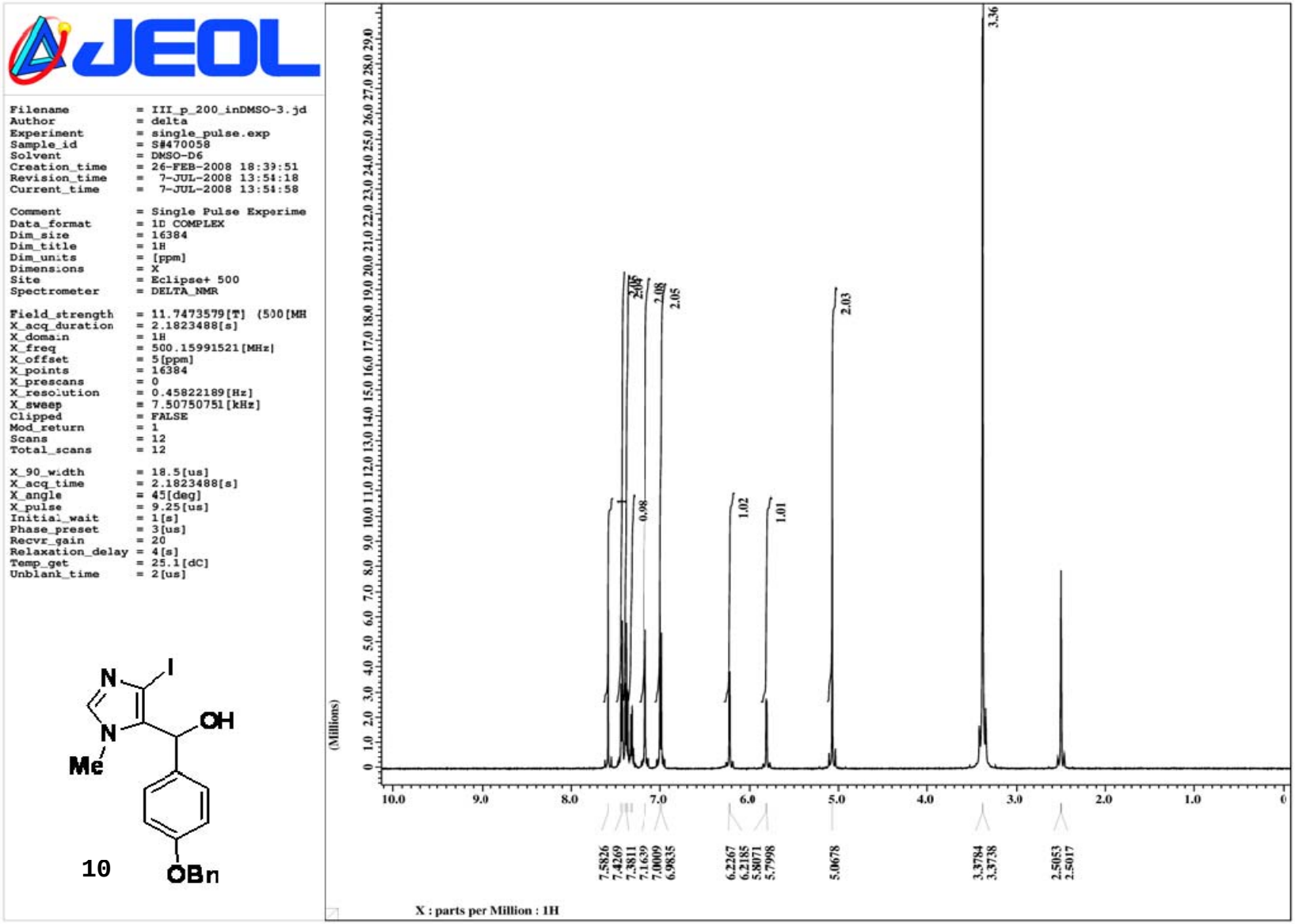
N(1)-C(2)	1.368(2)	C(7)-C(8)	1.387(2)
N(1)-C(1)	1.398(2)	C(7)-H(7)	0.9500
N(1)-C(4)	1.453(2)	C(8)-C(9)	1.392(2)
N(2)-C(1)	1.348(2)	C(8)-H(8)	0.9500
N(2)-C(3)	1.460(2)	C(9)-C(10)	1.390(2)
N(3)-C(1)	1.280(2)	C(10)-C(11)	1.384(2)
N(3)-H(3A)	0.8800	C(10)-H(10)	0.9500
N(3)-H(3B)	0.8800	C(11)-H(11)	0.9500
O(1)-C(2)	1.2165(19)	C(12)-C(13)	1.518(2)
O(2)-C(12)	1.4176(19)	C(12)-H(12)	1.0000
O(2)-C(19)	1.426(2)	C(13)-C(14)	1.389(2)
O(3)-C(16)	1.369(2)	C(13)-C(18)	1.401(2)
O(3)-C(20)	1.427(2)	C(14)-C(15)	1.390(2)
O(4)-C(9)	1.372(2)	C(14)-H(14)	0.9500
O(4)-H(4D)	0.76(3)	C(15)-C(16)	1.393(2)
O(5)-C(21)	1.400(2)	C(15)-H(15)	0.9500
O(5)-H(5D)	0.88(3)	C(16)-C(17)	1.396(2)
C(2)-C(3)	1.525(2)	C(17)-C(18)	1.382(2)
C(3)-C(5)	1.540(2)	C(17)-H(17)	0.9500
C(3)-C(12)	1.554(2)	C(18)-H(18)	0.9500
C(4)-H(4A)	0.9800	C(19)-H(19A)	0.9800
C(4)-H(4B)	0.9800	C(19)-H(19B)	0.9800
C(4)-H(4C)	0.9800	C(19)-H(19C)	0.9800
C(5)-C(6)	1.511(2)	C(20)-H(20A)	0.9800
C(5)-H(5A)	0.9900	C(20)-H(20B)	0.9800
C(5)-H(5B)	0.9900	C(20)-H(20C)	0.9800
C(6)-C(7)	1.393(2)	C(21)-H(21A)	0.9800
C(6)-C(11)	1.397(2)	C(21)-H(21B)	0.9800

C(21)-H(21C)	0.9800

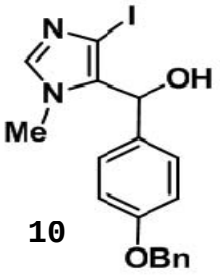
C(2)-N(1)-C(1)	111.30(13)	H(4B)-C(4)-H(4C)	109.5
C(2)-N(1)-C(4)	123.62(14)	C(6)-C(5)-C(3)	113.47(13)
C(1)-N(1)-C(4)	124.89(13)	C(6)-C(5)-H(5A)	108.9
C(1)-N(2)-C(3)	111.83(13)	C(3)-C(5)-H(5A)	108.9
C(1)-N(3)-H(3A)	120.0	C(6)-C(5)-H(5B)	108.9
C(1)-N(3)-H(3B)	120.0	C(3)-C(5)-H(5B)	108.9
H(3A)-N(3)-H(3B)	120.0	H(5A)-C(5)-H(5B)	107.7
C(12)-O(2)-C(19)	111.82(13)	C(7)-C(6)-C(11)	117.39(15)
C(16)-O(3)-C(20)	116.77(14)	C(7)-C(6)-C(5)	121.25(15)
C(9)-O(4)-H(4D)	104(2)	C(11)-C(6)-C(5)	121.35(15)
C(21)-O(5)-H(5D)	107.9(16)	C(8)-C(7)-C(6)	121.61(15)
N(3)-C(1)-N(2)	129.69(15)	C(8)-C(7)-H(7)	119.2
N(3)-C(1)-N(1)	122.20(15)	C(6)-C(7)-H(7)	119.2
N(2)-C(1)-N(1)	108.11(13)	C(7)-C(8)-C(9)	119.95(15)
O(1)-C(2)-N(1)	126.35(15)	C(7)-C(8)-H(8)	120.0
O(1)-C(2)-C(3)	126.69(14)	C(9)-C(8)-H(8)	120.0
N(1)-C(2)-C(3)	106.94(13)	O(4)-C(9)-C(10)	118.46(15)
N(2)-C(3)-C(2)	101.72(12)	O(4)-C(9)-C(8)	122.13(15)
N(2)-C(3)-C(5)	112.83(13)	C(10)-C(9)-C(8)	119.41(15)
C(2)-C(3)-C(5)	111.75(13)	C(11)-C(10)-C(9)	119.91(16)
N(2)-C(3)-C(12)	111.46(13)	C(11)-C(10)-H(10)	120.0
C(2)-C(3)-C(12)	107.32(12)	C(9)-C(10)-H(10)	120.0
C(5)-C(3)-C(12)	111.28(13)	C(10)-C(11)-C(6)	121.73(15)
N(1)-C(4)-H(4A)	109.5	C(10)-C(11)-H(11)	119.1
N(1)-C(4)-H(4B)	109.5	C(6)-C(11)-H(11)	119.1
H(4A)-C(4)-H(4B)	109.5	O(2)-C(12)-C(13)	112.20(13)
N(1)-C(4)-H(4C)	109.5	O(2)-C(12)-C(3)	105.87(12)
H(4A)-C(4)-H(4C)	109.5	C(13)-C(12)-C(3)	112.35(13)

O(2)-C(12)-H(12)	108.8	C(13)-C(18)-H(18)	119.4
C(13)-C(12)-H(12)	108.8	O(2)-C(19)-H(19A)	109.5
C(3)-C(12)-H(12)	108.8	O(2)-C(19)-H(19B)	109.5
C(14)-C(13)-C(18)	118.15(15)	H(19A)-C(19)-H(19B)	109.5
C(14)-C(13)-C(12)	120.76(14)	O(2)-C(19)-H(19C)	109.5
C(18)-C(13)-C(12)	121.08(14)	H(19A)-C(19)-H(19C)	109.5
C(13)-C(14)-C(15)	121.36(15)	H(19B)-C(19)-H(19C)	109.5
C(13)-C(14)-H(14)	119.3	O(3)-C(20)-H(20A)	109.5
C(15)-C(14)-H(14)	119.3	O(3)-C(20)-H(20B)	109.5
C(14)-C(15)-C(16)	119.68(15)	H(20A)-C(20)-H(20B)	109.5
C(14)-C(15)-H(15)	120.2	O(3)-C(20)-H(20C)	109.5
C(16)-C(15)-H(15)	120.2	H(20A)-C(20)-H(20C)	109.5
O(3)-C(16)-C(15)	124.14(15)	H(20B)-C(20)-H(20C)	109.5
O(3)-C(16)-C(17)	116.17(15)	O(5)-C(21)-H(21A)	109.5
C(15)-C(16)-C(17)	119.69(15)	O(5)-C(21)-H(21B)	109.5
C(18)-C(17)-C(16)	119.87(15)	H(21A)-C(21)-H(21B)	109.5
C(18)-C(17)-H(17)	120.1	O(5)-C(21)-H(21C)	109.5
C(16)-C(17)-H(17)	120.1	H(21A)-C(21)-H(21C)	109.5
C(17)-C(18)-C(13)	121.23(15)	H(21B)-C(21)-H(21C)	109.5
C(17)-C(18)-H(18)	119.4		

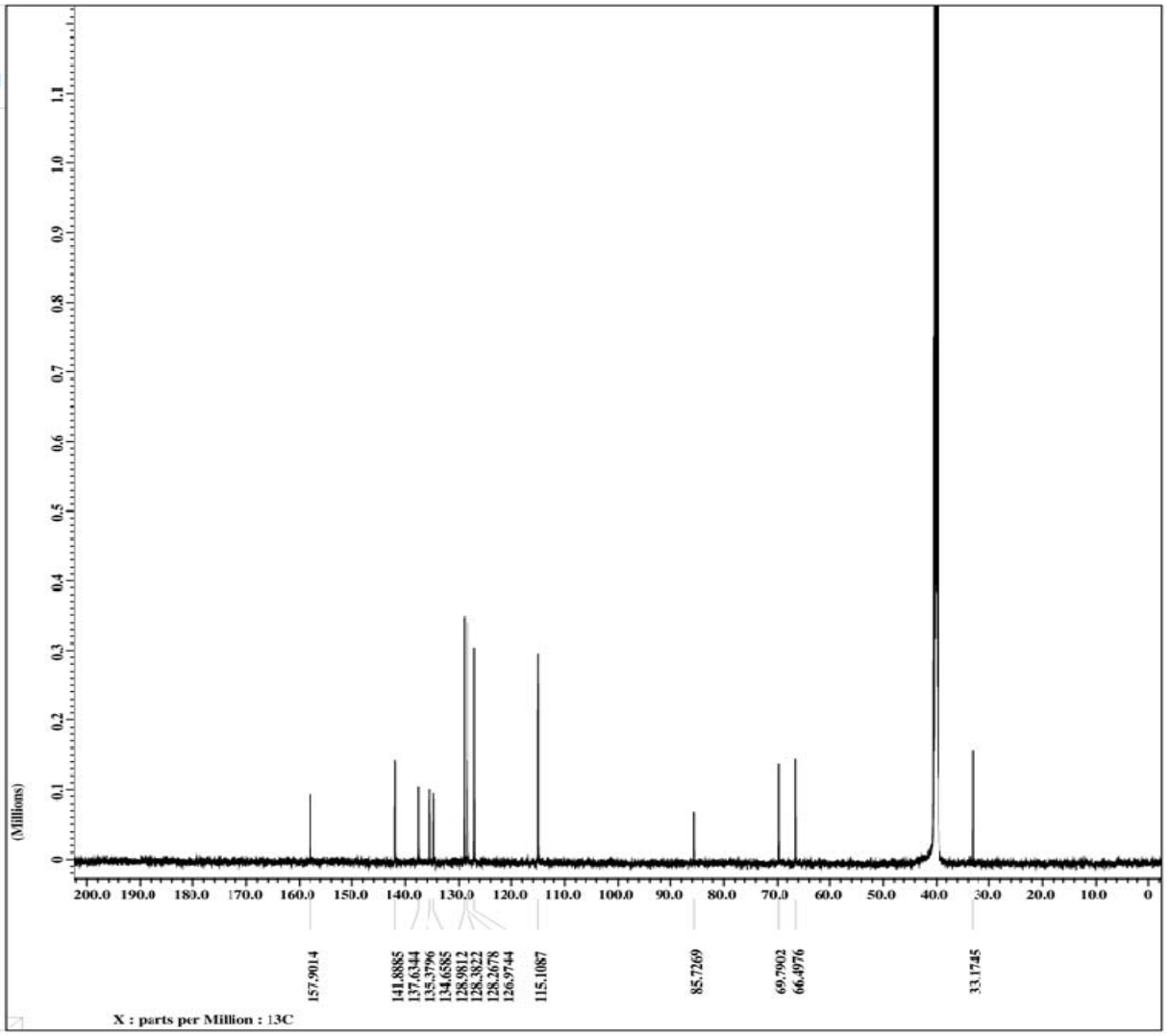
Symmetry transformations used to generate equivalent atoms:

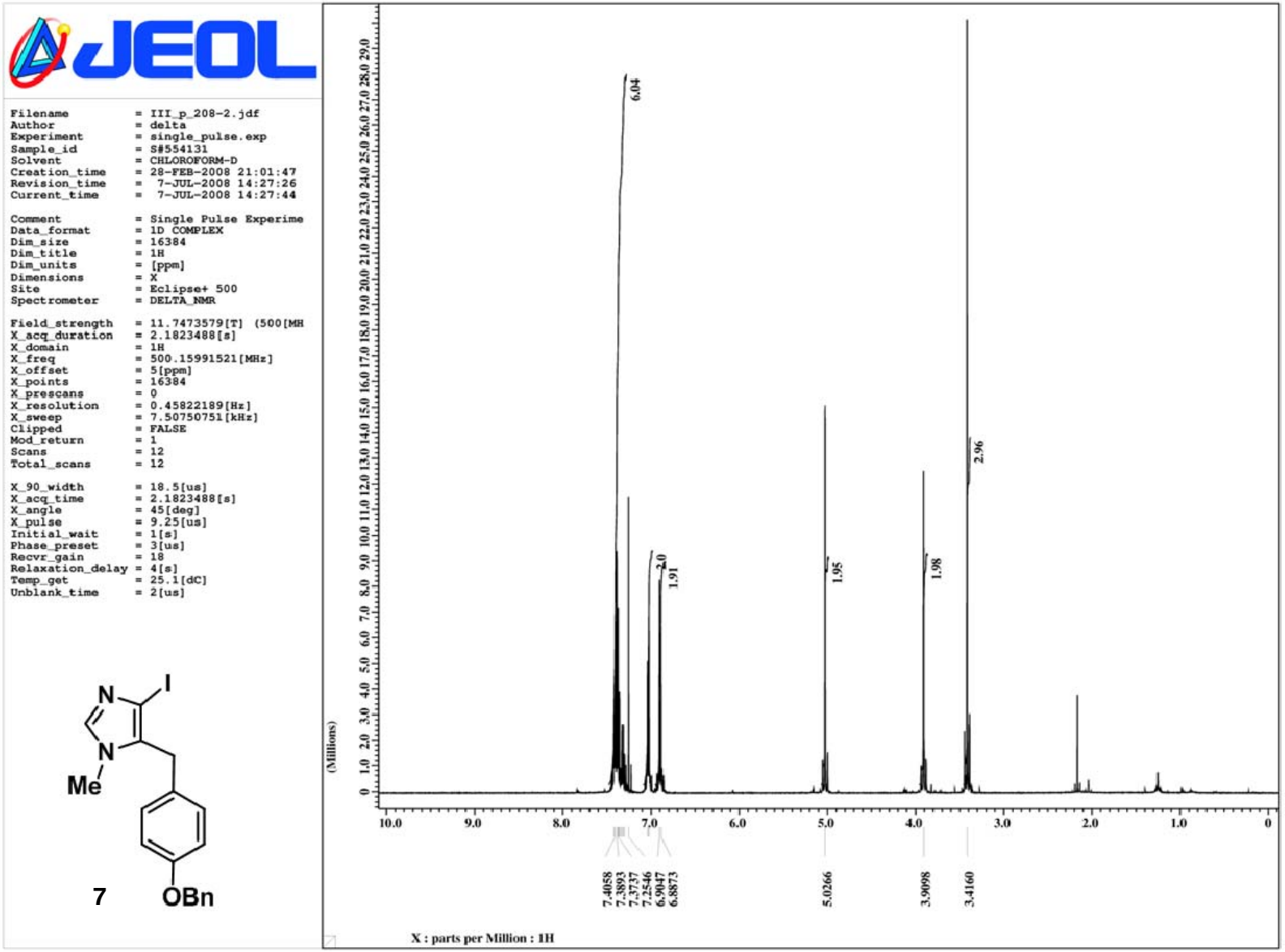

Filename = III_p_200_inDMSO-3.jd
Author = delta
Experiment = single_pulse.exp
Sample_id = S#470058
Solvent = DMSO-D6
Creation_time = 26-FEB-2008 18:39:51
Revision_time = 7-JUL-2008 13:54:18
Current_time = 7-JUL-2008 13:54:58
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 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 = 20
Relaxation_delay = 4[s]
Temp_get = 25.1[dC]
Unblank_time = 2[us]
N
I
N
OH
Me
10
OBn
(Millions)
3.36
2.07
2.05
2.08
2.05
2.03
0.98
1.02
1.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.5826
7.4269
7.3811
7.1639
7.0009
6.9835
6.2267
6.2185
5.8071
5.7998
5.0678
3.3784
3.3738
2.5053
2.5017
X : parts per Million : 1H

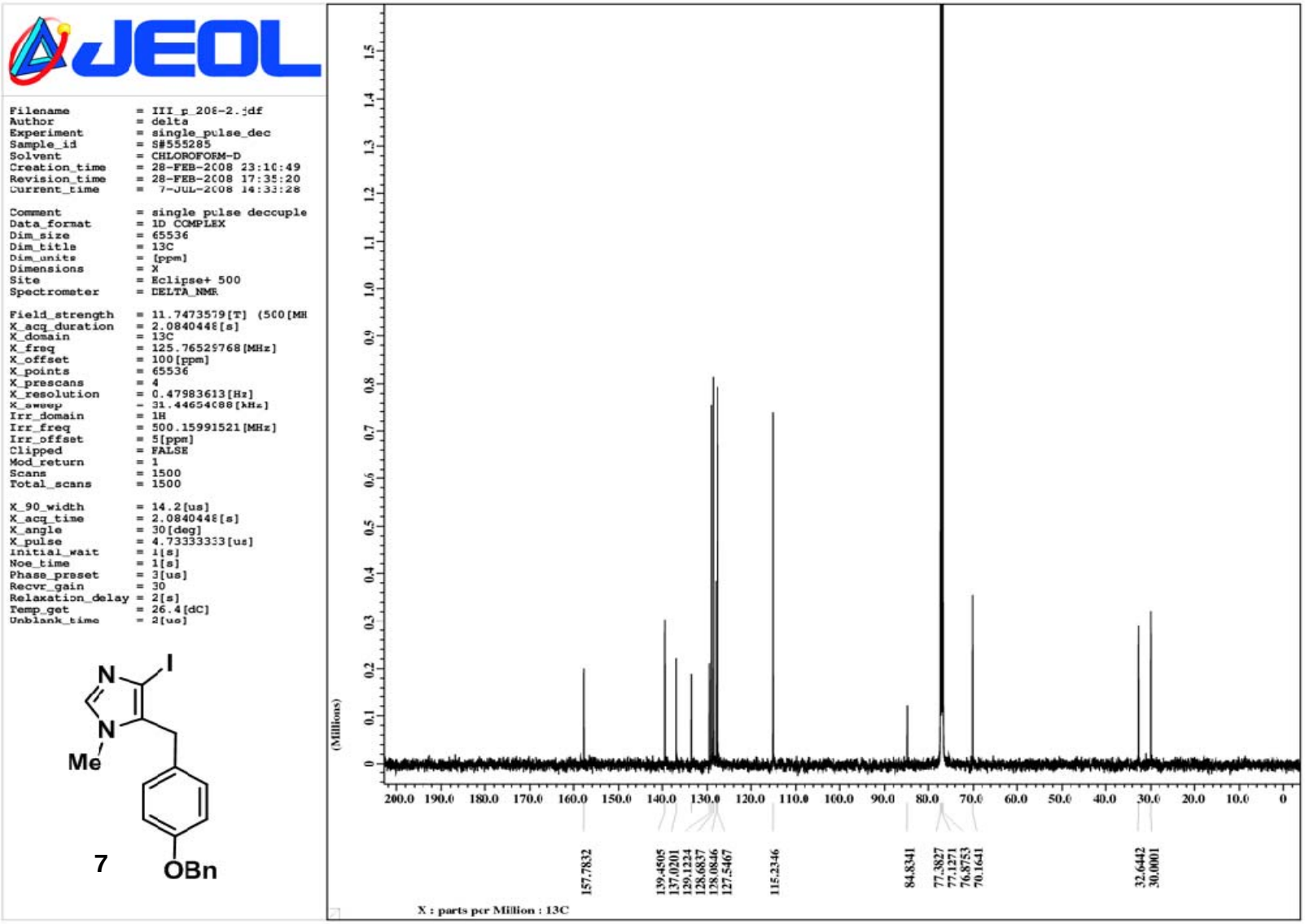
```
Filename          = III_p_200_inDMSO-2.jd
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#741288
Solvent           = DMSO-D6
Creation_time     = 27-FEB-2008 11:14:26
Revision_time     = 27-FEB-2008 09:29:53
Current_time      =  7-JUL-2008 14:18:48

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    = 11.7473579[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           = FALSE
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_presat      = 3[us]
Recvr_gain        = 29
Relaxation_delay  = 2[s]
Temp_get          = 26.4[dC]
Unblank_time      = 2[us]
```

