Supporting Information

One-Pot Multicomponent Synthesis of Tetra- and Penta-substituted 2-Aminopyrroles. A Short General Synthesis of Rigidins A, B, C, and D

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Materials and Methods

All reagents, solvents and catalysts were purchased from commercial sources (Acros Organics and Sigma-Aldrich) and used without purification. *2-amino-4`-benzyloxy-acetophenone HCl* for the synthesis of **1**, and *2-amino-3`-methoxy-4`-benzyloxy-acetophenone HCl* for the synthesis of **2** were purchased from Amatek Chemical and used without further purification. All reactions were performed in an oven dried flasks open to the atmosphere or under nitrogen and monitored by thin layer chromatography on TLC precoated (250 μm) silica gel 60 F_{254} glass-backed plates (EMD Chemicals Inc.). Visualization was accomplished with UV light. Flash column chromatography was performed on silica gel (32-63 μm, 60 Å pore size). ^{1}H and ^{13}C NMR spectra were recorded on Jeol Eclipse 300 or Bruker Avance III 400 spectrometers. Chemical shifts (δ) are reported in ppm relative to the TMS internal standard. Abbreviations are as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). HRMS analyses were performed at the Mass Spectrometry Facility, University of New Mexico. Samples were run on LCT Premier TOF mass spec.

General Procedure for the Synthesis of 1 and 2.

Hydrochloride of a substituted phenacyl amine (1 mmol) was dissolved in a mixture of H_2O/acetone (1:2, 6 mL). The solution was stirred and methanesulfonyl chloride (0.116 mL, 1.5 mmol) was added in one portion. The mixture was placed in an ice-bath and triethylamine (0.348 mL, 2.5 mmol) was added dropwise in 30 min. Once all reactants were added, acetone (14 mL) was added to the mixture and the ice-bath was removed. The reaction mixture was kept at room temperature for 10 h, and then the volatiles were evaporated under reduced pressure. To the obtained slurry were added EtOAc (20 mL) and saturated NH_4Cl (30 mL), the organic layer was separated and washed with saturated $NaHCO_3$ (30 mL), followed by washing with saturated NaCl (20 mL). The extraction was repeated twice with EtOAc (15 mL) and the combined organic phases were dried with $MgSO_4$. The evaporation of the solvent resulted in the desired product that 98% pure based on the NMR spectra.

***N*-2-[4-(Benzyloxy)phenyl]-2-oxoethylmethanesulfonamide (1).** 97% as light yellow needles, mp = 118-120 ^{o}C, R_f = 0.34 (MeOH/CH_2Cl_2=1/50). ^{1}H NMR ($CDCl_3$) δ: 3.02 (s, 3H), 4.63 (d, J = 4.1 Hz, 2H), 5.18 (s, 2H), 5.43 (bs, 1H), 7.07 (d, J = 8.6 Hz, 2H), 7.38-7.46 (m, 5H), 7.94 (d, J = 8.6 Hz, 2H). ^{13}C NMR (acetone-d_6) δ: 40.6, 49.8, 70.8, 115.7, 128.5, 128.7, 128.9, 129.4, 131.1, 137.6, 162.3, 164.2, 193.3. HRMS *m*/*z* (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+Na^+$) 342.0776, found 342.0780.

***N*-2-[4-(Benzyloxy)-3-methoxyphenyl]-2-oxoethylmethanesulfonamide (2).** 95% as light yellow needles, mp = 139-140 ^{o}C, R_f = 0.28 (MeOH/CH_2Cl_2=1/50). ^{1}H NMR ($CDCl_3$) δ: 3.01 (s, 3H), 3.97 (s, 3H), 4.63 (s, 2H), 5.27 (s, 2H), 5.39 (bs, 1H), 6.96 (d, J = 8.4 Hz, 1H), 7.35-7.44 (m, 5H), 7.51 (dd, J = 1.8 Hz, J = 8.3 Hz, 1H), 7,53 (d, J = 1.7 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ: 40.7, 49.8, 56.2, 70.9, 110.4, 112.4, 122.5, 127.3, 128.4, 128.8, 135.7, 150.0, 153.7, 191.7. HRMS *m*/*z* (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+Na^+$) 372.0882, found 372.0876.

General Procedure for the Synthesis of Pyrroles A_1-A_{17}.

To a stirred solution of N-aryl-, N-alkyl- or N-heteroarylsulfonamidoacetophenone (0.2 mmol), aldehyde (0.26 mmol) and malononitrile (0.26 mmol) in acetonitrile (3mL) was added Et_3N (0.073 mmol). The mixture was refluxed until the starting sulfonamide disappeared (TLC). After this time the reaction mixture was cooled to room temperature and DDQ (0.6 mmol) was added under the nitrogen atmosphere (in the case of **A_{16}** and **A_{17}** the first part of the reaction was done in EtOH and after the disappearance of the starting sulfonamide the reaction mixture was cooled and before the adding of DDQ the solvent was changed to acetonitrile). The reaction mixture was stirred at room temperature for two hours. After that it was concentrated and purified by flash chromotography on silica gel with the indicated mixture of solvents to afford pure pyrroles.

2-Amino-5-benzoyl-4-(4-chlorophenyl)-1-[(4-nitrophenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_1). 81% as yellow solid, mp = 142-144 ^oC decomp., (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 7.18-7.31 (m, 6H), 7.34-7.62 (m, 3H), 7.83 (s, 2H), 8.24 (d, J = 8.8 Hz, 2H), 8.53 (d, J = 8.8 Hz, 2H). ^{13}C NMR (DMSO-d_6) δ: 75.6, 114.9, 121.7, 125.5, 128.7, 129.0, 129.4, 131.5, 133.6, 134.1, 135.2, 138.2, 141.7, 151.7, 152.4, 185.5. HRMS m/z (ESI) calc'd for $C_{24}H_{16}ClN_4O_5S$ (M+H+) 507.0530, found 507.0533.

2-Amino-5-benzoyl-4-(3,4,5-trimethoxyphenyl)-1-[(2,4,6-triisopropilphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_2). 91% as brown solid, mp = 170-172 ^oC decomp., (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 1.01-1.08 (m, 18H), 2.72-2.81 (m, 1H), 3.45-3.84 (m, 11H), 6.29 (s, 2H), 7.07-7.30 (m, 8H). ^{13}C NMR (DMSO-d_6) δ: 23.6, 24.9, 29.2, 33.9, 56.2, 60.4, 75.4, 107.4, 115.3, 122.6, 124.6, 126.1, 128.3, 129.1, 131.9, 132.9, 134.8, 138.0, 138.1, 151.5, 152.5, 152.7, 155.5, 184.3. HRMS m/z (ESI) calc'd for $C_{36}H_{42}N_3O_6S$ (M+H+) 644.2794, found 644.2789.

2-Amino-5-benzoyl-4-(3,4,5-trimethoxyphenyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_3). 89% as brown solid, mp = 156-158 ^oC decomp., (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 3.55-3.89 (m, 12H), 6.49 (s, 2H), 7.25-7.74 (m, 11H), 8.0 (d, J = 15 Hz, 2H). ^{13}C NMR (DMSO-d_6) δ: 56.3, 56.7, 60.5, 74.9, 107.3, 115.5, 120.8, 126.0, 127.7, 129.0, 129.6, 130.9, 133.5, 133.9, 138.2, 138.7, 151.7, 152.8, 165.0, 186.5. HRMS m/z (ESI) calc'd for $C_{28}H_{26}N_3O_7S$ (M+H+) 548.1491, found 548.1474.

2-Amino-5-(4-methoxybenzoyl)-4-(3-bromo-4,5-dimethoxyphenyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_4). 78% as brown solid, mp = 138-140 ^oC, (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 3.61-3.85 (m, 12H), 6.84-7.62 (m, 10H), 7.93 (d, J = 5.5 Hz, 2H). ^{13}C NMR (DMSO-d_6) δ: 56.1, 56.5, 56.6, 60.5, 74.4, 113.9, 114.3, 115.5, 116.8, 121.3, 125.0, 127.5, 128.0, 130.9, 131.4, 132.0, 146.2, 151.3, 153.1, 163.7, 165.1, 185.2. HRMS m/z (ESI) calc'd for $C_{28}H_{25}BrN_3O_7S$ (M+H+) 626.0597, found 626.0576.

2-Amino-5-benzoyl-4-(3-bromo-4-methoxyphenyl)-1-methanesulfonyl-1*H*-pyrrole-3-carbonitrile (A_5). 37% as brown solid, mp = 166-168 ^oC, (CH_2Cl_2/EtOAc=20/1). ^1H NMR (DMSO-d_6) δ: 3.65 (s, 3H), 3.77 (s, 3H), 6.83-7.55 (m, 10H). ^{13}C NMR (DMSO-d_6) δ: 43.5, 56.7, 74.6, 110.4, 112.5, 124.6, 128.5, 129.7, 130.2, 133.2, 134.0, 138.0, 152.5, 155.7, 185.6. HRMS m/z (ESI) calc'd for $C_{20}H_{17}BrN_3O_4S$ (M+H+) 474.0123, found 474.0128.

2-Amino-5-benzoyl-4-(3,5-dibromophenyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_6). 56% as brown solid, mp = 112-114 ^oC, (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 3.89 (s, 3H), 7.23-7.73 (m, 12H), 8.03 (s, 2H). ^{13}C NMR (DMSO-d_6) δ: 56.7,

74.8, 115.6, 122.6, 127.4, 129.0, 129.5, 130.9, 131.5, 131.8, 133.7, 134.0, 134.6, 138.6, 151.8, 165.1, 185.9. HRMS *m*/*z* (ESI) calc'd for $C_{25}H_{18}Br_2N_3O_4S$ (M+H+) 613.9385, found 613.9360.

2-Amino-5-benzoyl-4-(5-bromo-3-pyridinyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_7). 41% as brown solid, mp = 90-92 ^oC, (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 3.89 (s, 3H), 7.24-7.76 (m, 10H), 8.39 (s, 1H), 8.57 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 56.7, 75.0, 115.0, 115.6, 119.9, 122.5, 127.4, 128.7, 129.0, 129.6, 130.4, 131.0, 133.7, 138.5, 139.6, 148.3, 150.7, 152.0, 165.1, 185.5. HRMS *m*/*z* (ESI) calc'd for $C_{24}H_{18}BrN_4O_4S$ (M+H+) 537.0232, found 537.0231.

2-Amino-5-benzoyl-4-(2,6-dichlorophenyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carbonitrile (A_8). 78% as pink solid, mp = 180-181 ^oC decomp., (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 3.87 (s, 3H), 7.21-7.63 (m, 10H), 7.81-7.91 (m, 4H). ^{13}C NMR (DMSO-d_6) δ: 56.7, 75.5, 114.2, 115.4, 123.9, 127.4, 128.5, 129.0, 129.6, 131.0, 131.9, 133.1, 133.5, 134.8, 138.4, 153.0, 165.1, 184.3. HRMS *m*/*z* (ESI) calc'd for $C_{25}H_{17}Cl_2N_3O_4SNa$ (M+Na+) 548.0215, found 548.0224.

2-Amino-5-benzoyl-4-(2,6-dichlorophenyl)-1-[(4-fluorophenyl)sulfonyl)-1*H*-pyrrole-3-carbonitrile (A_9). 55% as pink solid, mp = 166-168 ^oC, (CH_2Cl_2/EtOAc=10/1). ^1H NMR (DMSO-d_6) δ: 7.21-7.67 (m, 10H), 7.93-8.07 (m, 4H). ^{13}C NMR (DMSO-d_6) δ: 75.1, 114.0, 117.5, 117.8, 123.9, 128.5, 128.6, 129.0, 129.4, 131.7, 131.9, 132.0, 132.6, 133.2, 134.0, 134.7, 138.2, 153.2, 184.2. HRMS *m*/*z* (ESI) calc'd for $C_{24}H_{14}Cl_2FN_3O_3SNa$ (M+Na+) 536.0015, found 536.0015.

2-Amino-5-benzoyl-1-(butylsulfonyl)-4-(4-nitrophenyl)-1*H*-pyrrole-3-carbonitrile (A_{10}). 71% as brown solid, mp = 182-183 ^oC decomp., (CH_2Cl_2/EtOAc=50/1). ^1H NMR (DMSO-d_6) δ: 0.82-0.96 (m, 3H), 1.36-1.45 (m, 2H), 1.74-1.98 (m, 2H), 3.94-4.07(m, 2H), 7.14-7.62 (m, 9H), 7.94-8.01 (m, 2H). ^{13}C NMR (DMSO-d_6) δ: 13.8, 21.2, 24.5, 55.6, 74.5, 115.2, 122.9, 123.5, 128.7, 130., 131.1, 132.9, 133.5, 137.8, 138.2, 147.4, 152.6, 185.6. HRMS *m*/*z* (ESI) calc'd for $C_{22}H_{21}N_4O_5S$ (M+H+) 453.1233, found 453.1229.

2-Amino-5-benzoyl-1-[(4-methylphenyl)sulfonyl]-4-propyl-1*H*-pyrrole-3-carbonitrile (A_{11}). 55% as brown solid, mp = 105-107 ^oC, (CH_2Cl_2/EtOAc=50/1). ^1H NMR (DMSO-d_6) δ: 0.63 (t, J=12Hz, J=6Hz, 3H), 1.36-1.43 (q, J=6 Hz, J=9Hz, J=6Hz, 2H), 2.23-2.39 (m, 5H), 7.48-7.74 (m, 11H). ^{13}C NMR (DMSO-d_6) δ: 13.7, 21.7, 22.9, 27.4, 75.8, 115.1, 122.3, 127.9, 129.0, 129.2, 130.5, 132.6, 133.0, 139.8, 140.0, 146.8, 152.9, 185.6. HRMS *m*/*z* (ESI) calc'd for $C_{22}H_{22}N_3O_3S$ (M+H+) 408.1382, found 408.1375.

2-Amino-5-benzoyl-1-(methylsulfonyl)-4-(2-thienyl)-1*H*-pyrrole-3-carbonitrile (A_{12}). 50% as brown solid, mp = 118-119 ^oC decomp., (CH_2Cl_2/EtOAc=50/1). ^1H NMR (DMSO-d_6) δ: 3.73 (s, 3H), 6.79-6.90 (m, 2H), 7.23-7.61 (m, 8H). ^{13}C NMR (DMSO-d_6) δ: 43.3, 74.2, 115.5, 122.0, 126.5, 127.7, 128.8, 129.4, 129.8, 130.9, 133.6, 138.0, 152.2, 185.2. HRMS *m*/*z* (ESI) calc'd for $C_{17}H_{14}N_3O_3S_2$ (M+H+) 372.0477, found 372.0483.

4-[5-Amino-2-benzoyl-4-cyano-1-(2-thienylsulfonyl)-1*H*-pyrrol-3-yl]benzenecarboxylate (A_{13}). 96% as brown solid, mp = 158-160 ^oC decomp., (CH_2Cl_2/EtOAc=20/1). ^1H NMR (DMSO-d_6) δ: 3.73 (s, 3H), 7.26-7.77 (m, 12H), 7.98-7.99 (m, 1H), 8.21-8.22 (m, 1H). ^{13}C NMR (DMSO-d_6) δ: 52.8, 75.1, 121.3, 128.7, 129.0, 129.4, 129.5, 129.9, 130.1, 133.6, 134.5, 134.8, 135.4, 136.9, 138.4, 139.0, 152.2, 166.1, 185.5. HRMS *m*/*z* (ESI) calc'd for $C_{24}H_{18}N_3O_5S_2$ (M+H+) 492.0688, found 492.0689.

2-Amino-5-benzoyl-4-(2-chloro-6-fluorophenyl)-1-(methylsulfonyl)-1*H*-pyrrole-3-carbonitrile (A_{14}). 88% as brown solid, mp = 140-142 ^oC, (CH_2Cl_2/EtOAc=20/1). ^1H NMR (DMSO-d_6) δ: 3.96 (s, 3H), 6.93-7.34 (m, 6H), 7.48 (d, J=6 Hz, 2H), 7.84 (s, 2H). ^{13}C NMR

(DMSO-d_6) δ: 43.9, 74.7, 114.5, 114.6, 114.8, 124.6, 125.7, 128.0, 128.4, 129.0, 132.3, 133.4, 134.2, 137.6, 153.1, 184.7. HRMS *m*/*z* (ESI) calc'd for $C_{19}H_{14}ClFN_3O_3S$ (M+H+) 418.0428, found 418.0424.

2-Amino-5-benzoyl-4-(2,6-dichlorophenyl)-1-(methylsulfonyl)-1*H*-pyrrole-3-carbonitrile (A_{15}). 88% as brown solid, mp = 115-117 ^oC decomp., (CH_2Cl_2/EtOAc=20/1). ^1H NMR (DMSO-d_6) δ: 3.90 (s, 3H), 7.01-7.27 (m, 6H), 7.45 (d, J=6 Hz, 2H), 7.80 (s, 2H). ^{13}C NMR (DMSO-d_6) δ: 43.8, 74.3, 114.4, 124.5, 128.1, 128.4, 128.6, 129.8, 131.8, 132.4, 133.0, 134.7, 137.8, 153.2, 184.7. HRMS *m*/*z* (ESI) calc'd for $C_{19}H_{14}Cl_2N_3O_3S$ (M+H+) 434.0133, found 434.0125.

2-Amino-5-benzoyl-4-(4-bromophenyl)-1-[(4-methoxyphenyl)sulfonyl]-1*H*-pyrrole-3-carboxamide (A_{16}). 38% as brown oil, (CH_2Cl_2/EtOAc=20/1). ^1H NMR (DMSO-d_6) δ: 3.84 (s, 3H), 7.08-7.92 (m, 16H). ^{13}C NMR (DMSO-d_6) δ: 56.6, 95.88, 115.4, 121.8, 122.6, 127.7, 128.9, 129.3, 130.5, 131.4, 131.8, 133.1, 139.3, 150.0, 164.9, 166.8, 186.7. HRMS *m*/*z* (ESI) calc'd for $C_{25}H_{21}BrN_3O_5S$ (M+H+) 554.0385, found 554.0383.

Ethyl 2-amino-5-benzoyl-1-[(4-methoxyphenyl)sulfonyl]-4-(3,4,5-trimethylphenyl)-1*H*-pyrrole-3-carboxylate (A_{17}). 76% as brown oil, (hexane/EtOAc=10/1). ^1H NMR (acetone-d_6) δ: 0.90 (t, J=9 Hz, J=6 Hz, 3H), 3.54-4.01 (m, 17H), 6.39 (s, 2H), 7.04-7.44 (m, 6H), 7.67 (d, J = 9 Hz, 2H), 7.80 (d, J=9 Hz, 1H), 8.06 (d, J=9 Hz, 2H). ^{13}C NMR (DMSO-d_6) δ: 14.1, 56.2, 56.6, 59.5, 60.3, 93.2, 108.5, 113.2, 114.5, 115.4, 122.1, 127.5, 128.2, 128.6, 129.5, 129.8, 130.7, 133.1, 133.5, 137.2, 138.9, 150.7, 151.8, 164.9, 165.1, 187.2. HRMS *m*/*z* (ESI) calc'd for $C_{30}H_{31}N_2O_9S$ (M+H+) 595.1750, found 595.1769.

General Procedure for the Synthesis of Pyrroles (B_1-B_5)

To a stirred solution of N-aryl- or N-alkylsulfonamidoacetophenone (0.2 mmol), aldehyde (0.26 mmol) and malononitrile (0.26 mmol) in acetonitrile (3 mL) was added Et_3N (0.073 mmol). The mixture was refluxed until the starting sulfonamide disappeared (TLC). After that the reaction mixture was cooled to room temperature and co-evaporated three times with toluene to dryness. Then it was dissolved in DMF (1 mL) and to the stirred solution was added DBU (0.4 mmol) under the nitrogen atmosphere. The stirring was maintained at room temperature for 12 hours. After that the reaction mixture was concentrated and purified by flash chromatography on silica gel (CH_2Cl_2/EtOAc=4/1) to afford pure pyrroles.

2-Amino-5-benzoyl-4-butyl-1*H*-pyrrole-3-carbonitrile (B_1). 43% as brown solid, mp = 128-130 ^oC, (CH_2Cl_2/EtOAc=5/1). ^1H NMR (DMSO-d_6) δ: 0.66 (t, J=6 Hz, 3H), 1.23-1.38 (m, 2H), 2.22 (t, J=6 Hz, J= 9Hz, 2H), 6.33 (s, 2H), 7.40-7.62 (m, 5H), 10.55 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 14.2, 23.9, 28.4, 77.7, 116.7, 121.2, 128.0, 128.9, 131.2, 138.2, 140.8, 150.7, 183.6. HRMS *m*/*z* (ESI) calc'd for $C_{16}H_{18}N_3O$ (M+H+) 254.1293, found 254.1280.

2-Amino-5-benzoyl-4-(5-bromo-3-pyridinyl)-1*H*-pyrrole-3-carbonitrile (B_2). 80% as yellow solid, mp = 269-271 ^oC decomp., (CH_2Cl_2/EtOAc=5/1). ^1H NMR (DMSO-d_6) δ: 6.48 (s, 2H), 7.05-7.24 (m, 5H), 7.57 (s, 1H), 8.16 (s, 1H), 8.36 (s, 1H), 11.24 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 77.2, 116.3, 119.7, 121.8, 128.2, 129.0, 130.5, 131.0, 131.5, 138.7, 139.8, 148.7, 149.4, 151.1, 183.9. HRMS *m*/*z* (ESI) calc'd for $C_{17}H_{12}BrN_4O$ (M+H+) 367.0194, found 397.0199.

2-Amino-5-benzoyl-4-(2,6-dichlorophenyl)-1*H*-pyrrole-3-carbonitrile (B_3). 70% as yellow solid, mp = 210-212 ^oC decomp., (CH_2Cl_2/EtOAc=5/1). ^1H NMR (DMSO-d_6) δ: 6.49 (s,

2H), 7.03-7.29 (m, 8H), 11.27 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 77.3, 115.8, 121.6, 127.7, 128.4, 129.6, 131.1, 131.2, 131.7, 135.2, 138.7, 151.1, 183.8. HRMS *m*/*z* (ESI) calc'd for $C_{18}H_{12}Cl_2N_3O$ (M+H+) 356.0357, found 356.0363.

2-Amino-5-benzoyl-4-(3,5-dibromophenyl)-1*H*-pyrrole-3-carbonitrile (B_4). 70% as yellow solid, mp = 278-280 ^{o}C decomp., (CH_2Cl_2/EtOAc=5/1). ^{1}H NMR (DMSO-d_6) δ: 6.45 (s, 2H), 7.04-7.24 (m, 7H), 7.51 (s, 1H), 11.27 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 76.9, 116.3, 121.5, 122.1, 128.1, 128.8, 131.4, 132.0, 132.5, 132.8, 137.0, 138.9, 151.0, 184.0. HRMS *m*/*z* (ESI) calc'd for $C_{18}H_{11}Br_2N_3ONa$ (M+Na+) 465.9167, found 465.9171.

2-Amino-5-benzoyl-4-(3,4,5-trimethoxyphenyl)-1*H*-pyrrole-3-carbonitrile (B_5). 19-78% as yellow solid, mp = 245-247 ^{o}C decomp., (CH_2Cl_2/EtOAc=4/1). ^{1}H NMR (DMSO-d_6) δ: 3.53 (s, 6H), 3.56 (s, 3H), 6.29 (s, 2H), 6.43 (s, 2H), 7.08-7.29 (m, 5H), 11.03 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 56.2, 60.4, 77.1, 108.2, 116.7, 120.8, 127.8, 128.3, 129.0, 131.1, 136.3, 137.7, 139.3, 151.0, 152.6, 184.0. HRMS *m*/*z* (ESI) calc'd for $C_{21}H_{20}N_3O_4$ (M+H+) 378.1454, found 378.1463.

General Procedure for the Synthesis of Pyrroles B_5-B_{15}.

To a stirred solution of N-methylsulfonamidoacetophenone (0.2 mmol), aldehyde (0.26 mmol) and a corresponding derivative of cyanoacetic acid (0.26 mmol) in EtOH (3 mL) were added 0.1 mmol of anhydrous granulated K_2CO_3 in one portion. The mixture was refluxed for 14 hours. After this time reaction the mixture was cooled to room temperature and concentrated. The crude product was purified by flash chromotography on silica gel with the indicated mixture of solvents to afford pure pyrroles.

2-Amino-5-benzoyl-4-(3,4,5-trimethoxyphenyl)-1*H*-pyrrole-3-carbonitrile (B_5). 80% as yellow solid, mp = 245-247 ^{o}C decomp., (CH_2Cl_2/EtOAc=5/1). ^{1}H NMR (DMSO-d_6) δ: 3.53 (s, 6H), 3.56 (s, 3H), 6.29 (s, 2H), 6.43 (s, 2H), 7.08-7.29 (m, 5H), 11.03 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 56.2, 60.4, 77.1, 108.2, 116.7, 120.8, 127.8, 128.3, 129.0, 131.1, 136.3, 137.7, 139.3, 151.0, 152.6, 184.0. HRMS *m*/*z* (ESI) calc'd for $C_{21}H_{20}N_3O_4$ (M+H+) 378.1454, found 378.1463.

2-Amino-5-benzoyl-4-(2-chloro-6-fluorophenyl)-1*H*-pyrrole-3-carbonitrile (B_6). 88% as yellow solid, mp = 232-233 ^{o}C decomp., (CH_2Cl_2/EtOAc=5/1). ^{1}H NMR (DMSO-d_6) δ: 6.43 (s, 2H), 6.85-7.20 (m, 8H), 11.26 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 77.7, 114.2, 114.6, 115.8, 122.0, 125.2, 125.5, 127.7, 127.9, 131.3, 131.4, 131.5, 134.6, 138.5, 151.1, 183.8. HRMS *m*/*z* (ESI) calc'd for $C_{19}H_{11}ClFN_3OK$ (M+K+) 378.0212, found 378.0209.

2-Amino-5-benzoyl-4-(4-methoxyphenyl)-1*H*-pyrrole-3-carbonitrile (B_7). 55% as brown solid, mp = 200-202 ^{o}C decomp., (CH_2Cl_2/EtOAc=4/1). ^{1}H NMR (DMSO-d_6) δ: 3.61 (s, 6H), 6.36 (s, 2H), 6.59 (d, J=9 Hz, 2H), 6.88-7.21 (m, 8H), 10.92 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.6, 77.1, 113.7, 120.7, 125.2, 127.9, 129.0, 130.9, 131.4, 136.0, 138.9, 151.0, 159.1, 184.1. HRMS *m*/*z* (ESI) calc'd for $C_{19}H_{16}N_3O_2$ (M+H+) 318.1243, found 318.1240.

2-Amino-5-(4-methoxybenzoyl)-4-(3,4,5-trimethoxyphenyl)-1*H*-pyrrole-3-carbonitrile (B_8). 85% as yellow solid, mp = 238-240 ^{o}C decomp., (CH_2Cl_2/EtOAc=2/1). ^{1}H NMR (acetone-d_6) δ: 3.60 (s, 6H), 3.63 (s, 3H), 3.71 (s, 3H), 6.03 (s, 2H), 6.38 (s, 2H), 6.64 (d, J=9 Hz, 2H), 7.36 (d, J=9 Hz, 2H), 10.34 (s, 1H). ^{13}C NMR (acetone-d_6) δ: 54.9, 55.5, 59.7, 77.2, 108.0, 112.7,

115.7, 121.0, 128.2, 131.0, 131.4, 134.5, 138.2, 149.8, 153.0, 162.0, 183.1. HRMS *m*/*z* (ESI) calc'd for $C_{22}H_{22}N_3O_5$ (M+H+) 408.1559, found 408.1568.

2-Amino-5-benzoyl-4-(4-bromophenyl)-1*H*-pyrrole-3-carbonitrile (B_9). 73% as yellow solid, mp = 297-299 ^oC decomp., (CH_2Cl_2/EtOAc=3/1). ^1H NMR (DMSO-d_6) δ: 6.43 (s, 2H), 6.94-7.26 (m, 9H), 11.08 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 77.0, 116.6, 121.1, 121.4, 128.0, 129.1, 131.0, 131.1, 132.2, 132.4, 134.6, 138.8, 151.0, 184.0. HRMS *m*/*z* (ESI) calc'd for $C_{18}H_{13}BrN_3O$ (M+H+) 366.0242, found 366.0230.

[5-Amino-4-(4-methoxybenzoyl)-3-(4-methoxyphenyl)-1*H*-pyrrol-2-yl](4-methoxyphenyl) methanone (B_{10}). 48% as yellow solid, mp = 213-214 ^oC decomp., (CH_2Cl_2/EtOAc=2/1). ^1H NMR (DMSO-d_6) δ: 3.52 (s, 6H), 3.60 (s, 3H), 3.63 (s, 3H), 6.24 (d, J=9 Hz, 2H), 6.43-6.57 (m, 8H), 7.02 (d, J=9 Hz, 2H), 7.12 (d, J=9 Hz, 2H), 10.94 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.6, 55.7, 106.6, 112.8, 112.9, 113.0, 121.6, 127.0, 130.6, 131.0, 131.7, 132.6, 133.0, 133.1, 150.9, 158.1, 160.9, 161.3, 184.6, 191.6. HRMS *m*/*z* (ESI) calc'd for $C_{27}H_{25}N_2O_5$ (M+H+) 457.1763, found 457.1755.

[**5-Amino-3-(4-nitrophenyl)-4-(phenylsulfonyl)-1*H*-pyrrol-2-yl](phenyl)methanone (B_{11}).** 28% as yellow solid, mp = 176-178 ^oC decomp., (CH_2Cl_2/EtOAc=4/1). ^1H NMR (DMSO-d_6) δ: 6.25 (s, 2H), 6.87-7.07 (m, 7H), 7.32-7.46 (m, 5H), 7.70 (d, J=9 Hz, 2H), 11.35 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 102.4, 121.9, 122.8, 126.1, 127.8, 128.6, 129.6, 130.1, 130.8, 133.0, 133.2, 138.7, 140.1, 144.0, 146.7, 147.6, 184.8. HRMS *m*/*z* (ESI) calc'd for $C_{23}H_{18}N_3O_5S$ (M+H+) 448.0967, found 448.0964.

[**5-Amino-3-(4-bromophenyl)-4-(phenylsulfonyl)-1*H*-pyrrol-2-yl](phenyl)methanone (B_{12}).** 40% as yellow solid, mp = 272-273 ^oC decomp., (CH_2Cl_2/EtOAc=4/1). ^1H NMR (DMSO-d_6) δ: 6.15 (s, 2H), 6.67 (d, J=6 Hz, 2H), 6.96-7.49 (m, 13H), 11.19 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 102.3, 122.7, 126.1, 127.8, 128.6, 129.4, 129.9, 130.6, 131.4, 131.7, 133.1, 133.6, 138.8, 144.2, 147.4, 185.0. HRMS *m*/*z* (ESI) calc'd for $C_{23}H_{18}BrN_2O_3S$ (M+H+) 448.0222, found 448.0214.

[**5-Amino-3-(3,4,5-trimethoxyphenyl)-4-(phenylsulfonyl)-1*H*-pyrrol-2-yl](phenyl)methanone (B_{13}).** 52% as yellow solid, mp = 212-213 ^oC decomp., (CH_2Cl_2/EtOAc=4/1). ^1H NMR (acetone-d_6) δ: 3.54 (s, 6H), 3.63 (s, 3H), 6.11 (s, 2H), 6.27 (s, 2H), 7.01-7.49 (m, 10H), 10.44 (s, 1H). ^{13}C NMR (acetone-d_6) δ: 43.5, 56.20, 60.38, 103.5, 110.6, 123.3, 127.2, 127.9, 128.9, 129.3, 131.0, 133.0, 133.6, 138.9, 140.0, 145.1, 147.7, 152.9, 185.6. HRMS *m*/*z* (ESI) calc'd for $C_{26}H_{25}N_2O_6S$ (M+H+) 493.1433, found 493.1437.

Ethyl 2-amino-5-benzoyl-1-[(4-methoxyphenyl)sulfonyl]-4-(3,4,5-trimethoxyphenyl)-1*H*-pyrrole-3-carboxylate (B_{14}). 57% as yellow solid, mp = 161-163 ^oC decomp., (CH_2Cl_2/EtOAc=4/1). ^1H NMR (DMSO-d_6) δ: 0.91 (t, J=9 Hz, J=6 Hz, 3H), 3.48 (s, 3H), 3.50 (s, 6H), 3.92 (q, J=9 Hz, J=6Hz, J=6 Hz, 2H), 6.17 (s, 2H), 6.19 (s, 2H), 6.97-7.15 (m, 5H), 10.87 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 14.5, 56.1, 58.9, 60.2, 96.3, 109.4, 122.2, 127.4, 128.4, 130.2, 134.8, 136.9, 139.9, 150.3, 151.7, 165.4, 185.0. HRMS *m*/*z* (ESI) calc'd for $C_{23}H_{24}N_2O_6Na$ (M+Na+) 447.1532, found 447.1516.

2-Amino-5-benzoyl-4-(2,6-dichlorophenyl)-1*H*-pyrrole-3-carboxamide (B_{15}). 48% as white solid, mp = 224-226 ^oC decomp., (CH_2Cl_2/EtOAc=2/1). ^1H NMR (DMSO-d_6) δ: 6.38 (s, 2H), 7.05-7.28 (m, 10H), 11.09 (s, 1H). ^{13}C NMR (DMSO-d_6) δ: 98.8, 120.7, 125.9, 127.1, 127.7, 128.7, 130.4, 131.3, 133.0, 135.7, 139.8, 150.3, 167.2, 184.0. HRMS *m*/*z* (ESI) calc'd for $C_{18}H_{14}ClN_3O_2$ (M+H+) 374.0463, found 374.0465.

General Procedure for the Synthesis of Pyrroles B_{16}-B_{21}.

To a solution of **1** or **2** (0.676 mmol), selected aldehyde (0.879 mmol) and cyanoacetamide (0.072 g, 0.879 mmol) in EtOH was added anhydrous granulated K_2CO_3 (0.052g, 0.372 mmol) in one portion. The mixture was purged with nitrogen for 5 min, and then refluxed for 14 hours under the nitrogen atmosphere. After this time the reaction mixture was cooled to room temperature, the formed precipitate was collected by filtration and washed on the filter with EtOH (2 mL) and diethyl ether. The mother liquor was evaporated and the residue subjected to column chromatography with MeOH/CH_2Cl_2=1/70 to 1/50 gradient.

2-Amino-5-benzoyl-4-[4-(methoxy)phenyl]-1*H*-pyrrole-3-carboxamide (B_{16}). 93% as yellow solid, mp = 256-257 ^oC decomp., (MeOH/CH_2Cl_2=3/47). ^1H NMR (DMSO-d_6) δ: 3.66 (s, 3H), 6.37 (bs, 2H), 6.65 (d, J = 8.6 Hz, 2H), 6.93 (d, J = 8.6 Hz, 2H), 6.99 (t, J = 7.7 Hz, J = 15.3 Hz, 1H), 7.10 (d, J = 8.6 Hz, 2H), 7.16 (t, J = 7.6 Hz, J = 15.4 Hz, 2H), 10.84 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.5, 99.5, 114.0, 121.9, 126.4, 127.6, 128.3, 132.2, 139.7, 149.7, 159.0, 167.7, 184.3. HRMS m/z (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+H^+$) 336.1348, found 336.1348.

2-Amino-5-(4-methoxybenzoyl)-4-(4-methoxyphenyl)-1*H*-pyrrole-3-carboxamide (B_{17}). 89% as yellow solid, mp = 260-262 ^oC decomp., (MeOH/CH_2Cl_2=3/47). ^1H NMR (DMSO-d_6) δ: 3.66 (s, 3H), 3.68 (s, 3H), 6.33 (bs, 2H), 6.54 (d, J = 8.4 Hz, 2H), 6.70 (d, J = 8.3 Hz, 2H), 6.94 (d, J = 8.3 Hz, 2H), 7.10 (d, J = 8.4 Hz, 2H), 10.80 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.6, 99.1, 113.0, 114.1, 121.9, 126.7, 130.4, 131.7, 132.0, 132.3, 149.6, 159.2, 160.8, 167.9, 183.6. HRMS m/z (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+H^+$) 366.1454, found 366.1445.

2-Amino-5-[4-(benzyloxy)benzoyl]-4-[4-(benzyloxy)phenyl]-1*H*-pyrrole-3-carboxamide (B_{18}). 83% as yellow solid, mp = 234-235 ^oC decomp., (MeOH/CH_2Cl_2=1/50). ^1H NMR (DMSO-d_6) δ: 5.02 (s, 4H), 6.35 (bs, 2H), 6.64 (d, J = 6.3 Hz, 2H), 6.77 (d, J = 8.0 Hz, 2H), 6.96 (d, J = 6.6 Hz, 2H), 7.10 (d, J = 6.6 Hz, 2H), 7.35 (m, 10H), 10.79 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 69.3, 69.4, 98.5, 113.2, 114.3, 121.3, 126.4, 127.4, 127.5, 127.7, 127.8, 128.3, 129.8, 131.2, 131.7, 131.8, 136.6, 136.8, 149.0, 157.9, 159.4, 167.3, 183.0. HRMS m/z (ESI+) calc'd for $C_{14}H_7N_3O_3$ ($M+Na^+$) 540.1899, found 540.1898.

2-Amino-5-[4-(benzyloxy)-3-methoxybenzoyl]-4-[4-(benzyloxy)phenyl]-1*H*-pyrrole-3-carboxamide (B_{19}). 86% as yellow solid, mp = 208-209 ^oC decomp., (MeOH/CH_2Cl_2=1/50). ^1H NMR (DMSO-d_6) δ: 3.62 (s, 3H), 4.98 (s, 2H), 4.99 (s, 2H) 6.34 (bs, 2H), 6.71 (d, J = 1.7 Hz, 1H), 6.76 (m, 4H), 7.00 (d, J = 6.3 Hz, 2H), 7.34 (m, 10H), 10.86 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.2, 69.5, 70.1, 98.5, 111.6, 112.2, 114.2, 121.2, 121.4, 126.4, 127.6, 127.8, 128.3, 128.4, 131.3, 131.6, 132.4, 136.8, 136.8, 147.6, 149.1, 149.2, 157.8, 167.3, 183.1. HRMS m/z (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+Na^+$) 570.2005, found 570.2007.

2-Amino-5-[4-(benzyloxy)benzoyl]-4-[4-(benzyloxy)-3-methoxyphenyl]-1*H*-pyrrole-3-carboxamide (B_{20}). 86% as yellow solid, mp = 190-191 ^oC decomp., (MeOH/CH_2Cl_2=1/50). ^1H NMR (DMSO-d_6) δ: 3.53 (s, 3H), 4.99 (s, 2H), 5.01 (s, 2H), 6.36 (bs, 2H), 6.51 (s, 1H), 6.61 (d, J = 8.3 Hz, 2H), 6.70 (d, J = 7.8 Hz, 1H), 6.89 (d, J = 8.2 Hz, 1H), 7.13 (d, J = 8.3 Hz, 2H), 7.34 (m, 10H), 10.83 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.8, 69.9, 70.7 98.9, 113.6, 113.9, 115.4, 121.8, 123.3, 127.5, 128.0, 128.1, 128.2, 128.3, 128.8, 128.9, 130.2, 131.9, 132.6, 137.5, 148.0, 149.1, 149.6, 160.0, 167.8, 183.6. HRMS m/z (ESI^+) calc'd for $C_{14}H_7N_3O_3$ ($M+H^+$) 548.2185, found 548.2172.

2-Amino-5-[4-(benzyloxy)-3-methoxybenzoyl]-4-[4-(benzyloxy)-3-methoxyphenyl]-1*H*-pyrrole-3-carboxamide (B_{21}). Mixture of 2 mL of dioxane and 4 mL of ethanol was used as a

chromatography solvent. 84% as yellow solid, mp = 188-190 ^{o}C decomp., (MeOH/CH_2Cl_2=1/50). ^{1}H NMR (DMSO-d_6) δ: 3.53 (s, 3H), 3.61 (s, 3H), 4.95 (s, 2H), 4.98 (s, 2H), 6.35 (bs, 2H), 6.53 (s, 1H), 6.54-6.80 (m, 5H), 6.89 (d, J = 8.2 Hz, 2H), 7.34 (m, 10H), 10.83 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.6, 55.7, 70.8, 70.9, 98.9, 111.9, 112.8, 113.9, 115.2, 121.7, 121.8, 123.1, 128.1, 128.2, 128.3, 128.7, 128.8, 132.0, 133.3, 137.3, 137.5, 147.9, 148.1, 148.9, 149.6, 149.7, 167.8, 183.7. HRMS m/z (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+H$^+$) 578.2291, found 578.2289.

General Procedure for the Synthesis of Protected Rigidins C_1-C_4.

A selected pyrrolocarboxamide **B_{18}-B_{21}** (0.3478 mmol) was co-evaporated with toluene (5 mL) and dissolved in diglyme (3 mL, extra dry grade). Nitrogen was bubbled through the mixture for 5 min, and 0.20 mL (0.3825 mmol) of oxalyl chloride (2M in CH_2Cl_2) were added dropwise during an intense stirring of the mixture. After the addition the mixture was heated at 130 ^{o}C for 1h and then 110 ^{o}C for 5h under the atmosphere of nitrogen. After that time the solvent was evaporated under reduced pressure and 5 mL of 50% MeOH in CH_2Cl_2 were added to the solid in the flask. The formed precipitate was filtered off, rinsed with 2 mL of MeOH followed by 5 mL of diethyl ether. The mother liquor was evaporated and the residue was chromatographed on silica gel using 30/1 to 30/5 of CH_2Cl_2/MeOH gradient.

6-[4-(Benzyloxy)benzoyl]-5-[4-(benzyloxy)phenyl]-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (C_1). 81% as a brown powder, mp > 300 ^{o}C decomp., (MeOH/CH_2Cl_2=4/30). ^{1}H NMR (DMSO-d_6) δ: 5.02 (s, 2H), 5.06 (s, 2H), 6.69 (d, J = 8.8 Hz, 2H), 6.74 (d, J = 8.8 Hz, 2H), 7.07 (d, J = 8.7 Hz, 2H), 7.29-7.40 (m, 12H), 10.71 (bs, 1H), 11.31 (bs, 1H), 11.94 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 69.8, 70.0, 98.9, 113.8, 114.3, 128.0, 128.1, 128.3, 128.5, 128.9, 129.0, 131.7, 132.8, 136.8, 137.6, 151.3, 158.1, 160.3, 161.2, 185.7. HRMS m/z (ESI$^-$) calc'd for $C_{14}H_7N_3O_3$ (M-H$^+$) 542.1721, found 542.1714.

6-[4-(Benzyloxy)-3-methoxybenzoyl]-5-[4-(benzyloxy)phenyl]-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (C_2). 74% as a brown powder, mp > 300 ^{o}C decomp., (MeOH/CH_2Cl_2=4/30). ^{1}H NMR (DMSO-d_6) δ: 3.57 (s, 3H), 5.01 (s, 2H), 5.05 (s, 2H), 6.72 (s, 1H), 6.87-7.10 (m, 7H), 7.37 (m, 10H), 10.72 (bs, 1H), 11.27 (bs, 1H), 11.94 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 56.3, 70.3, 71.2, 99.1, 114.0, 114.1, 123.5, 125.4, 125.6, 127.9, 128.0, 128.2, 128.3, 128.9, 131.9, 132.8, 137.4, 137.8, 142.0, 149.2, 150.9, 151.7, 158.5, 160.1, 185.7. HRMS m/z (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+Na$^+$) 596.1798, found 596.1792.

6-[4-(Benzyloxy)benzoyl]-5-[4-(benzyloxy)-3-methoxyphenyl]-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (C_3). 76% as an yellow powder, mp > 300 ^{o}C decomp., (MeOH/CH_2Cl_2=4/30). ^{1}H NMR (DMSO-d_6) δ: 3.60 (s, 3H), 4.97 (s, 2H), 5.03 (s, 2H), 6.66-6.74 (m, 5H), 7.34-7.38 (m, 12H), 10.70 (bs, 1H), 11.48 (bs, 1H), 11.96 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.9, 70.0, 70.6, 98.9, 113.1, 114.2, 116.2, 124.4, 125.5, 125.8, 128.1, 128.3, 128.5, 128.9, 129.0, 129.1, 131.2, 131.6, 137.0, 137.7, 151.3, 160.3, 161.5, 185.8. HRMS m/z (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+Na$^+$) 596.1798, found 596.1804.

6-[4-(Benzyloxy)-3-methoxybenzoyl]-5-[4-(benzyloxy)-3-(methoxy)phenyl]-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (C_4). 72% as an yellow powder, mp > 300 ^{o}C decomp., (MeOH/CH_2Cl_2=4/30). ^{1}H NMR (DMSO-d_6) δ: 3.52 (s, 3H), 3.55 (s, 3H), 4.98 (s, 2H), 5.04 (s, 2H), 6.74 (s, 1H), 6.78 (s, 1H), 6.88 (d, J = 8.4 Hz, 1H), 6.93 (d, J = 1.5 Hz, 1H), 7.10

(dd, *J* = 1.6 Hz, *J* = 8.3 Hz, 1H), 7.31-7.39 (m, 10H), 10.73 (bs, 1H), 11.27 (bs, 1H), 11.96 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.6, 55.7, 70.5, 70.7, 98.8, 112.7, 113.2, 123.3, 124.3, 125.5, 125.6, 128.0, 128.1, 128.2, 128.4, 128.8, 128.9, 131.4, 137.0, 137.6, 141.9, 147.7, 148.1, 148.3, 151.1, 151.2, 160.2, 185.7. HRMS *m*/*z* (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+Na$^+$) 626.1903, found 626.1908.

Synthesis of Rigidins A, B, C and D.

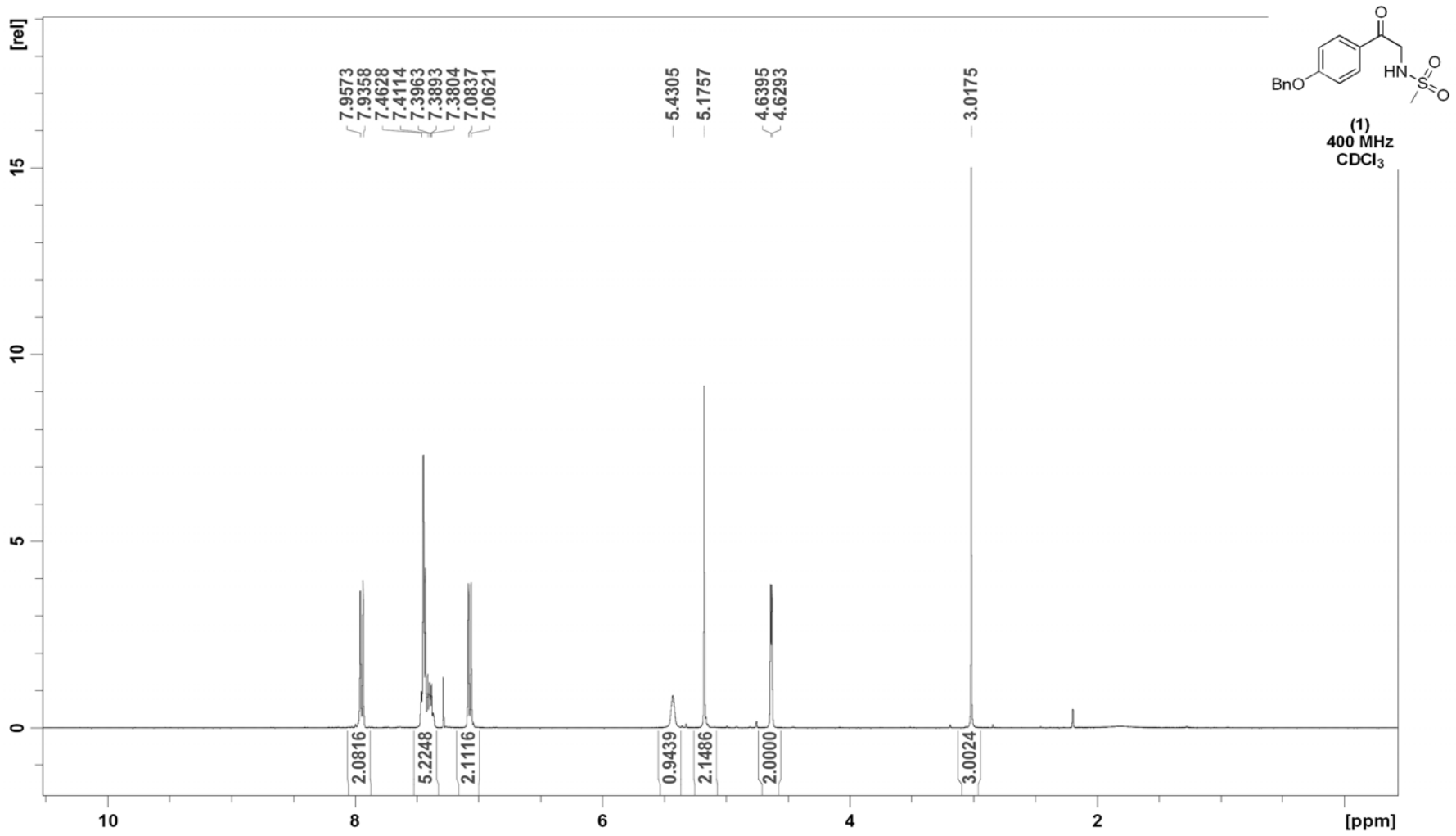
A selected protected rigidin **C_1-C_4** (0.0735 mmol) and Pd/C (20 mg, 10%) were suspended in DMF (0.3 mL) and MeOH (1 mL). The suspension was degassed three times by the freeze-pump-thaw method at -196 ^{o}C and then stirred under a hydrogen balloon for 3h. After that time the solvent was lyophilized, the crude residue was dissolved in MeOH/CH_2Cl_2 and subjected to silica gel column chromatography (MeOH/CH_2Cl_2/AcOH=10/50/1) to afford pure alkaloids.

6-(4-Hydroxybenzoyl)-5-(4-hydroxyphenyl)-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (Rigidin A). 94.7% (0.0254 g) as yellow solid, mp > 300 ^{o}C decomp., (CH_2Cl_2/MeOH/AcOH = 50/10/1, R_f = 0.43) ^{1}H NMR (DMSO-d_6) δ: 6.46 (d, *J* = 8.5 Hz, 2H), 6.47 (d, *J* = 8.6 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.6 Hz, 2H), 9.28 (bs, 1H), 10.02 (bs, 1H), 10.63 (bs, 1H), 11.32 (bs, 1H), 11.84 (bs, 1H). ^{13}C NMR (CD_3OD) δ: 98.5, 113.6, 114.1, 123.9, 128.5, 129.3, 131.5, 132.3, 156.0, 160.2, 160.8, 162.9, 185.4. HRMS *m*/*z* (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+Na$^+$) 386.0753, found 386.0742.

6-(4-Hydroxy-3-methoxybenzoyl)-5-(4-hydroxyphenyl)-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (Rigidin B). 95% as brown solid, mp > 300 ^{o}C decomp., (CH_2Cl_2/MeOH/AcOH = 50/10/1, R_f = 0.41). ^{1}H NMR (DMSO-d_6) δ: 3.56 (s, 3H), 6.47 (d, *J* = 8.5 Hz, 2H), 6.57 (d, *J* = 8.2 Hz, 1H), 6.93 (d, *J* = 1.6 Hz, 1H), 6.99-7.01 (m, 3H), 9.30 (bs, 1H), 10.54 (bs, 1H), 11.99 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.7, 98.8, 113.7, 114.3, 115.0, 123.6, 124.2, 125.3, 128.8, 129.6, 133.0, 147.0, 150.7, 152.7, 156.9, 160.7, 185.8. HRMS *m*/*z* (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+H$^+$) 394.1039, found 394.1032.

6-(4-Hydroxybenzoyl)-5-(4-hydroxy-3-methoxyphenyl)-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (Rigidin C). 96% as dark yellow solid, mp > 300 ^{o}C decomp., (CH_2Cl_2/MeOH/AcOH = 50/10/1, R_f = 0.41). ^{1}H NMR (DMSO-d_6) δ: 3.58 (s, 3H), 6.53 (m, 3H), 6.69-6.76 (m, 2H), 7.33 (d, *J* = 8.5 Hz, 2H), 8.69 (bs, 1H), 9.66 (bs, 1H), 10.21 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 56.2, 98.7, 114.8, 115.6, 116.4, 123.5, 125.5, 129.3, 131.7, 132.1, 135.4, 141.8, 147.1, 148.1, 151.1, 161.1, 185.7. HRMS *m*/*z* (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+H$^+$) 394.1039, found 394,1028.

6-(4-Hydroxy-3-methoxybenzoyl)-5-(4-hydroxy-3-methoxyphenyl)-1*H*-pyrrolo[2,3-*d*]pyrimidine-2,4(3*H*,7*H*)-dione (Rigidin D). 93% as maroon solid, mp > 300 ^{o}C decomp., (CH_2Cl_2/MeOH/AcOH = 50/10/1, R_f = 0.38). ^{1}H NMR (DMSO-d_6) δ: 3.52 (s, 3H), 3.54 (s, 3H), 6.49 (d, *J* = 8.1 Hz, 1H), 6.58 (d, *J* = 8.2 Hz, 1H), 6.66 (dd, *J* = 1.7 Hz, *J* = 8.1 Hz, 1H), 6.73 (d, *J* = 1.6 Hz, 1H), 6.93 (d, *J* = 1.6 Hz, 1H), 7.05 (dd, *J* = 1.6 Hz, *J* = 8.2 Hz, 1H), 8.89 (bs, 1H), 9.65 (bs, 1H), 10.60 (bs, 1H), 11.83 (bs, 1H). ^{13}C NMR (DMSO-d_6) δ: 55.8, 55.8, 98.8, 113.7, 114.7, 116.5, 123.7, 124.0, 124.8, 125.3, 128.8, 129.7, 146.3, 146.6, 146.9, 150.8, 160.6, 185.8. HRMS *m*/*z* (ESI$^+$) calc'd for $C_{14}H_7N_3O_3$ (M+Na$^+$) 446.0964, found 446.0964.


