Supporting Information

Diversity-Oriented Approach to Pyrrole-imidazole Alkaloid Frameworks

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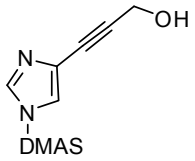
General Considerations: All reagents were purchased from commercial suppliers and were used as received unless otherwise noted. All reactions involving air- or water-sensitive compounds were conducted in oven-dried glassware under an atmosphere of dry argon or nitrogen. ^{1}H and ^{13}C NMR (δ in ppm) spectra were recorded in $CDCl_3$ (unless otherwise noted) at 500 and 125 MHz, respectively; using a JEOL Eclipse+ 500 spectrometer unless otherwise noted using residual $CHCl_3$ as reference. Infrared spectra were recorded either as solids or films for oil or liquids using Bruker Alpha spectrometer (ATR spectroscopy). High resolution mass spectra (HR-MS) were obtained by Dr. Powell through the mass spectrometry service at the University of Florida, Gainesville, Florida.

4-(3-Hydroxyprop-1-ynyl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (S1):



N_2 was bubbled through solution of 4-iodoimidazole (8.00 g, 26.4 mmol), propargyl alcohol (2.22 g, 39.6 mmol), $Pd(PPh_3)_2Cl_2$ (463 mg, 0.66 mmol), CuI (251 mg, 1.32 mmol) in THF/TEA (120 mL/120 mL). The reaction mixture was heated at 60 ºC for overnight. The reaction mixture was concentrated, the residue was preabsorbed on silica gel by dissolving it in methanol and purified by chromatography (EtOAc→EtOAc/MeOH, 98:2) providing **S1** (4.37 g, 72%) as a white solid; m.p. 129-131 °C; ^{1}H NMR (300 MHz): δ = 7.84 (s, 1H), 7.37 (s, 1H), 4.48 (d, *J* = 6.2 Hz, 2H), 2.86 (s, 6H), 2.79 (t, *J* = 6.2 Hz, 1H); ^{13}C NMR (75 MHz): δ = 136.6, 125.6, 120.9, 89.8, 77.3, 51.3, 38.3; IR (neat, cm^{-1}): 3242, 3150, 3122, 2927, 2222, 1685, 1548, 1386, 1266, 1170, 1087, 1030, 958, 843, 722; HR-ESIMS (m/z):

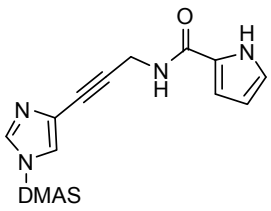
Calcd. for $C_8H_{12}N_3O_3S$ $[M+H]^+$ 230.0593, found 230.0591; Calcd. for $C_8H_{11}N_3O_3SNa$ $[M+Na]^+$ 252.0413, found 252.0407.

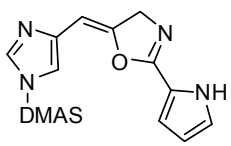
4-(3-Azidoprop-1-ynyl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (S2): To an ice-cooled solution of the alcohol **S1** (1.50 g, 6.55 mmol) in dry THF (50 mL), diphenyl phosphoryl azide (1.70 mL, 7.86 mmol) followed by DBU (1.17 mL, 7.86 mmol) was added dropwise. The solution was allowed to warm to r.t. and stirred overnight. The reaction mixture was partitioned between NH_4Cl solution and extracted with EtOAc. The organic layer was washed with brine solution, dried (Na_2SO_4), and concentrated to give brown solids. These solids were purified by flash chromatography providing azide **S2** (1.56 g, 94%) as a brown solid; m.p 75-76 °C; ^{1}H NMR (300 MHz): δ = 7.81 (s, 1H), 7.40 (s, 1H), 4.11 (s, 2H), 2.86 (s, 6H); ^{13}C (75 MHz) NMR: δ = 136.7, 125.0, 121.5, 83.4, 79.2, 40.5, 38.3; IR (neat, cm^{-1}): 3132, 3114, 2920, 2114, 2070, 1466, 1392, 1268, 1181, 1088, 963, 854, 719; HR-ESIMS (m/z): Calcd. for $C_8H_{11}N_6O_2S$ $[M+H]^+$ 255.0659, found 255.0661; Calcd. for $C_8H_{10}N_6O_2SNa$ $[M+Na]^+$ 277.0478, found 277.0490.

4-(3-Aminoprop-1-ynyl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (S3): PPh_3 (1.62 g, 6.19 mmol) was added to the solution of azide **S2** (1.05 g, 4.13 mmol) in dry THF (40 mL) and stirred for 4 h. H_2O (3 mL) was added to above solution and stirred overnight. The reaction mixture was concentrated purified on silica gel (EtOAc→EtOAc/MeOH/Et_3N, 70:25:5) to obtain

amine **S3** (819 mg, 87%) as a thick oil. ^{1}H NMR (300 MHz): δ = 7.79 (d, *J* = 1.1 Hz, 1H), 7.30 (d, *J* = 1.1 Hz, 1H), 3.61 (s, 2H), 2.84 (s, 6H), 1.56 (brs, 2H); ^{13}C NMR (75 MHz): δ = 136.5, 126.1, 120.2, 92.3, 74.5, 38.3, 32.2; IR (neat, cm^{-1}): 3369, 3303, 3126, 2923, 2233, 1671, 1602, 1541, 1387, 1325, 1170, 1085, 960, 723; HR-ESIMS (m/z): Calcd. for $C_8H_{13}N_4O_2S$ $[M+H]^+$ 229.0754, found 229.0747; Calcd. for $C_8H_{12}N_4O_2SNa$ $[M+Na]^+$ 251.0573, found 251.0566.

***N*-(3-(1-(*N*,*N*-Dimethylsulfamoyl)-1H-imidazol-4-yl)prop-2-ynyl)-1H-pyrrole-2-carboxamide (11):** A mixture of amine **S3** (750 mg, 3.28 mmol), trichloroacetyl pyrrole derivative (905 mg, 4.26 mmol) and K_2CO_3 (587 mg, 4.26 mmol) in dry DMF (12 mL) was stirred at r.t. overnight. To the resulting mixture was added half saturated NH_4Cl solution and the aqueous layer was repeatedly extracted with EtOAc. The organic extracts were combined, washed with brine solution, dried (Na_2SO_4), concentrated and purified on silica gel (CH_2Cl_2→hexane/EtOAc, 20:80) to give amide **11** (916 mg, 87%) as a white solid; m.p 179-182 °C; ^{1}H NMR (300 MHz, DMSO-d_6): δ = 11.49 (s, 1H), 8.50 (t, *J* = 5.6 Hz, 1H), 8.16 (d, *J* = 1.4 Hz, 1H), 7.91 (d, *J* = 1.4 Hz, 1H), 6.86-6.83 (m, 1H), 6.79-6.76 (m, 1H), 6.07-6.04 (m, 1H), 4.22 (d, *J* = 5.6 Hz, 2H), 2.78 (s, 6H); ^{13}C NMR (75 MHz): δ = 160.9, 137.8, 126.2, 125.1, 122.3, 122.2, 110.9, 109.2, 89.1, 74.9, 38.3, 28.9; IR (neat, cm^{-1}): 3236, 3140, 3049, 1620, 1563, 1421, 1390, 1261, 1178, 1088, 1037, 964, 837, 769, 725; HR-ESIMS (m/z): Calcd. for $C_{13}H_{16}N_5O_3S$ $[M+H]^+$ 322.0968, found 322.0969; Calcd. for $C_{13}H_{15}N_5O_3SNa$ $[M+Na]^+$ 344.0788, found 344.0804.



(*Z*)-4-((2-(1H-Pyrrol-2-yl)oxazol-5(4H)-ylidene)methyl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (14): 

$Pd(OAc)_2$ (1.98 mg, 0.02 mmol) was added to the solution of amide **11** (100 mg, 0.31 mmol) in CH_2Cl_2/TFA (1.5 mL/1 mL) and stirred for 1 h. After the starting material was consumed, the reaction mixture was concentrated and partitioned between half saturated $NaHCO_3$ solution and EtOAc. The organic layer was dried (Na_2SO_4), concentrated and purified by column chromatography (hexane/EtOAc, 20:80) providing oxazole **14** (87 mg, 87%) as a yellowish powder; m.p 193-195 °C; ^{1}H NMR (300 MHz, DMSO-d_6): δ = 11.98 (s, 1H), 8.12 (d, *J* = 1.0 Hz, 1H), 7.59 (s, 1H), 7.05-7.02 (m, 1H), 6.71-6.69 (m, 1H), 6.22-6.19 (m, 1H), 5.73 (t, *J* = 2.2 Hz, 1H), 4.73 (d, *J* = 2.2 Hz, 2H), 2.82 (s, 6H); ^{13}C NMR (75 MHz): δ = 156.9, 153.0, 137.1, 137.0, 124.2, 118.7, 114.9, 113.4, 109.9, 93.0, 58.2, 38.4; IR (neat, cm^{-1}): 3168, 3143, 2972, 1714, 1667, 1430, 1393, 1153, 1091, 991, 946, 803, 729; HR-ESIMS (m/z): Calcd. for $C_{13}H_{16}N_5O_3S$ $[M+H]^+$ 322.0968, found 322.0967; Calcd. for $C_{13}H_{15}N_5O_3SNa$ $[M+Na]^+$ 344.0788, found 344.0794.

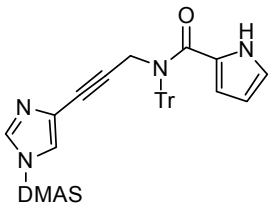
***N*-Tritylprop-2-yn-1-amine (S4):** To a solution of propargyl amine (14.78 g, 0.269 mol) in dry CH_2Cl_2 (100 mL), the solution of trityl chloride (35.80 g, 0.128 mol) in CH_2Cl_2 (75 mL) was added dropwise at 0 °C. the solution was stirred at r.t. overnight. The reaction mixture was partitioned with water (50 mL), and the organic layer was washed with brine, dried (Na_2SO_4) and concentrated. The residue was crystallized from hexane to give **S4** (34.65 g, 91%) as a white solid; m.p 73-75 °C; ^{1}H NMR: δ = 7.52-7.50 (m, 6H), 7.32-7.29 (m, 6H), 7.23-7.21 (m, 3H), 2.97 (s, 2H), 2.20 (t,

J = 2.5 Hz, 1H), 2.02 (brs, 1H); ^{13}C NMR: δ = 145.3, 128.7, 128.1, 126.7, 82.8, 71.1, 70.8, 33.7; IR (neat, cm^{-1}): 3326, 3289, 3278, 3054, 2840, 2125, 1594, 1487, 1447, 1209, 1097, 1029, 899, 768, 744, 694; HR-ESIMS (m/z): Calcd. for $C_{22}H_{19}NNa$ $[M+Na]^+$ 320.1410, found 320.1412; Calcd. for $C_{22}H_{19}NK$ $[M+K]^+$ 336.1149, found 336.1153.

***N*-(Prop-2-ynyl)-*N*-trityl-1H-pyrrole-2-carboxamide (S5):** To the mixture of pyrrole carboxylic acid (10.00 g, 90.09 mmol) in dry CH_2Cl_2 (125 mL), oxalyl chloride (19.65 mL, 225.2 mmol) was added followed by two drops of DMF. The solution was stirred for 3-4 h and then concentrated to obtain crude acid chloride derivative. The amine **S4** (10.72 g, 36.04 mmol) and triethylamine (17.54 mL, 126.1mmol) were dissolved in dry THF (150 mL) and cooled to 0 °C. The solution of crude acid chloride derivative in dry THF (100 mL) was added to above solution over a period of 20-30 min. The solution was warmed to r.t. and stirred overnight. The reaction mixture was worked up by adding NH_4Cl and repeatedly extracting with EtOAc. The combined organic extracts were washed with $NaHCO_3$ solution, followed by the brine solution, dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (hexane/EtOAc, 80:20) to obtain product **S5** (12.65 g, 90%) with an estimated 85% purity. This product was used in excess for next reaction. For characterization purpose amide **S5** was purified by repeated recrystallization from CH_2Cl_2; m.p 198-202 °C; ^{1}H NMR: δ = 9.48 (brs, 1H), 7.38-7.37 (m, 6H), 7.28-7.19 (m, 10H), 6.76-6.75 (m, 1H), 6.27-6.25 (m, 1H), 4.62 (d, *J* = 2.3 Hz, 2H), 2.17 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR: δ = 163.5, 143.1, 130.3, 127.5, 126.8, 126.2, 121.8, 112.8, 109.9, 80.9, 78.2, 72.9, 40.1; IR (neat, cm^{-1}): 3288, 3244, 3058, 3032, 2120, 1604, 1490, 1403,

1342, 1131, 917, 840, 724, 698; HR-ESIMS (m/z): Calcd. for $C_{27}H_{22}N_2ONa$ $[M+Na]^+$ 413.1624, found 413.1638; Calcd. for $C_{27}H_{22}N_2OK$ $[M+K]^+$ 429.1364, found 429.1360.

***N*-(3-(1-(*N*,*N*-Dimethylsulfamoyl)-1H-imidazol-4-yl)prop-2-ynyl)-*N*-trityl-1H-pyrrole-2-carboxamide (12):** 4-Iodoimidazole (1.28 g, 4.25 mmol), alkyne **S5** (2.48 g, 6.37 mmol), K_2CO_3 (1.17 g, 8.52 mmol), CuI (40 mg, 0.21 mmol) and $Pd(PPh_3)_2Cl_2$ (74 mg, 0.11 mmol) were placed in a reaction flask. To the above reaction mixture dry THF (40 mL) was added and N_2 was bubbled through it for 3-5 min. The heterogeneous mixture was stirred at 55 °C overnight. The reaction mixture was cooled to r.t. and water (15 mL) was added and the layers were separated. The aqueous layer was extracted with EtOAc. The combined organic extracts were dried (Na_2SO_4) and concentrated to provide brown solids. The crude product was purified by chromatography (CH_2Cl_2→hexane/EtOAc, 45:55) to give product **12** (1.46 g, 61%) as a pale yellowish solid; m.p 170-173 °C; ^{1}H NMR: δ = 9.43 (brs, 1H), 7.78 (d, *J* = 1.3 Hz, 1H), 7.41-7.39 (m, 6H), 7.29-7.25 (m, 6H), 7.23-7.20 (m, 4H), 7.19-7.18 (m, 1H), 6.80-6.78 (m, 1H), 6.28-6.26 (m, 1H), 4.82 (s, 2H), 2.88 (s, 6H); ^{13}C NMR: δ = 163.7, 143.0, 136.4, 130.3, 127.5, 126.8, 126.2, 125.7, 121.8, 121.1, 112.9, 110.2, 88.2, 78.0, 76.4, 40.9, 38.3; IR (neat, cm^{-1}): 3248, 3140, 3055, 1595, 1396, 1344, 1259, 1177, 1132, 1083, 958, 836, 745, 721; HR-ESIMS (m/z): Calcd. for $C_{32}H_{29}N_5O_3SNa$ $[M+Na]^+$ 586.1883, found 586.1899; Calcd. for $C_{32}H_{29}N_5O_3SK$ $[M+K]^+$ 602.1623, found 602.1610.

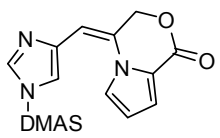


(*Z*)-*N*,*N*-Dimethyl-4-((1-oxo-2-trityl-2,3-dihydropyrrolo[1,2-a]pyrazin-4(1H)-ylidene)methyl)-1H-imidazole-1-sulfonamide (15): The alkyne **12** (2.68 g, 4.75 mmol) and Cs_2CO_3 (2.32 g, 7.12 mmol) were dissolved in dry DMF (30 mL) and stirred at r.t. for 30 min. The reaction mixture was quenched with NH_4Cl solution and extracted with EtOAc. The combined organic extracts were washed with brine solution, dried (Na_2SO_4), concentrated, and purified by column chromatography (hexane/EtOAc, 45:55) to give pyrazine **15** (2.17 g, 81%) as a pale yellow solid; m.p 181-184 °C; ^{1}H NMR: δ = 7.93 (s, 1H), 7.63-7.62 (m, 1H), 7.55-7.54 (m, 6H), 7.34 (s, 1H), 7.29-7.25 (m, 6H), 7.18-7.15 (m, 3H), 6.90-6.89 (m, 1H), 6.22-6.21 (m, 1H), 5.94 (s, 1H), 4.06 (s, 2H), 2.91 (s, 6H); ^{13}C NMR: δ = 161.1, 142.9, 137.4, 136.6, 130.3, 128.9, 127.9, 126.7, 126.5, 123.2, 116.9, 115.8, 111.0, 104.8, 76.0, 52.7, 38.4; IR (neat, cm^{-1}): 3148, 3116, 1698, 1663, 1640, 1463, 1382, 1169, 1078, 963, 777, 748, 706; HR-ESIMS (m/z): Calcd. for $C_{32}H_{29}N_5O_3SNa$ $[M+Na]^+$ 586.1883, found 586.1899; Calcd. for $C_{32}H_{29}N_5O_3SK$ $[M+K]^+$ 602.1623, found 602.1611.

3-(1-(*N*,*N*-Dimethylsulfamoyl)-1H-imidazol-4-yl)prop-2-ynyl-1H-pyrrole-2-carboxylate (13): To a mixture of imidazole propargyl alcohol derivative **S1** (1.20 g, 5.24 mmol), pyrrole carboxylic acid (640 mg, 5.76 mmol), DMAP (64 mg, 0.52 mmol) and CSA (73 mg, 0.31 mmol) in dry CH_2Cl_2 (120 mL), a solution of DCC (1.62 g, 7.86 mmol) in dry CH_2Cl_2 (10 mL) was added dropwise at -78 °C. The solution was then stirred at r.t. for 24 h. The mixture was filtered, the filtrate was concentrated and purified by column chromatography (hexane/EtOAc, 40:60) to obtain ester **13** (1.23 g, 73%) as a

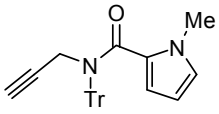
white solid; m.p 156-158 °C; ^{1}H NMR (300 MHz): δ = 9.32 (brs, 1H), 7.83 (d, *J* = 1.4 Hz, 1H), 7.40 (d, *J* = 1.4 Hz, 1H), 7.00-6.98 (m, 2H), 6.28-6.26 (m, 1H), 5.07 (s, 2H), 2.86 (s, 6H); ^{13}C NMR (75 MHz): δ = 160.2, 136.6, 125.3, 123.6, 121.9, 121.5, 116.3, 110.8, 85.4, 78.6, 52.4, 38.3; IR (neat, cm^{-1}): 3321, 3132, 3070, 2969, 2244, 1713, 1392, 1317, 1266, 1163, 1080, 960, 857, 748, 724; HR-ESIMS (m/z): Calcd. for $C_{13}H_{15}N_4O_4S$ $[M+H]^+$ 323.0809, found 323.0805; Calcd. for $C_{13}H_{14}N_4O_4SNa$ $[M+Na]^+$ 345.0628, found 345.0646.

(*Z*)-*N*,*N*-Dimethyl-4-((1-oxo-1H-pyrrolo[2,1-c][1,4]oxazin-4(3H)-ylidene)methyl)-1H-imidazole-1-sulfonamide (16): The mixture of ester **13** (100 mg, 0.31 mmol) and



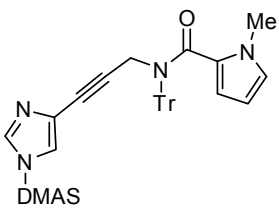
Cs_2CO_3 (131 mg, 0.40 mmol) in dry DMF (2.5 mL) was heated at 80 °C for 1.5 h. Then the reaction mixture was neutralized with 1N HCl at 0 °C and the resulting aqueous layer was repeatedly extracted with EtOAc. The organic layers were combined, washed with the brine solution, dried with Na_2SO_4 and purified on column (hexane/EtOAc, 30:70) to give morpholine **16** (65 mg, 65%) as a pale yellowish powder; m.p 156-157 °C; ^{1}H NMR: δ = 8.04-8.03 (m, 1H), 7.90 (s, 1H), 7.30 (s, 1H), 7.18-7.17 (m, 1H), 6.38 (t, *J* = 3.4 Hz, 1H), 6.13 (s, 1H), 4.92 (s, 2H), 2.88 (s, 6H); ^{13}C NMR (75 MHz): δ = 158.7, 136.7, 136.6, 126.7, 126.6, 119.7, 118.9, 118.1, 111.6, 107.5, 71.7, 38.3; IR (neat, cm^{-1}): 3142, 3127, 2927, 1700, 1519, 1462, 1391, 1291, 1241, 1173, 1078, 1016, 884, 755, 718; HR-ESIMS (m/z): Calcd. for $C_{13}H_{15}N_4O_4S$ $[M+H]^+$ 323.0809, found 323.0801; Calcd. for $C_{13}H_{14}N_4O_4SNa$ $[M+Na]^+$ 345.0628, found 345.0634.

1-Methyl-*N*-(prop-2-ynyl)-*N*-trityl-1H-pyrrole-2-carboxamide (S6): To an ice-cold



solution of a pyrrole derivative **S5** (≈85%, 1.10 g, 2.81 mmol) in dry THF (25 mL), NaH (60%, 146 mg, 3.65 mmol) was added and the solution was allowed to warm to 20 °C over a period of 10-15 min. The solution was cooled back to 0 °C and MeI (0.88 mL, 14.1 mmol) was added dropwise and stirring was continued for 1 h at r.t. The resulting mixture was quenched with NH_4Cl solution and extracted repeatedly with EtOAc. The organic extracts were combined, washed with brine solution, dried (Na_2SO_4) and concentrated to give a brown solid. The crude material was purified on silica gel (hexane/EtOAc, 70:30) to obtain methyl protected derivative **S6** (795 mg, 70%) as a white solid; m.p 200-202 °C; ^{1}H NMR: δ = 7.51-7.49 (m, 6H), 7.29-7.25 (m, 6H), 7.22-7.19 (m, 3H), 6.98 (dd, *J* = 4.0, 2.2 Hz, 1H), 6.68 (t, *J* = 2.1 Hz, 1H), 6.14 (dd, *J* = 4.0, 2.5 Hz, 1H), 4.47 (d, *J* = 2.3 Hz, 2H), 3.58 (s, 3H), 1.76 (t, *J* = 2.3 Hz, 1H); ^{13}C NMR: δ = 166.5, 143.3, 129.7, 127.9, 127.7, 127.0, 126.6, 112.7, 107.1, 80.7, 76.8, 70.6, 41.5, 35.8; IR (neat, cm^{-1}): 3311, 3128, 3056, 1620, 1414, 1337, 1249, 1116, 967, 904, 742, 697; HR-ESIMS (m/z): Calcd. for $C_{28}H_{24}N_2ONa$ $[M+Na]^+$ 427.1718, found 427.1798; Calcd. for $C_{28}H_{24}N_2OK$ $[M+K]^+$ 443.1520, found 443.1520.

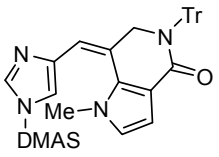
***N*-(3-(1-(*N*,*N*-Dimethylsulfamoyl)-1H-imidazol-4-yl)prop-2-ynyl)-1-methyl-*N*-trityl-1H-pyrrole-2-carboxamide (17):** 4-Iodoimidazole (462 mg, 1.53 mmol), alkyne **S6** (926



mg, 2.29 mmol), K_2CO_3 (422 mg, 3.06 mmol), CuI (15 mg, 0.08 mmol) and $Pd(PPh_3)_2Cl_2$ (27 mg, 0.04 mmol) were placed in a reaction flask. To the above reaction mixture dry THF (25 mL) was added and N_2 was bubbled through it for 3-5 min. The heterogeneous mixture was

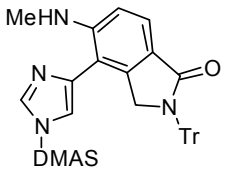
stirred at 55 °C overnight. The reaction mixture was cooled to r.t. and water (15 mL) was added and the layers separated. The aqueous layer was extracted with EtOAc. The combined organic extracts were dried (Na_2SO_4) and concentrated to brown solids. The crude product was purified by flash chromatography (CH_2Cl_2→hexane/EtOAc, 50:50) to give product **17** (566 mg, 64%) as a yellowish solid; m.p 189-191 °C; ^{1}H NMR: δ = 7.70 (d, *J* = 1.1 Hz, 1H), 7.54-7.52 (m, 6H), 7.29-7.26 (m, 6H), 7.20-7.18 (m, 3H), 6.99-6.98 (m, 2H), 6.67 (t, *J* = 2.1 Hz, 1H), 6.14 (dd, *J* = 4.0, 2.5 Hz, 1H), 4.67 (s, 2H), 3.58 (s, 3H), 2.85 (s, 6H); ^{13}C NMR: δ = 166.8, 143.3, 136.2, 129.7, 127.9, 127.7, 127.3, 126.6, 125.6, 121.1, 113.0, 107.3, 88.2, 76.6, 74.6, 42.3, 38.3, 35.7; IR (neat, cm^{-1}): 3148, 3085, 2980, 2239, 1736, 1659, 1437, 1392, 1175, 1087, 960, 828, 777, 704; HR-ESIMS (m/z): Calcd. for $C_{33}H_{31}N_5O_3SNa$ $[M+Na]^+$ 600.2040, found 600.2057; Calcd. for $C_{33}H_{31}N_5O_3SK$ $[M+K]^+$ 616.1779, found 616.1775.

(*Z*)-*N*,*N*-Dimethyl-4-((1-methyl-4-oxo-5-trityl-5,6-dihydro-1H-pyrrolo[3,2-c]pyridin-7(4H)-ylidene)methyl)-1H-imidazole-1-sulfonamide (18): The alkyne **17** (150 mg, 0.26 mmol) and $Pd(OAc)_2$ (9.0 mg, 0.013 mmol) were dissolved in dry DMF (3 mL) and stirred at 80 °C for 24 h. The reaction mixture was partitioned between H_2O and EtOAc. The organic extract was washed with brine solution, dried (Na_2SO_4), concentrated, and purified by column chromatography (hexane/EtOAc, 40:60) to give pyridine derivative **18** (79 mg, 52%) as a pale yellow solid; m.p 172-176 °C; ^{1}H NMR: δ = 7.92 (d, *J* = 1.1 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 6H), 7.23 (t, *J* = 7.6Hz, 6H), 7.18 (s, 1H), 7.14-7.12 (m, 3H), 6.56 (d, *J* = 2.9 Hz, 6H), 6.52 (d, *J* = 2.9 Hz, 1H), 6.40 (s, 1H), 4.05 (s, 2H), 3.15 (s, 3H), 2.93 (s, 6H); ^{13}C

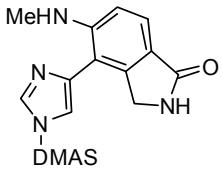


NMR: δ = 165.9, 143.8, 140.2, 136.9, 134.2, 129.0, 127.6, 126.6, 126.1, 126.0, 120.0, 116.4, 116.3, 109.0, 76.0, 57.1, 38.4, 36.4; IR (neat, cm^{-1}): 3111, 3022, 2921, 1650, 1595, 1448, 1389, 1262, 1170, 1074, 961, 800, 721; HR-ESIMS (m/z): Calcd. for $C_{33}H_{32}N_5O_3S$ $[M+H]^+$ 578.2220, found 578.2199; Calcd. for $C_{33}H_{31}N_5O_3SNa$ $[M+Na]^+$ 600.2040, found 600.2051.

***N*,*N*-Dimethyl-4-(5-(methylamino)-3-oxo-2-tritylisoindolin-4-yl)-1H-imidazole-1-sulfonamide (S7):** N_2 was bubbled through solution of the alkyne **17** (150 mg, 0.259 mmol), $AuCl(PPh_3)$ (13 mg, 0.025 mmol) in dry dioxane (15 mL) for 2-3 min and then the solution was heated to 115 °C in sealed tube for 24 h. The reaction mixture was concentrated and purified by column chromatography (hexane/EtOAc, 30:70) providing pale yellowish solid of aniline derivative **S7** (60 mg, 40%); ^1H NMR: δ = 7.94-7.92 (m, 2H), 7.66 (d, *J* = 8.6 Hz, 1H), 7.30-7.25 (m, 15H), 6.84 (s, 1H), 6.76 (d, *J* = 8.6 Hz, 1H), 4.27 (s, 2H), 2.96 (d, *J* = 4.6 Hz, 3H), 2.72 (s, 6H); ^{13}C NMR: δ = 169.7, 151.2, 143.4, 140.4, 140.2, 134.9, 130.1, 127.7, 127.1, 125.5, 121.2, 114.1, 110.8, 108.4, 73.8, 54.9, 38.4, 30.4; IR (neat, cm^{-1}): 3307, 3254, 3126, 2923, 2234, 1673, 1601, 1444, 1372, 1278, 1170, 1084, 961, 907, 751, 723; HR-ESIMS (m/z): Calcd. for $C_{33}H_{32}N_5O_3S$ $[M+H]^+$ 578.2220, found 578.2215; Calcd. for $C_{33}H_{31}N_5O_3SNa$ $[M+Na]^+$ 600.2040, found 600.2055.

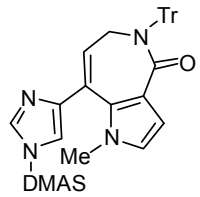


***N*,*N*-Dimethyl-4-(5-(methylamino)-3-oxoisoindolin-4-yl)-1H-imidazole-1-sulfonamide (24):** A solution of aniline derivative **S7** (55.0 mg, 0.095 mmol) in



TFA/CH_2Cl_2 (1 mL/2 mL) was stirred at r.t. for 3 h. Then the reaction mixture was concentrated, dissolved in EtOAc and partitioned with half saturated solution of $NaHCO_3$. The organic layer was dried (Na_2SO_4), concentrated and purified on silica gel (EtOAc/MeOH, 98:2) obtaining amide **24** (30 mg, 94%) as a white solid; m.p 226-227 °C; ^{1}H NMR: δ = 7.96 (s, 1H), 7.64 (d, *J* = 8.6 Hz, 1H), 7.27 (s, 1H), 6.73 (d, *J* = 8.6 Hz, 1H), 4.35 (s, 2H), 2.90 (s, 3H), 2.87 (s, 6H); ^{13}C NMR: δ = 151.3, 143.1, 139.7, 135.4, 125.1, 119.4, 114.7, 110.8, 109.6, 46.6, 38.3, 30.3, 29.7; IR (neat, cm^{-1}): 3305, 3175, 3066, 2920, 2439, 2320, 1668, 1597, 1487, 1383, 1279, 1164, 966, 822, 730; HR-ESIMS (m/z): Calcd. for $C_{14}H_{18}N_5O_3S$ $[M+H]^+$ 336.1125, found 336.1118; Calcd. for $C_{14}H_{17}N_5O_3SNa$ $[M+Na]^+$ 358.0944, found 358.0948.

***N*,*N*-Dimethyl-4-(1-methyl-4-oxo-5-trityl-1,4,5,6-tetrahydropyrrolo[3,2-c]azepin-8-yl)-1H-imidazole-1-sulfonamide (25):** A solution of alkyne **17** (100 mg, 0.173 mmol) and $AuCl_3$ (5.24 mg, 0.017mmol) in dry dioxane (10 mL) was heated to 60 °C in sealed tube for overnight. The reaction mixture was concentrated purified by flash chromatography (hexane/EtOAc, 25:75) providing azepin **25** in 43% yield; m.p 225-228 °C; ^{1}H NMR: δ = 7.97 (s, 1H), 7.39-7.38 (m, 6H), 7.25-7.21 (m, 6H), 7.17-7.13 (m, 4H), 6.61 (s, 2H), 6.52 (t, *J* = 7.5 Hz, 1H), 4.07-4.04 (m, 1H), 3.71-3.66 (m, 1H), 3.32 (s, 3H), 2.97 (s, 6H); ^{13}C NMR: δ = 166.4, 144.2, 141.7, 136.9, 130.2, 129.5, 128.5, 127.6, 127.5, 126.3, 125.1, 124.8, 115.2, 111.4, 77.6, 45.6, 38.4, 36.9; IR (neat, cm^{-1}): 3133, 3122, 2915, 1637, 1593, 1492, 1454, 1393, 1260, 1171, 1081, 955, 914, 837, 729; HR-ESIMS (m/z): Calcd. for



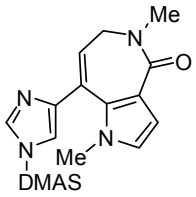
$C_{33}H_{32}N_5O_3S$ $[M+H]^+$ 578.2220, found 578.2207; Calcd. for $C_{33}H_{31}N_5O_3SNa$ $[M+Na]^+$ 600.2040, found 600.2058.

***N*-(3-(1-(*N*,*N*-Dimethylsulfamoyl)-1H-imidazol-4-yl)prop-2-ynyl)-*N*-1-dimethyl-1H-pyrrole-2-carboxamide (23):** 4-Iodoimidazole (343 mg, 1.14 mmol), alkyne (260 mg, 1.48 mmol), CuI (10.7 mg, 0.057 mmol) and $Pd(PPh_3)_2Cl_2$ (20.0 mg, 0.028 mmol) were placed in a reaction flask. To the above reaction mixture 1:1 mixture of dry THF (5 mL) and triethyl amine (5 mL) was added and N_2 was bubbled through it for 3-5 min. The solution was stirred at 60 °C for 15 h. The reaction mixture was concentrated and purified by flash chromatography (hexane/EtOAc, 30:70→EtOAc) to give product **23** (338 mg, 85%) as a pale yellowish solid; m.p 135-137 °C; ^{1}H NMR: δ = 7.83 (s, 1H), 7.34 (s, 1H), 6.70 (s, 1H), 6.57(s, 1H), 6.08 (t, *J* = 3.1 Hz, 1H), 4.54 (s, 2H), 3.79 (s, 3H), 3.23 (s, 3H), 2.87 (s, 6H); ^{13}C NMR (75 MHz): δ = 163.8, 136.6, 126.9, 125.7, 124.5, 121.0, 113.8, 107.1, 86.5, 38.3, 36.0; IR (neat, cm^{-1}): 3132, 3117, 2961, 1618, 1532, 1456, 1380, 1324, 1243, 1171, 1085, 958, 841, 722; HR-ESIMS (m/z): Calcd. for $C_{15}H_{20}N_5O_3S$ $[M+H]^+$ 350.1281, found 350.1283; Calcd. for $C_{15}H_{19}N_5O_3SNa$ $[M+Na]^+$ 372.1101, found 372.1119.