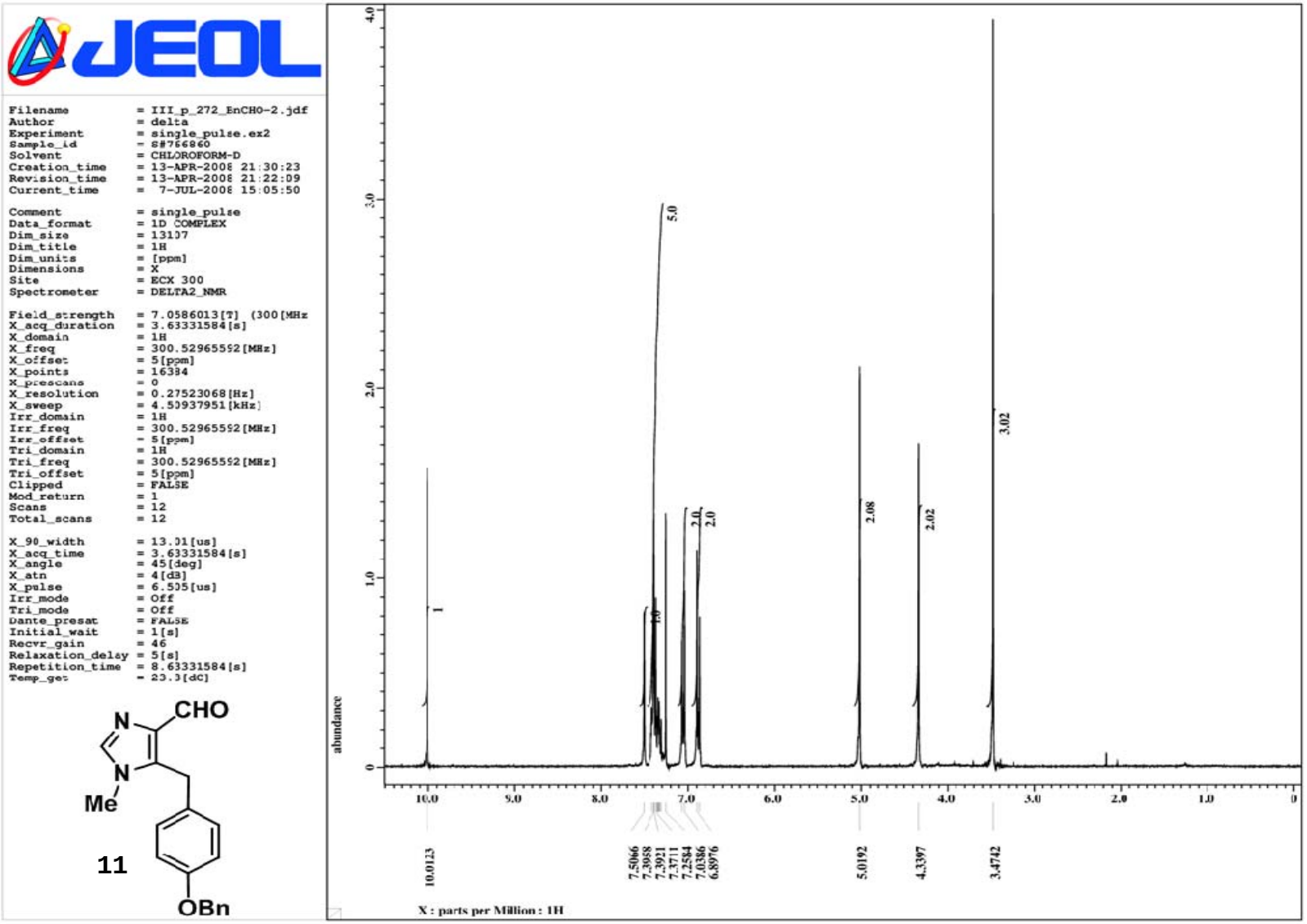


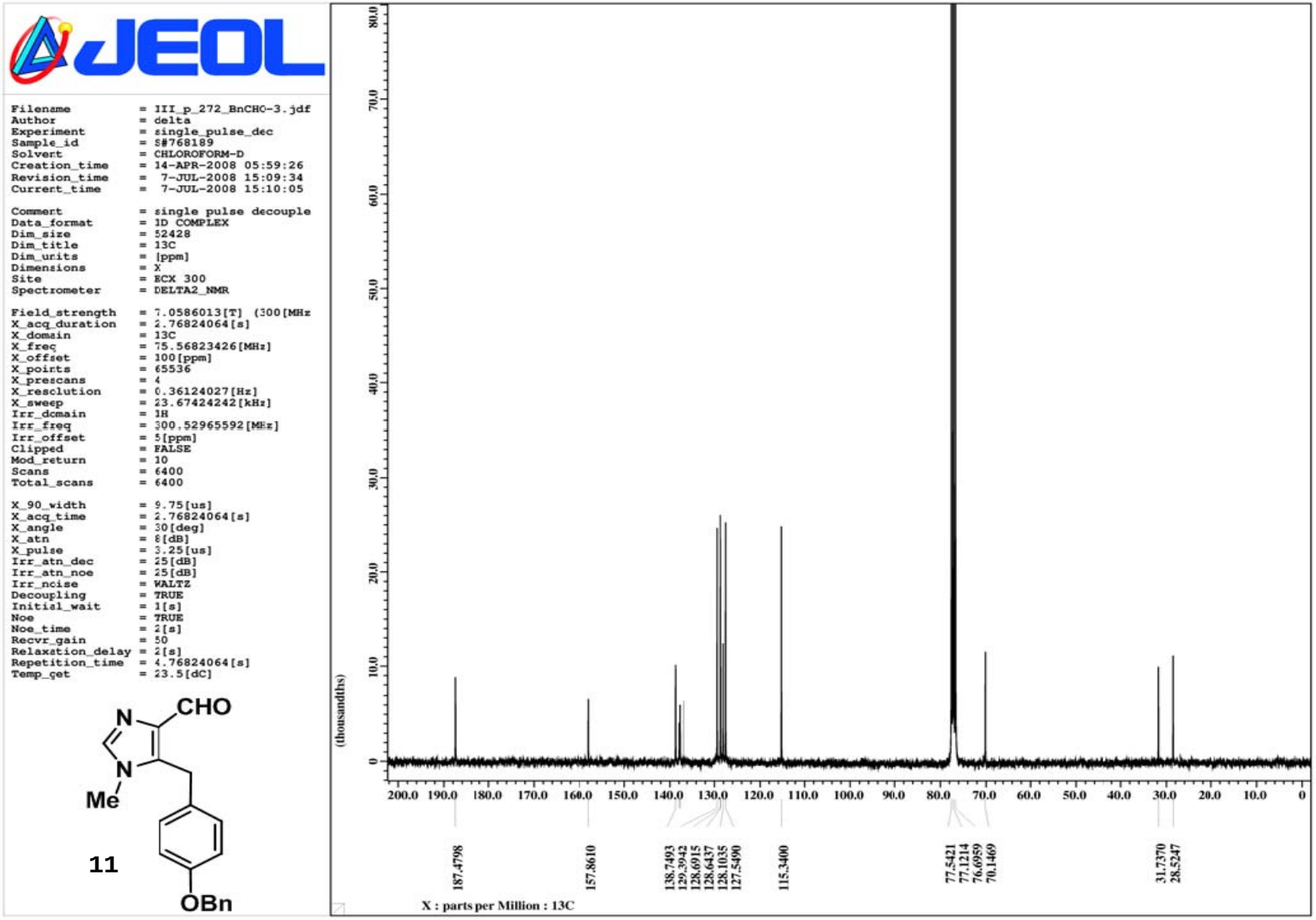
10

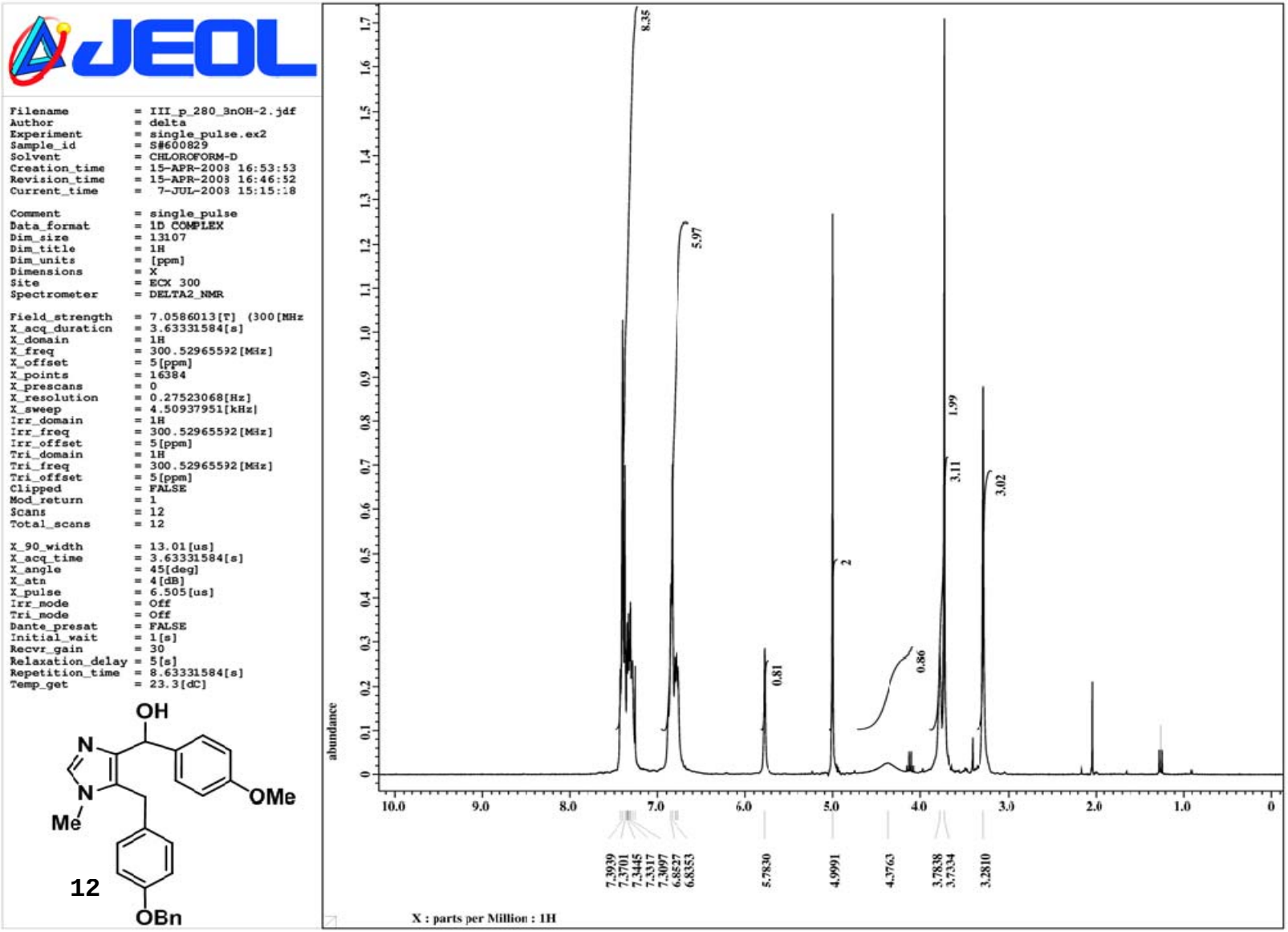


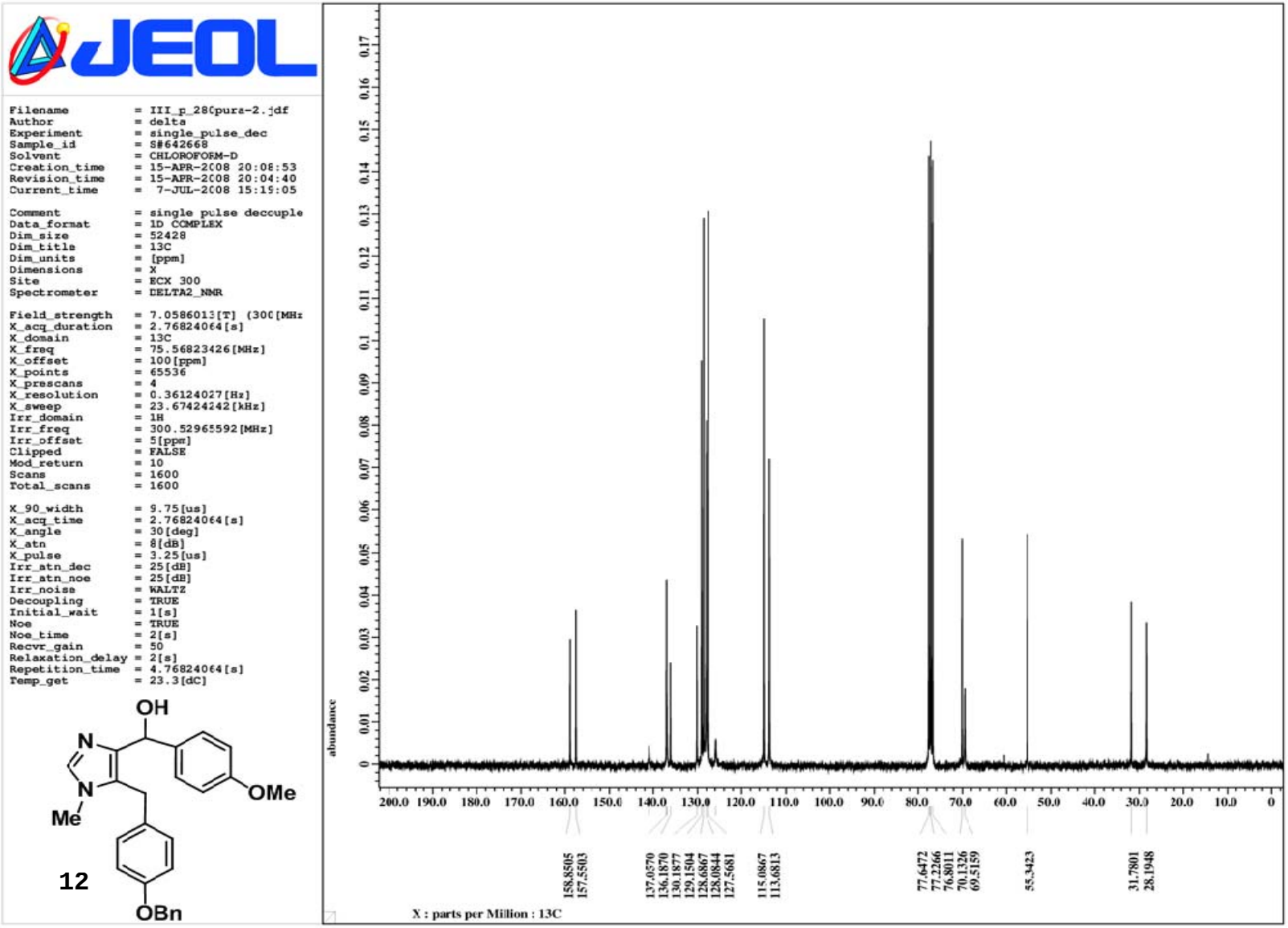

Filename = III_p_208-2.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#554131
Solvent = CHLOROFORM-D
Creation_time = 28-FEB-2008 21:01:47
Revision_time = 7-JUL-2008 14:27:26
Current_time = 7-JUL-2008 14:27:44
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 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 = 18
Relaxation_delay = 4[s]
Temp_get = 25.1[dC]
Unblank_time = 2[us]
N
I
N
Me
7
OBn
(Millions)
6.04
2.0
1.91
1.95
1.98
2.96
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4058
7.3893
7.3737
7.2546
6.9047
6.8873
5.0266
3.9098
3.4160
X : parts per Million : 1H

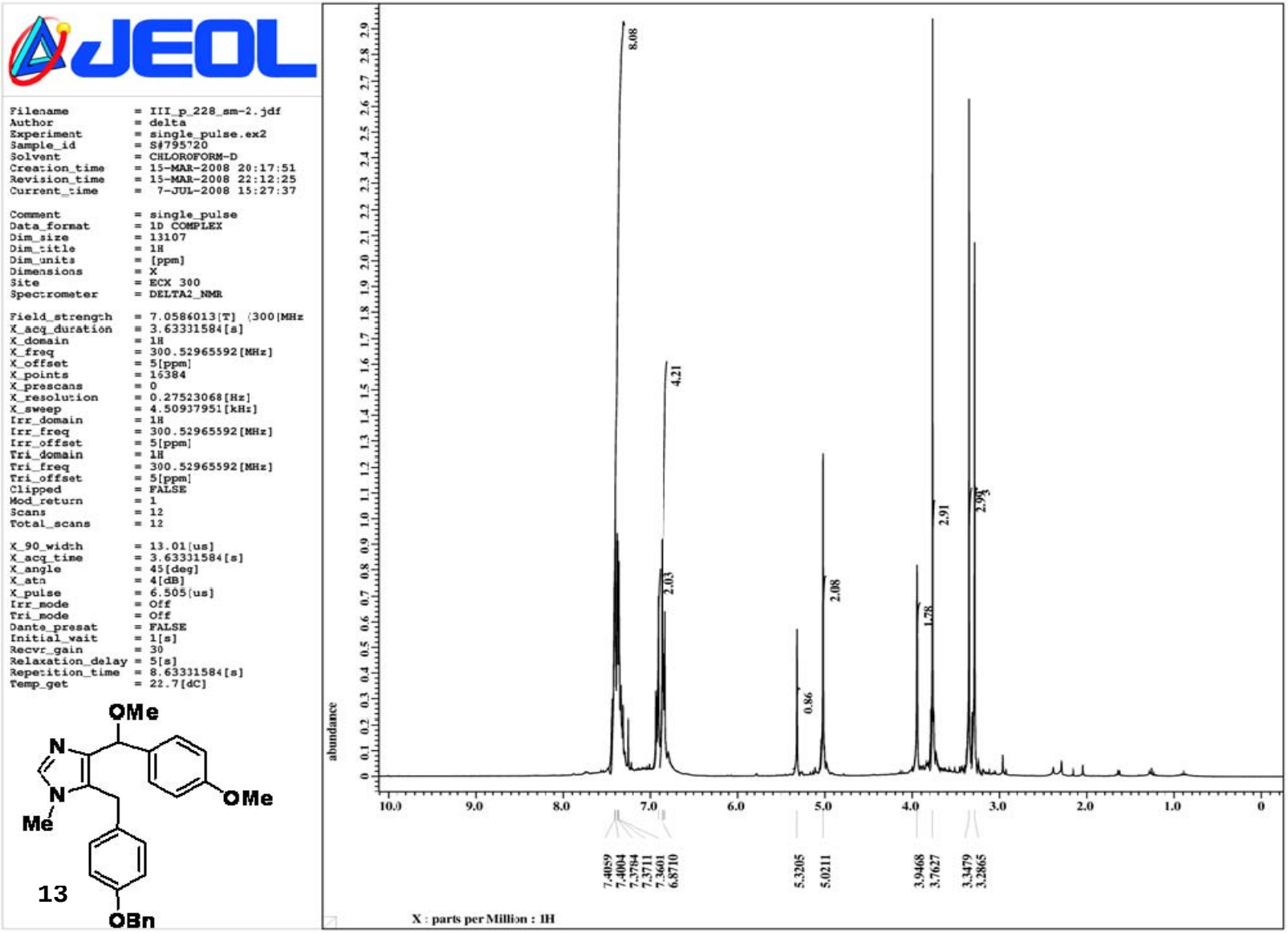

Filename = III_p_208-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#555285
Solvent = CHLOROFORM-D
Creation_time = 28-FEB-2008 23:10:49
Revision_time = 28-FEB-2008 17:35:20
Current_time = 7-JUL-2008 14:33:28
Comment = single pulse deccuple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 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 = FALSE
Mod_return = 1
Scans = 1500
Total_scans = 1500
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 = 26.4[dC]
Unblank_time = 2[us]
N
I
N
Me
7
OBn
(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 0
157.7832
139.4505
137.0201
129.1224
128.6837
128.0846
127.5467
115.2346
84.8341
77.3827
77.1271
76.8753
70.1641
32.6442
30.0001
X : parts per Million : 13C