[rel]
15
10
5
0
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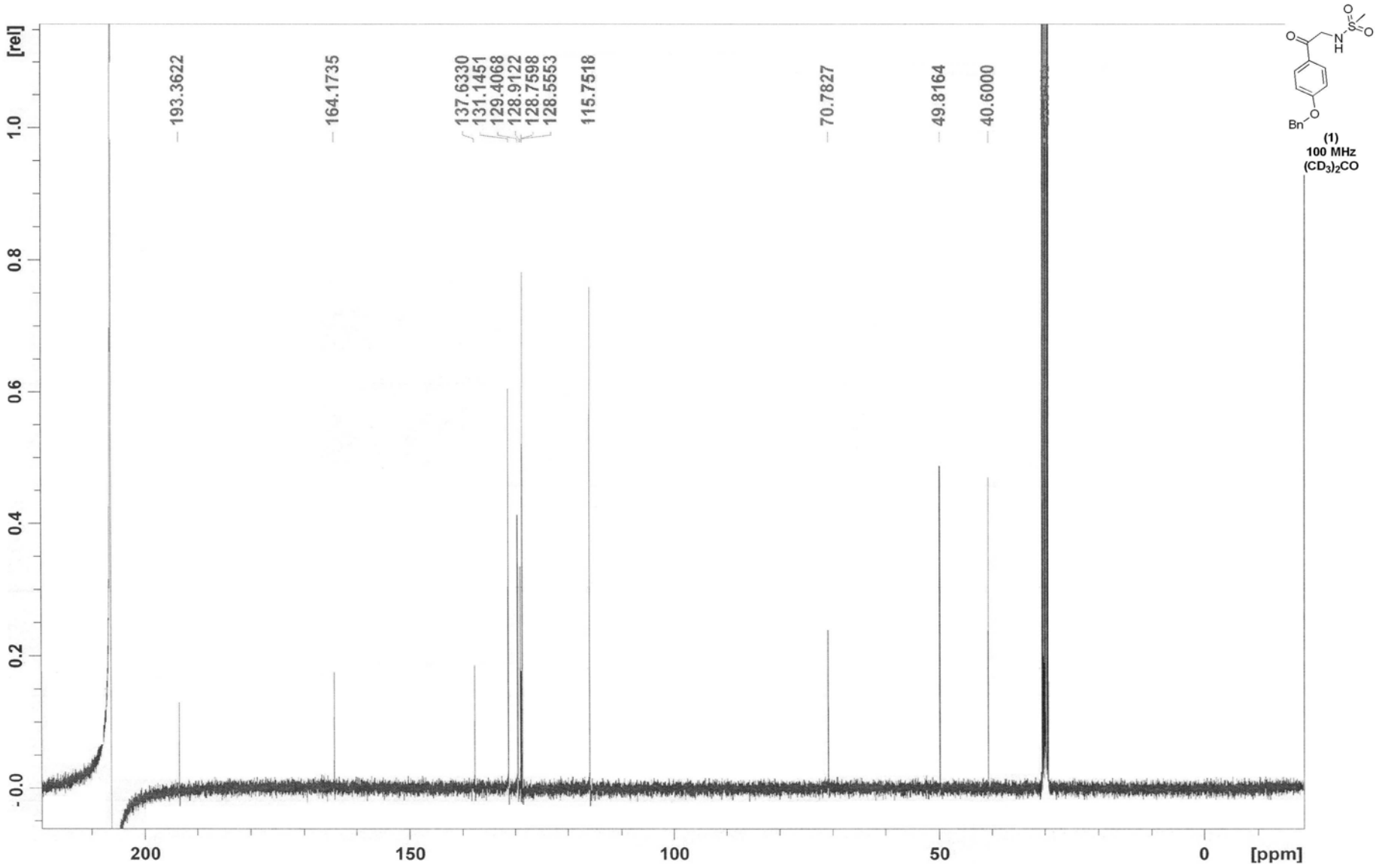
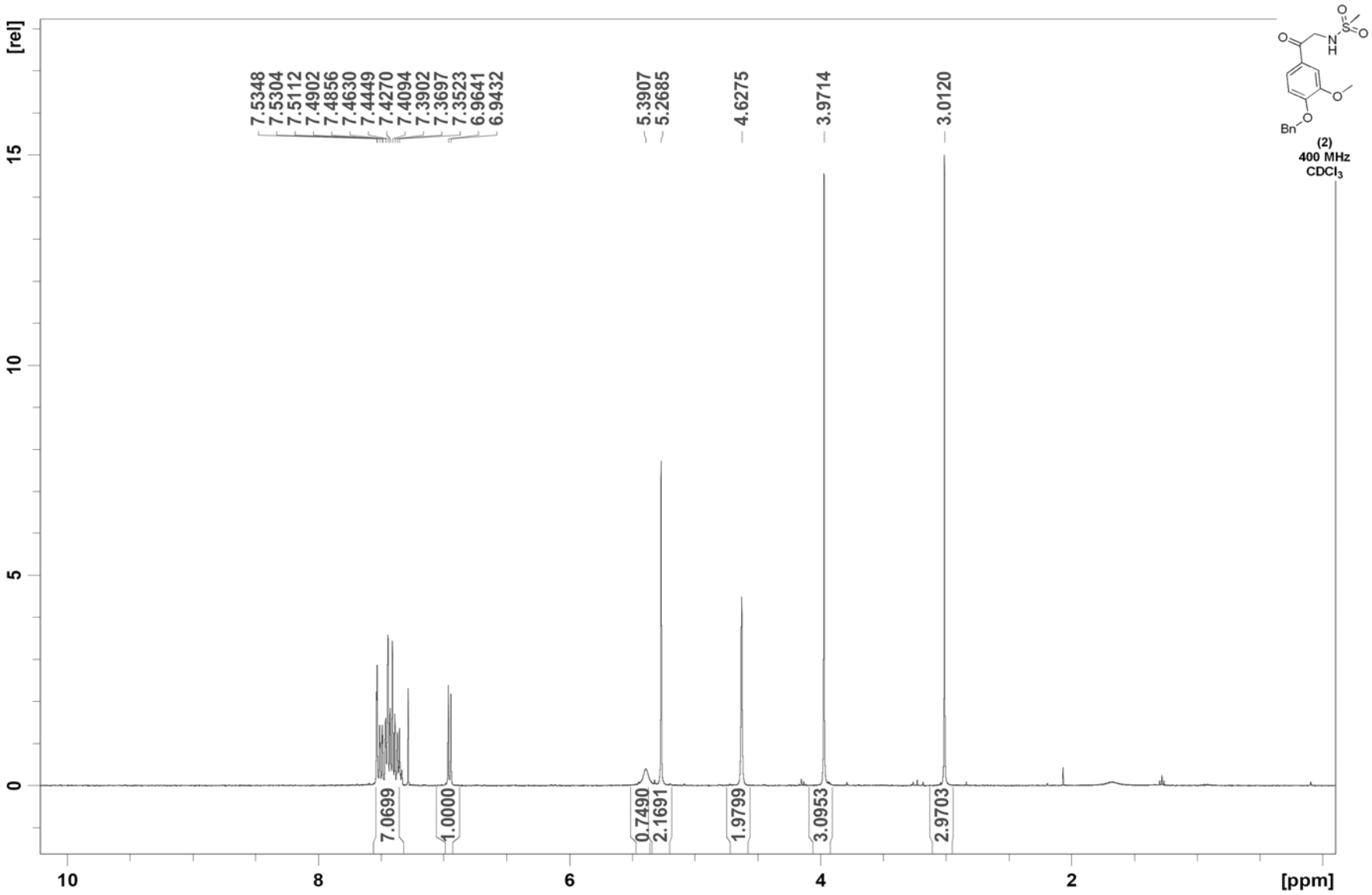
7.4628
7.4114
7.3963
7.3893
7.3804
7.0837
7.0621
5.4305
5.1757
4.6395
4.6293
3.0175
2.0816
5.2248
2.1116
0.9439
2.1486
2.0000
3.0024
10
8
6
4
2
[ppm]
BnO
HN
O
O
O
(1)
400 MHz
CDCl3


193.3622
164.1735
137.6330
131.1451
129.4068
128.9122
128.7598
128.5553
115.7518
70.7827
49.8164
40.6000
[rel]
1.0
0.8
0.6
0.4
0.2
-0.0
200
150
100
50
0
[ppm]
Bn
(1)
100 MHz
(CD3)2CO


[rel]
15
10
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7.5304
7.5112
7.4902
7.4856
7.4630
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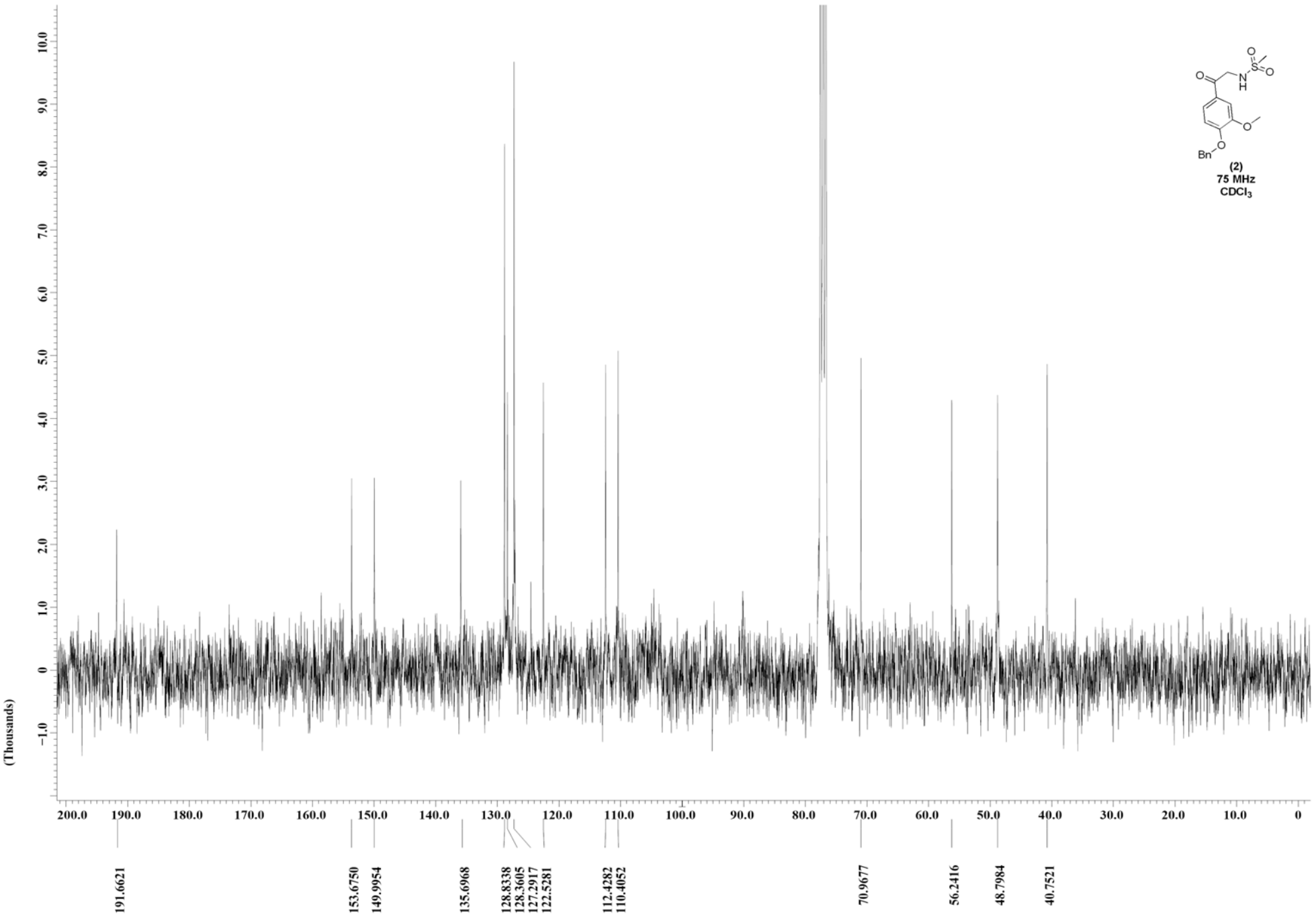
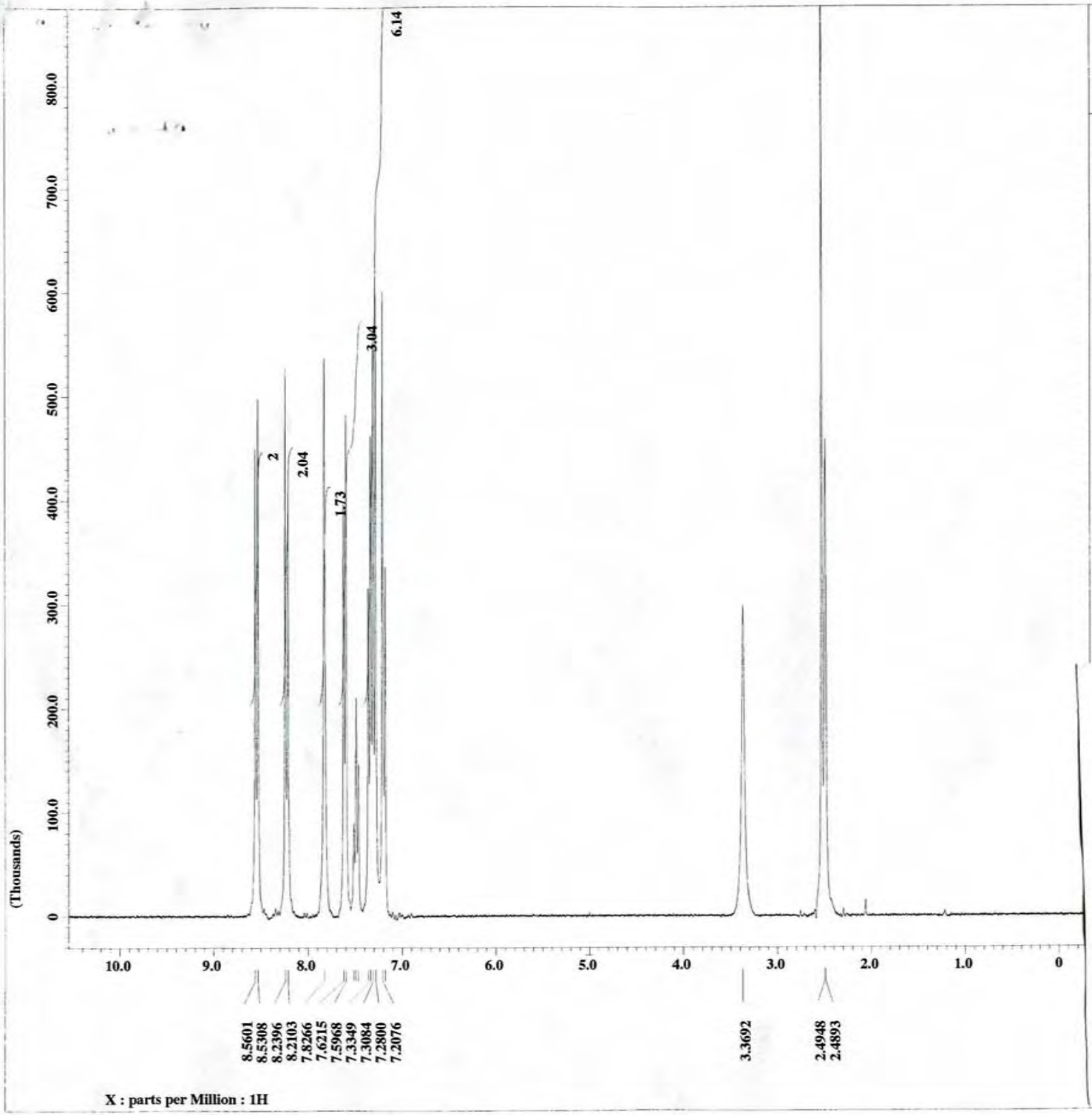
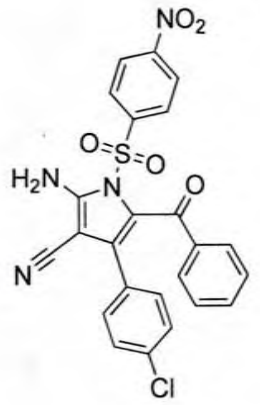
7.3902
7.3697
7.3523
6.9641
6.9432
5.3907
5.2685
4.6275
3.9714
3.0120
7.0699
1.0000
0.7490
2.1691
1.9799
3.0953
2.9703
10
8
6
4
2
[ppm]
Bn
(2)
400 MHz
CDCl3


(2)
75 MHz
CDCl3
(Thousands)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
191.6621
153.6750
149.9954
135.6968
128.8338
128.3605
127.2917
122.5281
112.4282
110.4052
70.9677
56.2416
48.7984
40.7521
X : parts per Million : 13C

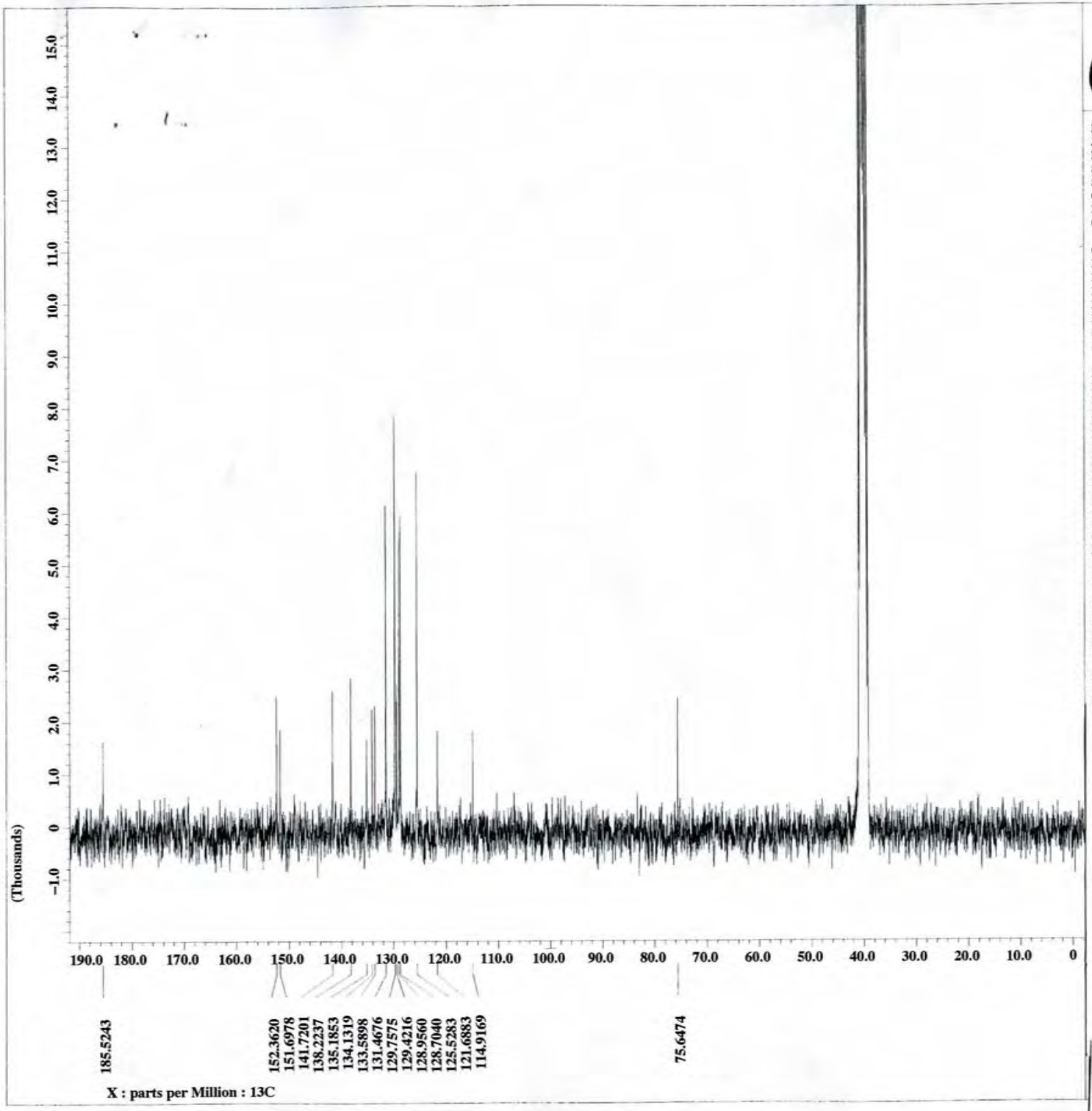


JEOL

Filename	= 1d_spectrum-157.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#411015
Solvent	= DMSO-D6
Creation_time	= 12-DEC-2010 11:18:33
Revision_time	= 12-DEC-2010 16:25:17
Current_time	= 12-DEC-2010 16:25:25
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15

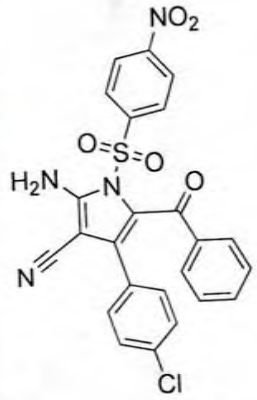


A_1
300 MHz, DMSO-d_6

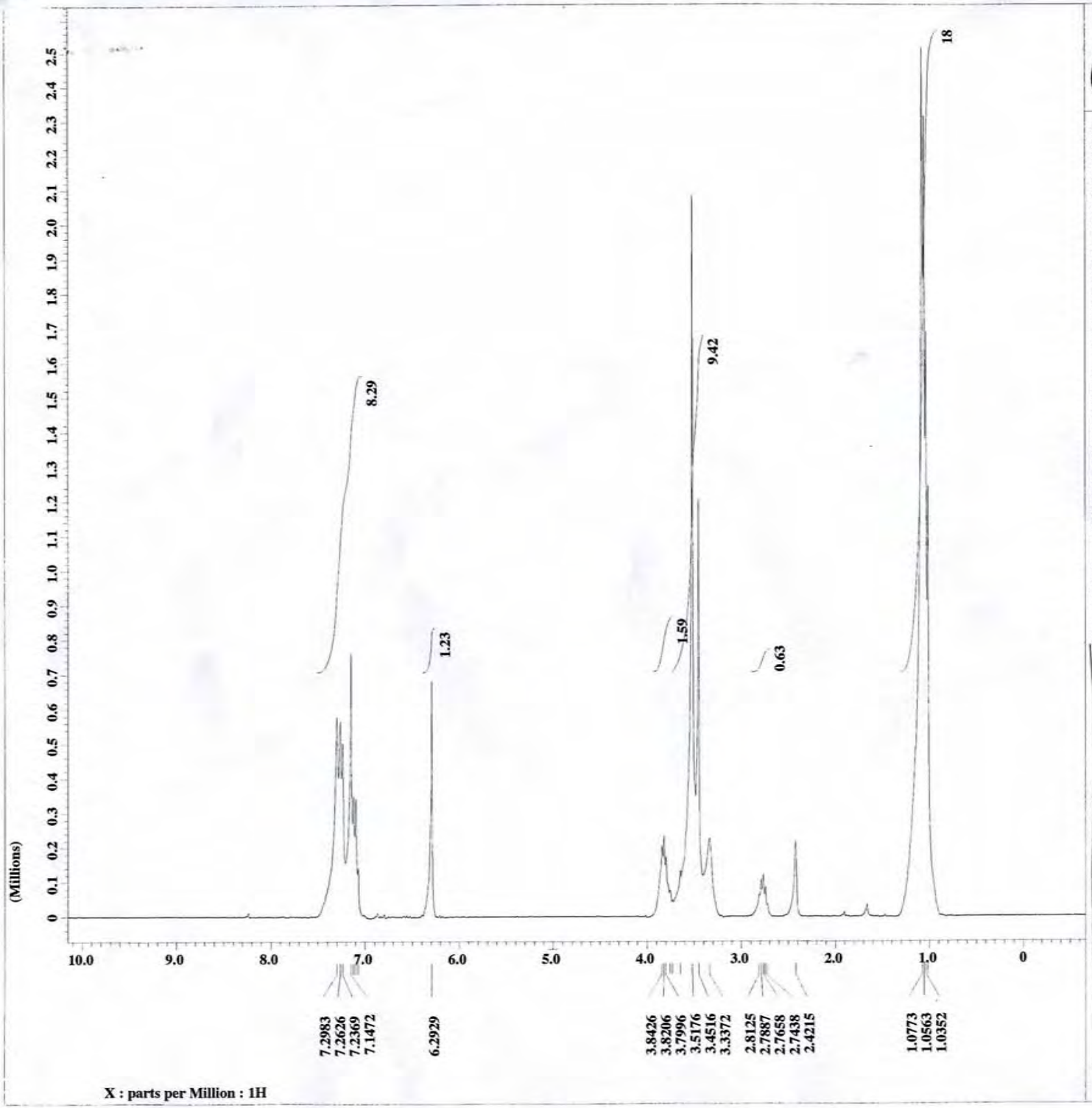


JEOL

Filename	= 1d_13c_spectrum-92.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#412896
Solvent	= DMSO-D6
Creation_time	= 12-DEC-2010 12:52:00
Revision_time	= 12-DEC-2010 16:18:47
Current_time	= 12-DEC-2010 16:18:51
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 2000
Total_scans	= 2000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]

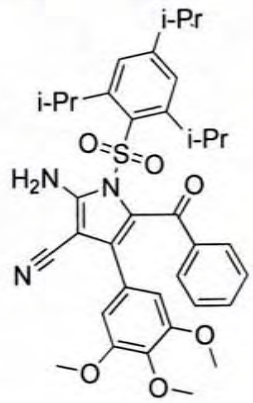


A_1
75 MHz, DMSO-d_6

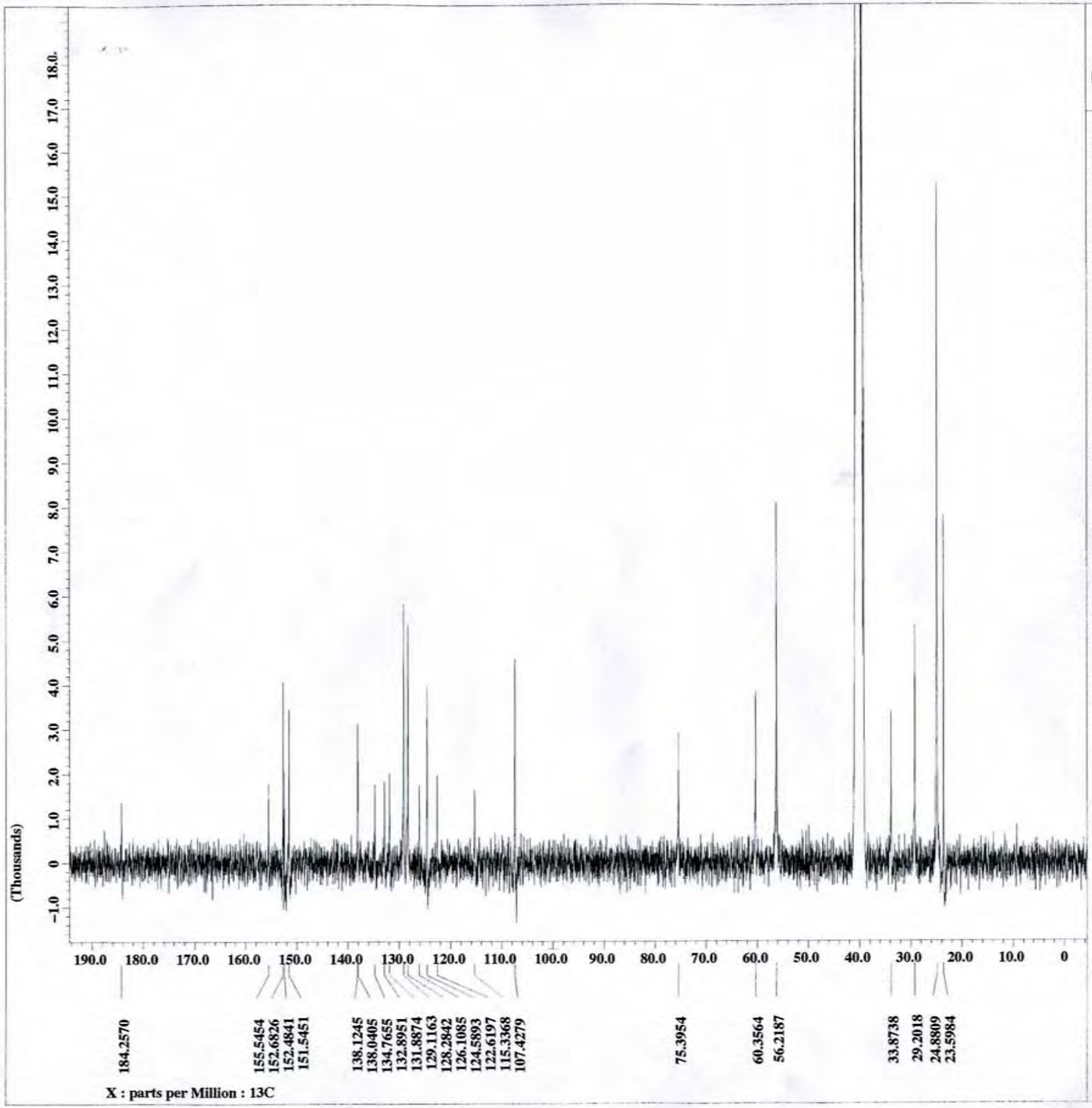


JEOL

Filename	= 1d_spectrum-96.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#406570
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 11:11:07
Revision_time	= 8-DEC-2010 11:38:55
Current_time	= 8-DEC-2010 11:38:59
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]

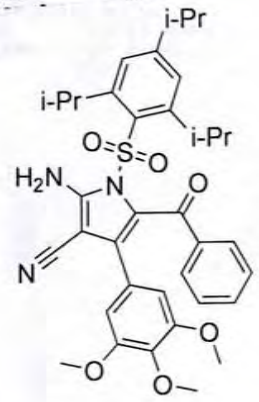


A_2
300 MHz, DMSO-d_6

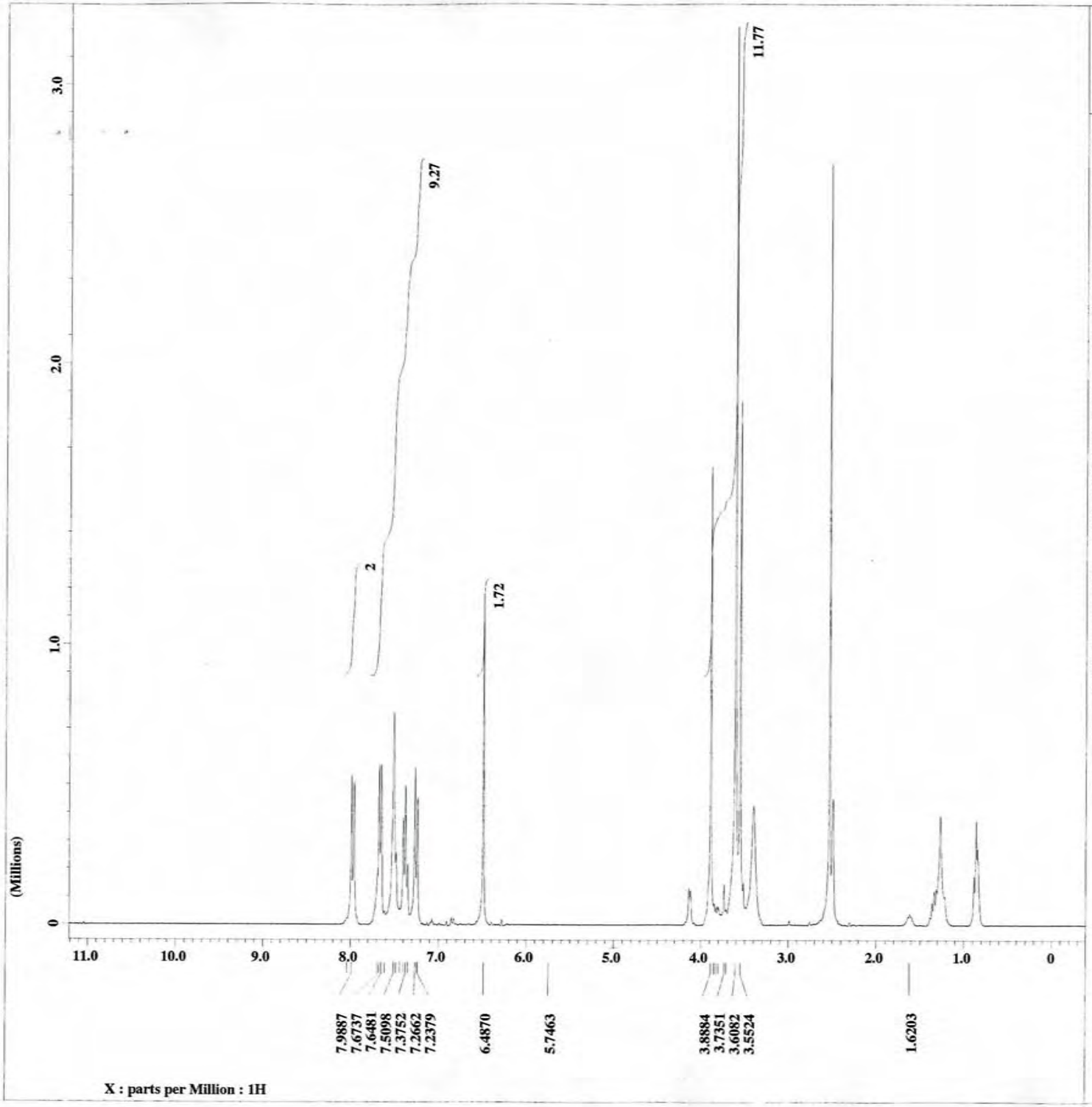


JEOL

Filename	= 1d_13c_spectrum-59.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#412344
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 12:51:02
Revision_time	= 8-DEC-2010 14:03:10
Current_time	= 8-DEC-2010 14:03:30
Comment	= Single Pulse with Bro
Data_format	= 1D REAL
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 2000
Total_scans	= 2000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15



A_2
75 MHz, DMSO-d_6



JEOL

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Filename          = 1d_spectrum-194.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#487424
Solvent           = DMSO-D6
Creation_time     = 17-DEC-2010 13:25:54
Revision_time     = 17-DEC-2010 13:41:47
Current_time      = 17-DEC-2010 13:41:51

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 21.8[dC]
Unblank_time      = 2[us]
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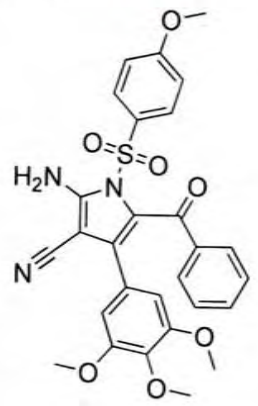
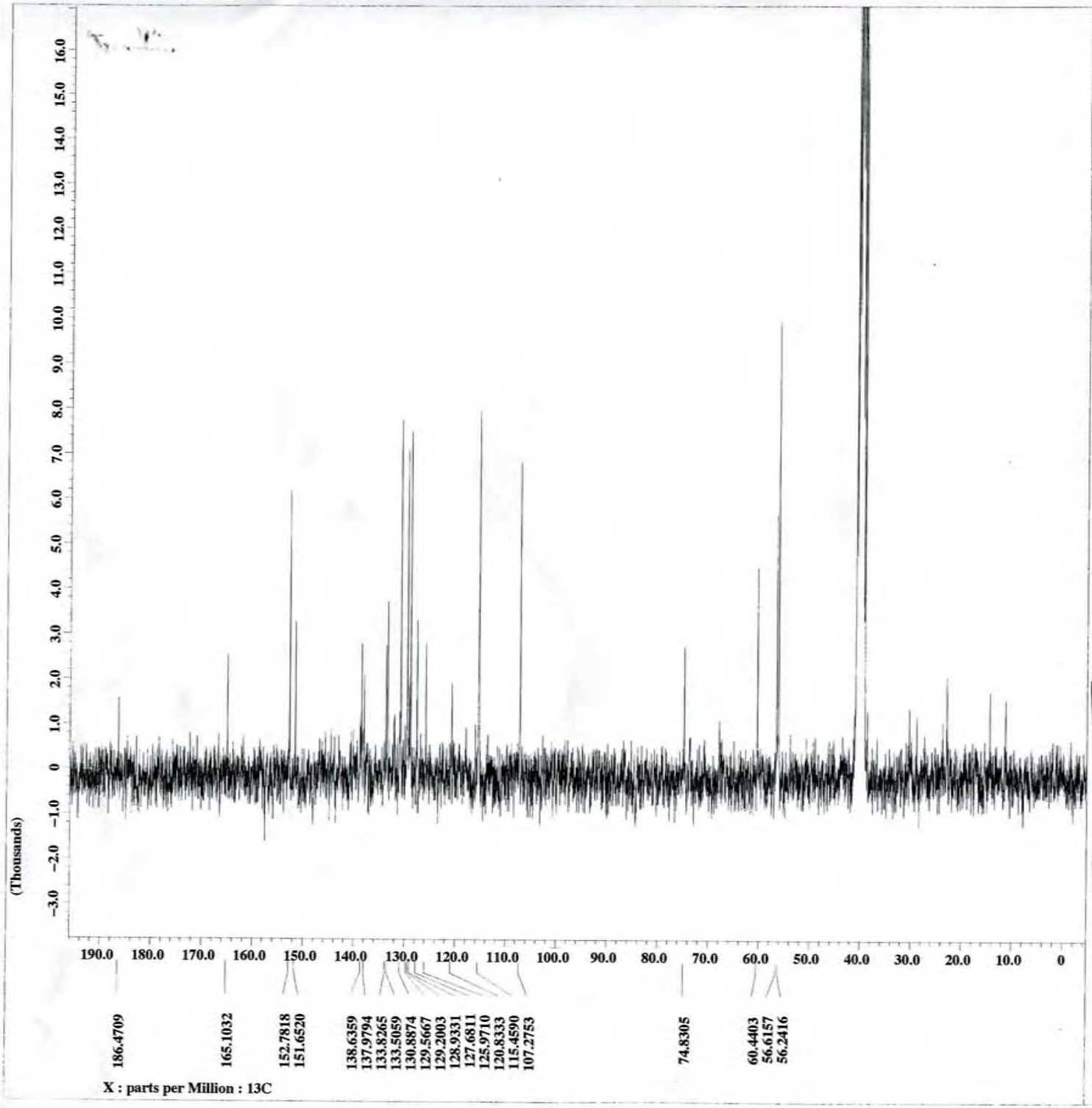
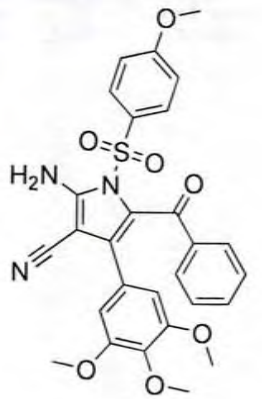


A_3
300 MHz, DMSO-d_6

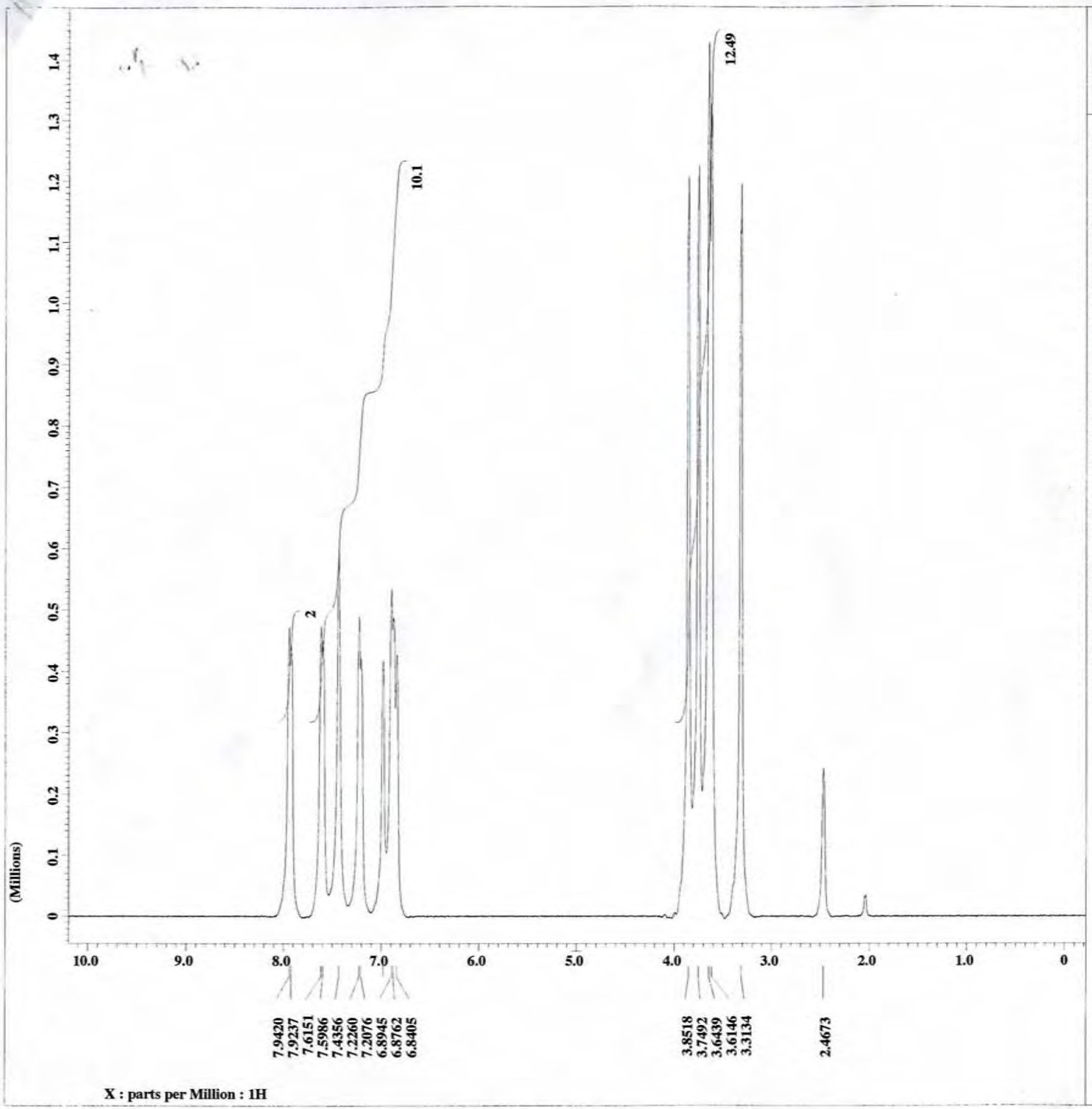


JEOL

Filename	= 1d_13c_spectrum-70.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#563071
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 16:16:45
Revision_time	= 9-DEC-2010 18:36:28
Current_time	= 9-DEC-2010 18:37:30
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1000
Total_scans	= 1000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]



A_3
75 MHz, DMSO-d_6



JEOL

Filename	= 1d_spectrum-145.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#469479
Solvent	= DMSO-D6
Creation_time	= 10-DEC-2010 12:56:00
Revision_time	= 10-DEC-2010 19:42:22
Current_time	= 10-DEC-2010 19:42:29
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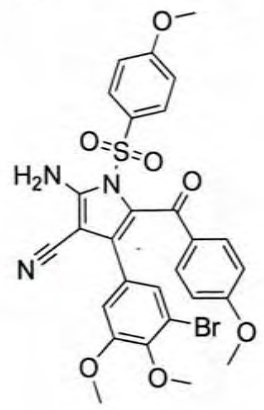
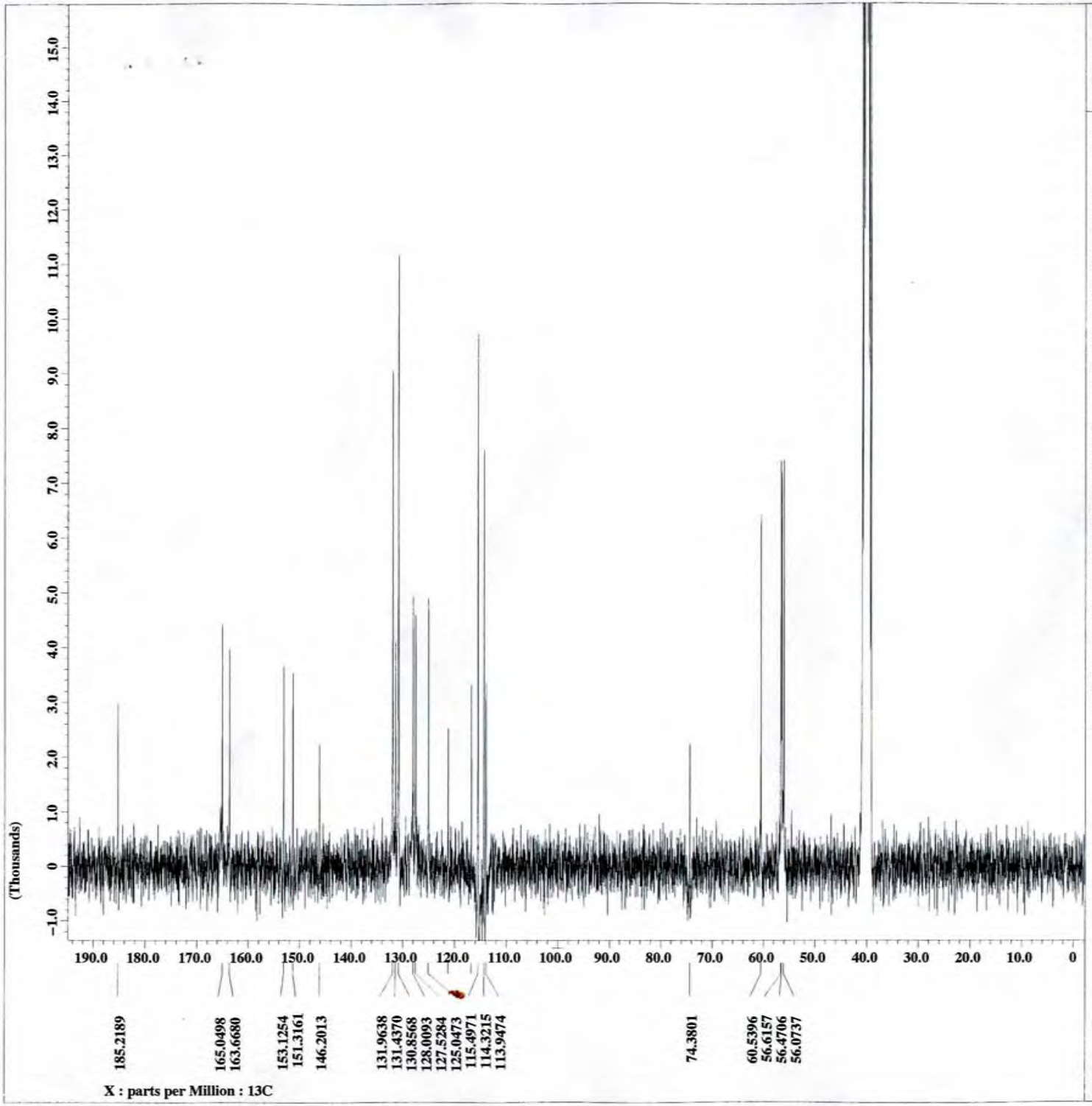
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 22.7[dC]



A_4
300 MHz, DMSO-d_6



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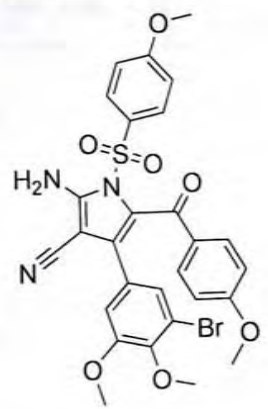
Author = alex
Experiment = single_pulse_dec
Sample_id = S#540132
Solvent = DMSO-D6
Creation_time = 10-DEC-2010 15:47:35
Revision_time = 10-DEC-2010 19:32:00
Current_time = 10-DEC-2010 19:32:09

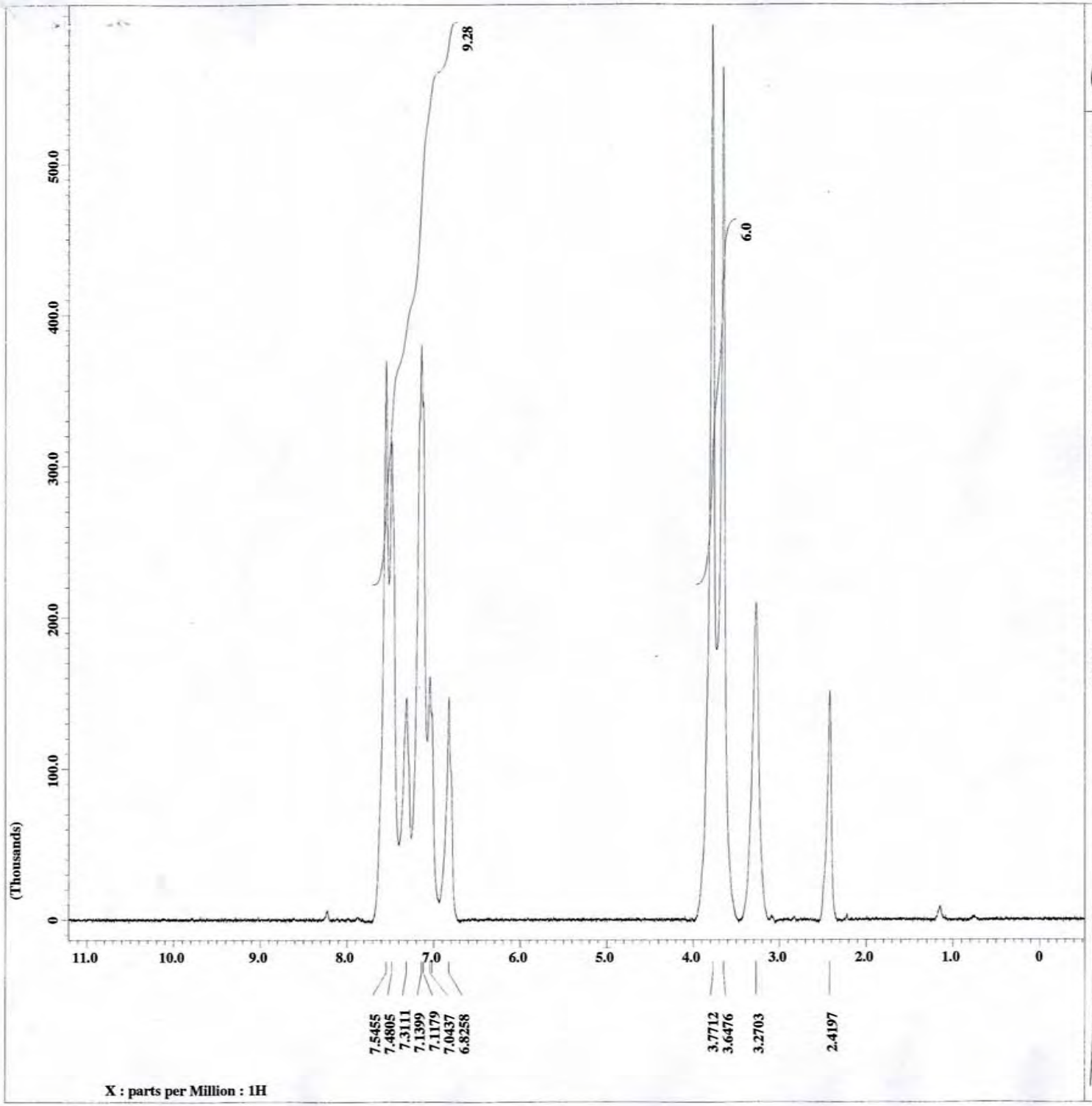
Comment = Single Pulse with Bro
Data_format = 1D REAL
Dim_size = 32768
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 300
Spectrometer = DELTA_NMR

Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.7334272[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.57689184[Hz]
X_sweep = 18.90359168[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 1200
Total_scans = 1200

X_90_width = 8.1[us]
X_acq_time = 1.7334272[s]
X_angle = 30[deg]
X_pulse = 2.7[us]
Initial_wait = 1[s]
Phase_preset = 3[us]