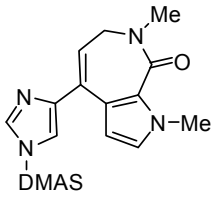
4-(1,5-Dimethyl-4-oxo-1,4,5,6-tetrahydropyrrolo[3,2-c]azepin-8-yl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (26) and 4-(1,7-Dimethyl-8-oxo-1,6,7,8-tetrahydropyrrolo[2,3-c]azepin-4-yl)-*N*,*N*-dimethyl-1H-imidazole-1-sulfonamide (27): A solution of alkyne **23** (200 mg, 0.57 mmol) and $AuCl_3$ (17.3 mg, 0.057mmol) in

dry dioxane (15 mL) was heated to 65 °C in sealed tube for overnight. The reaction mixture was concentrated purified by flash chromatography (hexane/EtOAc, 30:70→ EtOAc) providing two azepins **26** and **27**.

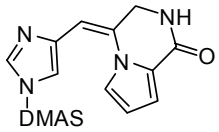
Azepine 26 (70 mg, 35%, pale yellowish solid): m.p 229-232 °C; ^{1}H NMR: δ = 7.89 (s, 1H), 7.00 (s, 1H), 6.74 (d, *J* = 2.9 Hz, 1H), 6.69 (d, *J* = 2.9 Hz, 1H), 6.58 (t, *J* = 7.6 Hz, 1H), 3.74 (d, *J* = 7.6 Hz, 1H), 3.35 (s, 3H), 3.16 (s, 3H), 2.85 (s, 6H); ^{13}C NMR: δ = 166.0, 141.3, 136.8, 130.6, 128.6, 125.3, 125.2, 123.8, 115.1, 109.9, 47.3, 38.3, 36.7, 35.4; IR (neat, cm^{-1}): 3131, 3088, 2955, 1603, 1556, 1496, 1379, 1262, 1169, 1087, 959, 861, 727; HR-ESIMS (m/z): Calcd. for $C_{15}H_{20}N_5O_3S$ $[M+H]^+$ 350.1281, found 350.1283; Calcd. for $C_{15}H_{19}N_5O_3SNa$ $[M+Na]^+$ 372.1101, found 372.1115.



Azepine 27 (82 mg, 41%, pale yellowish solid): m.p 212-215 °C; ^{1}H NMR: δ = 7.89 (d, *J* = 1.3 Hz, 1H), 7.25 (s, 1H), 6.77 (d, *J* = 2.9 Hz, 1H), 6.70 (t, *J* = 7.0 Hz, 1H), 6.31(d, *J* = 2.9 Hz, 1H), 3.97 (s, 3H), 3.78 (d, *J* = 7.0 Hz, 1H), 3.15 (s, 3H), 2.84 (s, 6H); ^{13}C NMR: δ = 162.2, 142.1, 136.7, 132.7, 127.3, 127.1, 124.5, 120.1, 115.3, 106.7, 47.3, 38.3, 36.7, 34.7; IR (neat, cm^{-1}): 3137, 3041, 2924, 1727, 1619, 1481, 1386, 1262, 1174, 1099, 959, 821, 721; HR-ESIMS (m/z): Calcd. for $C_{15}H_{20}N_5O_3S$ $[M+H]^+$ 350.1281, found 350.1286; Calcd. for $C_{15}H_{19}N_5O_3SNa$ $[M+Na]^+$ 372.1101, found 372.1118.

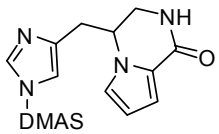


(*Z*)-*N*,*N*-Dimethyl-4-((1-oxo-2,3-dihydropyrrolo[1,2-a]pyrazin-4(1H)-ylidene)methyl)-1H-imidazole-1-sulfonamide (28): The pyrazine derivative **15** (800 mg, 1.42 mmol)



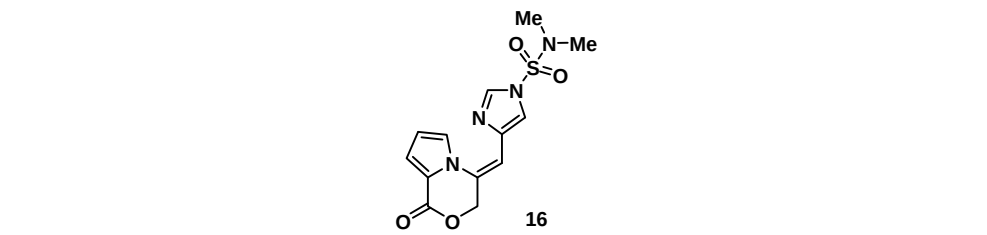
was dissolved in mixture of CH_2Cl_2 (5 mL), TFA (7 mL) and H_2O (2.5 mL) and stirred for 1.5 h and then it was concentrated. The residue was dissolved in CH_2Cl_2 and neutralized with $NaHCO_3$ solution. The organic layer was washed with brine solution, dried (Na_2SO_4) and purified on column (EtOAc→EtOAc/MeOH, 90:10) to give amide **28** (355 mg, 78%) as a white solid; m.p 172-175 °C; ^{1}H NMR: δ = 7.87 (s, 1H), 7.59 (s, 1H), 7.47-7.46 (m, 1H), 7.18 (s, 1H), 6.97-6.96 (m, 1H), 6.26 (t, *J* = 3.2 Hz, 1H), 5.99 (s, 1H), 4.19 (s, 2H), 2.84 (s, 6H); ^{13}C NMR: δ = 161.3, 137.2, 136.5, 129.4, 124.6, 124.1, 116.6, 114.6, 110.8, 106.3, 47.6, 38.3; IR (neat, cm^{-1}): 3294, 3191, 3127, 3059, 2928, 1659, 1556, 1431, 1387, 1230, 1173, 1081, 999, 843, 725, 668; HR-ESIMS (m/z): Calcd. for $C_{13}H_{16}N_5O_3S$ $[M+H]^+$ 322.0968, found 322.0963; Calcd. for $C_{13}H_{15}N_5O_3SNa$ $[M+Na]^+$ 344.0788, found 344.0794.

***N*,*N*-Dimethyl-4-((1-oxo-1,2,3,4-tetrahydropyrrolo[1,2-a]pyrazin-4-yl)methyl)-1H-imidazole-1-sulfonamide (29):** The olefin **28** (100 mg, 0.31 mmol) was subjected to

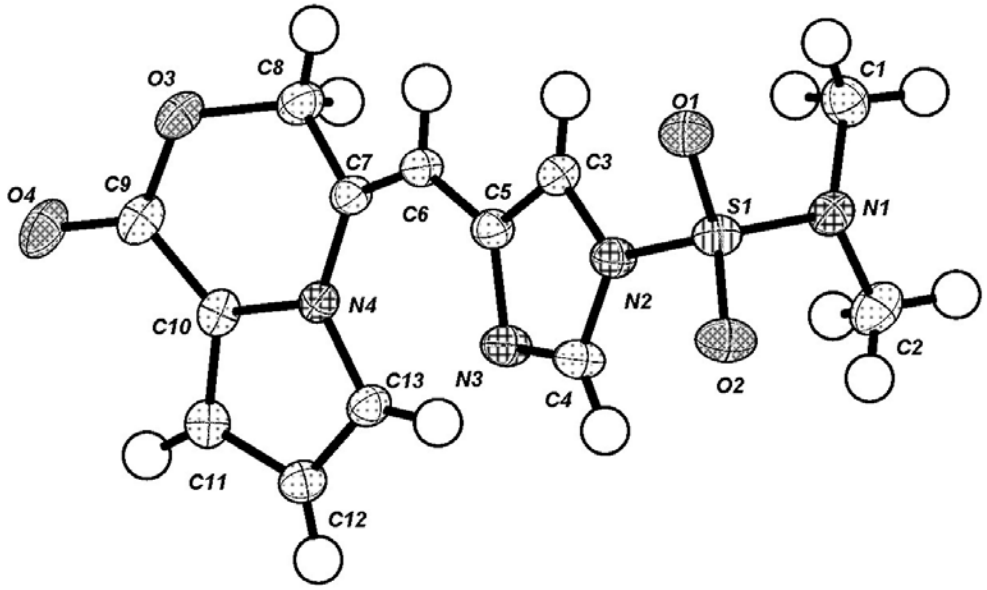


reduction by dissolving it in ethanol (4 mL) in presence of 10% Pd/C (30 mg, 0.03 mmol) with stirring at r.t. under 1 atm H_2 (balloon) for 5 h. The reaction mixture was filtered through a pad of Celite and the filtrate was concentrated. The residue was purified on silica gel (EtOAc/MeOH, 90:10) to provide pyrazine derivative **29** (75 mg, 75%) as a white powder; ^{1}H NMR: δ = 7.86 (s, 1H), 6.91 (dd, *J* = 4.0, 1.4 Hz, 1H), 6.76 (s 1H), 6.55-6.54 (m, 1H), 6.11-6.10 (s, 1H),

5.84 (brs, 1H), 4.64-4.62 (m, 1H), 3.92 (dd, *J* = 12.6, 4.6 Hz, 1H), 3.53-3.50 (m, 1H), 3.11-3.07 (m, 1H), 3.04-3.00 (m, 1H), 2.81 (s, 6H); ^{13}C NMR: δ = 161.2, 139.1, 136.9, 123.3, 123.2, 115.6, 113.9, 109.4, 53.6, 44.2, 38.2, 32.3.



16



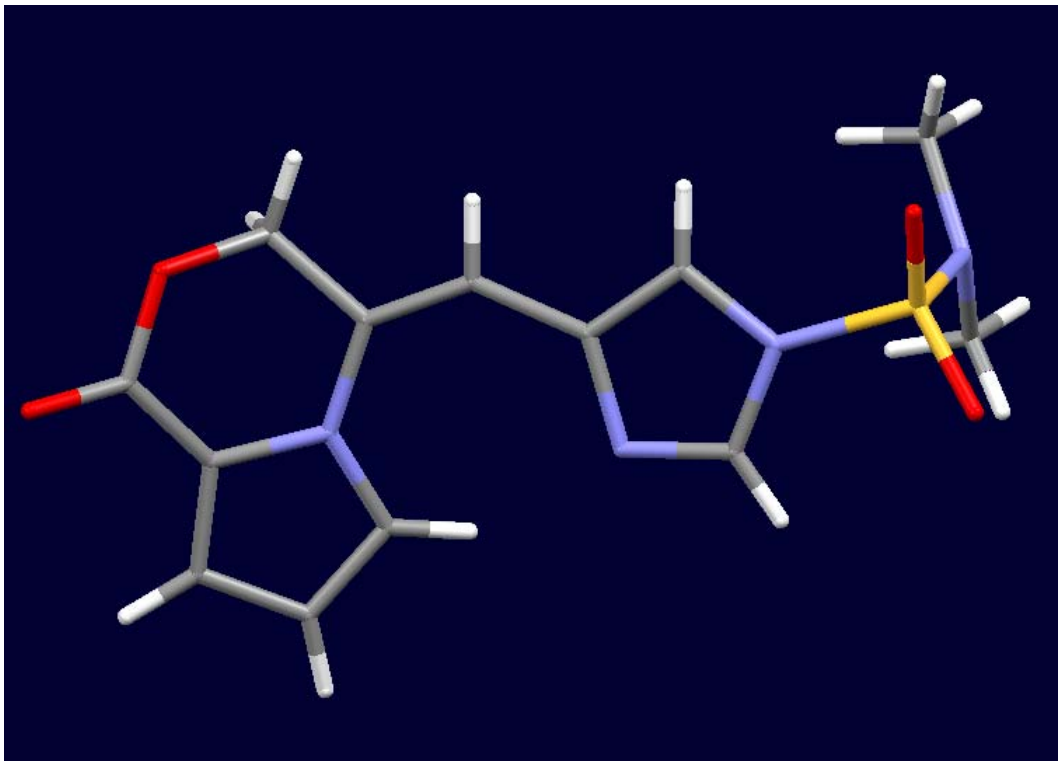


Figure S1: X-ray crystal structure of compound **16** (CIF = compound 16)

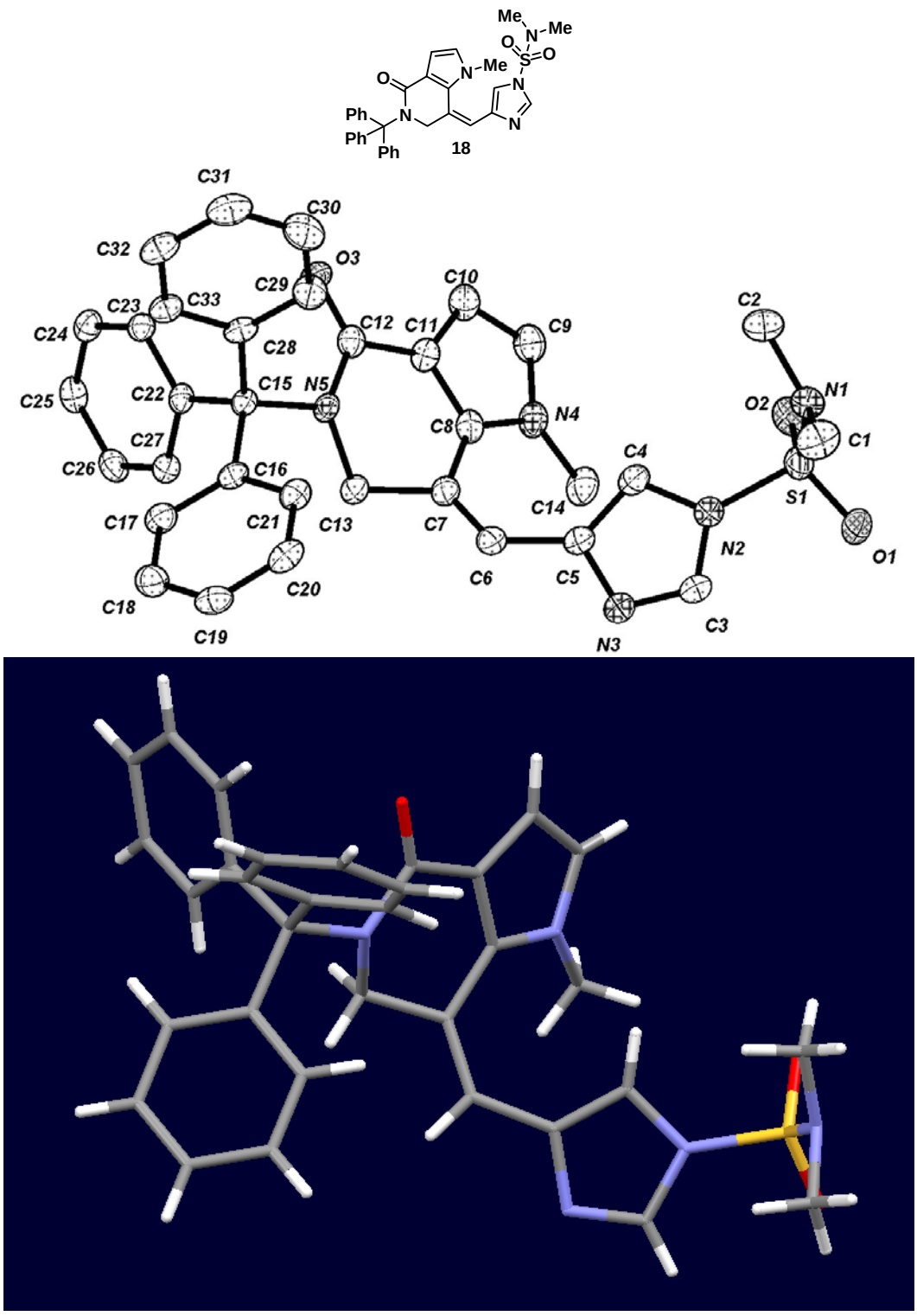


Figure S2: X-ray crystal structure of compound **18** (Cif = compound 18)

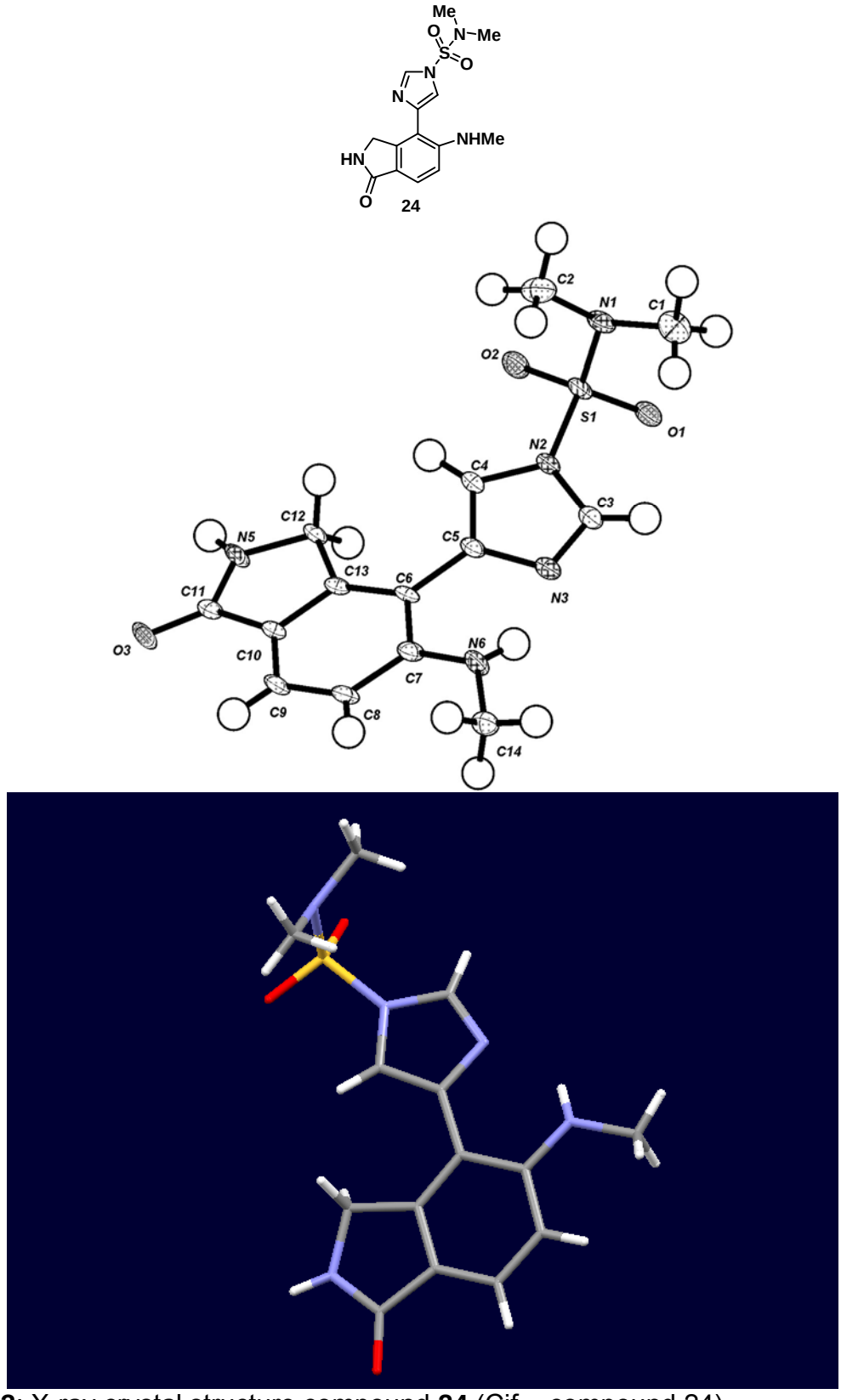


Figure S3: X-ray crystal structure compound **24** (Cif = compound 24)

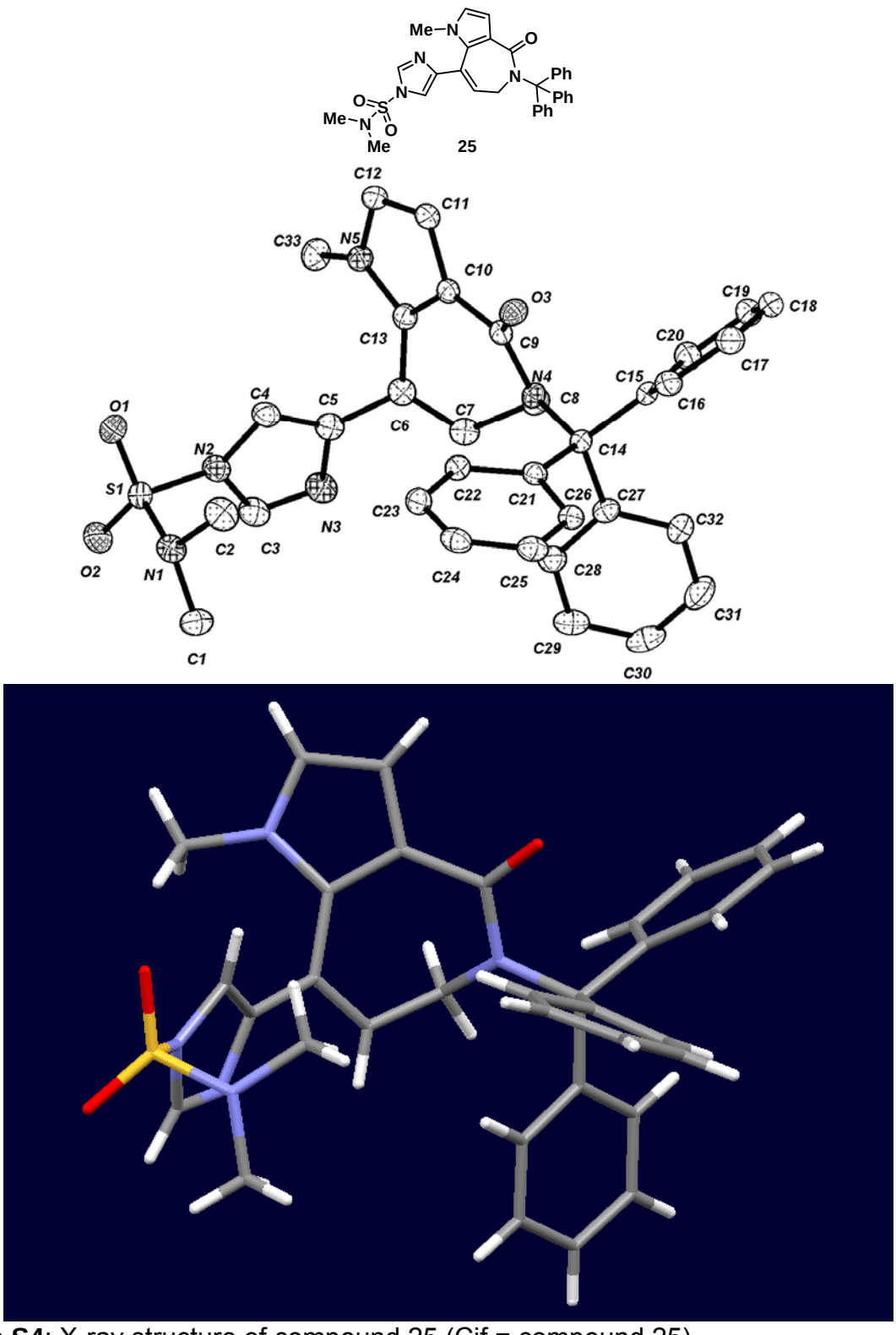


Figure S4: X-ray structure of compound 25 (Cif = compound 25)

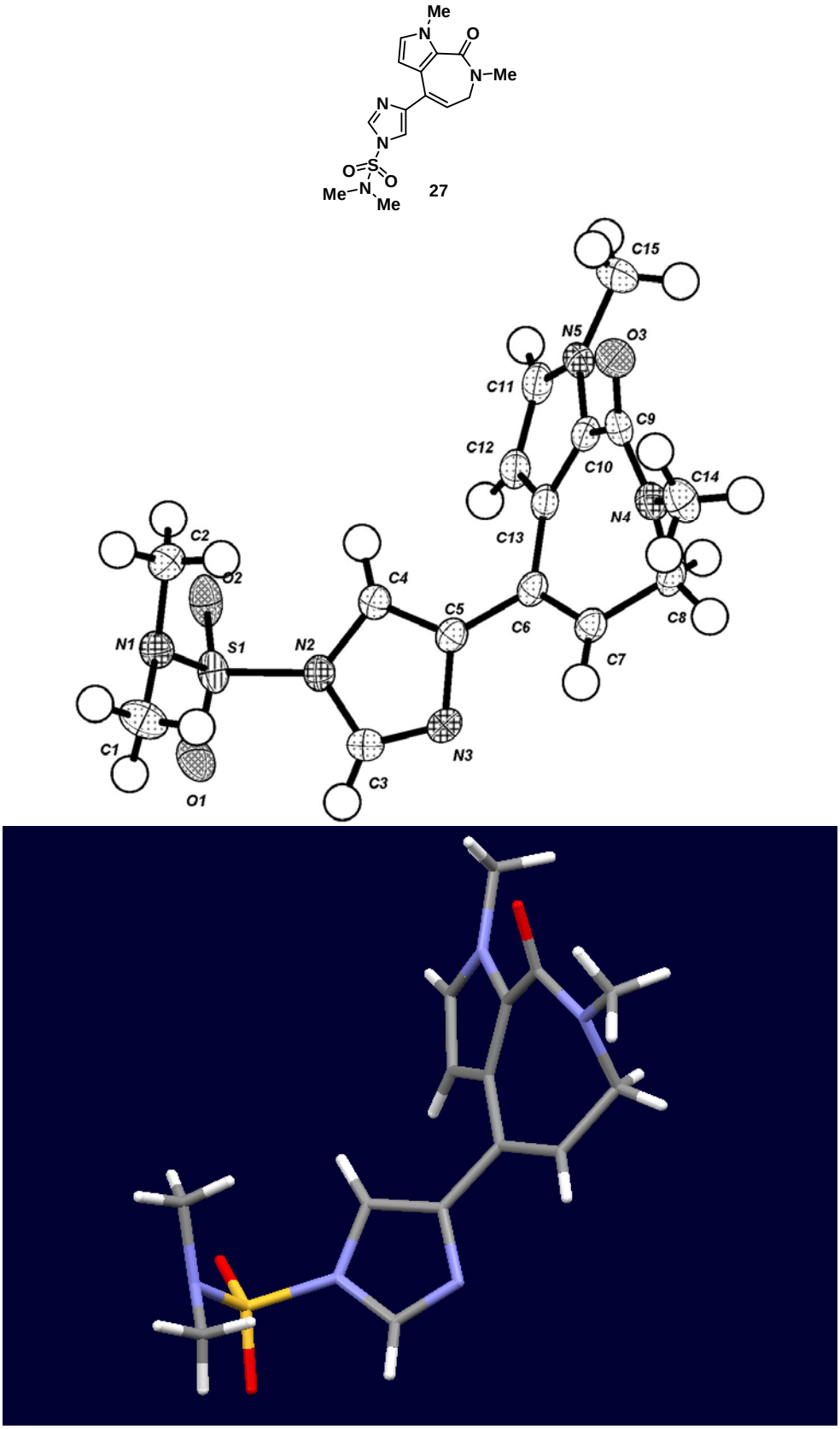


Figure S5: X-ray crystal structure of compound **27** (Cif = compound 27)

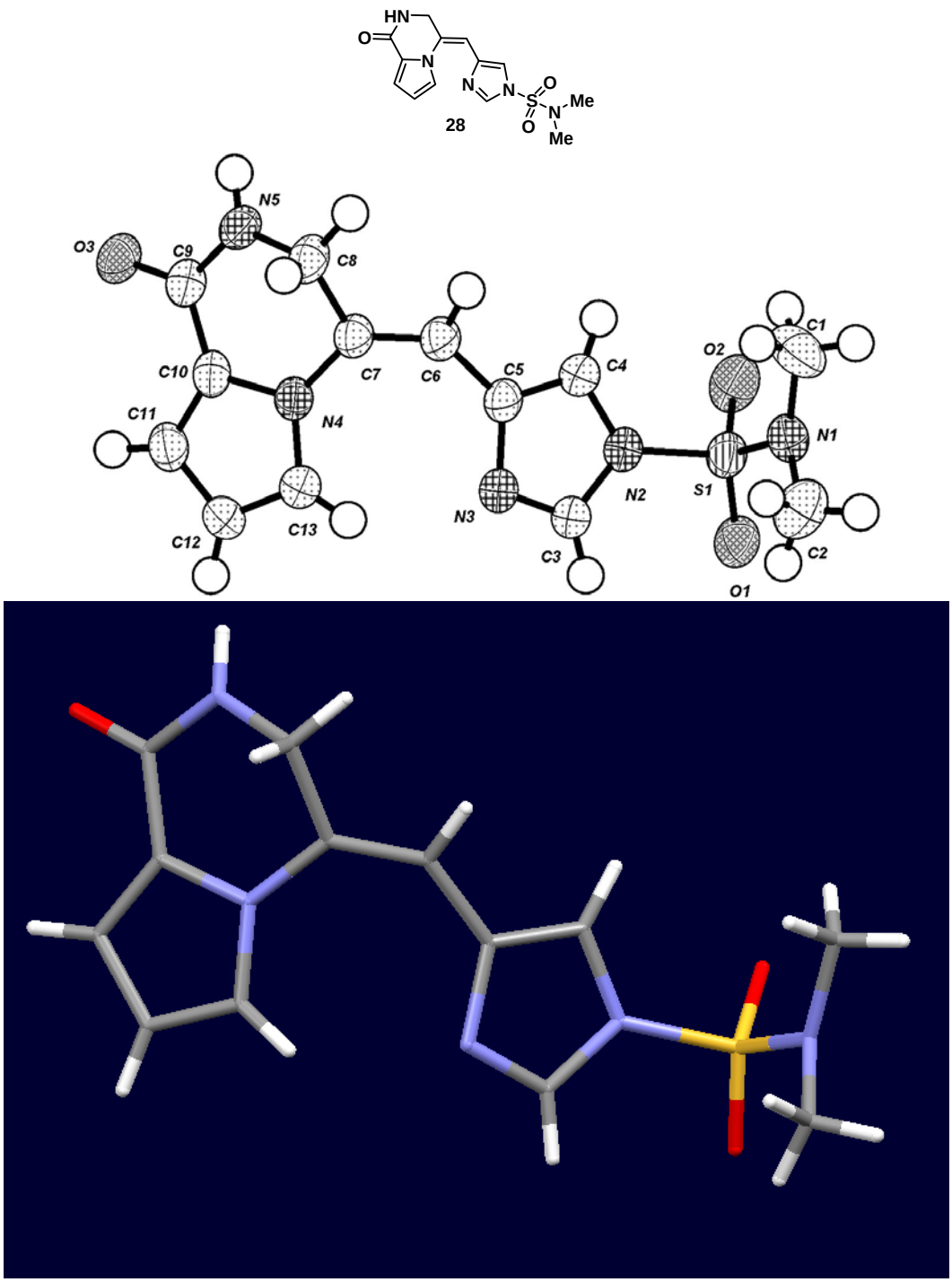
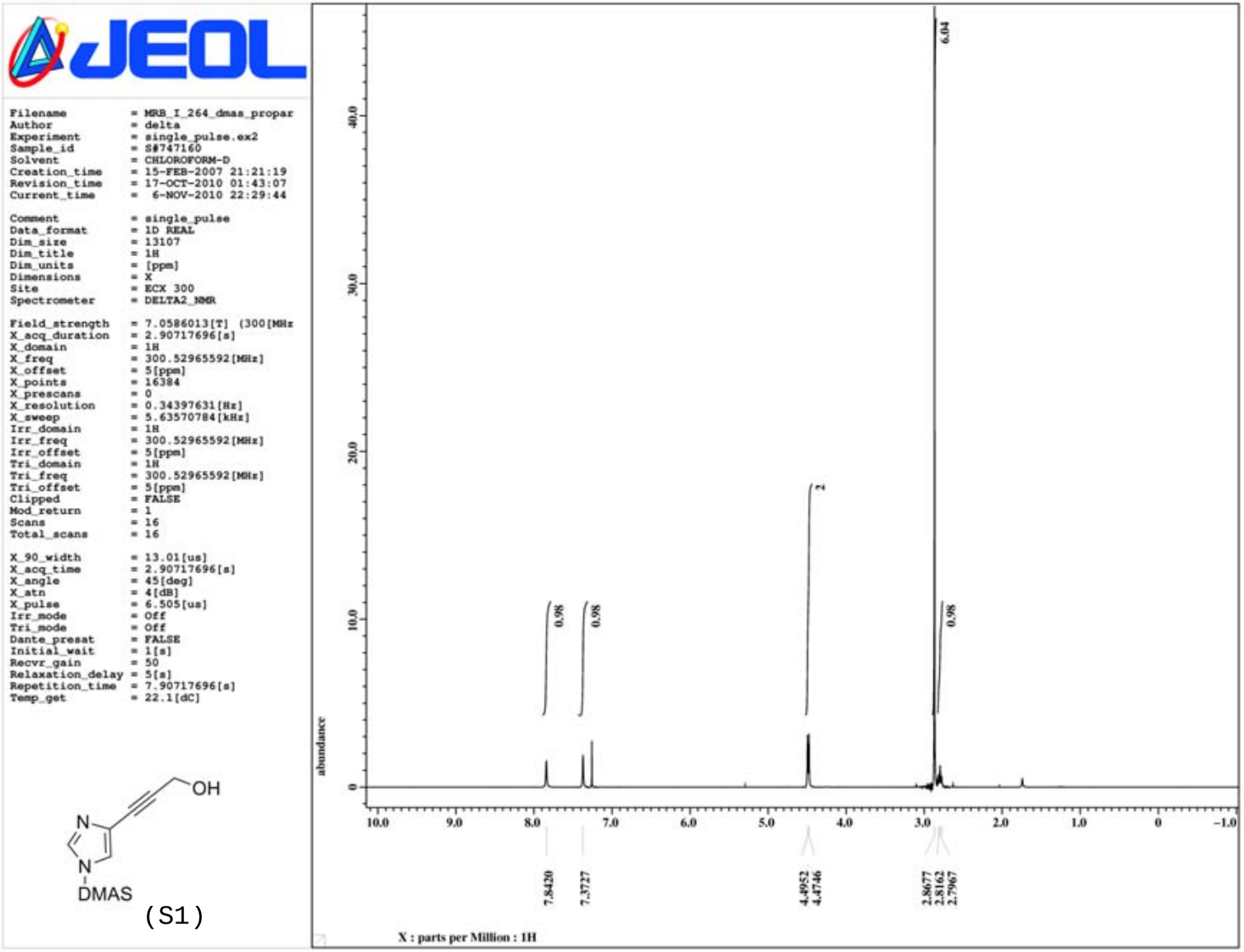