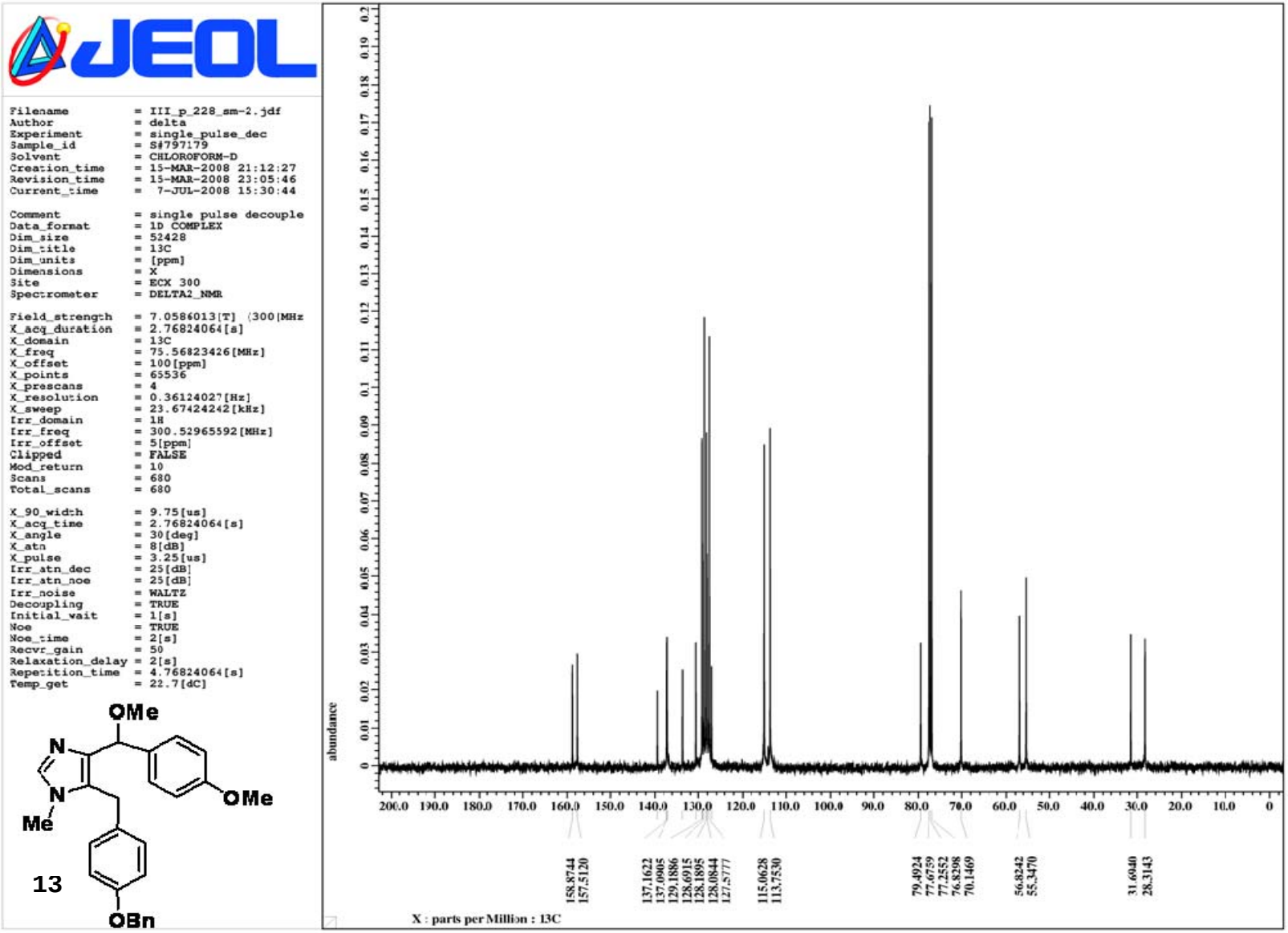

Filename = III_p_272_EnCHO-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#766860
Solvent = CHLOROFORM-D
Creation_time = 13-APR-2008 21:30:23
Revision_time = 13-APR-2008 21:22:09
Current_time = 7-JUL-2008 15:05:50
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 300.52965552[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 = 300.52965552[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 300.52965552[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 13.31[us]
X_acq_time = 3.63331584[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.655[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 23.3[dC]
CHO
N
N
Me
OBn
11
abundance
5.0
1
1.0
2.0
2.0
2.08
2.02
3.02
10.0123
7.5066
7.3958
7.3921
7.3711
7.2584
7.0386
6.8976
5.0192
4.3397
3.4742
X : parts per Million : 1H

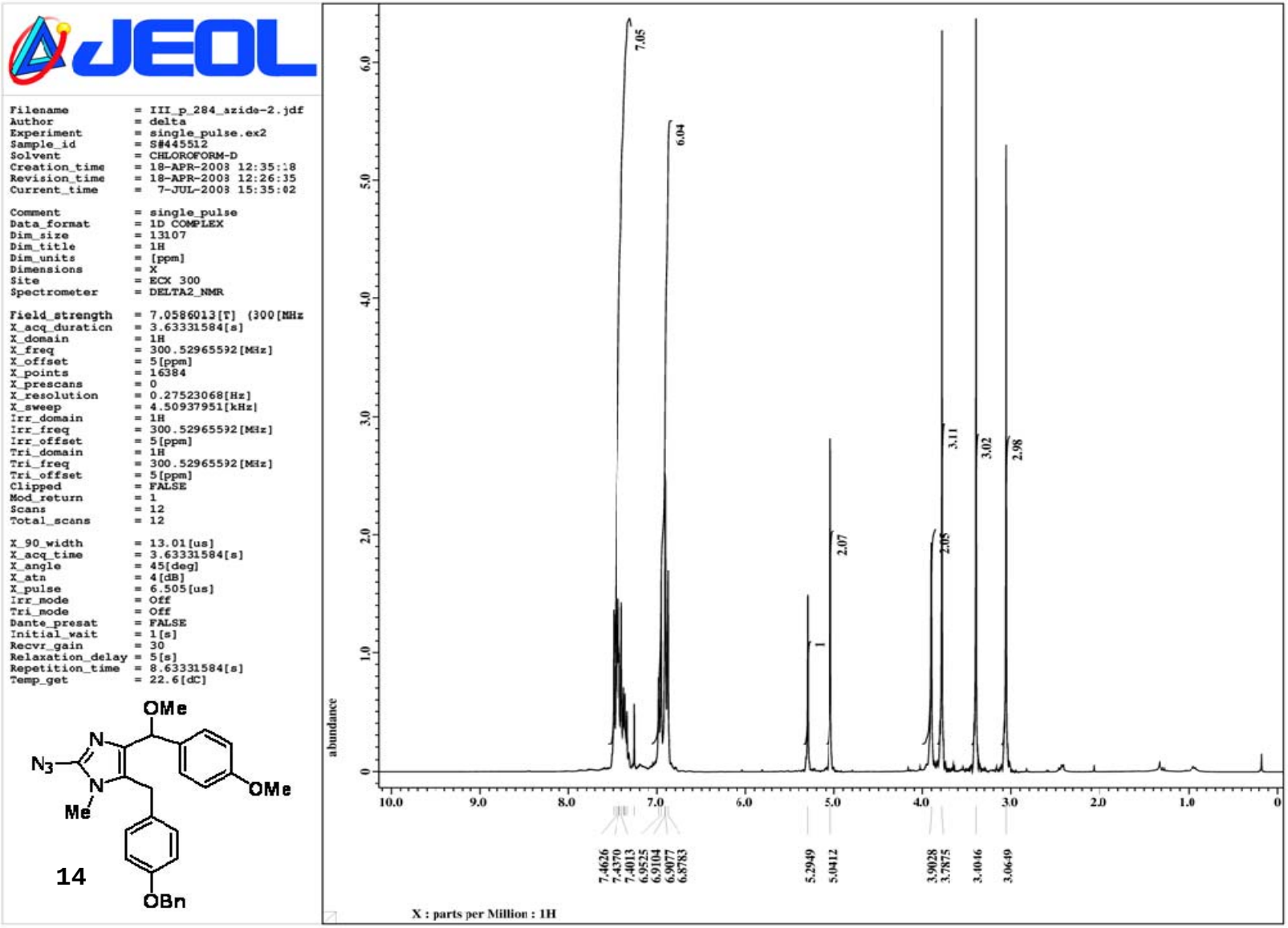

Filename = III_p_272_BnCHO-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#768189
Solvent = CHLOROFORM-D
Creation_time = 14-APR-2008 05:59:26
Revision_time = 7-JUL-2008 15:09:34
Currert_time = 7-JUL-2008 15:10:05
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 6400
Total_scans = 6400
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[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 = 23.5[dC]
N
CHO
N
Me
11
OBn
(thousandths)
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
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
187.4798
157.8610
138.7493
129.3942
128.6915
128.6437
128.1035
127.5490
115.3400
77.5421
77.1214
76.6959
70.1469
31.7370
28.5247
X : parts per Million : 13C

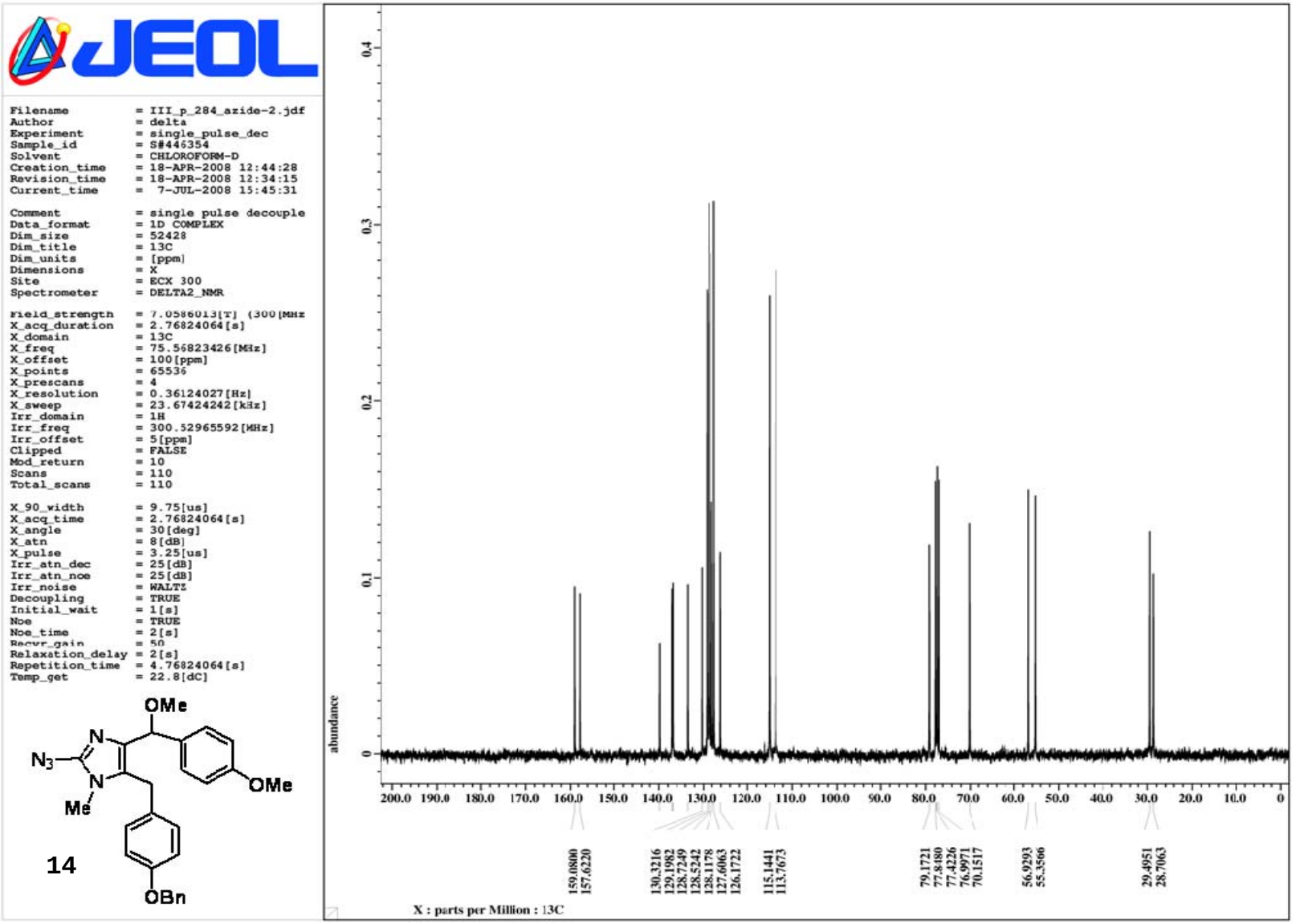

Filename = III_p_280_3nOH-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#600829
Solvent = CHLOROFORM-D
Creation_time = 15-APR-2003 16:53:53
Revision_time = 15-APR-2003 16:46:52
Current_time = 7-JUL-2003 15:15:18
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 30
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 23.3[dC]
OH
N
N
Me
OMe
12
OBn
abundance
8.35
5.97
2
0.81
0.86
1.99
3.11
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3939
7.3701
7.3445
7.3317
7.3097
6.8527
6.8353
5.7830
4.9991
4.3763
3.7838
3.7334
3.2810
X : parts per Million : 1H

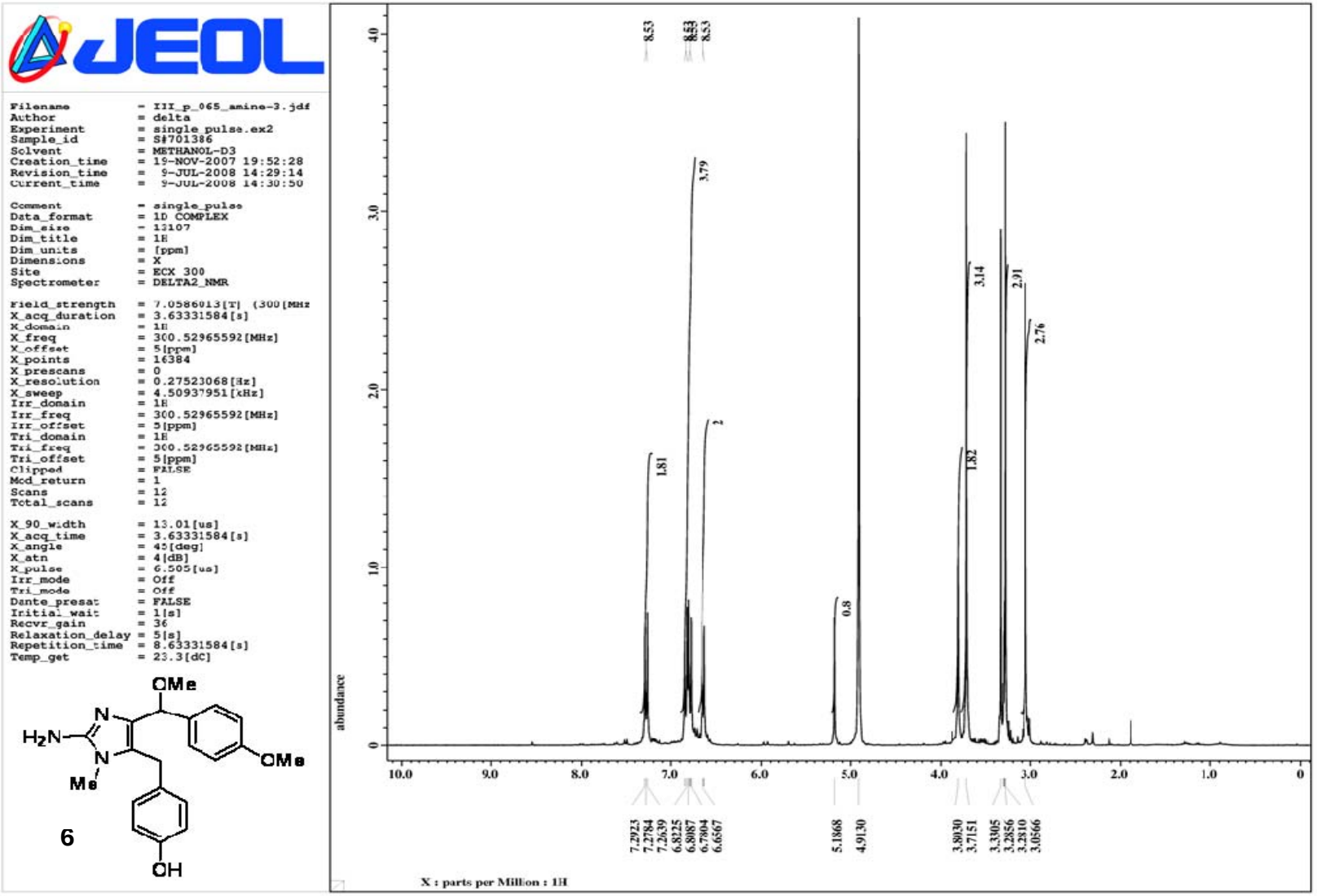

Filename = III_p_28(pure-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#642668
Solvent = CHLOROFORM-D
Creation_time = 15-APR-2008 20:08:53
Revision_time = 15-APR-2008 20:04:40
Current_time = 7-JUL-2008 15:15:05
Comment = single pulse deccuple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 1600
Total_scans = 1600
X_90_width = 9.75[us]
X_acq_time = 2.76824064[s]
X_angle = 30[deg]
X_atn = 6[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]
OH
N
N
Me
OMe
OBn
12
abundance
0 0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.1 0.11 0.12 0.13 0.14 0.15 0.16 0.17
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
158.8505
157.5503
137.0570
136.1870
130.1877
129.1504
128.6867
128.0844
127.5681
115.0867
113.6813
77.6472
77.2266
76.8011
70.1326
69.5159
55.3423
31.7801
28.1948
X : parts per Million : 13C

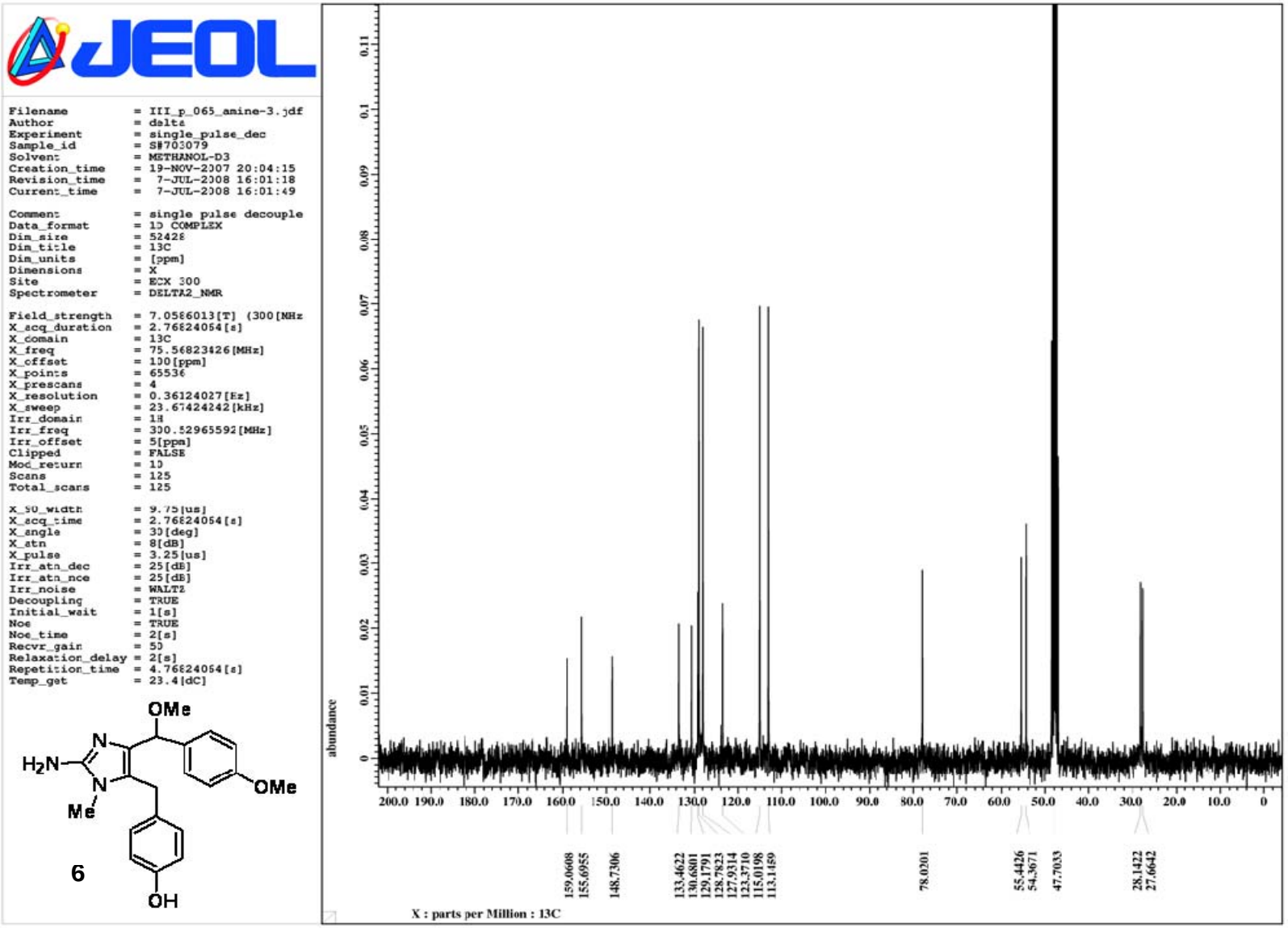

Filename = III_p_228_sm-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#795720
Solvent = CHLOROFORM-D
Creation_time = 15-MAR-2008 20:17:51
Revision_time = 15-MAR-2008 22:12:25
Current_time = 7-JUL-2008 15:27:37
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 30
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 22.7[dC]
OMe
N
N
Me
OMe
13
OBn
abundance
8.08
4.21
2.03
0.86
2.08
1.78
2.91
2.98
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4089
7.4004
7.3784
7.3711
7.3601
6.8710
5.3205
5.0211
3.9468
3.7627
3.3479
3.2865
X : parts per Million : 1H