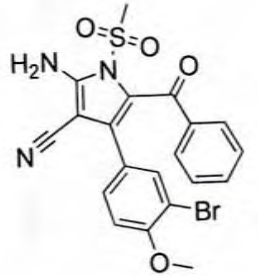


A_4
75 MHz, DMSO-d_6

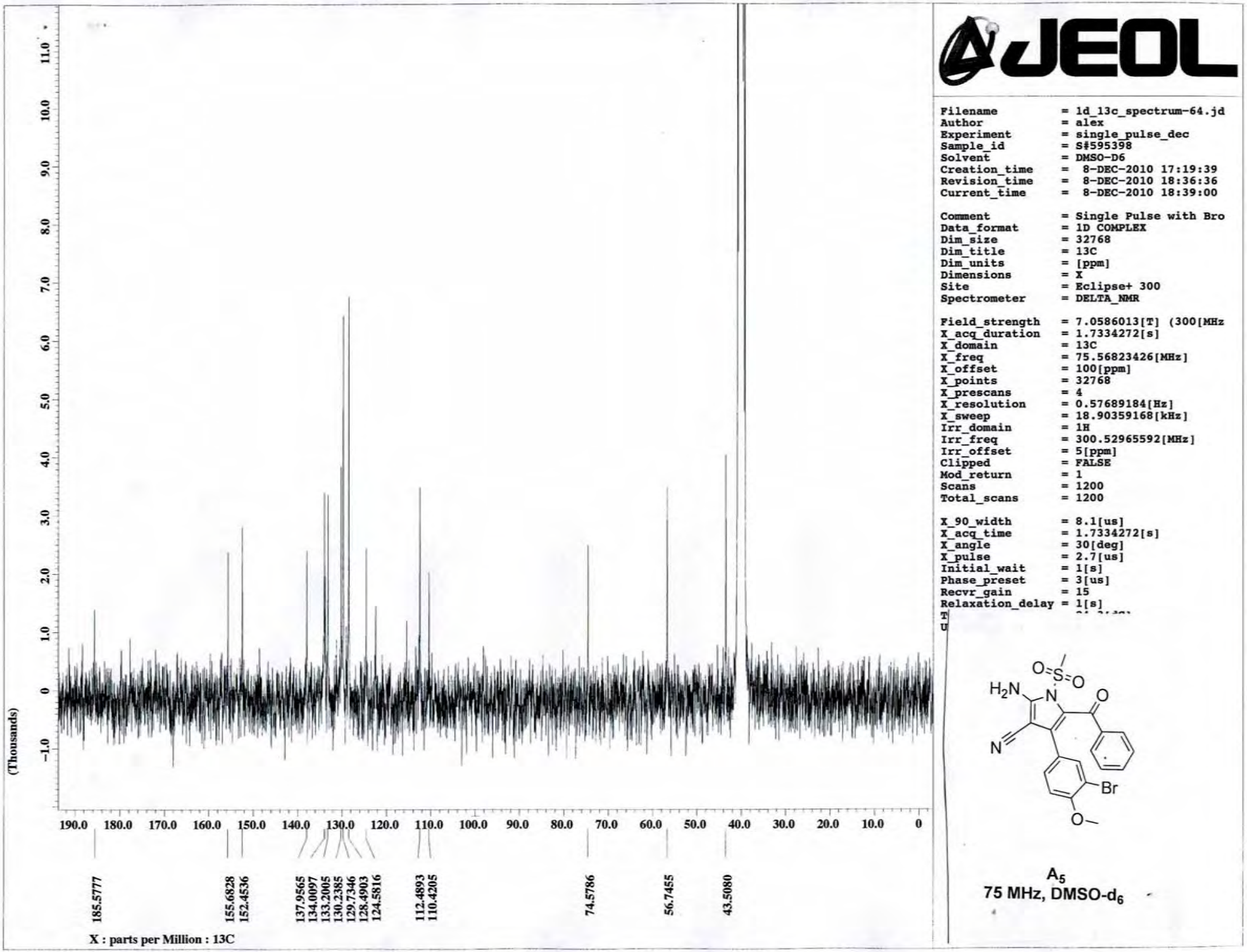


JEOL

Filename	= 1d_spectrum-104.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#594298
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 16:23:58
Revision_time	= 8-DEC-2010 18:41:52
Current_time	= 8-DEC-2010 18:42:04
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15



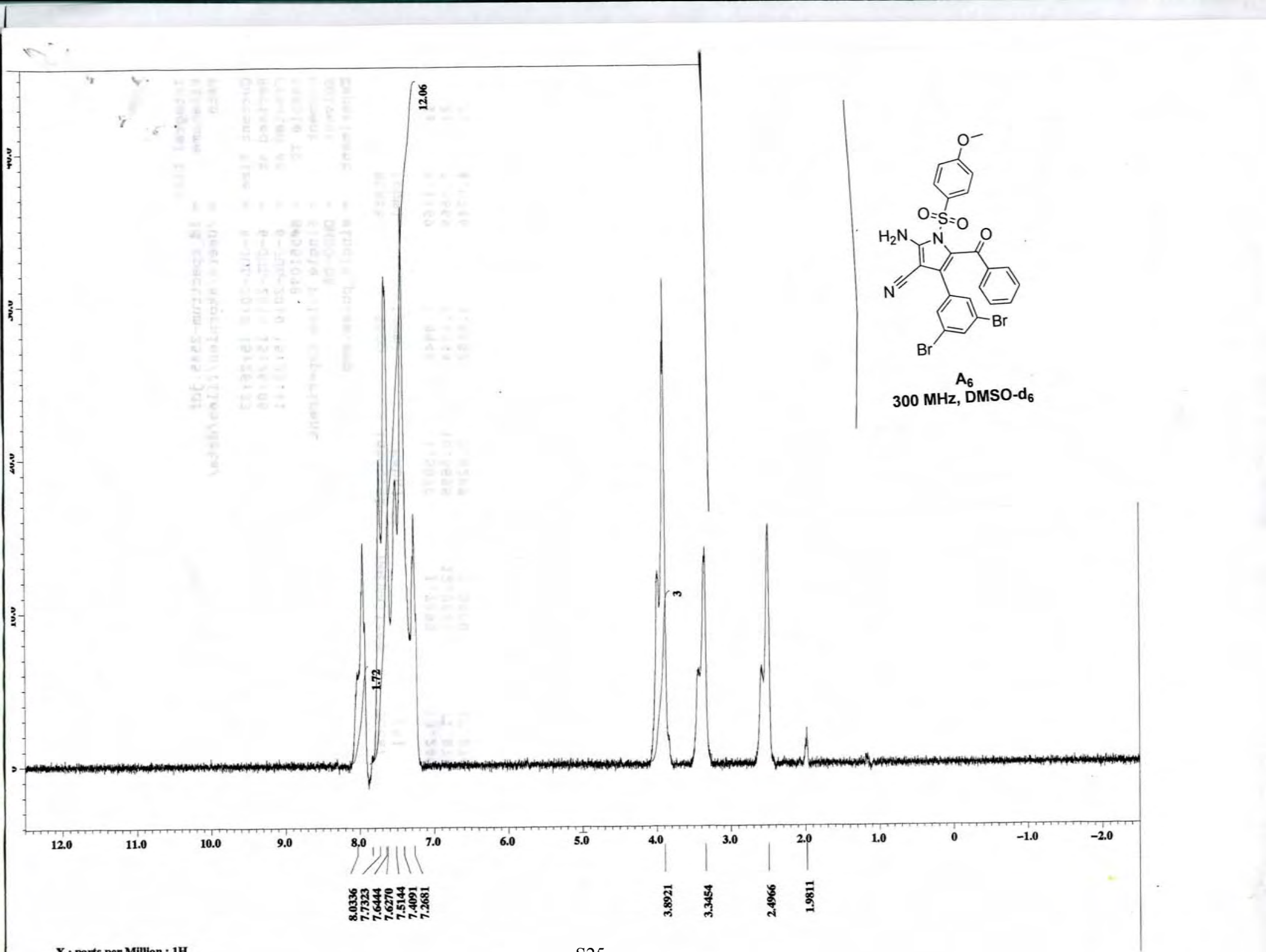
A_5
300 MHz, DMSO-d_6

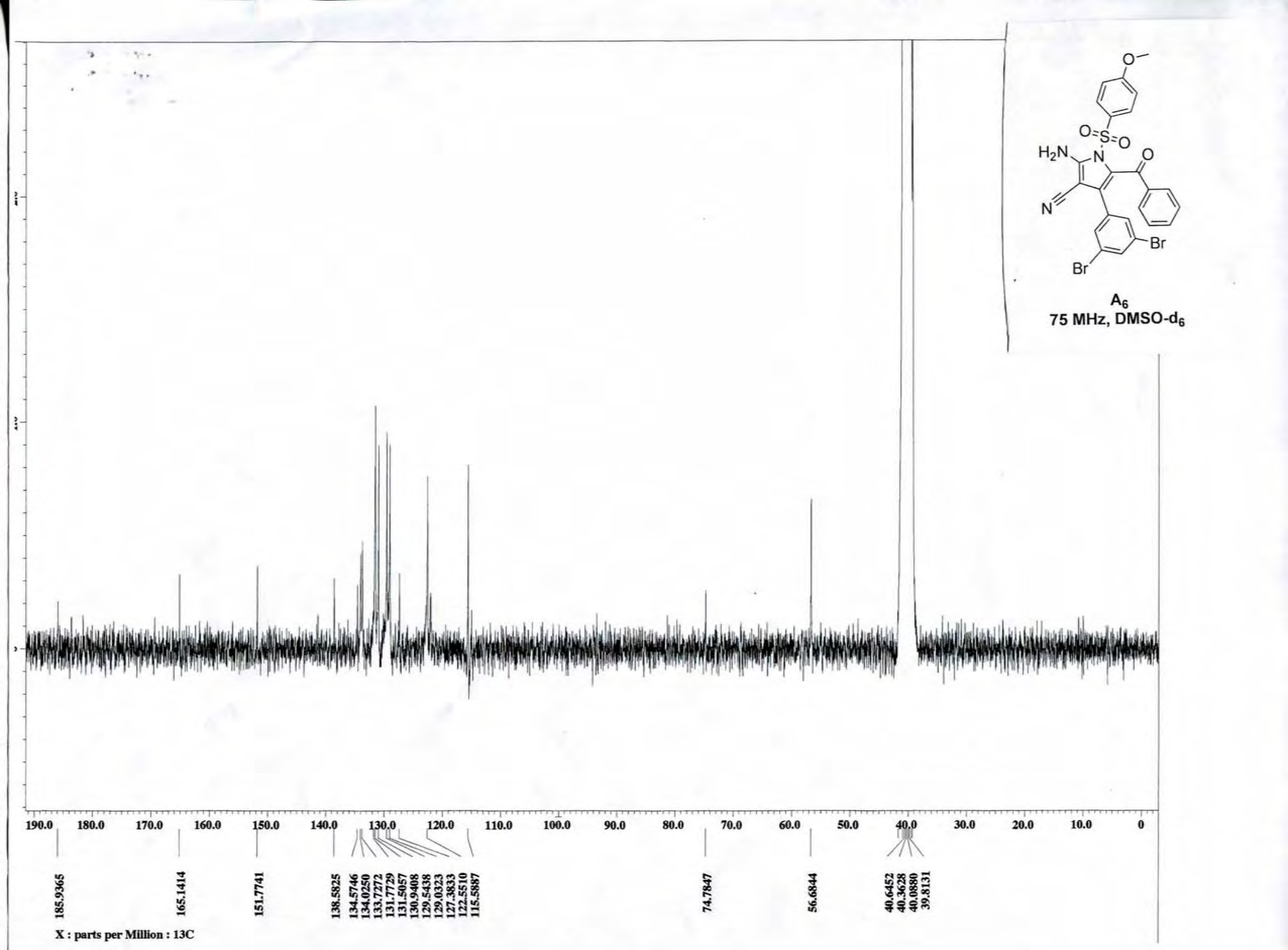


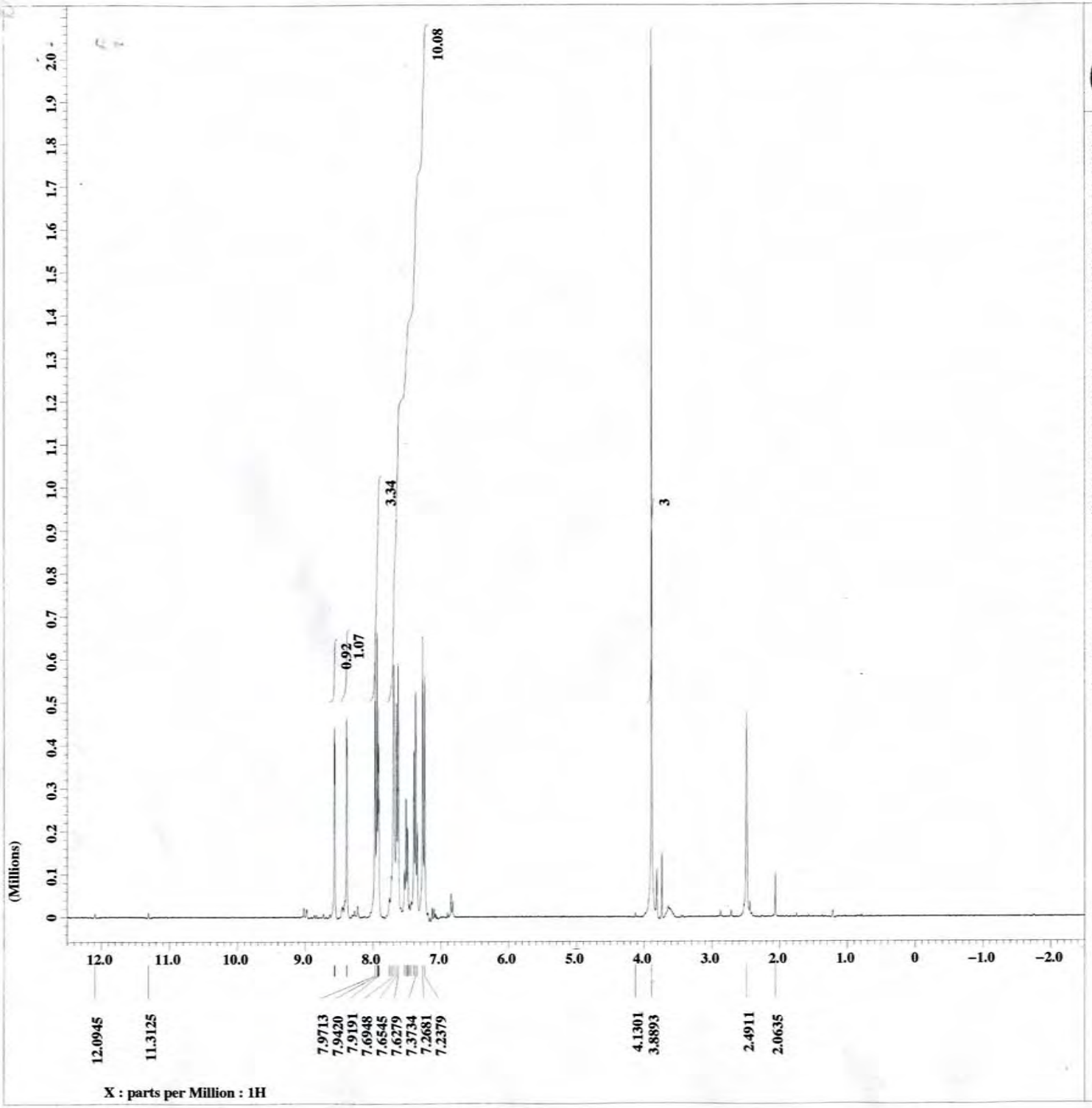
JEOL

Filename	= 1d_13c_spectrum-64.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#595398
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 17:19:39
Revision_time	= 8-DEC-2010 18:36:36
Current_time	= 8-DEC-2010 18:39:00
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]

A_5
75 MHz, DMSO-d_6

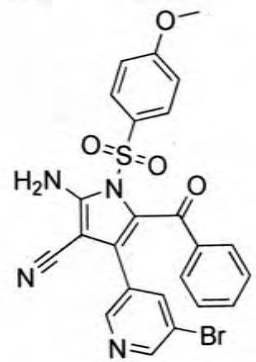

12.06
1.72
3
8.0336
7.7323
7.6444
7.6270
7.5144
7.4091
7.2681
3.8921
3.3454
2.4966
1.9811
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
−2.0
X : parts per Million : 1H
O
O=S=O
H2N
N
O
N
Br
Br
A6
300 MHz, DMSO-d6


A6
75 MHz, DMSO-d6
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
185.9365
165.1414
151.7741
138.5825
134.5746
134.0250
133.7272
131.7729
131.5057
130.9408
129.5438
129.0323
127.3833
122.5510
115.5887
74.7847
56.6844
40.6452
40.3628
40.0880
39.8131
X : parts per Million : 13C

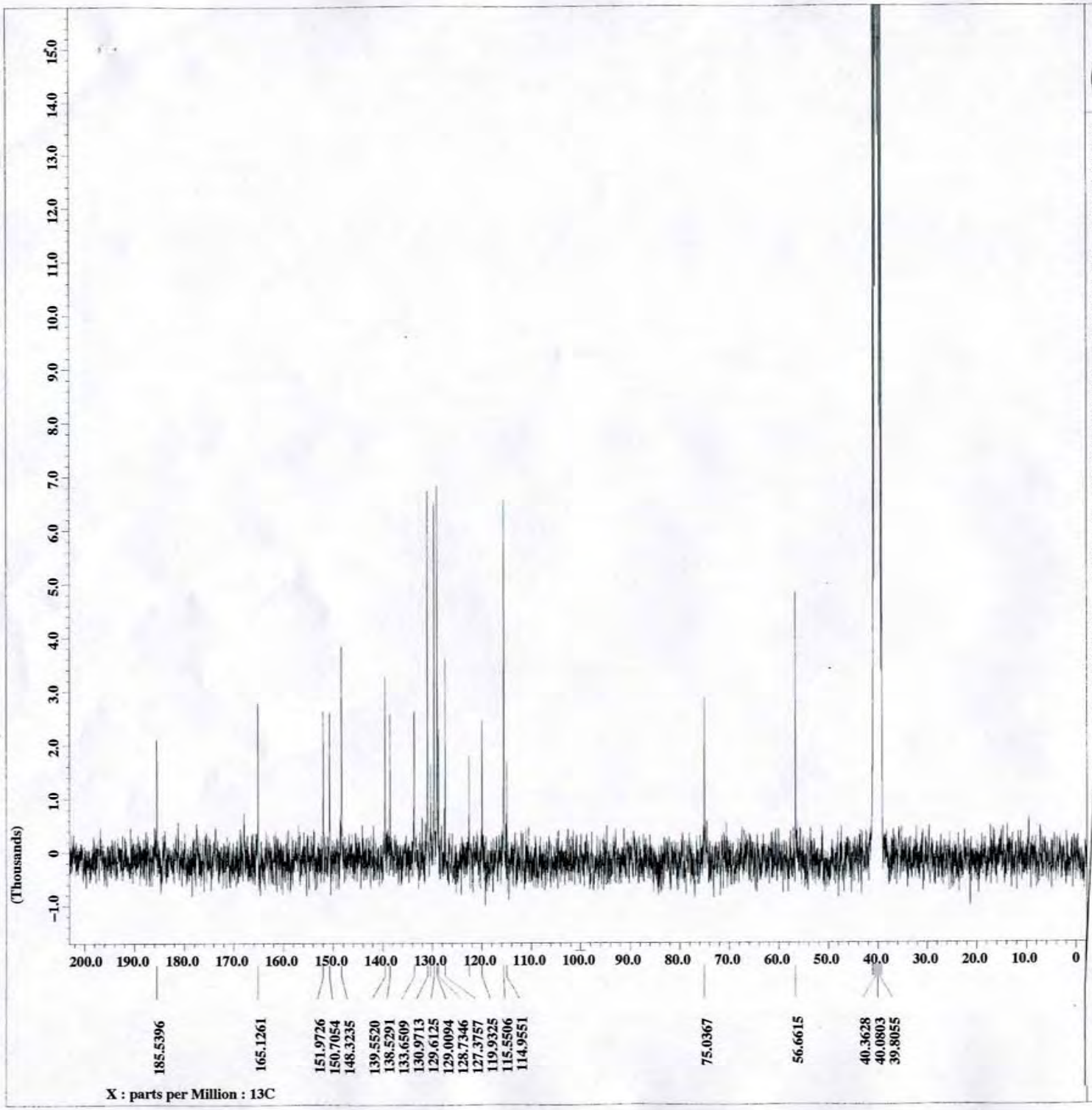


JEOL

Filename	= 1d_spectrum-148.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#500377
Solvent	= DMSO-D6
Creation_time	= 11-DEC-2010 13:47:28
Revision_time	= 11-DEC-2010 14:06:20
Current_time	= 11-DEC-2010 14:06:27
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 22.7[dC]

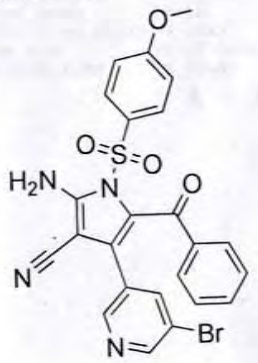


A_7
300 MHz, DMSO-d_6

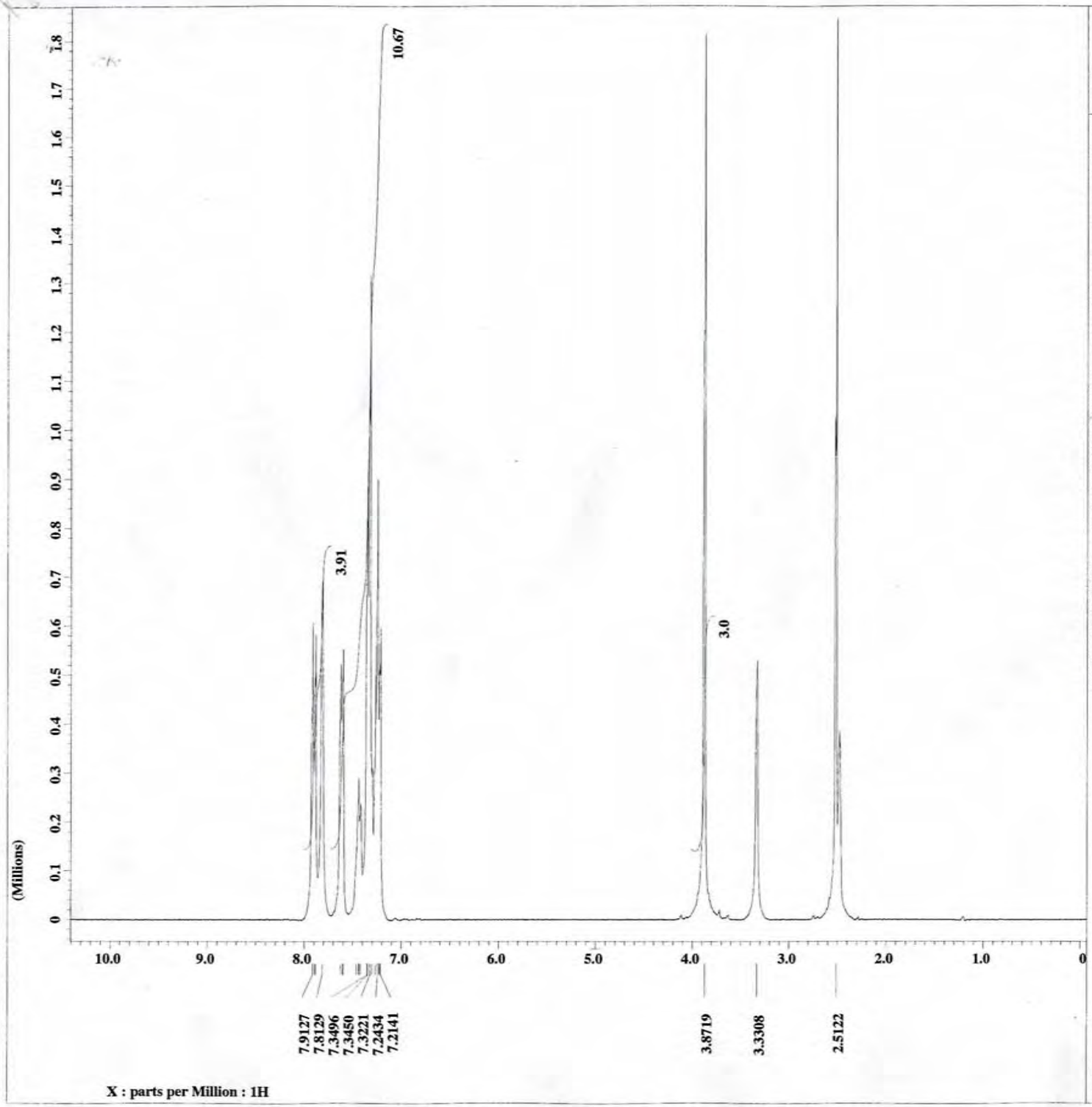


JEOL

Filename	= 1d_13c_spectrum-88.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#535311
Solvent	= DMSO-D6
Creation_time	= 11-DEC-2010 16:16:00
Revision_time	= 11-DEC-2010 17:19:02
Current_time	= 11-DEC-2010 17:19:14
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 2000
Total_scans	= 2000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]

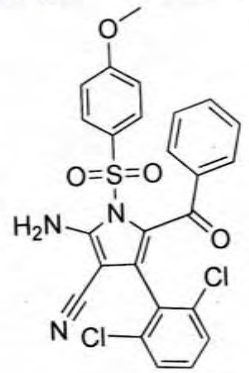


A_7
75 MHz, DMSO-d_6

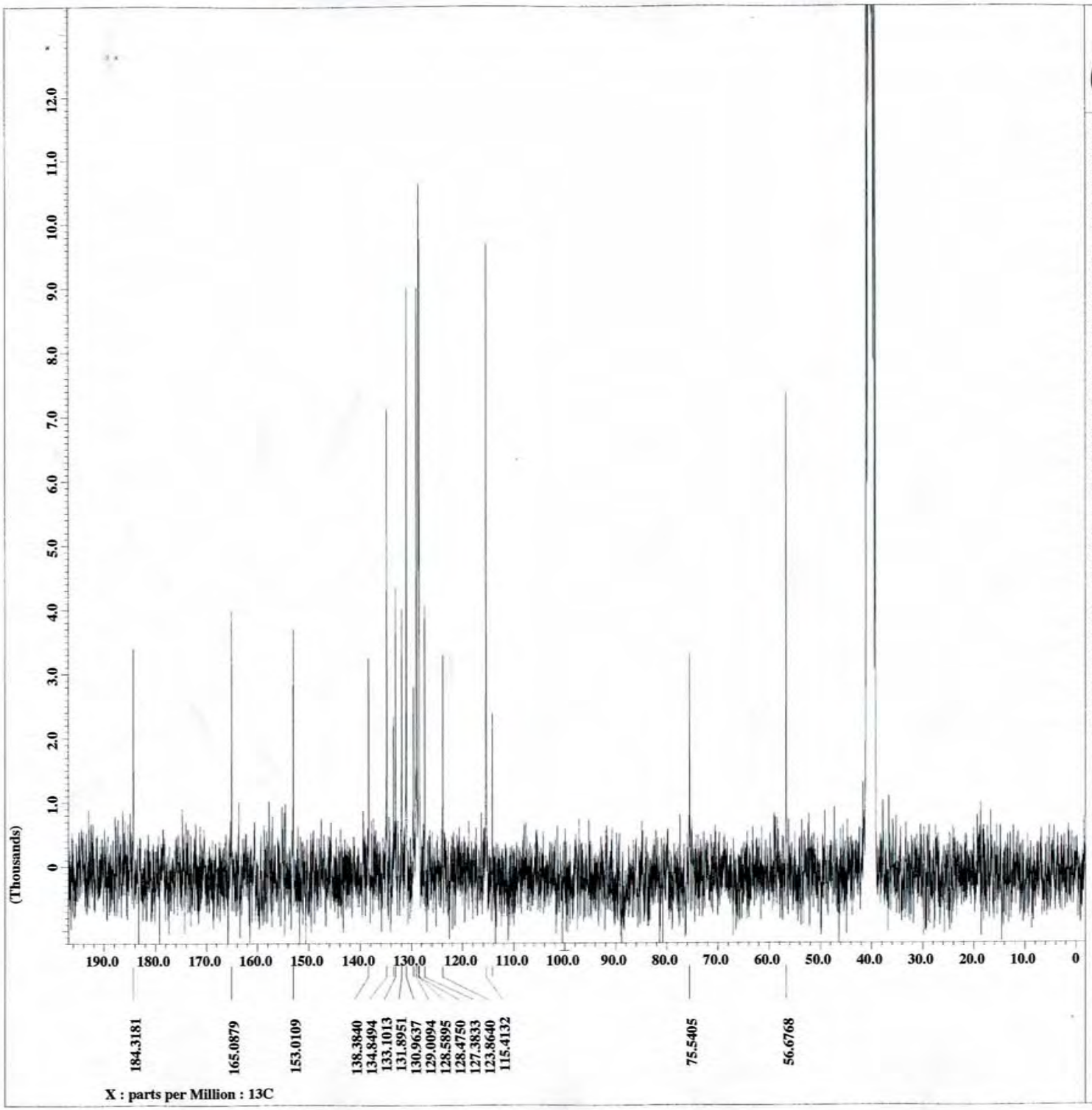


JEOL

Filename	= 1d_spectrum-134.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#678091
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 18:43:42
Revision_time	= 9-DEC-2010 19:32:05
Current_time	= 9-DEC-2010 19:32:23
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 23.2[dC]

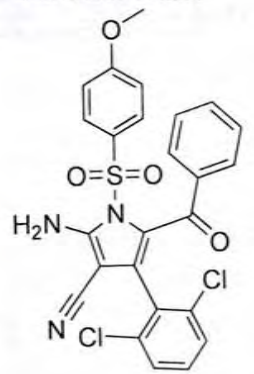


A_8
300 MHz, DMSO-d_6

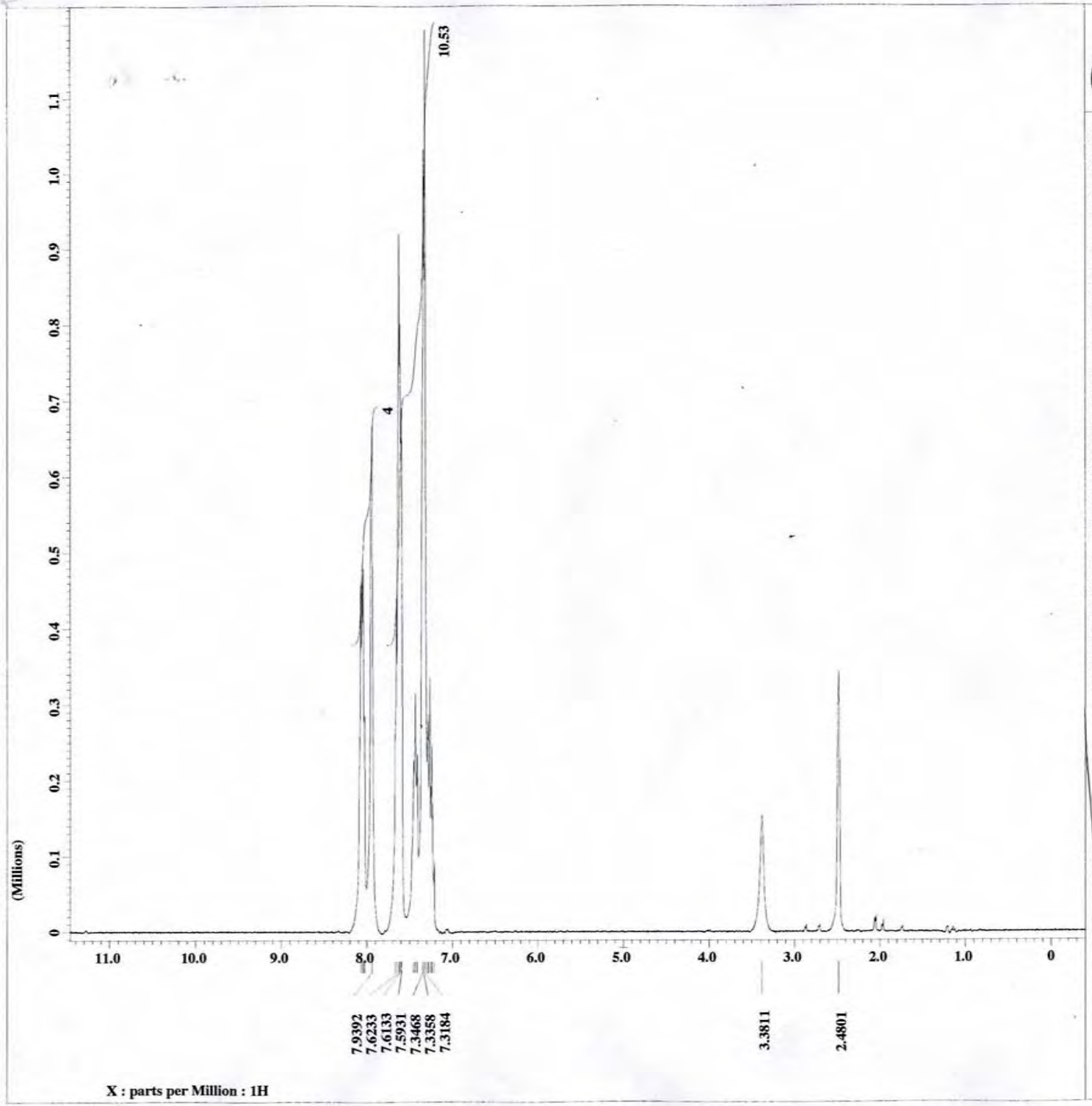


JEOL

Filename	= 1d_13c_spectrum-73.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#679231
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 19:30:20
Revision_time	= 9-DEC-2010 19:43:29
Current_time	= 9-DEC-2010 19:43:34
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1000
Total_scans	= 1000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation delay	= 1[s]

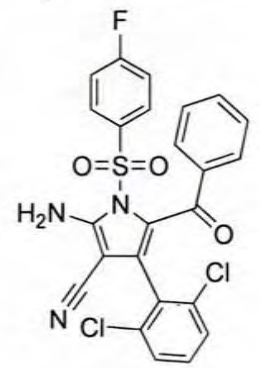


A_8
75 MHz, DMSO-d_6

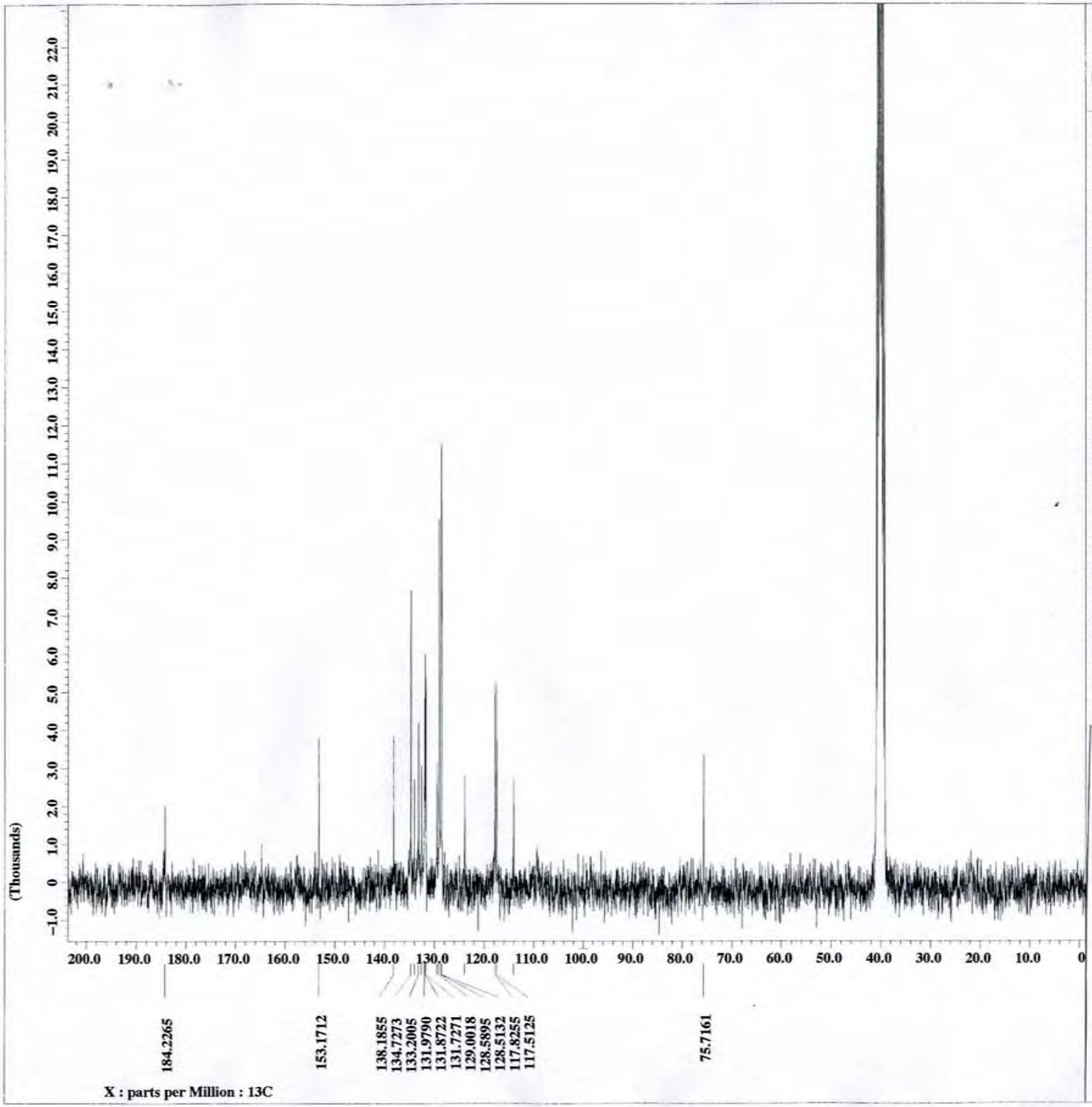


JEOL

```
Filename          = 1d_spectrum-123.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#454507
Solvent           = DMSO-D6
Creation_time     =  9-DEC-2010 12:31:02
Revision_time     =  9-DEC-2010 14:49:46
Current_time      =  9-DEC-2010 14:49:57

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 22.6[dC]
Unblank_time      = 2[us]
```

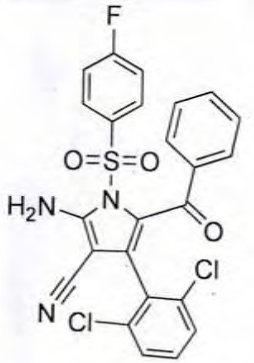


A_9
300 MHz, DMSO-d_6

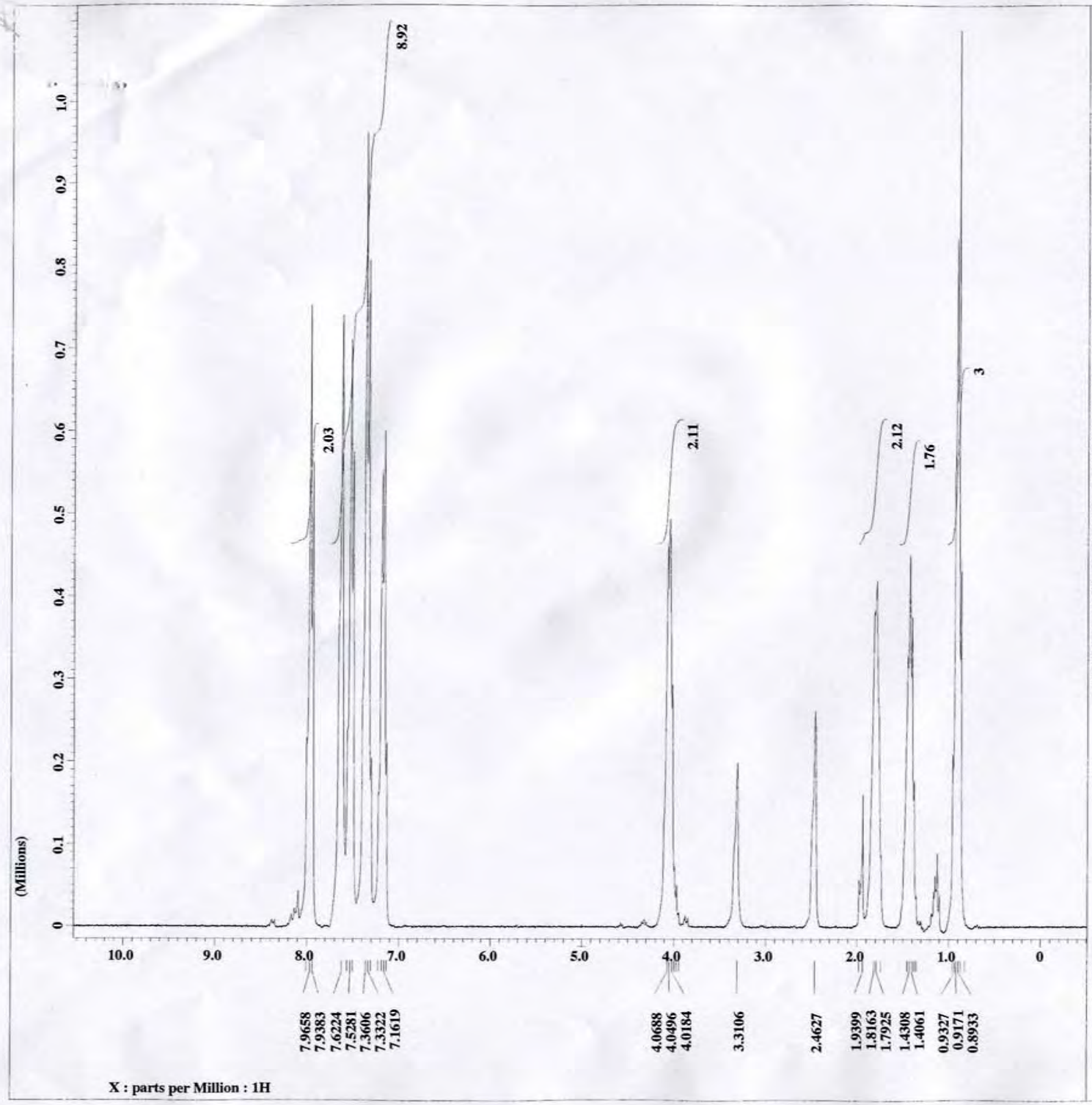


JEOL

Filename	= 1d_13c_spectrum-70.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#455639
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 13:26:47
Revision_time	= 9-DEC-2010 14:40:14
Current_time	= 9-DEC-2010 14:40:33
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 23.8[dC]

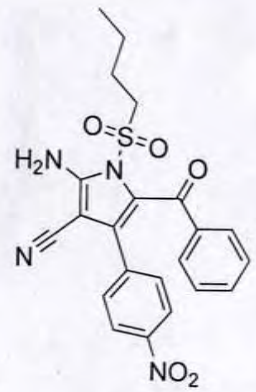


A_9
75 MHz, DMSO-d_6

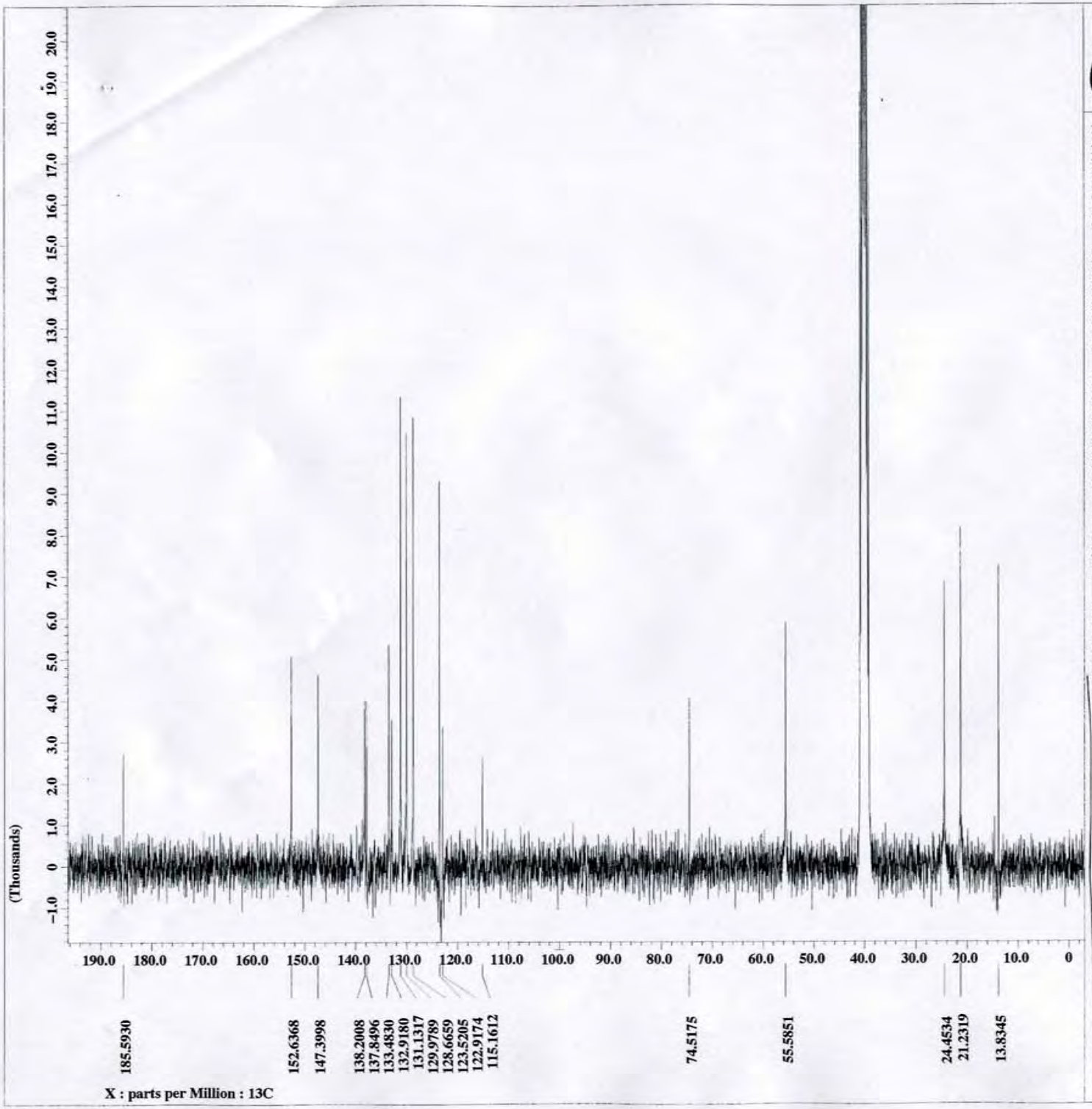


JEOL

Filename	= 1d_spectrum-118.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#414169
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 11:23:49
Revision_time	= 9-DEC-2010 12:35:01
Current_time	= 9-DEC-2010 12:35:15
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 22.1[dC]
Unblank_time	= 2[us]

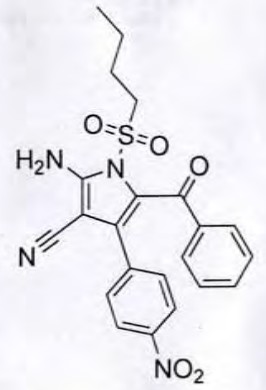


A_{10}
300 MHz, DMSO-d_6

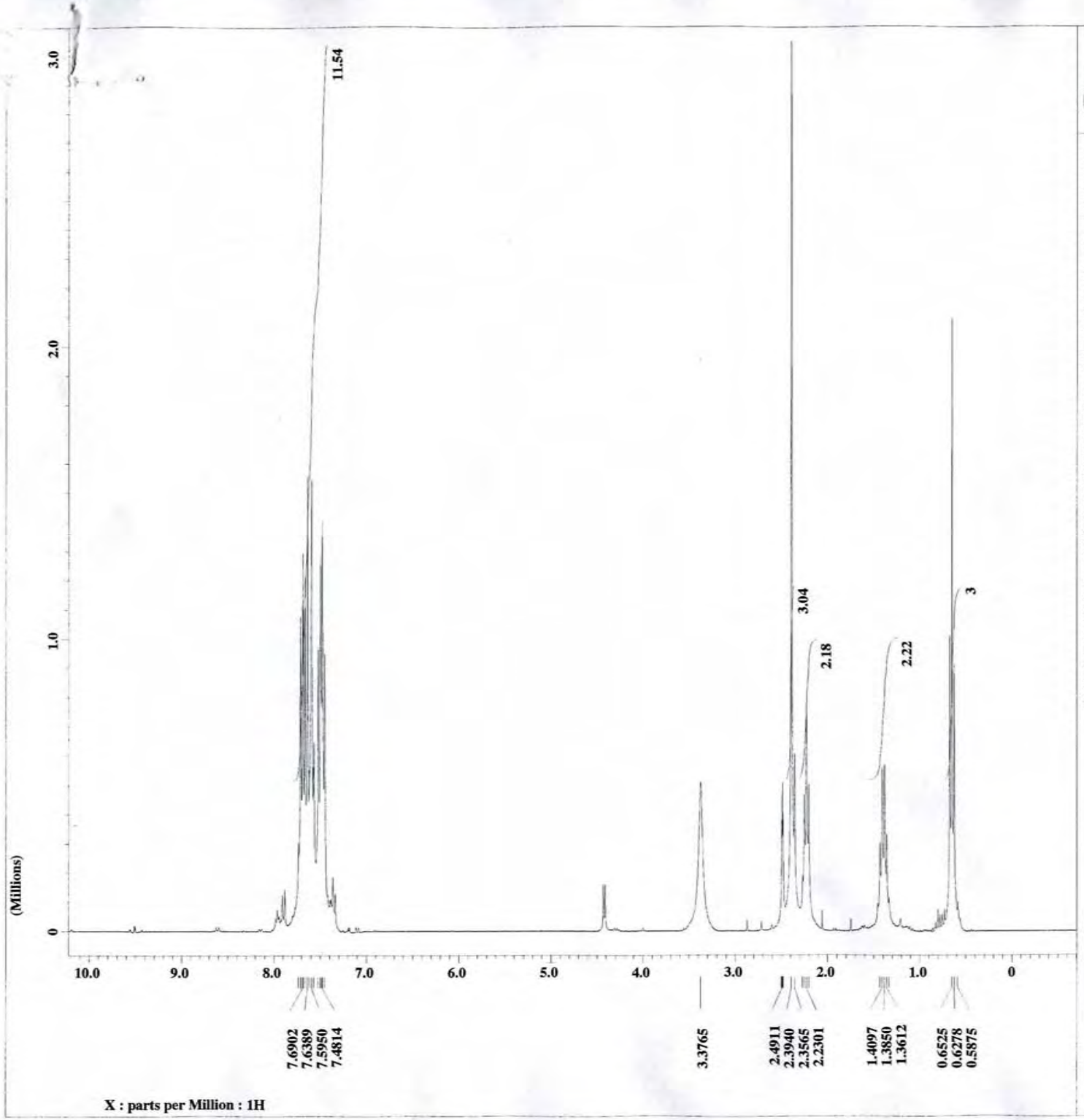


JEOL

Filename	= 1d_13c_spectrum-69.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#416841
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 12:12:58
Revision_time	= 9-DEC-2010 12:27:31
Current_time	= 9-DEC-2010 12:27:35
Comment	= Single Pulse with Bro
Data_format	= 1D REAL
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1000
Total_scans	= 1000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]

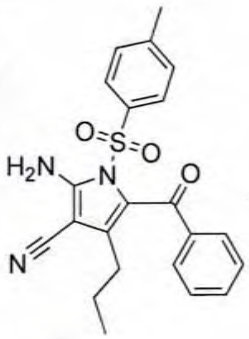


A_{10}
75 MHz, DMSO-d_6

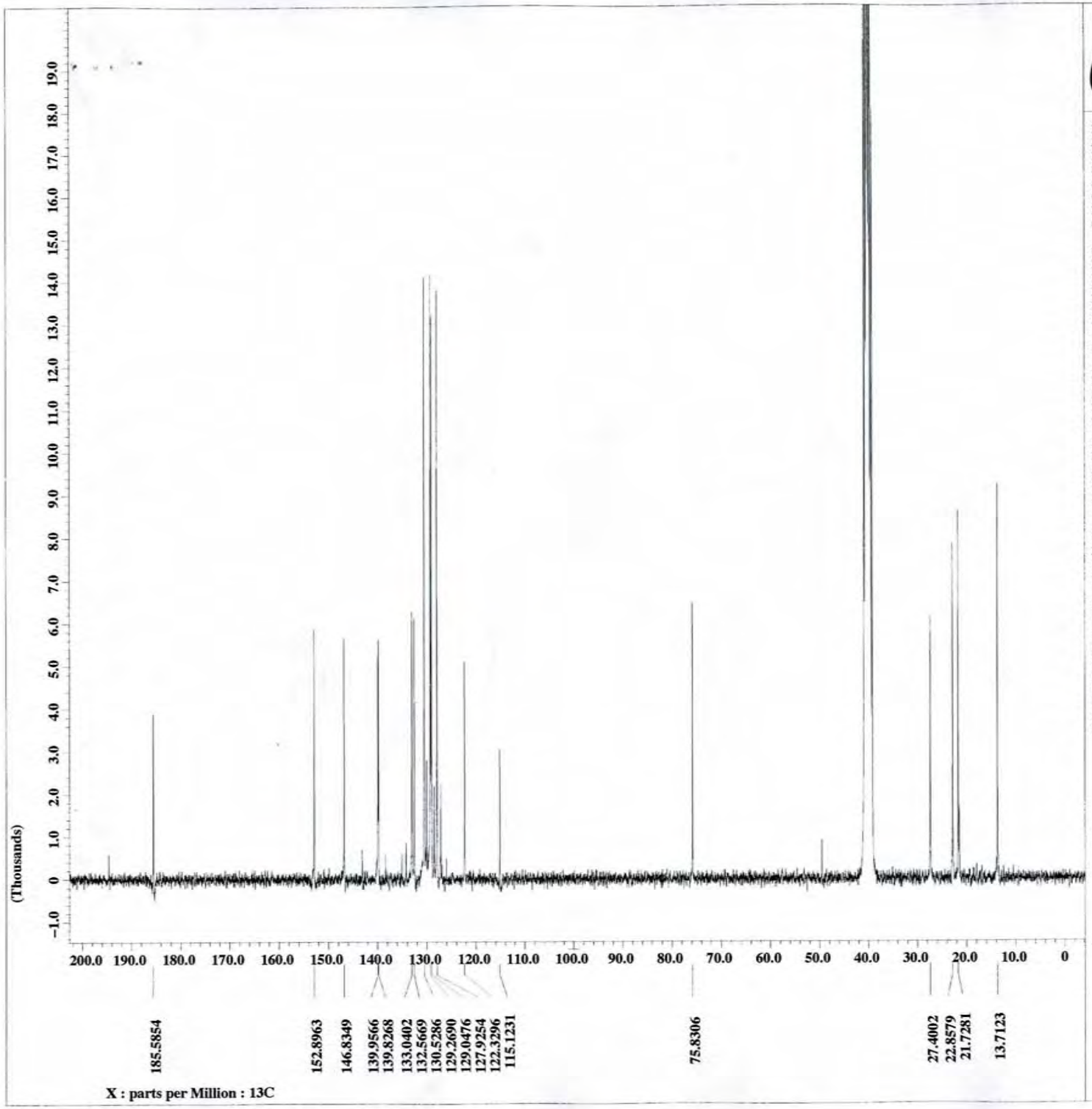


JEOL

```
Filename          = 1d_spectrum-201.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#671616
Solvent           = DMSO-D6
Creation_time     = 17-DEC-2010 18:32:53
Revision_time     = 18-DEC-2010 17:16:36
Current_time      = 18-DEC-2010 17:16:53

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23[dC]
Unblank_time      = 2[us]
```