Figure S6: X-ray crystal structure of compound **28** (Cif = compound 28)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(S1)
abundance
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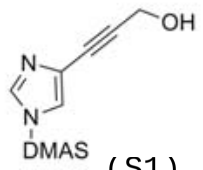
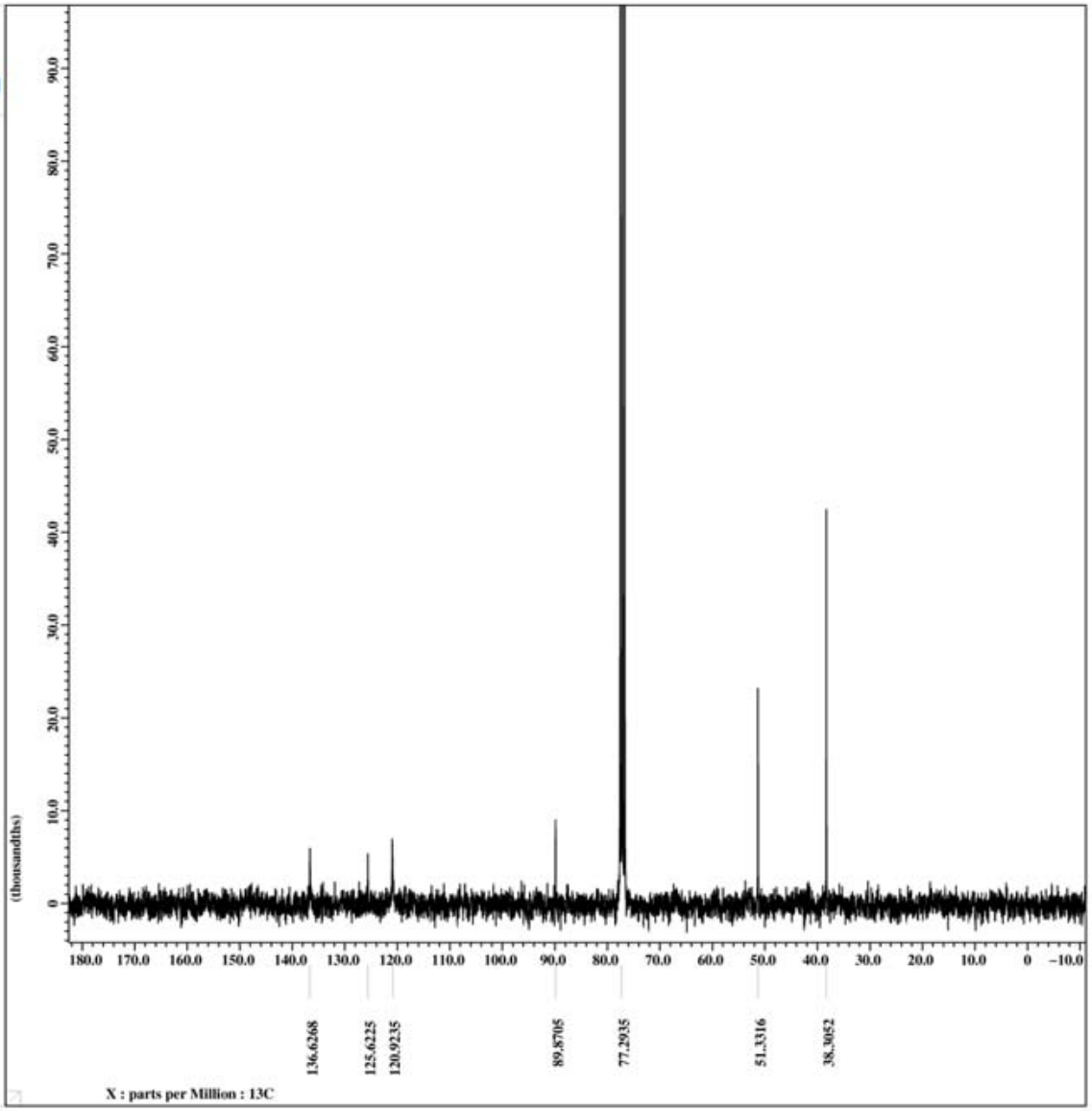
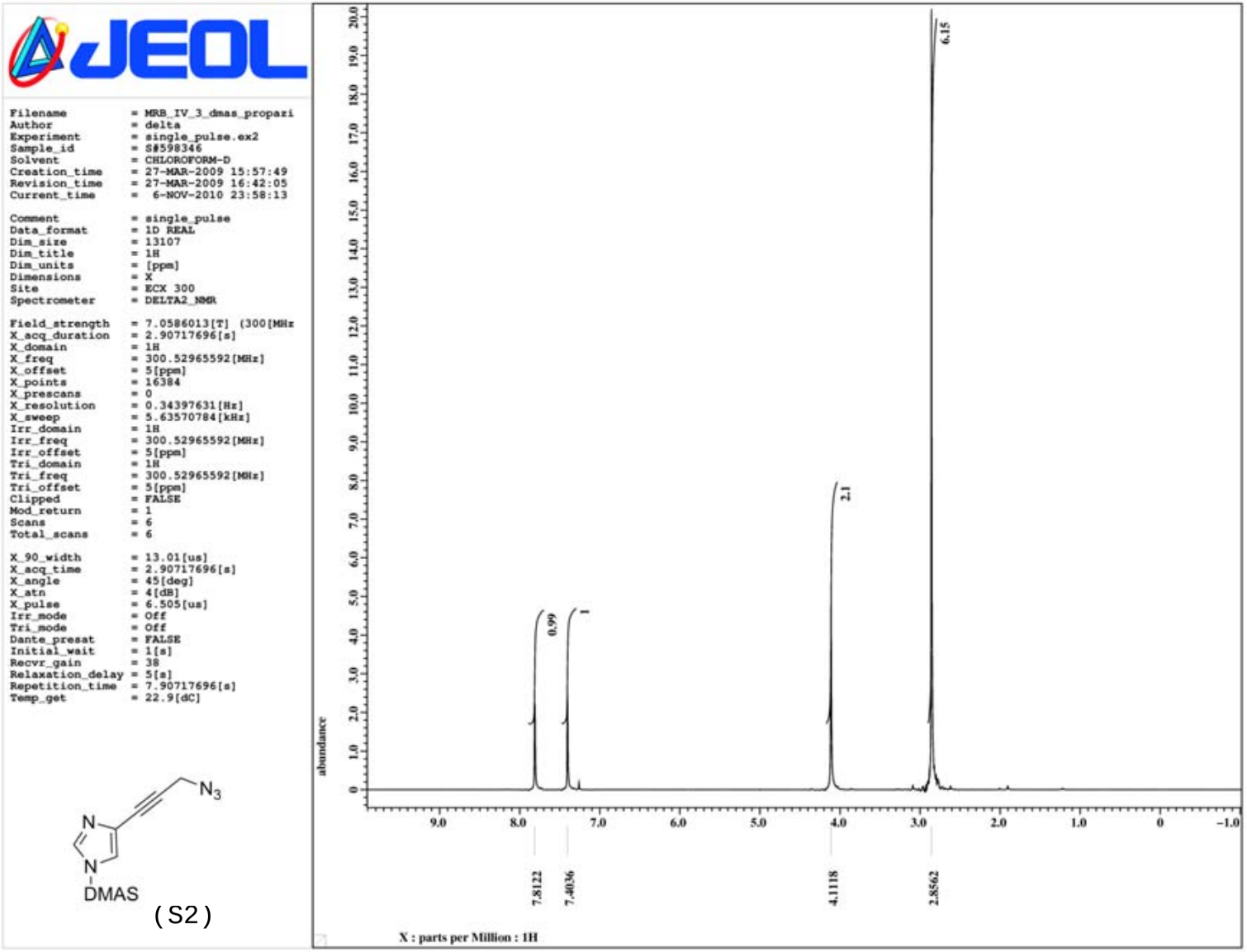
30.0
20.0
10.0
0
6.04
0.98
0.98
0.98
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
7.8420
7.3727
4.4952
4.4746
2.8677
2.8162
2.7967
X : parts per Million : 1H

```
Filename          = MRB_I_264_dmas_propar
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#747198
Solvent           = CHLOROFORM-D
Creation_time     = 15-FEB-2007 21:47:41
Revision_time     = 10-OCT-2010 10:14:11
Current_time      =  6-NOV-2010 22:30:16

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             = 324
Total_scans       = 324

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]
```

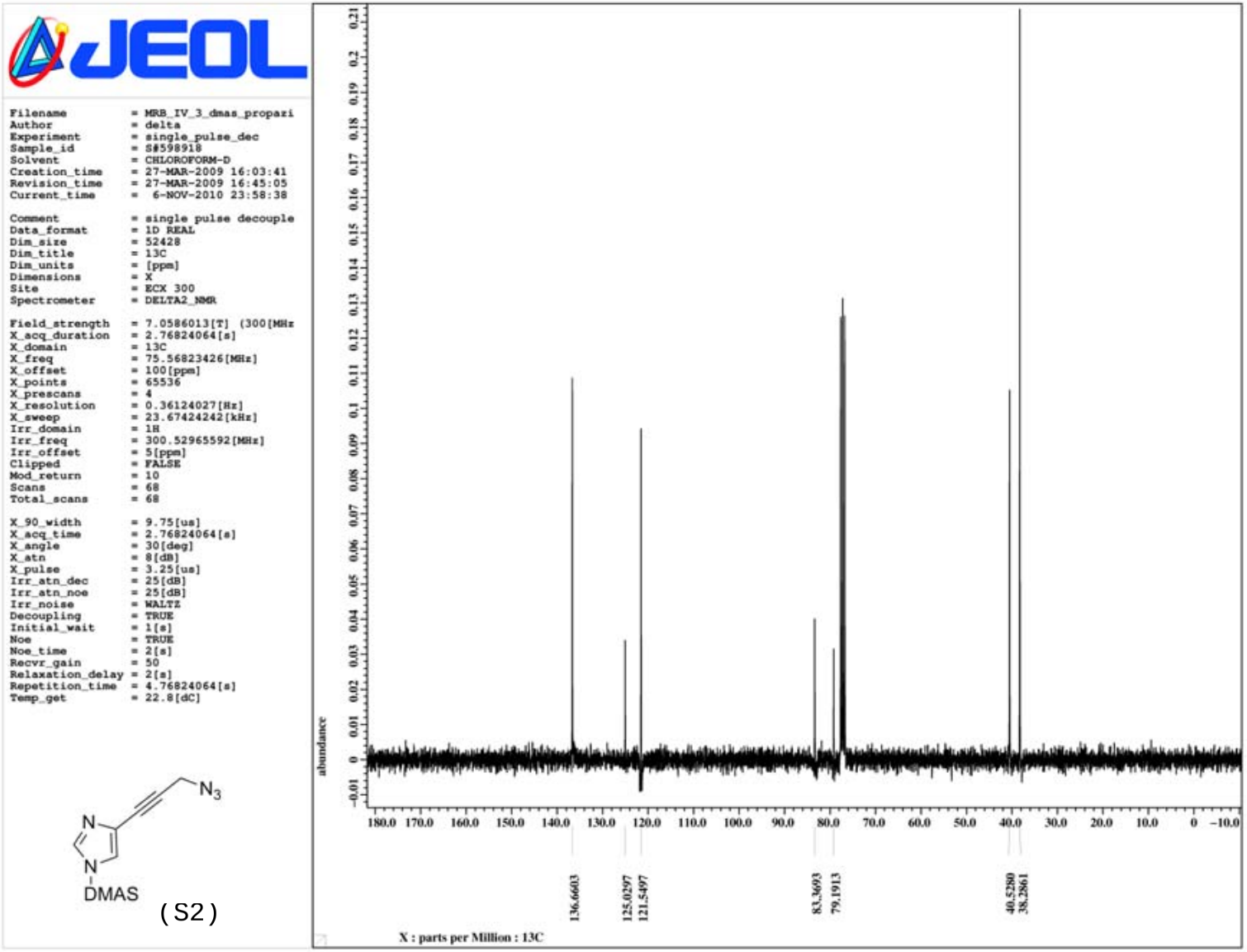


(S1)

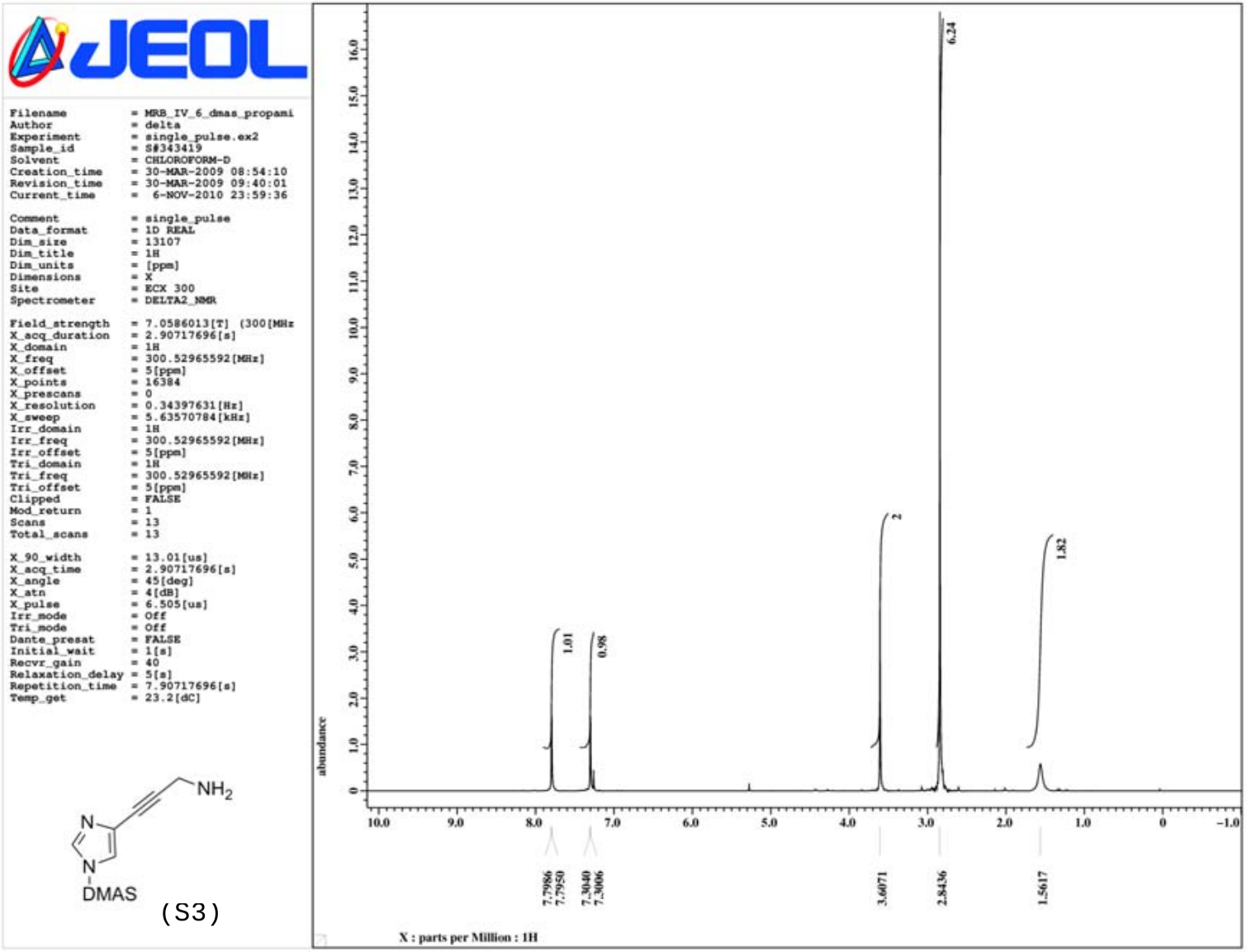


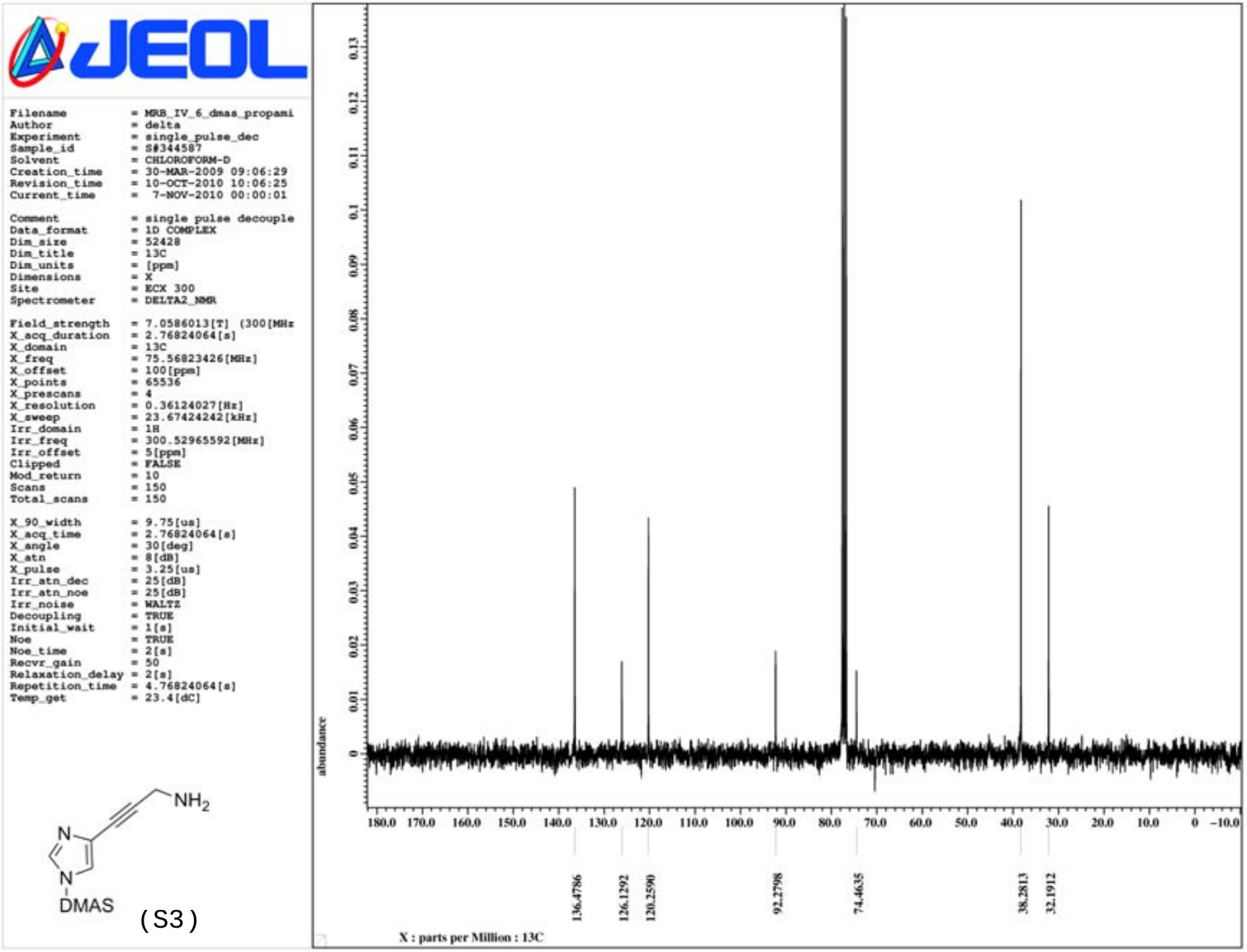

Filename = MRB_IV_3_dmas_propazi
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#598346
Solvent = CHLOROFORM-D
Creation_time = 27-MAR-2009 15:57:49
Revision_time = 27-MAR-2009 16:42:05
Current_time = 6-NOV-2010 23:58:13
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[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 = 6
Total_scans = 6
X_90_width = 13.01[us]
X_acq_time = 2.90717696[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 = 7.90717696[s]
Temp_get = 22.9[dC]
N3
N
N
DMAS
abundance
6.15
2.1
0.99
1
7.8122
7.4036
4.1118
2.8562
X : parts per Million : 1H

(S2)

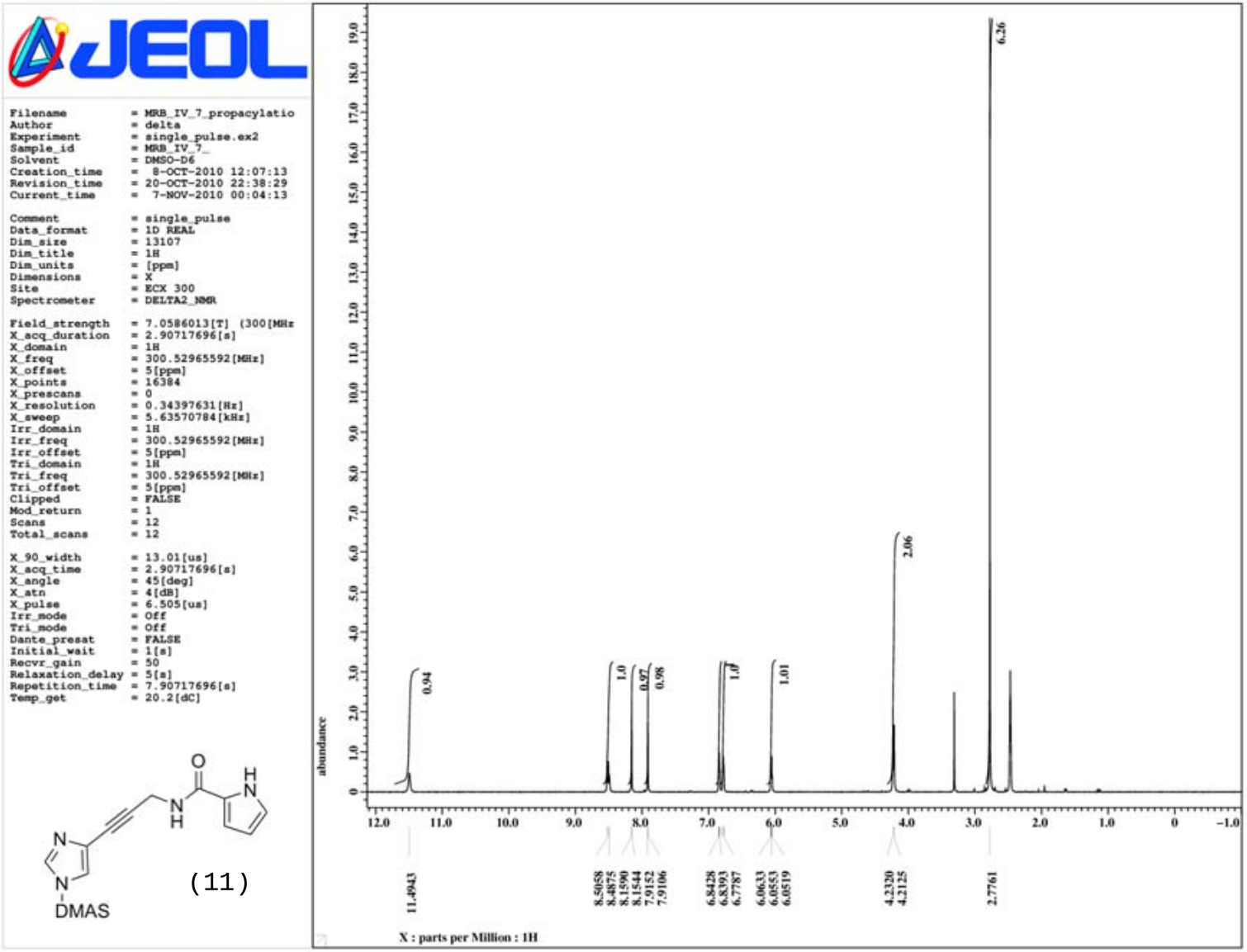

Filename = MRB_IV_3_dmas_propazi
Author = delta
Experiment = single_pulse_dec
Sample_id = S#598918
Solvent = CHLOROFORM-D
Creation_time = 27-MAR-2009 16:03:41
Revision_time = 27-MAR-2009 16:45:05
Current_time = 6-NOV-2010 23:58:38
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 52428
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 = 68
Total_scans = 68
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]
N3
N
N
DMAS
abundance
136.6603
125.0297
121.5497
83.3693
79.1913
40.5280
38.2861
X : parts per Million : 13C

(S2)


Filename = MRB_IV_6_dmas_propami
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#343419
Solvent = CHLOROFORM-D
Creation_time = 30-MAR-2009 08:54:10
Revision_time = 30-MAR-2009 09:40:01
Current_time = 6-NOV-2010 23:59:36
Comment = single_pulse
Data_format = 1D REAL
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 = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[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 = 13
Total_scans = 13
X_90_width = 13.01[us]
X_acq_time = 2.90717696[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 = 40
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 23.2[dC]
NH2
N
N
DMAS
(S3)
abundance
6.24
2
1.82
1.01
0.98
7.7986
7.7950
7.3040
7.3006
3.6071
2.8436
1.5617
X : parts per Million : 1H

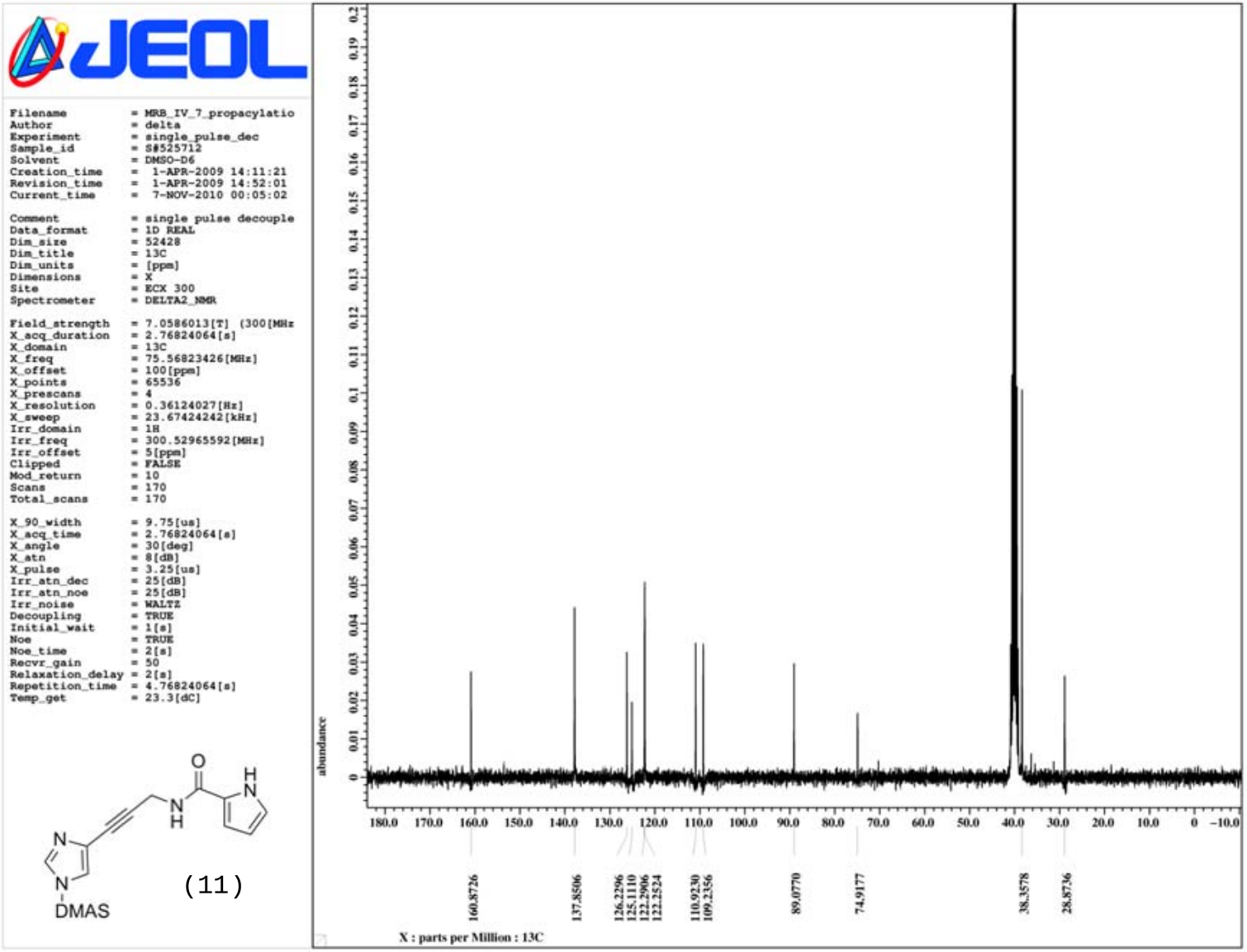

Filename = MRB_IV_6_dmas_propami
Author = delta
Experiment = single_pulse_dec
Sample_id = S#344587
Solvent = CHLOROFORM-D
Creation_time = 30-MAR-2009 09:06:29
Revision_time = 10-OCT-2010 10:06:25
Current_time = 7-NOV-2010 00:00:01
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 52428
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 300
Spectrometer = DELTA2_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 2.76824064[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 = 150
Total_scans = 150
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 = 23.4[dC]
NH2
N
N
DMAS
abundance
136.4786
126.1292
120.2590
92.2798
74.4635
38.2813
32.1912
X : parts per Million : 13C

(S3)



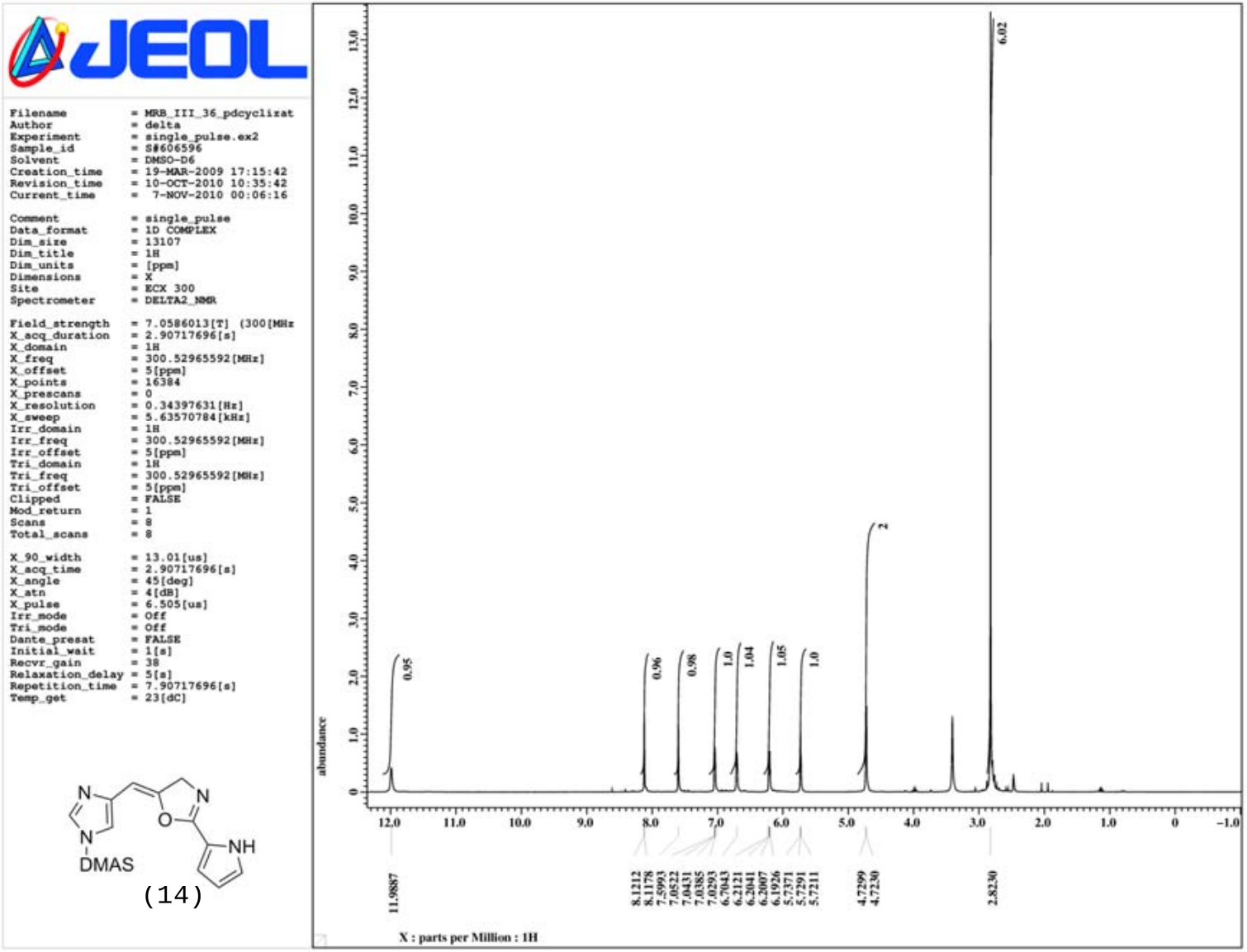
```
Filename          = MRB_IV_7_propacylatio
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = MRB_IV_7_
Solvent           = DMSO-D6
Creation_time     =  8-OCT-2010 12:07:13
Revision_time     = 20-OCT-2010 22:38:29
Current_time      =  7-NOV-2010 00:04:13

Comment           = single_pulse
Data_format       = 1D REAL
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    = 2.90717696[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.34397631[Hz]
X_sweep           = 5.63570784[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        = 2.90717696[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   = 7.90717696[s]
Temp_get          = 20.2[dC]
```