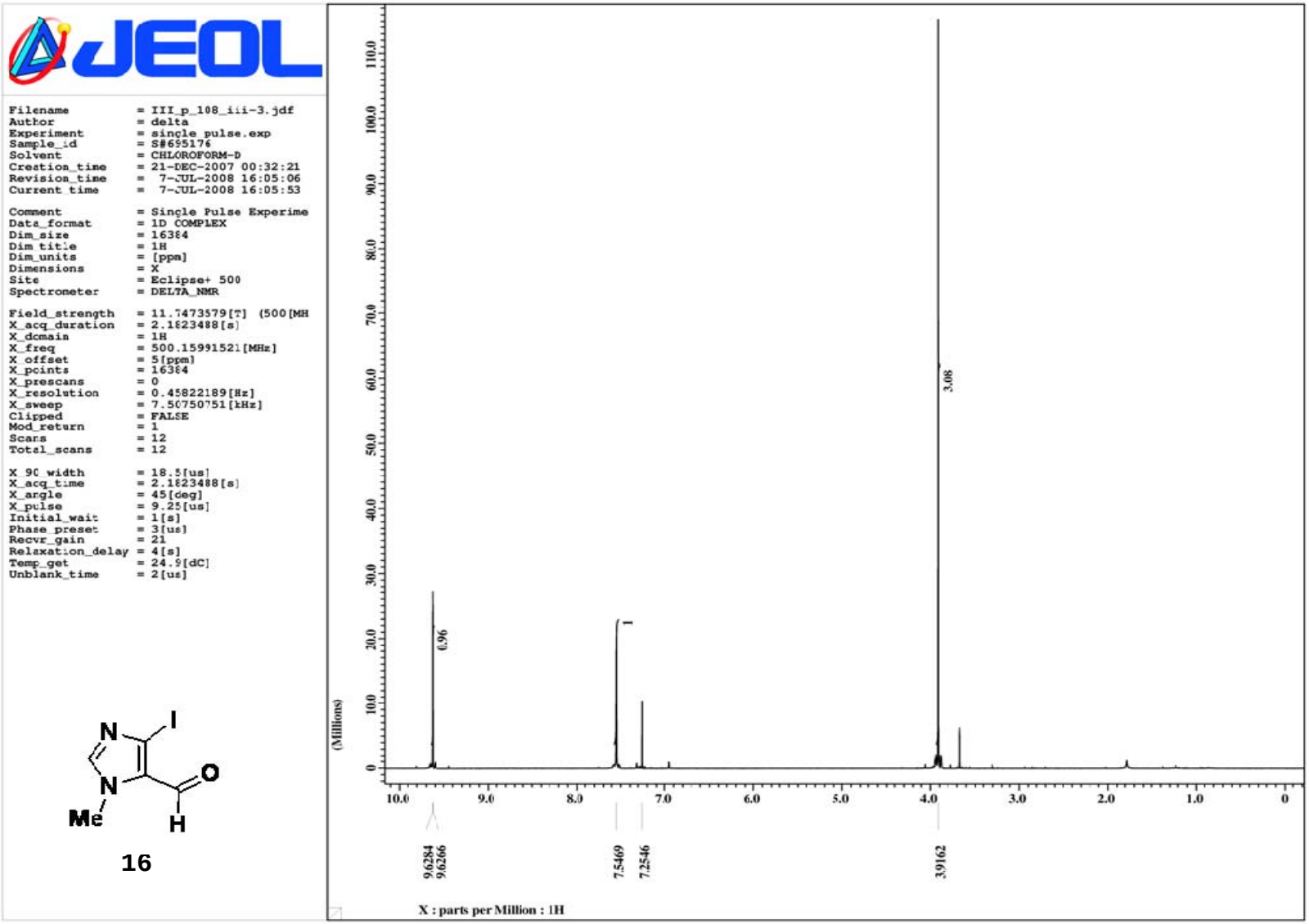

Filename = III_p_228_sm-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#797179
Solvent = CHLOROFORM-D
Creation_time = 15-MAR-2008 21:12:27
Revision_time = 15-MAR-2008 23:05:46
Current_time = 7-JUL-2008 15:30:44
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 680
Total_scans = 680
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 = 22.7[dC]
OMe
N
N
Me
OMe
13
OBn
abundance
158.8744
157.5120
137.1622
137.0905
129.1886
128.6915
128.1895
128.0844
127.5777
115.0628
113.7530
79.4924
77.6759
77.2552
76.8398
70.1469
56.8242
55.3470
31.6940
28.3143
X : parts per Million : 13C

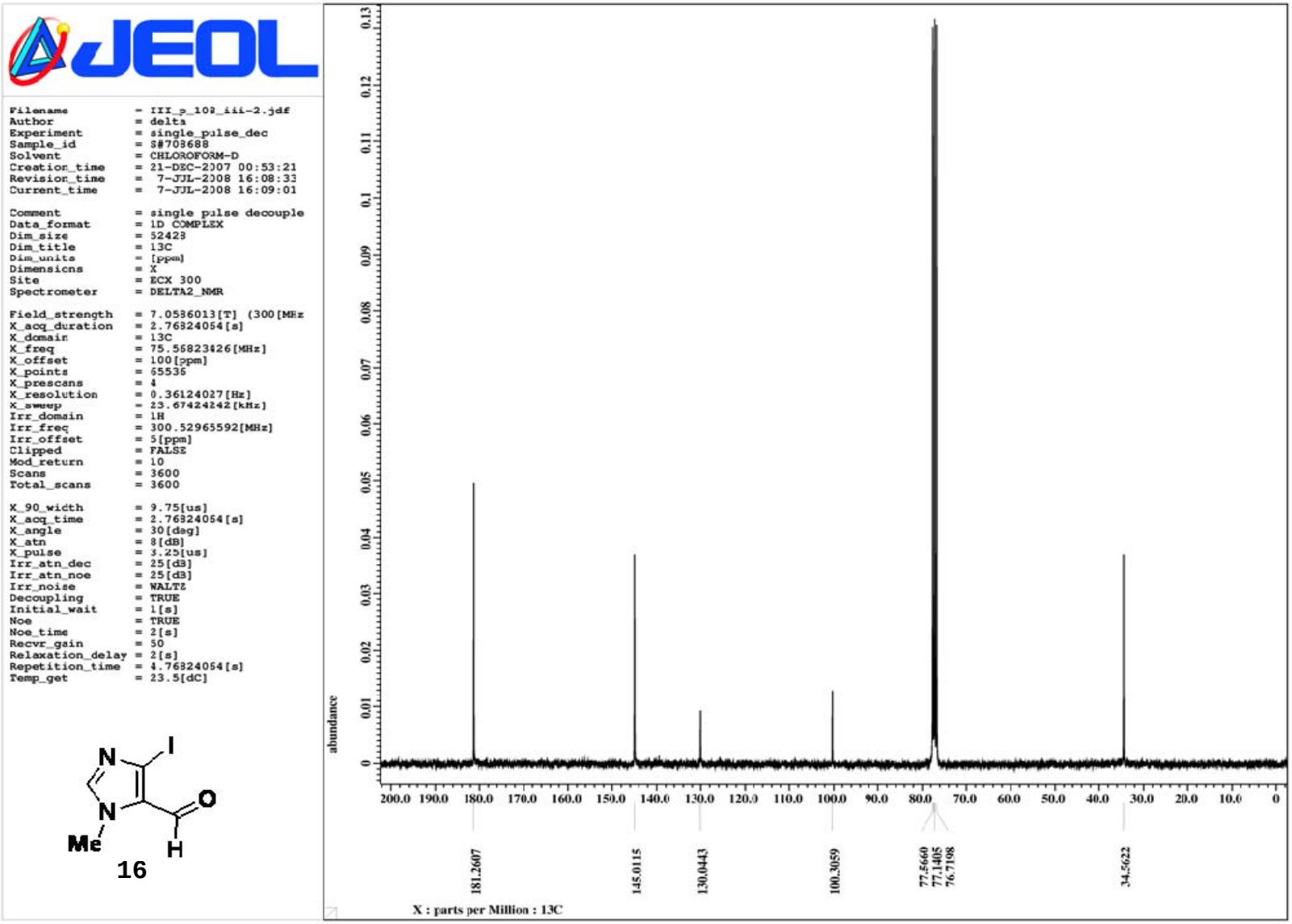

Filename = III_p_284_azide-2.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#445512
Solvent = CHLOROFORM-D
Creation_time = 18-APR-2003 12:35:18
Revision_time = 18-APR-2003 12:26:35
Current_time = 7-JUL-2003 15:35:02
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 30
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 22.6[dC]
OMe
N
N3
N
Me
OMe
14
OBn
abundance
7.05
6.04
3.11
3.02
2.98
2.07
2.05
1
7.4626
7.4370
7.4013
6.9525
6.9104
6.9077
6.8783
5.2949
5.0412
3.9028
3.7875
3.4046
3.0649
X : parts per Million : 1H

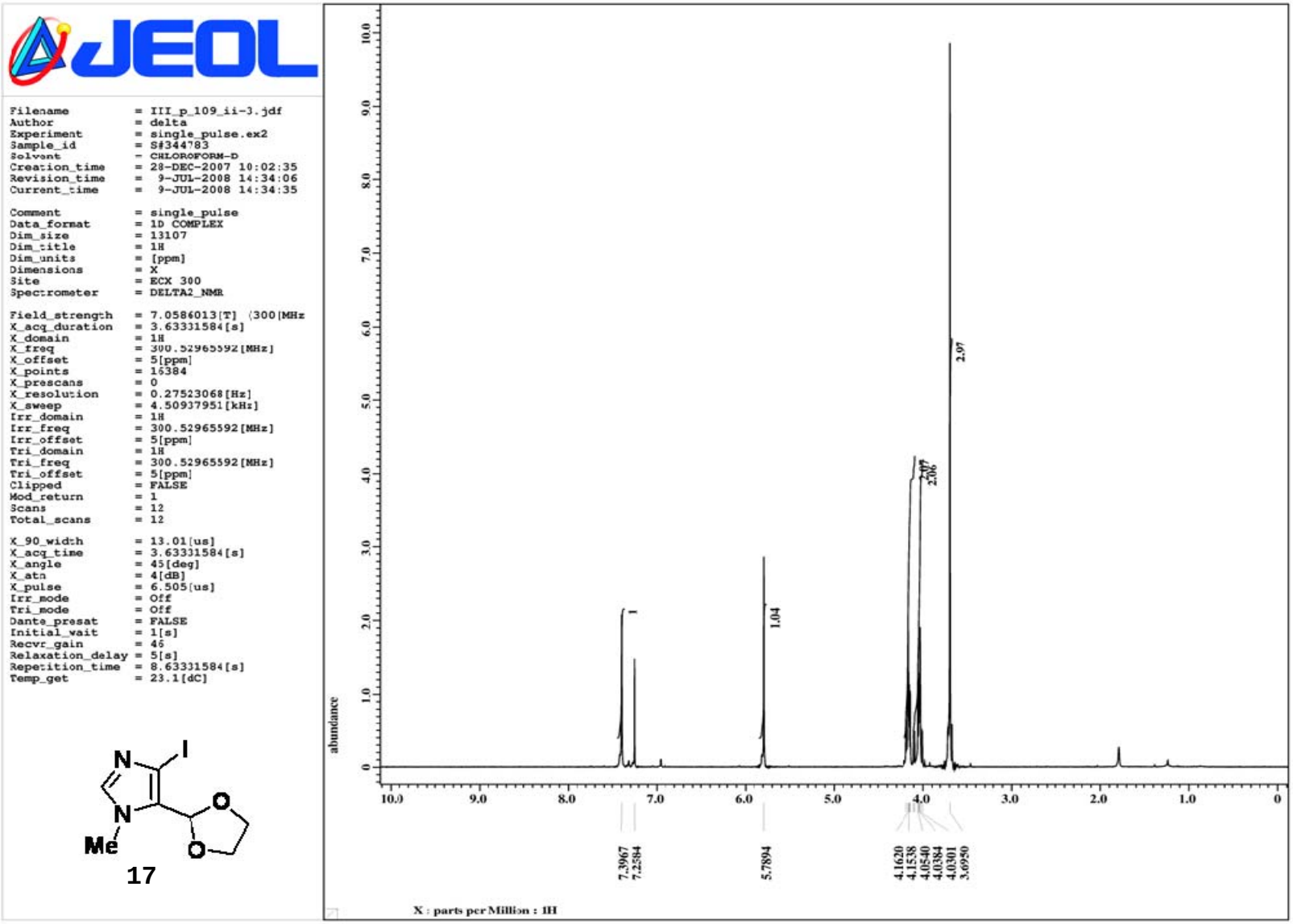

Filename = III_p_284_azide-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#446354
Solvent = CHLOROFORM-D
Creation_time = 18-APR-2008 12:44:28
Revision_time = 18-APR-2008 12:34:15
Current_time = 7-JUL-2008 15:45:31
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 110
Total_scans = 110
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 = 22.8[dC]
OMe
N
N3
N
Me
OMe
OBn
14
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
159.0800
157.6220
130.3216
129.1982
128.7249
128.5242
128.1178
127.6063
126.1722
115.1441
113.7673
79.1721
77.8480
77.4226
76.9971
70.1517
56.9293
55.3566
29.4951
28.7063
X : parts per Million : 13C

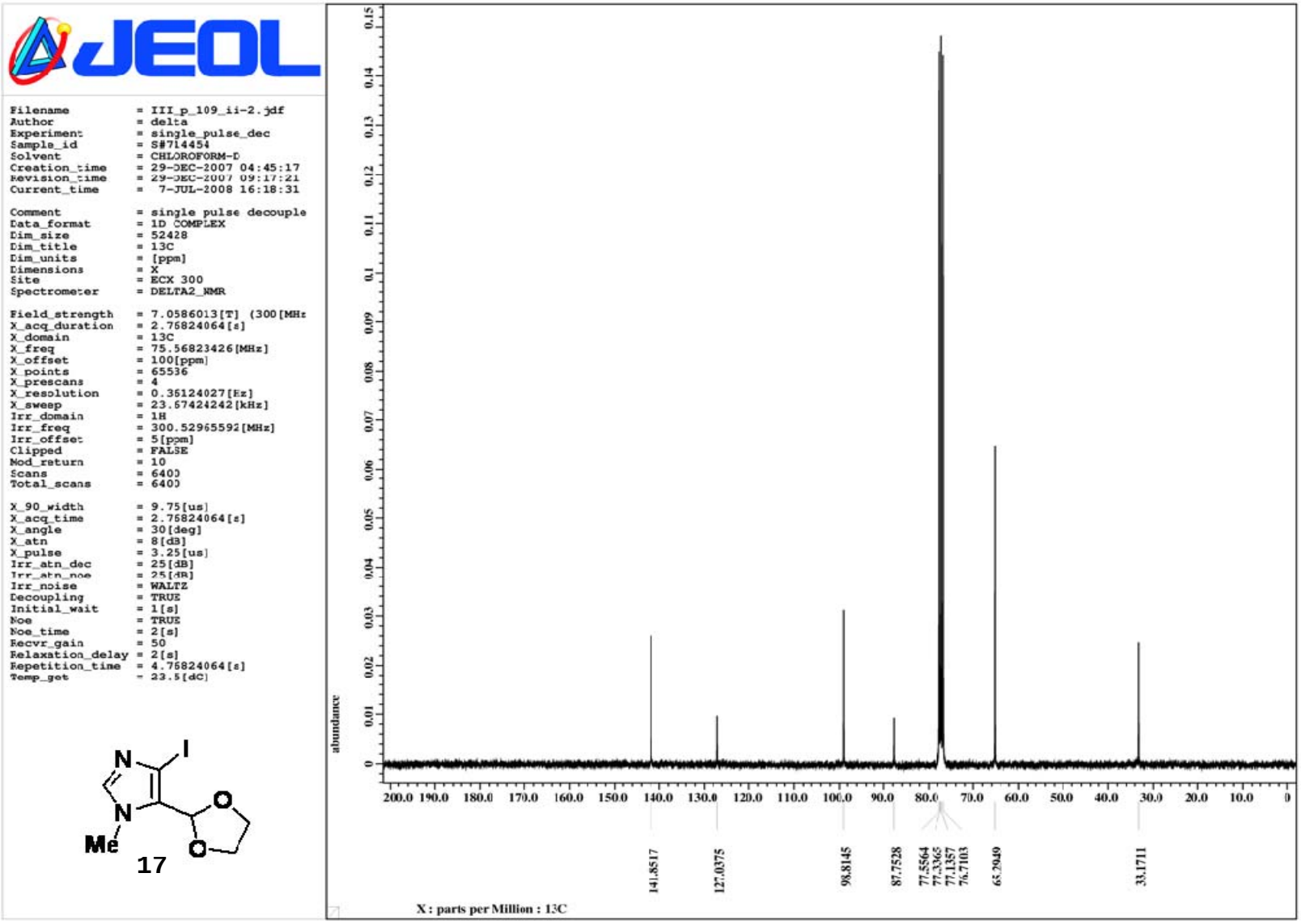

Filename = III_p_065_amine-3.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#701386
Solvent = METHANOL-D3
Creation_time = 19-NOV-2007 19:52:28
Revision_time = 9-JUL-2008 14:29:14
Current_time = 9-JUL-2008 14:30:50
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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
Mcd_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 = 36
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 23.3[dC]
OMe
N
H2N
N
Me
OMe
6
OH
8.53
3.79
1.81
2
0.8
3.14
1.82
2.91
2.76
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2923
7.2784
7.2639
6.8225
6.8087
6.7804
6.6567
5.1868
4.9130
3.8030
3.7151
3.3305
3.2856
3.2810
3.0566
X : parts per Million : 1H

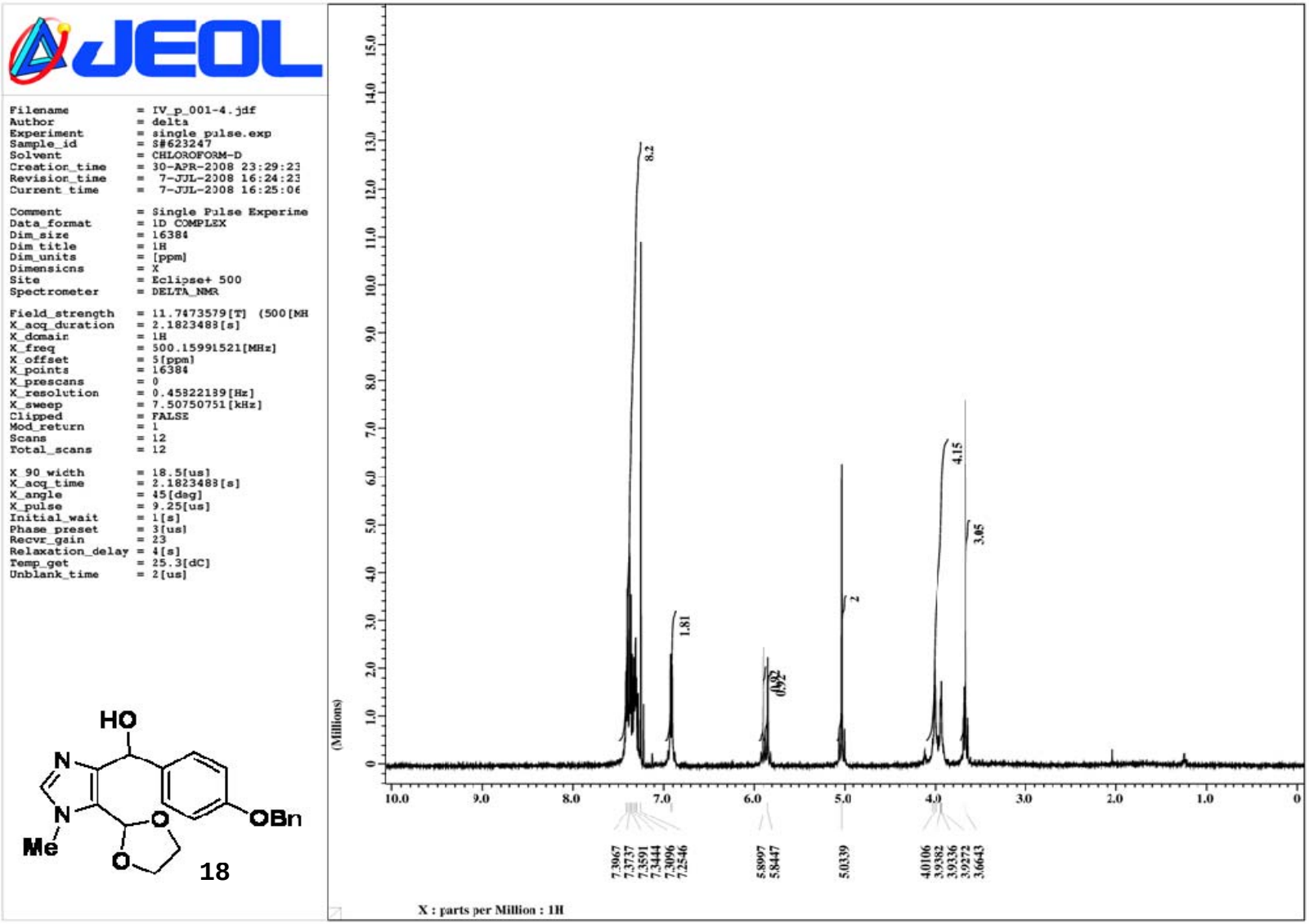

Filename = III_p_065_amine-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#703079
Solvent = METHANOL-D3
Creation_time = 19-NOV-2007 20:04:15
Revision_time = 7-JUL-2008 16:01:18
Current_time = 7-JUL-2008 16:01:49
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824054[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 = 1D
Scans = 125
Total_scans = 125
X_90_width = 9.75[us]
X_acq_time = 2.76824054[s]
X_angle = 30[deg]
X_atn = 8[dB]
X_pulse = 3.25[us]
Irr_atn_dec = 25[dB]
Irr_atn_nce = 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 = 4.76824054[s]
Temp_get = 23.4[dC]
OMe
N
H2N
N
Me
OMe
6
OH
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
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
159.0608
155.6955
148.7306
133.4622
130.6801
129.1791
128.7823
127.9314
123.3710
115.0198
113.1489
78.0301
55.4426
54.3671
47.7033
28.1422
27.6642
X : parts per Million : 13C