A_{11}
300 MHz, DMSO-d_6

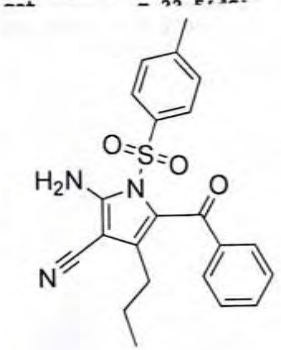


Filename = 1d_13c_spectrum-109.j
Author = alex
Experiment = single_pulse_dec
Sample_id = S#675148
Solvent = DMSO-D6
Creation_time = 18-DEC-2010 06:46:52
Revision_time = 18-DEC-2010 17:10:36
Current_time = 18-DEC-2010 17:11:38

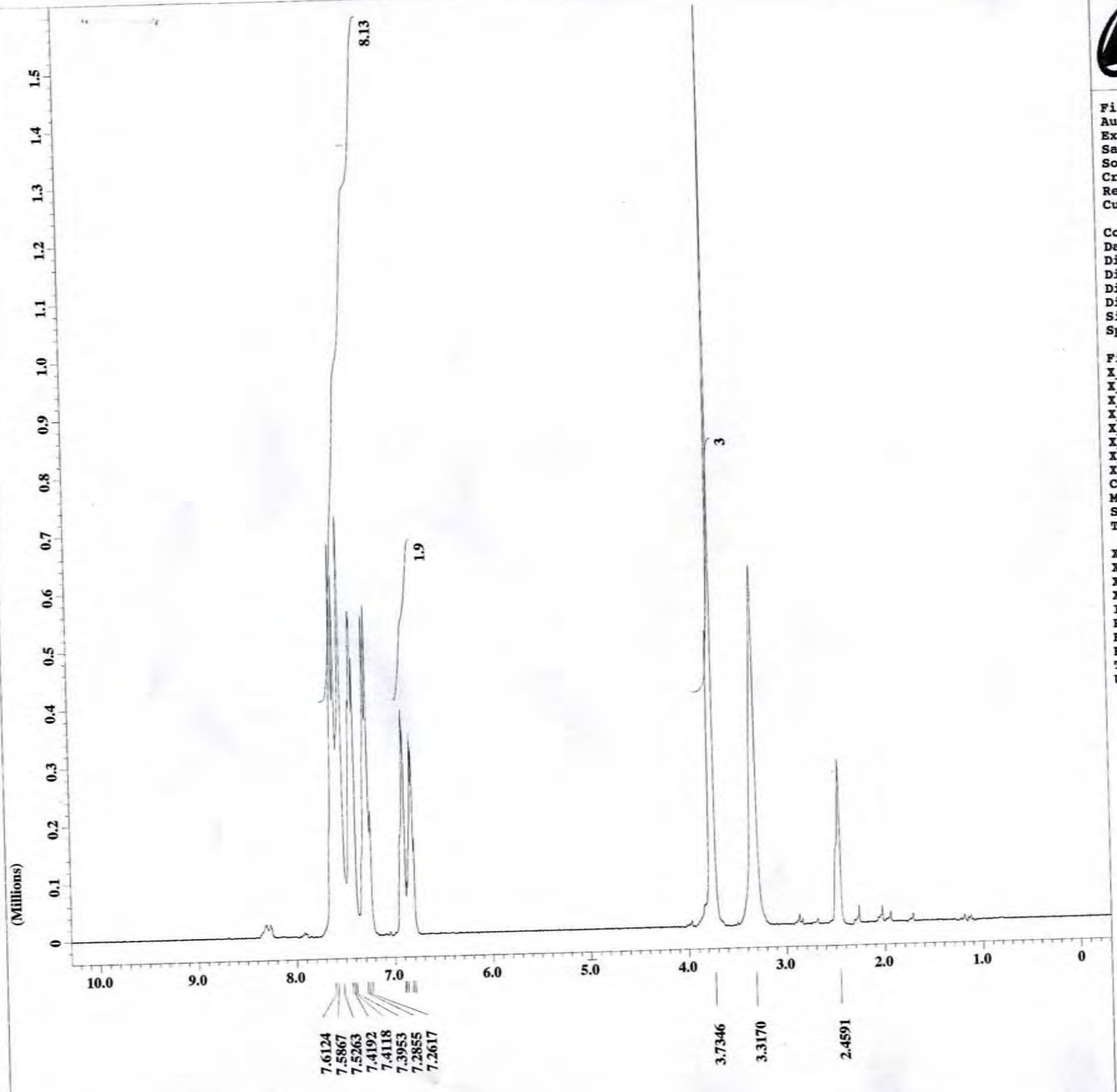
Comment = Single Pulse with Bro
Data_format = 1D REAL
Dim_size = 32768
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 300
Spectrometer = DELTA_NMR

Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.7334272[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.57689184[Hz]
X_sweep = 18.90359168[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 16000
Total_scans = 16000

X_90_width = 8.1[us]
X_acq_time = 1.7334272[s]
X_angle = 30[deg]
X_pulse = 2.7[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 15
Relaxation_delay = 1[s]

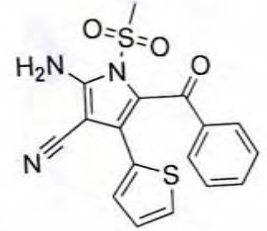


A_{11}
75 MHz, DMSO-d_6

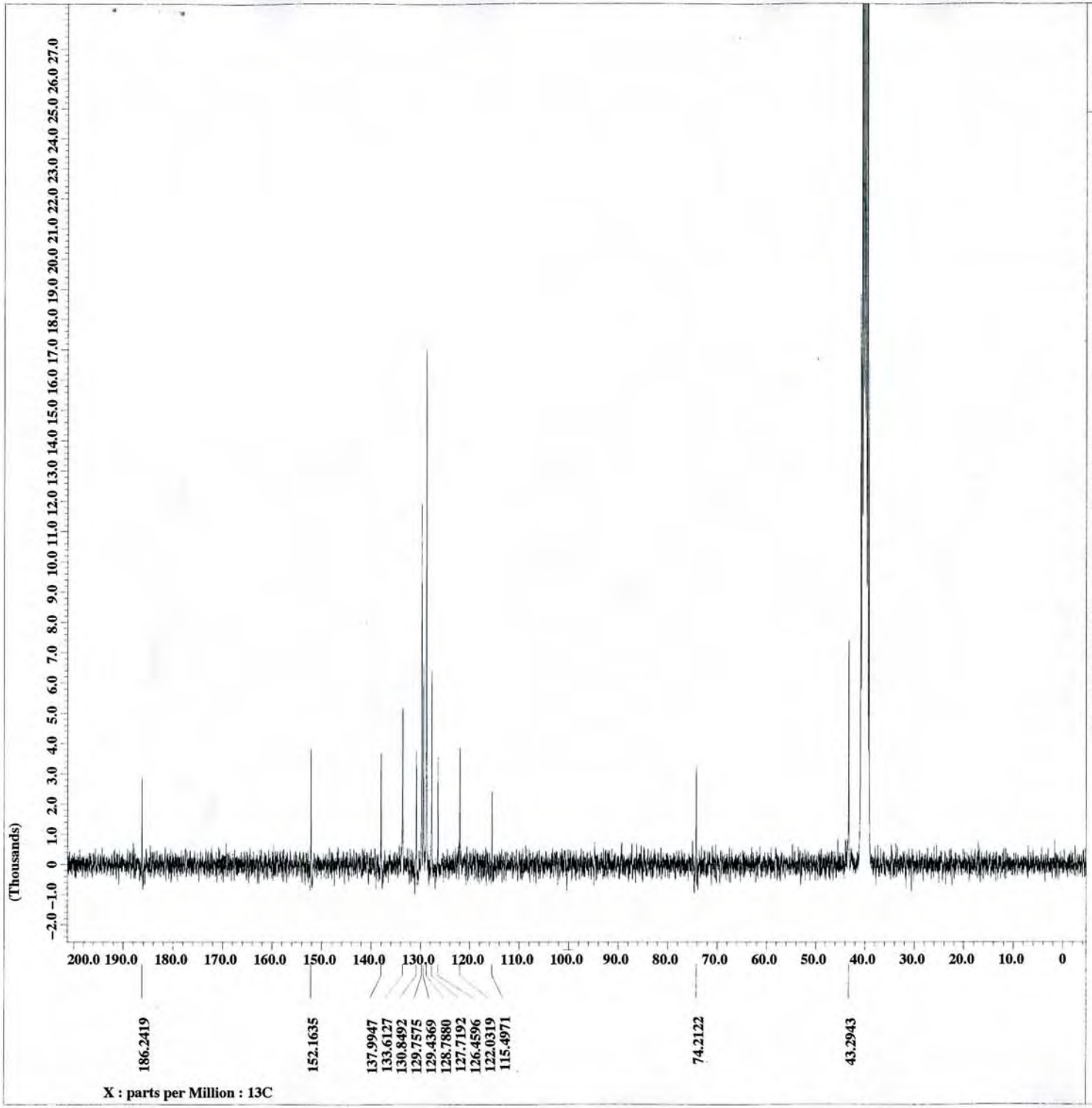


JEOL

Filename	= 1d_spectrum-145.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#345560
Solvent	= DMSO-D6
Creation_time	= 10-DEC-2010 09:29:32
Revision_time	= 10-DEC-2010 19:46:17
Current_time	= 10-DEC-2010 19:46:25
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 21.2[dC]
Unblank_time	= 2[us]



A_{12}
300 MHz, DMSO-d_6

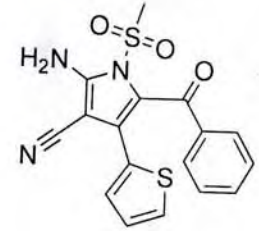


Filename = 1d_13c_spectrum-81.jd
Author = alex
Experiment = single_pulse_dec
Sample_id = S#347199
Solvent = DMSO-D6
Creation_time = 10-DEC-2010 11:02:29
Revision_time = 10-DEC-2010 19:29:32
Current_time = 10-DEC-2010 19:29:43

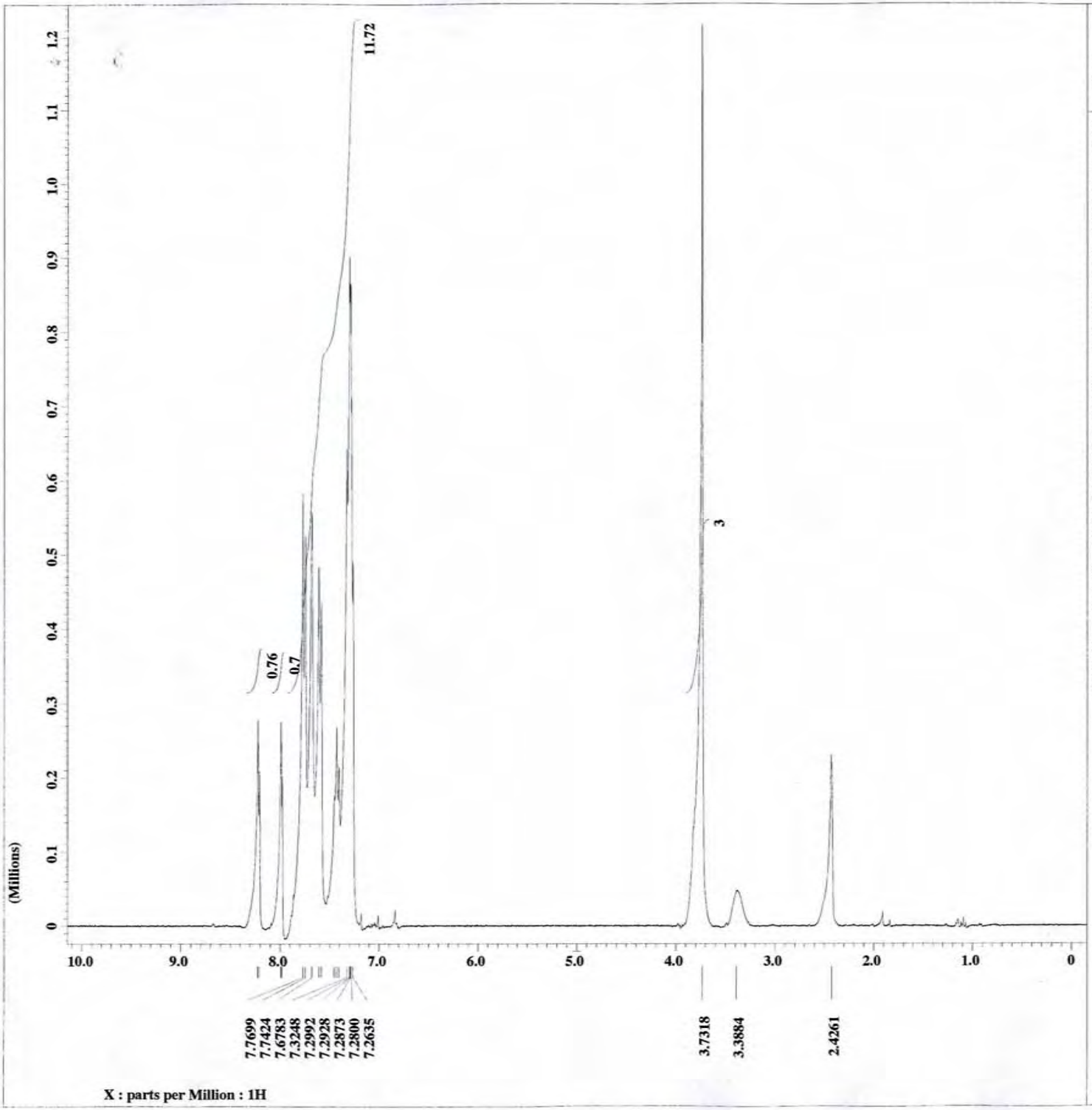
Comment = Single Pulse with Bro
Data_format = 1D REAL
Dim_size = 32768
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 300
Spectrometer = DELTA_NMR

Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 1.7334272[s]
X_domain = 13C
X_freq = 75.56823426[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.57689184[Hz]
X_sweep = 18.90359168[kHz]
Irr_domain = 1H
Irr_freq = 300.52965592[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 2000
Total_scans = 2000

X_90_width = 8.1[us]
X_acq_time = 1.7334272[s]
X_angle = 30[deg]
X_pulse = 2.7[us]
Initial_wait = 1[s]
Phase_preset = 3[us]
Recvr_gain = 15
Relaxation_delay = 1[s]
Temp_get = 23.1[dC]
Unblank_time = 2[us]

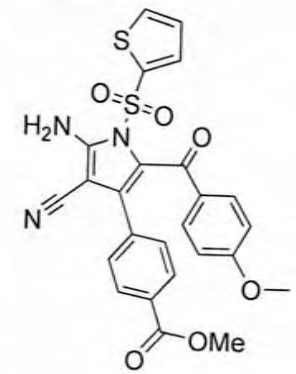


A_{12}
75 MHz, DMSO-d_6

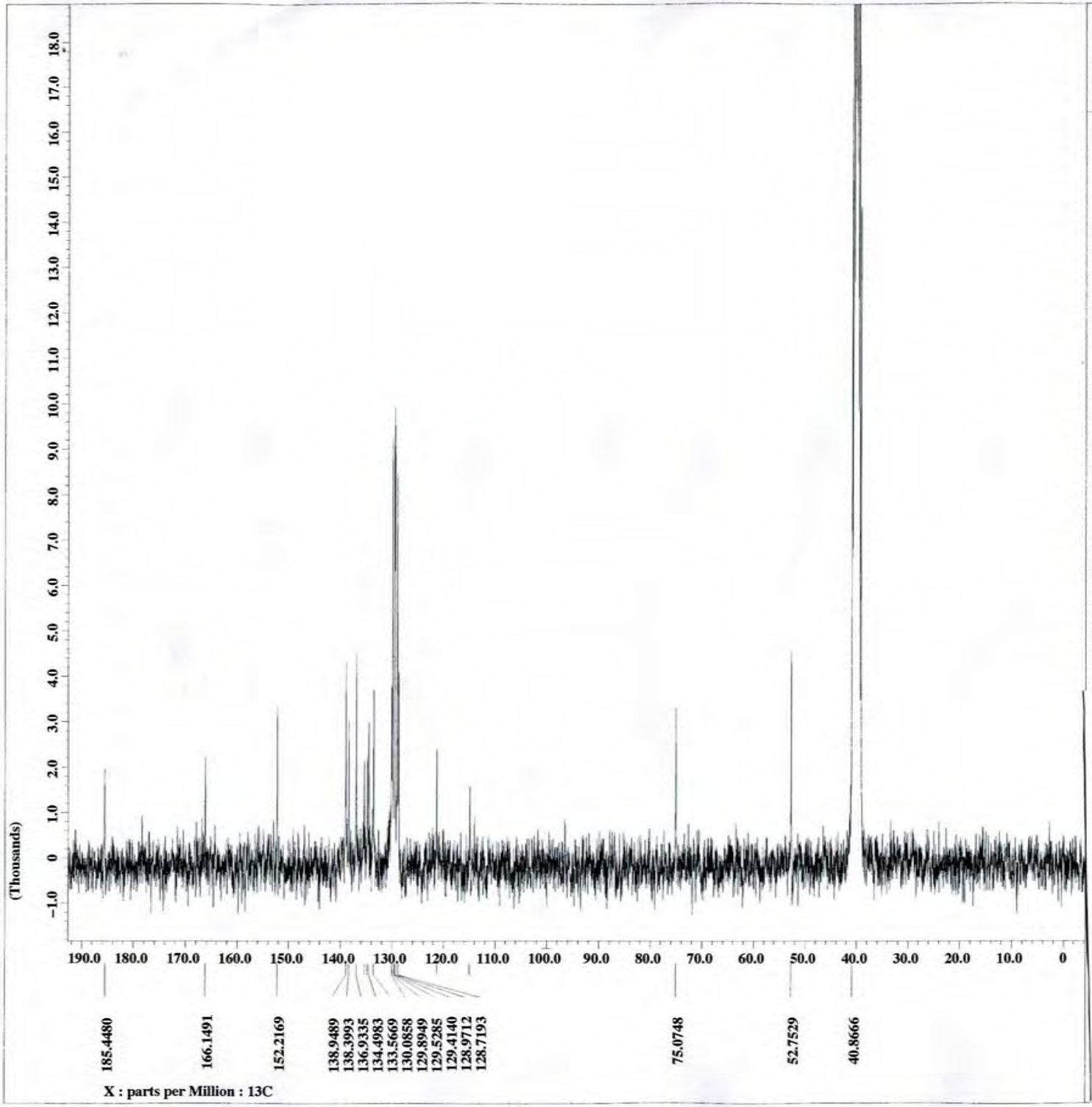


JEOL

```
Filename          = 1d_spectrum-87.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#389823
Solvent           = DMSO-D6
Creation_time     =  7-DEC-2010 10:43:46
Revision_time     =  7-DEC-2010 12:12:35
Current_time      =  7-DEC-2010 12:12:45

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23.3[dC]
Unbl
```

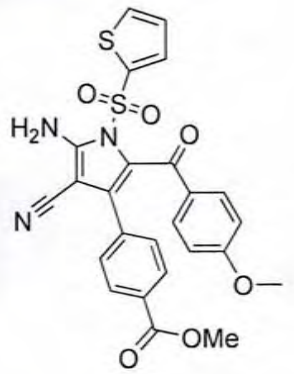


A_{13}
300 MHz, DMSO-d_6

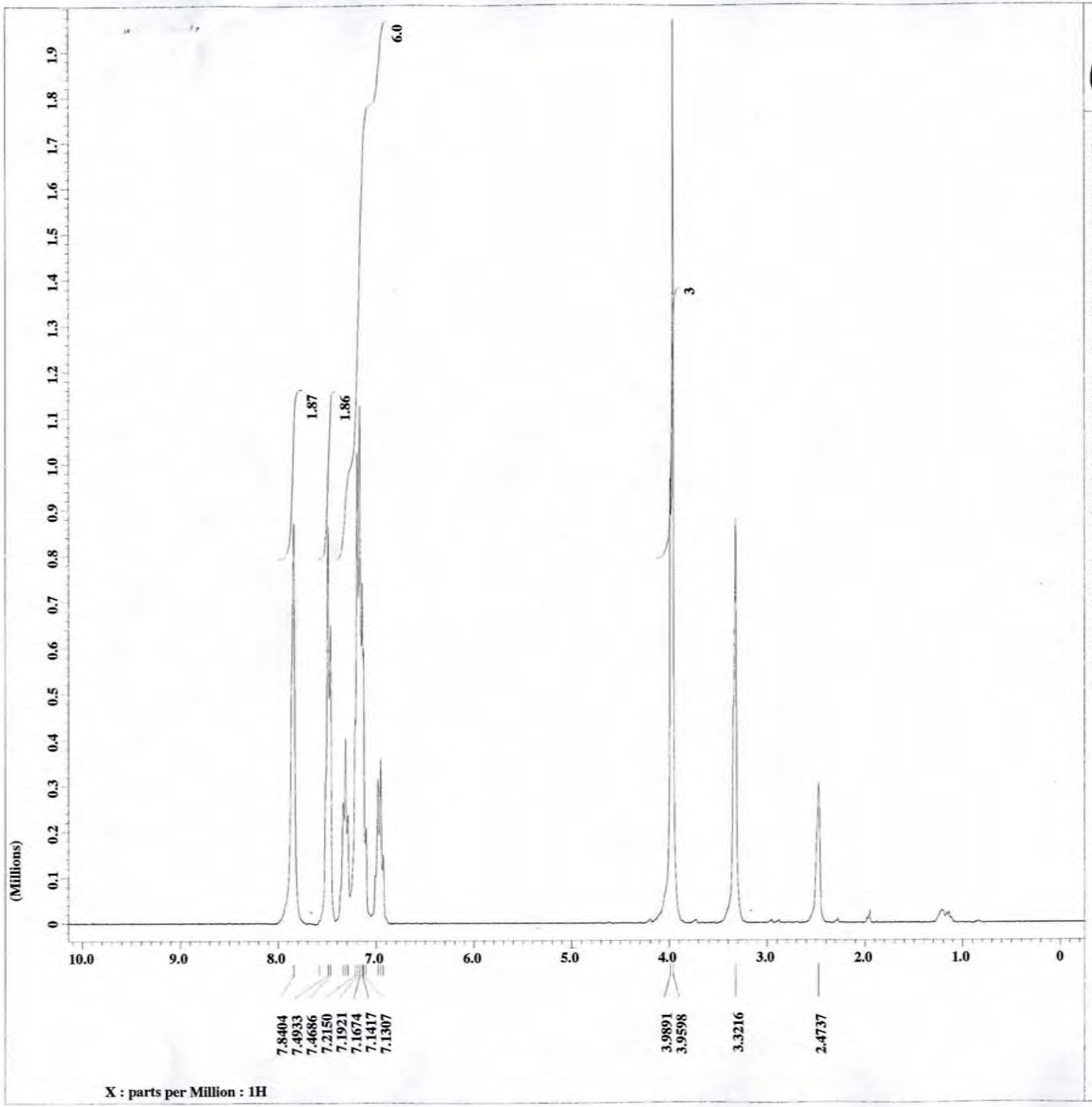


JEOL

Filename	= 1d_13c_spectrum-53.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#391453
Solvent	= DMSO-D6
Creation_time	= 7-DEC-2010 11:39:48
Revision_time	= 7-DEC-2010 12:07:28
Current_time	= 7-DEC-2010 12:07:55
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15

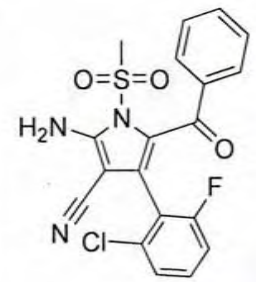


A_{13}
75 MHz, DMSO-d_6

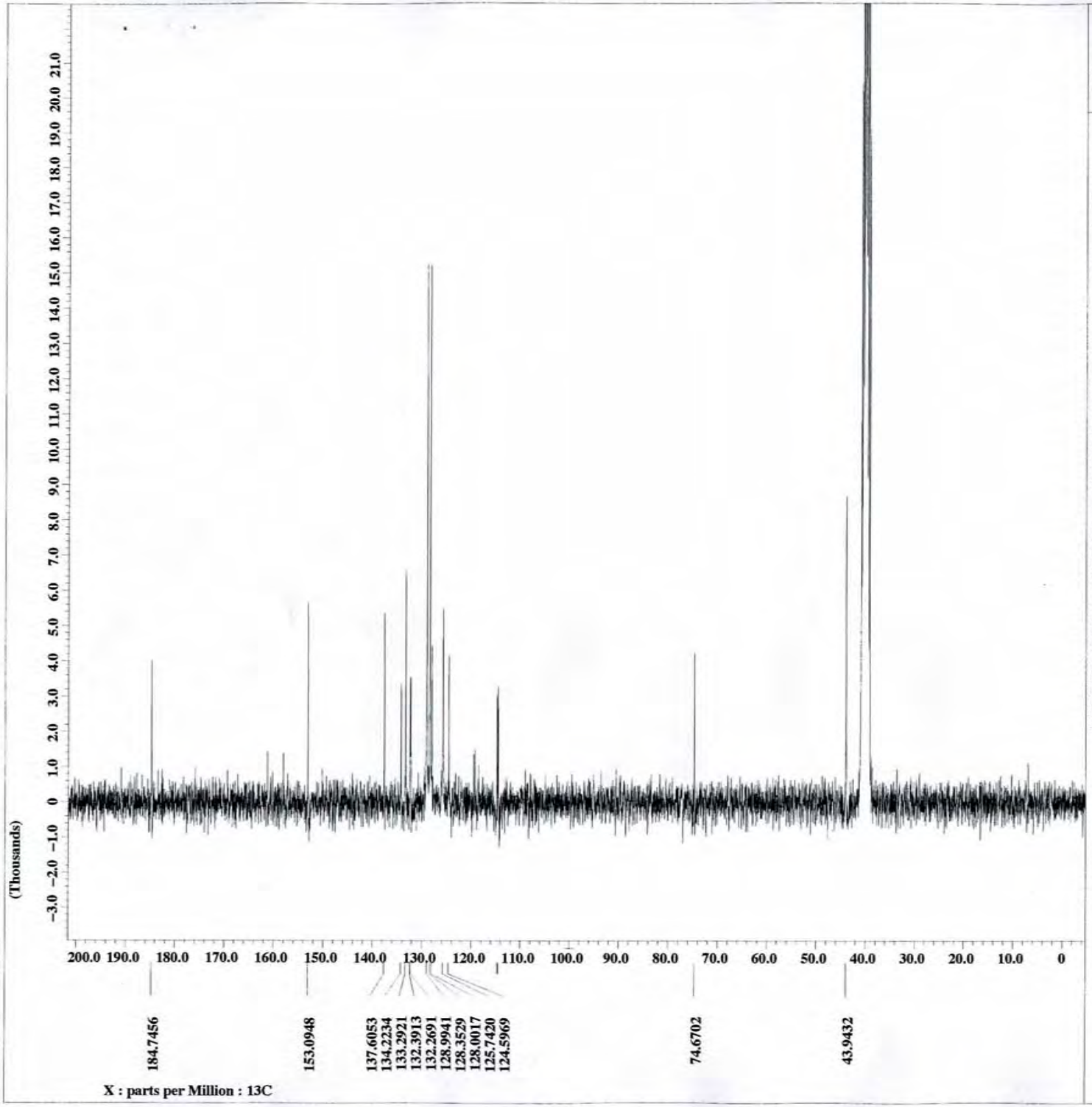


JEOL

Filename	= 1d_spectrum-89.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#348461
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 09:34:15
Revision_time	= 8-DEC-2010 10:49:55
Current_time	= 8-DEC-2010 10:50:00
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 21.4[dC]
Unblank_time	= 2[us]

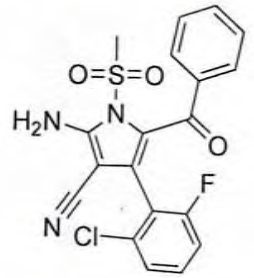


A_{14}
300 MHz, DMSO-d_6

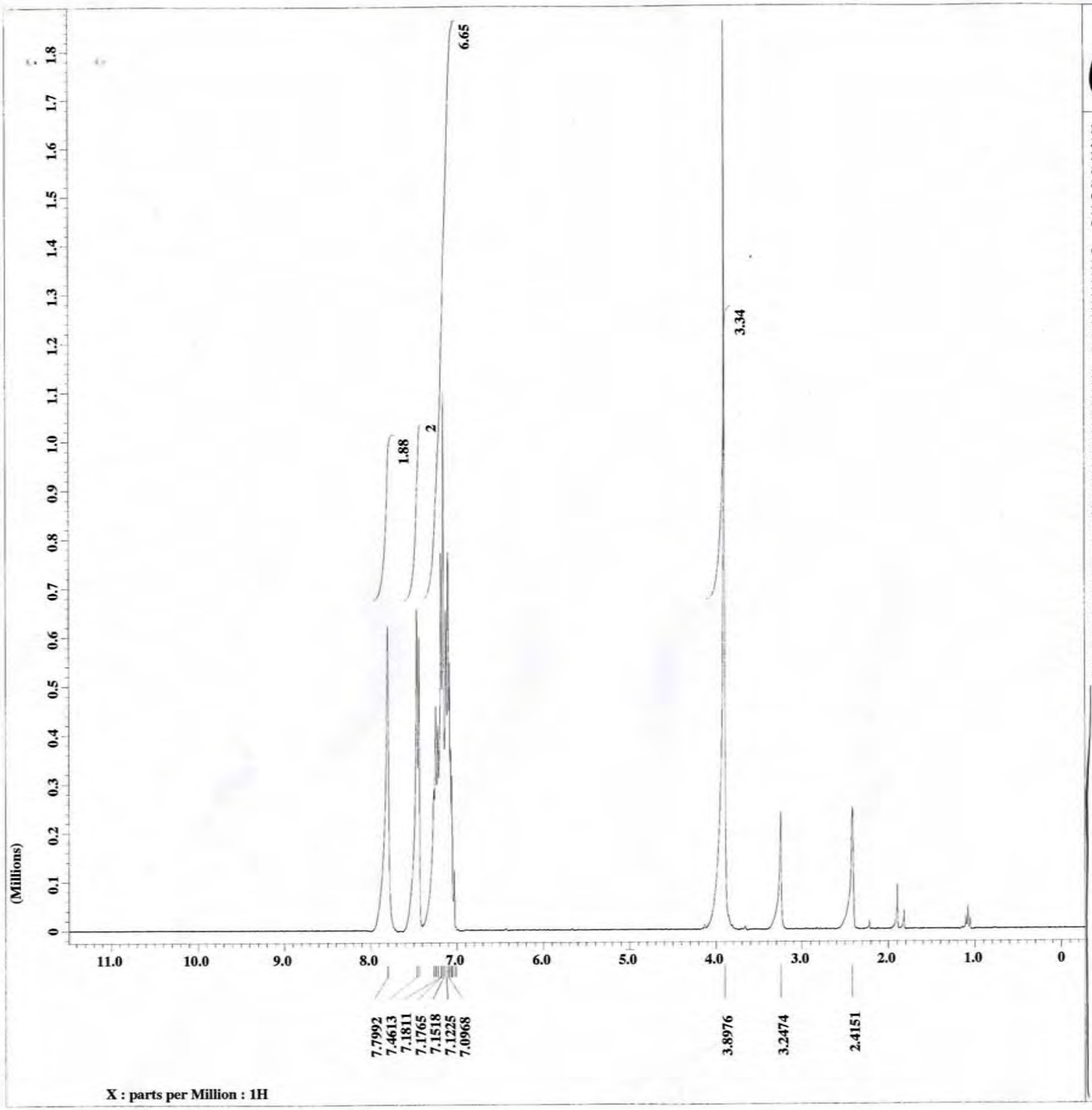


JEOL

Filename	= 1d_13c_spectrum-58.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#349586
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 10:29:58
Revision_time	= 8-DEC-2010 10:41:06
Current_time	= 8-DEC-2010 10:53:38
Comment	= Single Pulse with Bro
Data_format	= 1D REAL
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 22.9[dC]
Unblank_time	= 2[us]

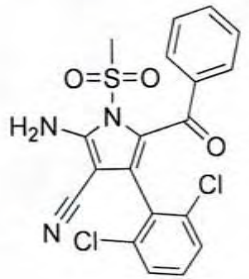


A_{14}
75 MHz, DMSO-d_6

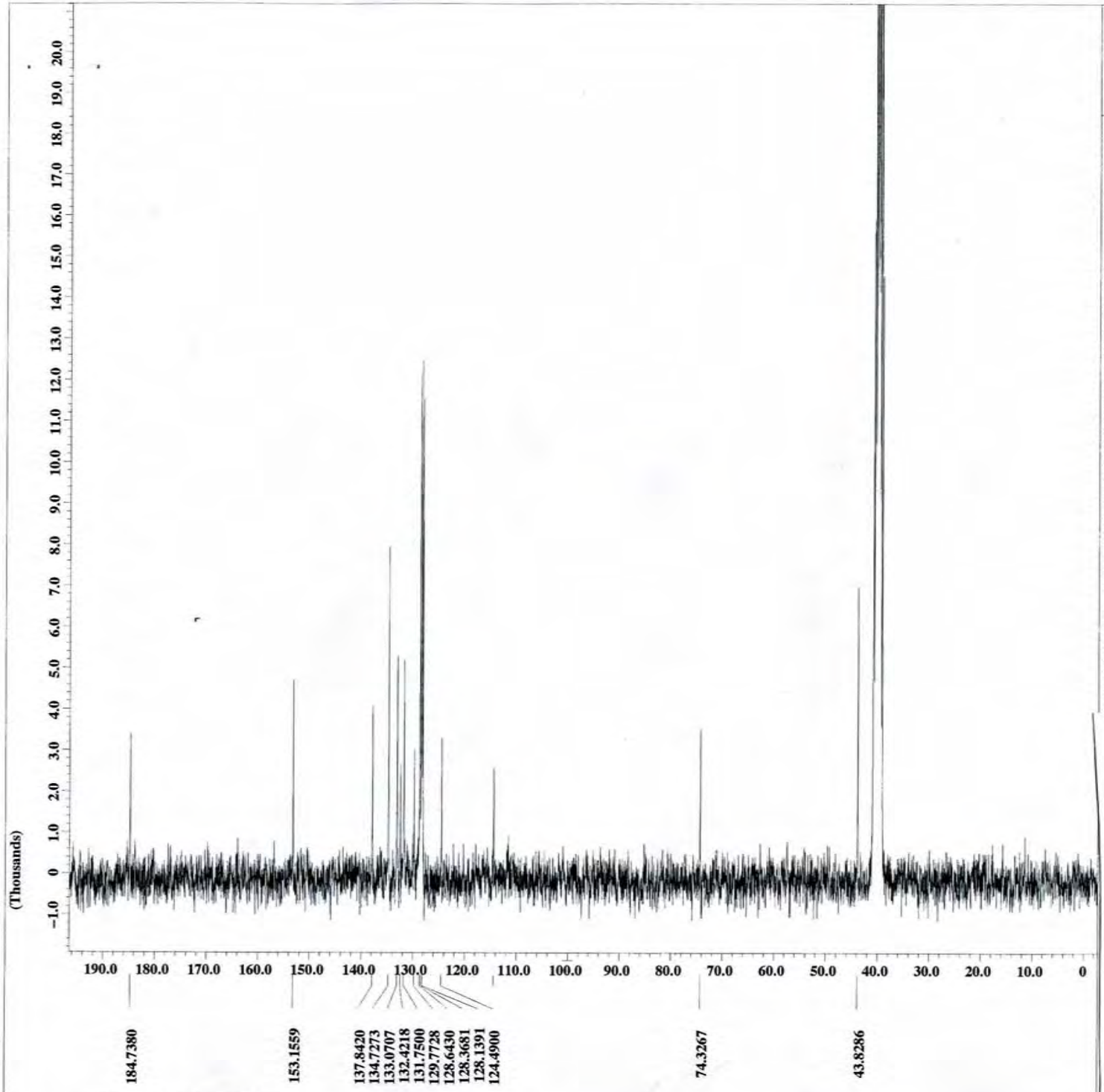


JEOL

Filename	= 1d_spectrum-86.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#598507
Solvent	= DMSO-D6
Creation_time	= 7-DEC-2010 16:31:04
Revision_time	= 7-DEC-2010 16:49:16
Current_time	= 7-DEC-2010 16:49:30
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 23.8[dC]

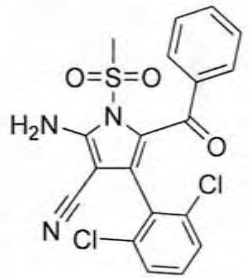


A_{15}
300 MHz, DMSO-d_6

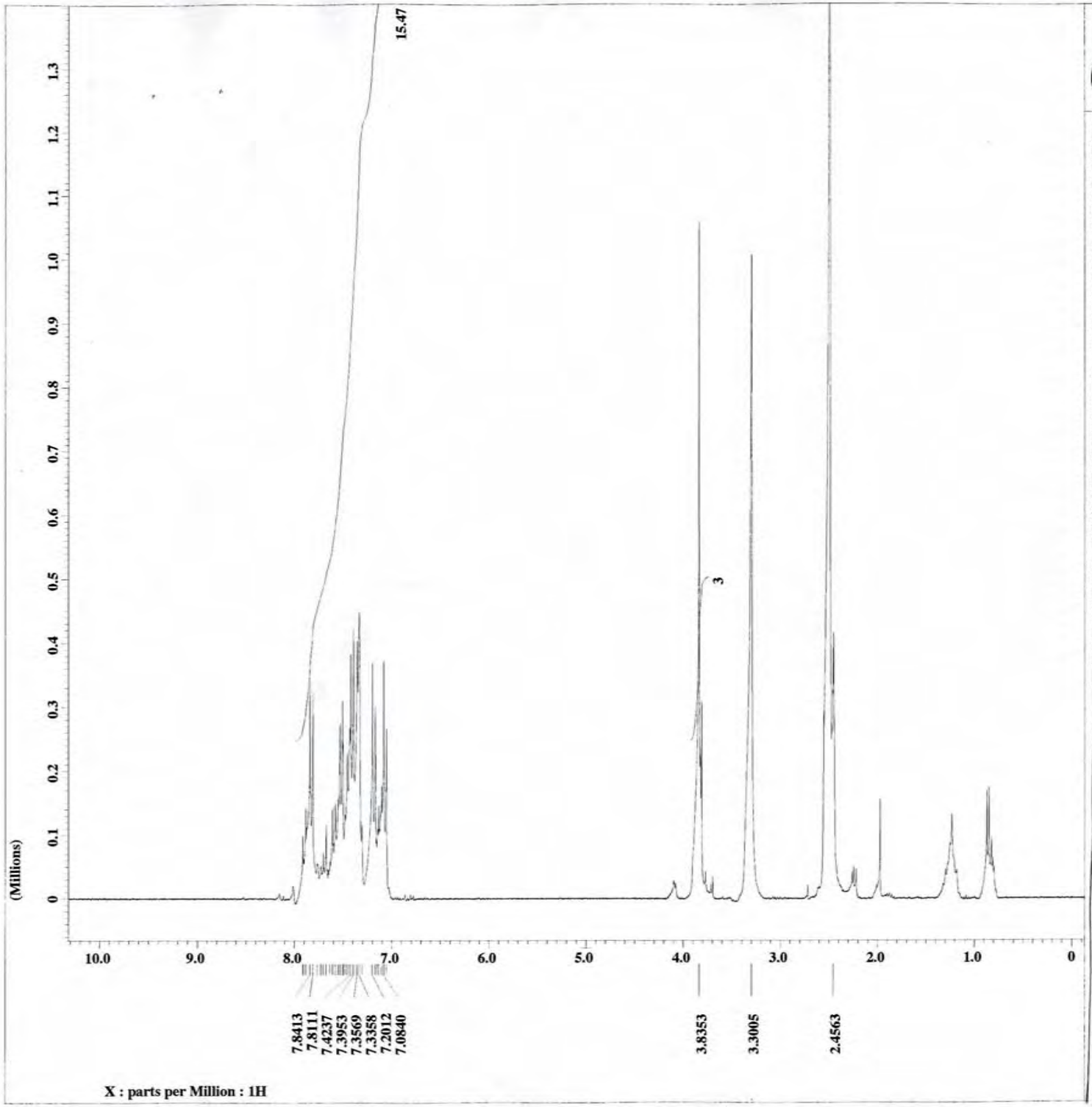


JEOL

Filename	= 1d_13c_spectrum-55.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#599615
Solvent	= DMSO-D6
Creation_time	= 7-DEC-2010 17:26:48
Revision_time	= 7-DEC-2010 17:37:36
Current_time	= 7-DEC-2010 17:37:43
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]

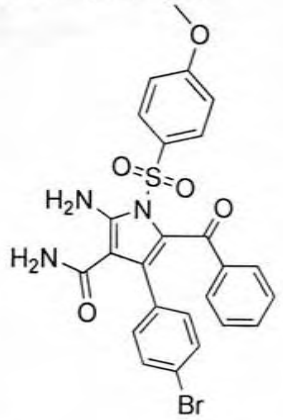


A_{15}
75 MHz, DMSO-d_6

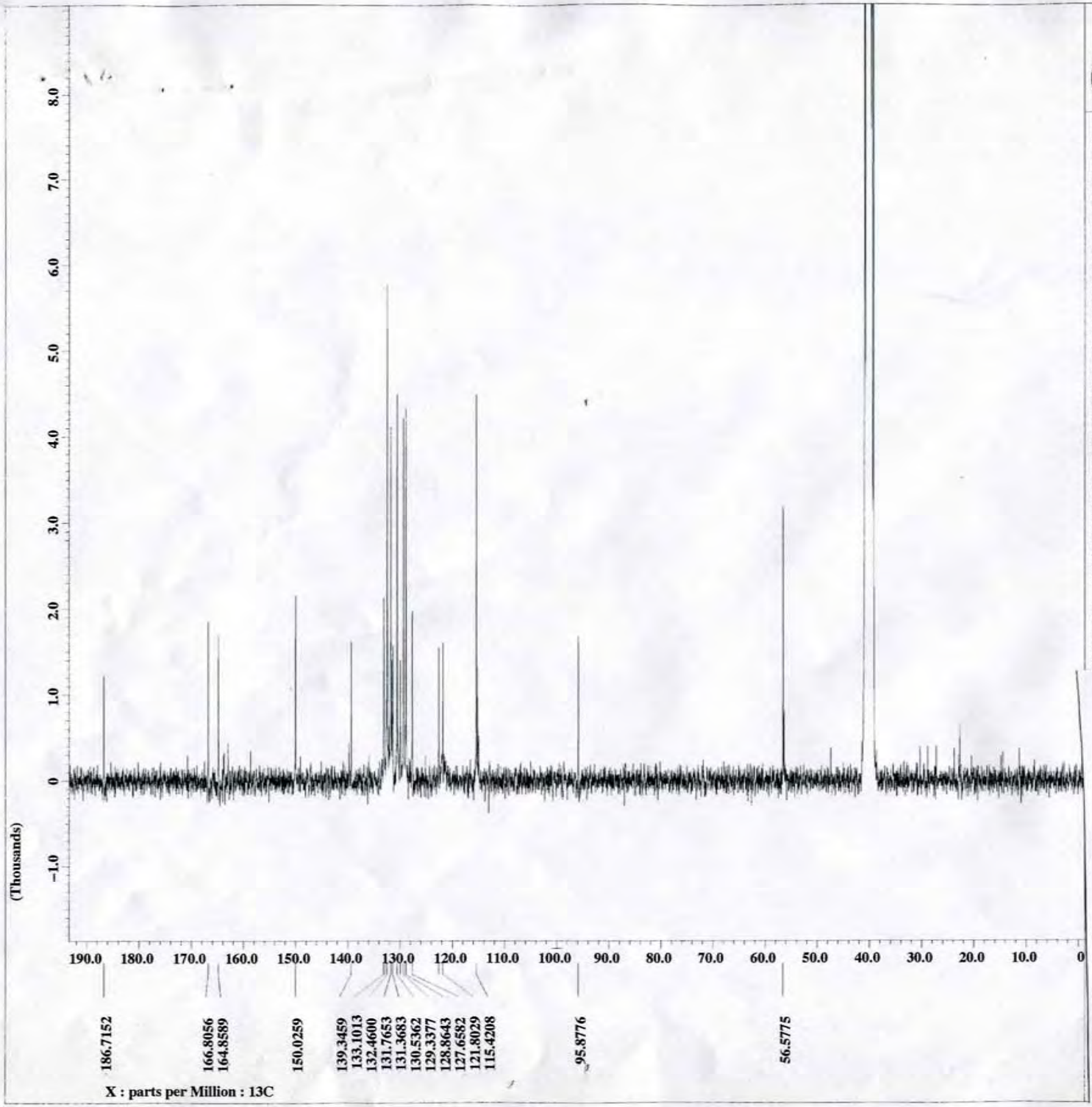


JEOL

```
Filename          = 1d_spectrum-173.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#415190
Solvent           = DMSO-D6
Creation_time     = 14-DEC-2010 11:25:29
Revision_time     = 14-DEC-2010 11:51:28
Current_time      = 14-DEC-2010 11:51:44

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
```

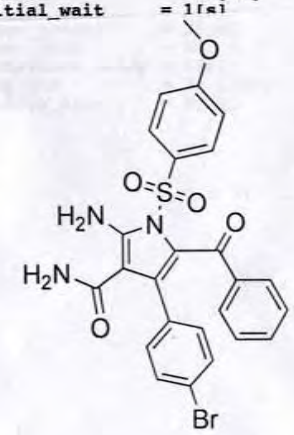


A_{16}
300 MHz, DMSO-d_6

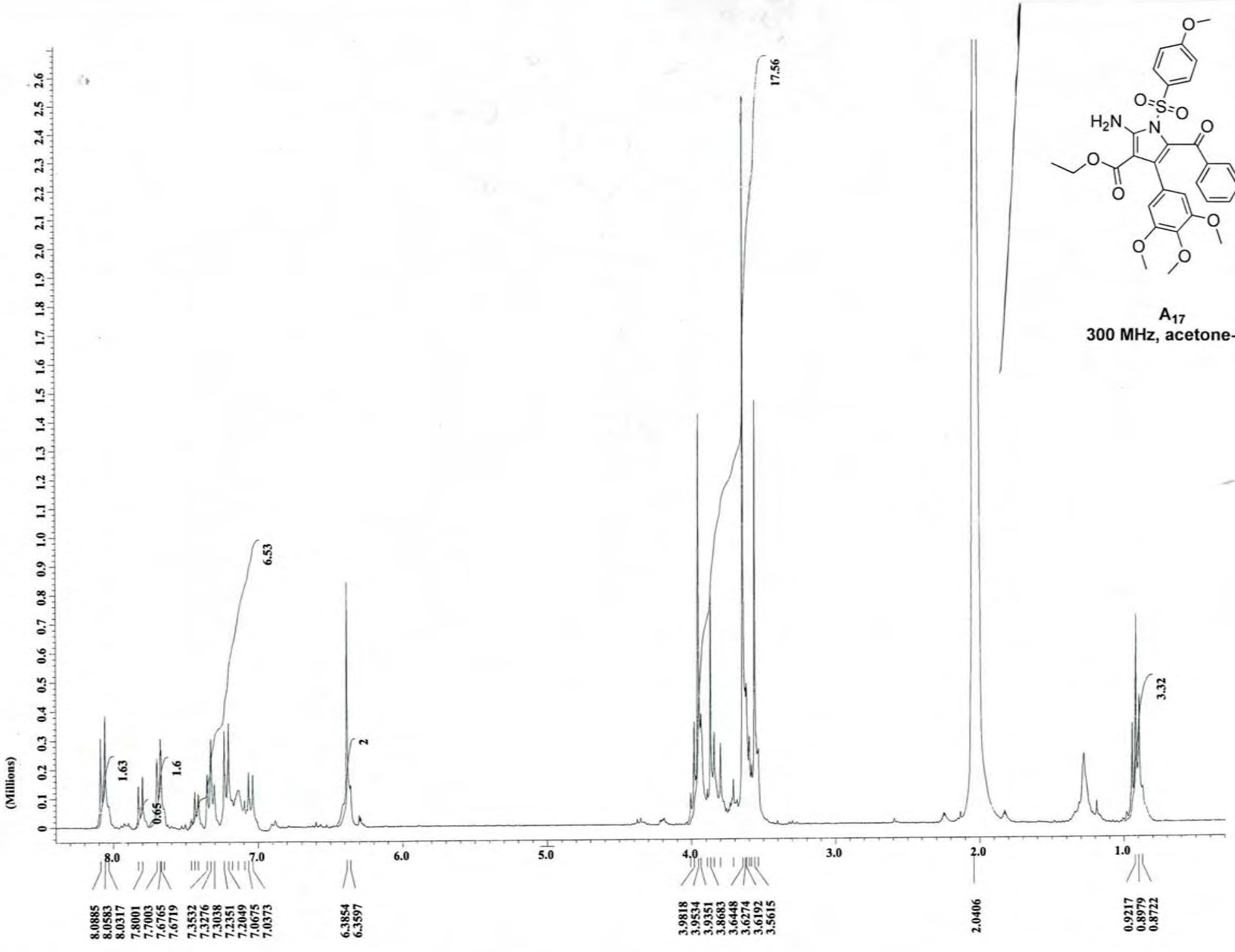


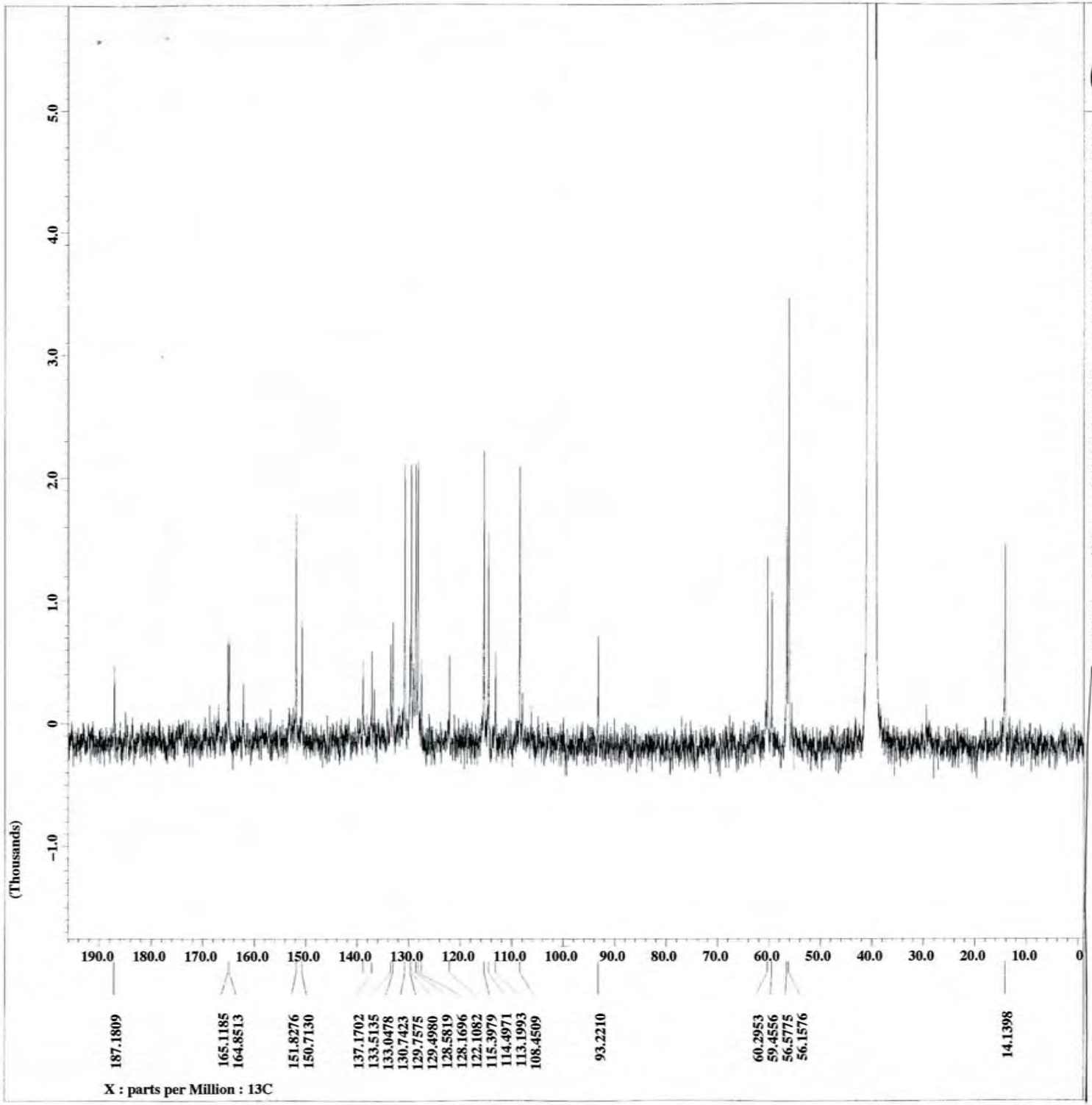
JEOL

```
Filename          = 1d_13c_spectrum-91.jd
Author            = alex
Experiment        = single_pulse_dec
Sample_id         = S#638404
Solvent           = DMSO-D6
Creation_time     = 12-DEC-2010 05:45:38
Revision_time     = 12-DEC-2010 11:27:20
Current_time      = 12-DEC-2010 11:27:31

Comment           = Single Pulse with Bro
Data_format       = 1D REAL
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 16000
Total_scans       = 16000

X_90_width        = 8.1[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 2.7[us]
Initial_wait      = 1[s]
```