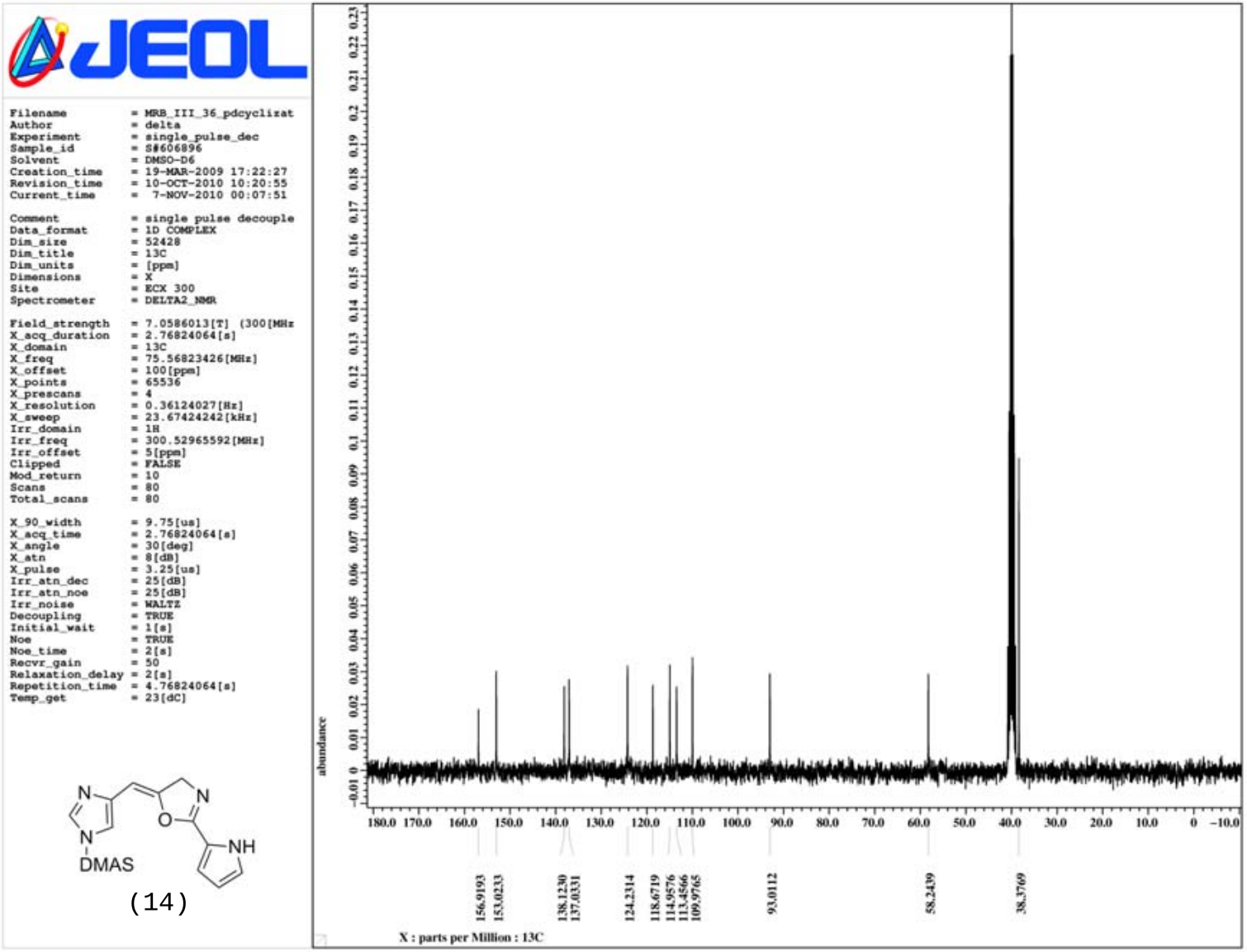

(11)



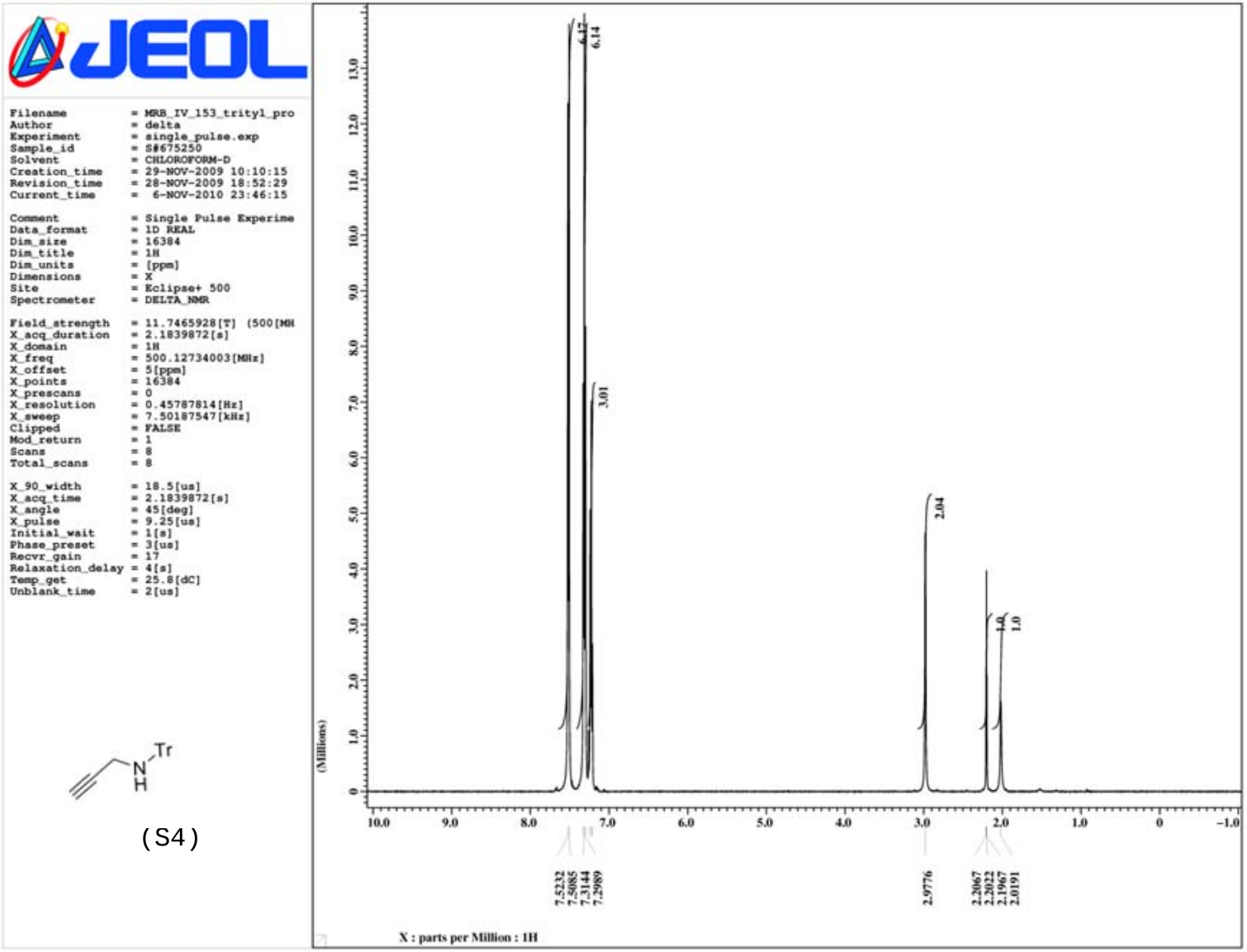
Filename	= MRB_IV_7_propacylatio
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#525712
Solvent	= DMSO-D6
Creation_time	= 1-APR-2009 14:11:21
Revision_time	= 1-APR-2009 14:52:01
Current_time	= 7-NOV-2010 00:05:02
Comment	= single pulse decouple
Data_format	= 1D REAL
Dim_size	= 52428
Dim_title	= 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	= 170
Total_scans	= 170
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]

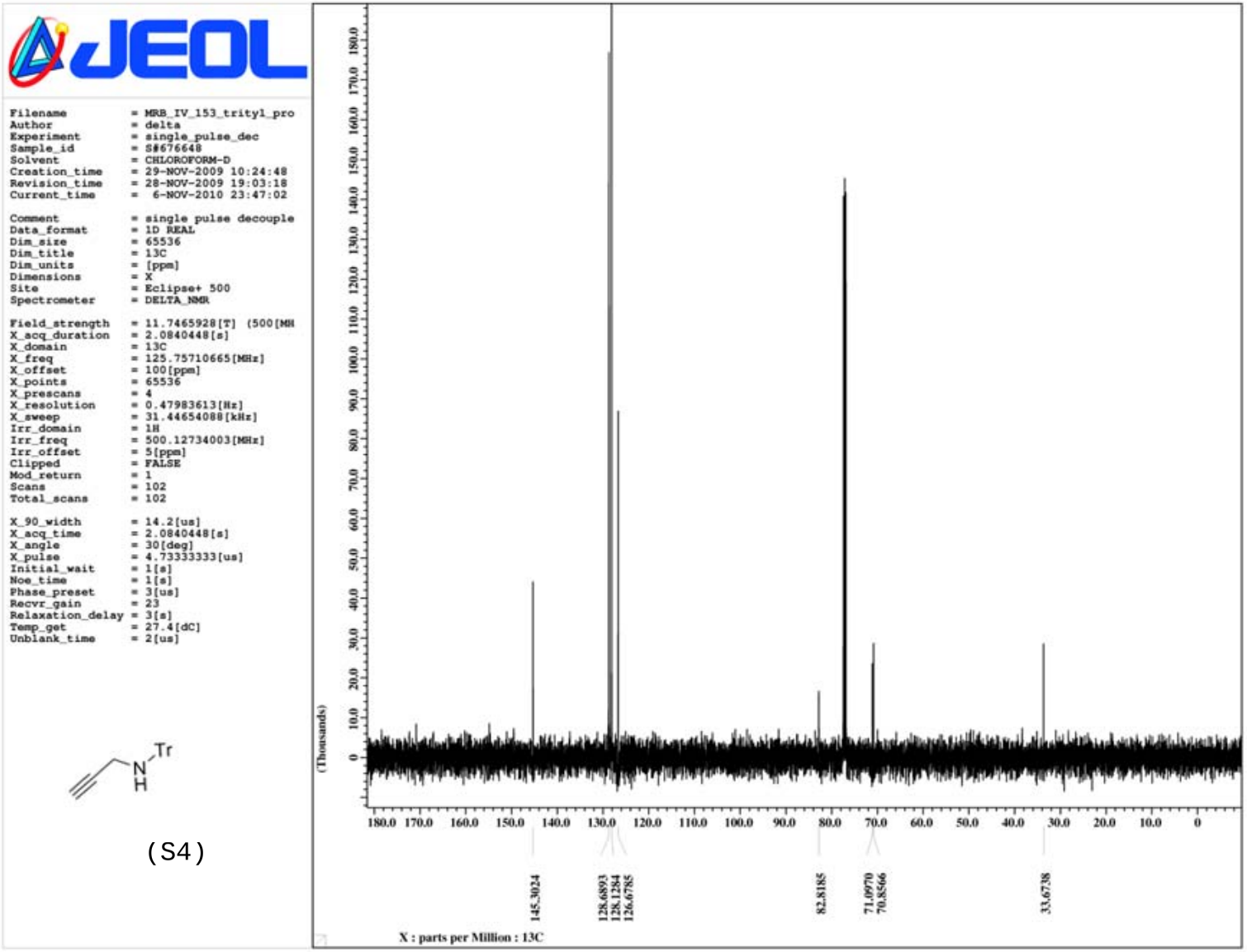
DMAS (11)


Filename = MRB_III_36_pdcyclizat
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#606596
Solvent = DMSO-D6
Creation_time = 19-MAR-2009 17:15:42
Revision_time = 10-OCT-2010 10:35:42
Current_time = 7-NOV-2010 00:06:16
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 = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[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 = 8
Total_scans = 8
X_90_width = 13.01[us]
X_acq_time = 2.90717696[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 = 7.90717696[s]
Temp_get = 23[dC]
N
N
O
N
NH
DMAS
(14)
abundance
0.95
0.96
0.98
1.0
1.04
1.05
1.0
2
6.02
11.9887
8.1212
8.1178
7.5993
7.0522
7.0431
7.0385
7.0293
6.7043
6.2121
6.2041
6.2007
6.1926
5.7371
5.7291
5.7211
4.7299
4.7230
2.8230
X : parts per Million : 1H


Filename = MRB_III_36_pdcyclizat
Author = delta
Experiment = single_pulse_dec
Sample_id = S#606896
Solvent = DMSO-D6
Creation_time = 19-MAR-2009 17:22:27
Revision_time = 10-OCT-2010 10:20:55
Current_time = 7-NOV-2010 00:07:51
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 = 80
Total_scans = 80
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[dC]
N
N
DMAS
O
N
NH
abundance
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 −10.0
156.9193
153.0233
138.1230
137.0331
124.2314
118.6719
114.9576
113.4566
109.9765
93.0112
58.2439
38.3769
X : parts per Million : 13C

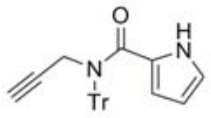
(14)


Filename = MRB_IV_153_trityl_pro
Author = delta
Experiment = single_pulse.exp
Sample_id = S#675250
Solvent = CHLOROFORM-D
Creation_time = 29-NOV-2009 10:10:15
Revision_time = 28-NOV-2009 18:52:29
Current_time = 6-NOV-2010 23:46:15
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 = 8
Total_scans = 8
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 = 17
Relaxation_delay = 4[s]
Temp_get = 25.8[dC]
Unblank_time = 2[us]
Tr
N
H
(S4)
6.17
6.14
3.01
2.04
1.4
1.0
(Millions)
7.5232
7.5085
7.3144
7.2989
2.9776
2.2067
2.2022
2.1967
2.0191
X : parts per Million : 1H

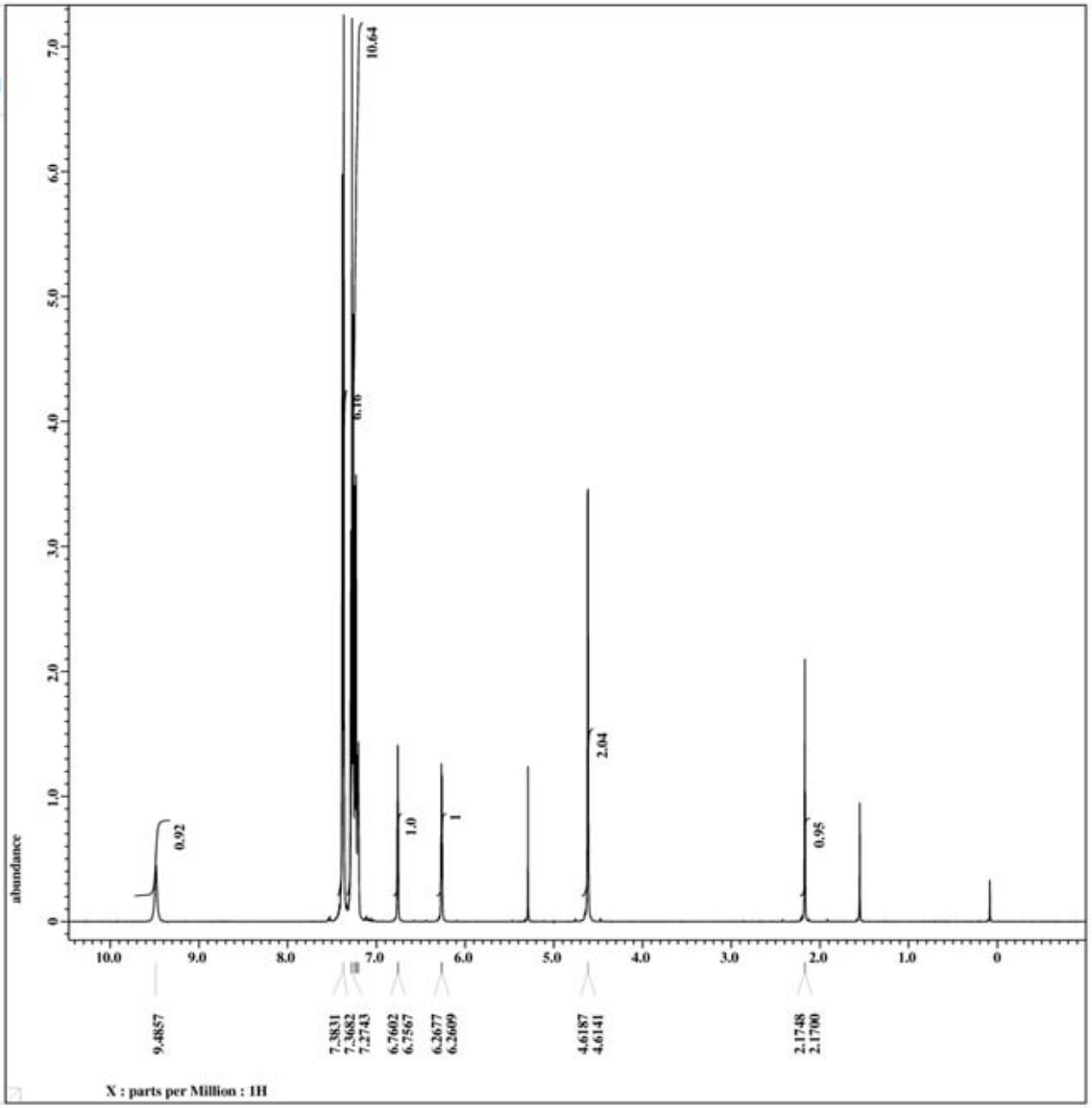

Filename = MRB_IV_153_trityl_pro
Author = delta
Experiment = single_pulse_dec
Sample_id = S#676648
Solvent = CHLOROFORM-D
Creation_time = 29-NOV-2009 10:24:48
Revision_time = 28-NOV-2009 19:03:18
Current_time = 6-NOV-2010 23:47:02
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 65536
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 = FALSE
Mod_return = 1
Scans = 102
Total_scans = 102
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 = 23
Relaxation_delay = 3[s]
Temp_get = 27.4[dC]
Unblank_time = 2[us]
Tr
N
H
(Thousands)
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
145.3024
128.6893
128.1284
126.6785
82.8185
71.0970
70.8566
33.6738
X : parts per Million : 13C

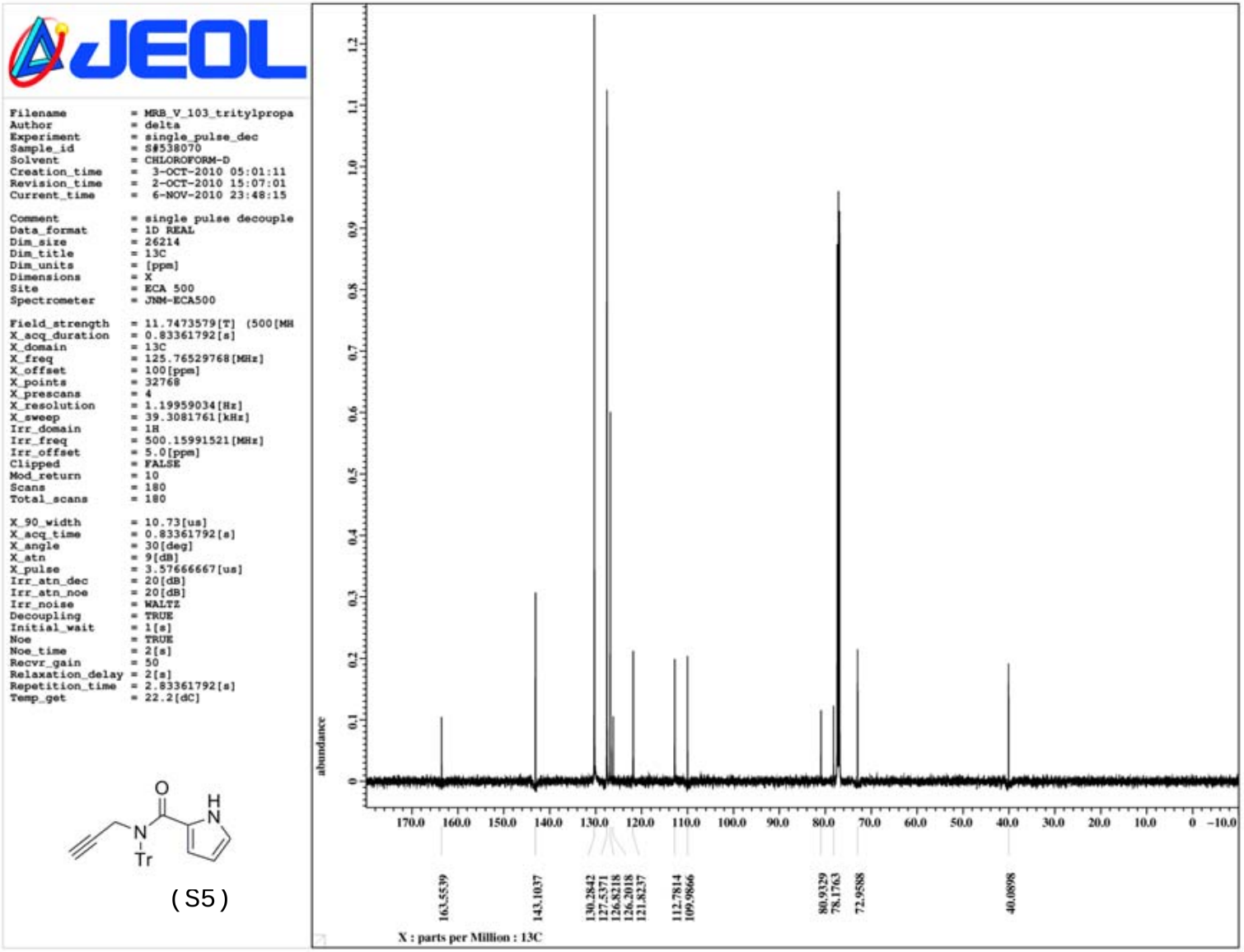
(S4)

Filename	= MRB_V_103_tritylpropa
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#537243
Solvent	= CHLOROFORM-D
Creation_time	= 3-OCT-2010 04:52:28
Revision_time	= 2-OCT-2010 15:02:42
Current_time	= 6-NOV-2010 23:47:50
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 21.6[dC]

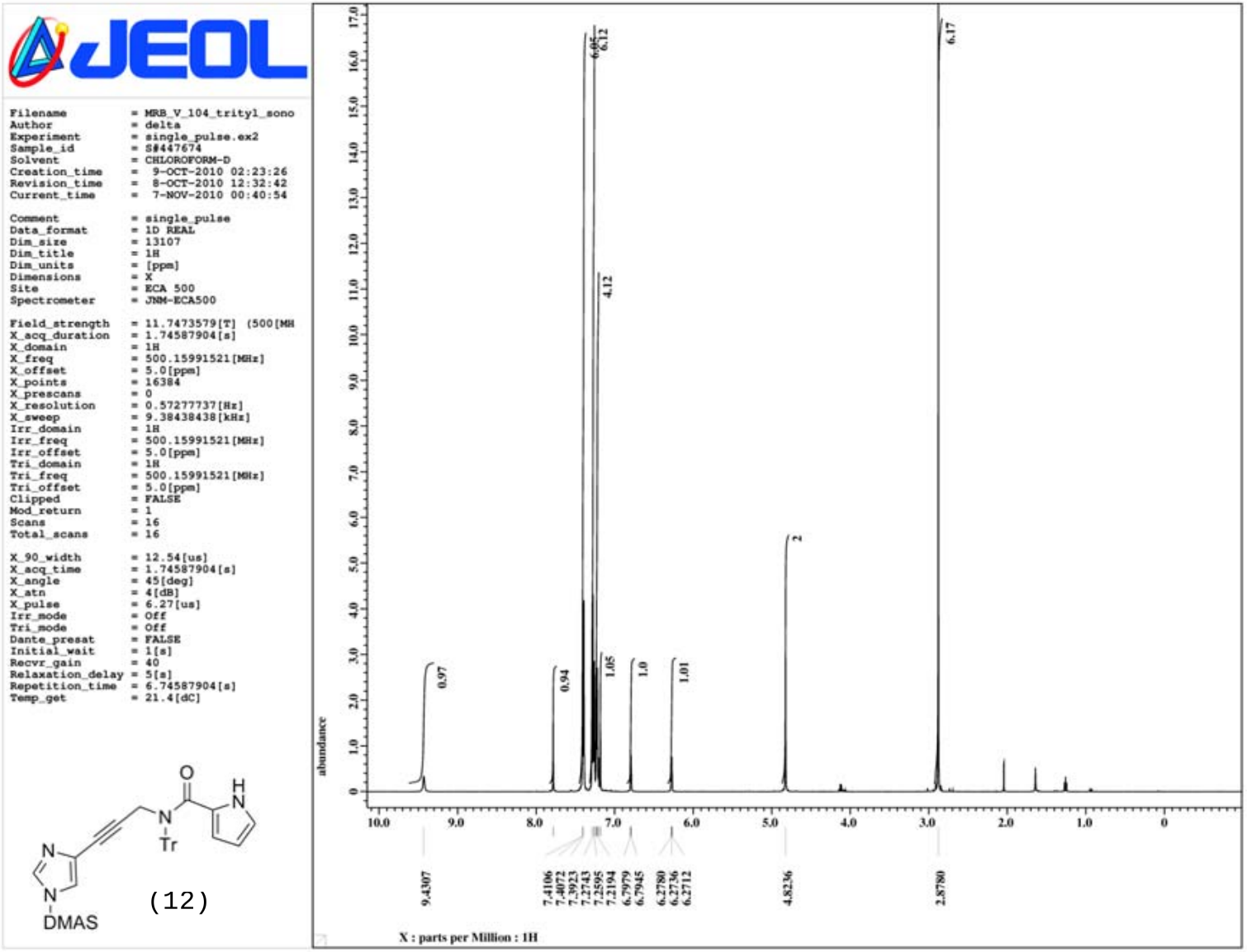


(S5)



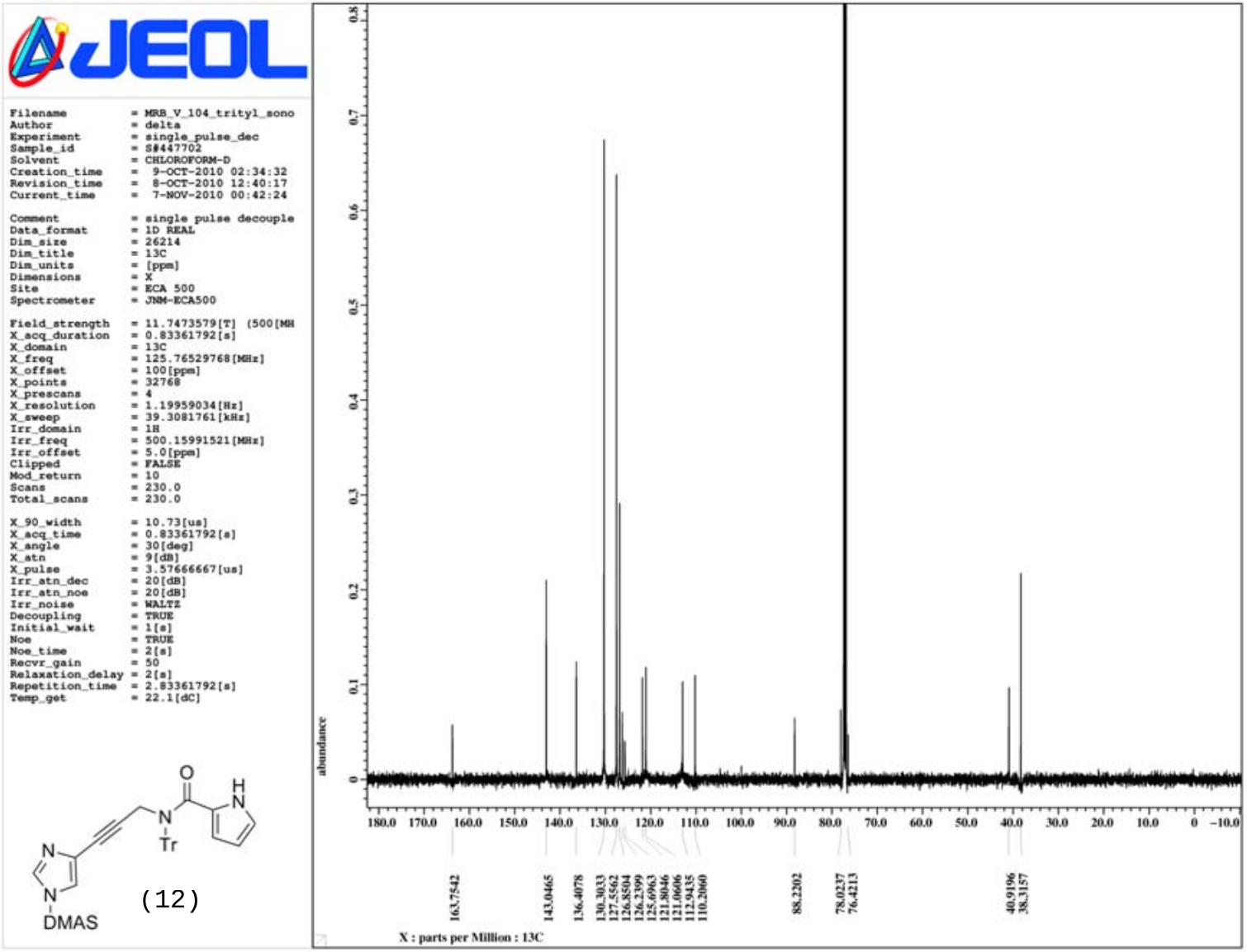

Filename = MRB_V_103_tritylpropa
Author = delta
Experiment = single_pulse_dec
Sample_id = S#538070
Solvent = CHLOROFORM-D
Creation_time = 3-OCT-2010 05:01:11
Revision_time = 2-OCT-2010 15:07:01
Current_time = 6-NOV-2010 23:48:15
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 180
Total_scans = 180
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.2[dC]
O
H
N
N
Tr
abundance
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 -10.0
163.5539
143.1037
130.2842
127.5371
126.8218
126.2018
121.8237
112.7814
109.9866
80.9329
78.1763
72.9588
40.0898
X : parts per Million : 13C

(S5)



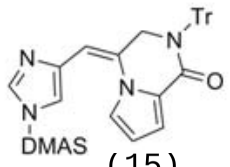
Filename	= MRB_V_104_trityl_sono
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#447674
Solvent	= CHLOROFORM-D
Creation_time	= 9-OCT-2010 02:23:26
Revision_time	= 8-OCT-2010 12:32:42
Current_time	= 7-NOV-2010 00:40:54
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 21.4[dC]

(12)

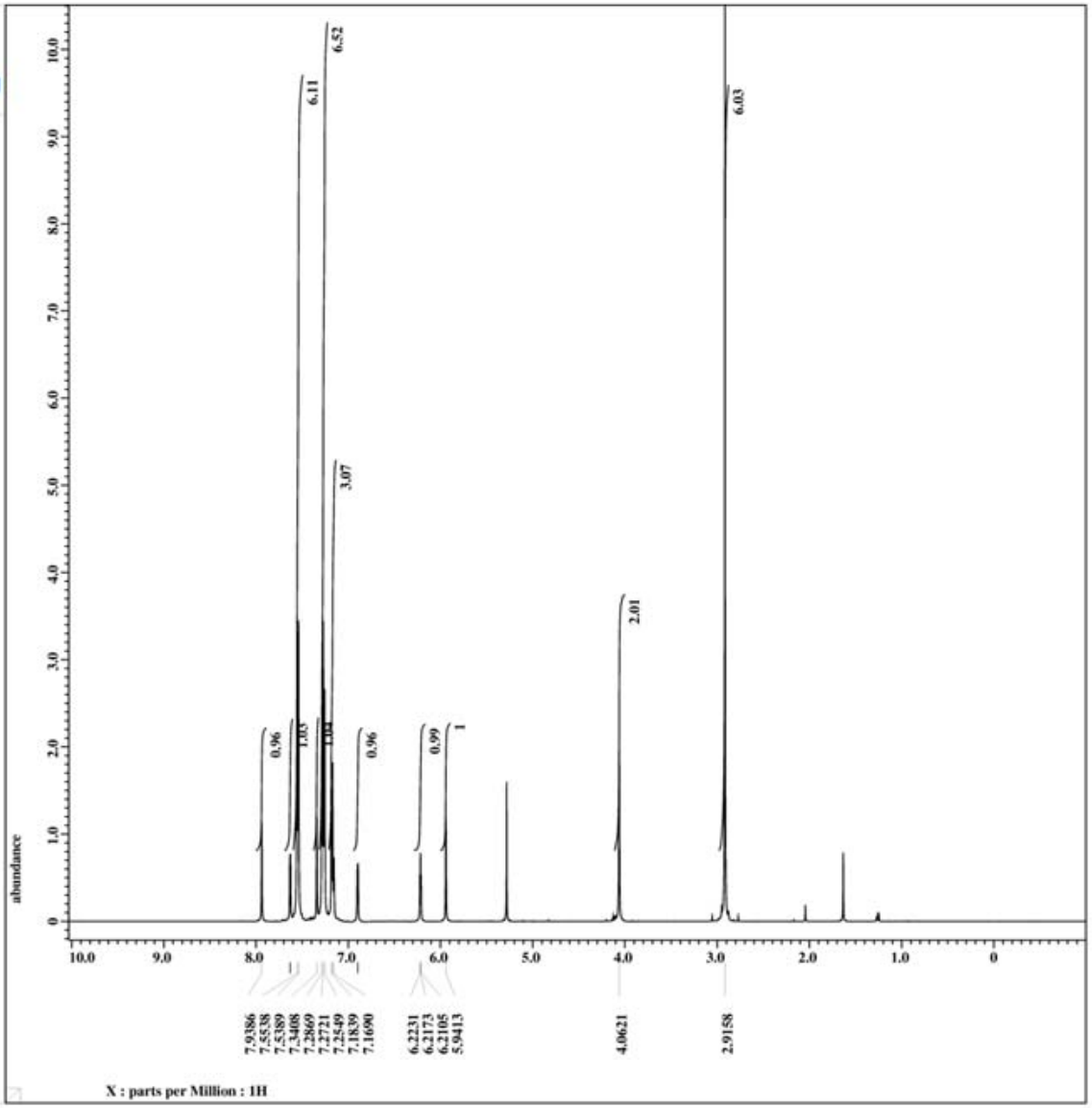

Filename = MRB_V_104_trityl_sono
Author = delta
Experiment = single_pulse_dec
Sample_id = S#447702
Solvent = CHLOROFORM-D
Creation_time = 9-OCT-2010 02:34:32
Revision_time = 8-OCT-2010 12:40:17
Current_time = 7-NOV-2010 00:42:24
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 230.0
Total_scans = 230.0
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.1[dC]
O
H
N
N
Tr
N
N
DMAS
(12)
abundance
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
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 −10.0
163.7542
143.0465
136.4078
130.3033
127.5562
126.8504
126.2399
125.6963
121.8046
121.0606
112.9435
110.2060
88.2202
78.0237
76.4213
40.9196
38.3157
X : parts per Million : 13C