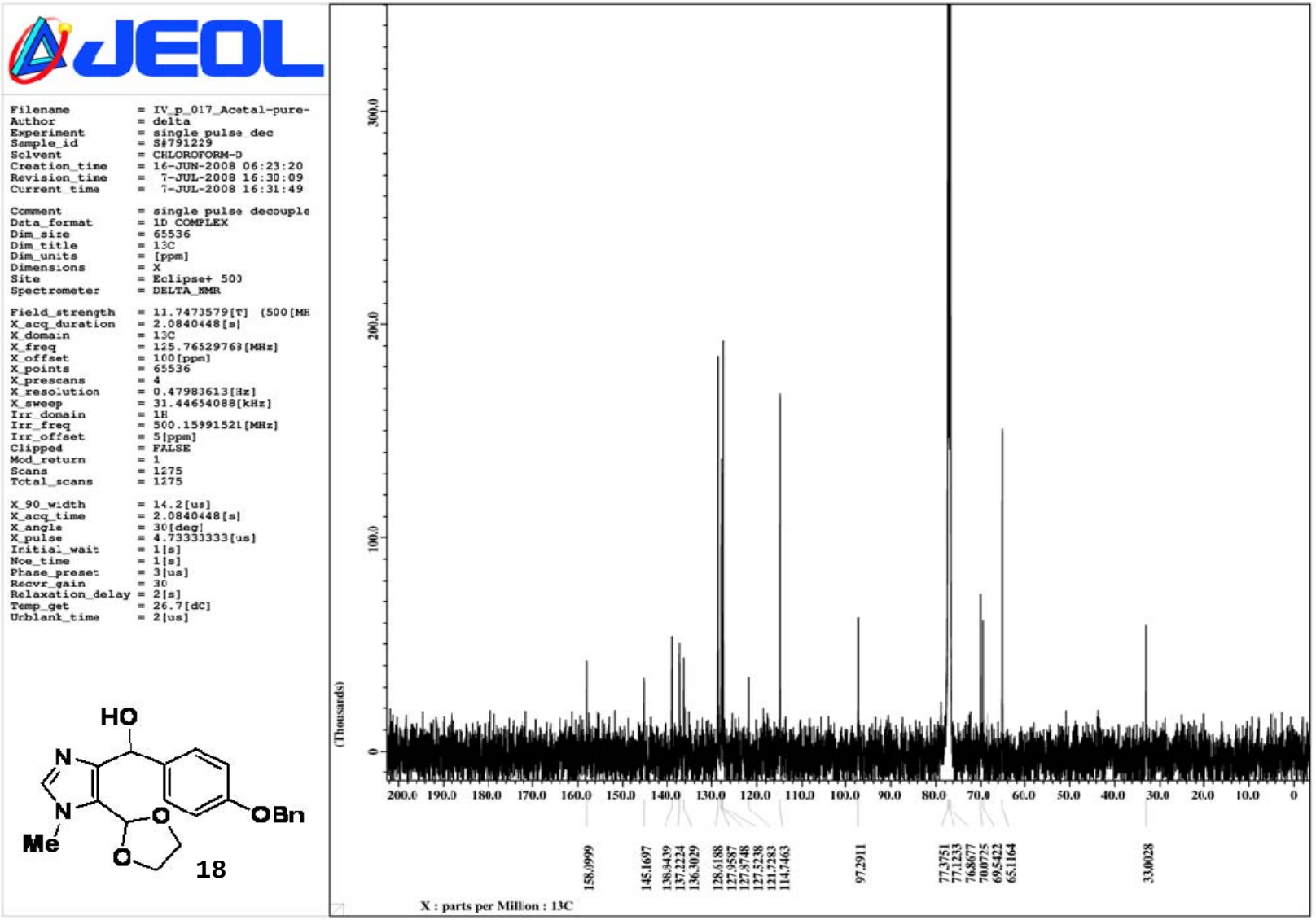

Filename = III_p_108_iii-3.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#695176
Solvent = CHLOROFORM-D
Creation_time = 21-DEC-2007 00:32:21
Revision_time = 7-JUL-2008 16:05:06
Current_time = 7-JUL-2008 16:05:53
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
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 = 21
Relaxation_delay = 4[s]
Temp_get = 24.9[dC]
Unblank_time = 2[us]
N
I
N
O
Me
H
16
(Millions)
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
0.96
1
3.08
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.6284
9.6266
7.5469
7.2546
3.9162
X : parts per Million : 1H

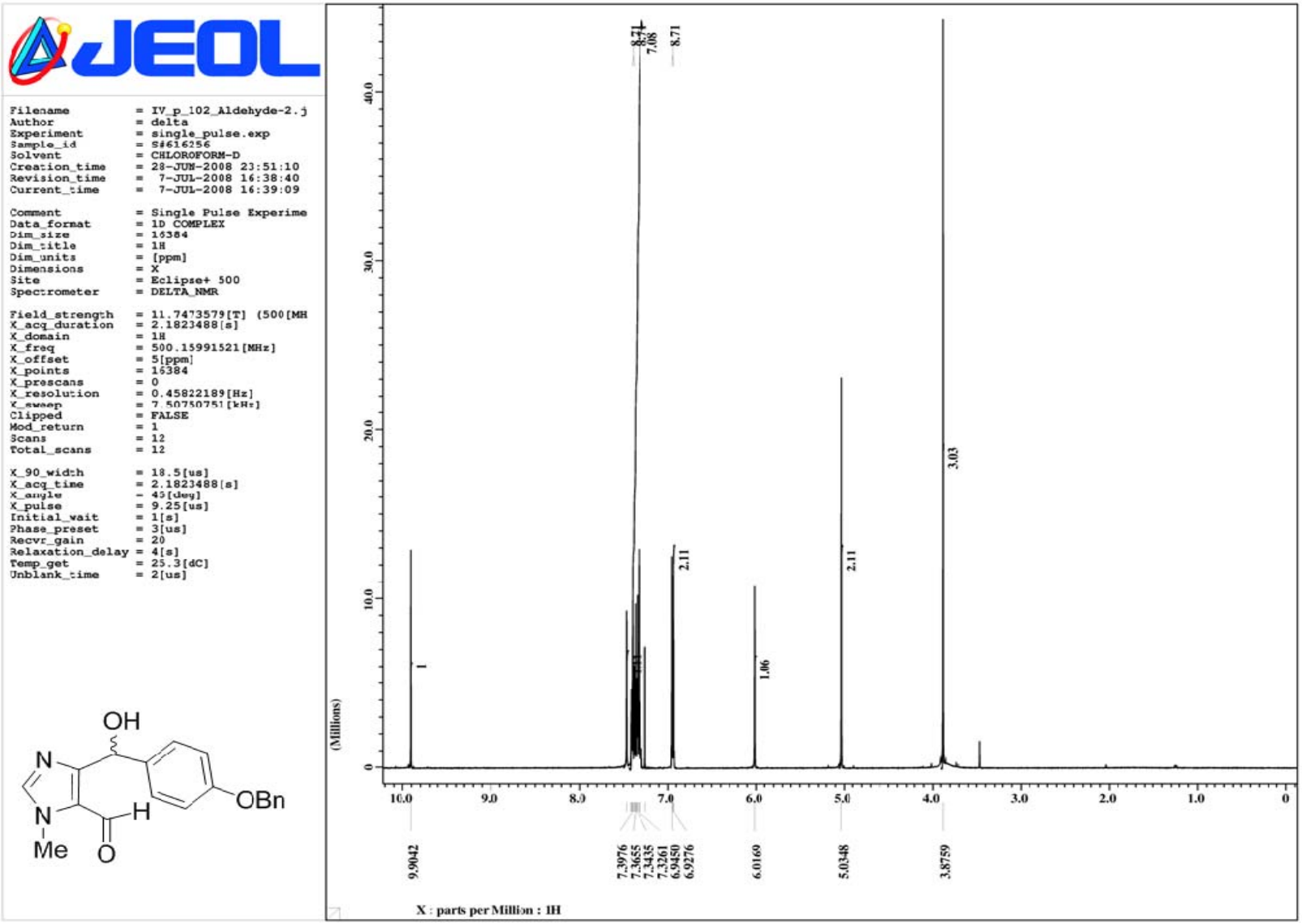

Filename = III_p_103_iii-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#703688
Solvent = CHLOROFORM-D
Creation_time = 21-DEC-2007 00:53:21
Revision_time = 7-JUL-2008 16:08:33
Current_time = 7-JUL-2008 16:09:01
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824054[s]
X_domain = 13C
X_freq = 75.55823426[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 = 3600
Total_scans = 3600
X_90_width = 9.75[us]
X_acq_time = 2.76824054[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 = 4.76824054[s]
Temp_get = 23.5[dC]
N
I
N
O
Me
H
16
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
0.13
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
181.2607
145.0115
130.0443
100.3059
77.5660
77.1405
76.7198
34.5622
X : parts per Million : 13C

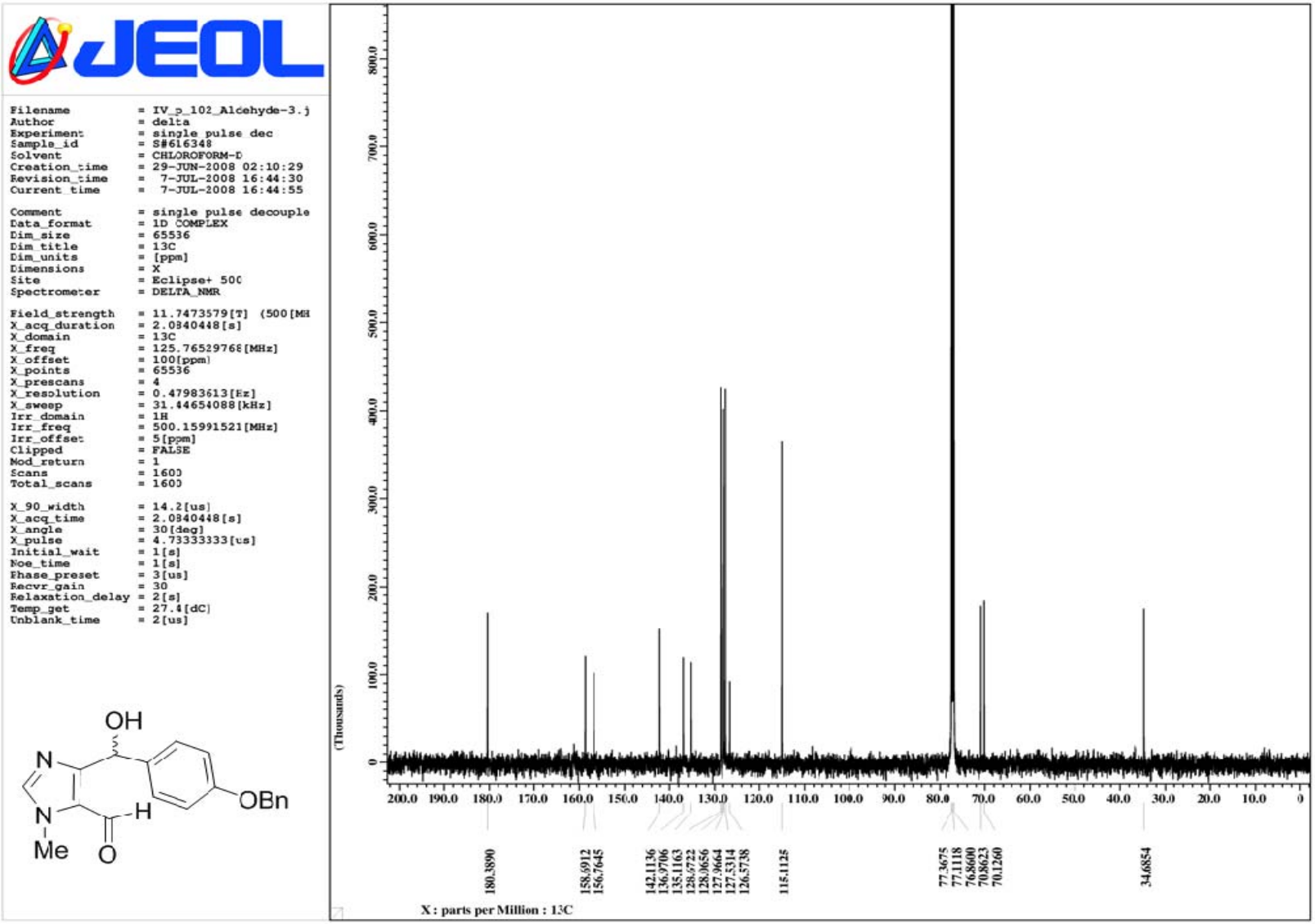

Filename = III_p_109_ii-3.jdf
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#344783
Solvent = CHLOROFORM-D
Creation_time = 28-DEC-2007 10:02:35
Revision_time = 9-JUL-2008 14:34:06
Current_time = 9-JUL-2008 14:34:35
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 46
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 23.1[dC]
N
I
N
O
Me
O
17
abundance
1
1.04
2.97
2.07
2.06
7.3967
7.2284
5.7894
4.1620
4.1538
4.0540
4.0384
4.0301
3.6950
X : parts per Million : 1H

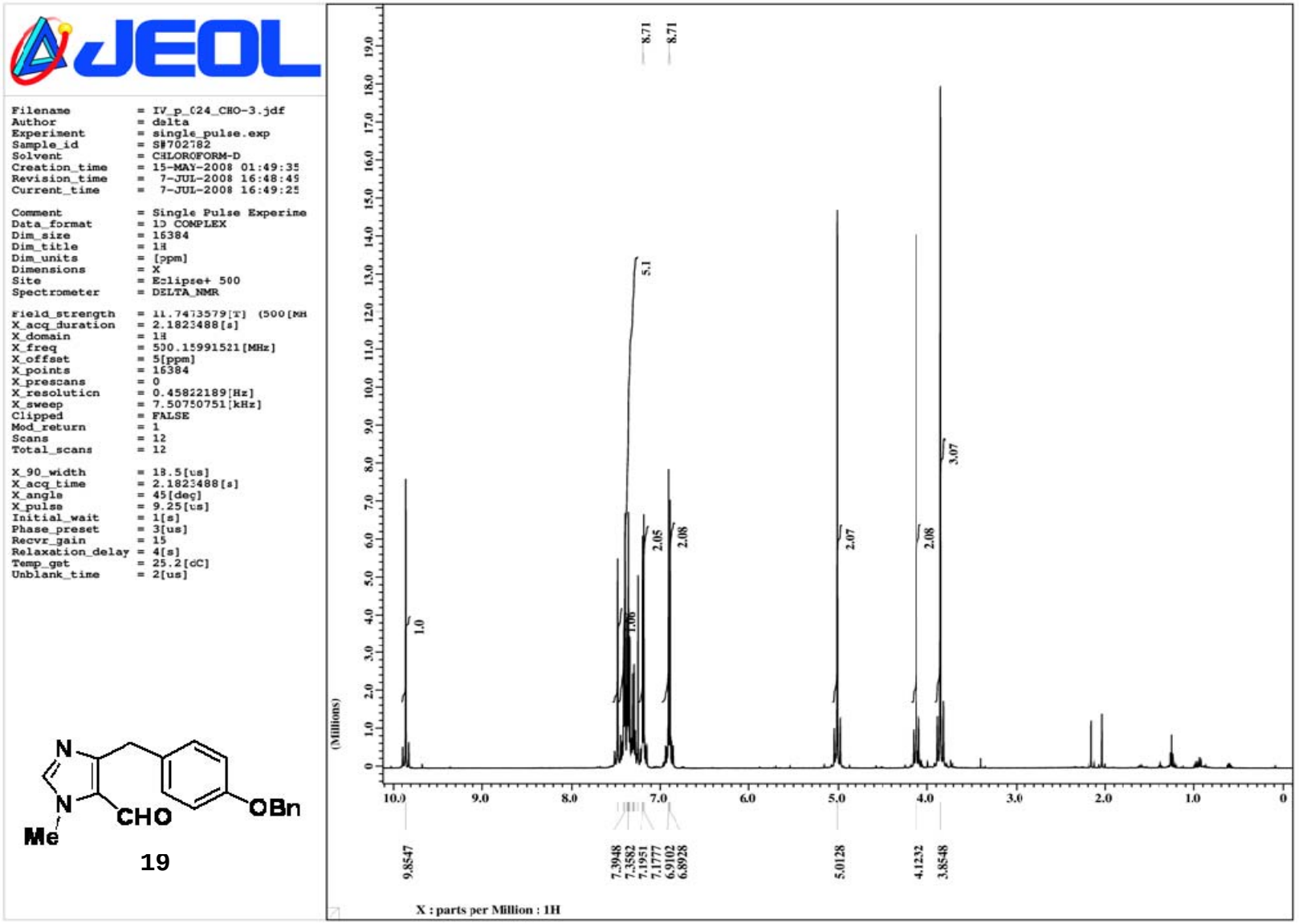

Filename = III_p_109_ii-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#714454
Solvent = CHLOROFORM-D
Creation_time = 29-DEC-2007 04:45:17
Revision_time = 29-DEC-2007 09:17:21
Current_time = 7-JUL-2008 16:18:31
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 6400
Total_scans = 6400
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_set = 23.5[dC]
N
I
N
O
O
Me
17
abundance
0
0.01
0.02
0.03
0.04
0.05
0.06
0.07
0.08
0.09
0.1
0.11
0.12
0.13
0.14
0.15
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
141.8517
127.0375
98.8145
87.7528
77.5564
77.3365
77.1357
76.7103
65.2949
33.1711
X : parts per Million : 13C

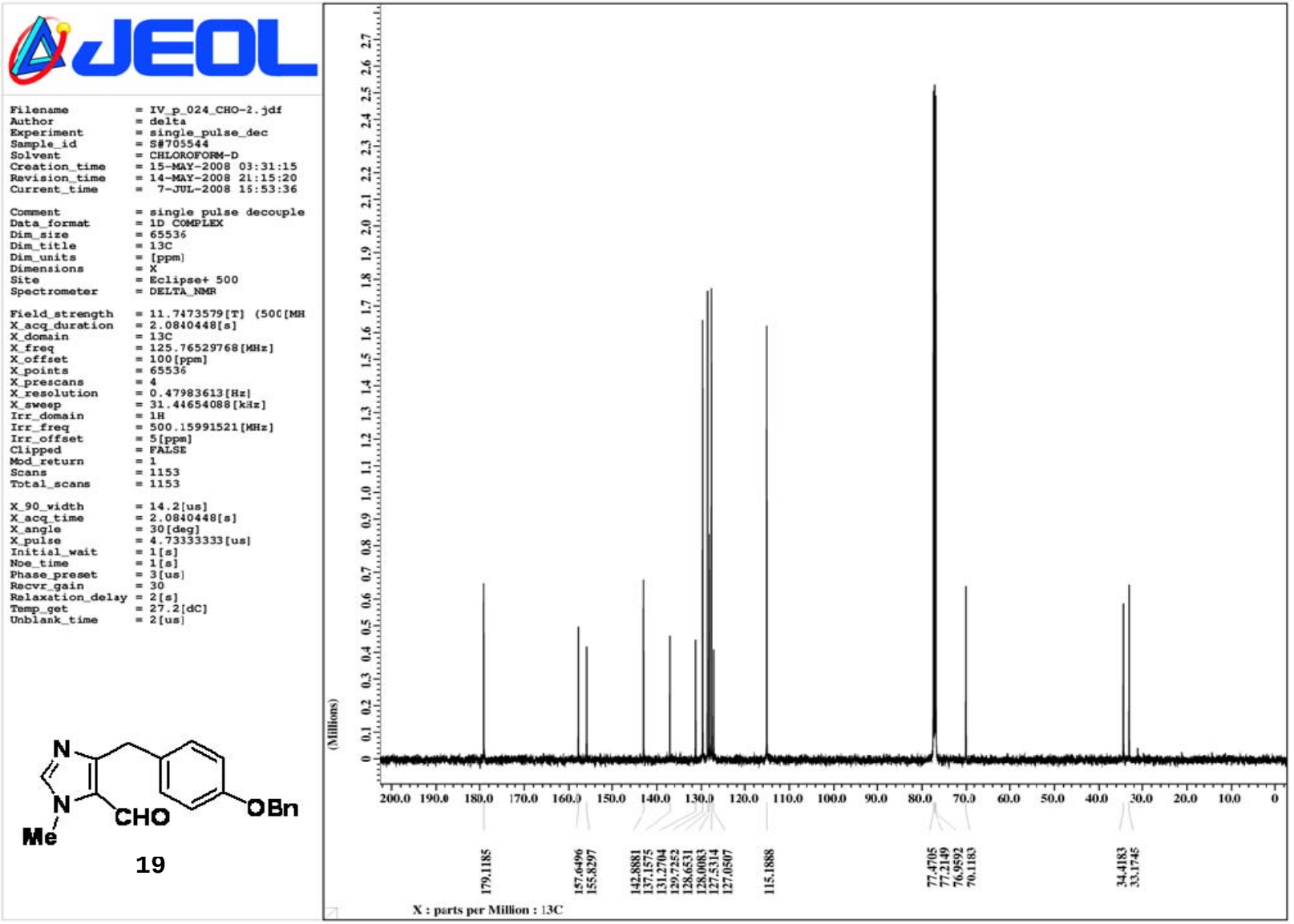

Filename = IV_p_001-4.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#623247
Solvent = CHLOROFORM-D
Creation_time = 30-APR-2008 23:29:23
Revision_time = 7-JUL-2008 16:24:23
Current_time = 7-JUL-2008 16:25:06
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.1823483[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.45822139[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 = 2.1823483[s]
X_angle = 45[deg]
X_pulse = 9.25[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 23
Relaxation_delay = 4[s]
Temp_get = 25.3[dC]
Unblank_time = 2[us]
HO
N
N
Me
O
O
OBn
18
(Millions)
8.2
1.81
2
4.15
3.05
7.3967
7.3737
7.3591
7.3444
7.3096
7.2546
5.8997
5.8447
5.0339
4.0106
3.9382
3.9336
3.9272
3.6643
X : parts per Million : 1H