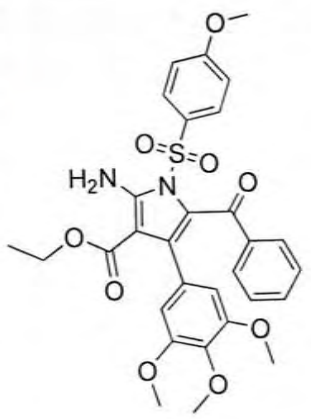
A_{16}
75 MHz, DMSO-d_6


A17
300 MHz, acetone-d6
(Millions)
X : parts per Million : 1H
8.0885
8.0583
8.0317
7.8001
7.7003
7.6765
7.6719
7.3532
7.3276
7.3038
7.2351
7.2049
7.0675
7.0373
6.3854
6.3597
3.9818
3.9534
3.9351
3.8683
3.6448
3.6274
3.6192
3.5615
2.0406
0.9217
0.8979
0.8722
1.63
0.65
1.6
6.53
2
17.56
3.32

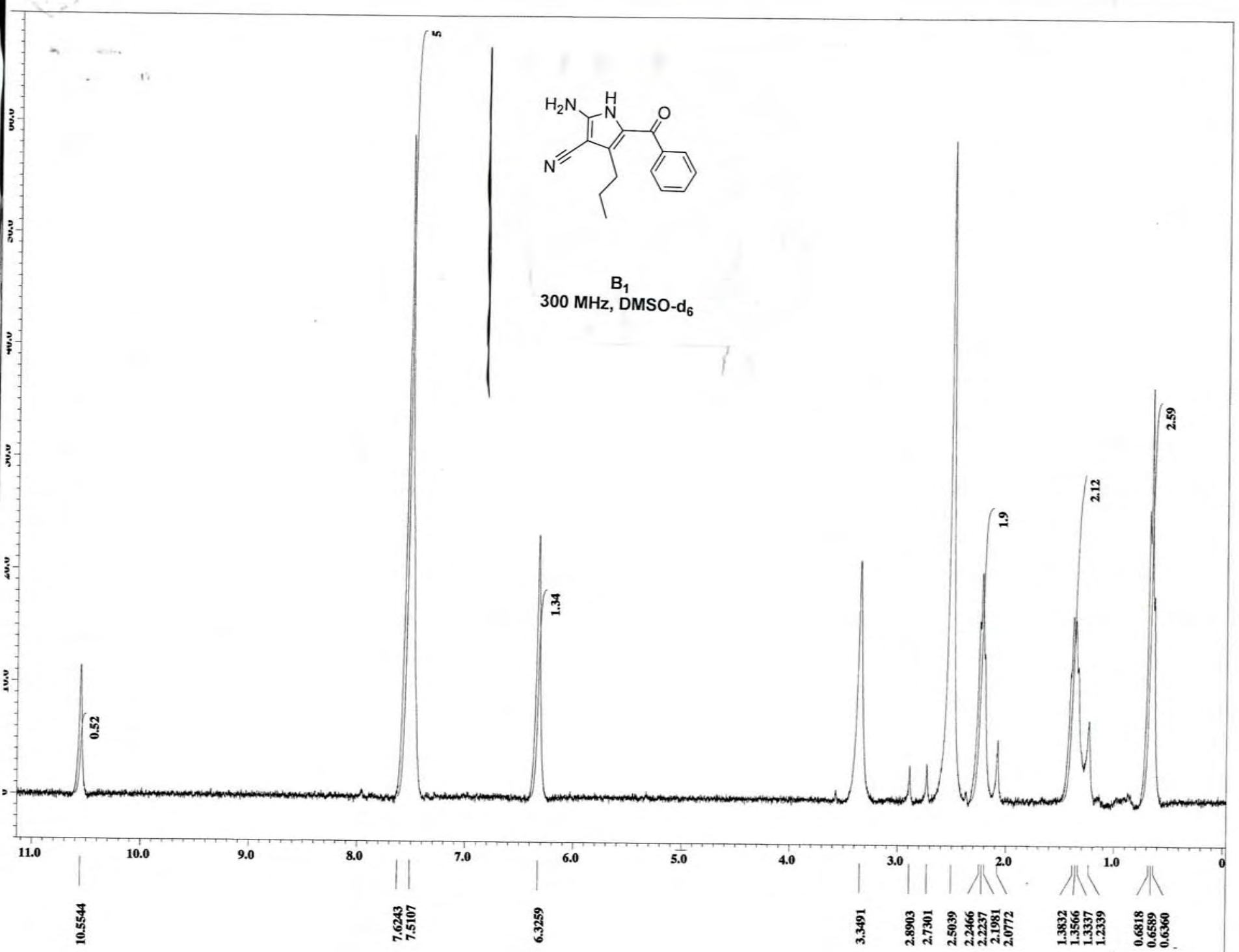


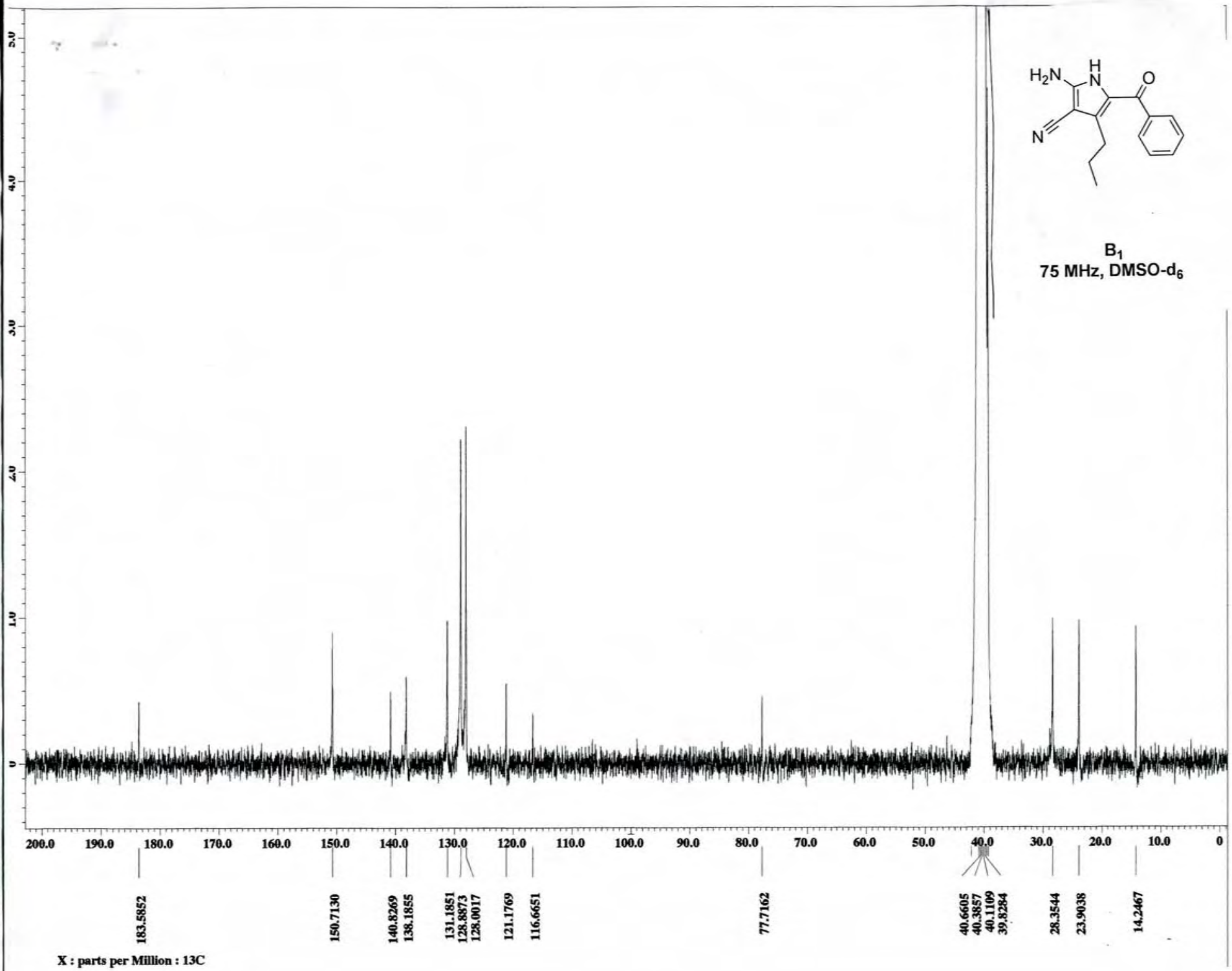
JEOL

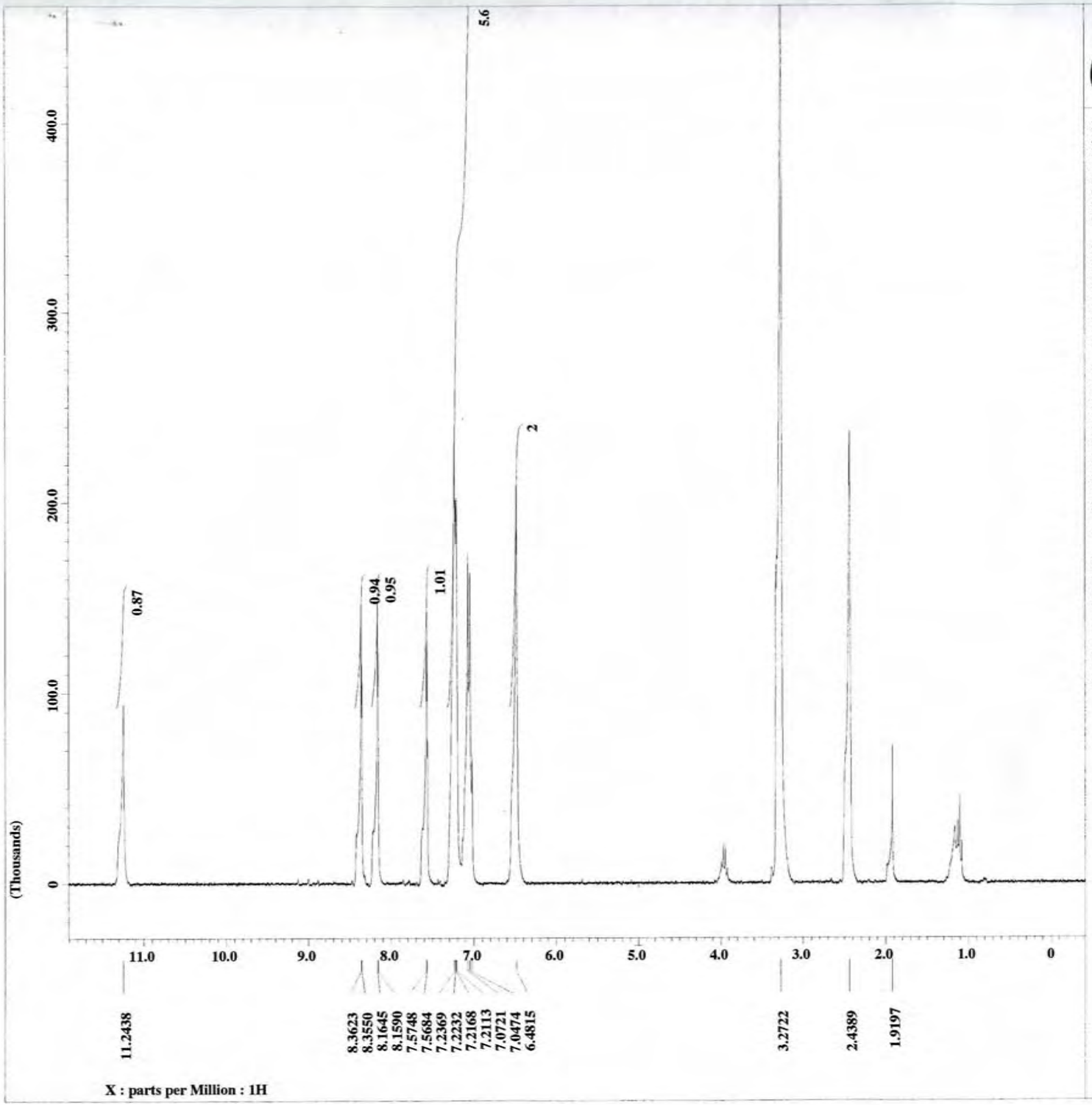
Filename	= 1d_13c_spectrum-110.j
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#589070
Solvent	= DMSO-D6
Creation_time	= 13-DEC-2010 07:25:39
Revision_time	= 20-DEC-2010 15:30:47
Current_time	= 20-DEC-2010 15:32:14
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 20000
Total_scans	= 20000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]



A_{17}
75 MHz, DMSO-d_6


B1
300 MHz, DMSO-d6
0.52
1.34
1.9
2.12
2.59
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.5544
7.6243
7.5107
6.3259
3.3491
2.8903
2.7301
2.5039
2.2466
2.2237
2.1981
2.0772
1.3832
1.3566
1.3337
1.2339
0.6818
0.6589
0.6360
X : parts per Million : 1H

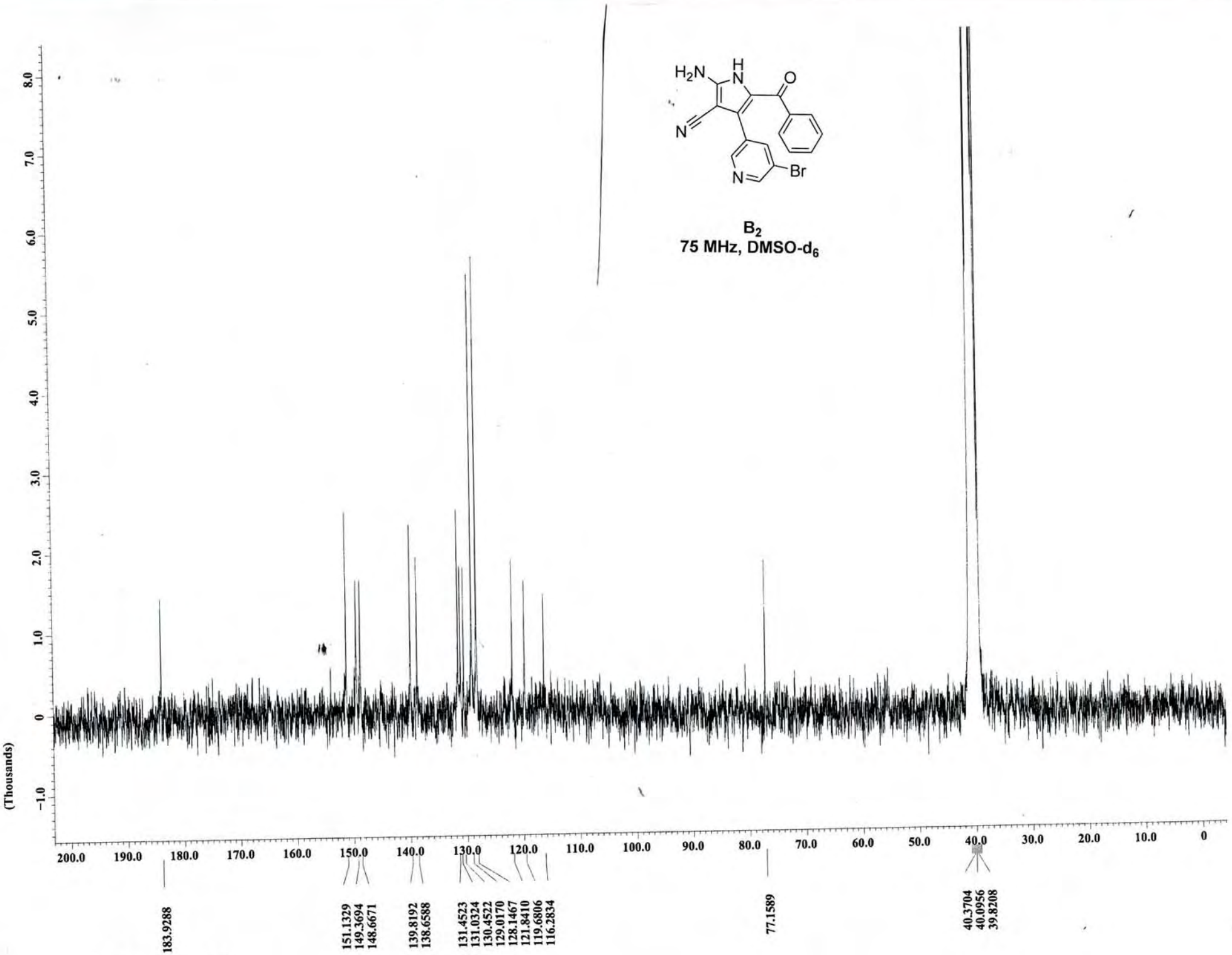

B1
75 MHz, DMSO-d6
183.5852
150.7130
140.8269
138.1855
131.1851
128.8873
128.0017
121.1769
116.6651
77.7162
40.6605
40.3857
40.1109
39.8284
28.3544
23.9038
14.2467
X : parts per Million : 13C



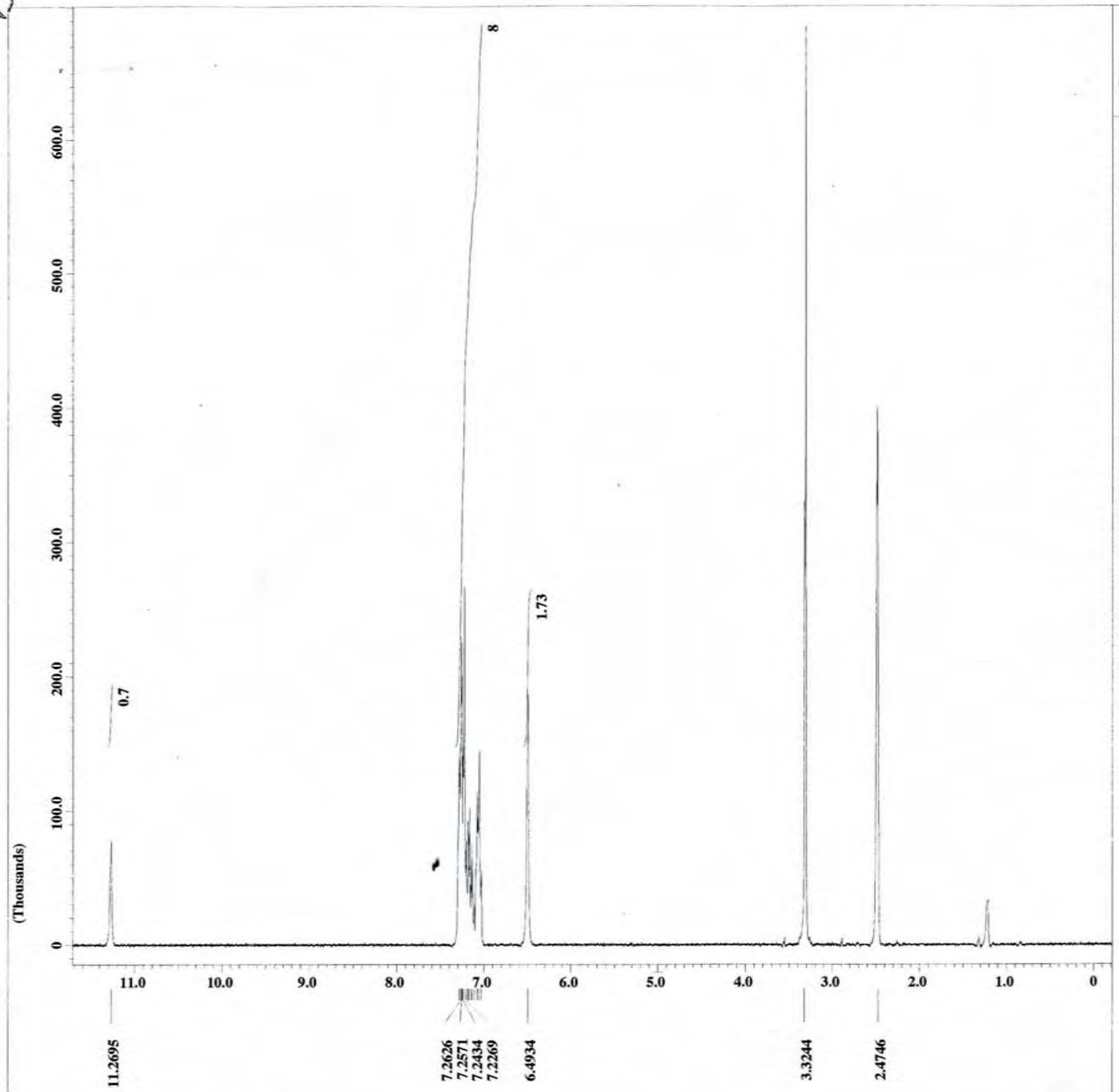
JEOL

Filename	= 1d_spectrum-211.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#622076
Solvent	= DMSO-D6
Creation_time	= 21-DEC-2010 17:11:21
Revision_time	= 21-DEC-2010 17:26:29
Current_time	= 21-DEC-2010 17:26:48
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 23.7[dC]
Unblank_time	= 2[us]

B_2
300 MHz, DMSO-d_6

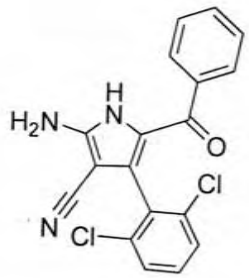

B_2
75 MHz, DMSO-d_6
(Thousands)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
183.9288
151.1329
149.3694
148.6671
139.8192
138.6588
131.4523
131.0324
130.4522
129.0170
128.1467
121.8410
119.6806
116.2834
77.1589
40.3704
40.0956
39.8208
X : parts per Million : 13C

B3

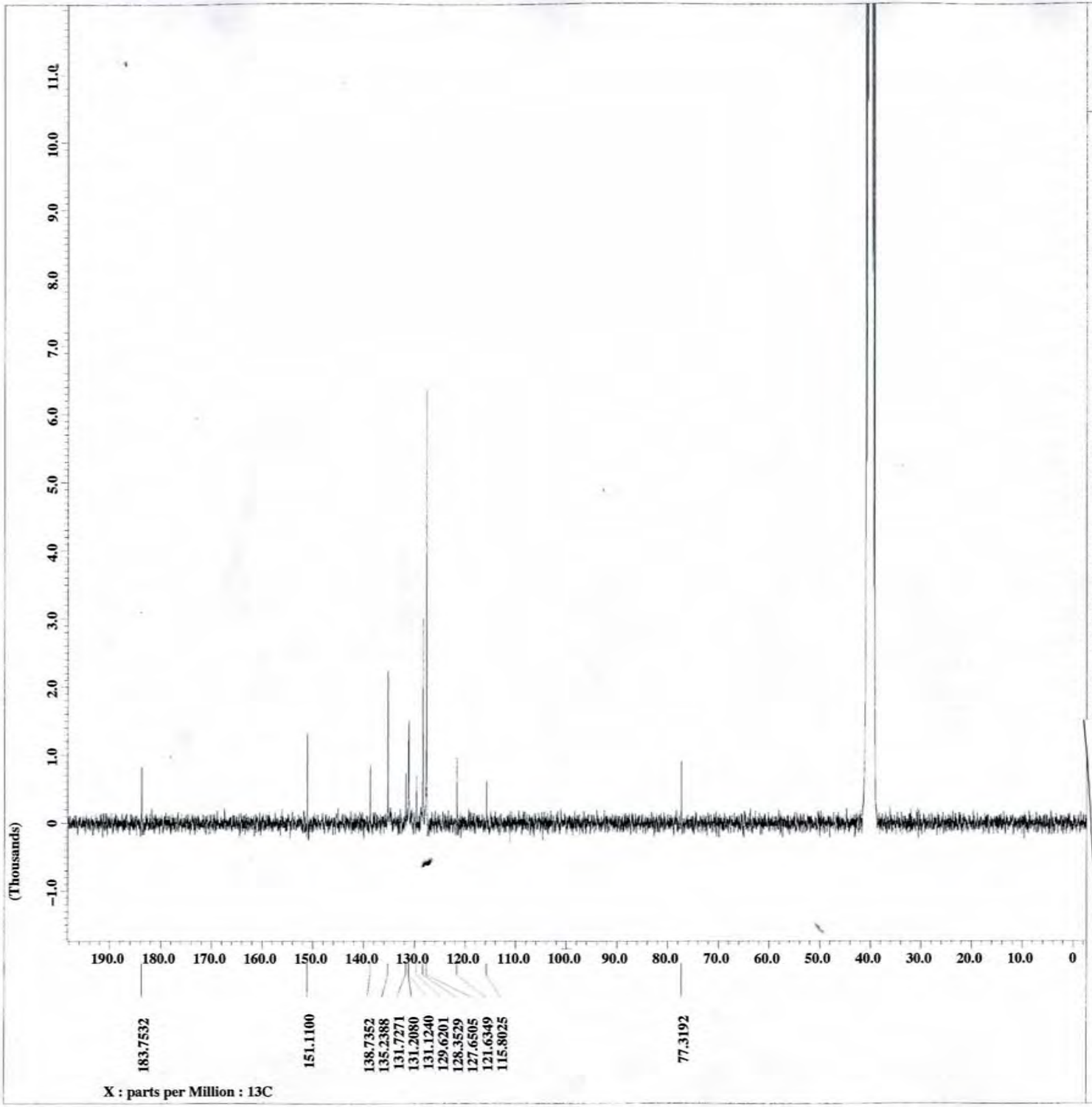


JEOL

Filename	= 1d_spectrum-151.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#705386
Solvent	= DMSO-D6
Creation_time	= 10-DEC-2010 19:29:07
Revision_time	= 11-DEC-2010 14:03:14
Current_time	= 11-DEC-2010 14:03:21
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 22.7[dC]
Unblank_time	= 2[us]

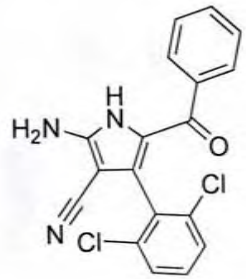


B_3
300 MHz, DMSO-d_6

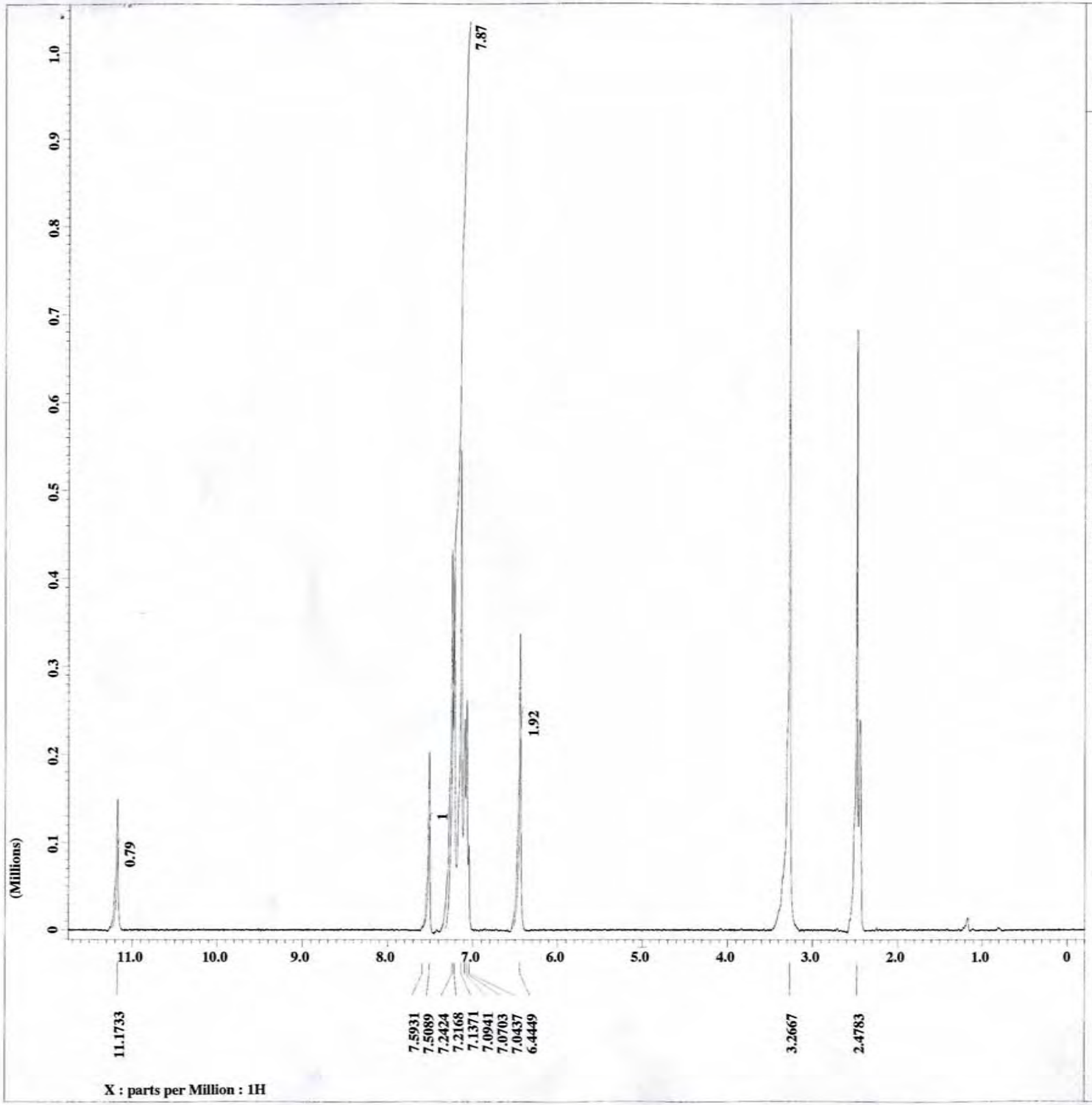


JEOL

Filename	= 1d_13c_spectrum-86.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#706810
Solvent	= DMSO-D6
Creation_time	= 11-DEC-2010 10:41:50
Revision_time	= 11-DEC-2010 13:49:26
Current_time	= 11-DEC-2010 13:49:53
Comment	= Single Pulse with Bro
Data_format	= 1D REAL
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 20000
Total_scans	= 20000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 23[dC]



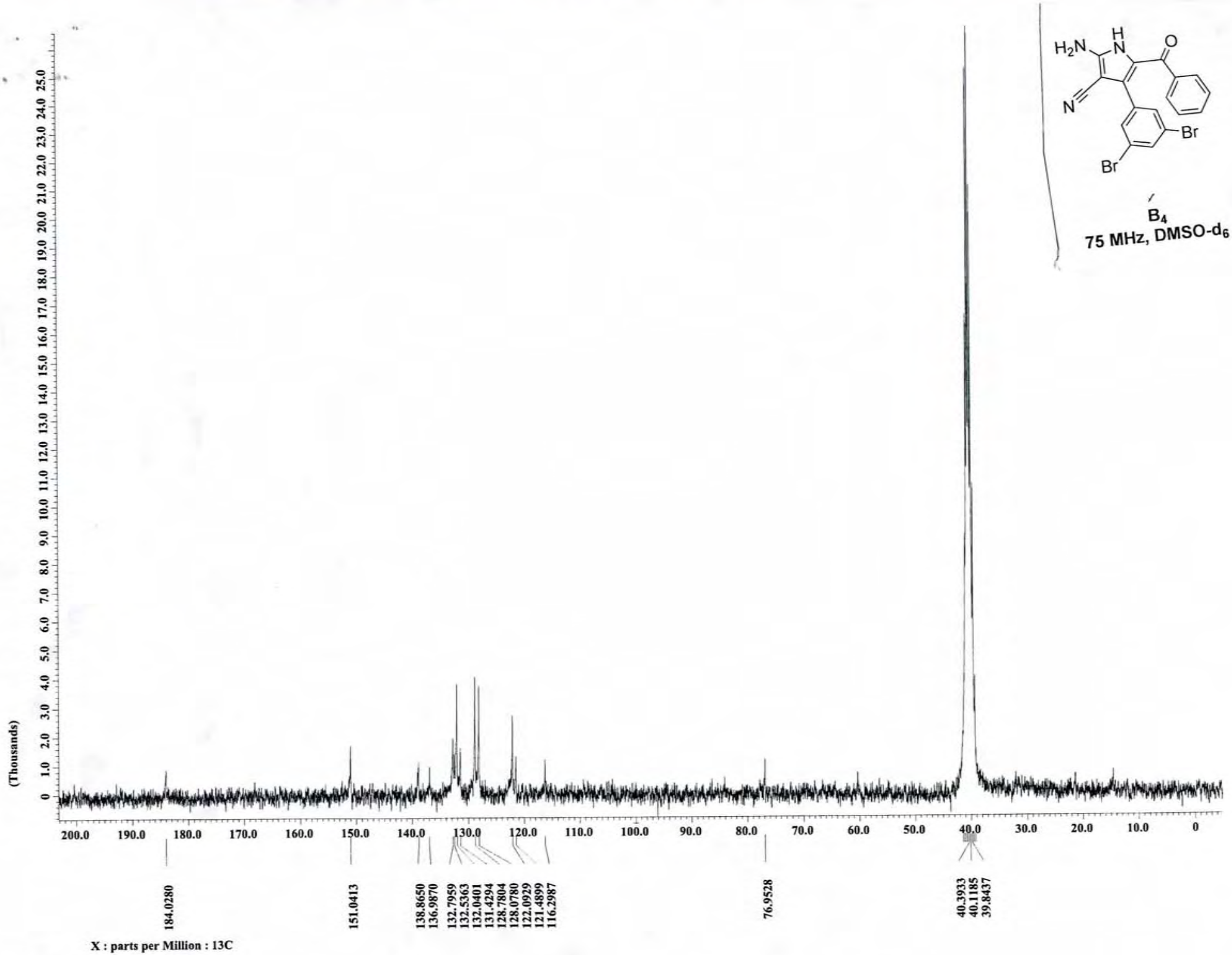
B_3
75 MHz, DMSO-d_6

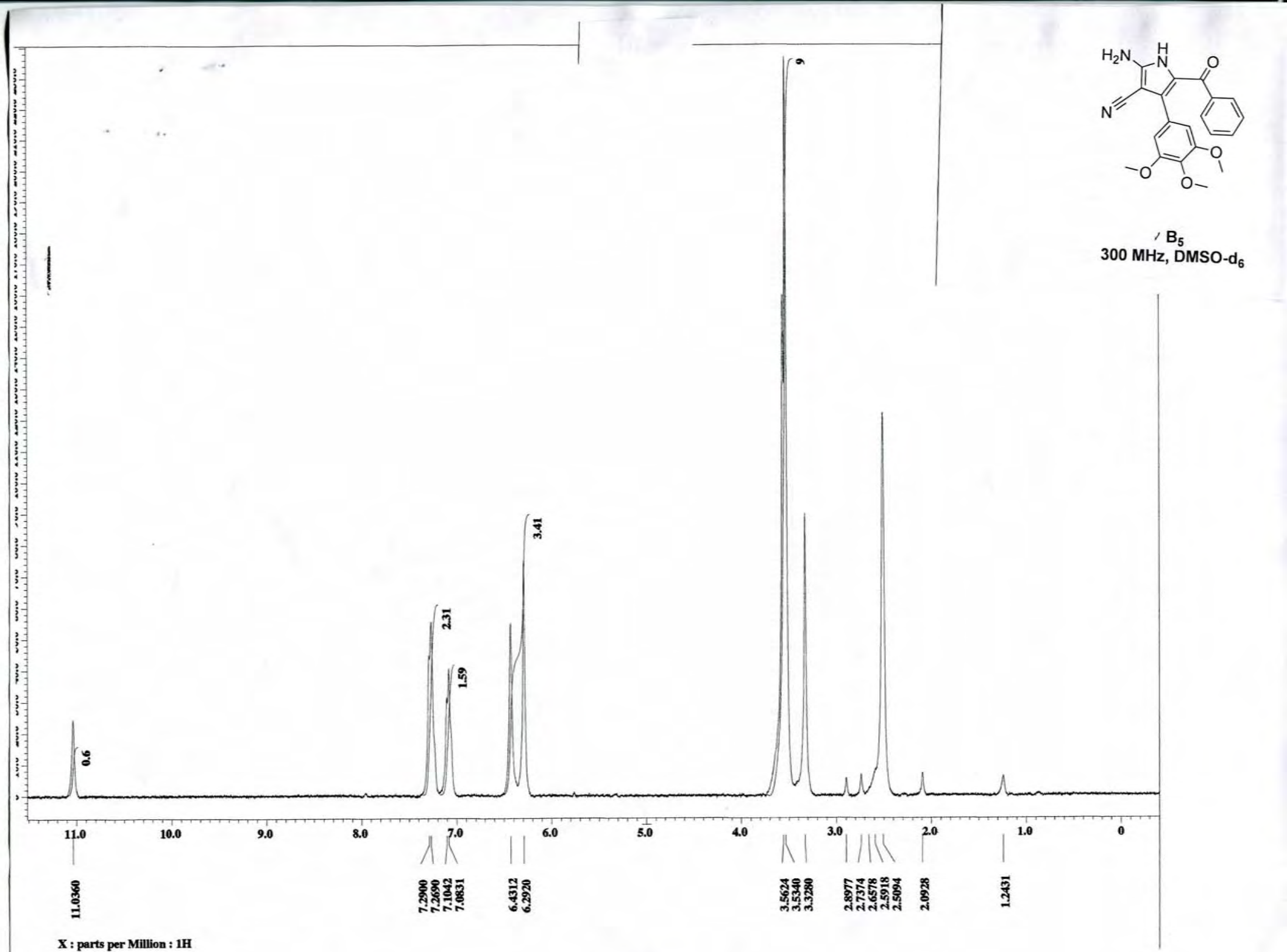


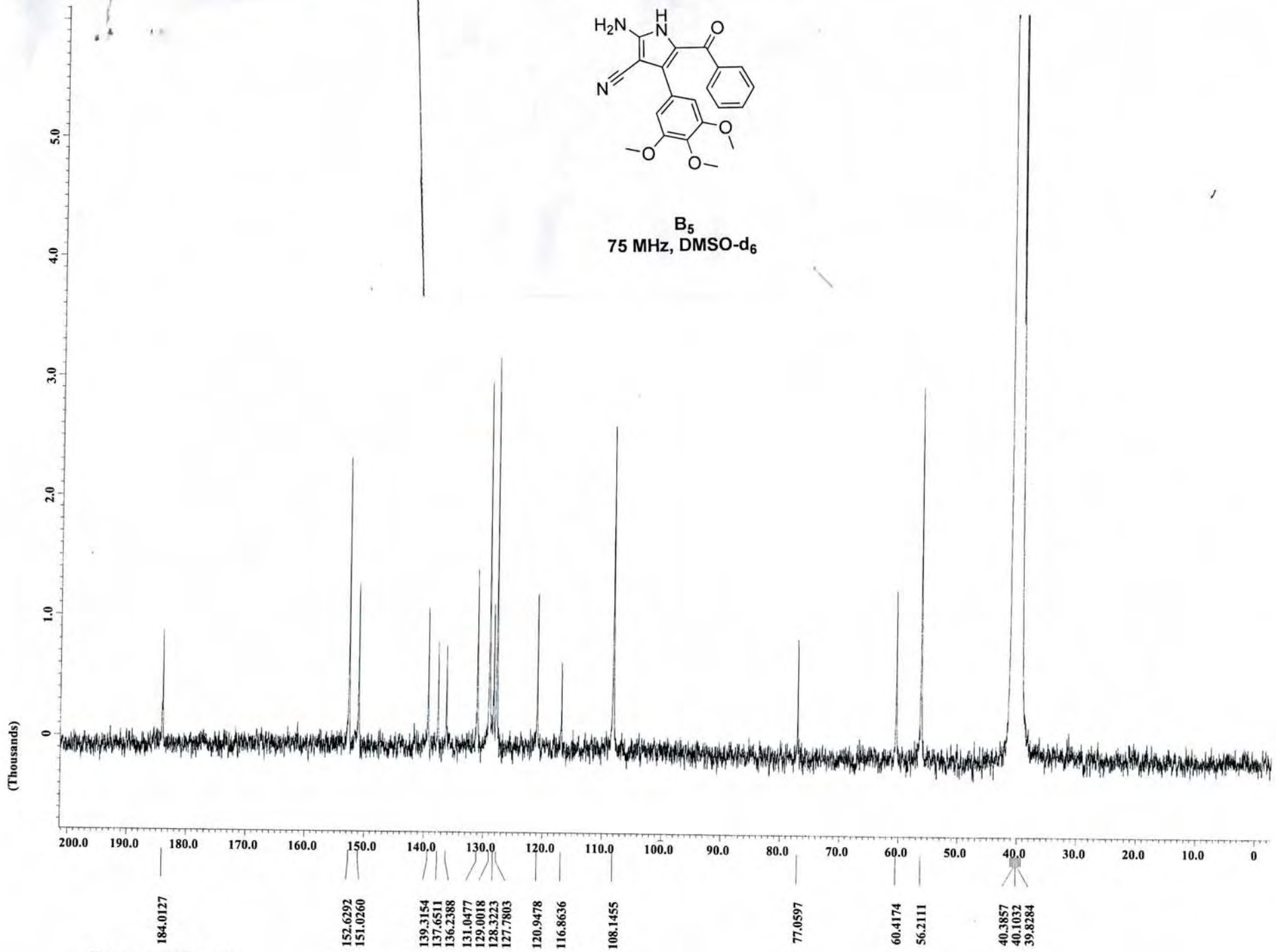
JEOL

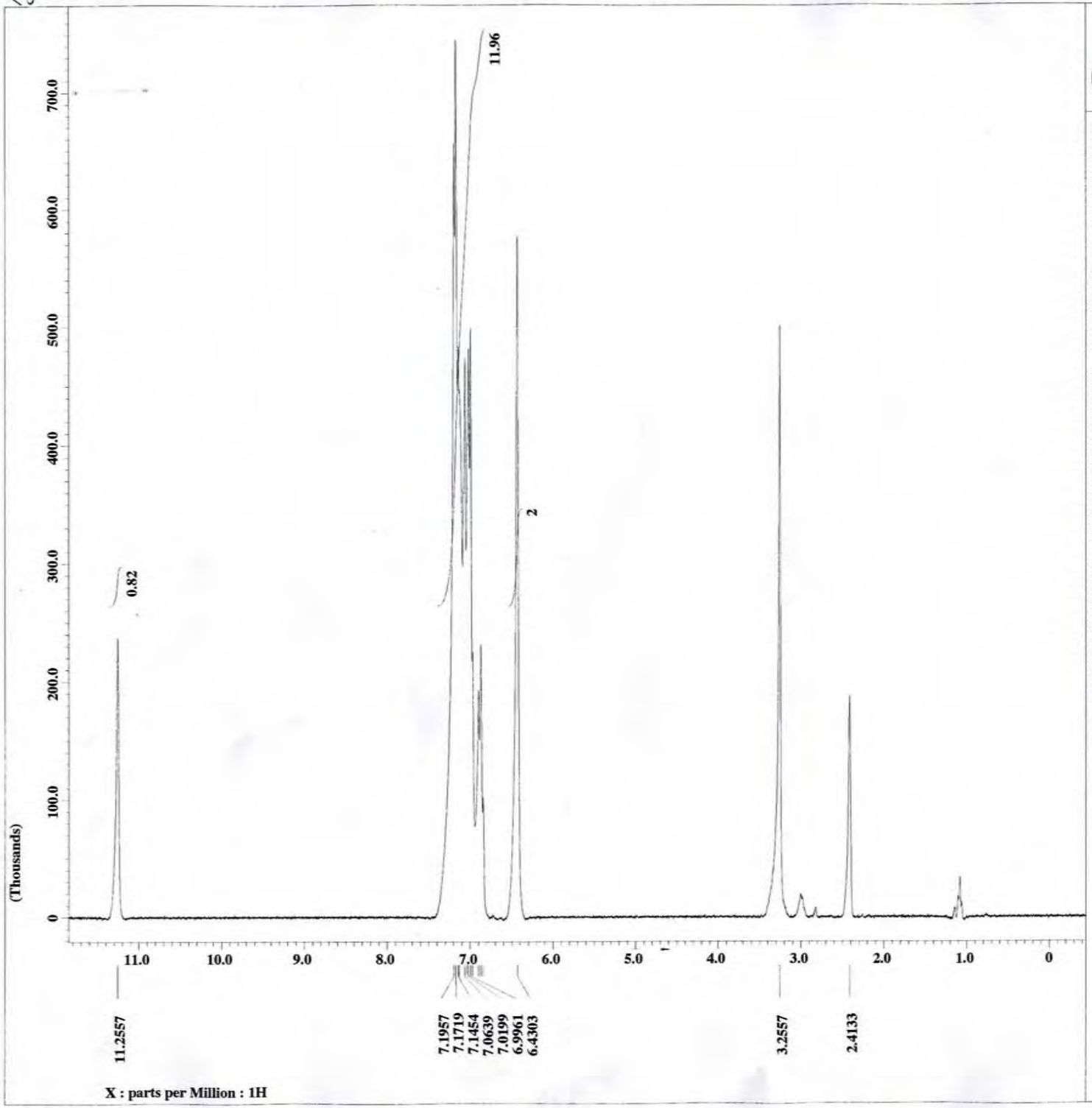
Filename	= 1d_spectrum-203.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#616018
Solvent	= DMSO-D6
Creation_time	= 20-DEC-2010 17:00:15
Revision_time	= 20-DEC-2010 17:13:25
Current_time	= 20-DEC-2010 17:13:58
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 24.4[dC]
Unblank_time	= 2[us]

B_4
300 MHz, DMSO-d_6


B4
75 MHz, DMSO-d6
(Thousands)
X : parts per Million : 13C
184.0280
151.0413
138.8650
136.9870
132.7959
132.5363
132.0401
131.4294
128.7804
128.0780
122.0929
121.4899
116.2987
76.9528
40.3933
40.1185
39.8437

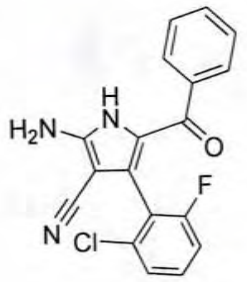

B5
300 MHz, DMSO-d6
0.6
2.31
1.59
3.41
9
11.0360
7.2900
7.2690
7.1042
7.0831
6.4312
6.2920
3.5624
3.5340
3.3280
2.8977
2.7374
2.6578
2.5918
2.5094
2.0928
1.2431
X : parts per Million : 1H


B5
75 MHz, DMSO-d6
(Thousands)
5.0
4.0
3.0
2.0
1.0
0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
184.0127
152.6292
151.0260
139.3154
137.6511
136.2388
131.0477
129.0018
128.3223
127.7803
120.9478
116.8636
108.1455
77.0597
60.4174
56.2111
40.3857
40.1032
39.8284
X : parts per Million : 13C

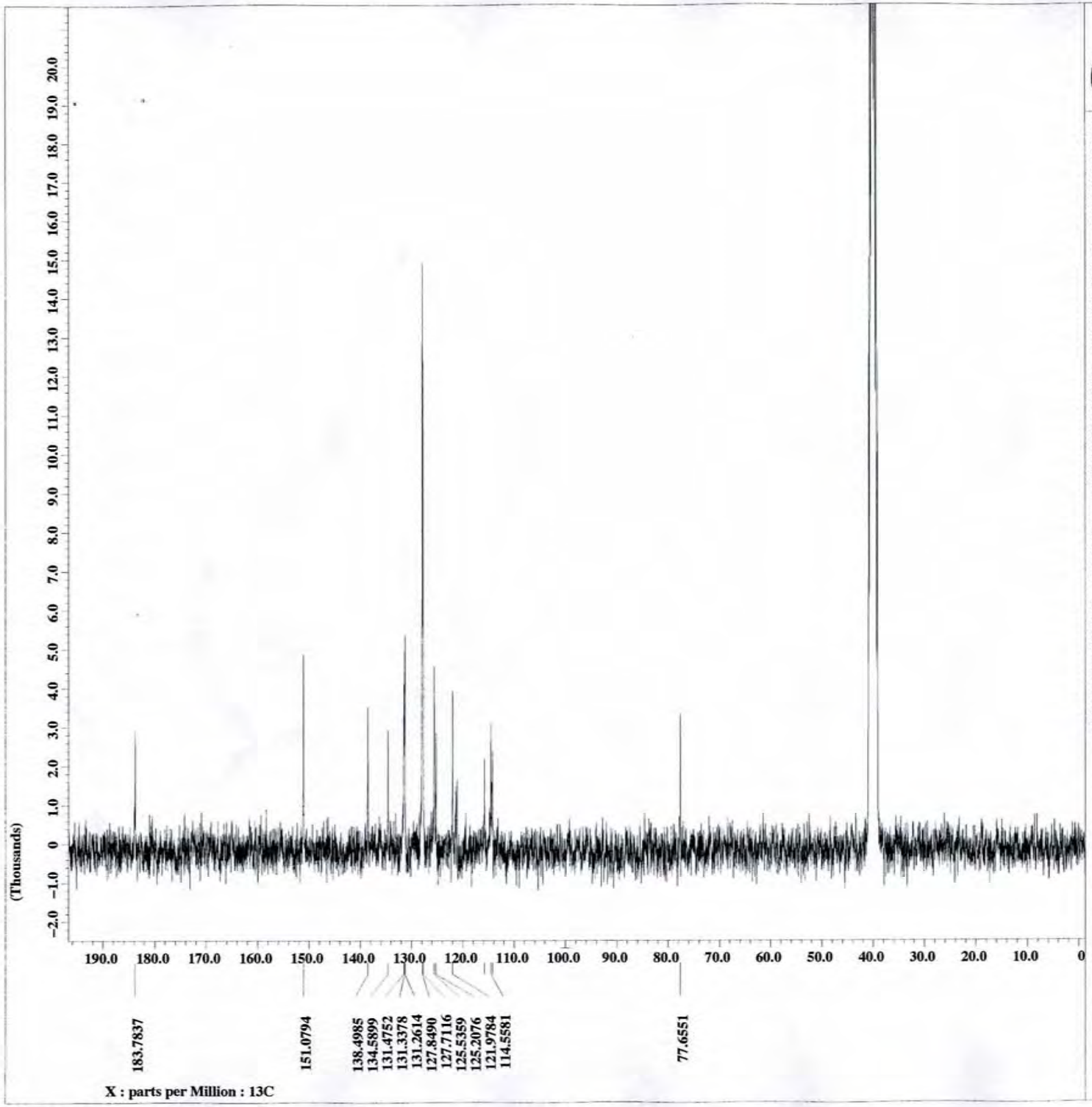


JEOL

```
Filename          = 1d_spectrum-103.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#549206
Solvent           = DMSO-D6
Creation_time     =  8-DEC-2010 15:08:51
Revision_time     =  8-DEC-2010 16:40:24
Current_time      =  8-DEC-2010 16:40:34

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23.5[dC]
Unblank_time      = 2[us]
```