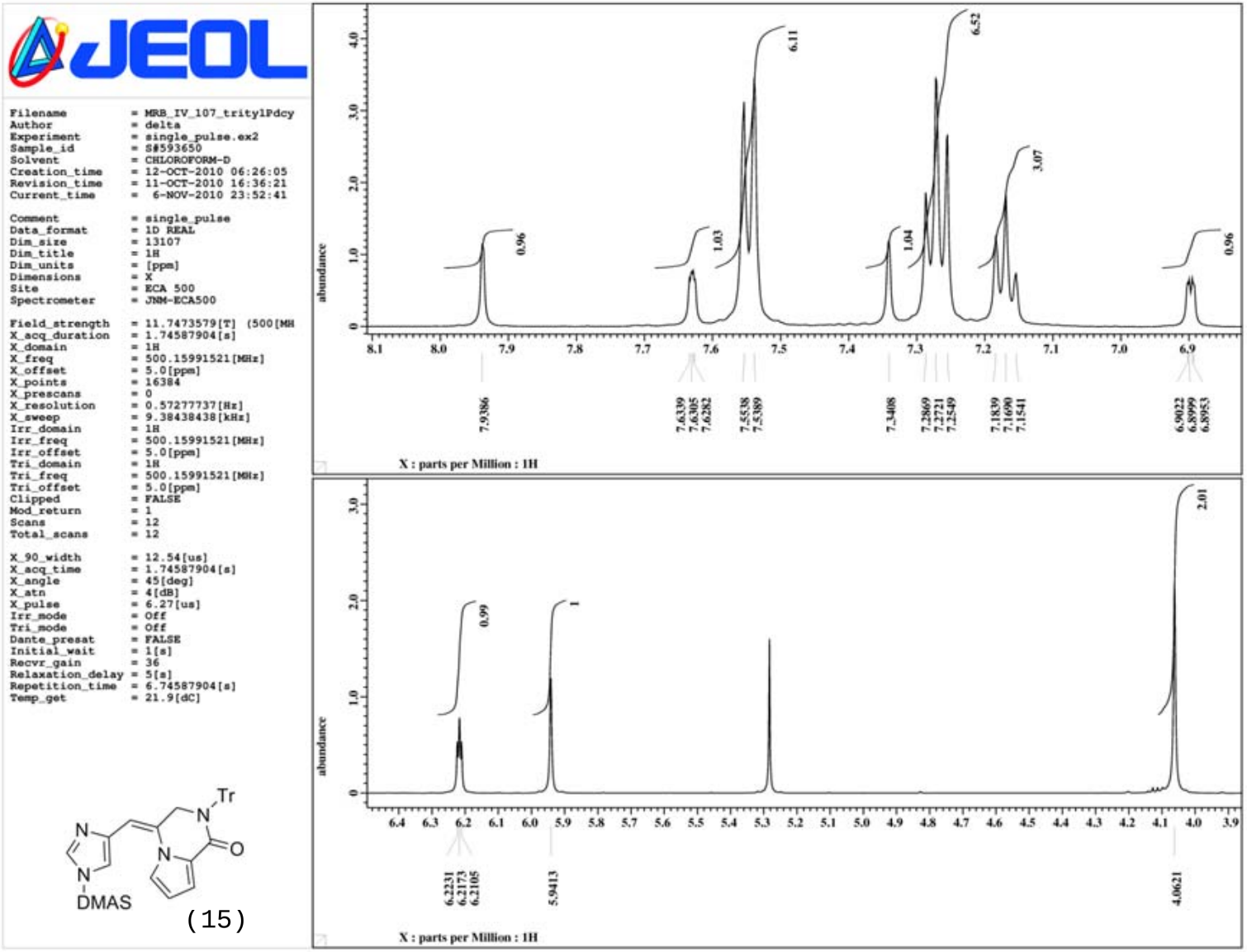
JEOL

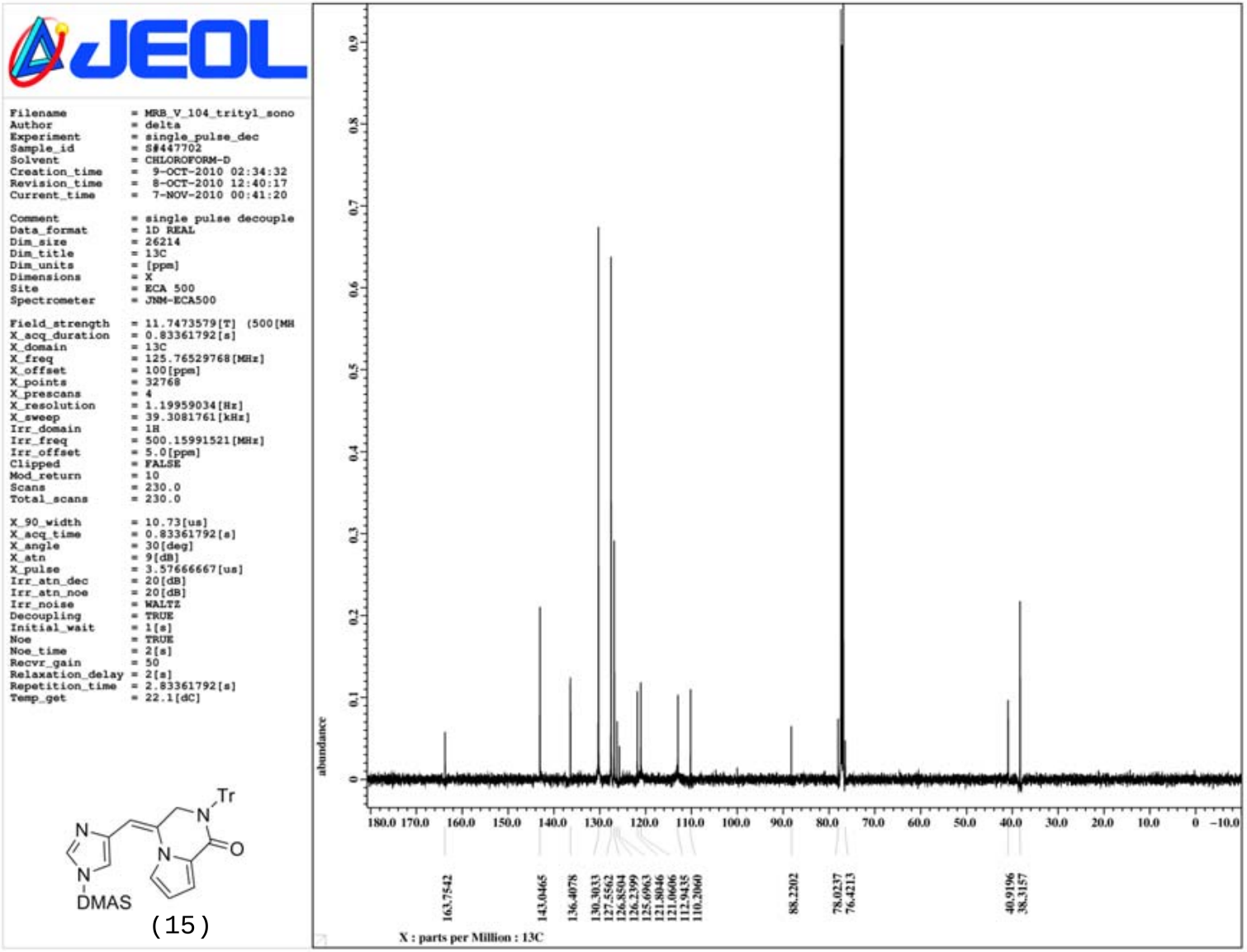
Filename	= MRB_IV_107_tritylPdcy
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#593650
Solvent	= CHLOROFORM-D
Creation_time	= 12-OCT-2010 06:26:05
Revision_time	= 11-OCT-2010 16:36:21
Current_time	= 7-NOV-2010 00:37:46
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 12
Total_scans	= 12
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 36
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 21.9[dC]



(15)

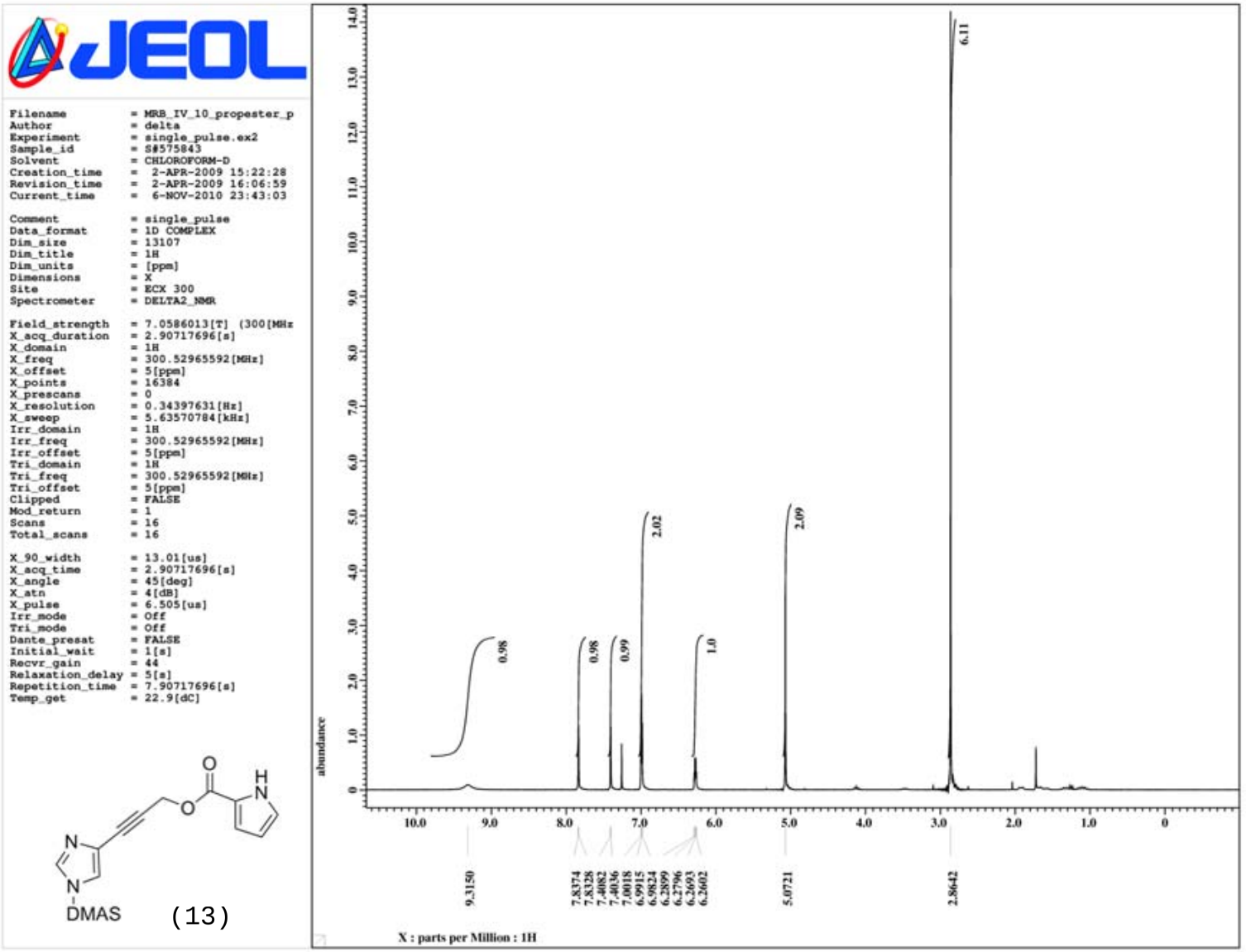


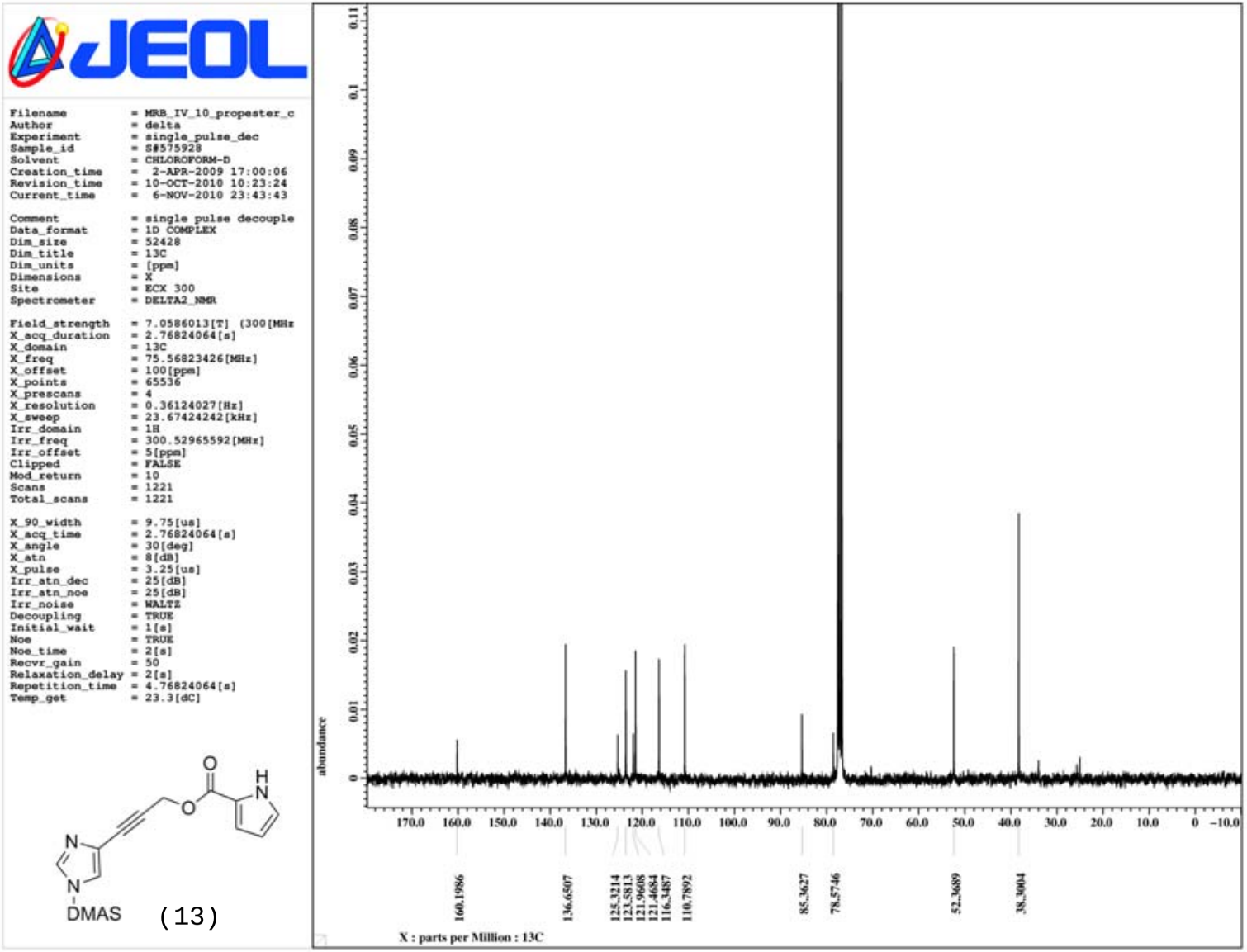

Filename = MRB_IV_107_tritylPdcy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#593650
Solvent = CHLOROFORM-D
Creation_time = 12-OCT-2010 06:26:05
Revision_time = 11-OCT-2010 16:36:21
Current_time = 6-NOV-2010 23:52:41
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 12
Total_scans = 12
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
N
N
N
Tr
O
DMAS
(15)
abundance
0.96
1.03
6.11
1.04
6.52
3.07
0.96
7.9386
7.6339
7.6305
7.6282
7.5538
7.5389
7.3408
7.2869
7.2721
7.2549
7.1839
7.1690
7.1541
6.9022
6.8999
6.8953
X : parts per Million : 1H
abundance
0.99
1
2.01
6.2231
6.2173
6.2105
5.9413
4.0621
X : parts per Million : 1H



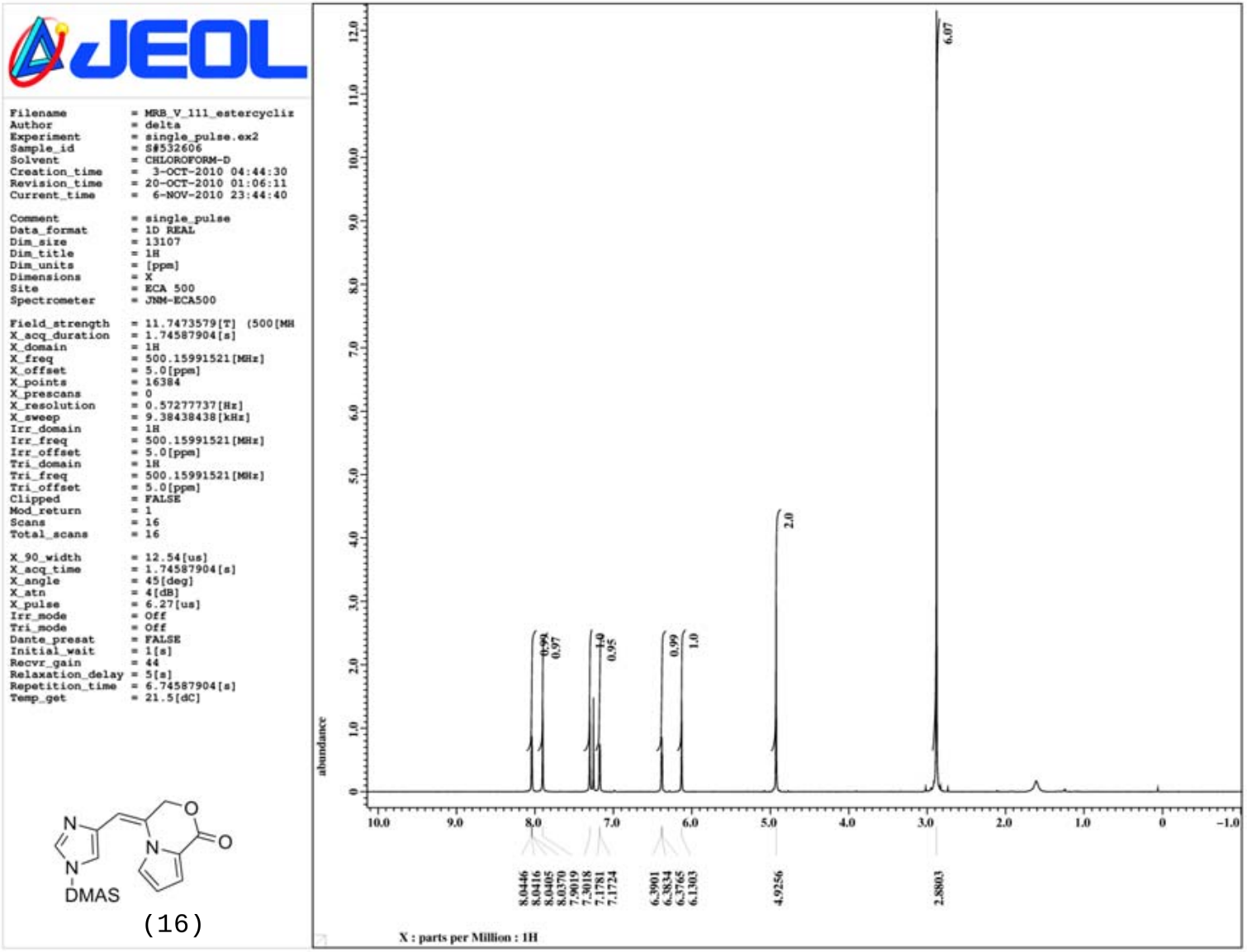
```
Filename          = MRB_V_104_trityl_sono
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#447702
Solvent           = CHLOROFORM-D
Creation_time     =  9-OCT-2010 02:34:32
Revision_time     =  8-OCT-2010 12:40:17
Current_time      =  7-NOV-2010 00:41:20

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 230.0
Total_scans       = 230.0

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[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   = 2.83361792[s]
Temp_get          = 22.1[dC]
```

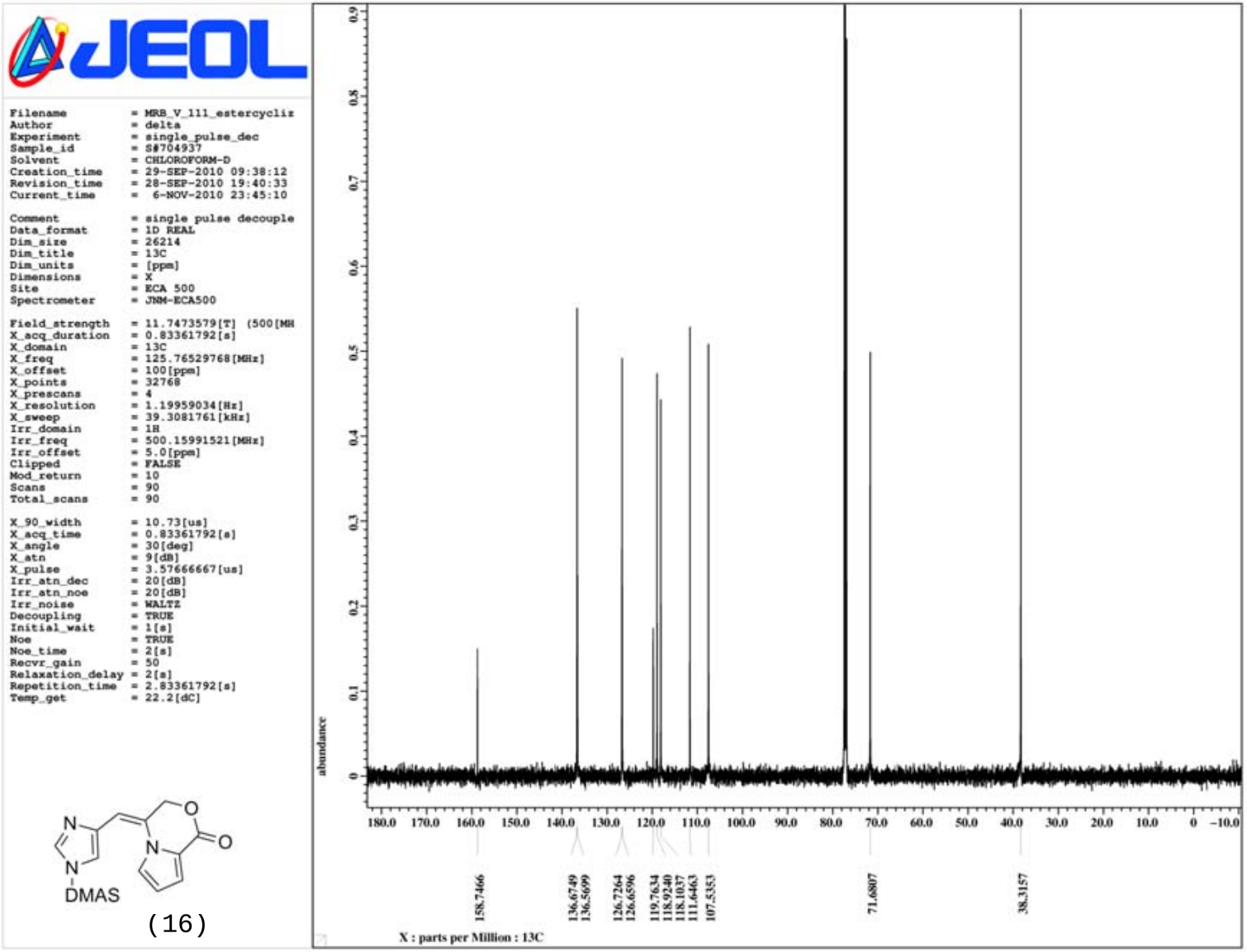

(15)


Filename = MRB_IV_10_propester_p
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#575843
Solvent = CHLOROFORM-D
Creation_time = 2-APR-2009 15:22:28
Revision_time = 2-APR-2009 16:06:59
Current_time = 6-NOV-2010 23:43:03
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 = 2.90717696[s]
X_domain = 1H
X_freq = 300.52965592[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.34397631[Hz]
X_sweep = 5.63570784[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 = 16
Total_scans = 16
X_90_width = 13.01[us]
X_acq_time = 2.90717696[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 = 44
Relaxation_delay = 5[s]
Repetition_time = 7.90717696[s]
Temp_get = 22.9[dC]
O
H
N
O
N
N
DMAS
(13)
abundance
0.98
0.98
0.99
2.02
1.0
2.09
6.11
9.3150
7.8374
7.8328
7.4082
7.4036
7.0018
6.9915
6.9824
6.2899
6.2796
6.2693
6.2602
5.0721
2.8642
X : parts per Million : 1H


Filename = MRB_IV_10_propester_c
Author = delta
Experiment = single_pulse_dec
Sample_id = S#575928
Solvent = CHLOROFORM-D
Creation_time = 2-APR-2009 17:00:06
Revision_time = 10-OCT-2010 10:23:24
Current_time = 6-NOV-2010 23:43:43
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 = 1221
Total_scans = 1221
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]
O
O
H
N
N
N
DMAS
abundance
0.11
0.1
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
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
−10.0
160.1986
136.6507
125.3214
123.5813
121.9608
121.4684
116.3487
110.7892
85.3627
78.5746
52.3689
38.3004
X : parts per Million : 13C

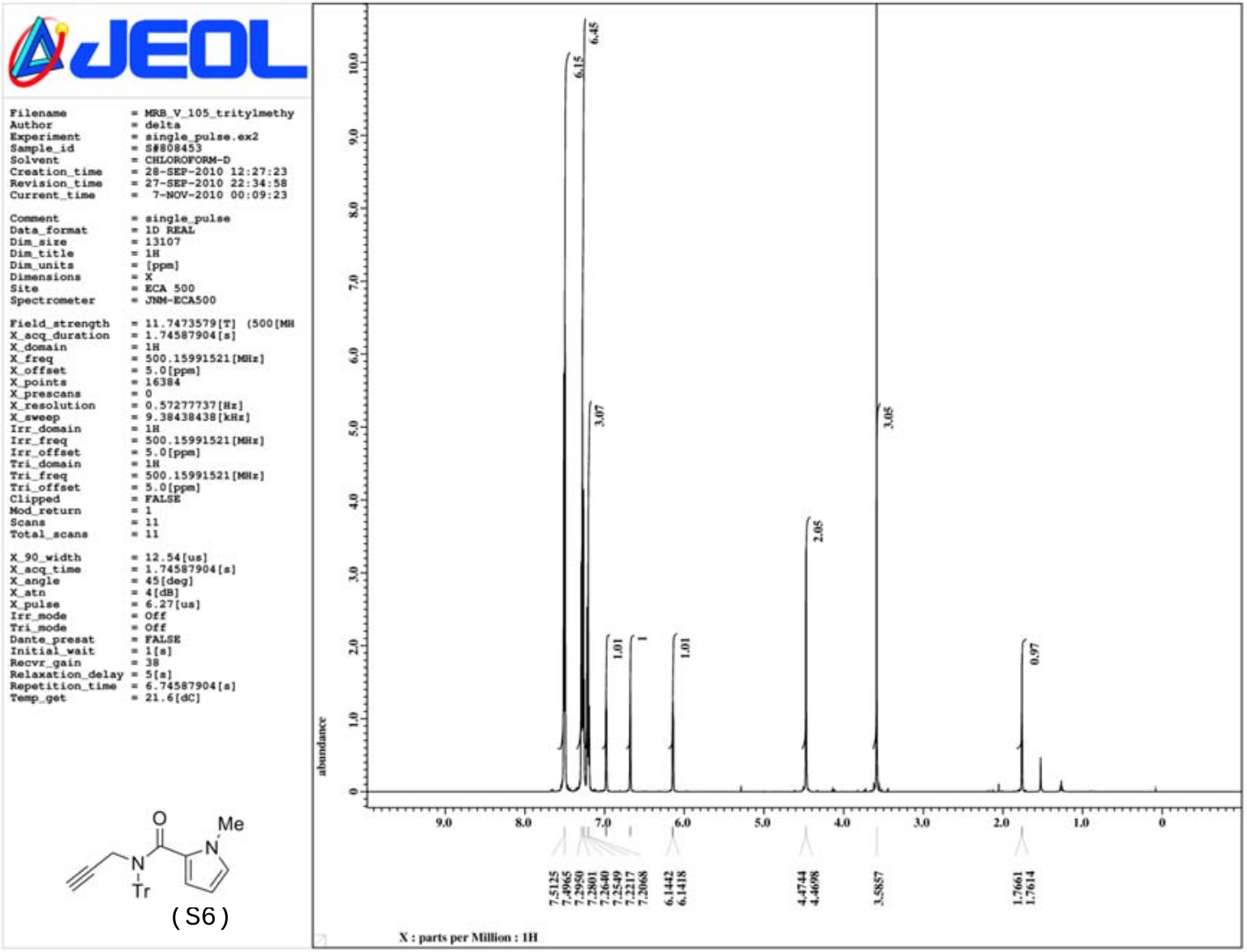
(13)

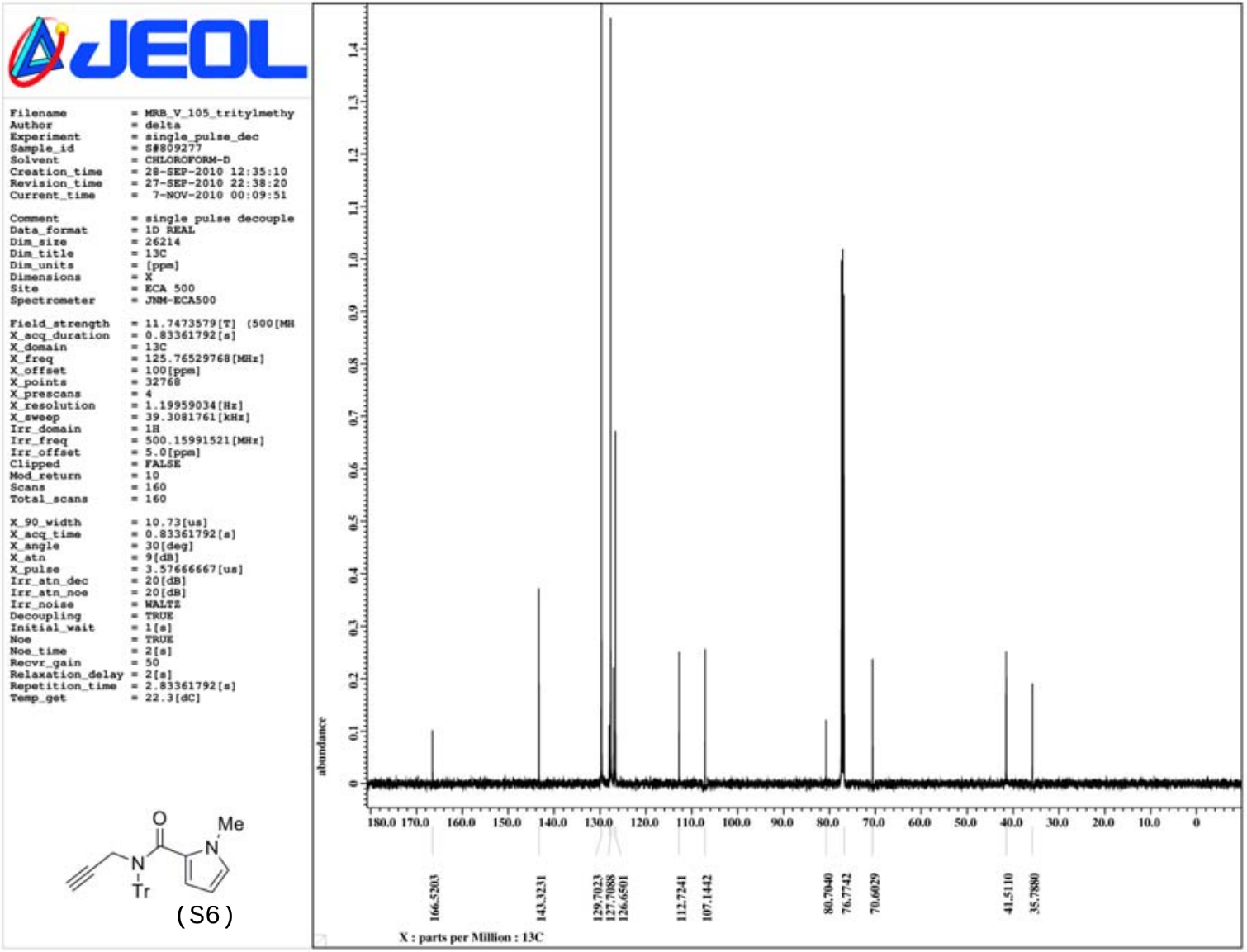

Filename = MRB_V_111_estercyclic
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#532606
Solvent = CHLOROFORM-D
Creation_time = 3-OCT-2010 04:44:30
Revision_time = 20-OCT-2010 01:06:11
Current_time = 6-NOV-2010 23:44:40
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 44
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.5[dC]
N
N
DMAS
O
N
O
(16)
abundance
6.07
2.0
0.94
0.97
1.0
0.95
0.99
1.0
8.0446
8.0416
8.0405
8.0370
7.9019
7.3018
7.1781
7.1724
6.3901
6.3834
6.3765
6.1303
4.9256
2.8803
X : parts per Million : 1H



```
Filename          = MRB_V_111_estercycliz
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#704937
Solvent           = CHLOROFORM-D
Creation_time     = 29-SEP-2010 09:38:12
Revision_time     = 28-SEP-2010 19:40:33
Current_time      =  6-NOV-2010 23:45:10

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 90
Total_scans       = 90

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[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   = 2.83361792[s]
Temp_get          = 22.2[dC]
```