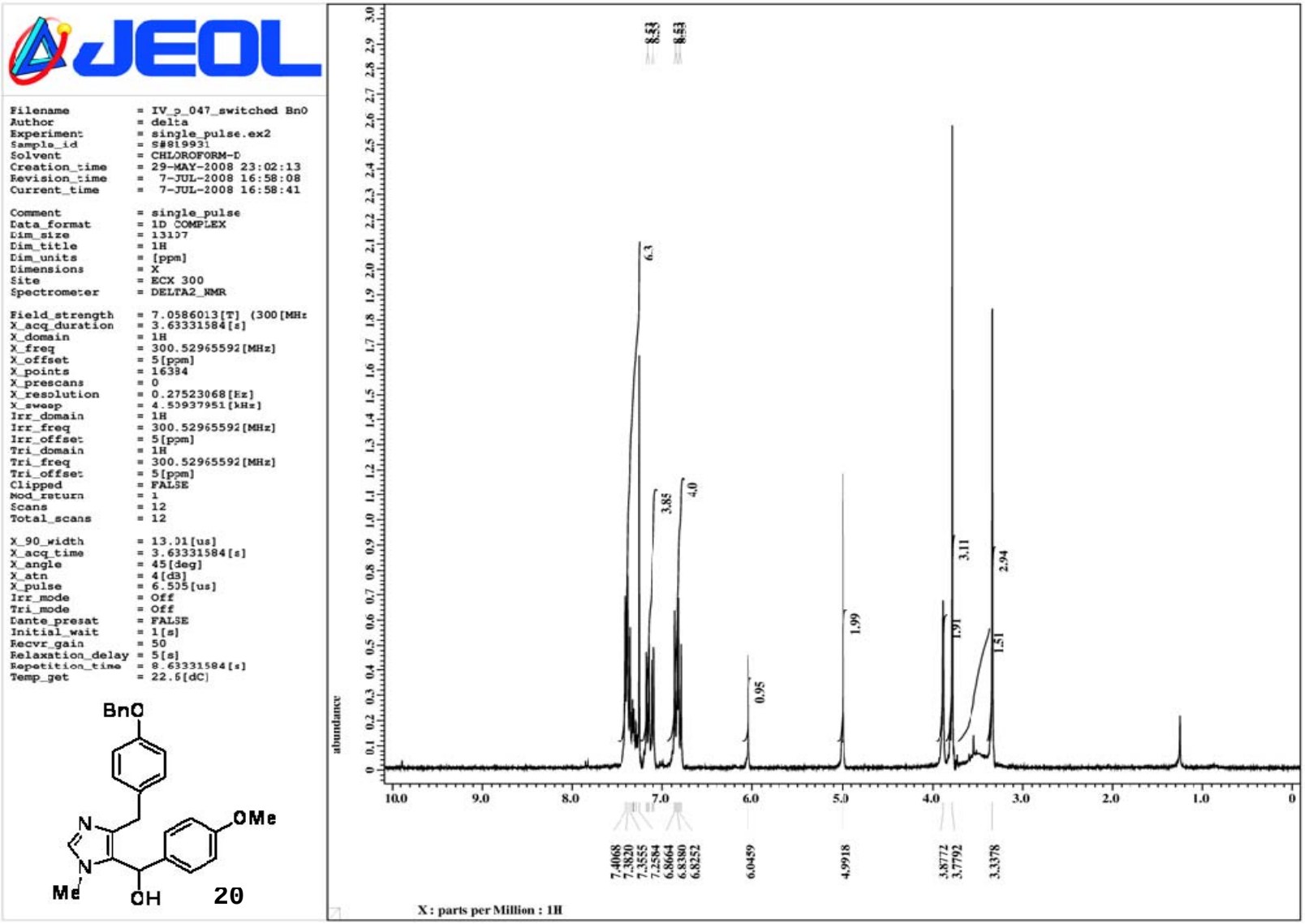

Filename = IV_p_017_Acetal-pure-
Author = delta
Experiment = single pulse dec
Sample_id = S#791229
Solvent = CHLOROFORM-D
Creation_time = 16-JUN-2008 06:23:20
Revision_time = 7-JUL-2008 16:30:09
Current_time = 7-JUL-2008 16:31:49
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 500
Spectrometer = DELTA_NMR
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 2.0840448[s]
X_domain = 13C
X_freq = 125.76529763[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 = FALSE
Mod_return = 1
Scans = 1275
Total_scans = 1275
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 = 26.7[dC]
Urblank_time = 2[us]
HO
N
N
Me
O
O
OBn
18
(Thousands)
300.0
200.0
100.0
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
158.0999
145.1697
138.3439
137.1224
136.3029
128.6188
127.9587
127.3748
127.5238
121.7283
114.7463
97.2911
77.3751
77.1233
76.8677
70.0725
69.5422
65.1164
33.0028
X : parts per Million : 13C


Filename = IV_p_102_Aldehyde-2.j
Author = delta
Experiment = single_pulse.exp
Sample_id = S#616256
Solvent = CHLOROFORM-D
Creation_time = 28-JUN-2008 23:51:10
Revision_time = 7-JUL-2008 16:38:40
Current_time = 7-JUL-2008 16:39:09
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 = 20
Relaxation_delay = 4[s]
Temp_get = 25.3[dC]
Unblank_time = 2[us]
OH
N
N
Me
O
H
OBn
(Millions)
40.0
30.0
20.0
10.0
0
8.71
8.71
7.08
8.71
3.03
2.11
2.11
1.06
1
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.9042
7.3976
7.3655
7.3435
7.3261
6.9450
6.9276
6.0160
5.0348
3.8759
X : parts per Million : 1H

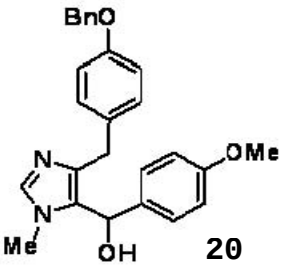

Filename = IV_p_102_Alcehyde-3.j
Author = delta
Experiment = single pulse dec
Sample_id = S#616348
Solvent = CHLOROFORM-D
Creation_time = 29-JUN-2008 02:10:29
Revision_time = 7-JUL-2008 16:44:30
Current_time = 7-JUL-2008 16:44:55
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
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 = FALSE
Mod_return = 1
Scans = 1600
Total_scans = 1600
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 = 27.4[dC]
Unblank_time = 2[us]
OH
N
N
Me
O
H
OBn
(Thousands)
800.0
700.0
600.0
500.0
400.0
300.0
200.0
100.0
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
180.3890
158.6912
156.7645
142.1136
136.9706
135.1163
128.6722
128.0656
127.9664
127.5314
126.5738
115.1125
77.3675
77.1118
76.8600
70.8623
70.1260
34.6854
X : parts per Million : 13C


Filename = IV_p_C24_CHO-3.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#702782
Solvent = CHLOROFORM-D
Creation_time = 15-MAY-2008 01:49:35
Revision_time = 7-JUL-2008 16:48:45
Current_time = 7-JUL-2008 16:49:25
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 = 25.2[dC]
Unblank_time = 2[us]
N
N
Me
CHO
OBn
19
8.71
8.71
5.1
2.05
2.08
2.07
2.08
3.07
1.0
1.06
(Millions)
9.8547
7.3948
7.3582
7.1951
7.1777
6.9102
6.8928
5.0128
4.1232
3.8548
X : parts per Million : 1H

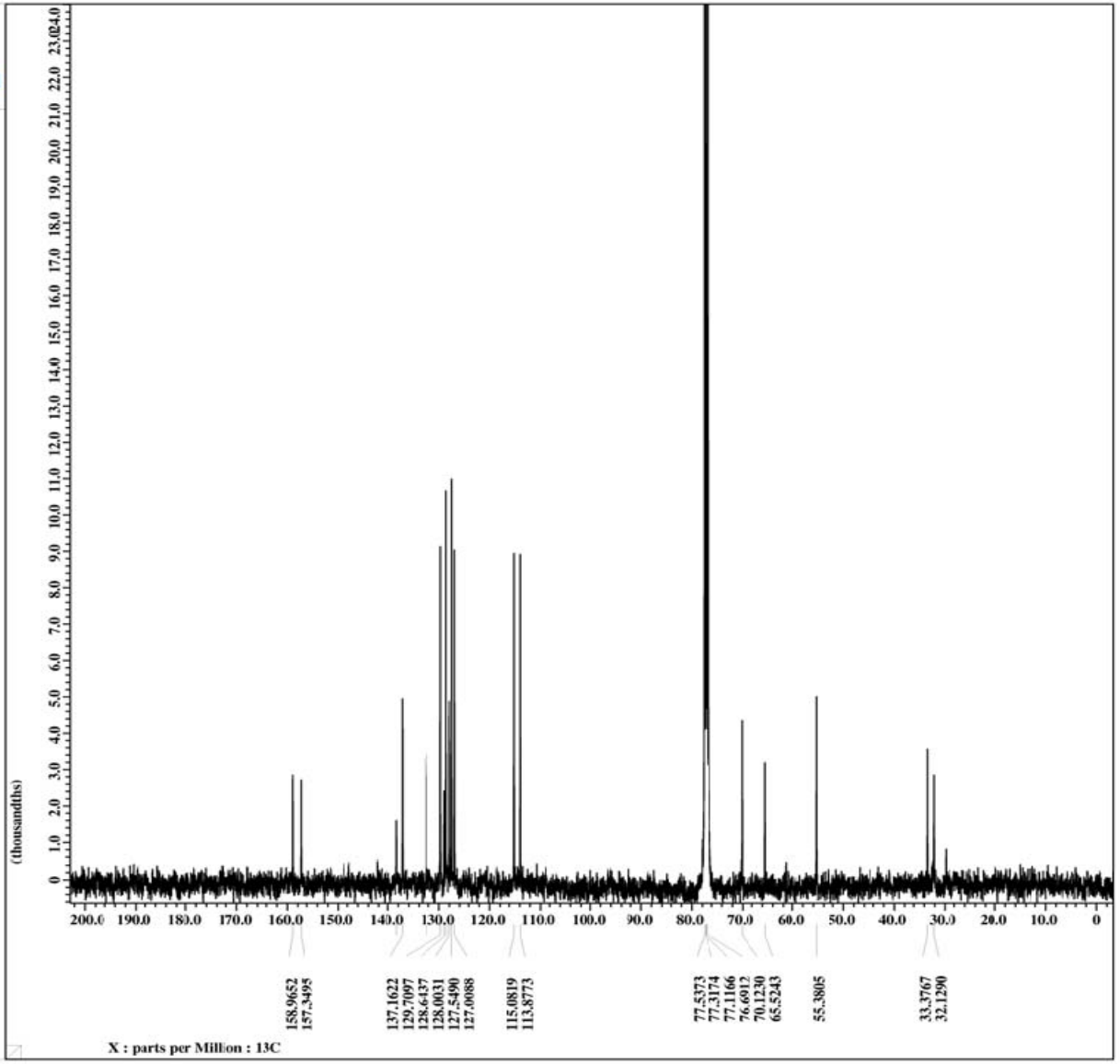

Filename = IV_p_024_CHO-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#705544
Solvent = CHLOROFORM-D
Creation_time = 15-MAY-2008 03:31:15
Revision_time = 14-MAY-2008 21:15:20
Current_time = 7-JUL-2008 15:53:36
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
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 = FALSE
Mod_return = 1
Scans = 1153
Total_scans = 1153
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 = 27.2[dC]
Unblank_time = 2[us]
N
N
CHO
OBn
Me
19
(Millions)
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0 2.1 2.2 2.3 2.4 2.5 2.6 2.7
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
179.1185
157.6496
155.8297
142.8881
137.1575
131.2704
129.7252
128.6531
128.0083
127.5314
127.0507
115.1888
77.4705
77.2149
76.9592
70.1183
34.4183
33.1745
X : parts per Million : 13C

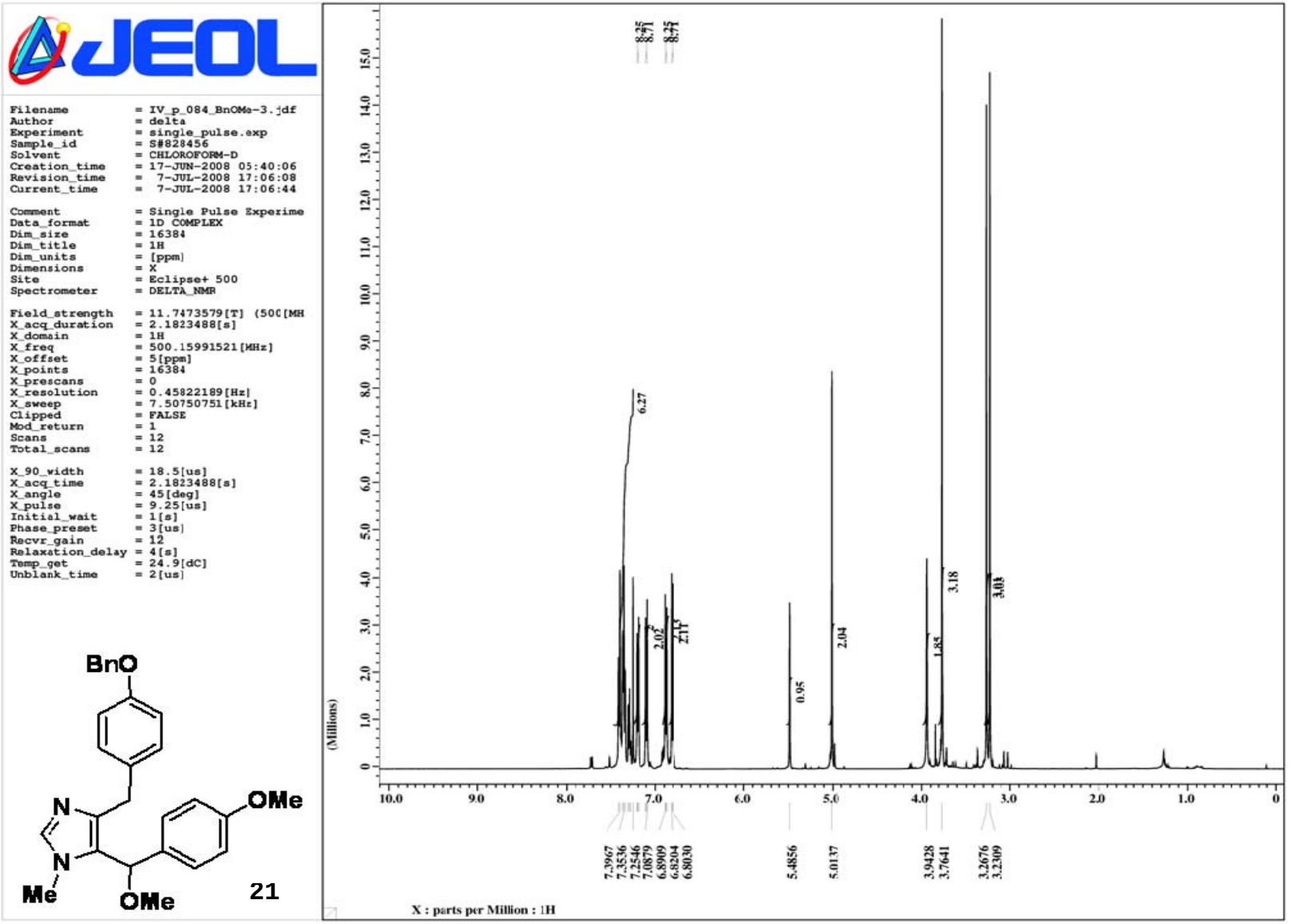

Filename = IV_p_047_switched BnO
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#819931
Solvent = CHLOROFORM-D
Creation_time = 29-MAY-2008 23:02:13
Revision_time = 7-JUL-2008 16:58:08
Current_time = 7-JUL-2008 16:58:41
Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 13107
Dim_title = 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 = 50
Relaxation_delay = 5[s]
Repetition_time = 8.63331584[s]
Temp_get = 22.5[dC]
BnO
OMe
N
N
Me
OH
20
abundance
6.3
3.85
4.0
0.95
1.99
1.91
3.11
2.94
1.51
7.4068
7.3820
7.3555
7.2584
6.8664
6.8380
6.8252
6.0459
4.9918
3.8772
3.7792
3.3378
X : parts per Million : 1H

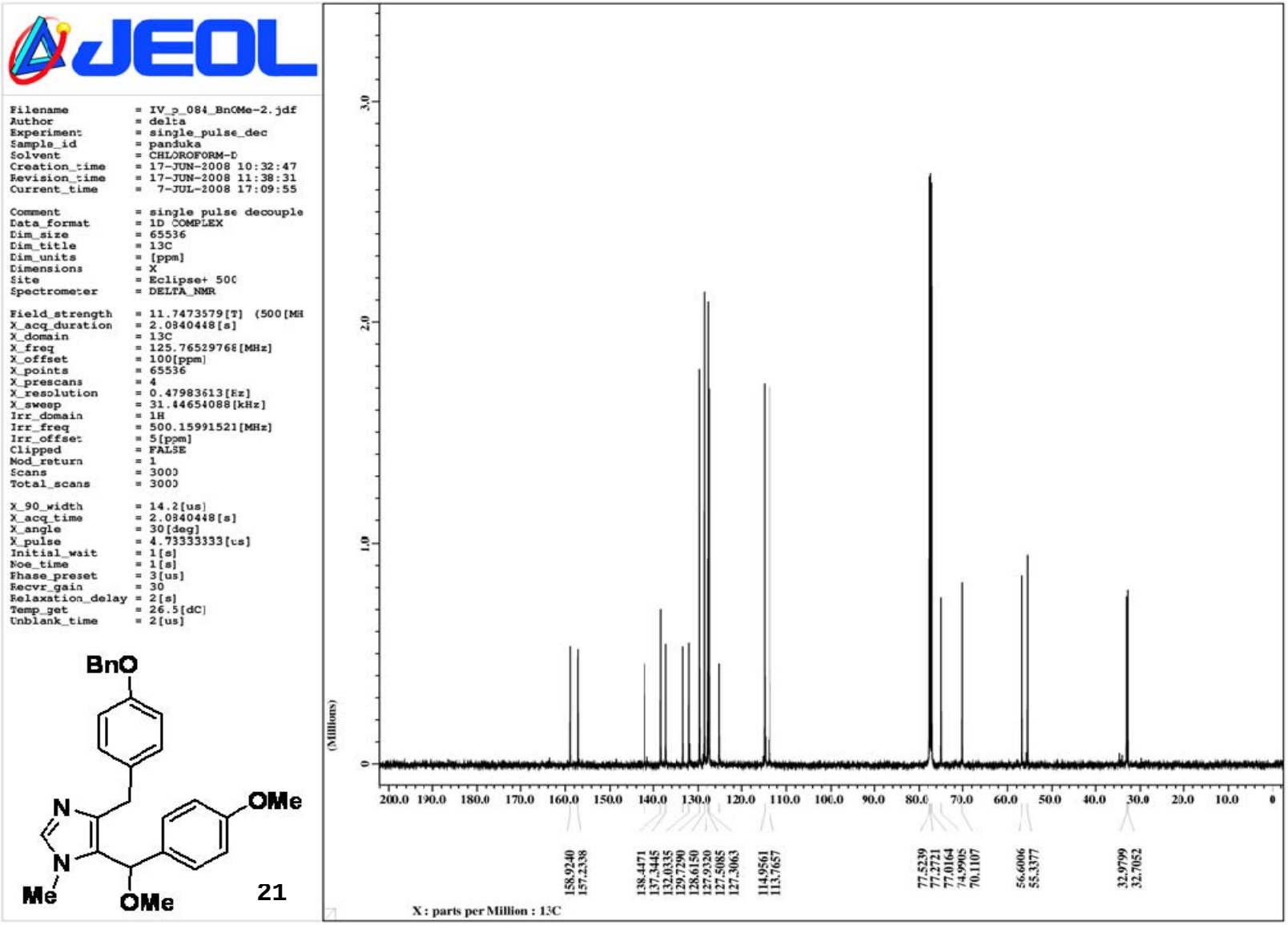
```
Filename          = IV_p_047_swithced BnO
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#821236
Solvent           = CHLOROFORM-D
Creation_time     = 30-MAY-2008 07:32:33
Revision_time     = 30-MAY-2008 09:46:59
Current_time      =  7-JUL-2008 17:01:35

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             = 6400
Total_scans       = 6400

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          = 24.5[dC]
```