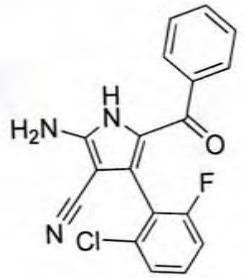


B_6
300 MHz, DMSO-d_6

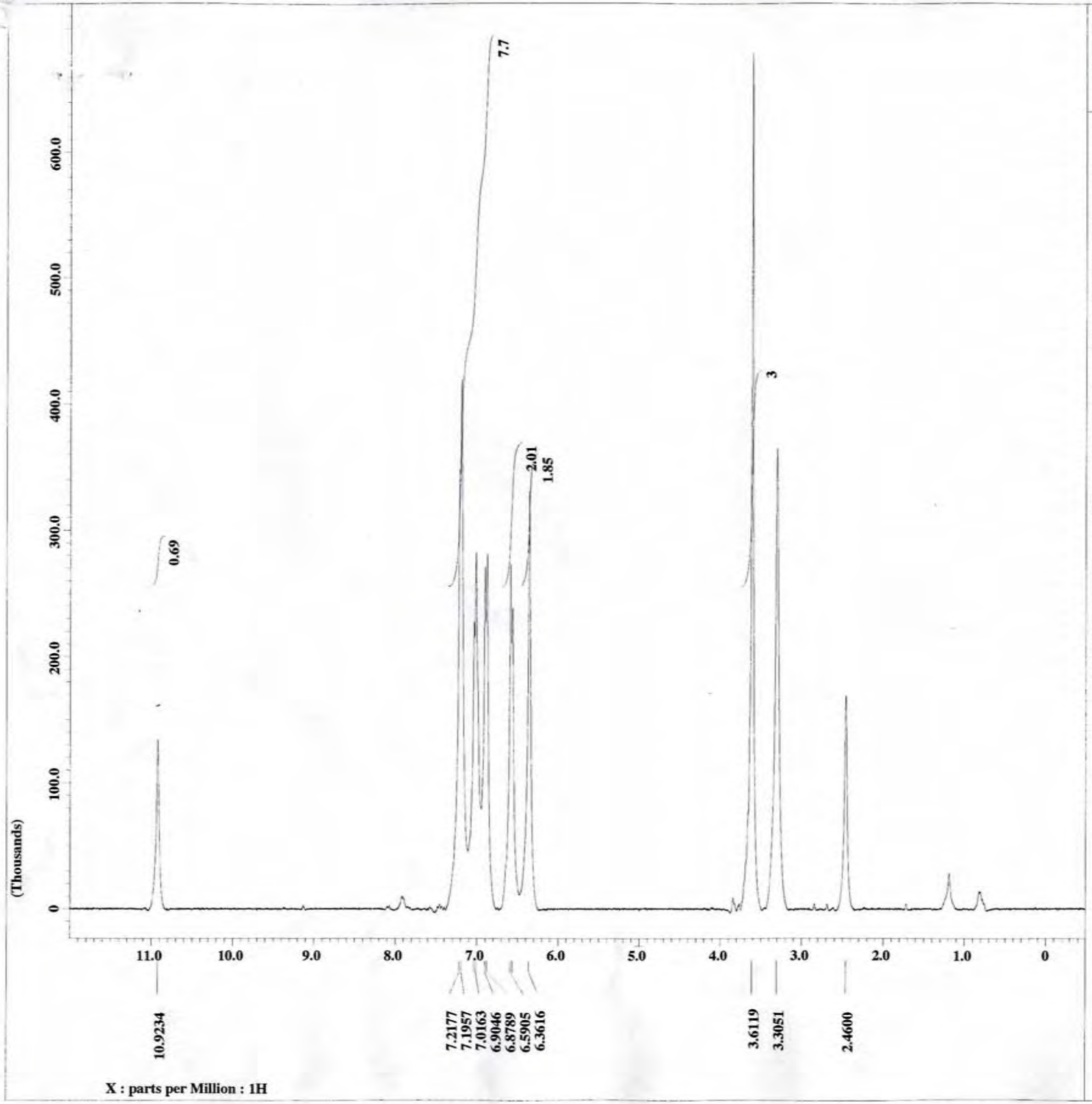


JEOL

Filename	= 1d_13c_spectrum-61.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#550704
Solvent	= DMSO-D6
Creation_time	= 8-DEC-2010 16:05:11
Revision_time	= 8-DEC-2010 16:33:05
Current_time	= 8-DEC-2010 16:34:11
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1200
Total_scans	= 1200
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 24.3[dC]
Unblank_time	= 2[us]

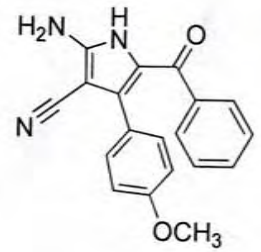


B_6
75 MHz, DMSO-d_6

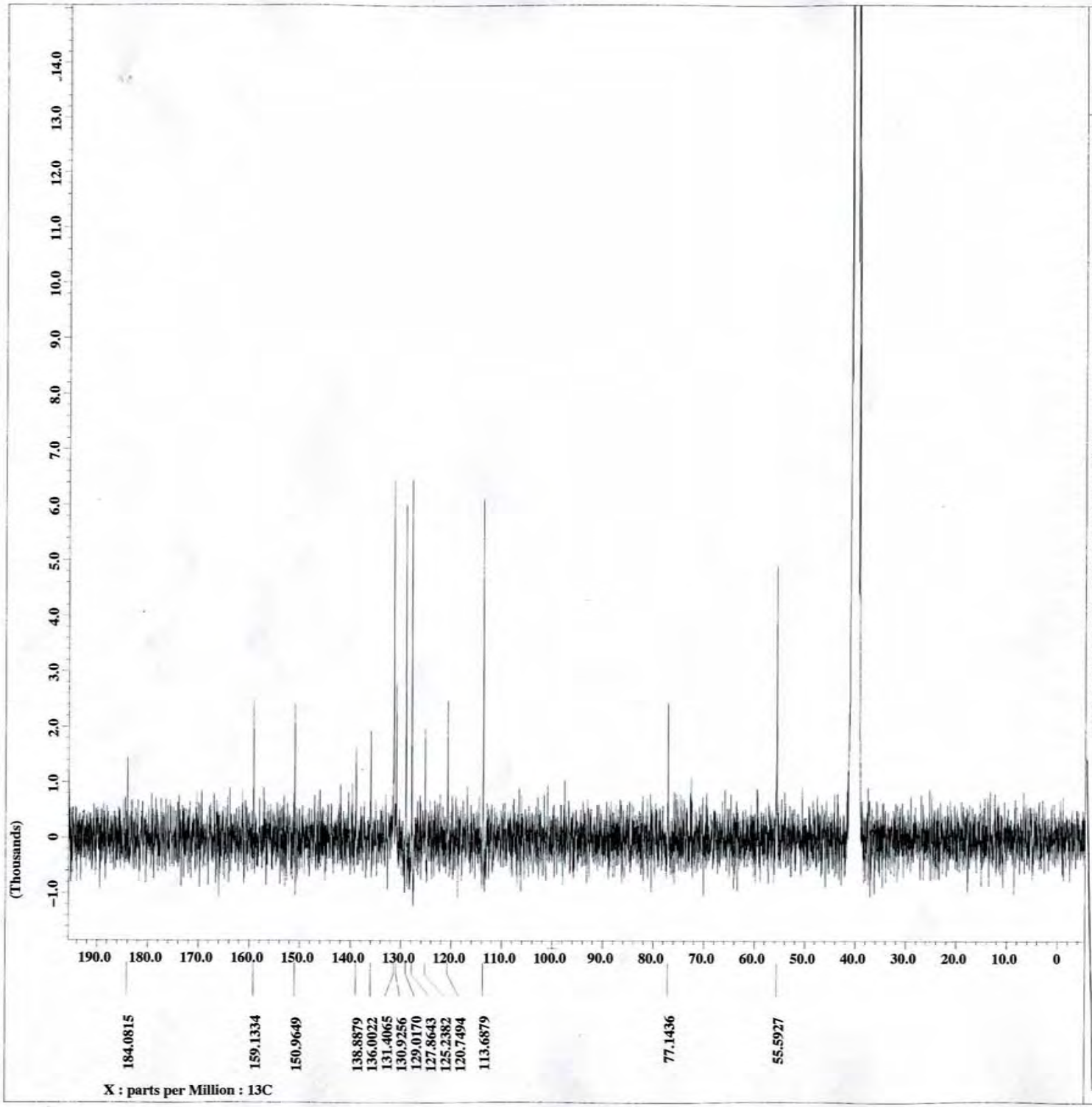


JEOL

Filename	= 1d_spectrum-213.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#624708
Solvent	= DMSO-D6
Creation_time	= 21-DEC-2010 17:15:42
Revision_time	= 21-DEC-2010 17:31:15
Current_time	= 21-DEC-2010 17:31:39
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16
Total_scans	= 16
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 23.8[dC]
Unblank_time	= 2[us]

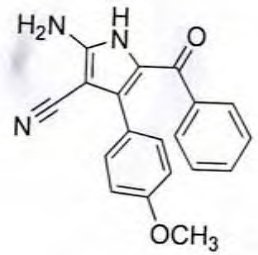


B_7
300 MHz, DMSO-d_6

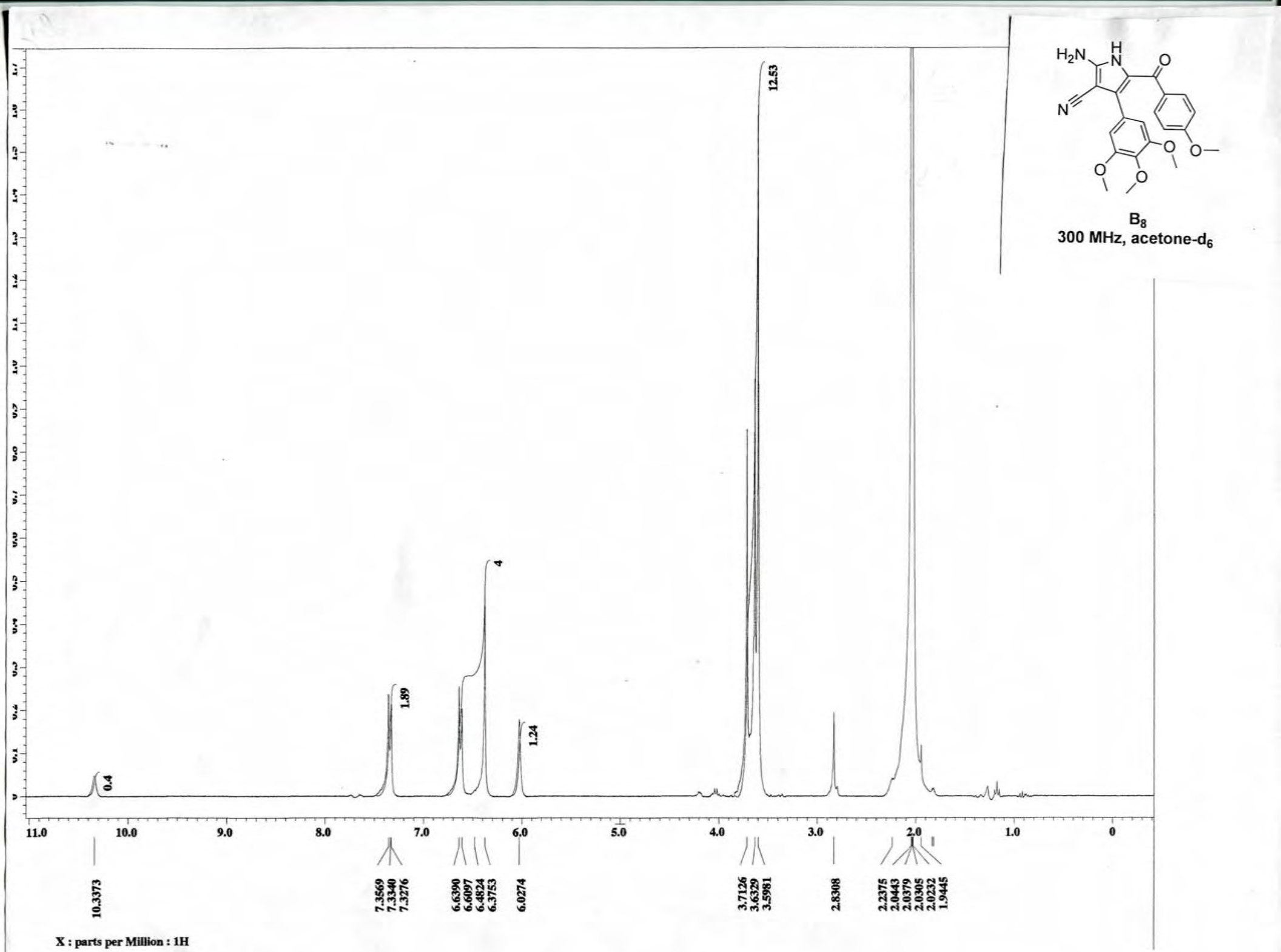


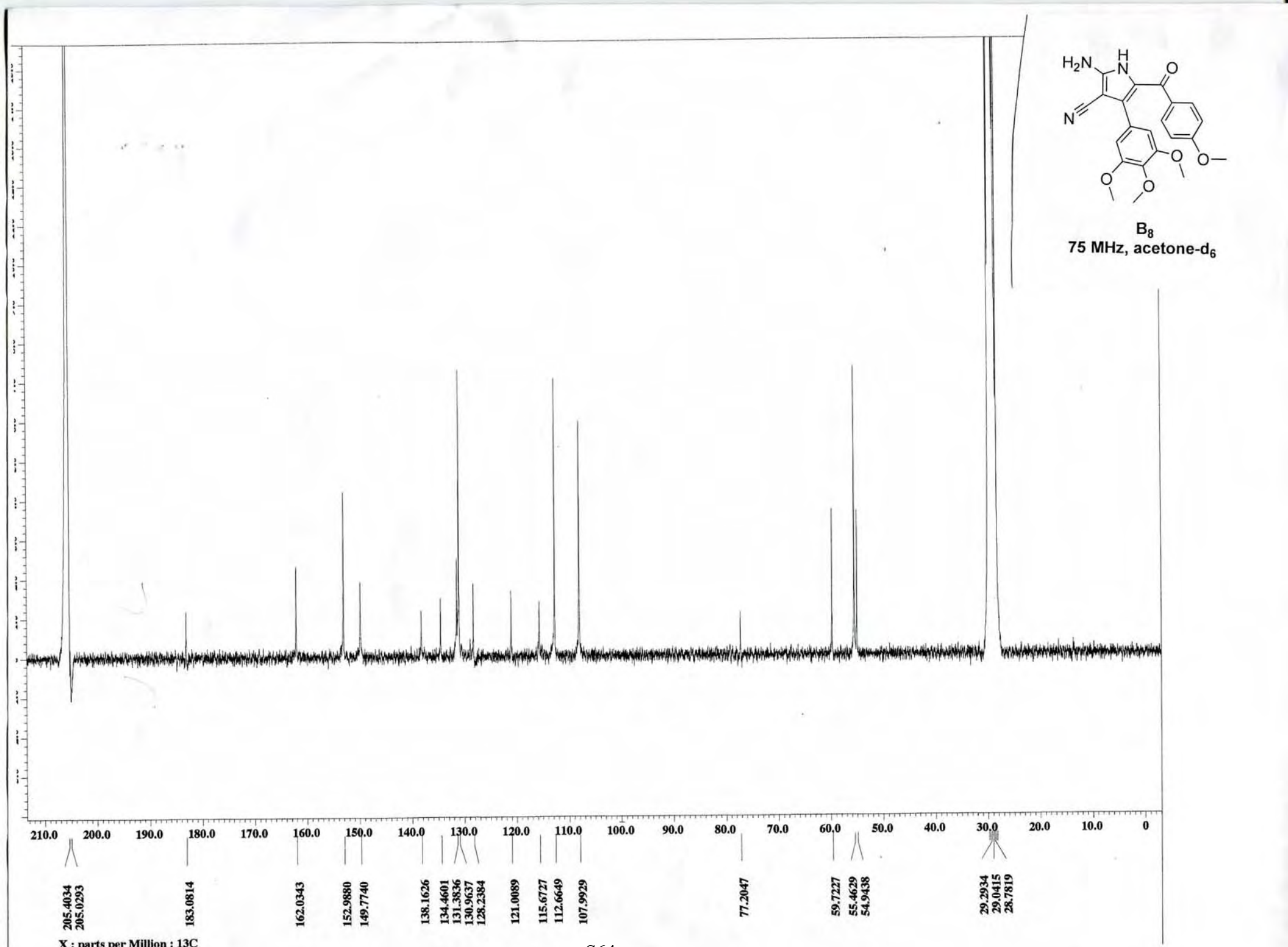
JEOL

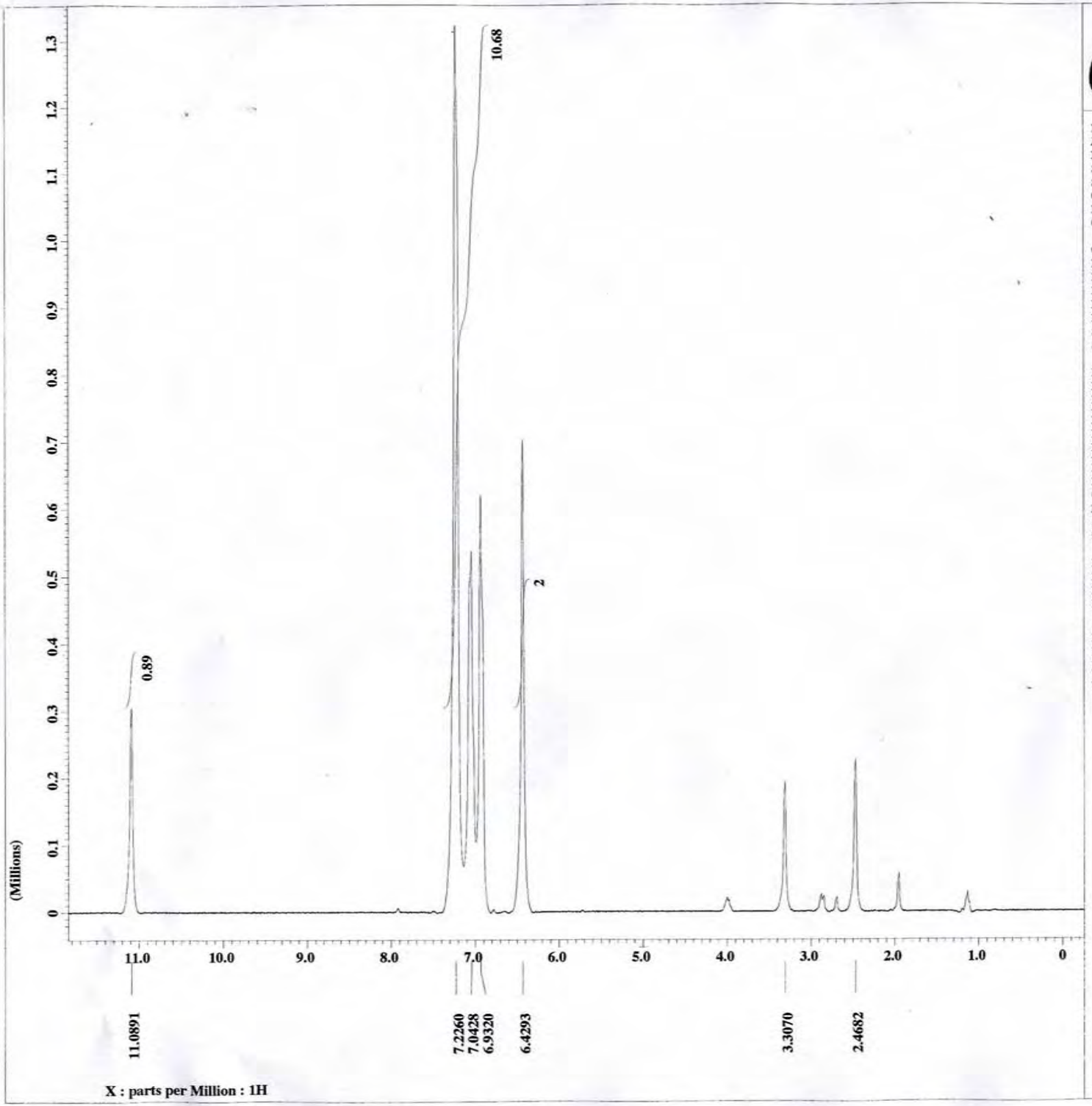
Filename	= 1d_13c_spectrum-116.j
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#626490
Solvent	= DMSO-D6
Creation_time	= 21-DEC-2010 18:02:25
Revision_time	= 21-DEC-2010 18:15:45
Current_time	= 21-DEC-2010 18:16:22
Comment	= Single Pulse with Bro
Data_format	= 1D REAL
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1000
Total_scans	= 1000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 25.1[dC]
Unblank_time	= 2[us]



B_7
75 MHz, DMSO-d_6

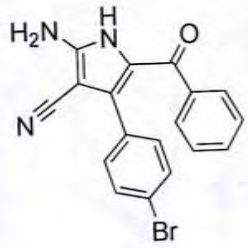

B8
300 MHz, acetone-d6
12.53
4
1.89
1.24
0.4
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.3373
7.3569
7.3340
7.3276
6.6390
6.6097
6.4824
6.3753
6.0274
3.7126
3.6329
3.5981
2.8308
2.2375
2.0443
2.0379
2.0305
2.0232
1.9445
X : parts per Million : 1H


B8
75 MHz, acetone-d6
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
205.4034
205.0293
183.0814
162.0343
152.9880
149.7740
138.1626
134.4601
131.3836
130.9637
128.2384
121.0089
115.6727
112.6649
107.9929
77.2047
59.7227
55.4629
54.9438
29.2934
29.0415
28.7819
X : parts per Million : 13C

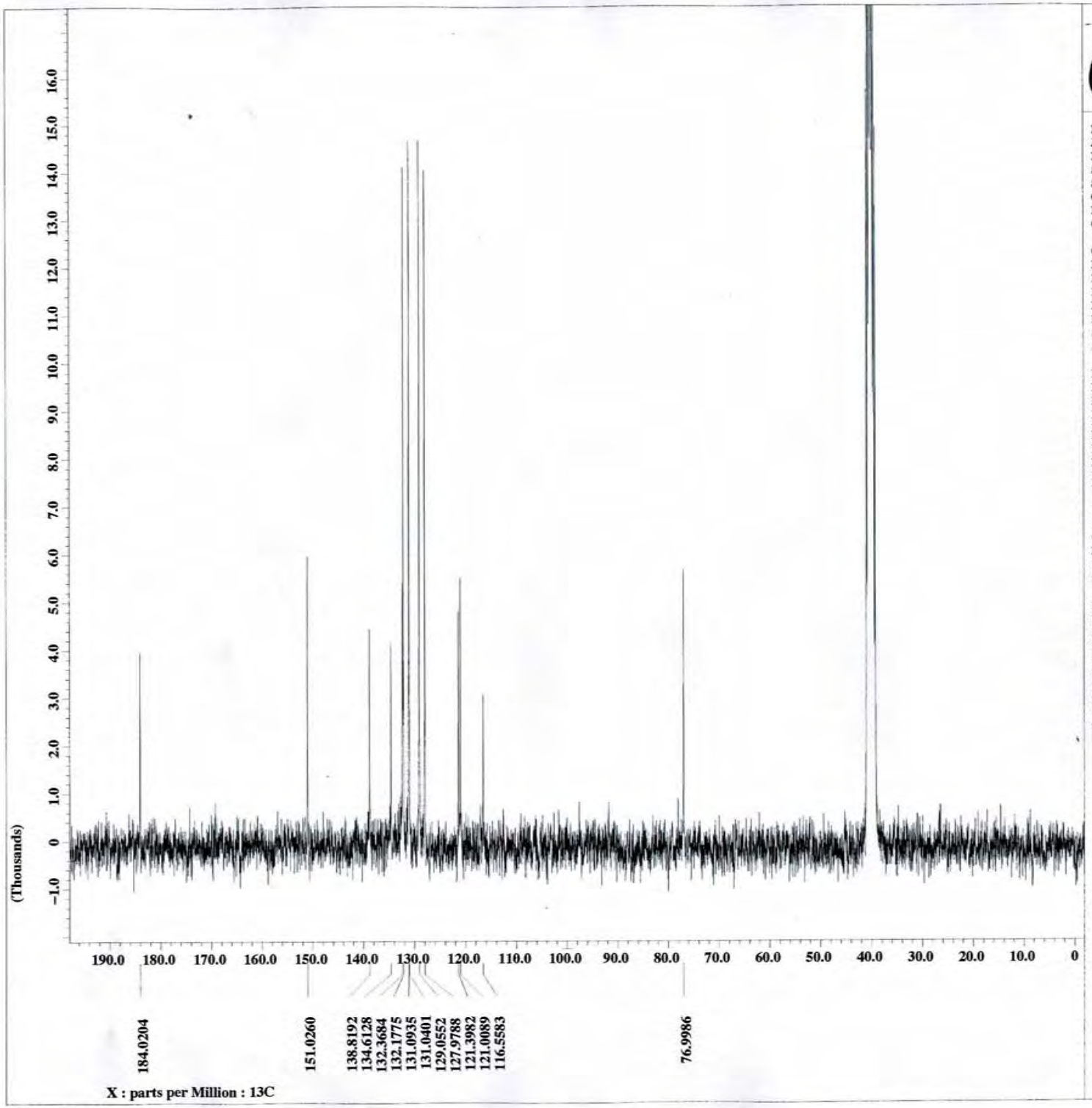


JEOL

```
Filename          = 1d_spectrum-85.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#433379
Solvent           = DMSO-D6
Creation_time     =  7-DEC-2010 11:55:50
Revision_time     =  7-DEC-2010 12:18:24
Current_time      =  7-DEC-2010 12:18:58

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23.7[dC]
Unblank_time      = 2[us]
```

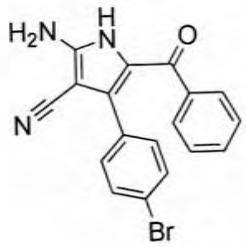


B_9
300 MHz, DMSO-d_6

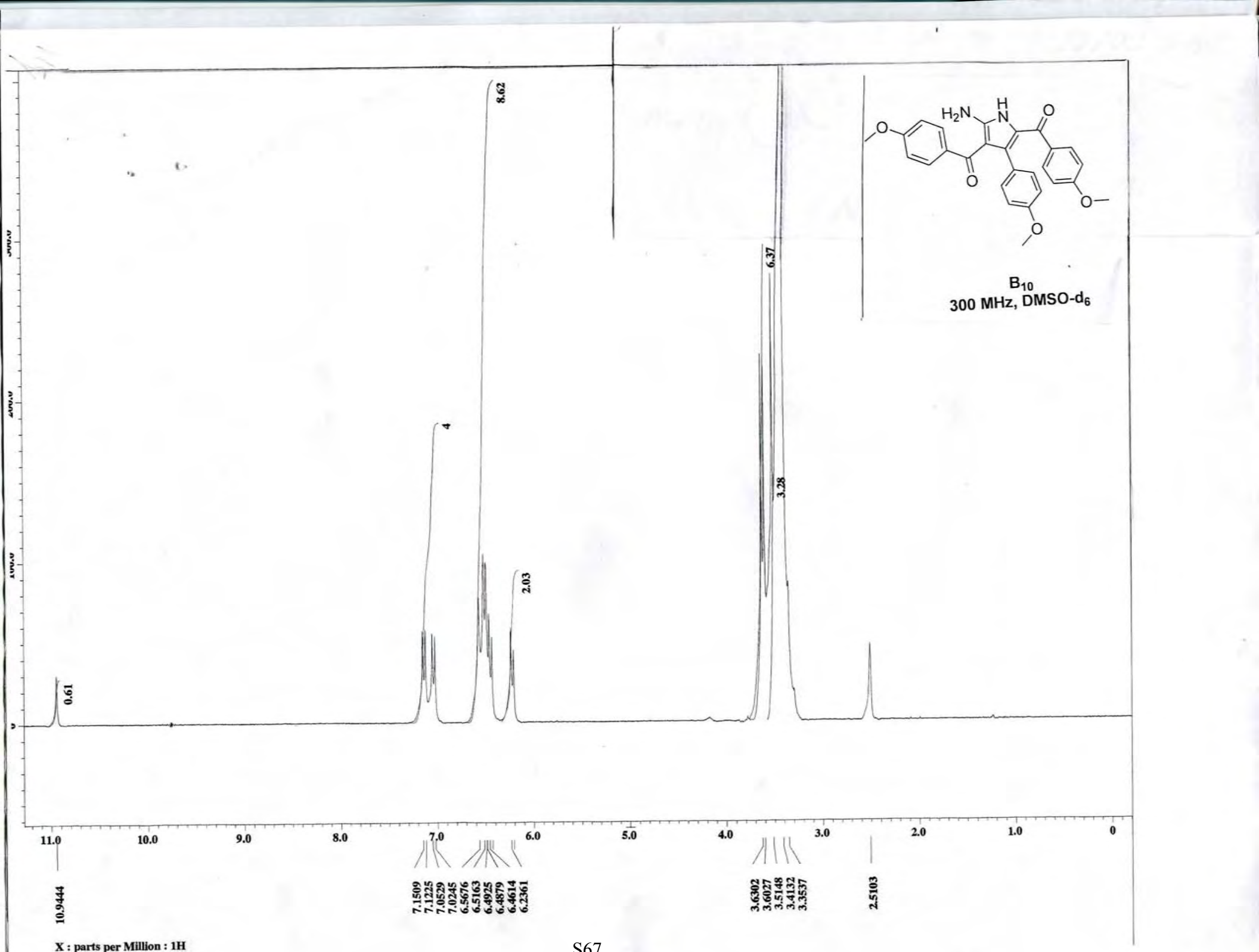


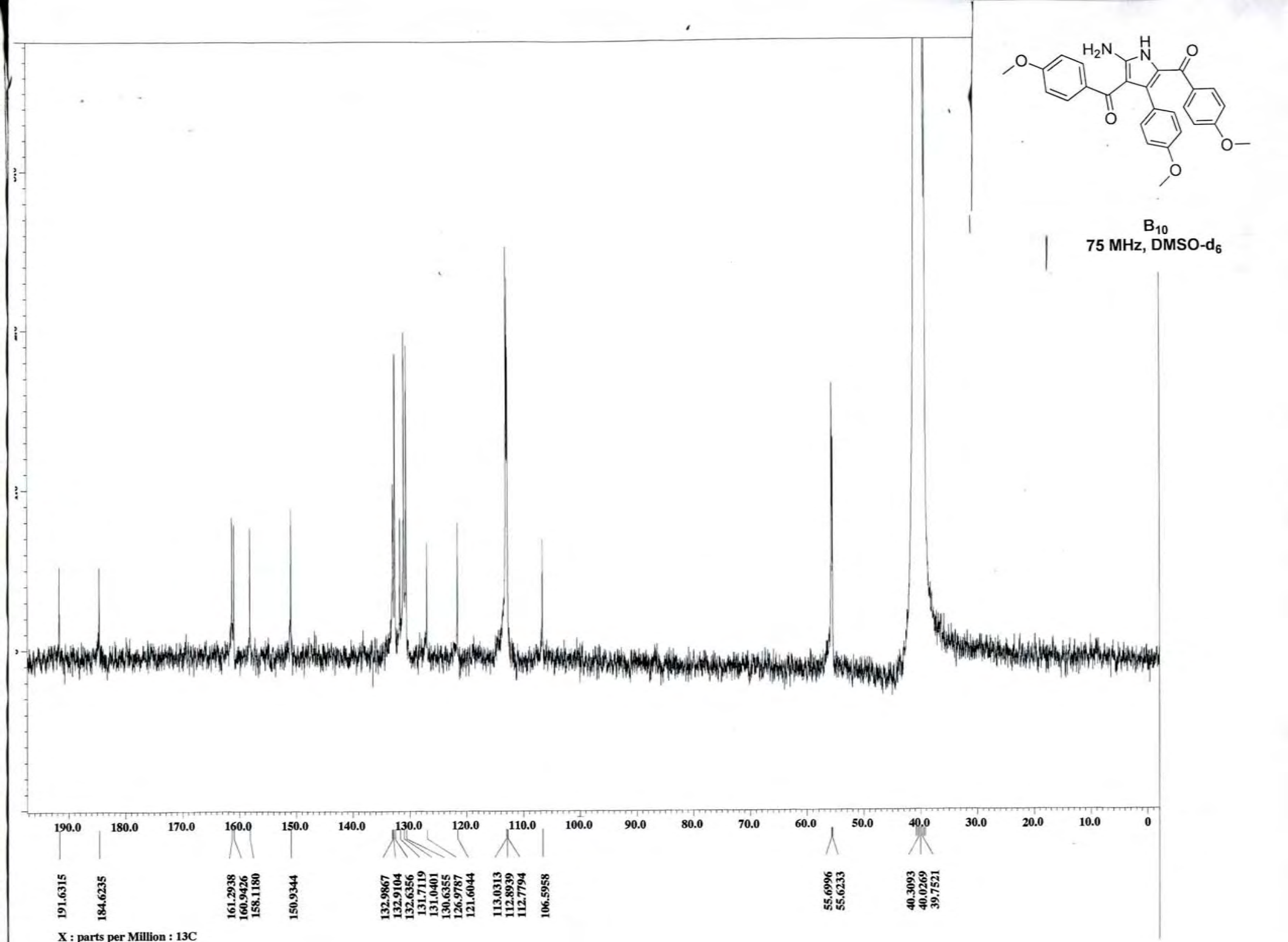
JEOL

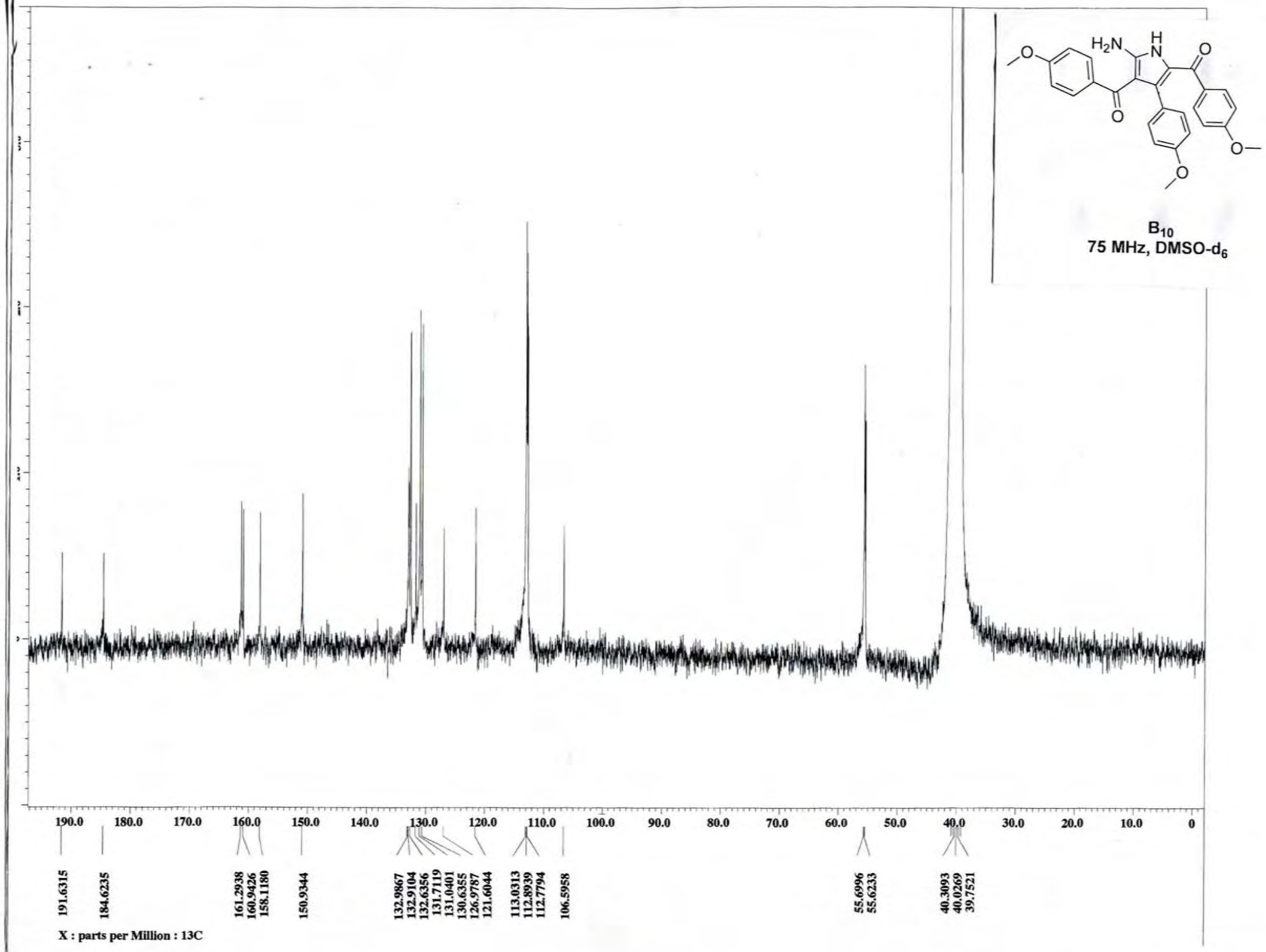
```
Filename          = 1d_13c_spectrum-53.jd
Author            = alex
Experiment        = single_pulse_dec
Sample_id         = S#434576
Solvent           = DMSO-D6
Creation_time     =  7-DEC-2010 13:09:54
Revision_time     =  7-DEC-2010 16:41:26
Current_time      =  7-DEC-2010 16:41:37

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1600
Total_scans       = 1600

X_90_width        = 8.1[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 2.7[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 1[s]
Temp_get          = 24.9[dC]
Unblank_time      = 2[us]
```

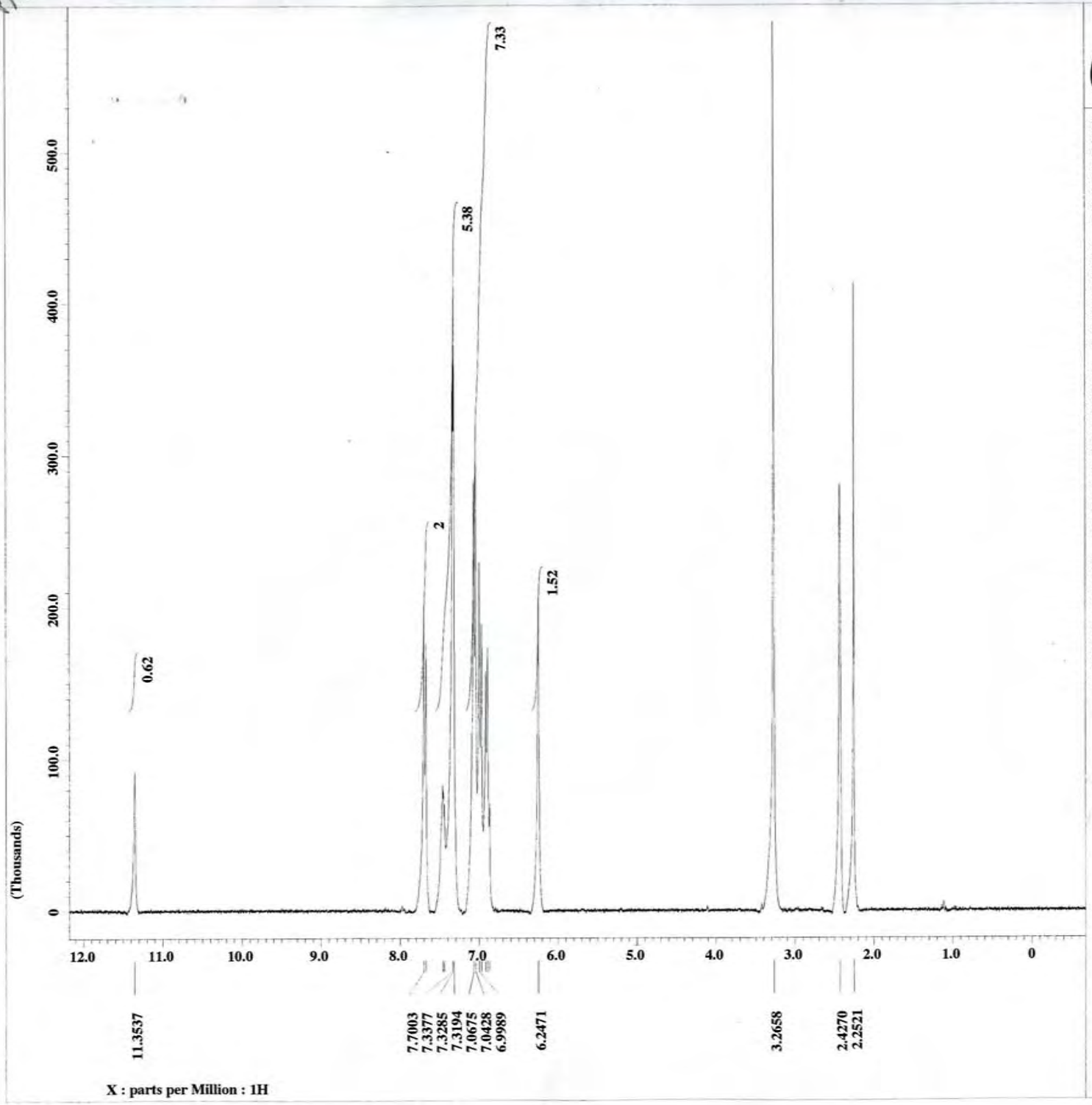


B_9
75 MHz, DMSO-d_6


8.62
6.37
4
3.28
2.03
0.61
B10
300 MHz, DMSO-d6
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.9444
7.1509
7.1225
7.0529
7.0245
6.5676
6.5163
6.4925
6.4879
6.4614
6.2361
3.6302
3.6027
3.5148
3.4132
3.3537
2.5103
X : parts per Million : 1H

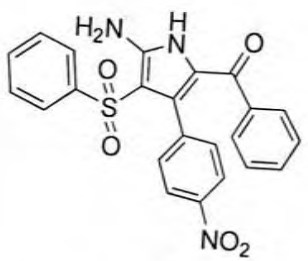

B10
75 MHz, DMSO-d6
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
191.6315
184.6235
161.2938
160.9426
158.1180
150.9344
132.9867
132.9104
132.6356
131.7119
131.0401
130.6355
126.9787
121.6044
113.0313
112.8939
112.7794
106.5958
55.6996
55.6233
40.3093
40.0269
39.7521
X : parts per Million : 13C


B10
75 MHz, DMSO-d6
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
191.6315
184.6235
161.2938
160.9426
158.1180
150.9344
132.9867
132.9104
132.6356
131.7119
131.0401
130.6355
126.9787
121.6044
113.0313
112.8939
112.7794
106.5958
55.6996
55.6233
40.3093
40.0269
39.7521
X : parts per Million : 13C

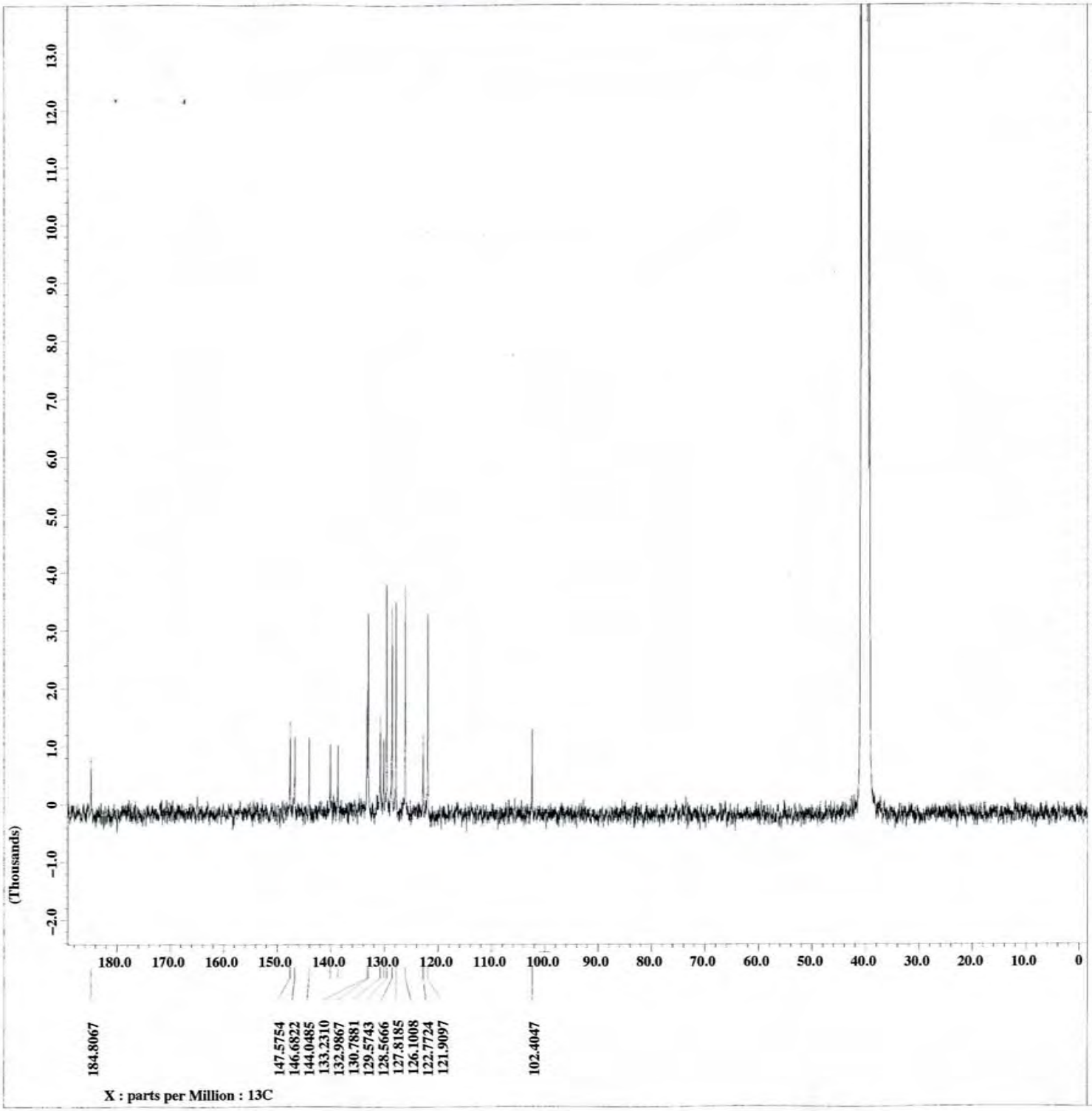


JEOL

Filename	= 1d_spectrum-132.jdf
Author	= alex
Experiment	= single_pulse.exp
Sample_id	= S#674454
Solvent	= DMSO-D6
Creation_time	= 9-DEC-2010 18:37:37
Revision_time	= 9-DEC-2010 18:57:36
Current_time	= 9-DEC-2010 18:57:42
Comment	= Single Pulse Experime
Data_format	= 1D REAL
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 16[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 8[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 4[s]
Temp_get	= 23.3[dC]
Unblank_time	= 2[us]

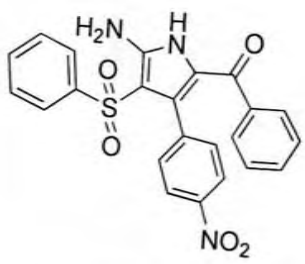


B_{11}
300 MHz, DMSO-d_6

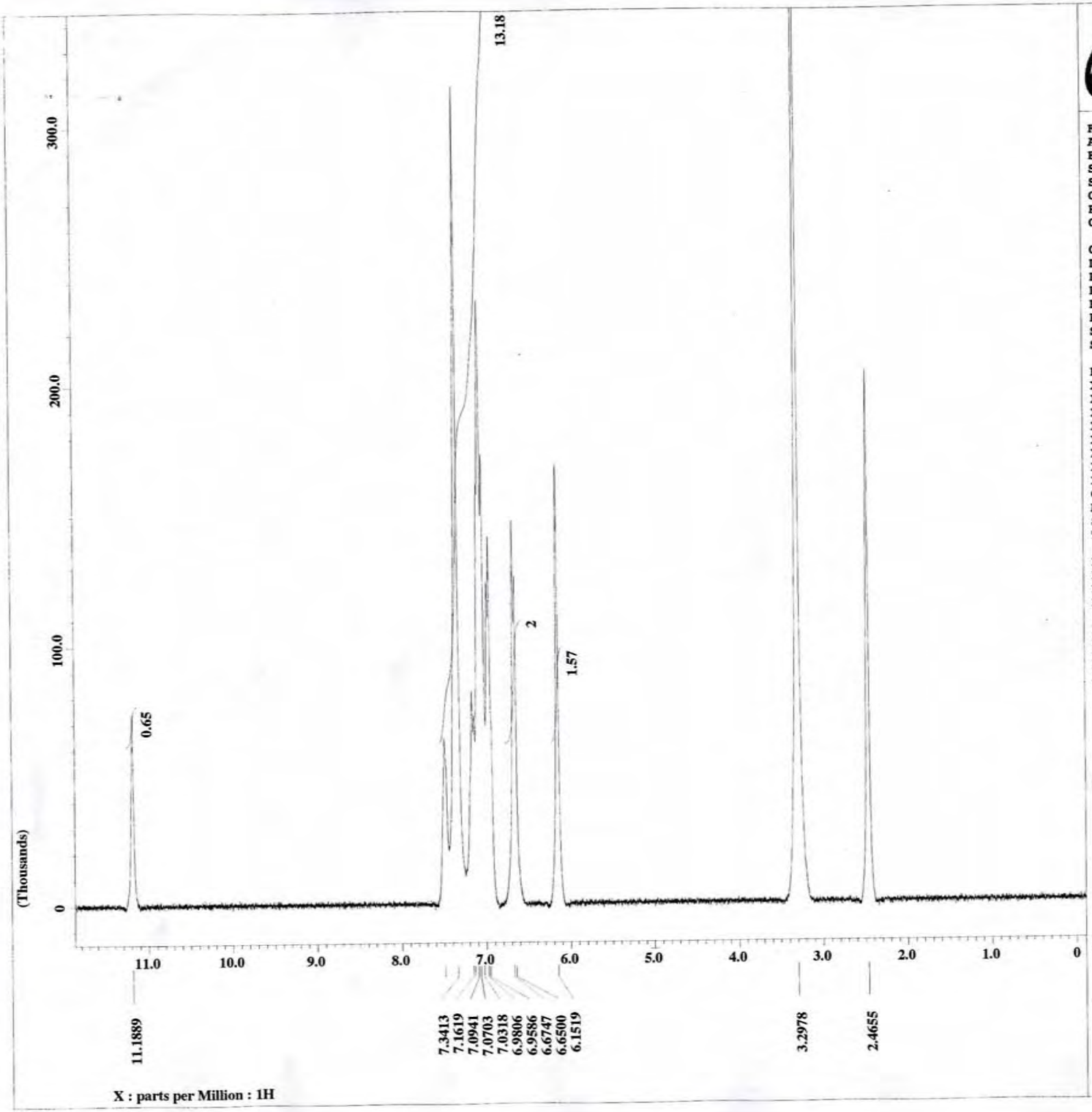


JEOL

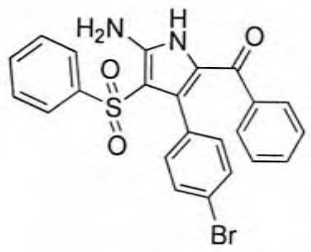
Filename	= 1d_13c_spectrum-74.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#709603
Solvent	= DMSO-D6
Creation_time	= 10-DEC-2010 07:44:17
Revision_time	= 10-DEC-2010 09:36:55
Current_time	= 10-DEC-2010 09:37:11
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 16000
Total_scans	= 16000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 21.9[dC]
Unblank_time	= 2[us]



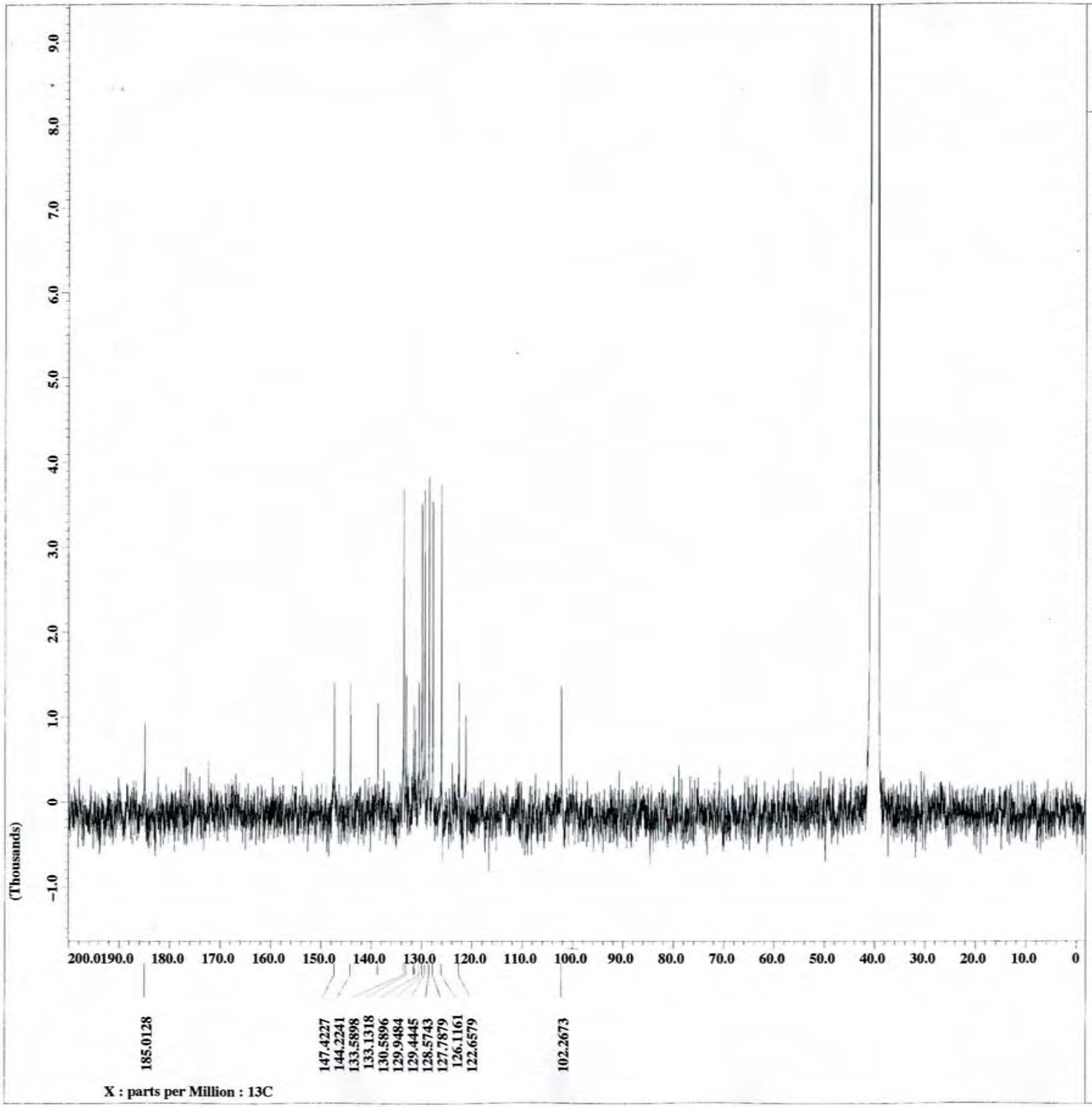
B_{11}
75 MHz, DMSO-d_6




```
Filename          = 1d_spectrum-188.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#431531
Solvent           = DMSO-D6
Creation_time     = 15-DEC-2010 11:52:44
Revision_time     = 15-DEC-2010 15:23:10
Current_time      = 15-DEC-2010 15:23:25

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23[dC]
Unblank_time      = 2[us]
```