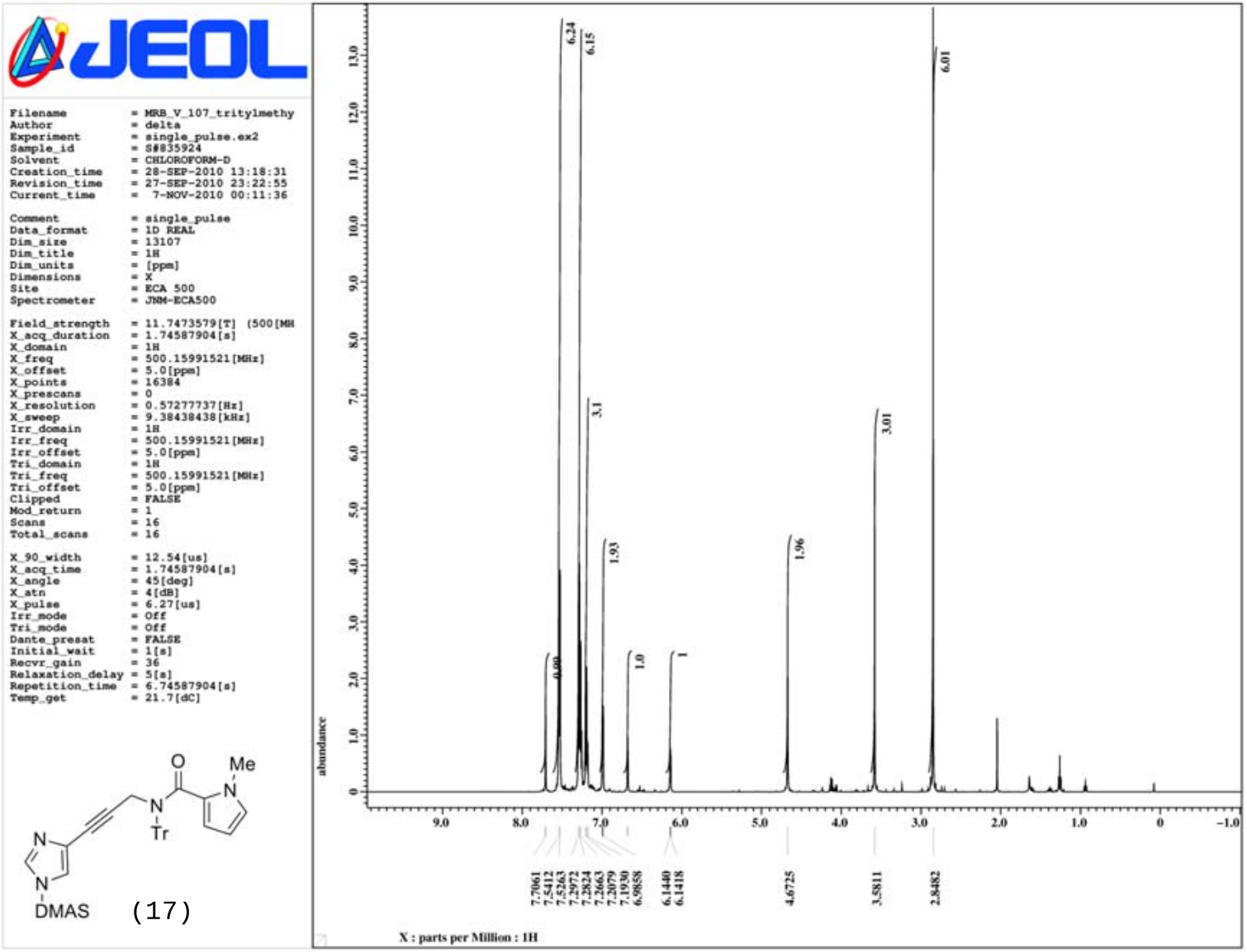

(16)

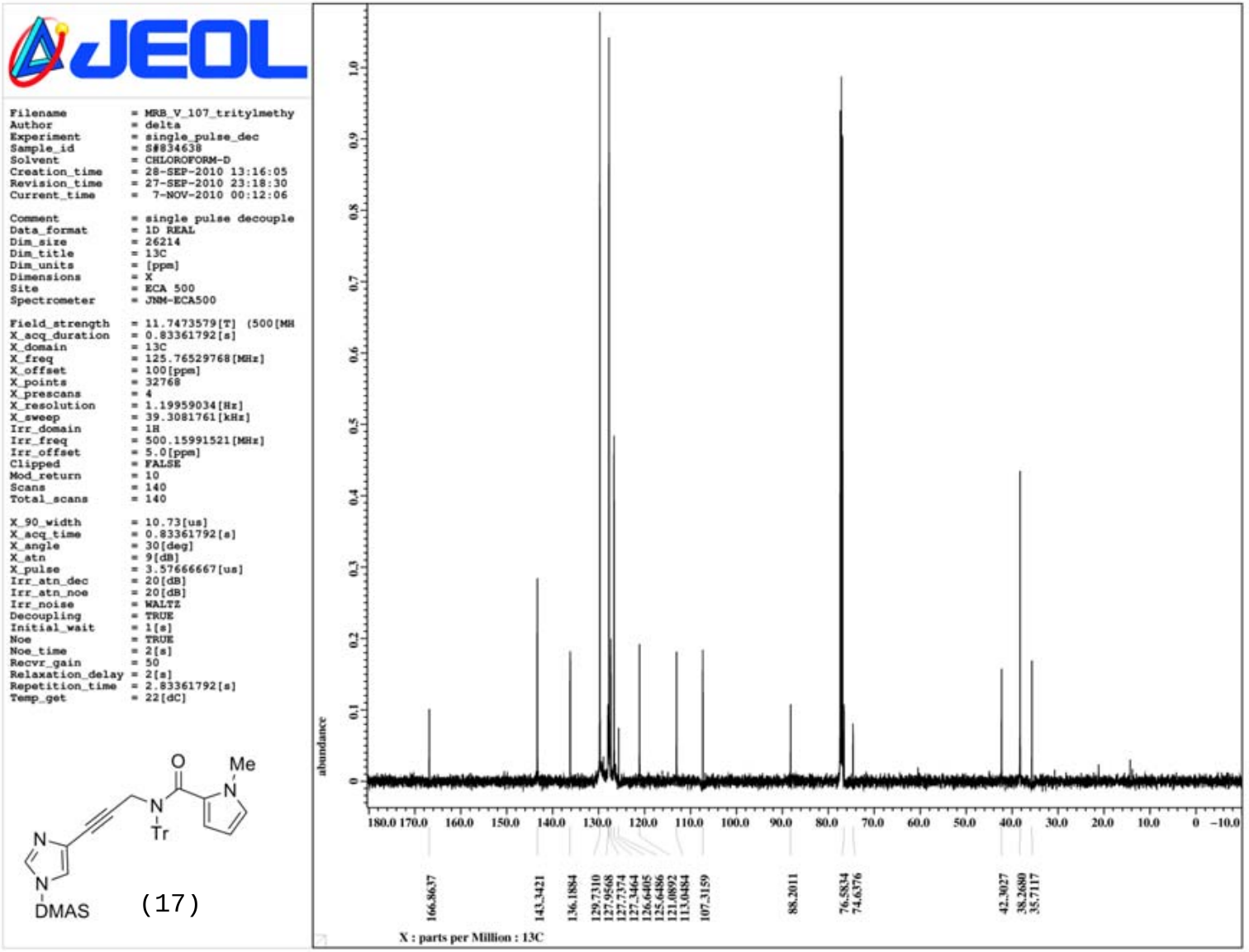

Filename = MRB_V_105_tritylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#808453
Solvent = CHLOROFORM-D
Creation_time = 28-SEP-2010 12:27:23
Revision_time = 27-SEP-2010 22:34:58
Current_time = 7-NOV-2010 00:09:23
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 11
Total_scans = 11
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 38
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.6[dC]
O
Me
N
N
Tr
(S6)
abundance
6.45
6.15
3.07
1.01
1
1.01
2.05
3.05
0.97
7.5125
7.4965
7.2950
7.2801
7.2640
7.2549
7.2317
7.2068
6.1442
6.1418
4.4744
4.4698
3.5857
1.7661
1.7614
X : parts per Million : 1H

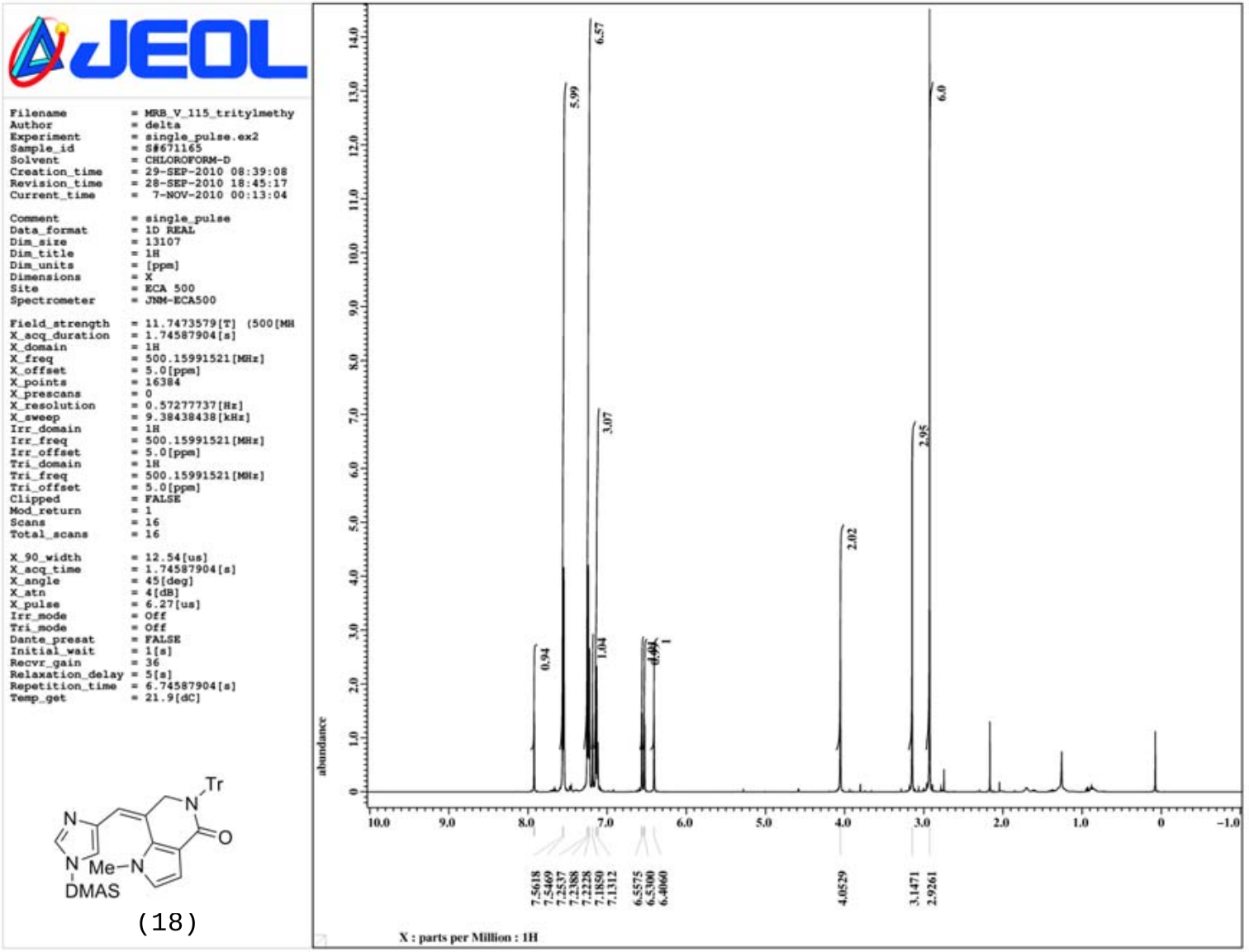


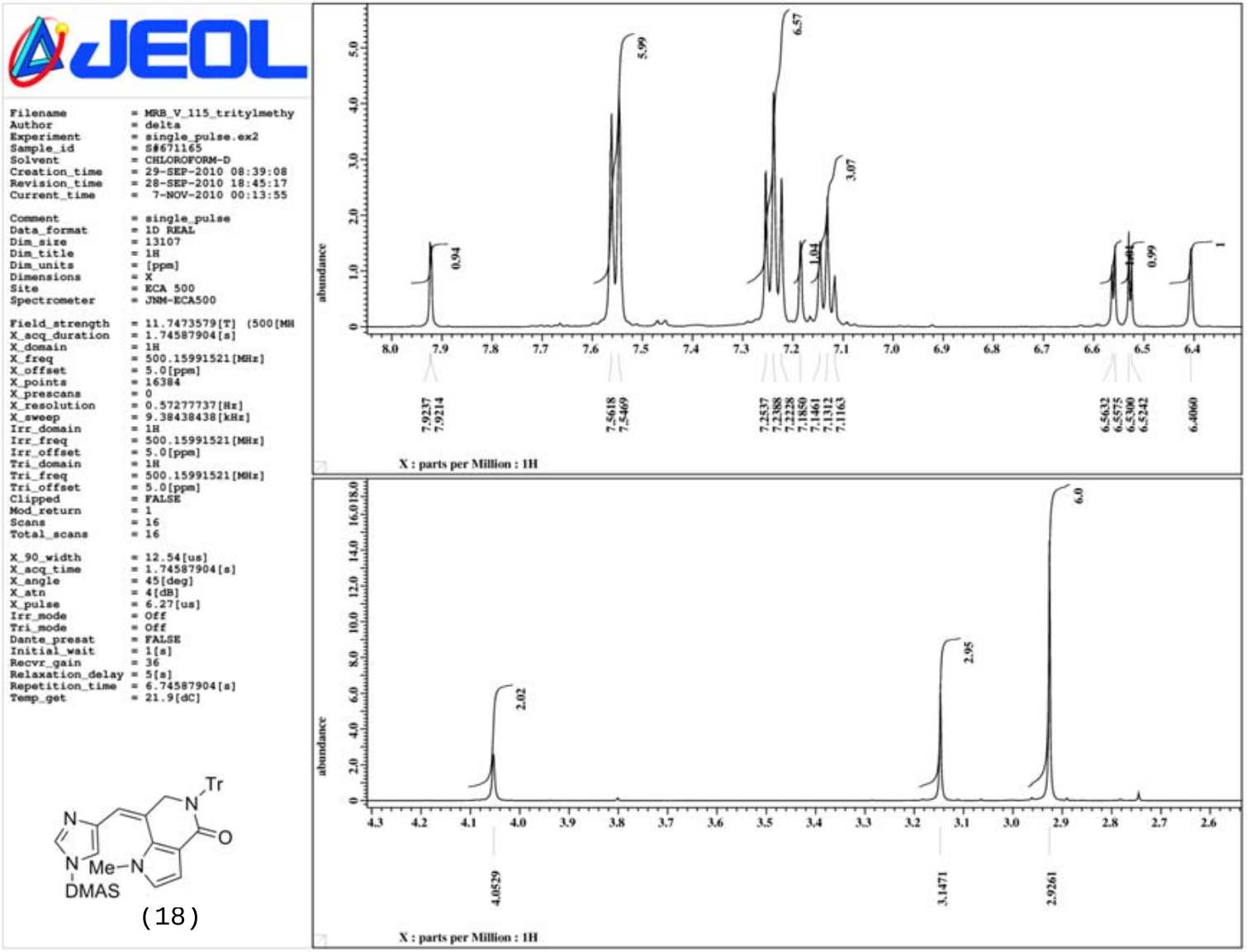
```
Filename          = MRB_V_105_tritylmethy
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#809277
Solvent           = CHLOROFORM-D
Creation_time     = 28-SEP-2010 12:35:10
Revision_time     = 27-SEP-2010 22:38:20
Current_time      =  7-NOV-2010 00:09:51

Comment           = single pulse decouple
Data_format       = 1D REAL
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 160
Total_scans       = 160

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[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   = 2.83361792[s]
Temp_get          = 22.3[dC]
```


(S6)

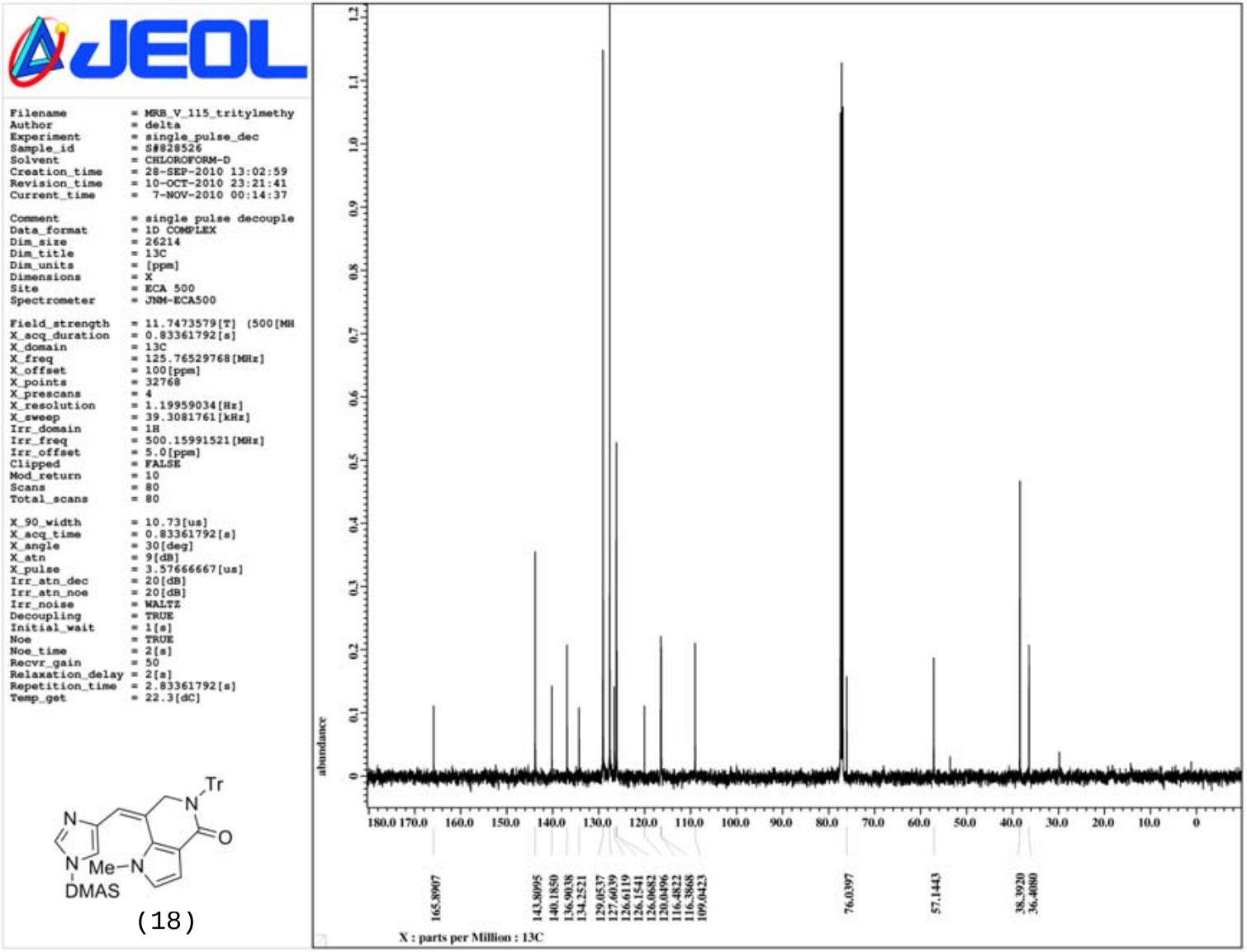

Filename = MRB_V_107_tritylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#835924
Solvent = CHLOROFORM-D
Creation_time = 28-SEP-2010 13:18:31
Revision_time = 27-SEP-2010 23:22:55
Current_time = 7-NOV-2010 00:11:36
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.7[dC]
O
Me
N
N
Tr
N
N
DMAS
(17)
abundance
6.24
6.15
3.1
1.93
1.0
1
1.96
3.01
6.01
0.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
7.7061
7.5412
7.5363
7.2972
7.2824
7.2663
7.2079
7.1930
6.9858
6.1440
6.1418
4.6725
3.5811
2.8482
X : parts per Million : 1H


Filename = MRB_V_107_tritylmethy
Author = delta
Experiment = single_pulse_dec
Sample_id = S#834638
Solvent = CHLOROFORM-D
Creation_time = 28-SEP-2010 13:16:05
Revision_time = 27-SEP-2010 23:18:30
Current_time = 7-NOV-2010 00:12:06
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 140
Total_scans = 140
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22[dC]
O
Me
N
N
Tr
N
N
DMAS
(17)
abundance
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 −10.0
166.8637
143.3421
136.1884
129.7310
127.9568
127.7374
127.3464
126.6405
125.6486
121.0892
113.0484
107.3159
88.2011
76.5834
74.6376
42.3027
38.2680
35.7117
X : parts per Million : 13C


Filename = MRB_V_115_tritylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#671165
Solvent = CHLOROFORM-D
Creation_time = 29-SEP-2010 08:39:08
Revision_time = 28-SEP-2010 18:45:17
Current_time = 7-NOV-2010 00:13:04
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
Tr
N
N
O
N
Me
N
DMAS
(18)
abundance
6.57
5.99
6.0
3.07
2.95
2.02
0.94
1.04
1
7.5618
7.5469
7.2537
7.2388
7.2228
7.1850
7.1312
6.5575
6.5300
6.4060
4.0529
3.1471
2.9261
X : parts per Million : 1H

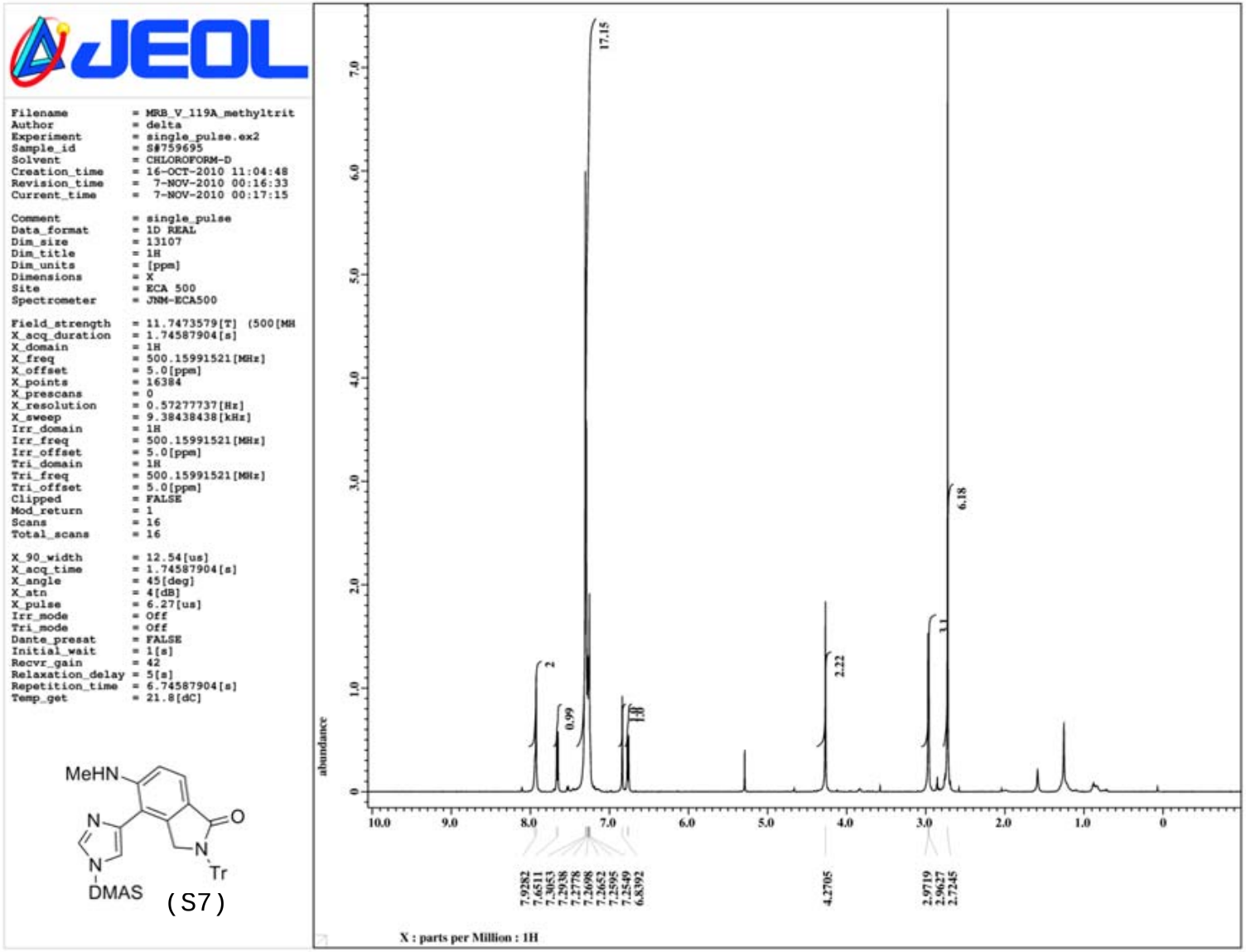

Filename = MRB_V_115_tritylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#671165
Solvent = CHLOROFORM-D
Creation_time = 29-SEP-2010 08:39:08
Revision_time = 28-SEP-2010 18:45:17
Current_time = 7-NOV-2010 00:13:55
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 36
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.9[dC]
abundance
X : parts per Million : 1H
7.9237
7.9214
7.5618
7.5469
7.2537
7.2388
7.2228
7.1850
7.1461
7.1312
7.1163
6.5632
6.5575
6.5300
6.5242
6.4060
0.94
5.99
6.57
3.07
1.04
1.01
0.99
1
abundance
X : parts per Million : 1H
4.0529
3.1471
2.9261
2.02
2.95
6.0
N
N
Me
N
Tr
N
O
DMAS

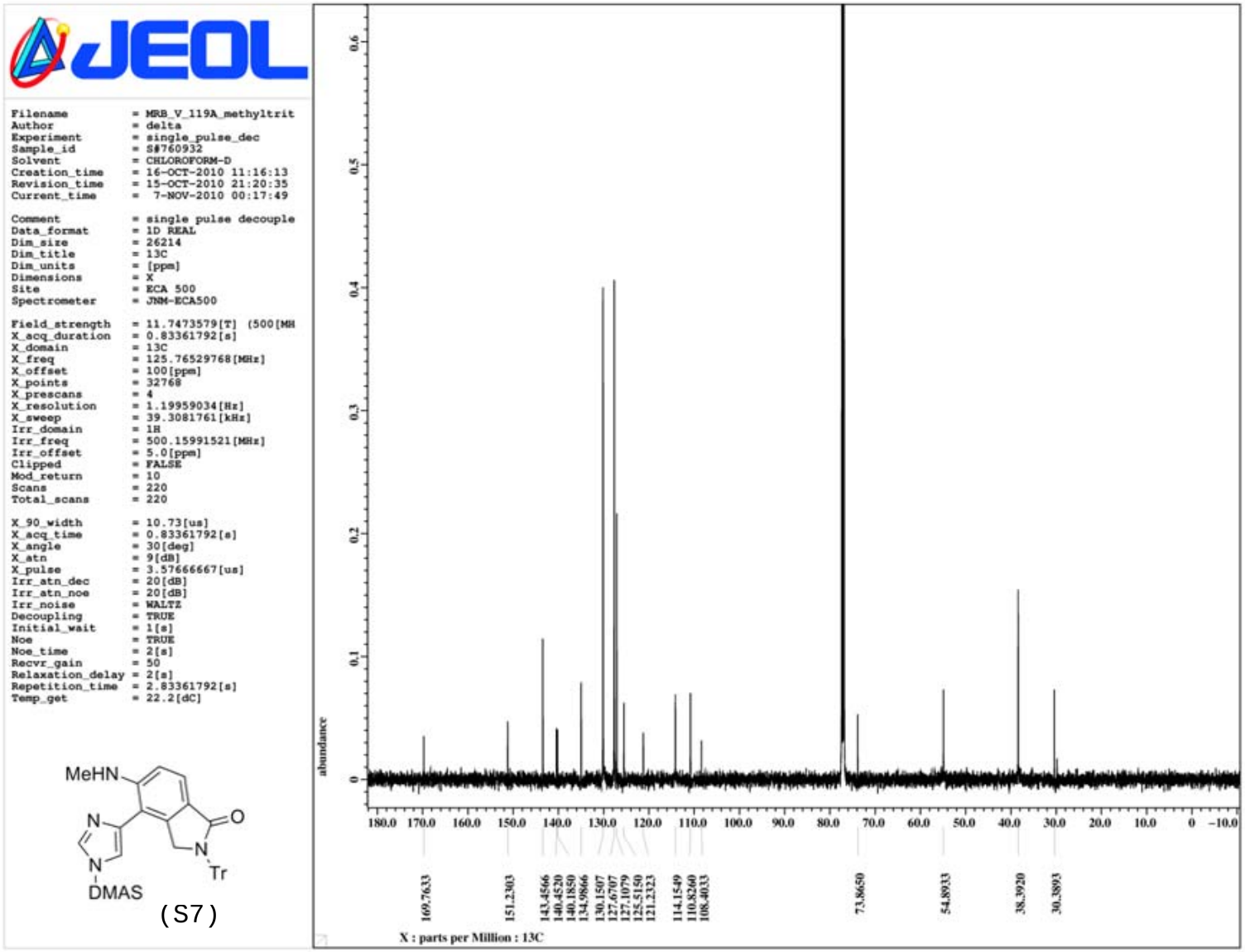
(18)

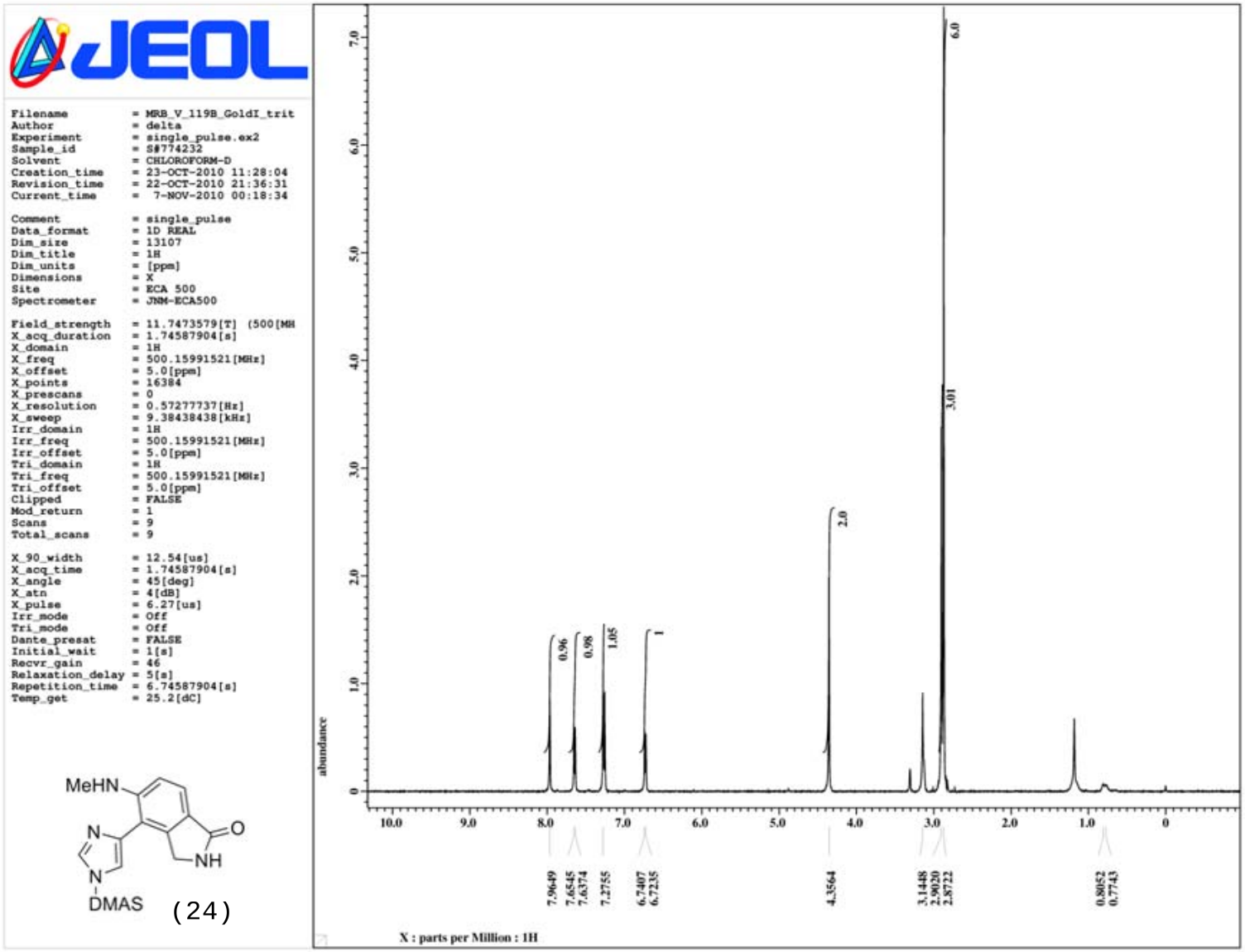


```
Filename          = MRB_V_115_tritylmethy
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#828526
Solvent           = CHLOROFORM-D
Creation_time     = 28-SEP-2010 13:02:59
Revision_time     = 10-OCT-2010 23:21:41
Current_time      =  7-NOV-2010 00:14:37

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 0.83361792[s]
X_domain          = 13C
X_freq            = 125.76529768[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 1.19959034[Hz]
X_sweep           = 39.3081761[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 10
Scans             = 80
Total_scans       = 80

X_90_width        = 10.73[us]
X_acq_time        = 0.83361792[s]
X_angle           = 30[deg]
X_atn             = 9[dB]
X_pulse           = 3.57666667[us]
Irr_atn_dec       = 20[dB]
Irr_atn_noe       = 20[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   = 2.83361792[s]
Temp_get          = 22.3[dC]
```


(18)