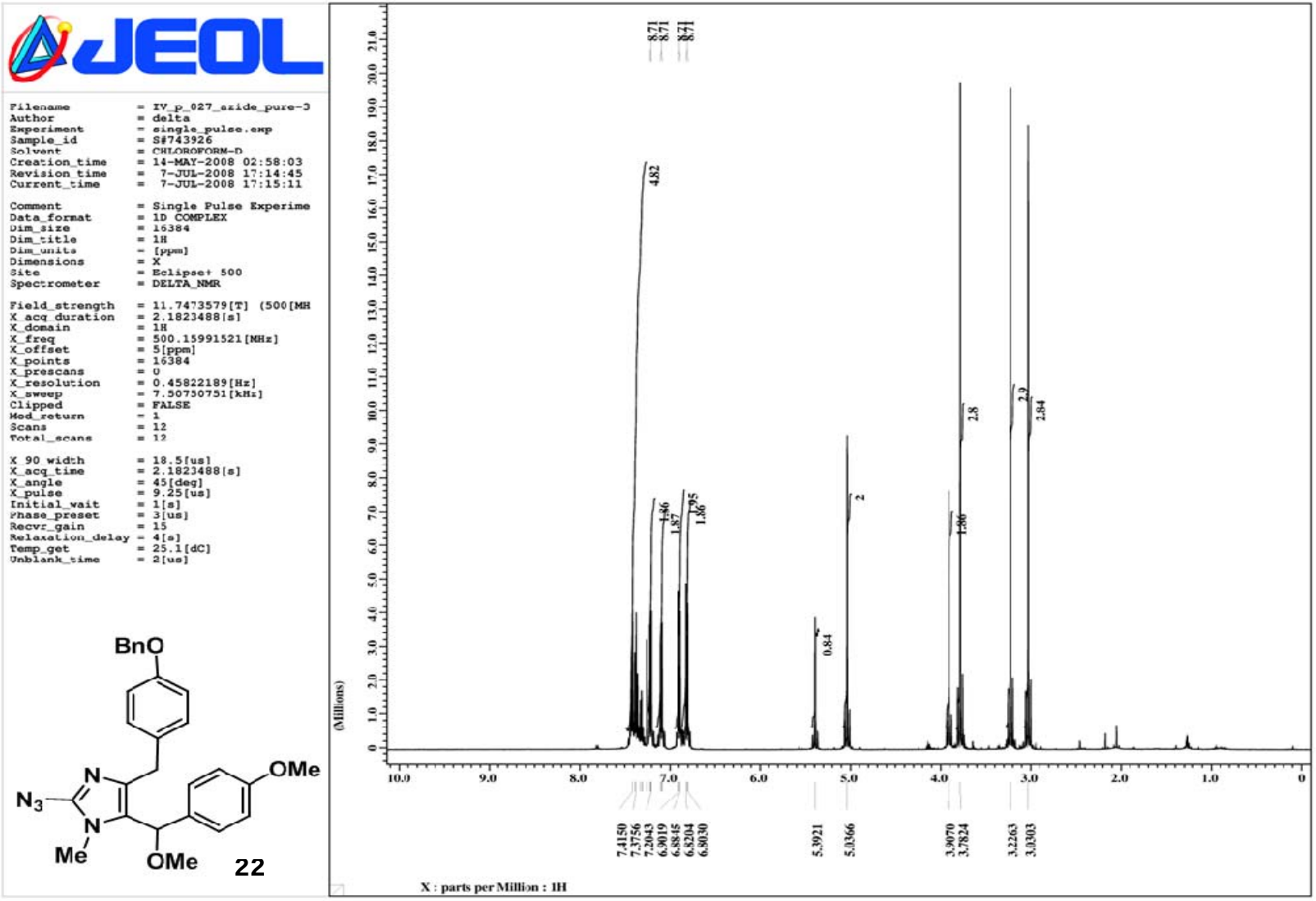


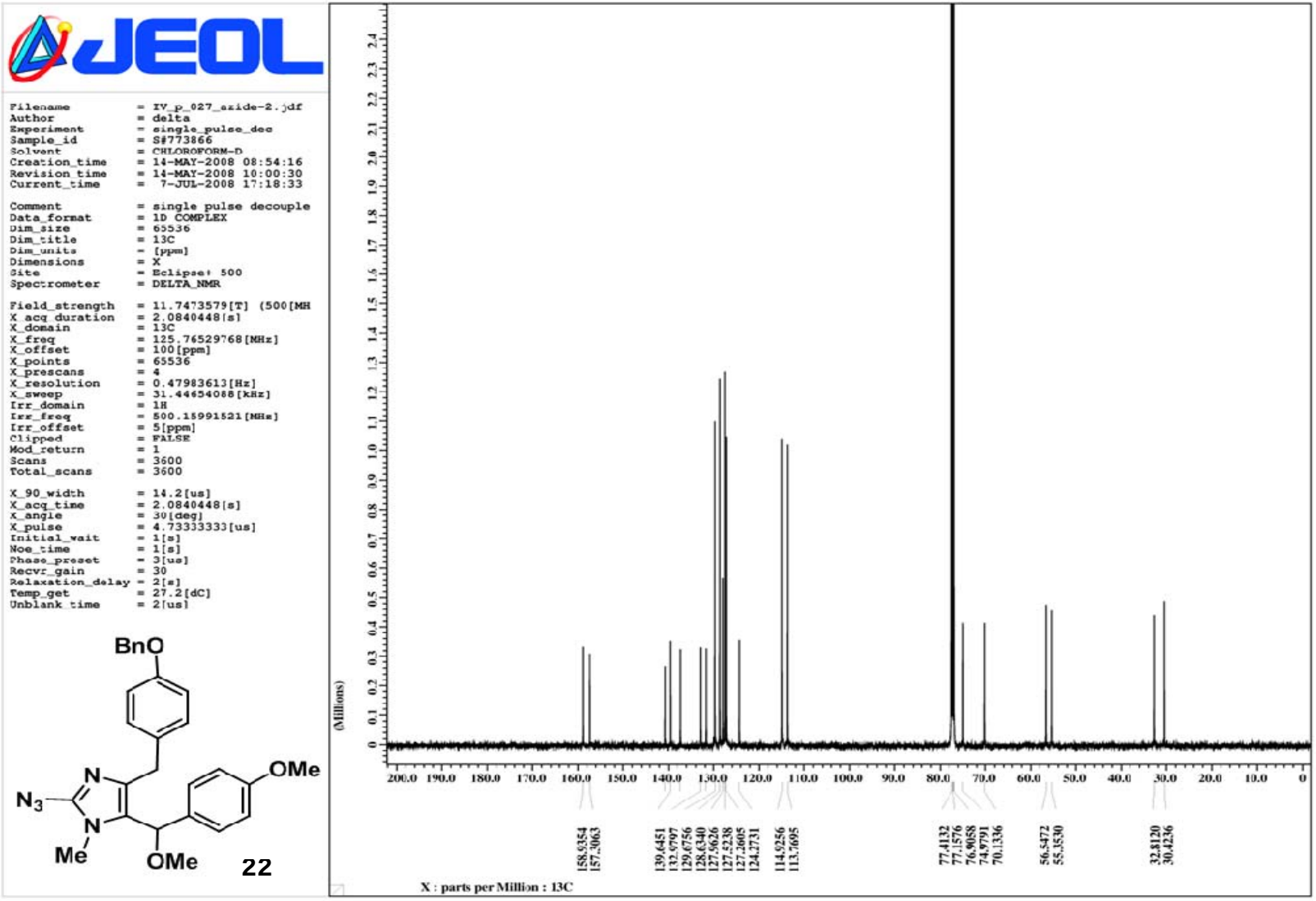
20

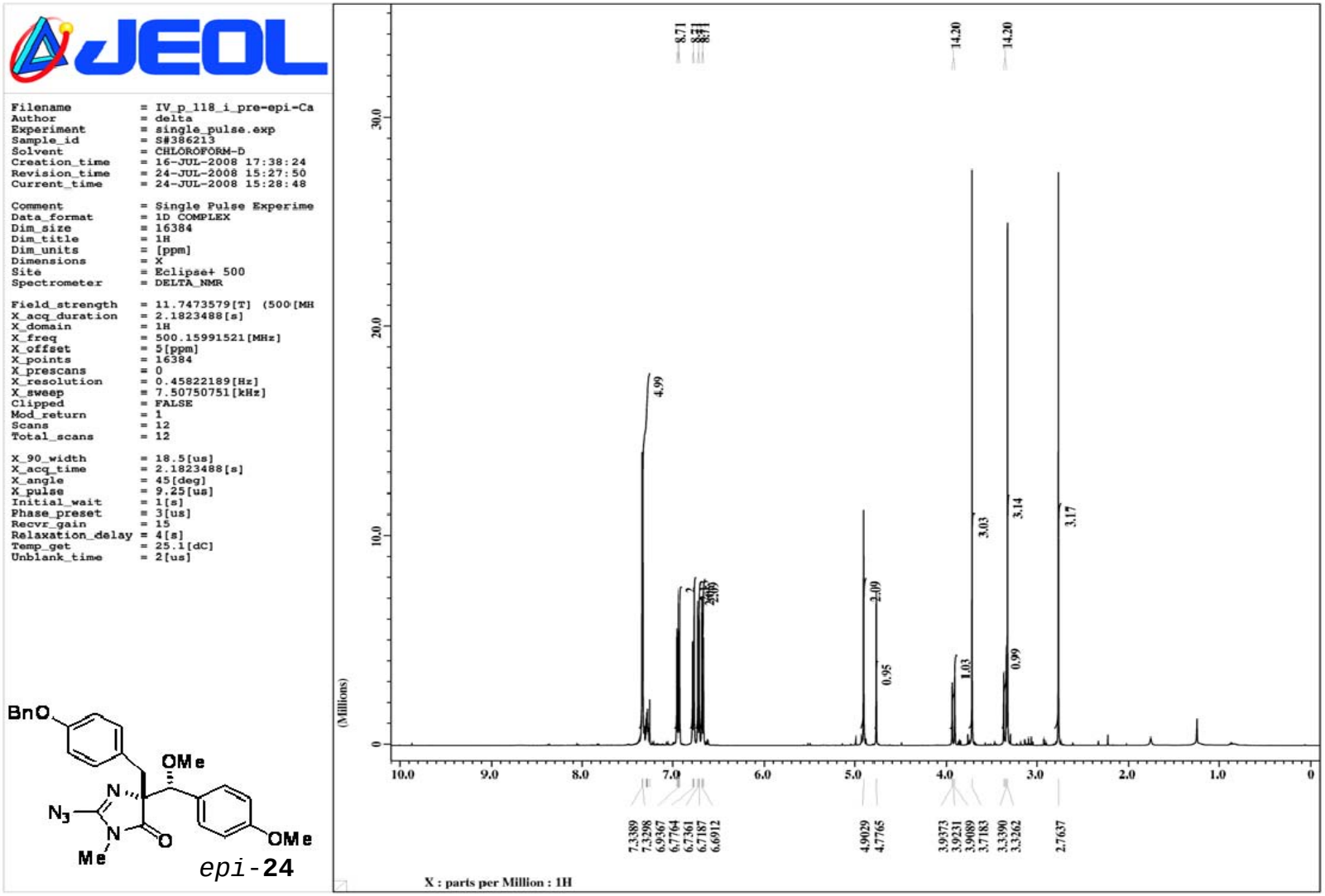


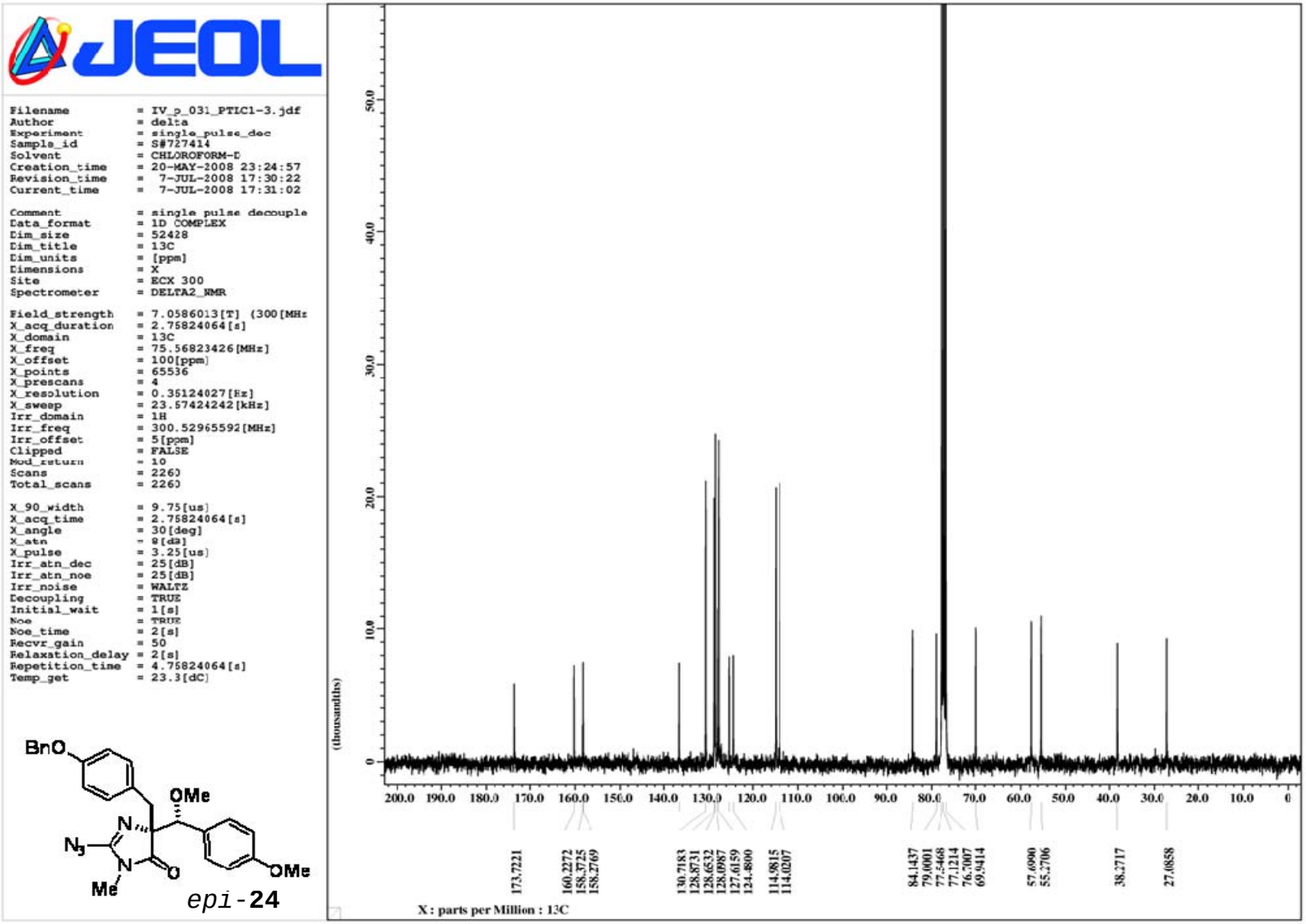

Filename = IV_p_084_BnOMe-3.jdf
Author = delta
Experiment = single_pulse.exp
Sample_id = S#828456
Solvent = CHLOROFORM-D
Creation_time = 17-JUN-2008 05:40:06
Revision_time = 7-JUL-2008 17:06:08
Current_time = 7-JUL-2008 17:06:44
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
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 = 24.9[dC]
Unblank_time = 2[us]
BnO
N
N
Me
OMe
OMe
21
(Millions)
6.27
2.02
2.11
0.95
2.04
1.85
3.18
3.03
7.3967
7.3536
7.2546
7.0879
6.8909
6.8204
6.8030
5.4856
5.0137
3.9428
3.7641
3.2676
3.2309
X : parts per Million : 1H

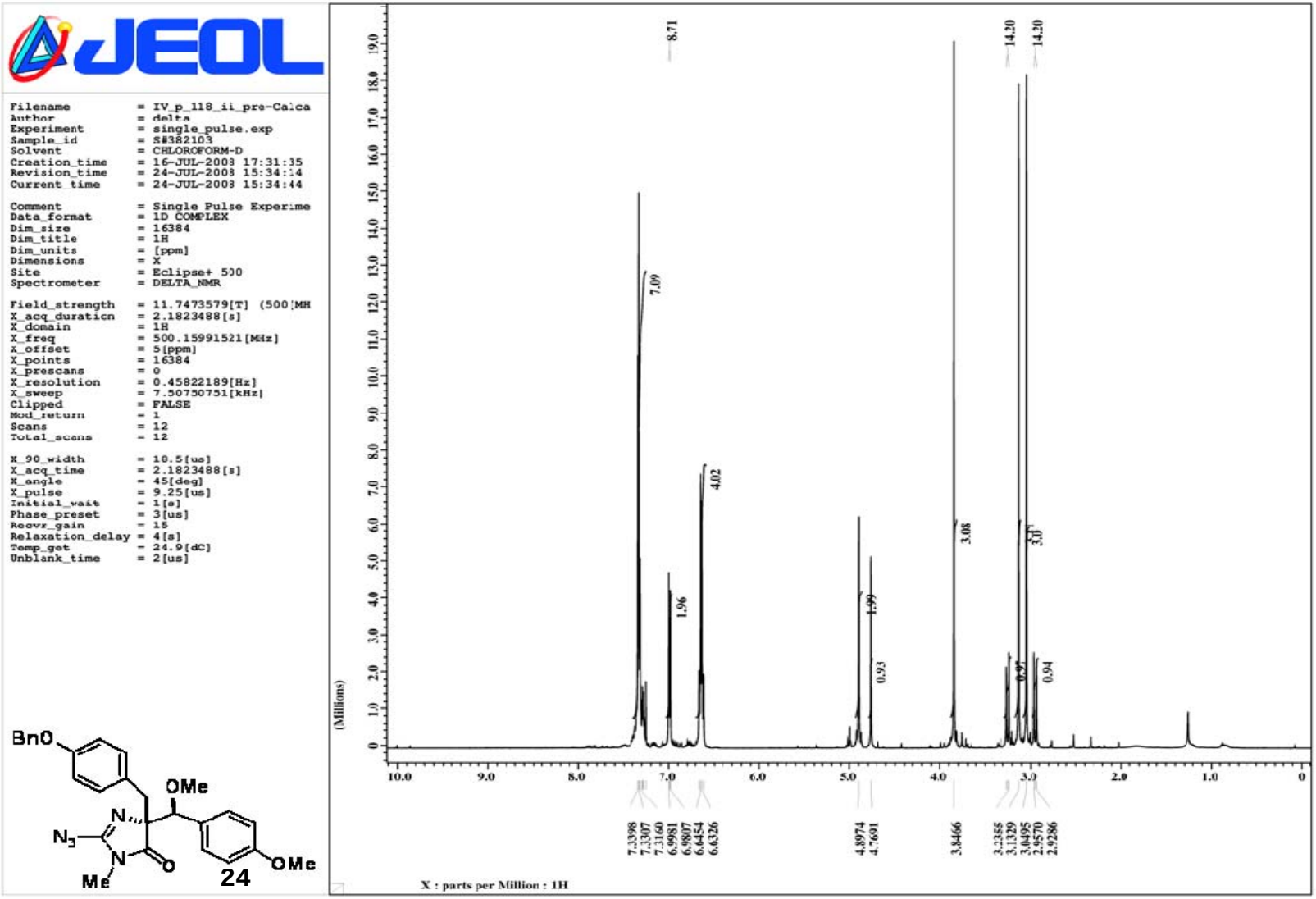

Filename = IV_p_084_BnOMe-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = panduka
Solvent = CHLOROFORM-D
Creation_time = 17-JUN-2008 10:32:47
Revision_time = 17-JUN-2008 11:38:31
Current_time = 7-JUL-2008 17:09:55
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
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 = FALSE
Nod_return = 1
Scans = 3000
Total_scans = 3000
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 = 26.5[dC]
Unblank_time = 2[us]
BnO
N
N
Me
OMe
OMe
21
(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
158.9240
157.2338
138.4471
137.3445
132.0335
129.7290
128.6150
127.9330
127.5085
127.3063
114.9561
113.7657
77.5239
77.2721
77.0164
74.9905
70.1107
56.6006
55.3377
33.9799
32.7052
X : parts per Million : 13C

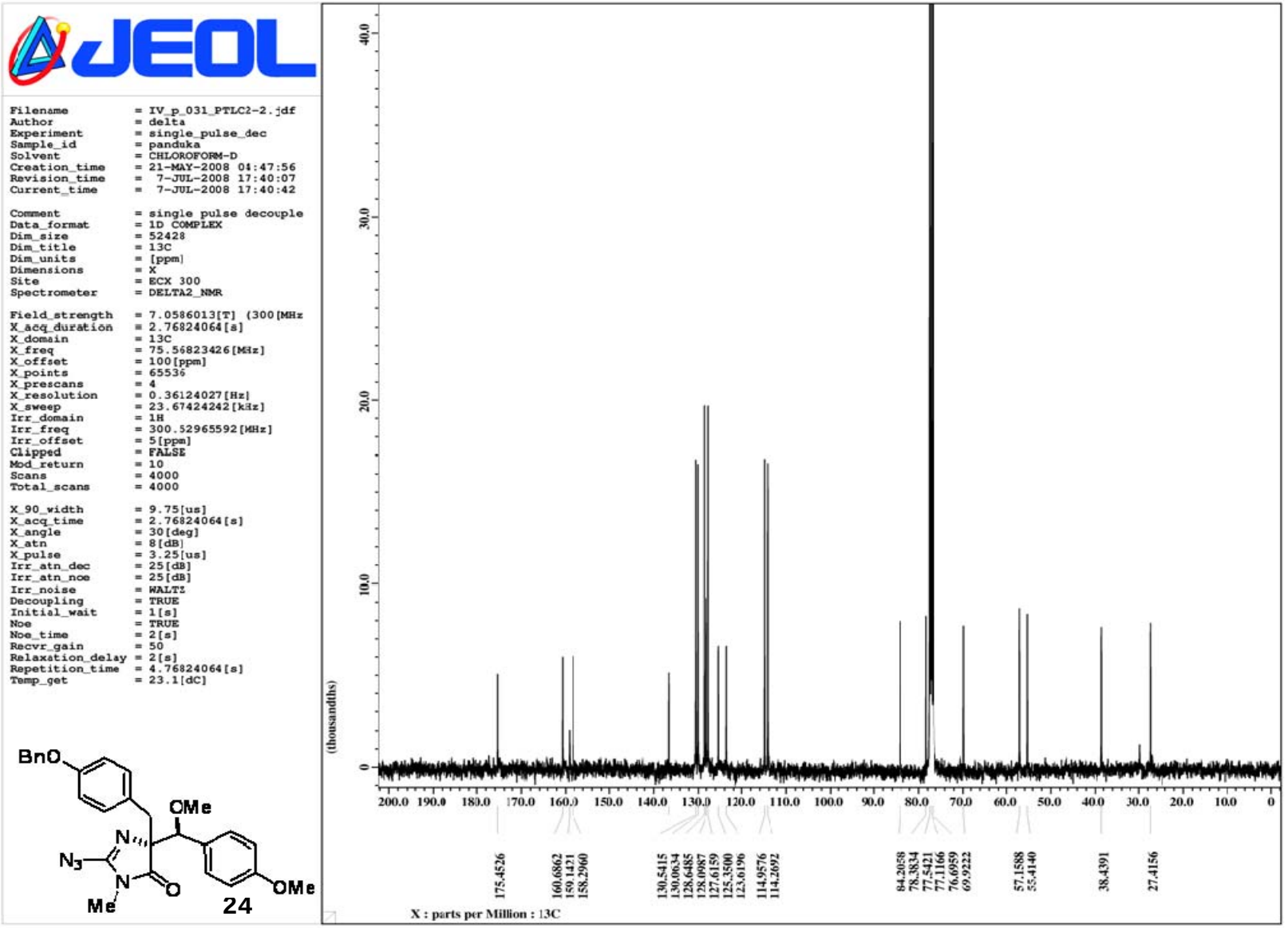

Filename = IV_p_027_azide_pure-3
Author = delta
Experiment = single_pulse.exp
Sample_id = S#743926
Solvent = CHLOROFORM-D
Creation_time = 14-MAY-2008 02:58:03
Revision_time = 7-JUL-2008 17:14:45
Current_time = 7-JUL-2008 17:15:11
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
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 = 25.1[dC]
Unblank_time = 2[us]
BnO
N
N3
N
Me
OMe
OMe
22
(Millions)
8.71
8.71
8.71
8.71
4.82
1.86
1.87
1.95
1.86
0.84
2
1.80
2.8
2.9
2.84
7.4150
7.3756
7.2043
6.9019
6.8845
6.8204
6.8030
5.3921
5.3366
3.9070
3.7824
3.2263
3.0303
X : parts per Million : 1H