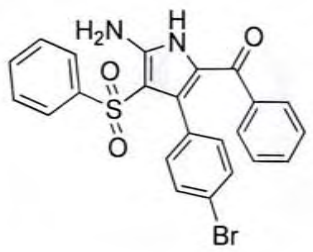


B_{12}
300 MHz, DMSO-d_6

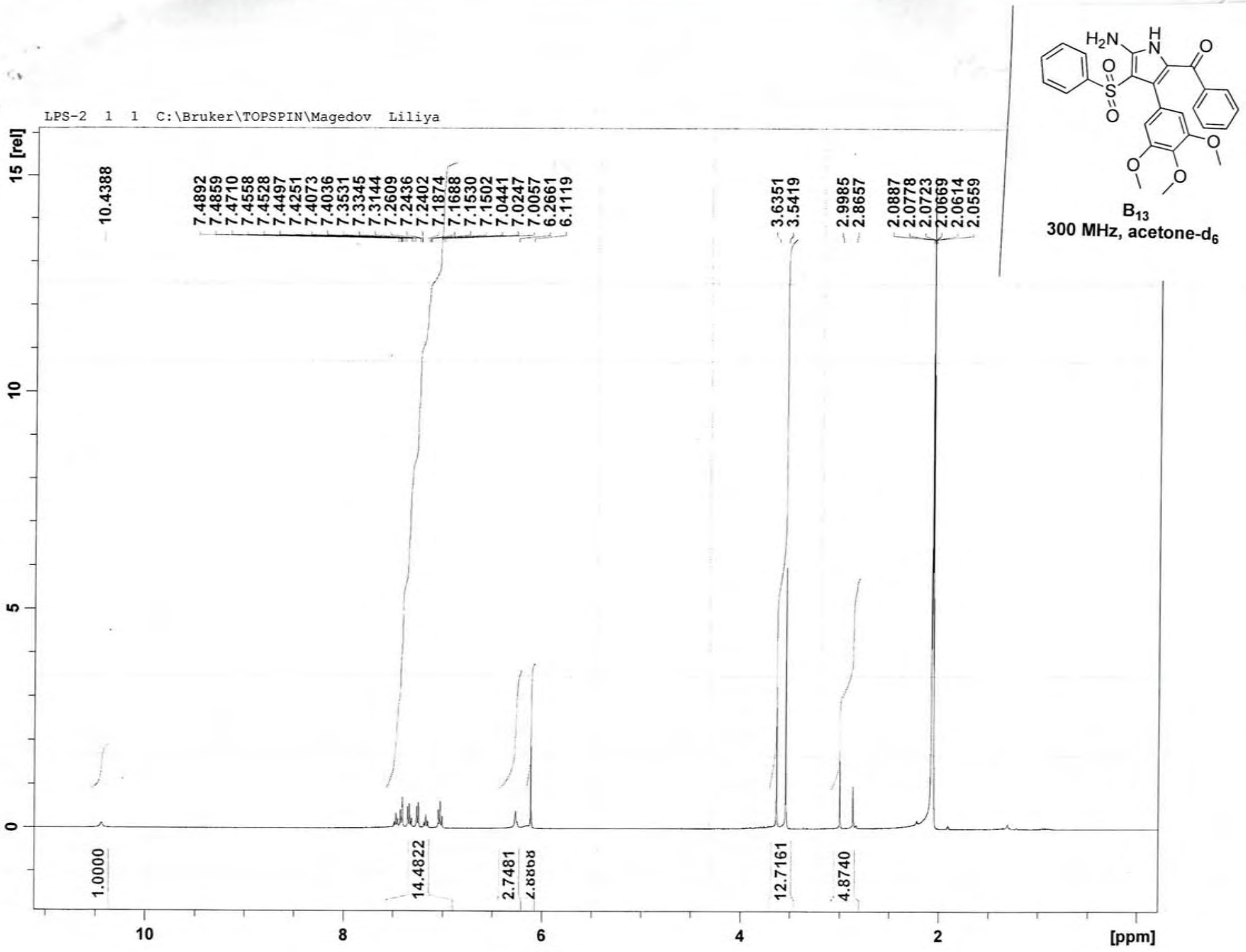


JEOL

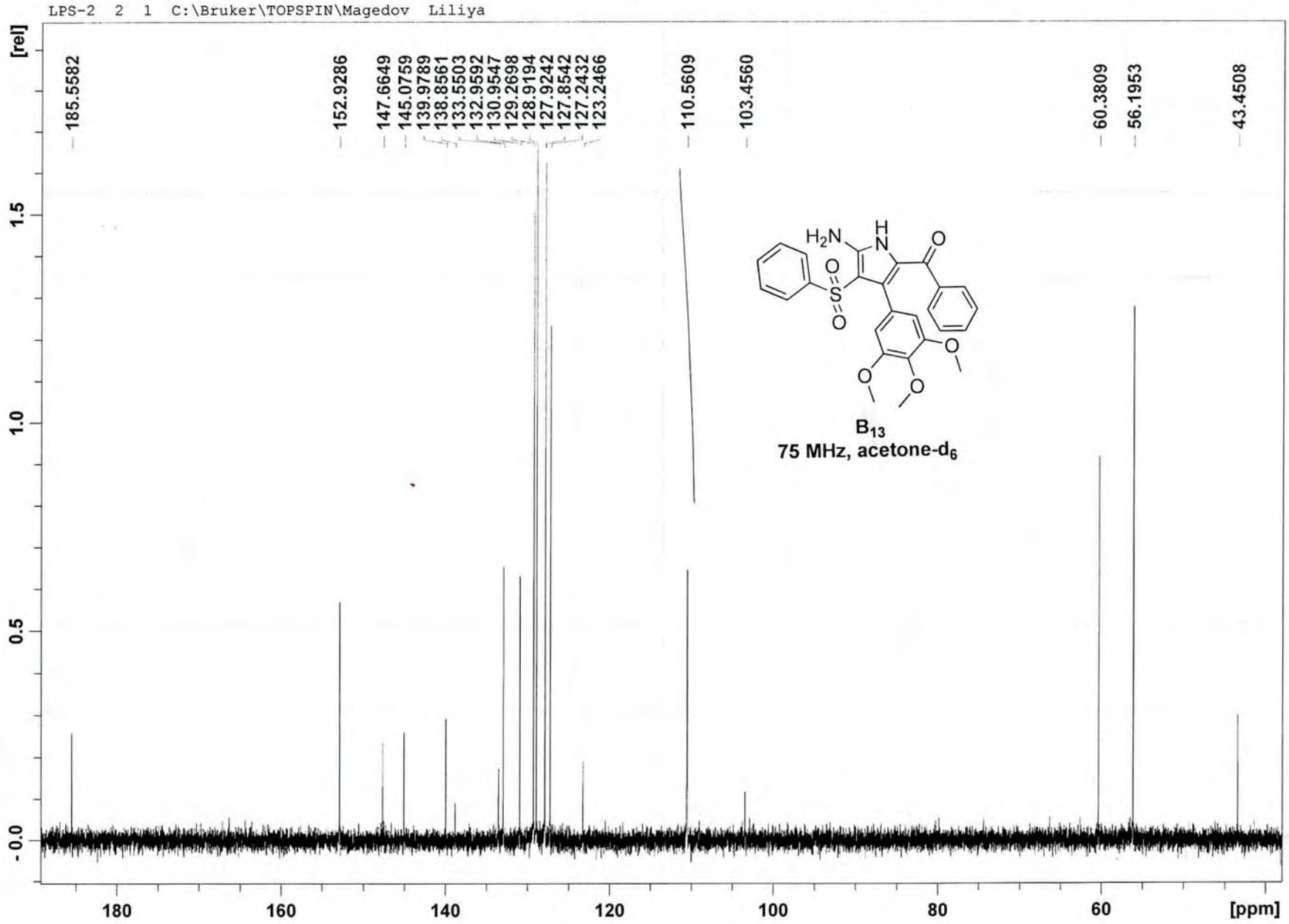
Filename	= 1d_13c_spectrum-99.jd
Author	= alex
Experiment	= single_pulse_dec
Sample_id	= S#434222
Solvent	= DMSO-D6
Creation_time	= 15-DEC-2010 14:58:38
Revision_time	= 15-DEC-2010 15:19:37
Current_time	= 15-DEC-2010 15:19:42
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 4000
Total_scans	= 4000
X_90_width	= 8.1[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 2.7[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 15
Relaxation_delay	= 1[s]
Temp_get	= 24.4[dC]
Unblank_time	= 2[us]

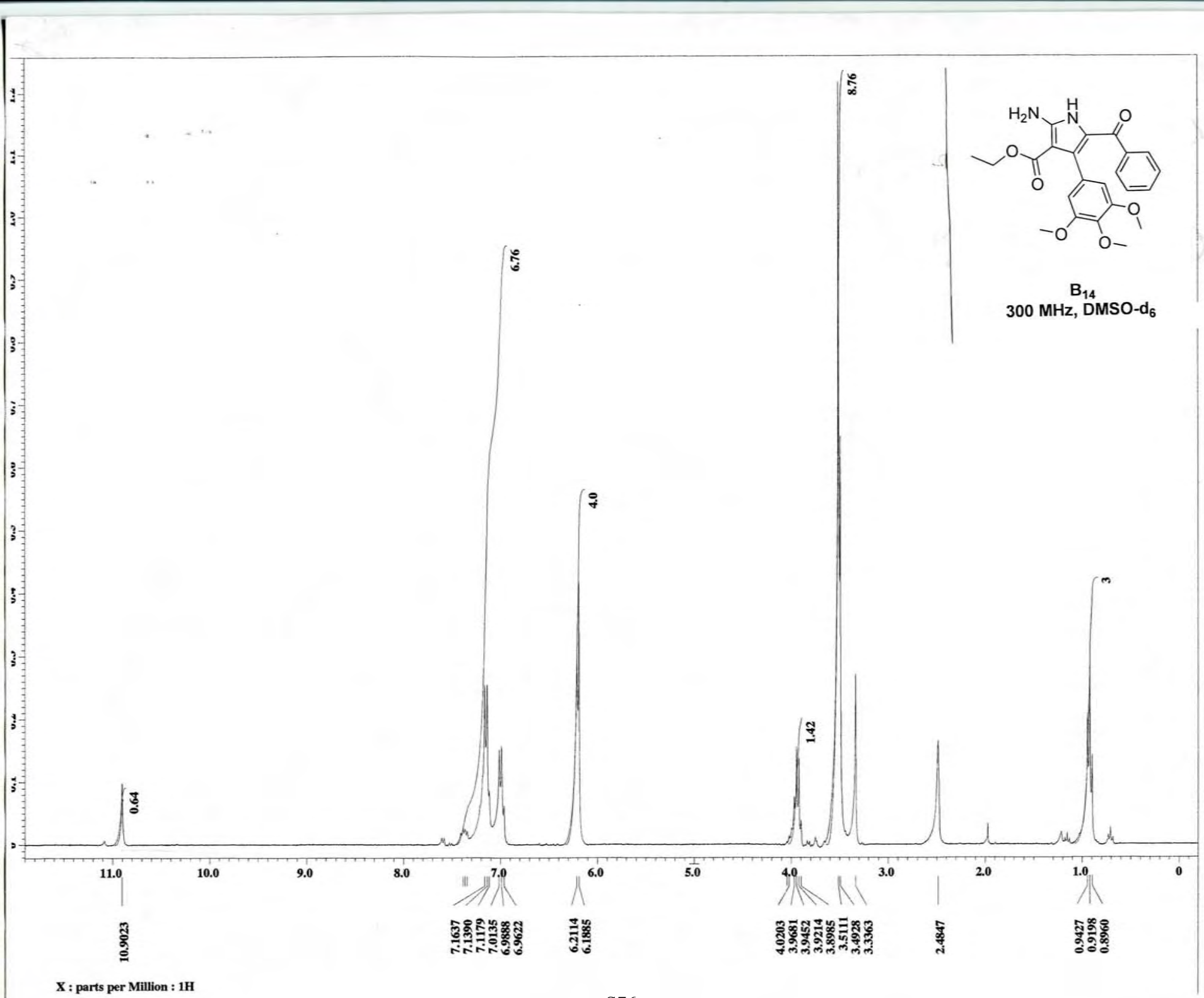


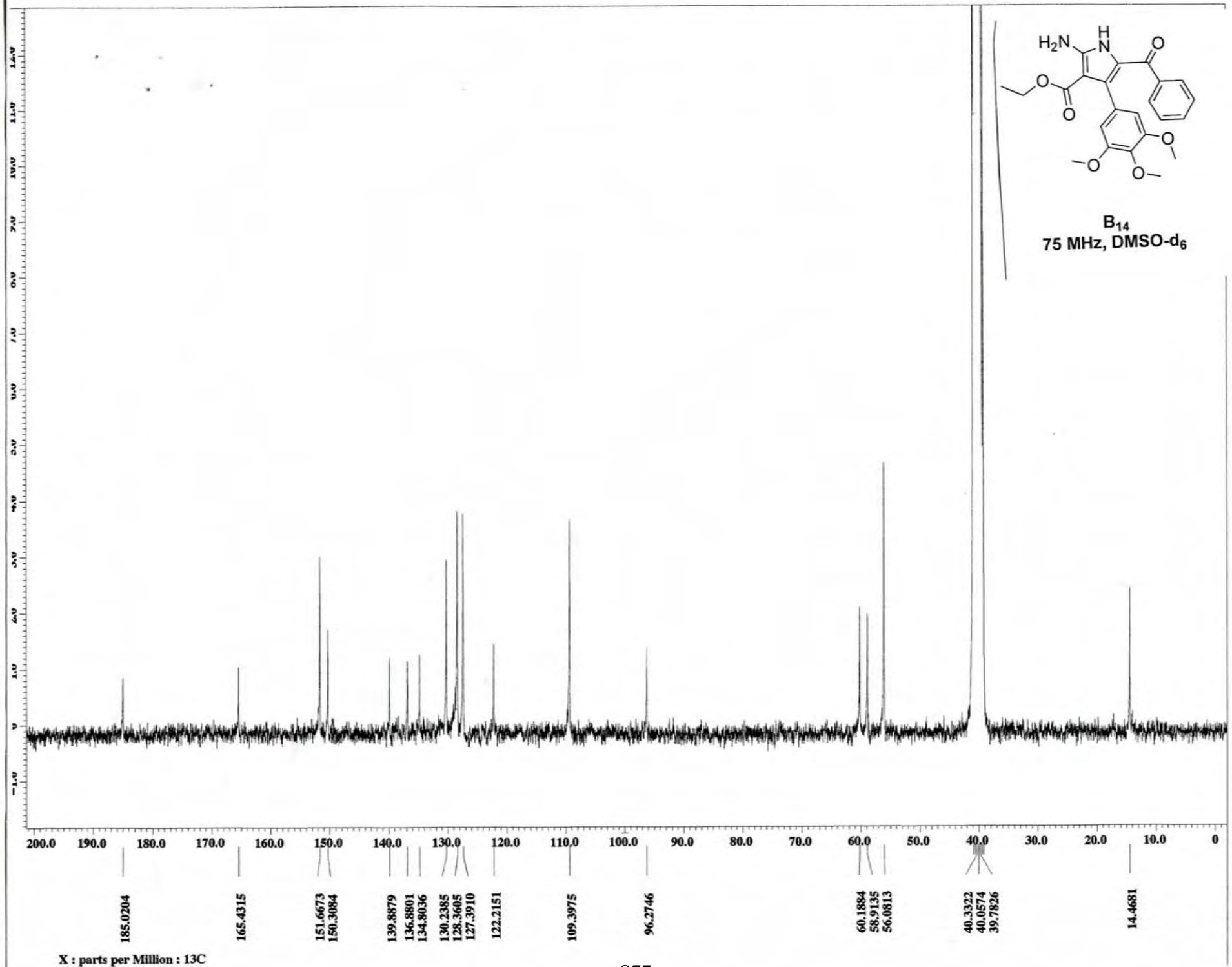
B_{12}
75 MHz, DMSO-d_6

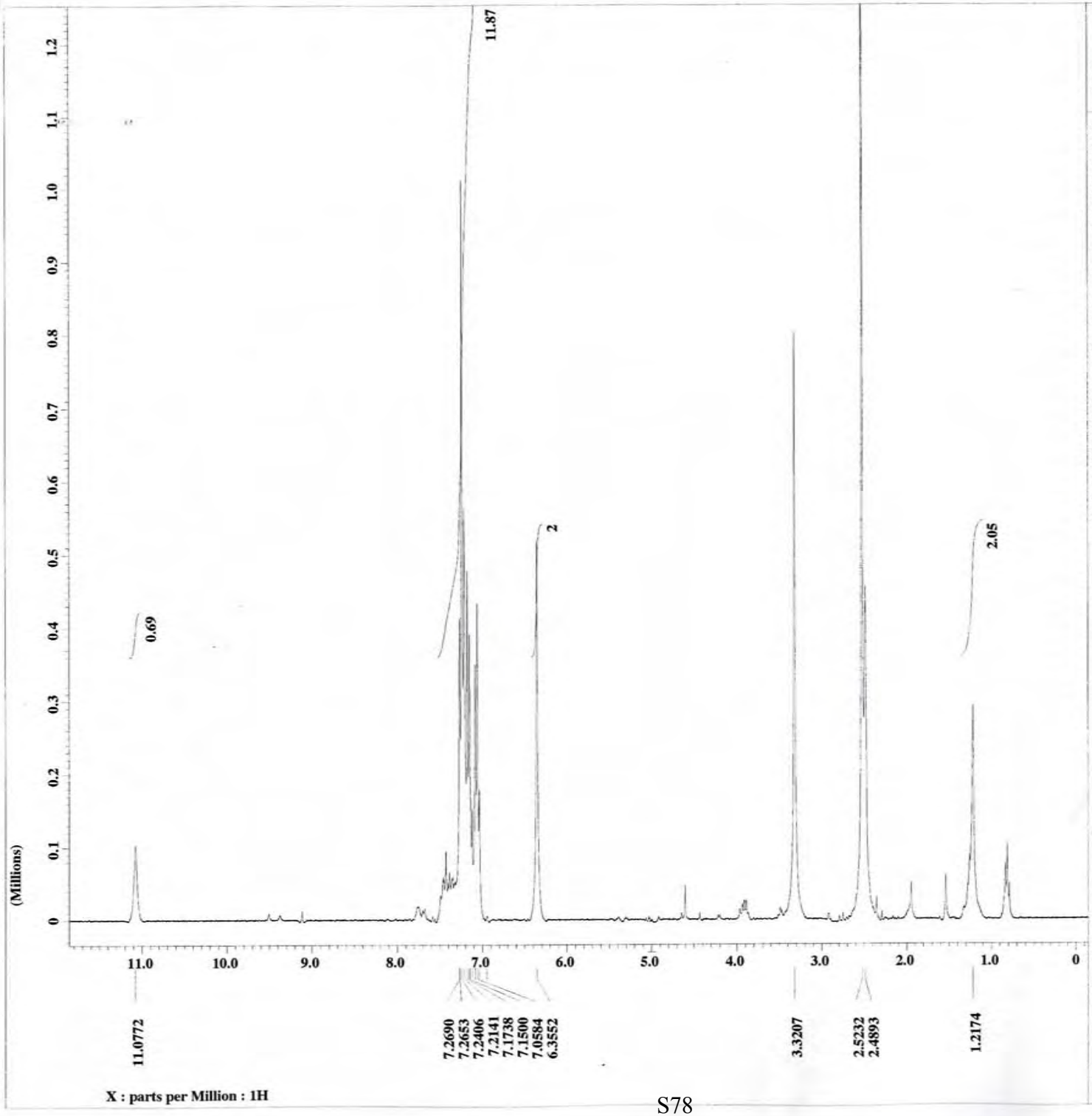


B_{13}
300 MHz, acetone-d_6


LPS-2 2 1 C:\Bruker\TOPSPIN\Magedov Liliya
[rel]
1.5
1.0
0.5
- 0.0
185.5582
152.9286
147.6649
145.0759
139.9789
138.8561
133.5503
132.9592
130.9547
129.2698
128.9194
127.9242
127.8542
127.2432
123.2466
110.5609
103.4560
60.3809
56.1953
43.4508
180
160
140
120
100
80
60
[ppm]
B13
75 MHz, acetone-d6

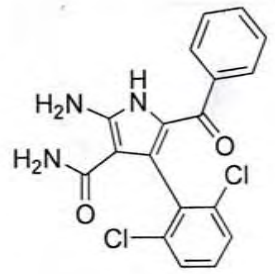

8.76
6.76
4.0
1.42
3
0.64
B14
300 MHz, DMSO-d6
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.9023
7.1637
7.1390
7.1179
7.0135
6.9888
6.9622
6.2114
6.1885
4.0203
3.9681
3.9452
3.9214
3.8985
3.5111
3.4928
3.3363
2.4847
0.9427
0.9198
0.8960
X : parts per Million : 1H


B14
75 MHz, DMSO-d6
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
185.0204
165.4315
151.6673
150.3084
139.8879
136.8801
134.8036
130.2385
128.3605
127.3910
122.2151
109.3975
96.2746
60.1884
58.9135
56.0813
40.3322
40.0574
39.7826
14.4681
X : parts per Million : 13C

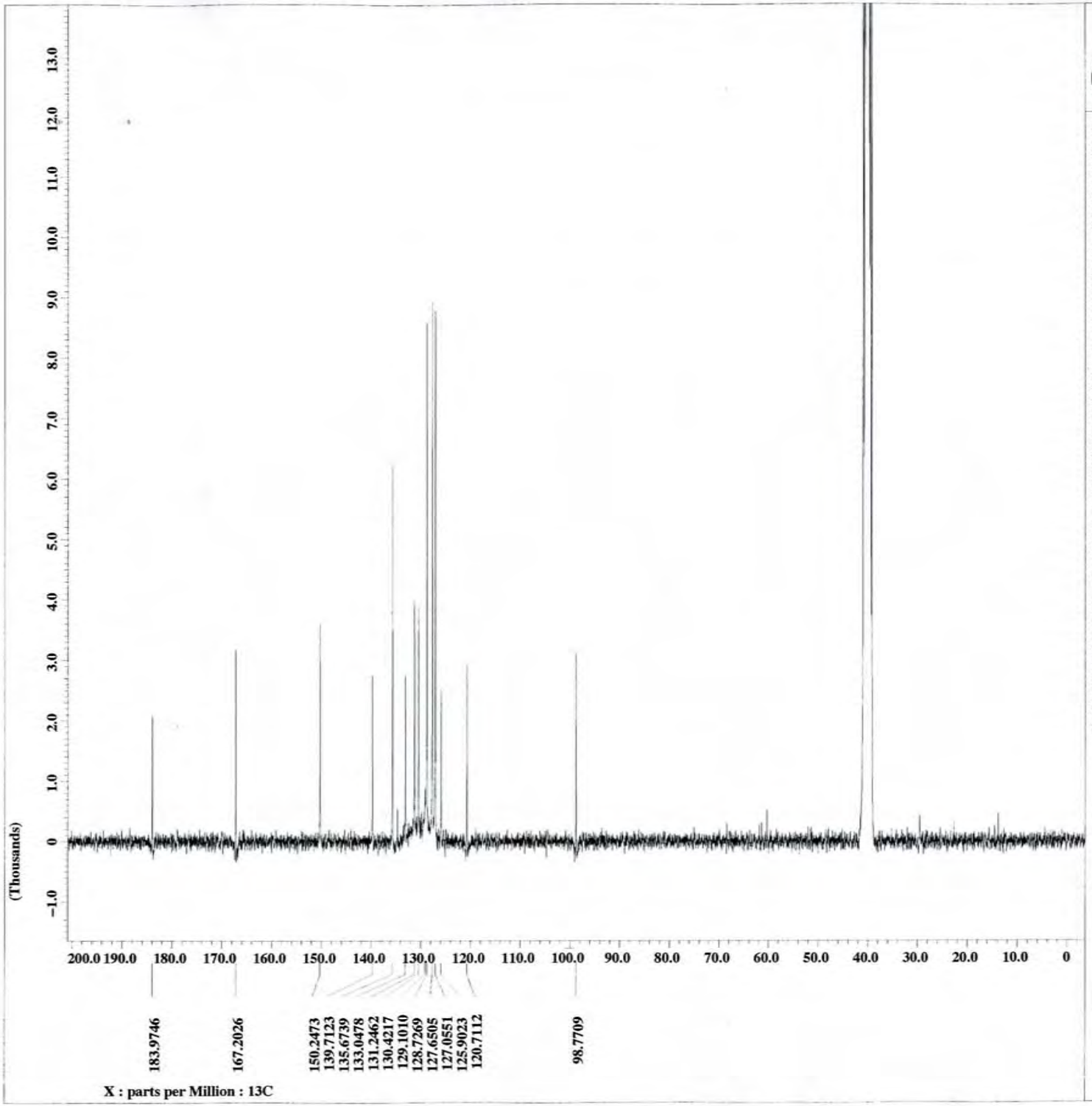


JEOL

```
Filename          = 1d_spectrum-168.jdf
Author            = alex
Experiment        = single_pulse.exp
Sample_id         = S#660649
Solvent           = DMSO-D6
Creation_time     = 13-DEC-2010 18:14:35
Revision_time     = 14-DEC-2010 10:24:48
Current_time      = 14-DEC-2010 10:24:55

Comment           = Single Pulse Experime
Data_format       = 1D REAL
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 16[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 8[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 4[s]
Temp_get          = 23.3[dC]
Unblank_time      = 2[us]
```

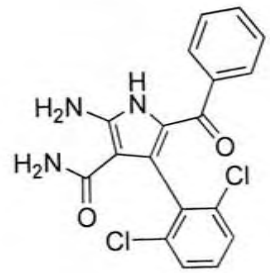


B_{15}
300 MHz, DMSO-d_6

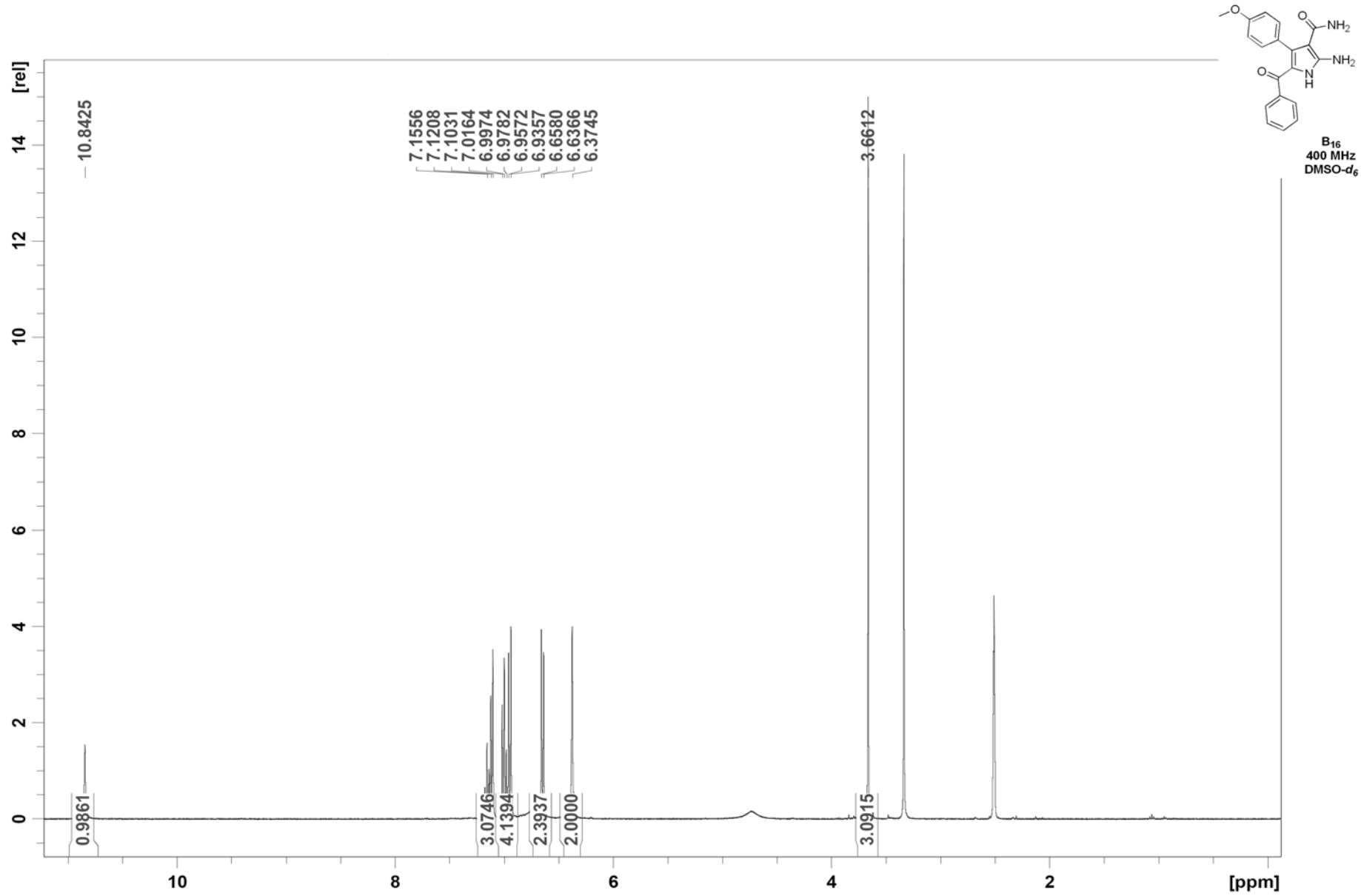


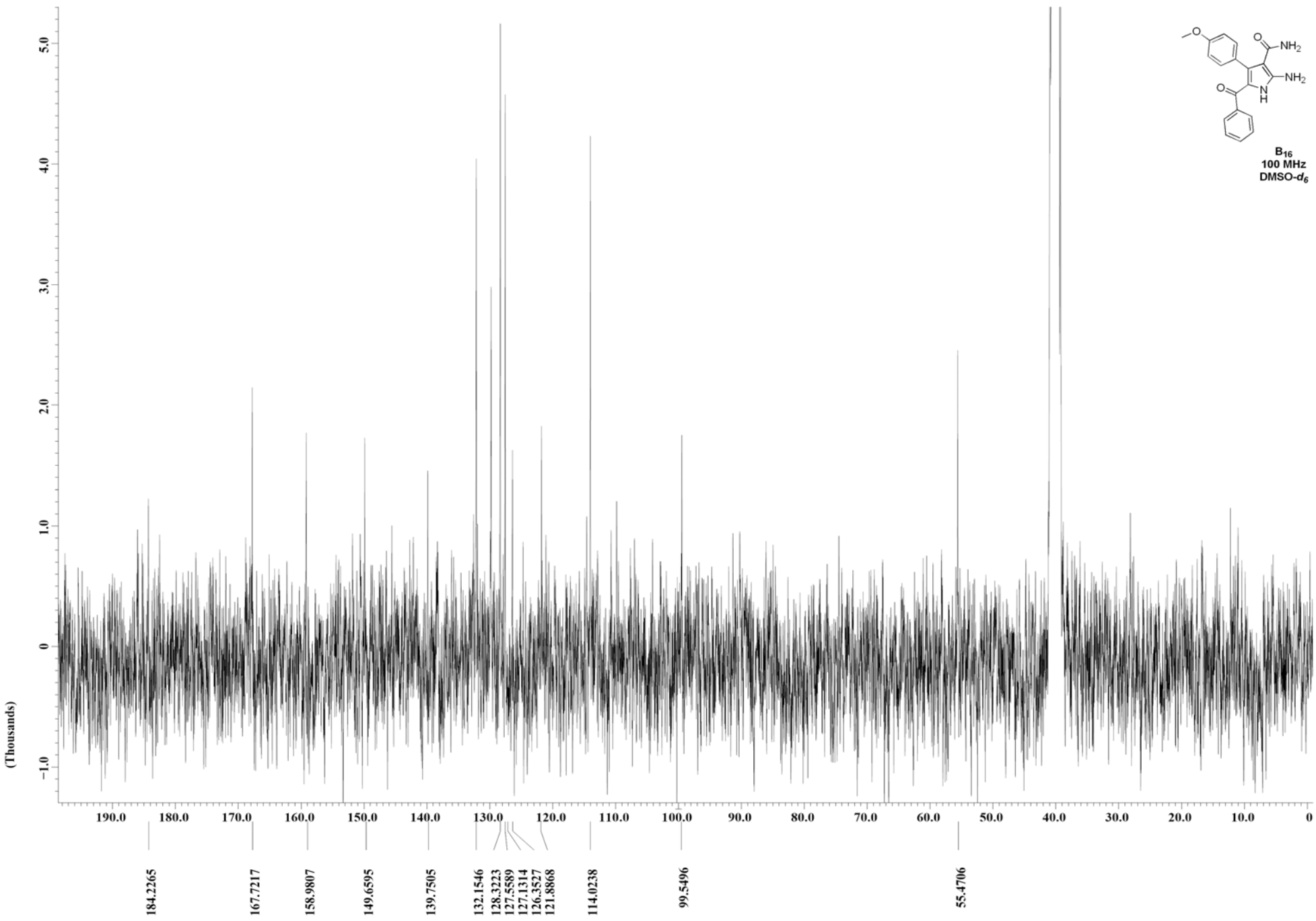
JEOL

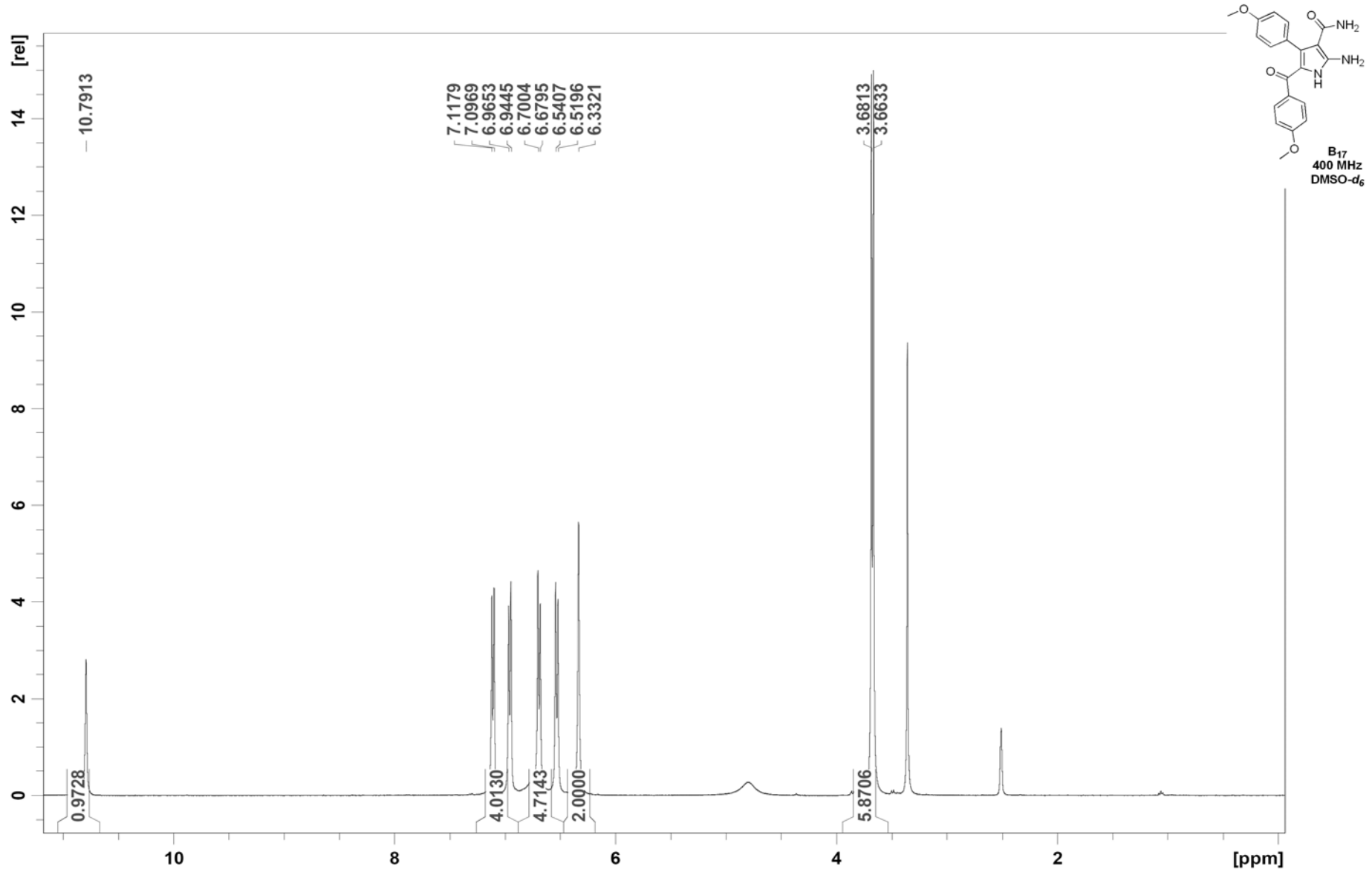
```
Filename          = 1d_13c_spectrum-95.jd
Author            = alex
Experiment        = single_pulse_dec
Sample_id         = S#663827
Solvent           = DMSO-D6
Creation_time     = 14-DEC-2010 09:30:13
Revision_time     = 14-DEC-2010 10:18:43
Current_time      = 14-DEC-2010 10:18:48

Comment           = Single Pulse with Bro
Data_format       = 1D REAL
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 20000
Total_scans       = 20000

X_90_width        = 8.1[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 2.7[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 15
Relaxation_delay  = 1[s]
Temp_get          = 22.8[dC]
Unblank_time      = 2[us]
```

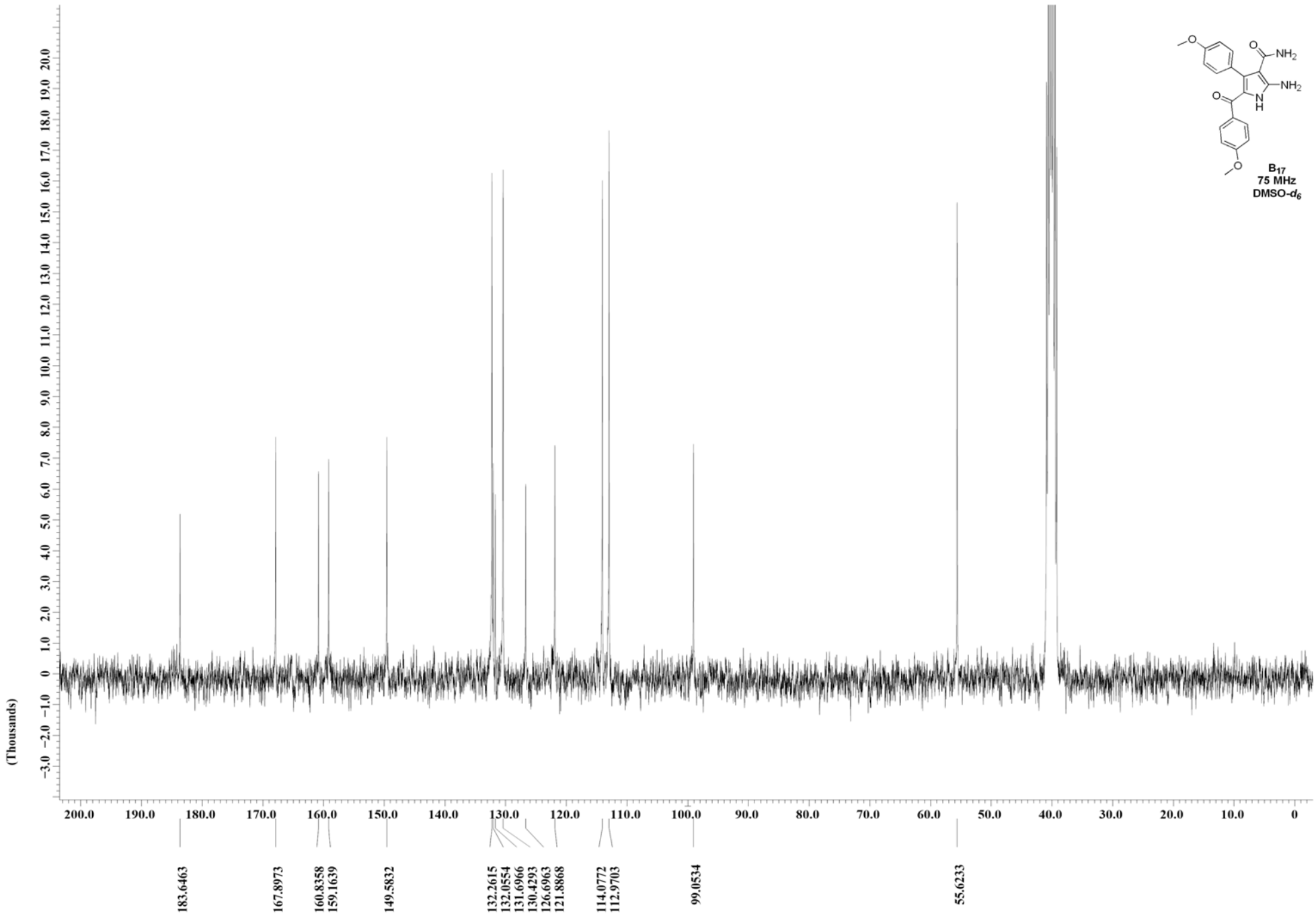


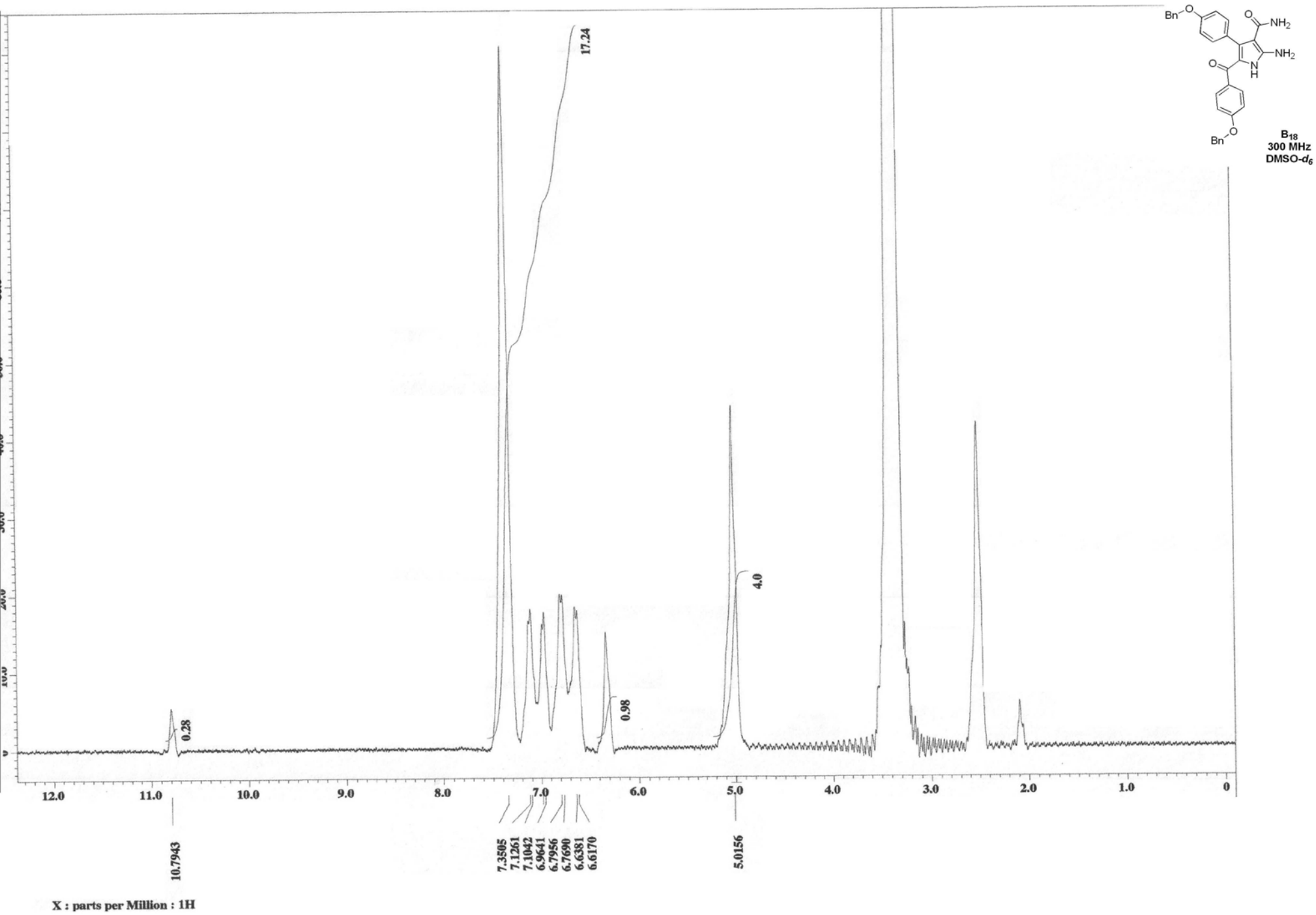
B_{15}
75 MHz, DMSO-d_6

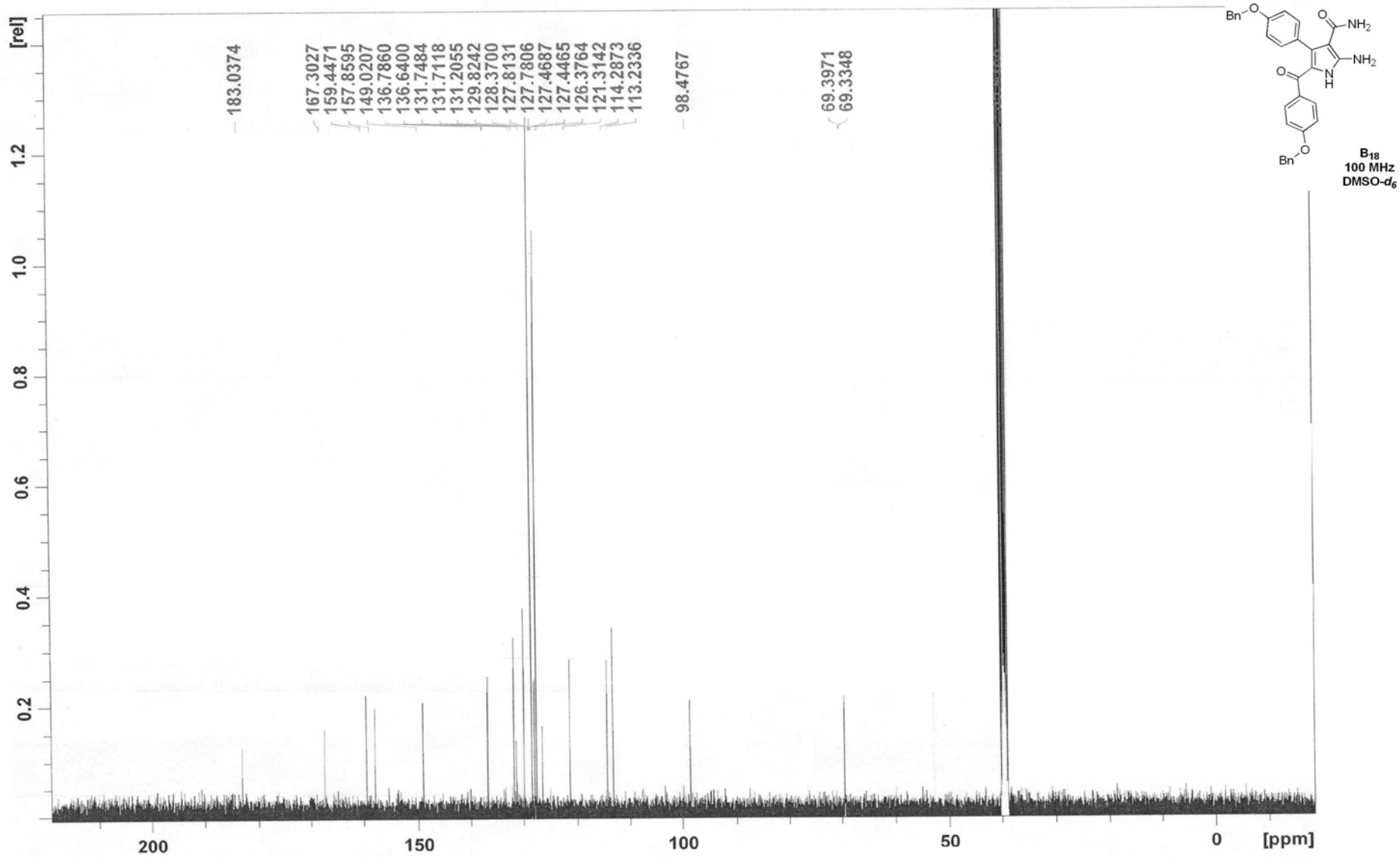

10.8425
7.1556
7.1208
7.1031
7.0164
6.9974
6.9782
6.9572
6.9357
6.6580
6.6366
6.3745
3.6612
0.9861
3.0746
4.1394
2.3937
2.0000
3.0915
[rel]
[ppm]
B16
400 MHz
DMSO-d6

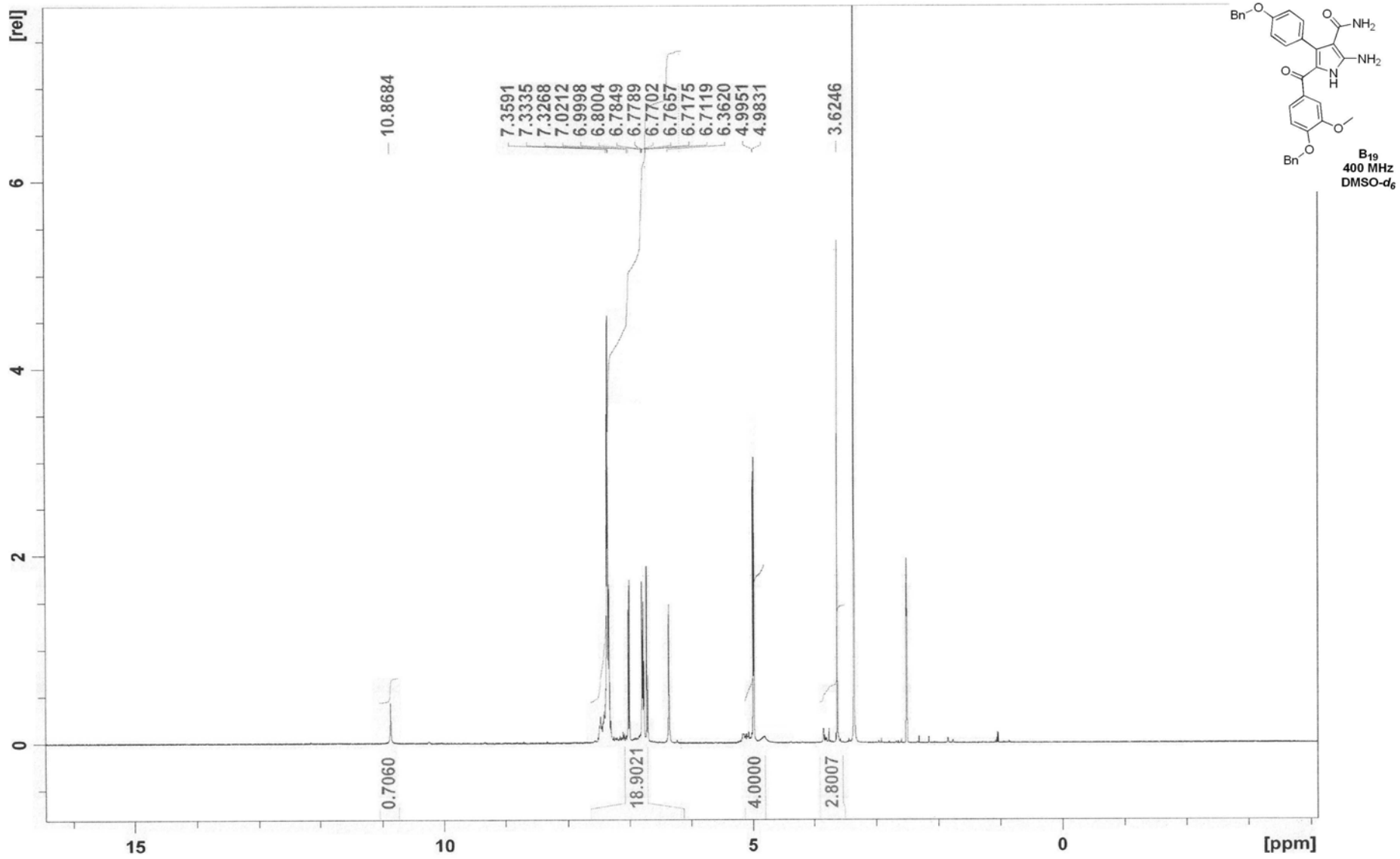

B16
100 MHz
DMSO-d6
(Thousands)
5.0
4.0
3.0
2.0
1.0
0
−1.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
184.2265
167.7217
158.9807
149.6595
139.7505
132.1546
128.3223
127.5589
127.1314
126.3527
121.8868
114.0238
99.5496
55.4706
X : parts per Million : 13C