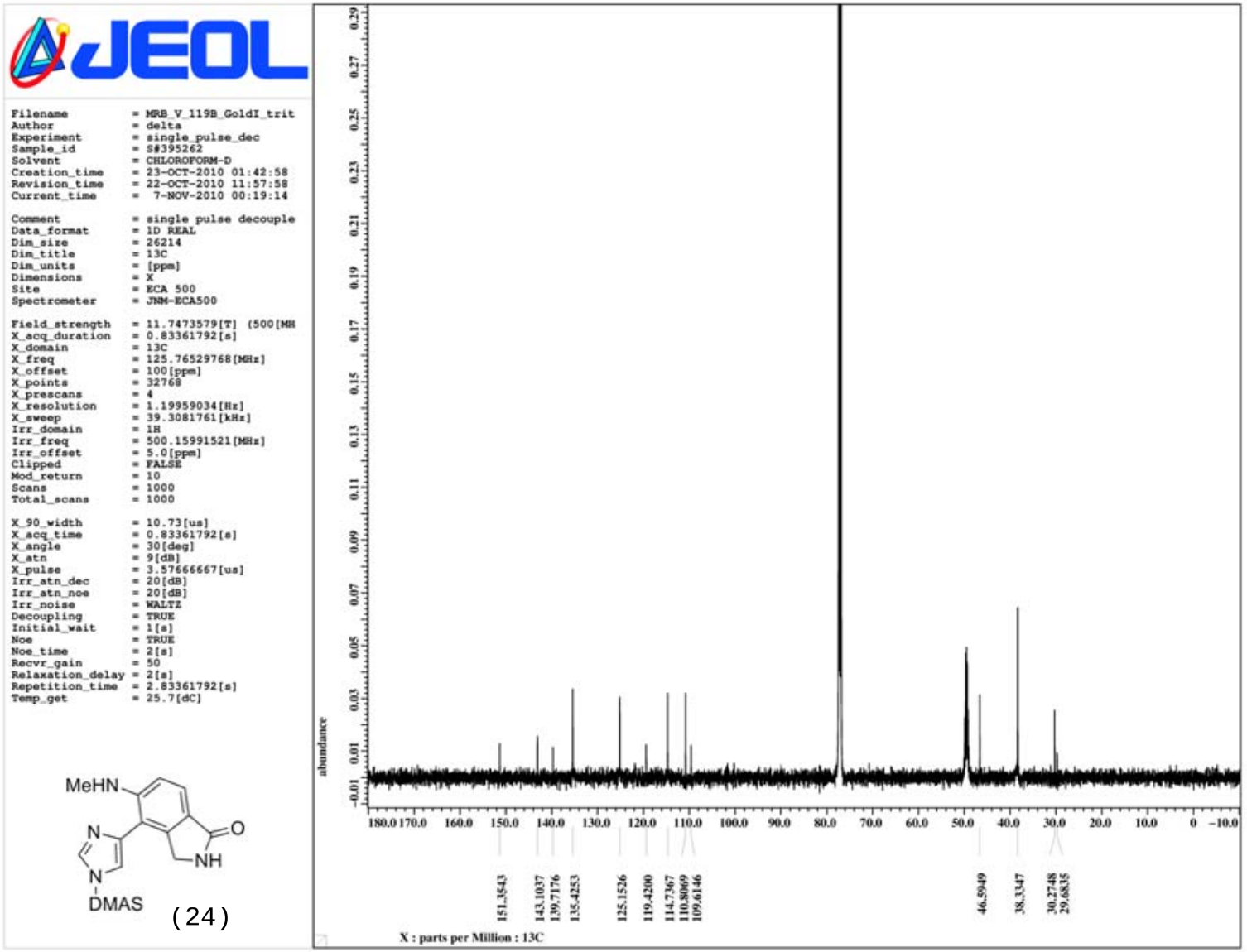

Filename = MRB_V_119A_methyltrit
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#759695
Solvent = CHLOROFORM-D
Creation_time = 16-OCT-2010 11:04:48
Revision_time = 7-NOV-2010 00:16:33
Current_time = 7-NOV-2010 00:17:15
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.8[dC]
MeHN
N
N
DMAS
O
N
Tr
(S7)
abundance
17.15
6.18
2
0.99
2.22
1.1
7.9282
7.6511
7.3053
7.2938
7.2778
7.2698
7.2652
7.2595
7.2549
6.8392
4.2705
2.9719
2.9627
2.7245
X : parts per Million : 1H

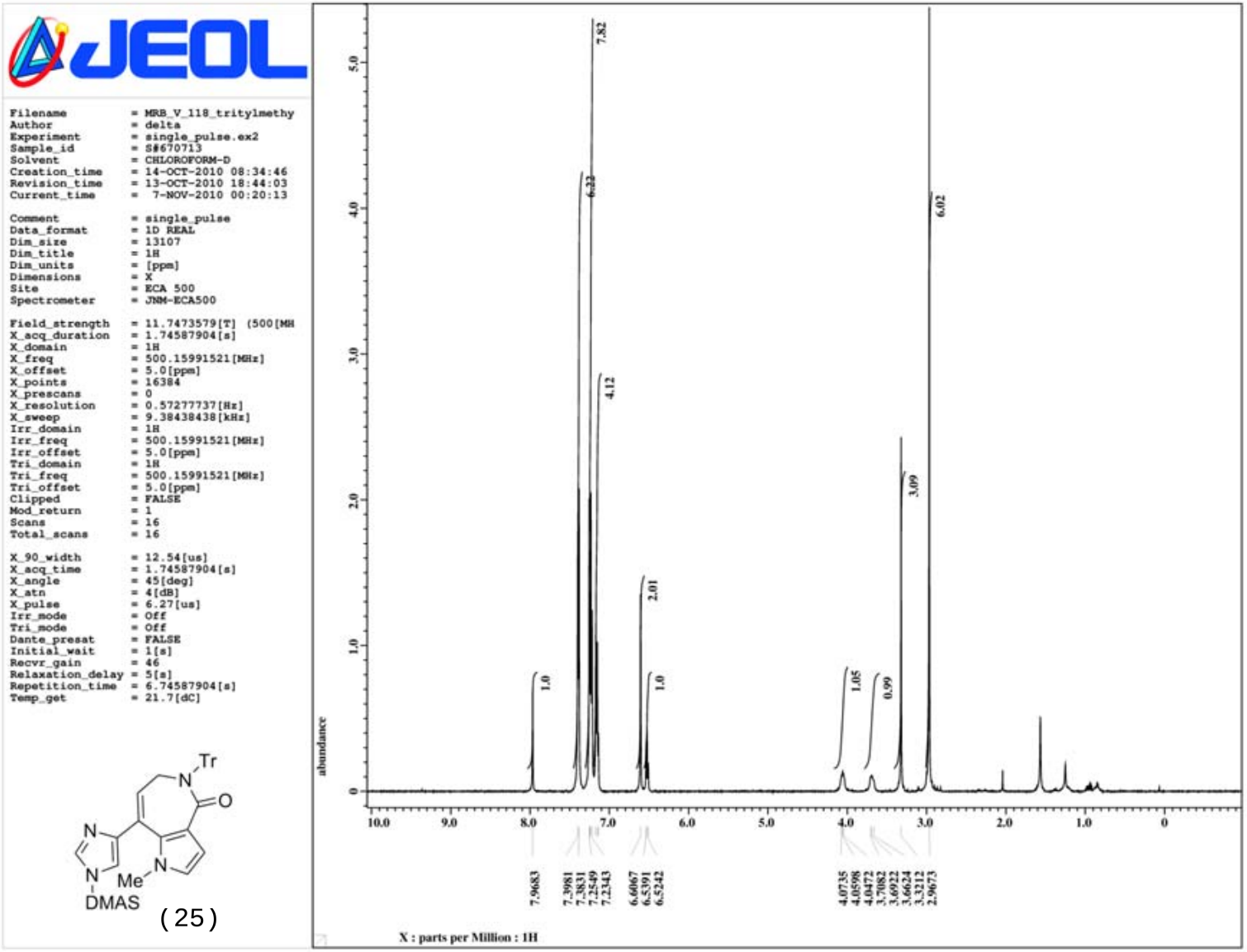

Filename = MRB_V_119A_methyltrit
Author = delta
Experiment = single_pulse_dec
Sample_id = S#760932
Solvent = CHLOROFORM-D
Creation_time = 16-OCT-2010 11:16:13
Revision_time = 15-OCT-2010 21:20:35
Current_time = 7-NOV-2010 00:17:49
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 220
Total_scans = 220
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.2[dC]
MeHN
N
N
DMAS
O
N
Tr
(S7)
abundance
0.6
0.5
0.4
0.3
0.2
0.1
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 −10.0
169.7633
151.2303
143.4566
140.4520
140.1850
134.9866
130.1507
127.6707
127.1079
125.5150
121.2323
114.1549
110.8260
108.4033
73.8650
54.8933
38.3920
30.3893
X : parts per Million : 13C

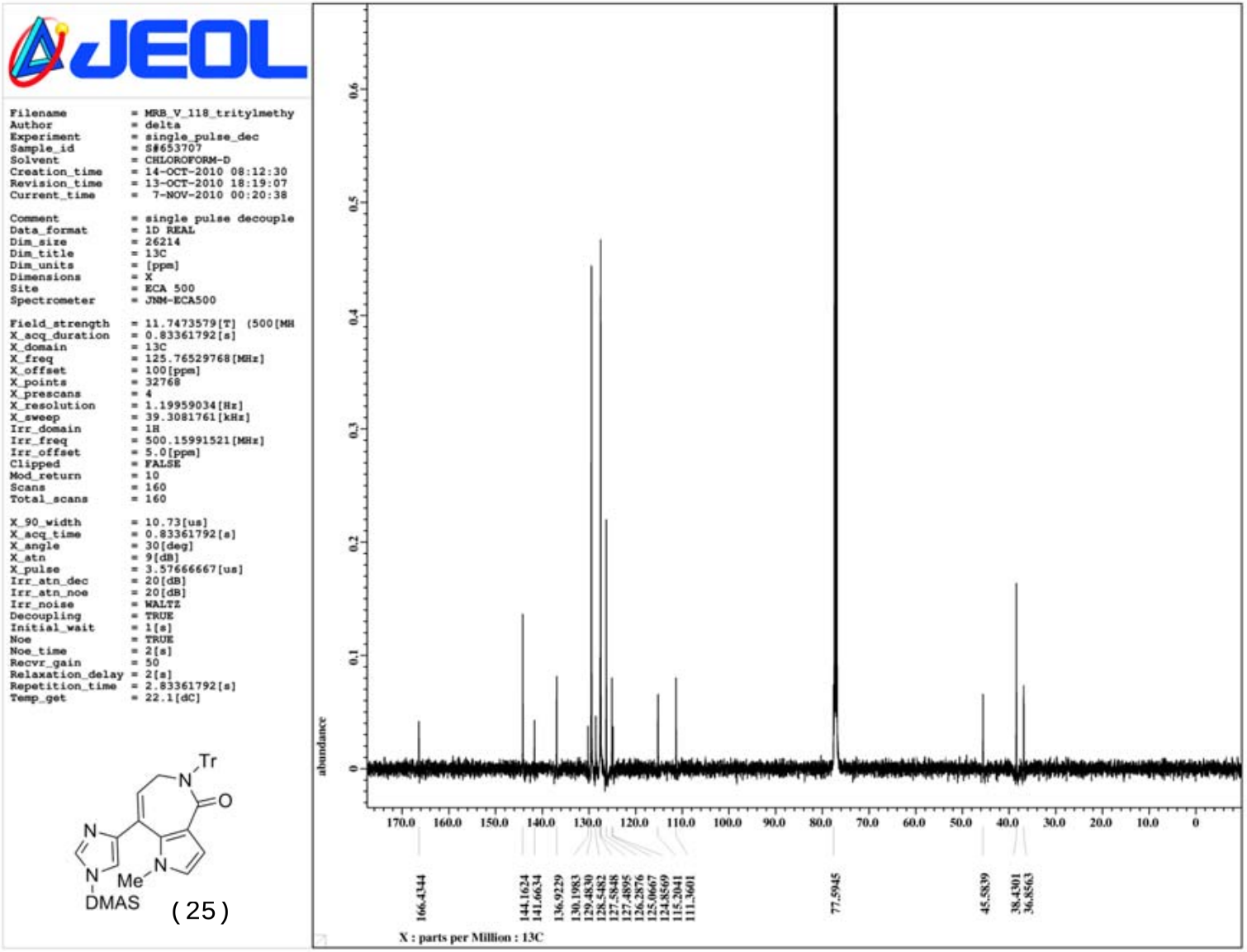


```
Filename          = MRB_V_119B_GoldI_trit
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#774232
Solvent           = CHLOROFORM-D
Creation_time     = 23-OCT-2010 11:28:04
Revision_time     = 22-OCT-2010 21:36:31
Current_time      =  7-NOV-2010 00:18:34

Comment           = single_pulse
Data_format       = 1D REAL
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T] (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 9
Total_scans       = 9

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 46
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 25.2[dC]
```


(24)

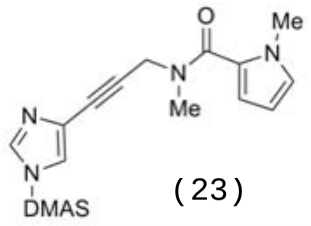

Filename = MRB_V_119B_GoldI_trit
Author = delta
Experiment = single_pulse_dec
Sample_id = S#395262
Solvent = CHLOROFORM-D
Creation_time = 23-OCT-2010 01:42:58
Revision_time = 22-OCT-2010 11:57:58
Current_time = 7-NOV-2010 00:19:14
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 1000
Total_scans = 1000
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 25.7[dC]
MeHN
N
N
DMAS
O
NH
(24)
abundance
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 −10.0
151.3543
143.1037
139.7176
135.4253
125.1526
119.4200
114.7367
110.8069
109.6146
46.5949
38.3347
30.2748
29.6835
X : parts per Million : 13C


Filename = MRB_V_118_tritylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#670713
Solvent = CHLOROFORM-D
Creation_time = 14-OCT-2010 08:34:46
Revision_time = 13-OCT-2010 18:44:03
Current_time = 7-NOV-2010 00:20:13
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.7[dC]
Tr
N
O
N
N
Me
N
DMAS
(25)
7.82
6.32
4.12
6.02
3.09
2.01
1.0
1.0
1.05
0.99
abundance
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.9683
7.3981
7.3831
7.2549
7.2343
6.6067
6.5391
6.5242
4.0735
4.0598
4.0472
3.7082
3.6922
3.6624
3.3312
2.9673
X : parts per Million : 1H

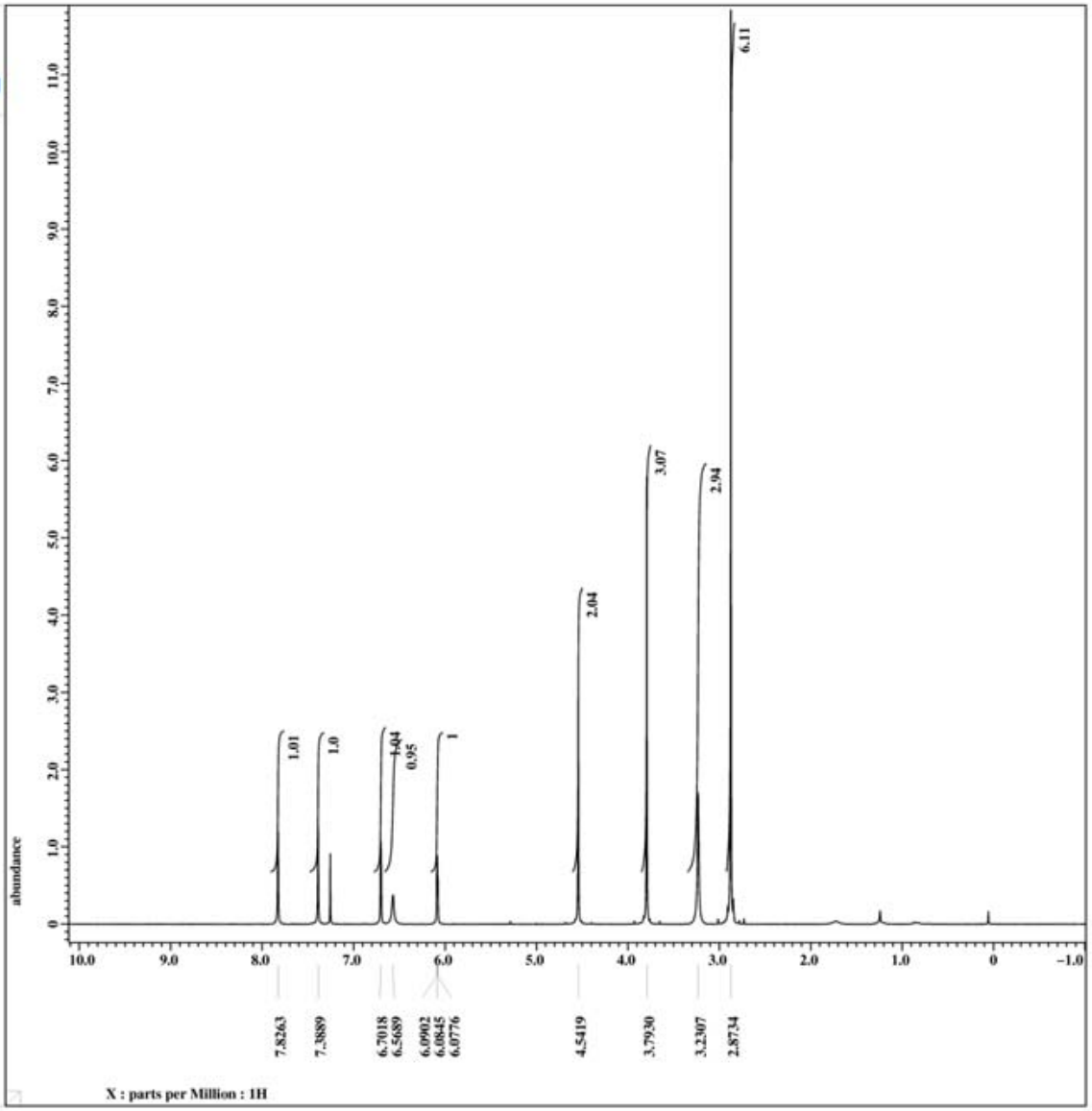

Filename = MRB_V_118_tritylmethy
Author = delta
Experiment = single_pulse_dec
Sample_id = S#653707
Solvent = CHLOROFORM-D
Creation_time = 14-OCT-2010 08:12:30
Revision_time = 13-OCT-2010 18:19:07
Current_time = 7-NOV-2010 00:20:38
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.1[dC]
Tr
N
O
N
N
Me
N
DMAS
(25)
abundance
0.6
0.5
0.4
0.3
0.2
0.1
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
166.4344
144.1624
141.6634
136.9229
130.1983
129.4830
128.5482
127.5848
127.4895
126.2876
125.0667
124.8569
115.2041
111.3601
77.5945
45.5839
38.4301
36.8563
X : parts per Million : 13C

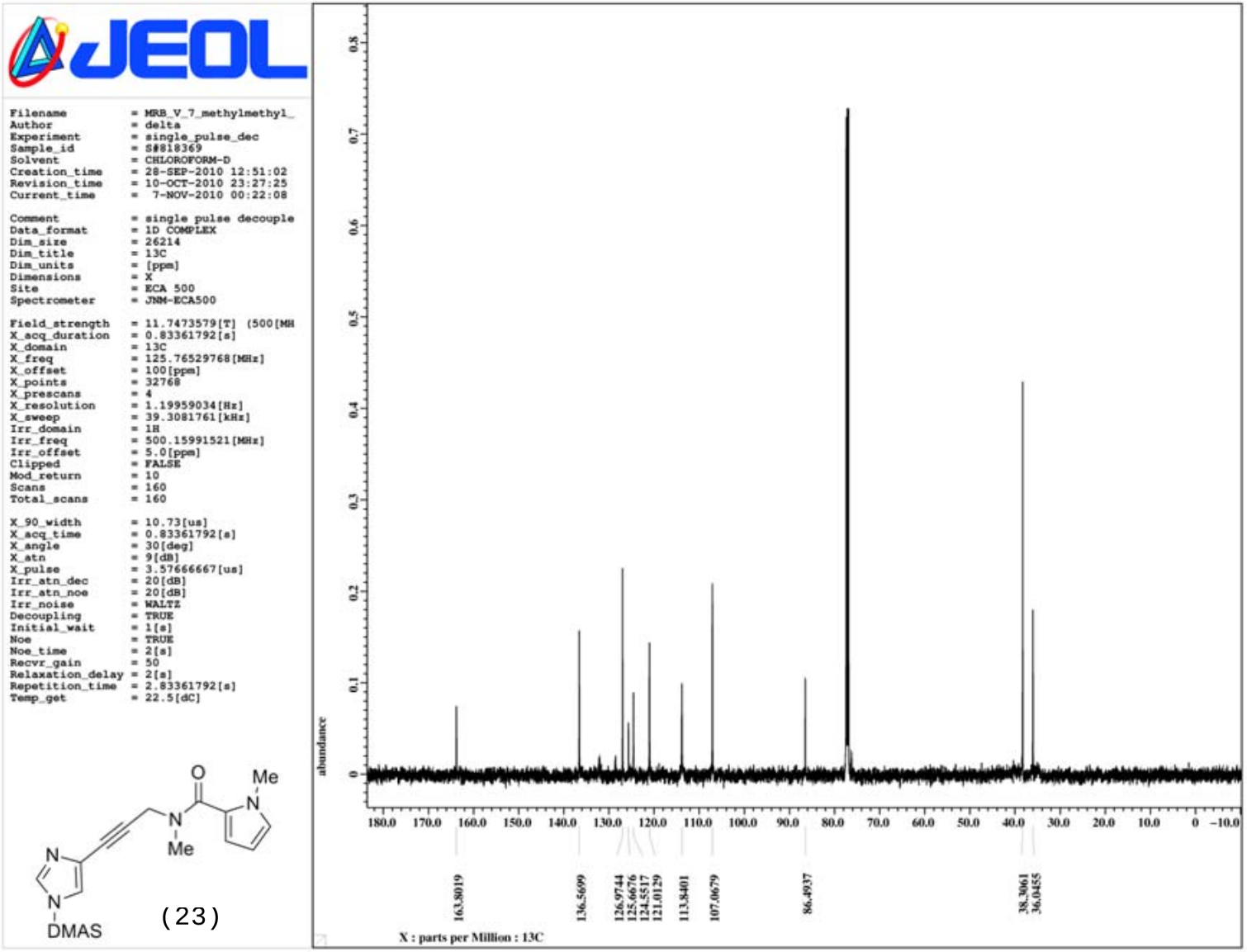
JEOL

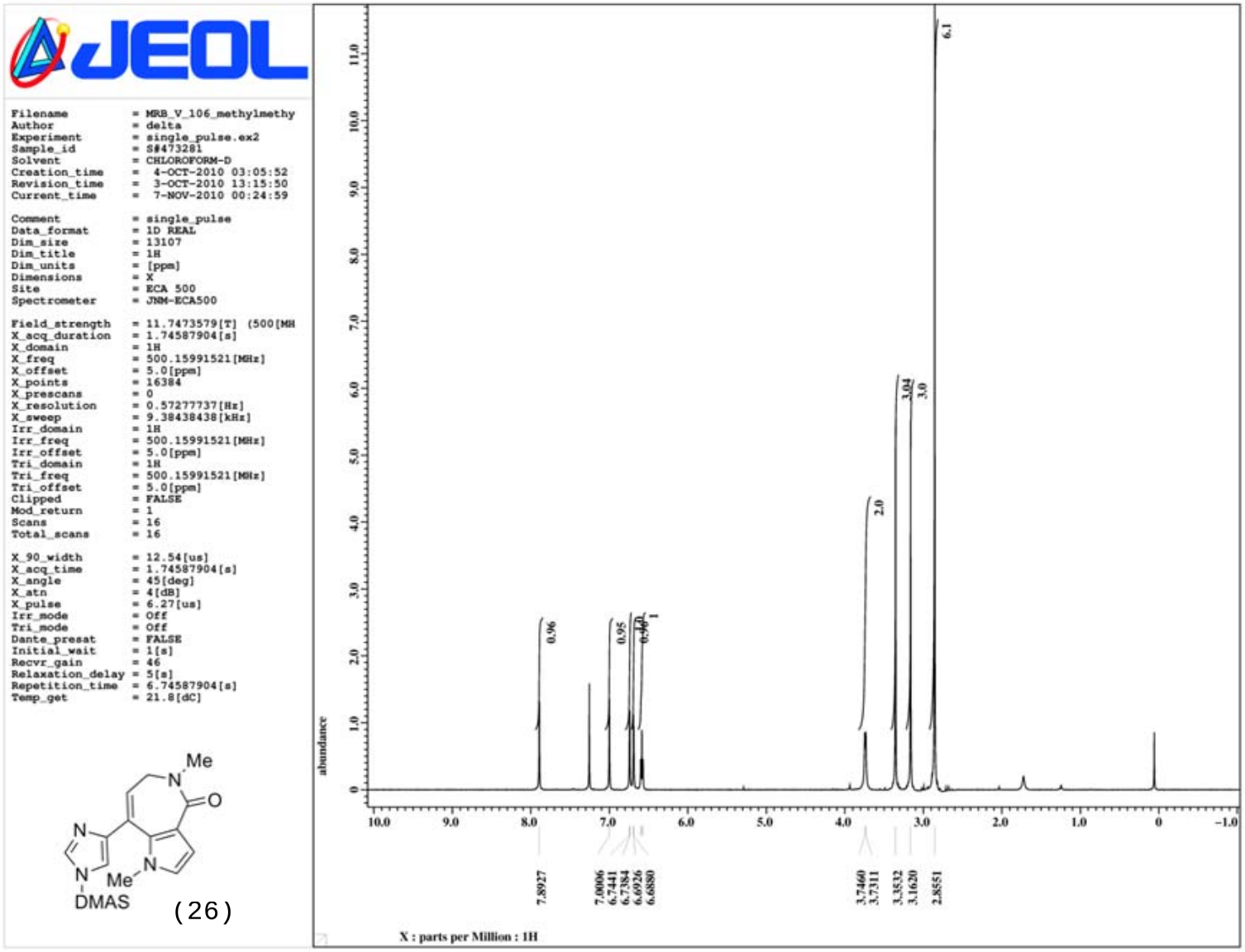
Filename	= MRB_V_7_methtlmethyl_
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#680671
Solvent	= CHLOROFORM-D
Creation_time	= 29-SEP-2010 08:54:45
Revision_time	= 28-SEP-2010 18:59:36
Current_time	= 7-NOV-2010 00:21:28
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 12
Total_scans	= 12
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 21.8[dC]

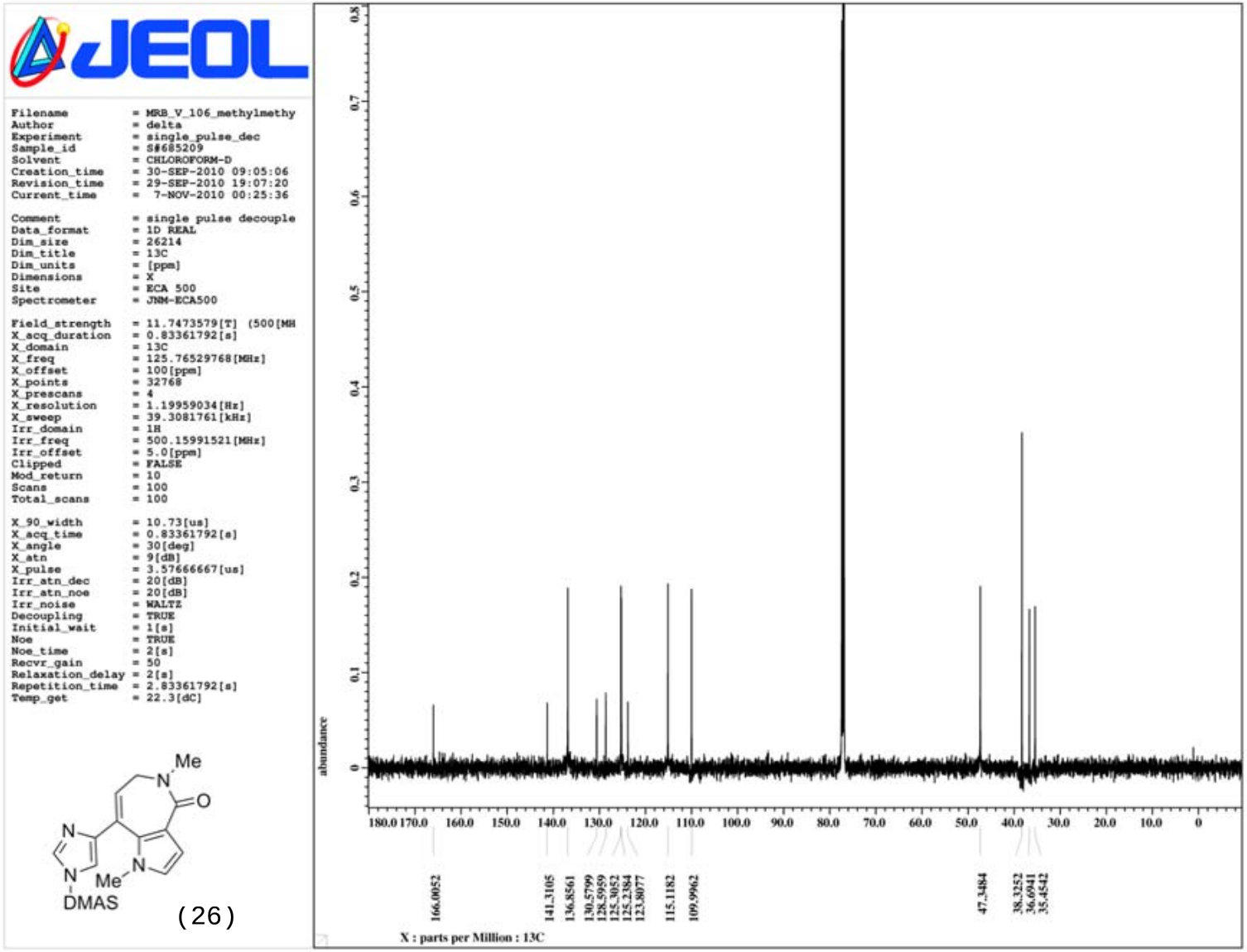


(23)



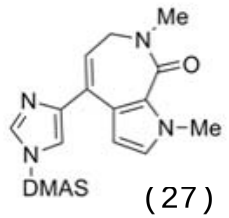

Filename = MRB_V_7_methylmethyl_
Author = delta
Experiment = single_pulse_dec
Sample_id = S#818369
Solvent = CHLOROFORM-D
Creation_time = 28-SEP-2010 12:51:02
Revision_time = 10-OCT-2010 23:27:25
Current_time = 7-NOV-2010 00:22:08
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 160
Total_scans = 160
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.5[dC]
O
Me
N
N
Me
N
N
DMAS
(23)
abundance
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
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 −10.0
163.8019
136.5699
126.9744
125.6676
124.5517
121.0129
113.8401
107.0679
86.4937
38.3061
36.0455
X : parts per Million : 13C


Filename = MRB_V_106_methylmethy
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#473281
Solvent = CHLOROFORM-D
Creation_time = 4-OCT-2010 03:05:52
Revision_time = 3-OCT-2010 13:15:50
Current_time = 7-NOV-2010 00:24:59
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 46
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.8[dC]
Me
N
O
N
N
Me
N
DMAS
(26)
abundance
0.96
0.95
2.0
3.04
3.0
6.1
7.8927
7.0006
6.7441
6.7384
6.6926
6.6880
3.7460
3.7311
3.3532
3.1620
2.8551
X : parts per Million : 1H

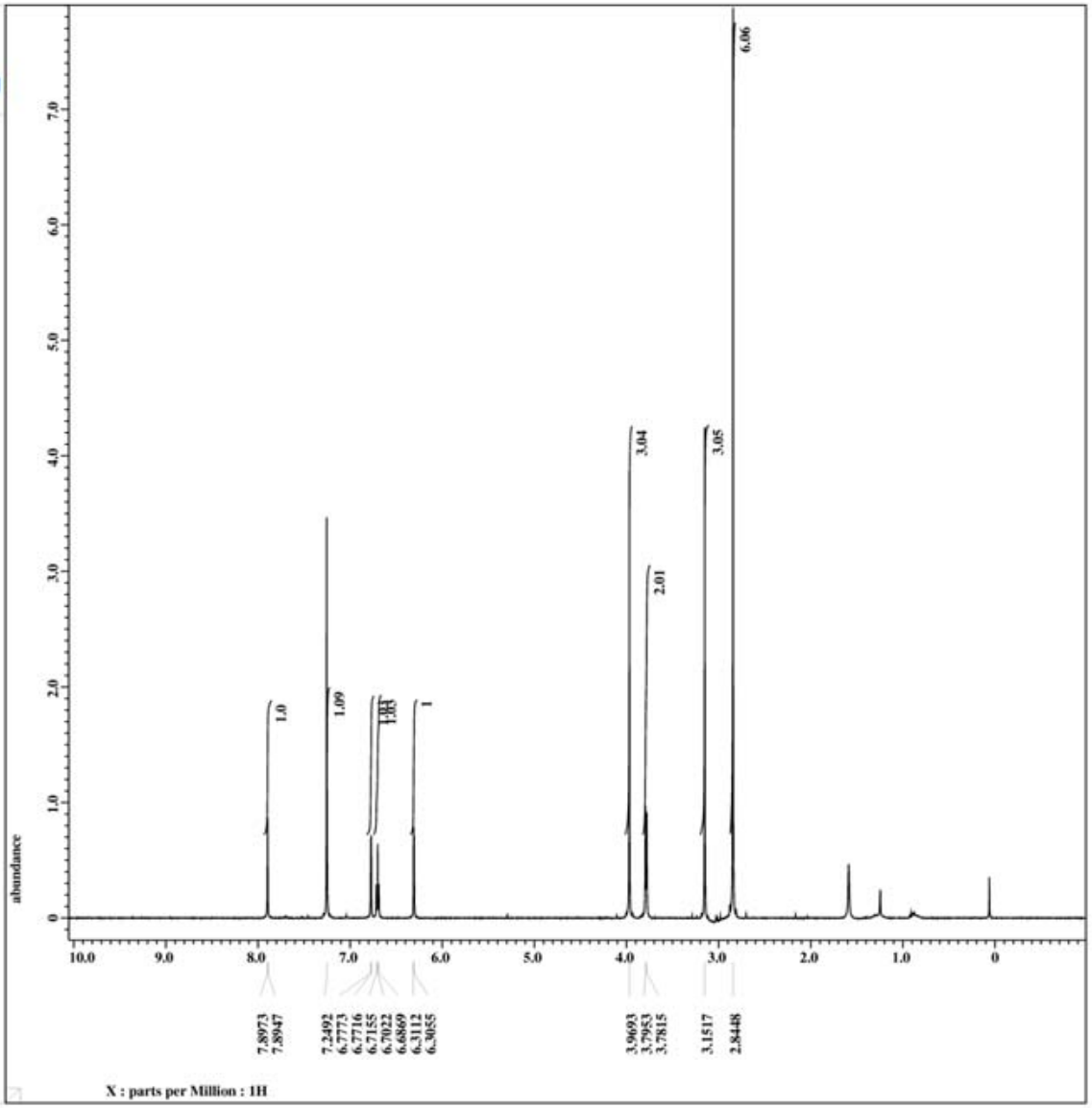

Filename = MRB_V_106_methylmethy
Author = delta
Experiment = single_pulse_dec
Sample_id = S#685209
Solvent = CHLOROFORM-D
Creation_time = 30-SEP-2010 09:05:06
Revision_time = 29-SEP-2010 19:07:20
Current_time = 7-NOV-2010 00:25:36
Comment = single pulse decouple
Data_format = 1D REAL
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain = 13C
X_freq = 125.76529768[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 1.19959034[Hz]
X_sweep = 39.3081761[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 10
Scans = 100
Total_scans = 100
X_90_width = 10.73[us]
X_acq_time = 0.83361792[s]
X_angle = 30[deg]
X_atn = 9[dB]
X_pulse = 3.57666667[us]
Irr_atn_dec = 20[dB]
Irr_atn_noe = 20[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 = 2.83361792[s]
Temp_get = 22.3[dC]
Me
N
O
N
N
Me
N
DMAS
abundance
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
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
166.0052
141.3105
136.8561
130.5799
128.5959
125.3052
125.2384
123.8077
115.1182
109.9962
47.3484
38.3252
36.6941
35.4542
X : parts per Million : 13C