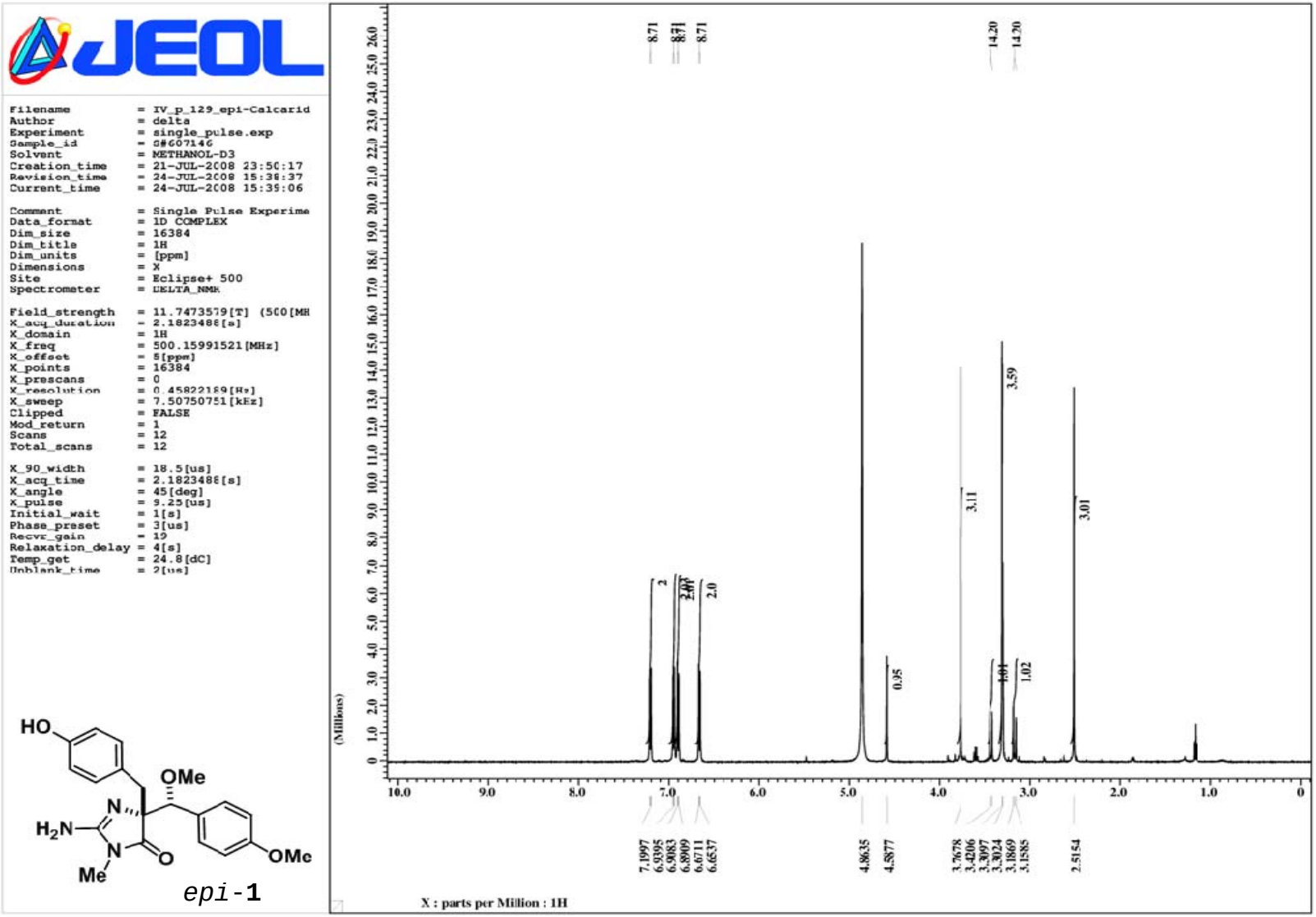

Filename = IV_p_027_azide-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#773866
Solvent = CHLOROFORM-D
Creation_time = 14-MAY-2008 08:54:16
Revision_time = 14-MAY-2008 10:00:30
Current_time = 7-JUL-2008 17:18:33
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 65536
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 = FALSE
Mod_return = 1
Scans = 3600
Total_scans = 3600
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 = 27.2[dC]
Unblank_time = 2[us]
BnO
N
N3
N
Me
OMe
OMe
22
(Millions)
158.5354
157.3063
139.6451
132.9797
129.6756
128.6340
127.9626
127.5238
127.2605
124.2731
114.9256
113.7695
77.4132
77.1576
76.9058
74.8791
70.1336
56.5472
55.3530
32.8120
30.4236
X : parts per Million : 13C

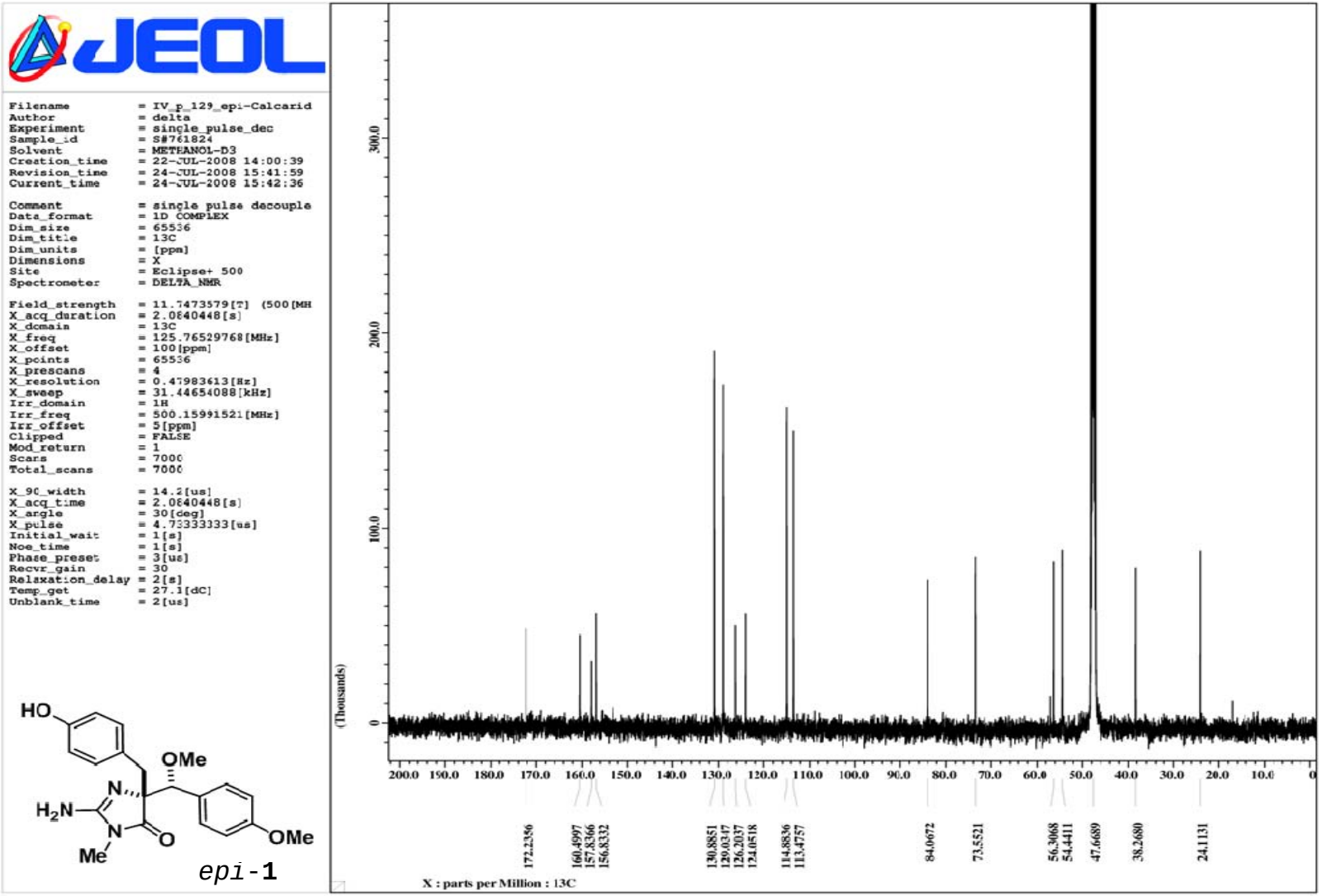

Filename = IV_p_118_i_pre-epi-Ca
Author = delta
Experiment = single_pulse.exp
Sample_id = S#386213
Solvent = CHLOROFORM-D
Creation_time = 16-JUL-2008 17:38:24
Revision_time = 24-JUL-2008 15:27:50
Current_time = 24-JUL-2008 15:28:48
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
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 = 25.1[dC]
Unblank_time = 2[us]
BnO
OMe
N
N3
N
O
OMe
Me
epi-24
(Millions)
30.0
20.0
10.0
0
8.71
14.20
14.20
4.99
3.03
3.14
3.17
2.09
0.95
1.03
0.99
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.3389
7.3298
6.9367
6.7764
6.7361
6.7187
6.6912
4.9029
4.7765
3.9373
3.9231
3.9089
3.7183
3.3390
3.3262
2.7637
X : parts per Million : 1H

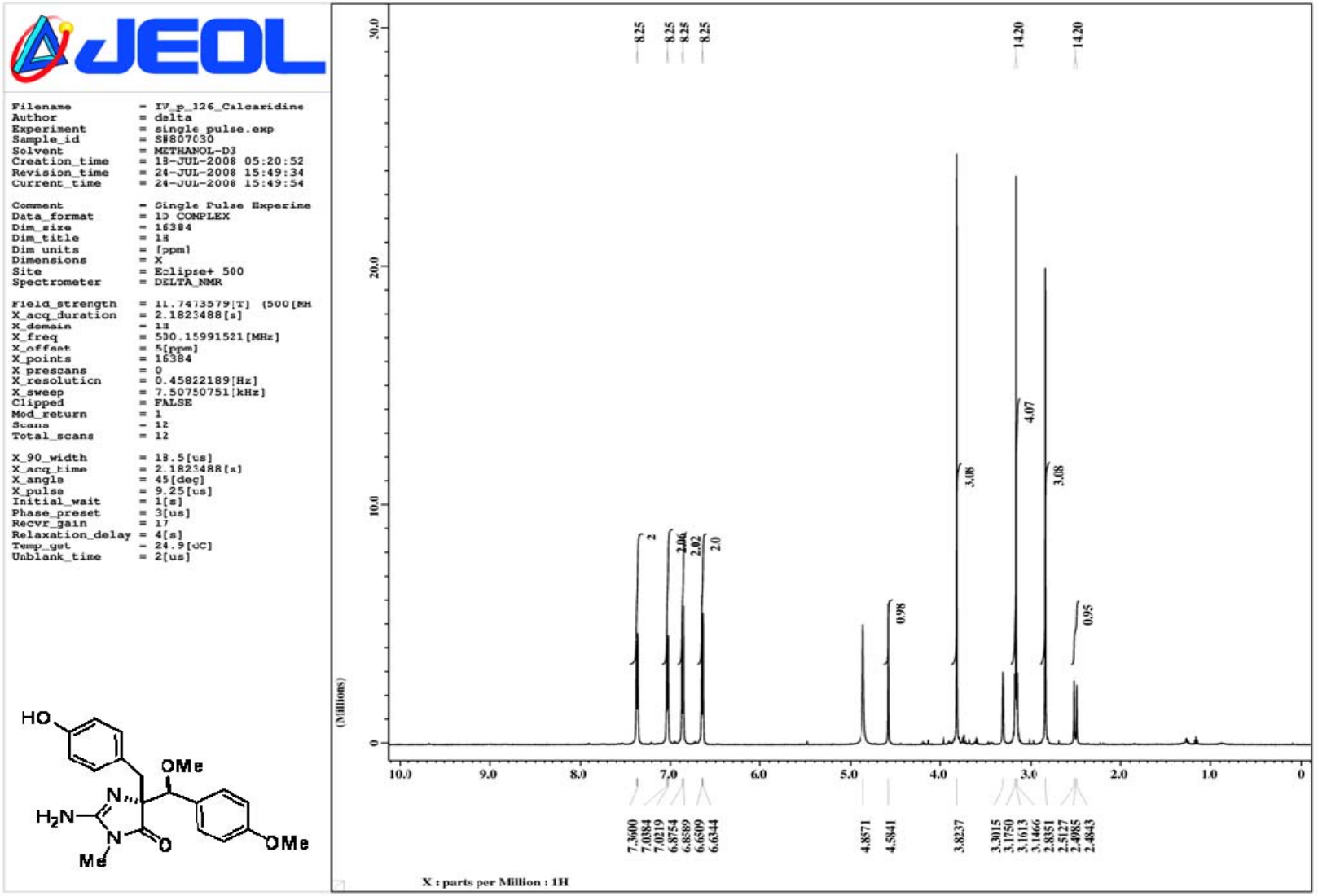

Filename = IV_p_031_PTIC1-3.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = S#727414
Solvent = CHLOROFORM-D
Creation_time = 20-MAY-2008 23:24:57
Revision_time = 7-JUL-2008 17:30:22
Current_time = 7-JUL-2008 17:31:02
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.75824064[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 = 23.57424242[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 2260
Total_scans = 2260
X_90_width = 9.75[us]
X_acq_time = 2.75824064[s]
X_angle = 30[deg]
X_atn = 9[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 = 4.75824064[s]
Temp_get = 23.3[dC]
BnO
OMe
N
N3
N
O
Me
OMe
epi-24
(thousandths)
50.0
40.0
30.0
20.0
10.0
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
173.7221
160.2272
158.3725
158.2769
130.7183
128.8731
128.6532
128.0987
127.6159
124.4800
114.9815
114.0207
84.1437
79.0001
77.5468
77.1214
76.7007
69.9414
57.6990
55.2706
38.2717
27.0858
X : parts per Million : 13C


Filename = IV_p_118_ii_pre-Calca
Author = delta
Experiment = single_pulse.exp
Sample_id = S#382103
Solvent = CHLOROFORM-D
Creation_time = 16-JUL-2003 17:31:35
Revision_time = 24-JUL-2003 15:34:14
Current time = 24-JUL-2003 15:34:44
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 = 10.5[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 = 24.9[dC]
Unblank_time = 2[us]
BnO
OMe
N
N3
N
Me
O
OMe
24
(Millions)
8.71
7.09
4.02
1.96
1.99
0.93
3.08
14.20
14.20
3.01
0.97
0.94
7.3398
7.3307
7.3160
6.9981
6.9807
6.6454
6.6326
4.8974
4.7691
3.8466
3.2355
3.1329
3.0495
2.9570
2.9286
X : parts per Million : 1H

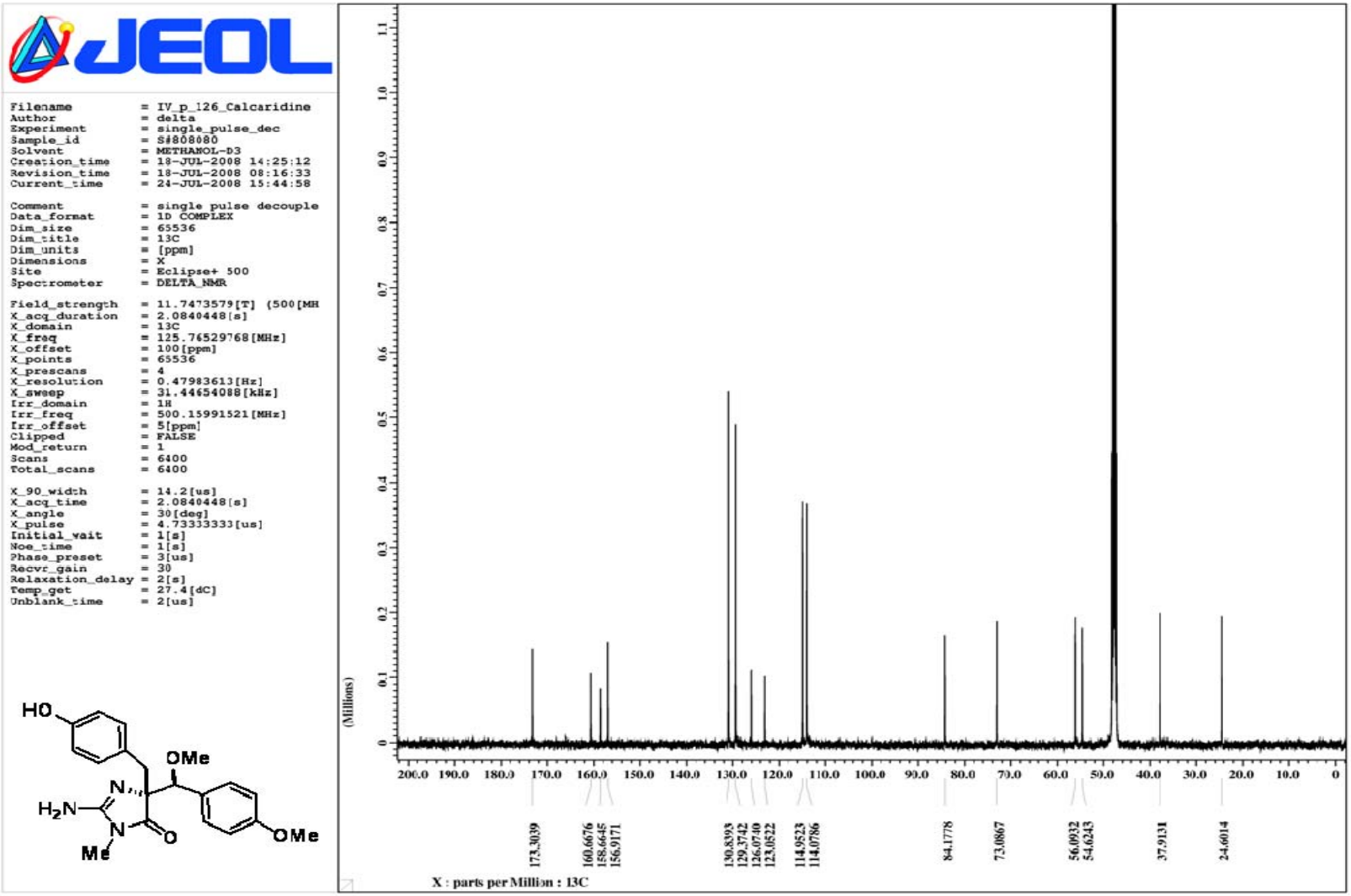

Filename = IV_p_031_PTLC2-2.jdf
Author = delta
Experiment = single_pulse_dec
Sample_id = panduka
Solvent = CHLOROFORM-D
Creation_time = 21-MAY-2008 04:47:56
Revision_time = 7-JUL-2008 17:40:07
Current_time = 7-JUL-2008 17:40:42
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
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 = 4000
Total_scans = 4000
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 = 23.1[dC]
BnO
OMe
N
N3
N
O
Me
OMe
24
(thousandths)
40.0
30.0
20.0
10.0
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
175.4526
160.6862
159.1421
158.2960
130.5415
130.0634
128.6485
128.0987
127.6159
125.3500
123.6196
114.9576
114.2692
84.2058
78.3834
77.5421
77.1166
76.6959
69.9222
57.1588
55.4140
38.4391
27.4156
X : parts per Million : 13C


Filename = IV_p_129_epi-Calcarid
Author = delta
Experiment = single_pulse.exp
Sample_id = d#607146
Solvent = METHANOL-D3
Creation_time = 21-JUL-2008 23:50:17
Revision_time = 24-JUL-2008 15:38:37
Current_time = 24-JUL-2008 15:39:06
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 = 19
Relaxation_delay = 4[s]
Temp_get = 24.8[dC]
Unblank_time = 2[us]
HO
OMe
N
H2N
N
O
OMe
Me
epi-1
(Millions)
8.71
8.71
8.71
8.71
14.20
14.20
3.59
3.11
3.01
2.0
0.95
1.02
7.1997
6.5395
6.5083
6.8909
6.6711
6.6537
4.8635
4.8877
3.7678
3.4206
3.3097
3.3024
3.1869
3.1585
2.5154
X : parts per Million : 1H


Filename = IV_p_129_epi-Calcarid
Author = delta
Experiment = single_pulse_dec
Sample_id = S#761824
Solvent = METHANOL-D3
Creation_time = 22-JUL-2008 14:00:39
Revision_time = 24-JUL-2008 15:41:59
Current_time = 24-JUL-2008 15:42:36
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 = FALSE
Mod_return = 1
Scans = 7000
Total_scans = 7000
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 = 27.1[dC]
Unblank_time = 2[us]
HO
OMe
N
H2N
N
O
OMe
Me
epi-1
(Thousands)
300.0
200.0
100.0
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
172.2356
160.4997
157.8366
156.8332
130.8851
129.0347
126.2037
124.0518
114.8836
113.4757
84.0672
73.5521
56.3068
54.4411
47.6689
38.2680
24.1131
X : parts per Million : 13C



Calcaridine (**1**)



Calcaridine (**1**)