(26)

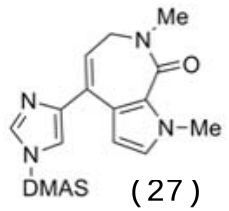
```
Filename          = MRB_V_106_methylmethy
Author            = delta
Experiment        = single_pulse.ex2
Sample_id         = S#459203
Solvent           = CHLOROFORM-D
Creation_time     =  4-OCT-2010 02:42:10
Revision_time     =  3-OCT-2010 12:52:50
Current_time      =  7-NOV-2010 00:22:55

Comment           = single_pulse
Data_format       = 1D REAL
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = JNM-ECA500

Field_strength    = 11.7473579[T]  (500[MH
X_acq_duration    = 1.74587904[s]
X_domain          = 1H
X_freq            = 500.15991521[MHz]
X_offset          = 5.0[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.57277737[Hz]
X_sweep           = 9.38438438[kHz]
Irr_domain        = 1H
Irr_freq          = 500.15991521[MHz]
Irr_offset        = 5.0[ppm]
Tri_domain        = 1H
Tri_freq          = 500.15991521[MHz]
Tri_offset        = 5.0[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16
Total_scans       = 16

X_90_width        = 12.54[us]
X_acq_time        = 1.74587904[s]
X_angle           = 45[deg]
X_atn             = 4[dB]
X_pulse           = 6.27[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 48
Relaxation_delay  = 5[s]
Repetition_time   = 6.74587904[s]
Temp_get          = 22[dC]
```



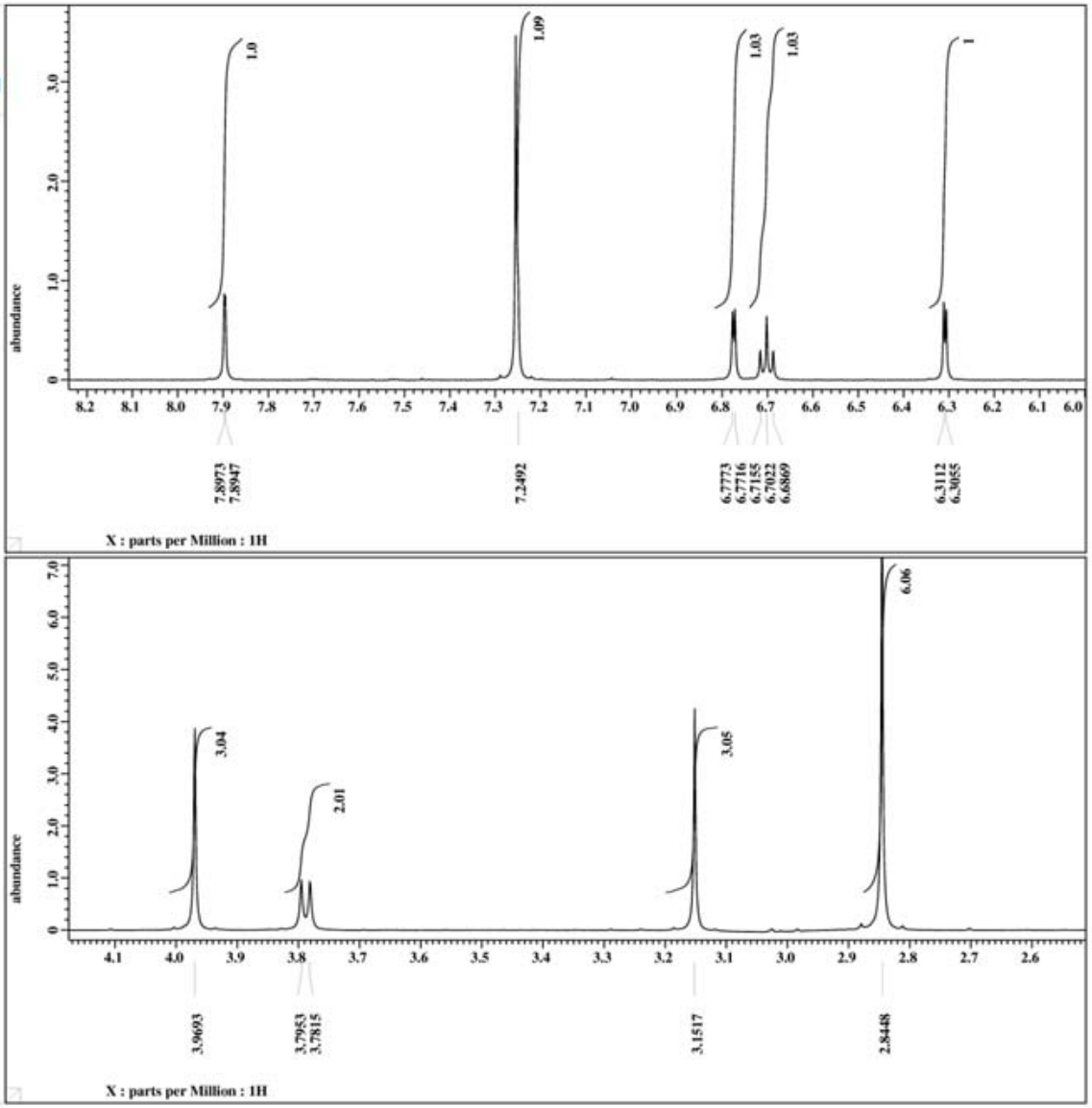
(27)

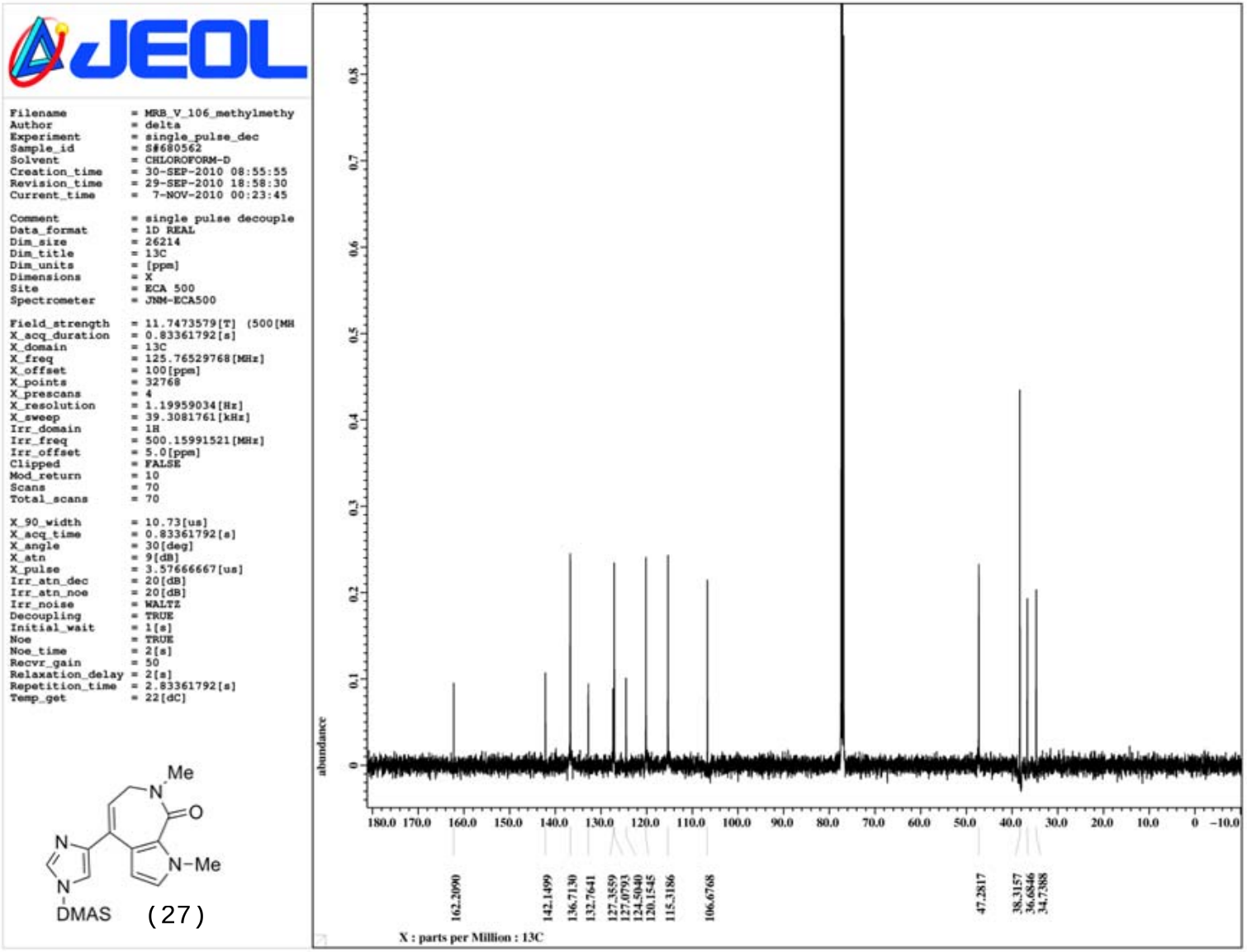


Filename	= MRB_V_106_methylmethy
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#459203
Solvent	= CHLOROFORM-D
Creation_time	= 4-OCT-2010 02:42:10
Revision_time	= 3-OCT-2010 12:52:50
Current_time	= 7-NOV-2010 00:23:23
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 48
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 22[dC]



DMAS (27)

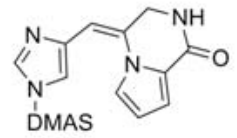




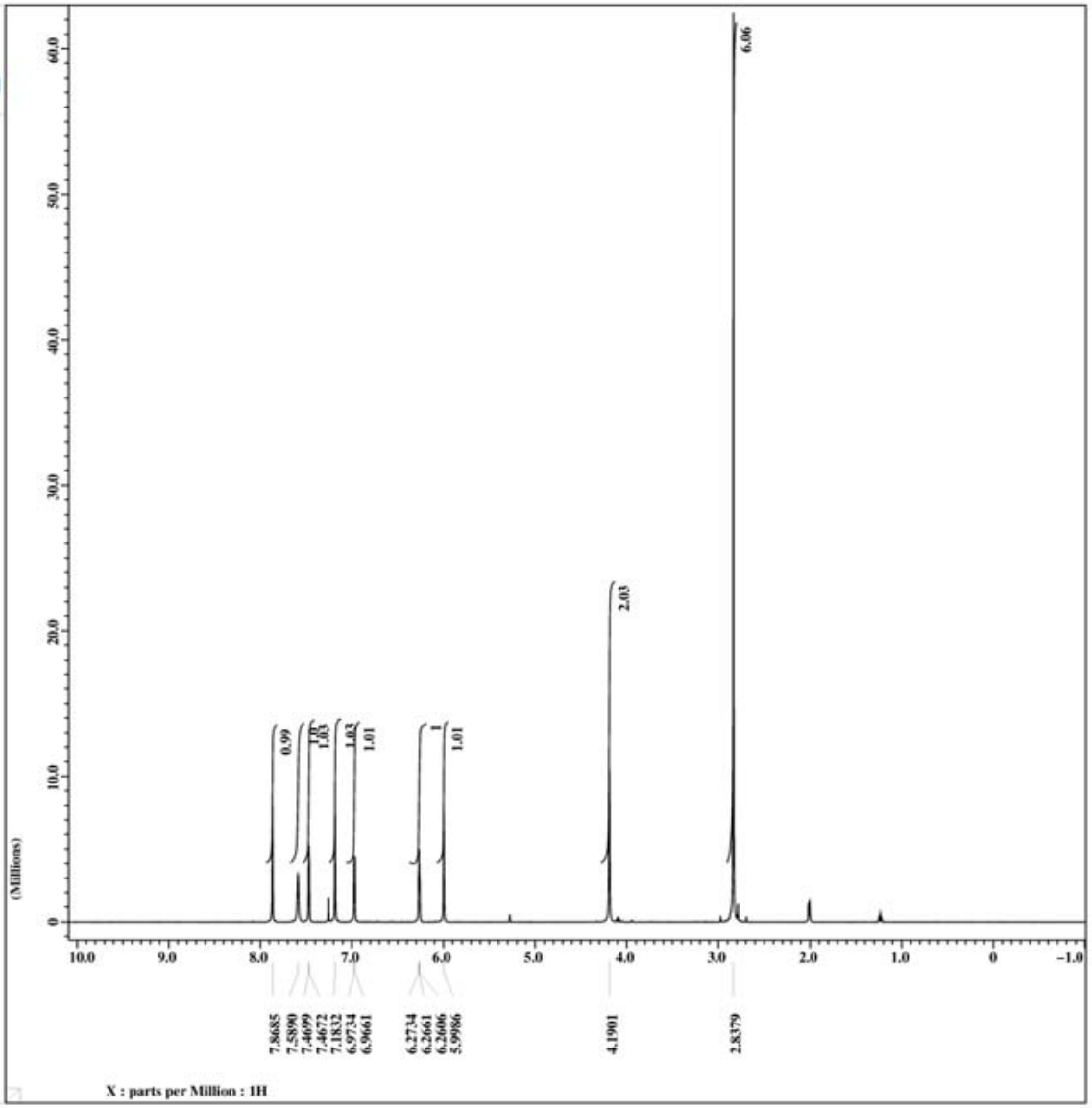
Filename	= MRB_V_106_methylmethy
Author	= delta
Experiment	= single_pulse_dec
Sample_id	= S#680562
Solvent	= CHLOROFORM-D
Creation_time	= 30-SEP-2010 08:55:55
Revision_time	= 29-SEP-2010 18:58:30
Current_time	= 7-NOV-2010 00:23:45
Comment	= single pulse decouple
Data_format	= 1D REAL
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 0.83361792[s]
X_domain	= 13C
X_freq	= 125.76529768[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 1.19959034[Hz]
X_sweep	= 39.3081761[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 10
Scans	= 70
Total_scans	= 70
X_90_width	= 10.73[us]
X_acq_time	= 0.83361792[s]
X_angle	= 30[deg]
X_atn	= 9[dB]
X_pulse	= 3.57666667[us]
Irr_atn_dec	= 20[dB]
Irr_atn_noe	= 20[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	= 2.83361792[s]
Temp_get	= 22[dC]

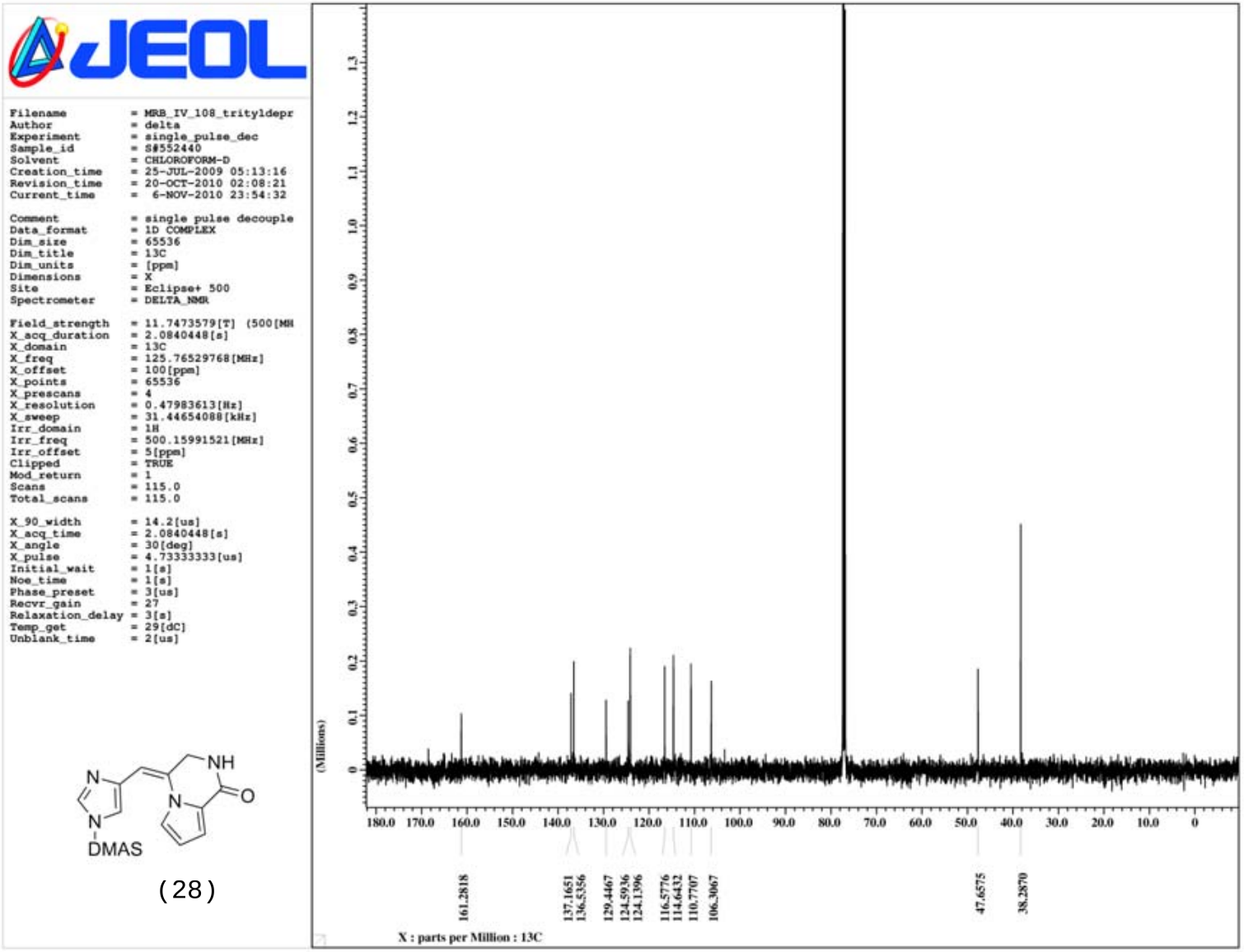
DMAS (27)

```
Filename          = MRB_IV_108_trityldepr
Author            = delta
Experiment        = single_pulse.exp
Sample_id         = S#552389
Solvent           = CHLOROFORM-D
Creation_time     = 25-JUL-2009 04:59:45
Revision_time     = 24-JUL-2009 15:26:16
Current_time      =  6-NOV-2010 23:54:01

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    = 11.7473579[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             = 8
Total_scans       = 8

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          = 27.4[dC]
Unblank_time      = 2[us]
```



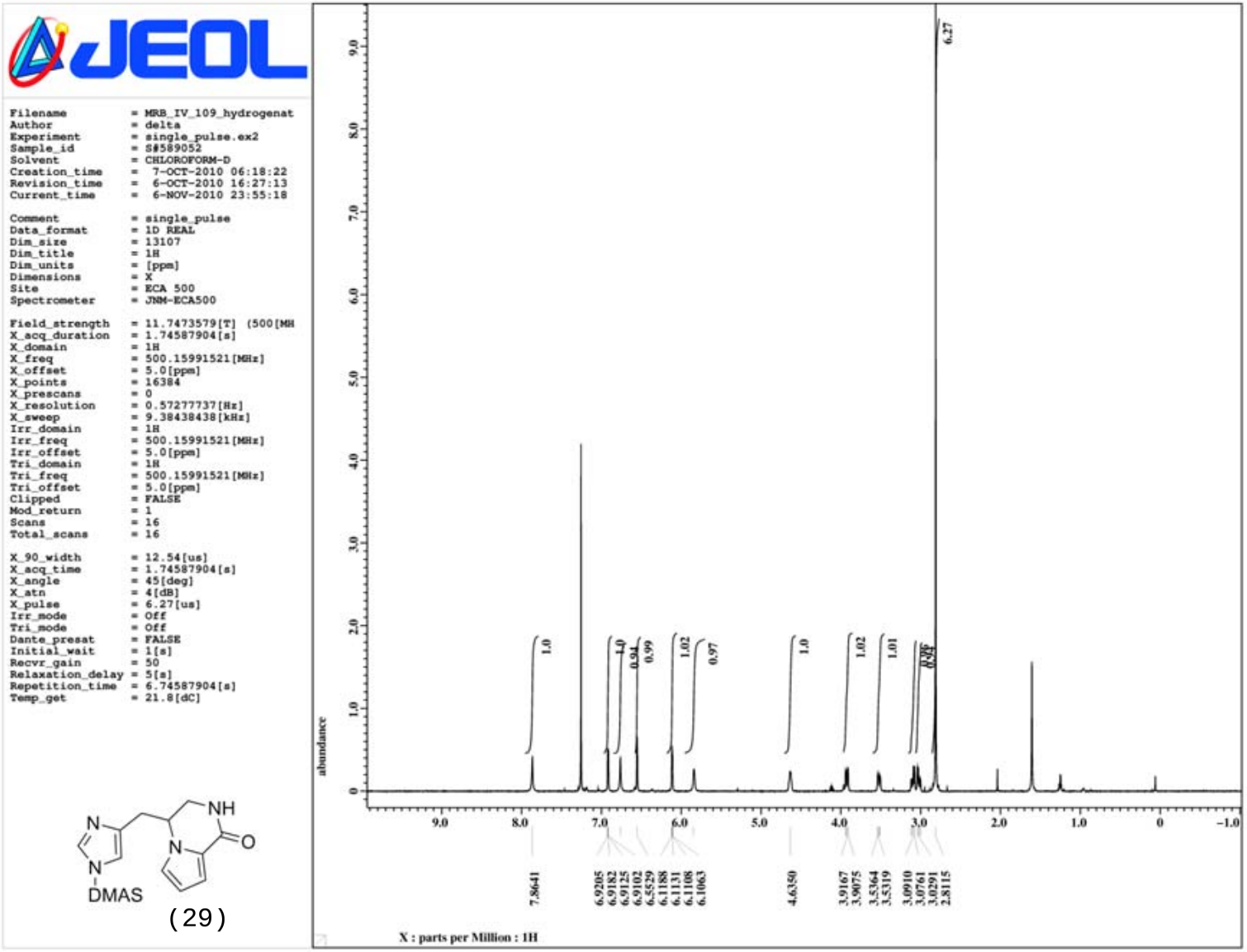
(28)



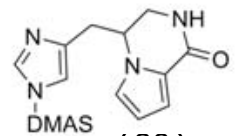


```
Filename          = MRB_IV_108_trityldepr
Author            = delta
Experiment        = single_pulse_dec
Sample_id         = S#552440
Solvent           = CHLOROFORM-D
Creation_time     = 25-JUL-2009 05:13:16
Revision_time     = 20-OCT-2010 02:08:21
Current_time      =  6-NOV-2010 23:54:32

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             = 115.0
Total_scans       = 115.0

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        = 27
Relaxation_delay  = 3[s]
Temp_get          = 29[dC]
Unblank_time      = 2[us]
```

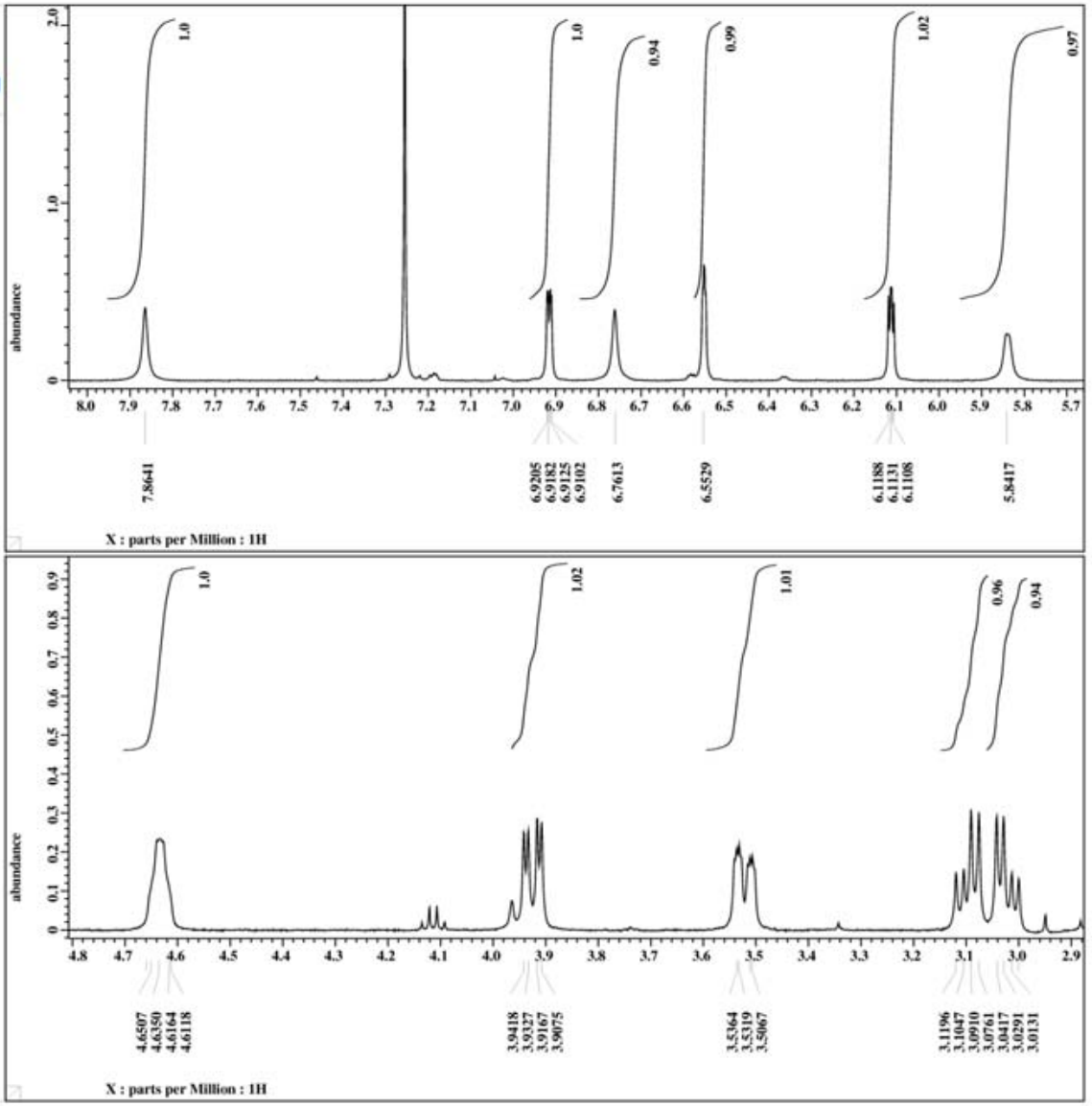

(28)

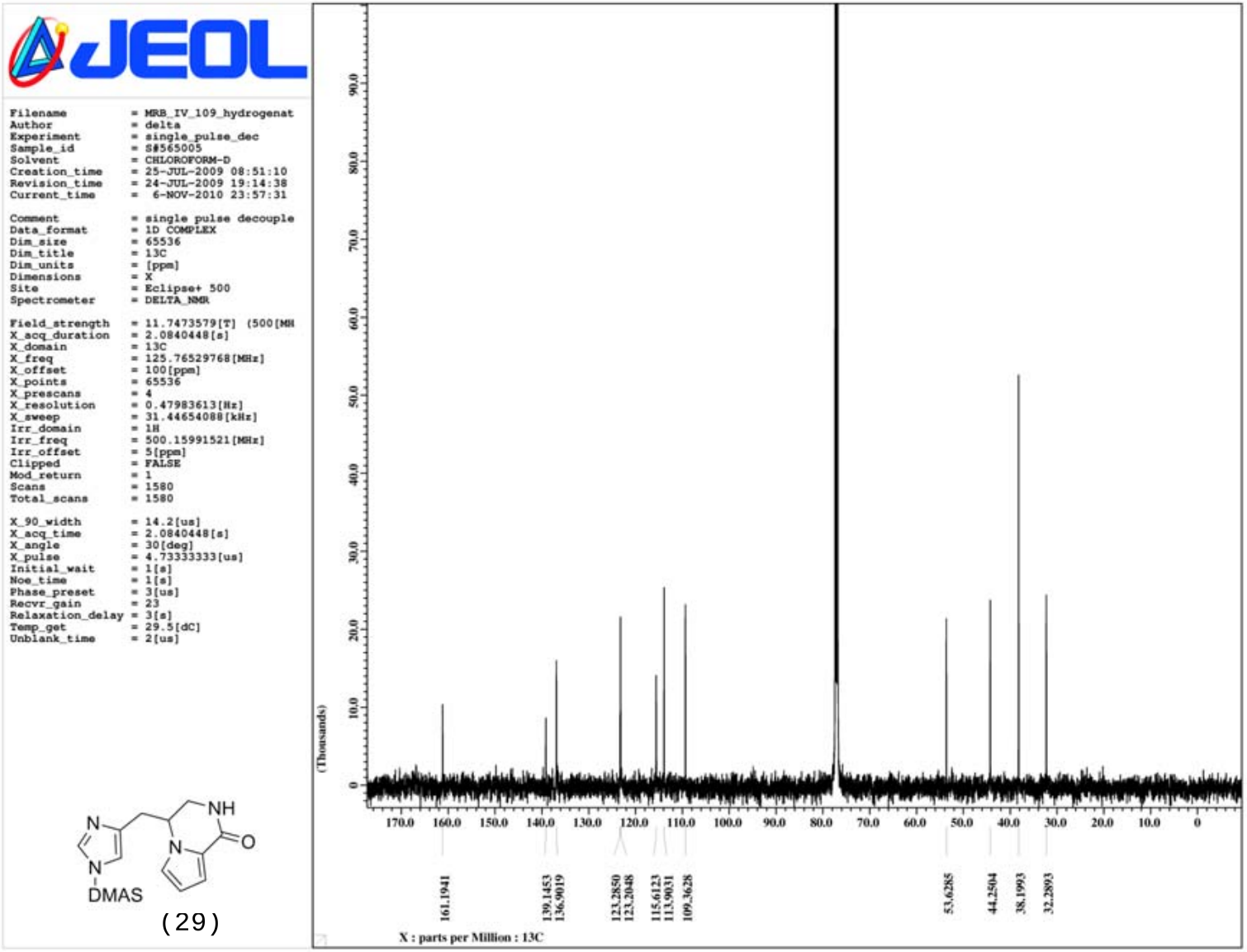

Filename = MRB_IV_109_hydrogenat
Author = delta
Experiment = single_pulse.ex2
Sample_id = S#589052
Solvent = CHLOROFORM-D
Creation_time = 7-OCT-2010 06:18:22
Revision_time = 6-OCT-2010 16:27:13
Current_time = 6-NOV-2010 23:55:18
Comment = single_pulse
Data_format = 1D REAL
Dim_size = 13107
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = JNM-ECA500
Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 5.0[ppm]
X_points = 16384
X_prescans = 0
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16
Total_scans = 16
X_90_width = 12.54[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 4[dB]
X_pulse = 6.27[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 50
Relaxation_delay = 5[s]
Repetition_time = 6.74587904[s]
Temp_get = 21.8[dC]
NH
N
N
O
N
DMAS
(29)
abundance
6.27
1.0
1.0
0.94
0.99
1.02
0.97
1.0
1.02
1.01
7.8641
6.9205
6.9182
6.9125
6.9102
6.5529
6.1188
6.1131
6.1108
6.1063
4.6350
3.9167
3.9075
3.5364
3.5319
3.0910
3.0761
3.0291
2.8115
X : parts per Million : 1H

Filename	= MRB_IV_109_hydrogenat
Author	= delta
Experiment	= single_pulse.ex2
Sample_id	= S#589052
Solvent	= CHLOROFORM-D
Creation_time	= 7-OCT-2010 06:18:22
Revision_time	= 6-NOV-2010 23:56:15
Current_time	= 6-NOV-2010 23:56:29
Comment	= single_pulse
Data_format	= 1D REAL
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= JNM-ECA500
Field_strength	= 11.7473579[T] (500[MH
X_acq_duration	= 1.74587904[s]
X_domain	= 1H
X_freq	= 500.15991521[MHz]
X_offset	= 5.0[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.57277737[Hz]
X_sweep	= 9.38438438[kHz]
Irr_domain	= 1H
Irr_freq	= 500.15991521[MHz]
Irr_offset	= 5.0[ppm]
Tri_domain	= 1H
Tri_freq	= 500.15991521[MHz]
Tri_offset	= 5.0[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 12.54[us]
X_acq_time	= 1.74587904[s]
X_angle	= 45[deg]
X_atn	= 4[dB]
X_pulse	= 6.27[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 6.74587904[s]
Temp_get	= 21.8[dC]



(29)




Filename = MRB_IV_109_hydrogenat
Author = delta
Experiment = single_pulse_dec
Sample_id = S#565005
Solvent = CHLOROFORM-D
Creation_time = 25-JUL-2009 08:51:10
Revision_time = 24-JUL-2009 19:14:38
Current_time = 6-NOV-2010 23:57:31
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 = 1580
Total_scans = 1580
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 = 23
Relaxation_delay = 3[s]
Temp_get = 29.5[dC]
Unblank_time = 2[us]
NH
N
N
O
N
DMAS
(29)
(Thousands)
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
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
161.1941
139.1453
136.9019
123.2850
123.2048
115.6123
113.9031
109.3628
53.6285
44.2504
38.1993
32.2893
X : parts per Million : 13C