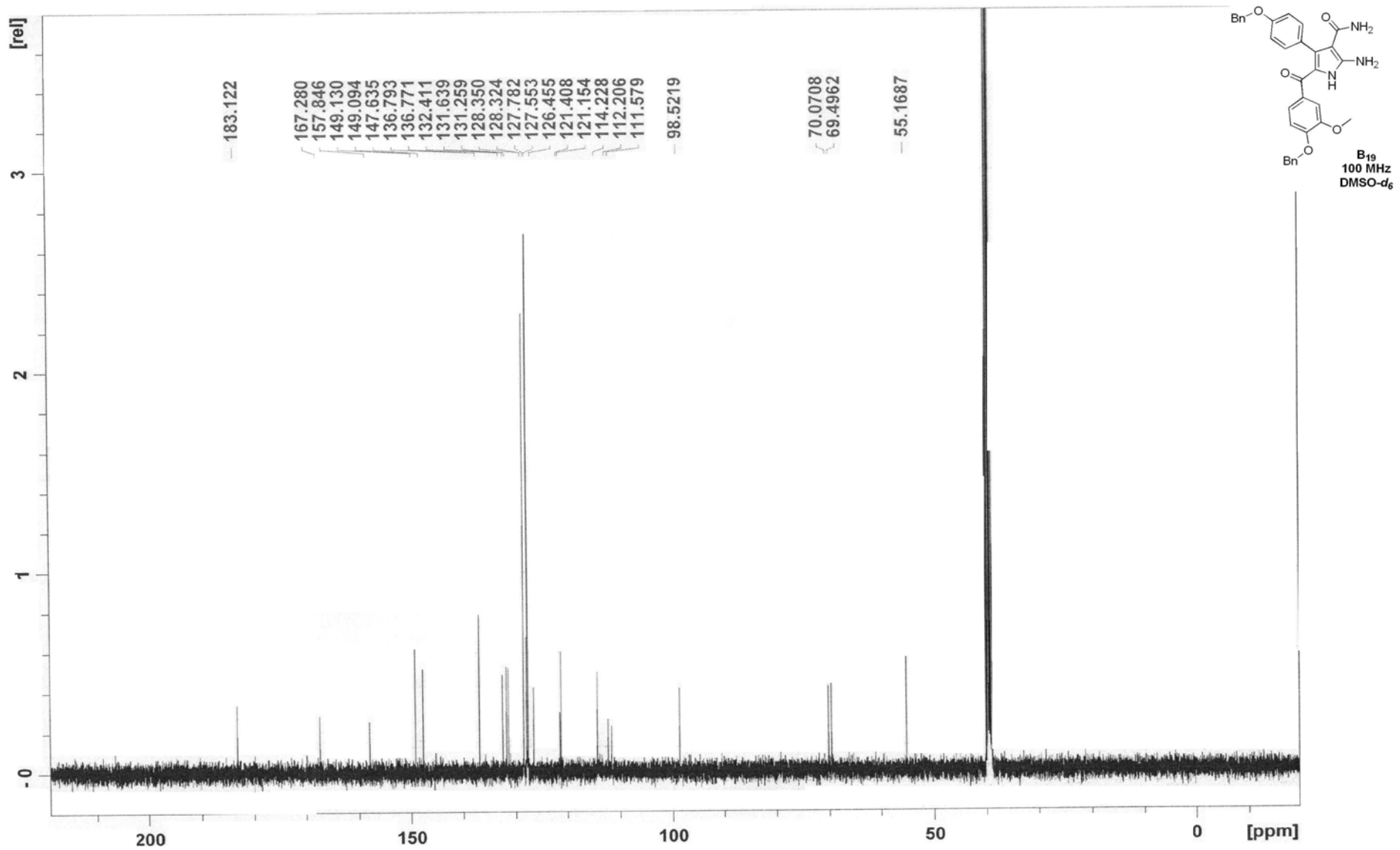

10.7913
7.1179
7.0969
6.9653
6.9445
6.7004
6.6795
6.5407
6.5196
6.3321
3.6813
3.6633
0.9728
4.0130
4.7143
2.0000
5.8706
[rel]
[ppm]
B17
400 MHz
DMSO-d6

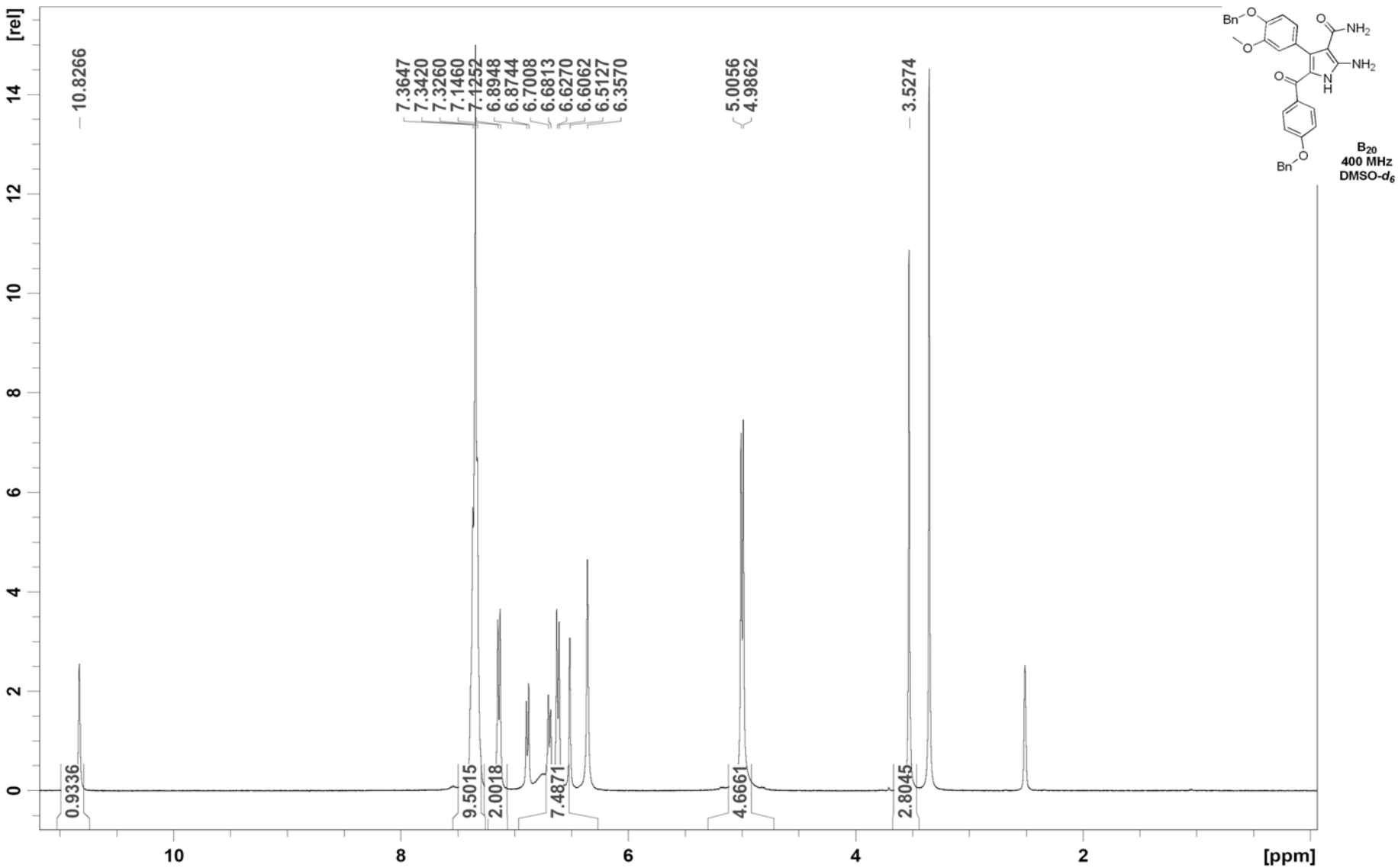

B17
75 MHz
DMSO-d6
(Thousands)
X : parts per Million : 13C
183.6463
167.8973
160.8358
159.1639
149.5832
132.2615
132.0554
131.6966
130.4293
126.6963
121.8868
114.0772
112.9703
99.0534
55.6233

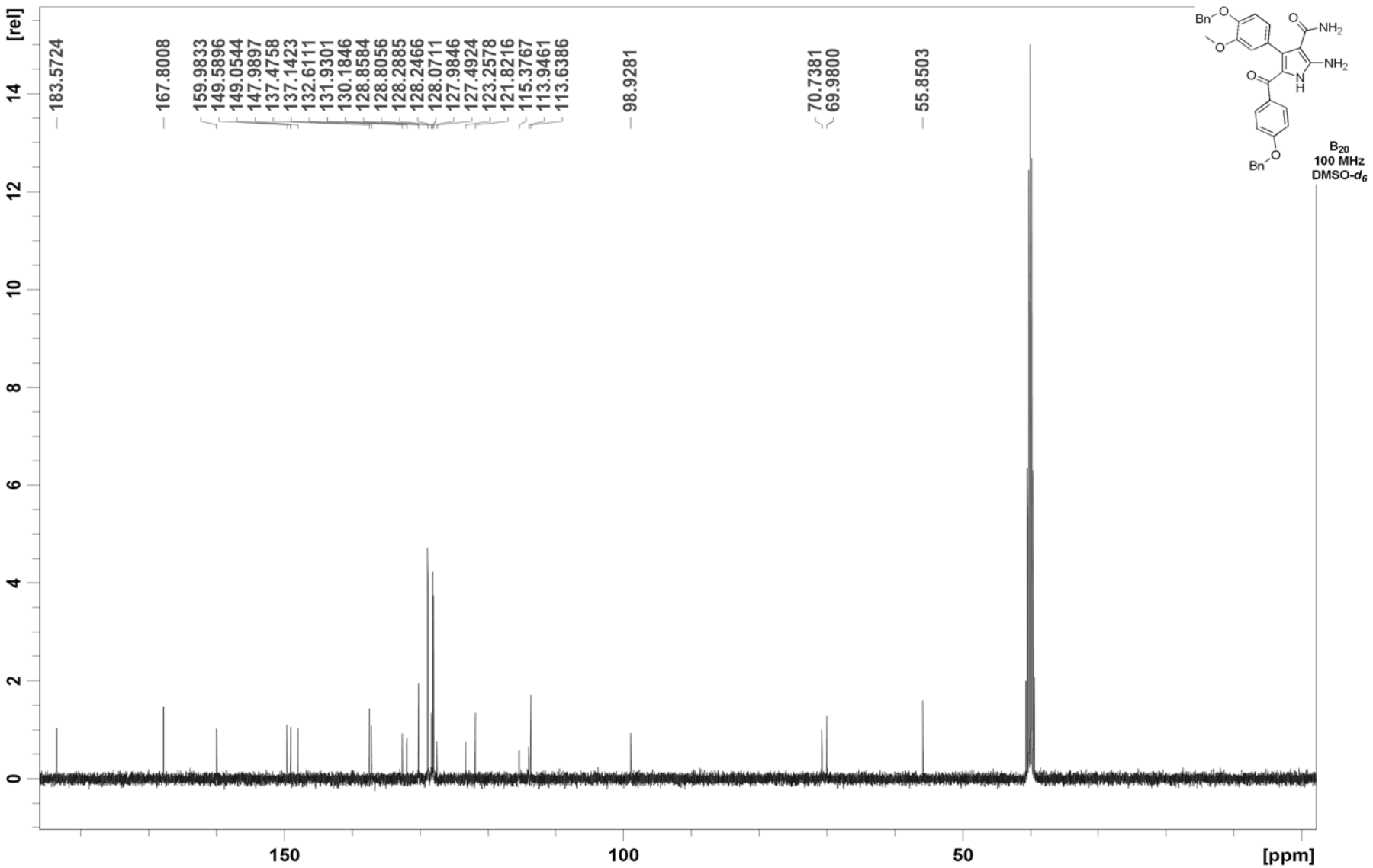

B18
300 MHz
DMSO-d6
17.24
4.0
0.98
0.28
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.7943
7.3505
7.1261
7.1042
6.9641
6.7956
6.7690
6.6381
6.6170
5.0156
X : parts per Million : 1H

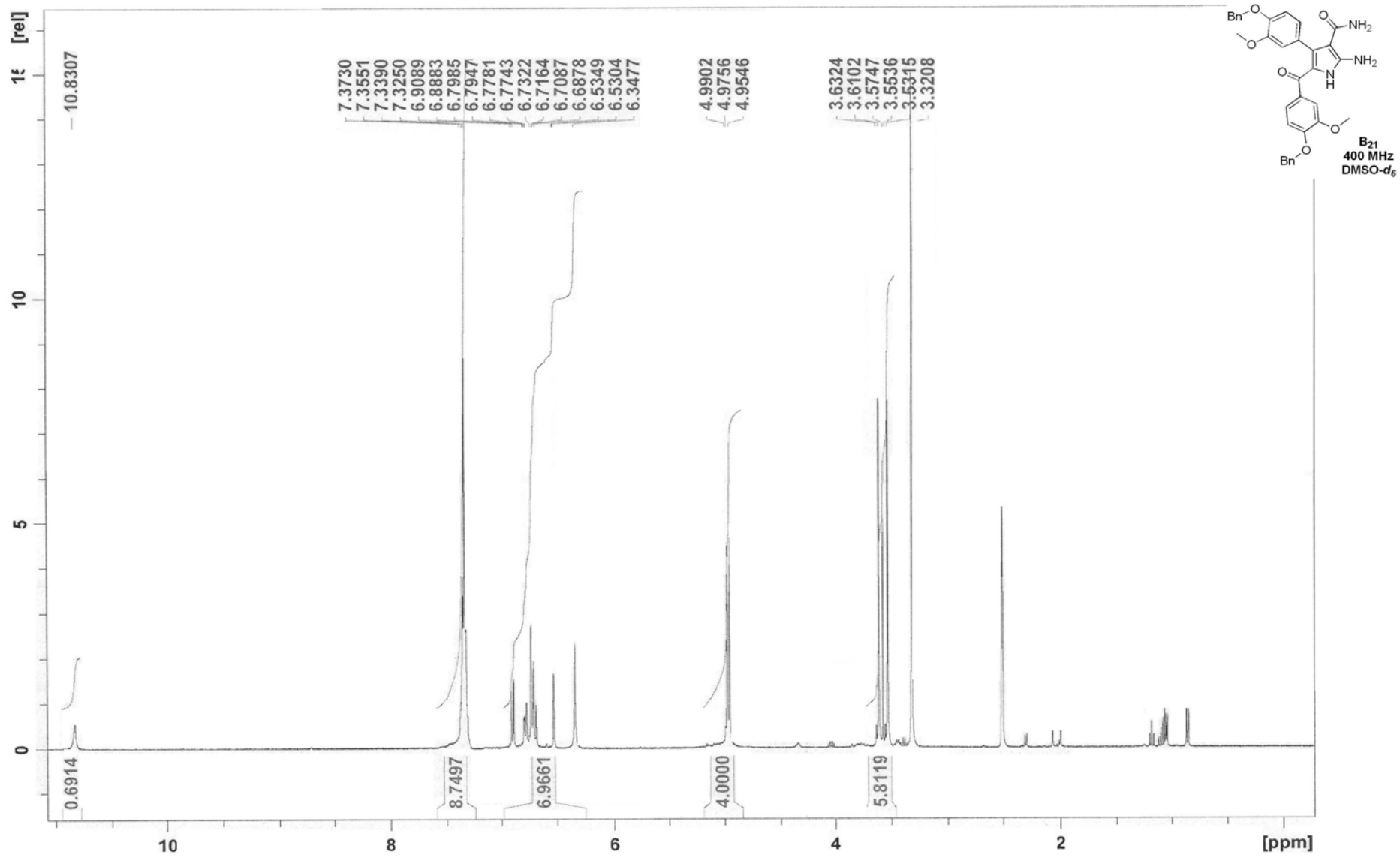

183.0374
167.3027
159.4471
157.8595
149.0207
136.7860
136.6400
131.7484
131.7118
131.2055
129.8242
128.3700
127.8131
127.7806
127.4687
127.4465
126.3764
121.3142
114.2873
113.2336
98.4767
69.3971
69.3348
[rel]
1.2
1.0
0.8
0.6
0.4
0.2
200
150
100
50
0
[ppm]
Bn
O
NH2
NH2
N
H
O
O
Bn
B18
100 MHz
DMSO-d6

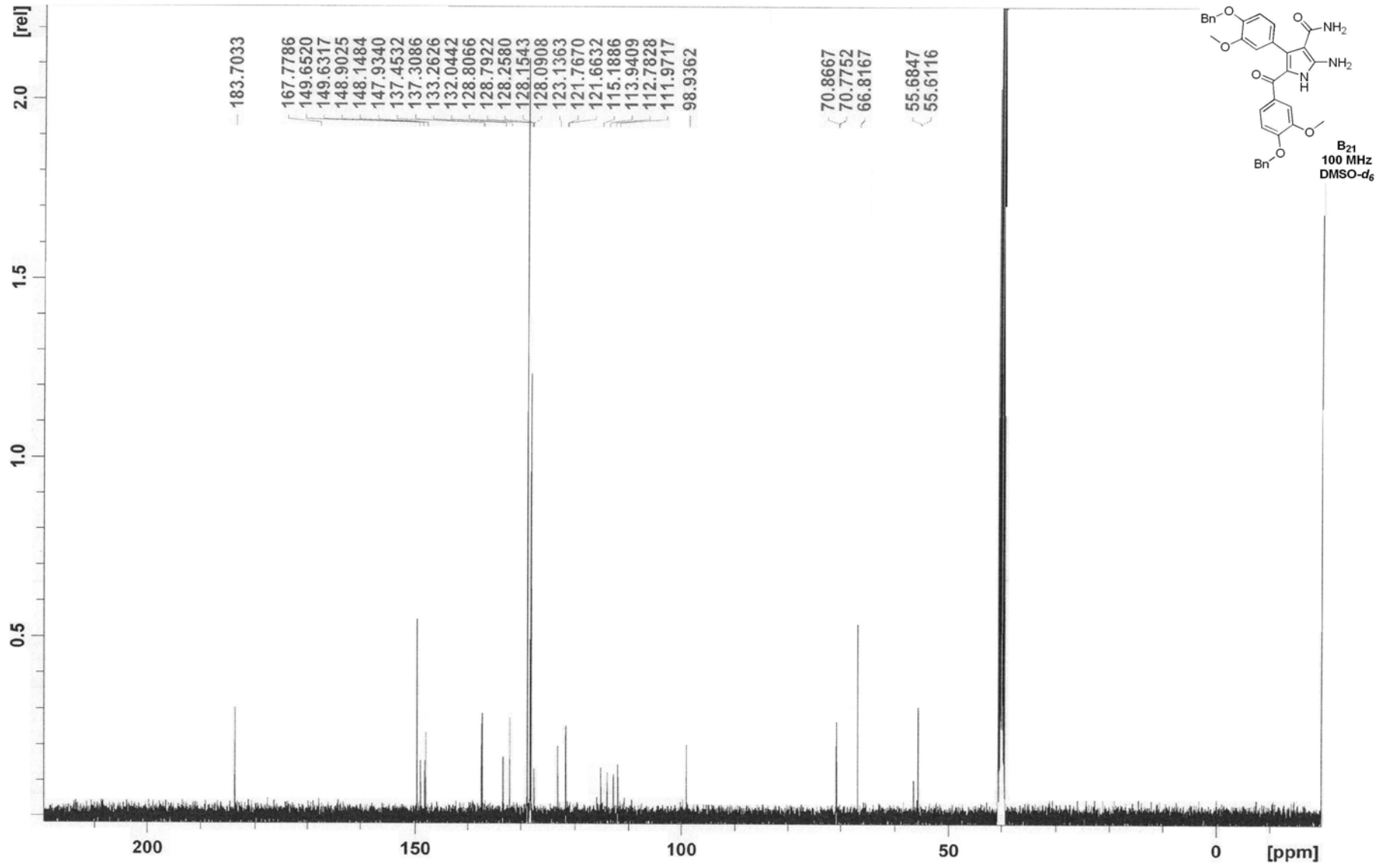

[rel]
10.8684
7.3591
7.3335
7.3268
7.0212
6.9998
6.8004
6.7849
6.7789
6.7702
6.7657
6.7175
6.7119
6.3620
4.9951
4.9831
3.6246
0.7060
18.9021
4.0000
2.8007
[ppm]
B19
400 MHz
DMSO-d6

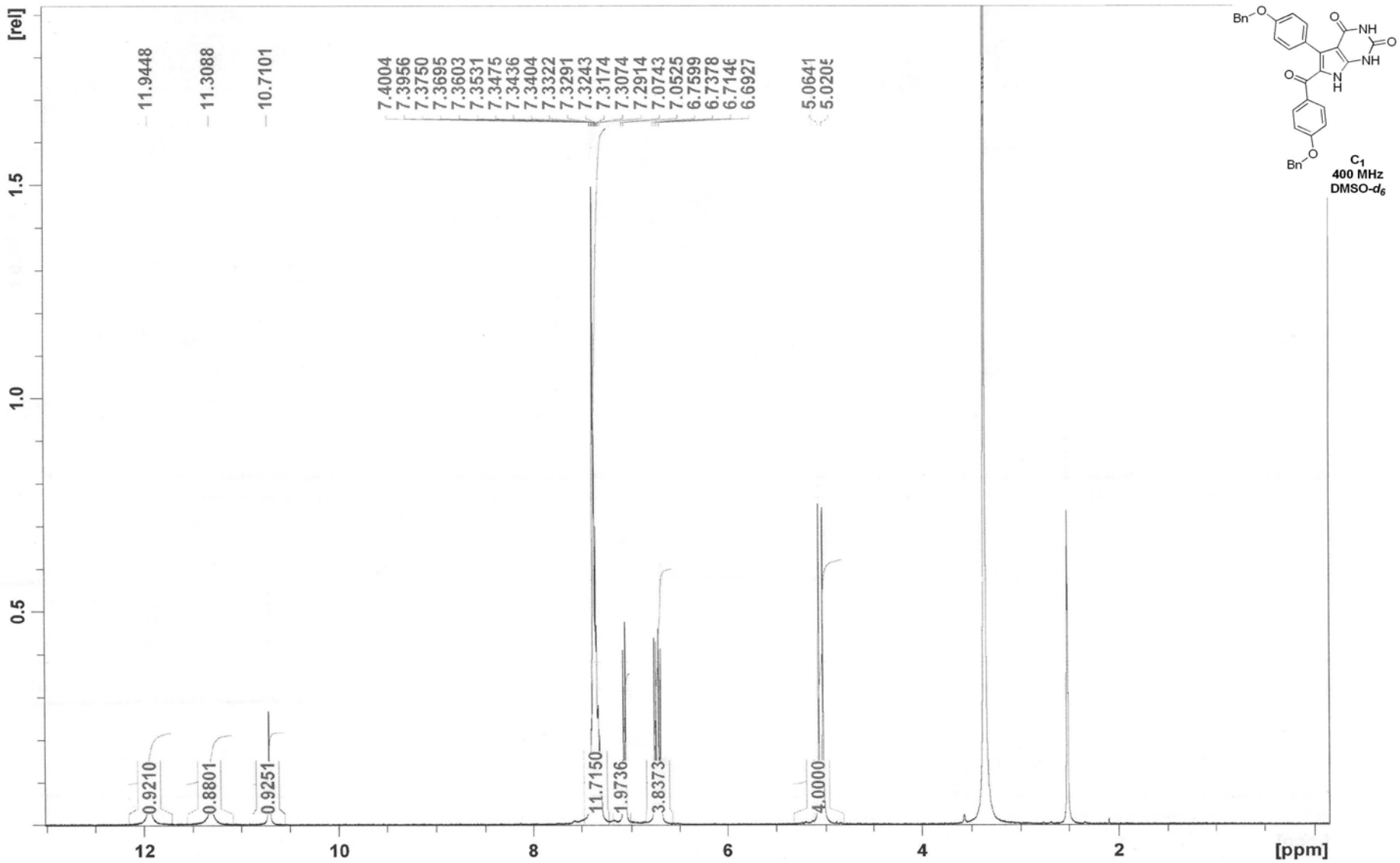

[rel]
3
2
1
-0
183.122
167.280
157.846
149.130
149.094
147.635
136.793
136.771
132.411
131.639
131.259
128.350
128.324
127.782
127.553
126.455
121.408
121.154
114.228
112.206
111.579
98.5219
70.0708
69.4962
55.1687
200
150
100
50
0
[ppm]
Bn
O
NH2
NH2
N
H
O
O
Bn
O
B19
100 MHz
DMSO-d6

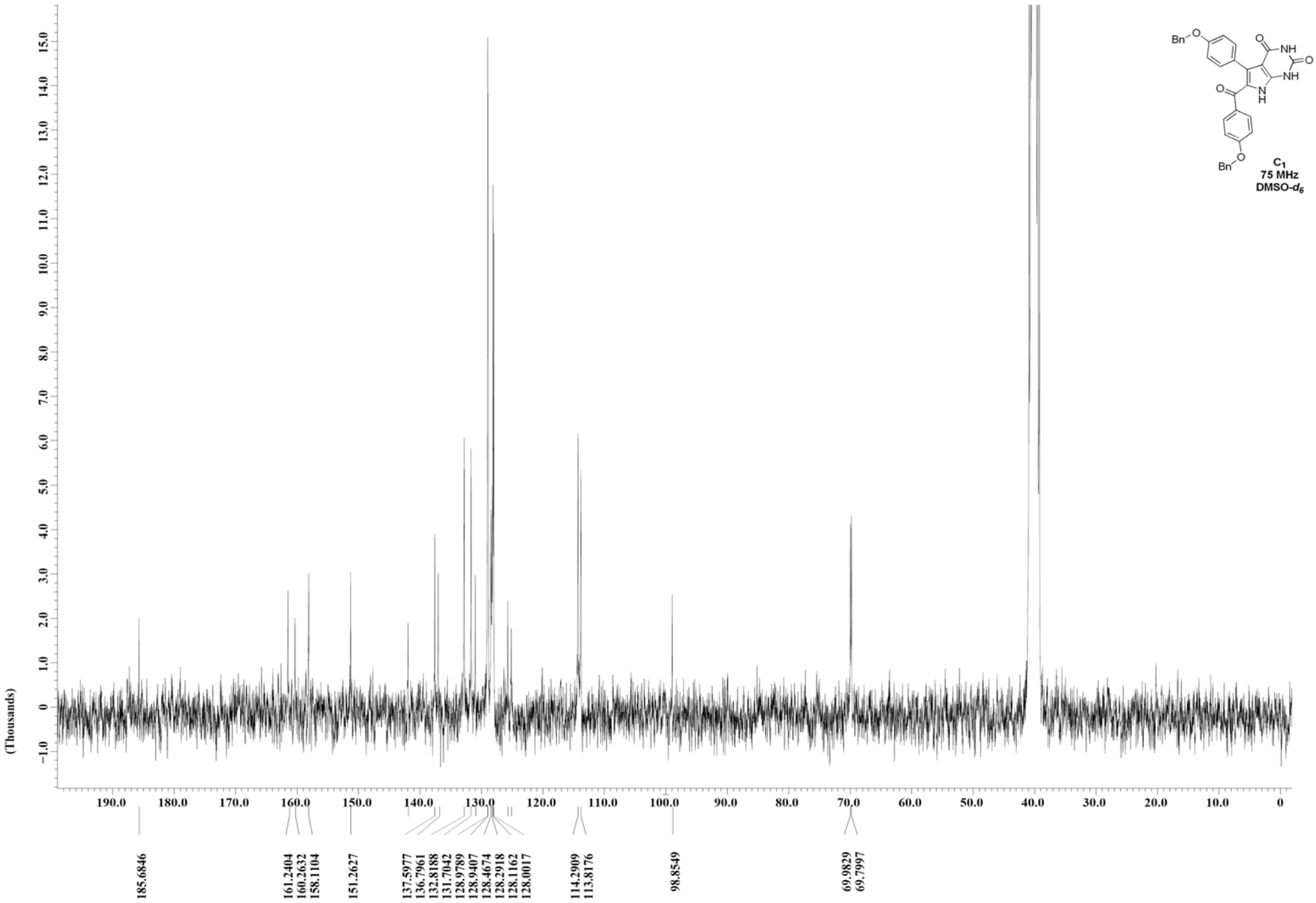

[rel]
10.8266
7.3647
7.3420
7.3260
7.1460
7.1252
6.8948
6.8744
6.7008
6.6813
6.6270
6.6062
6.5127
6.3570
5.0056
4.9862
3.5274
0.9336
9.5015
2.0018
7.4871
4.6661
2.8045
[ppm]
B20
400 MHz
DMSO-d6

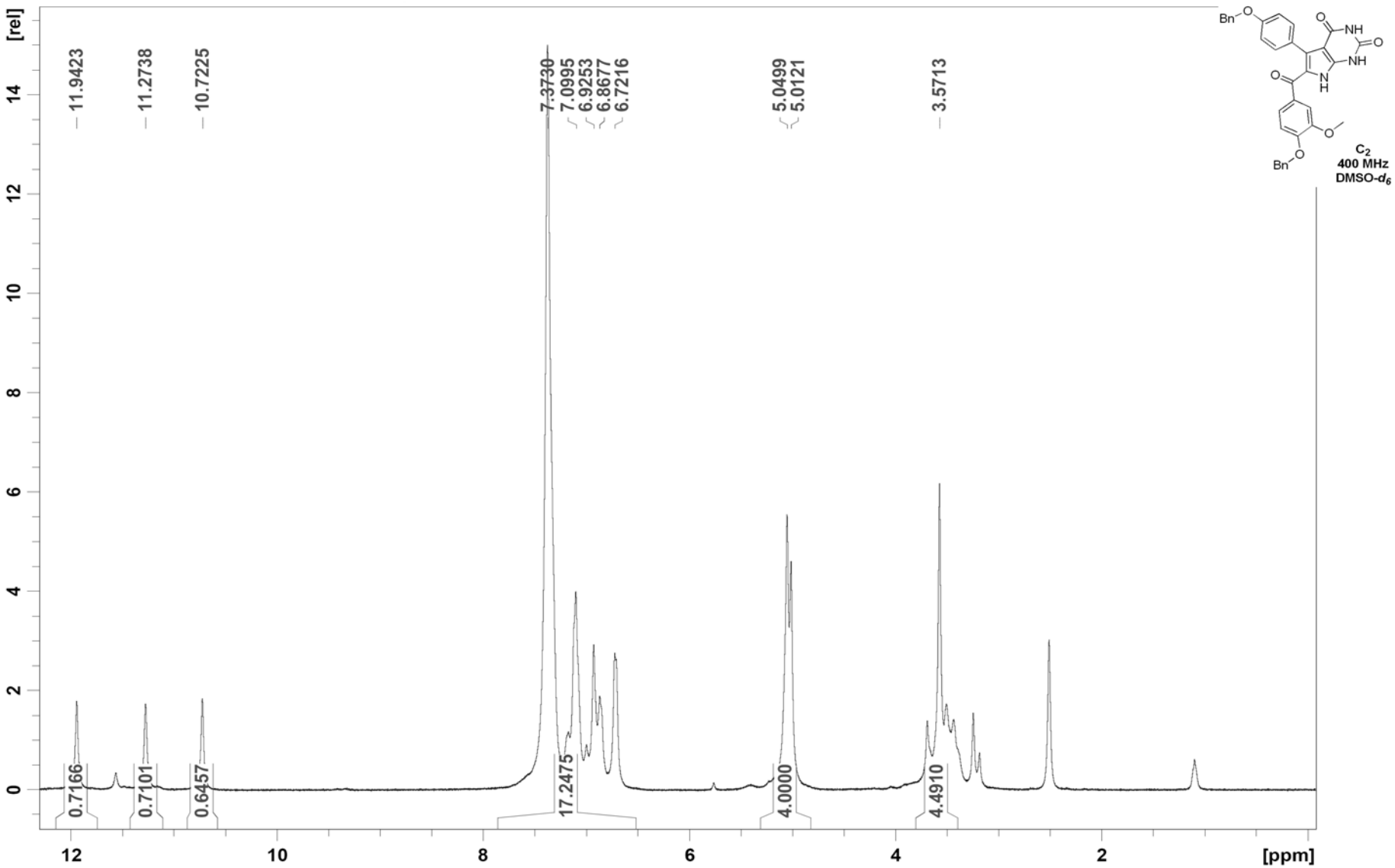

183.5724
167.8008
159.9833
149.5896
149.0544
147.9897
137.4758
137.1423
132.6111
131.9301
130.1846
128.8584
128.8056
128.2885
128.2466
128.0711
127.9846
127.4924
123.2578
121.8216
115.3767
113.9461
113.6386
98.9281
70.7381
69.9800
55.8503
[rel]
[ppm]
B20
100 MHz
DMSO-d6

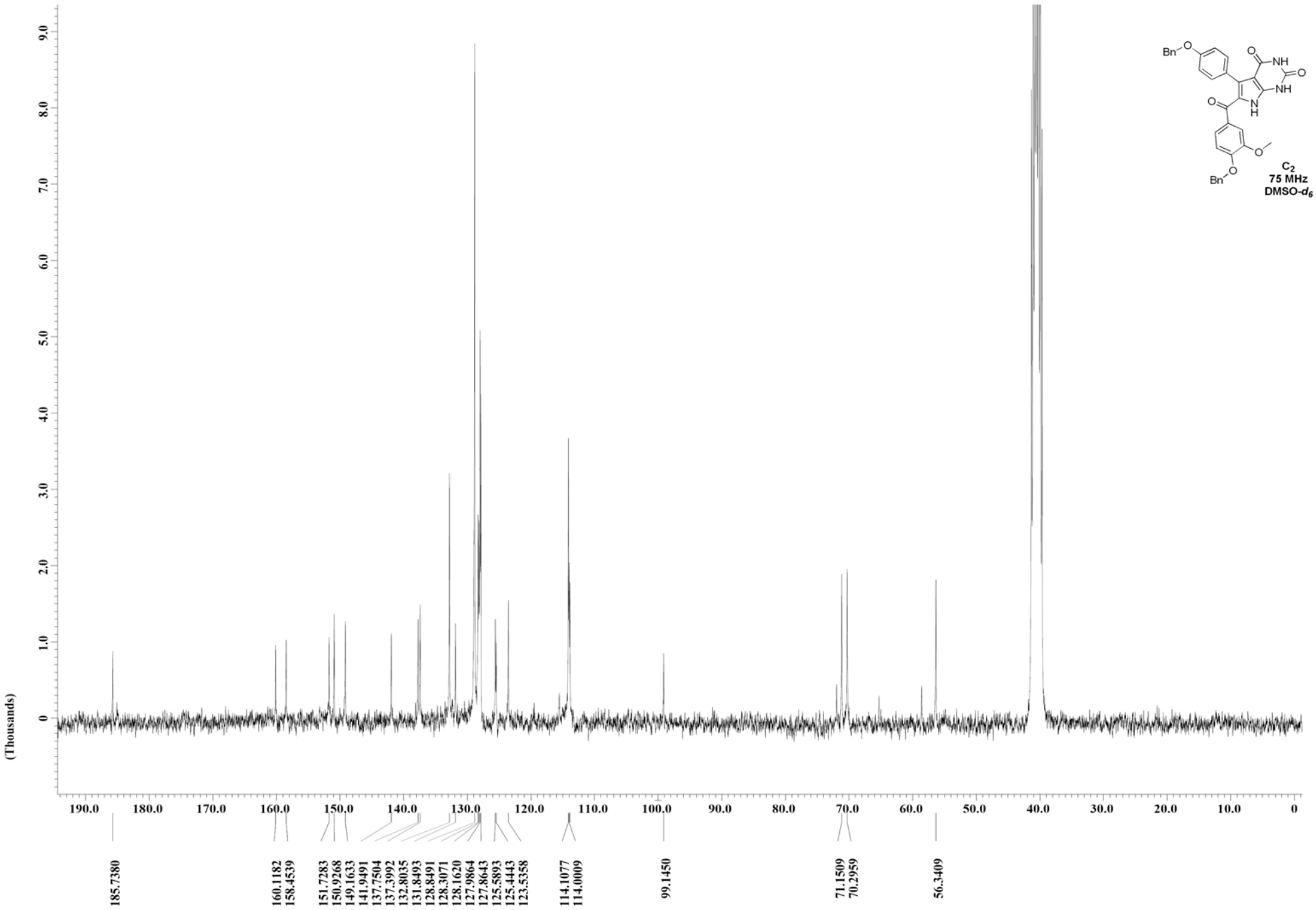

[rel]
15
10
5
0
10.8307
7.3730
7.3551
7.3390
7.3250
6.9089
6.8883
6.7985
6.7947
6.7781
6.7743
6.7322
6.7164
6.7087
6.6878
6.5349
6.5304
6.3477
4.9902
4.9756
4.9546
3.6324
3.6102
3.5747
3.5536
3.5315
3.3208
0.6914
8.7497
6.9661
4.0000
5.8119
10
8
6
4
2
[ppm]
Bn
O
NH2
NH2
N
H
B21
400 MHz
DMSO-d6

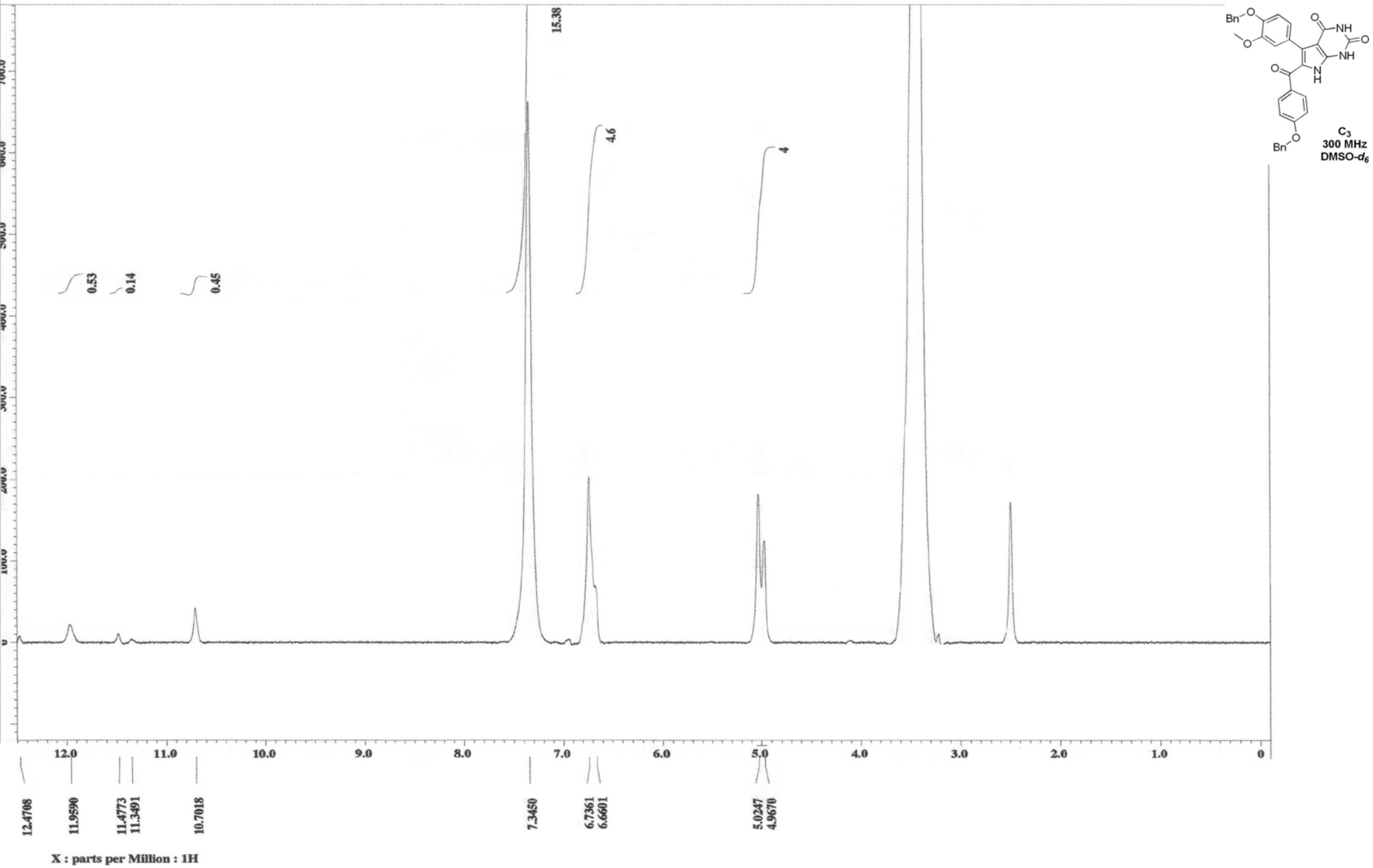

183.7033
167.7786
149.6520
149.6317
148.9025
148.1484
147.9340
137.4532
137.3086
133.2626
132.0442
128.8066
128.7922
128.2580
128.1543
128.0908
123.1363
121.7670
121.6632
115.1886
113.9409
112.7828
111.9717
98.9362
70.8667
70.7752
66.8167
55.6847
55.6116
[rel]
2.0
1.5
1.0
0.5
200
150
100
50
0
[ppm]
Bn
O
NH2
NH2
N
H
B21
100 MHz
DMSO-d6

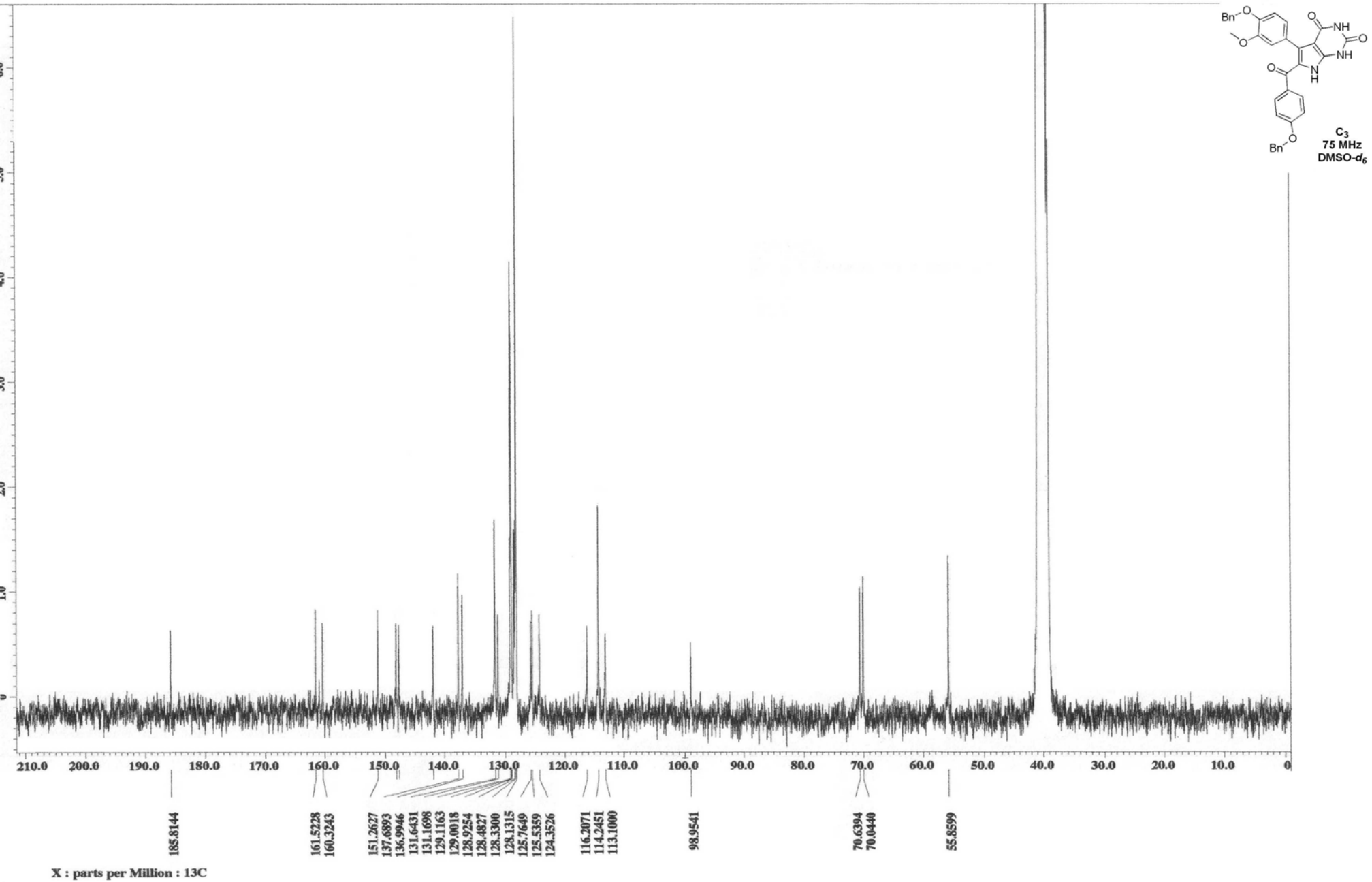

[rel]
1.5
1.0
0.5
11.9448
11.3088
10.7101
7.4004
7.3956
7.3750
7.3695
7.3603
7.3531
7.3475
7.3436
7.3404
7.3322
7.3291
7.3243
7.3174
7.3074
7.2914
7.0743
7.0525
6.7599
6.7378
6.7146
6.6927
5.0641
5.0205
0.9210
0.8801
0.9251
11.7150
1.9736
3.8373
4.0000
12
10
8
6
4
2
[ppm]
Bn
O
NH
O
NH
O
Bn
C1
400 MHz
DMSO-d6

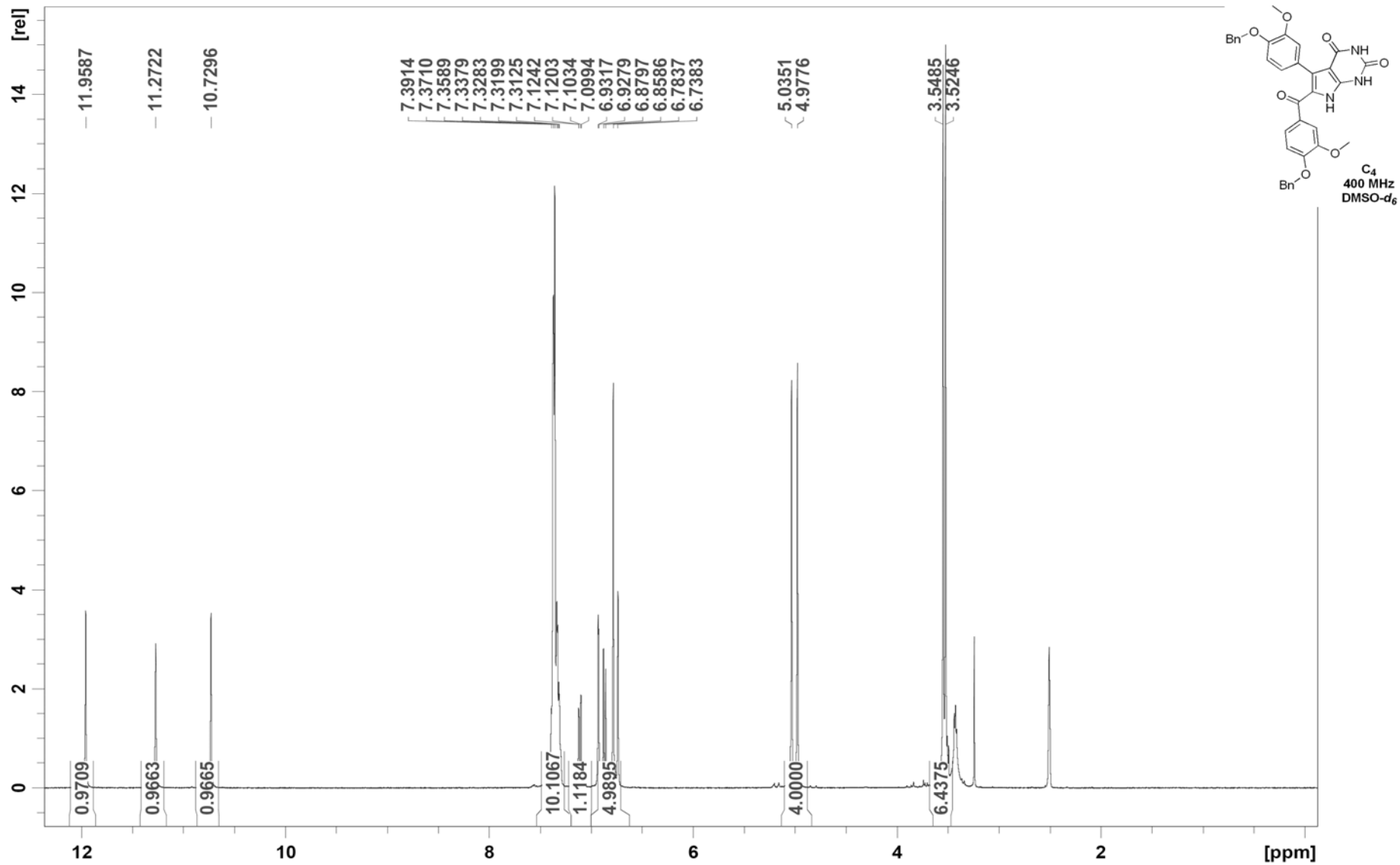

Bn
O
NH
O
NH
O
Bn
O
C1
75 MHz
DMSO-d6
(Thousands)
15.0
14.0
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
−1.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
185.6846
161.2404
160.2632
158.1104
151.2627
137.5977
136.7961
132.8188
131.7042
128.9789
128.9407
128.4674
128.2918
128.1162
128.0017
114.2909
113.8176
98.8549
69.9829
69.7997
X : parts per Million : 13C

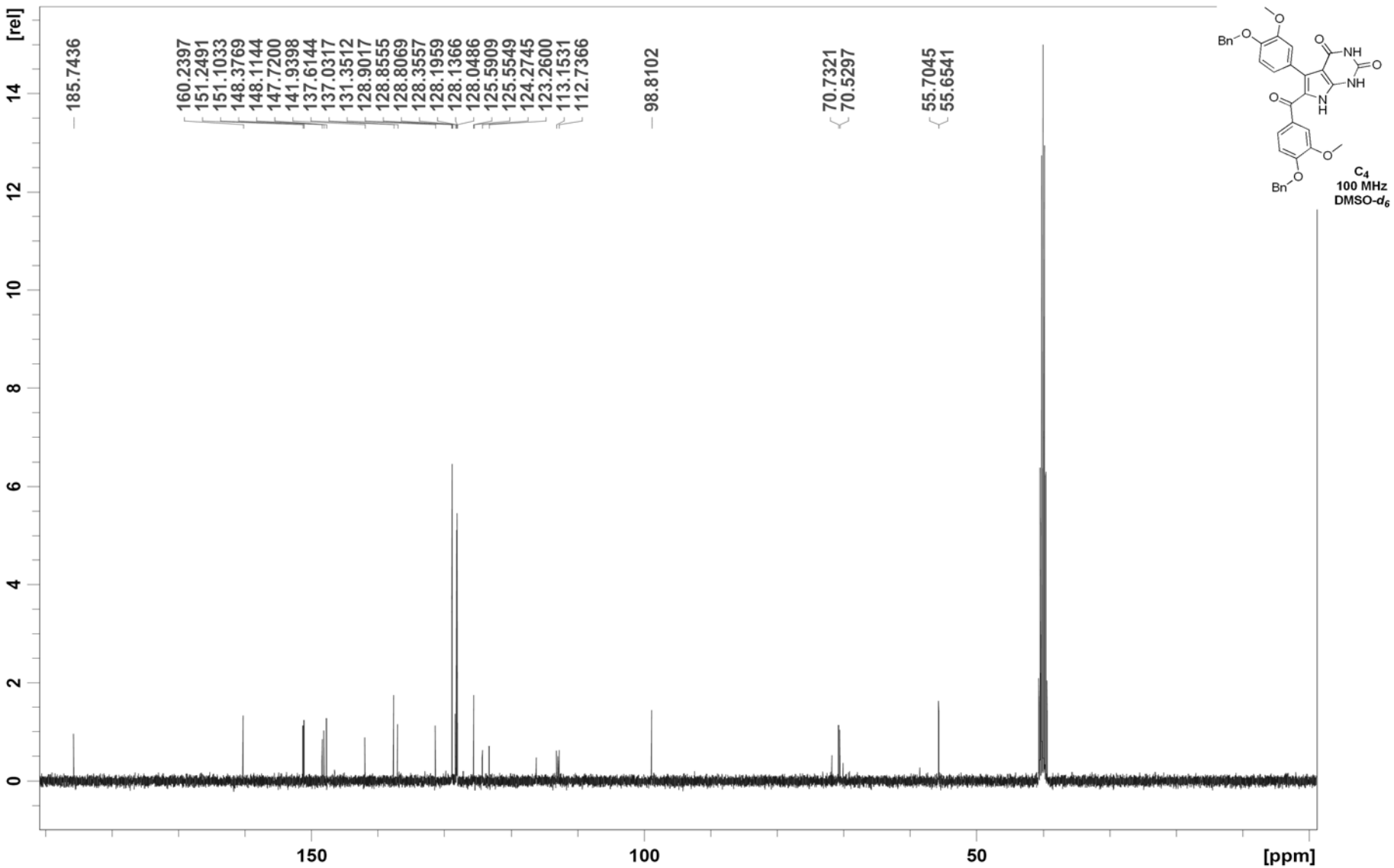

11.9423
11.2738
10.7225
7.3730
7.0995
6.9253
6.8677
6.7216
5.0499
5.0121
3.5713
0.7166
0.7101
0.6457
17.2475
4.0000
4.4910
[rel]
[ppm]
Bn
NH
NH
C2
400 MHz
DMSO-d6

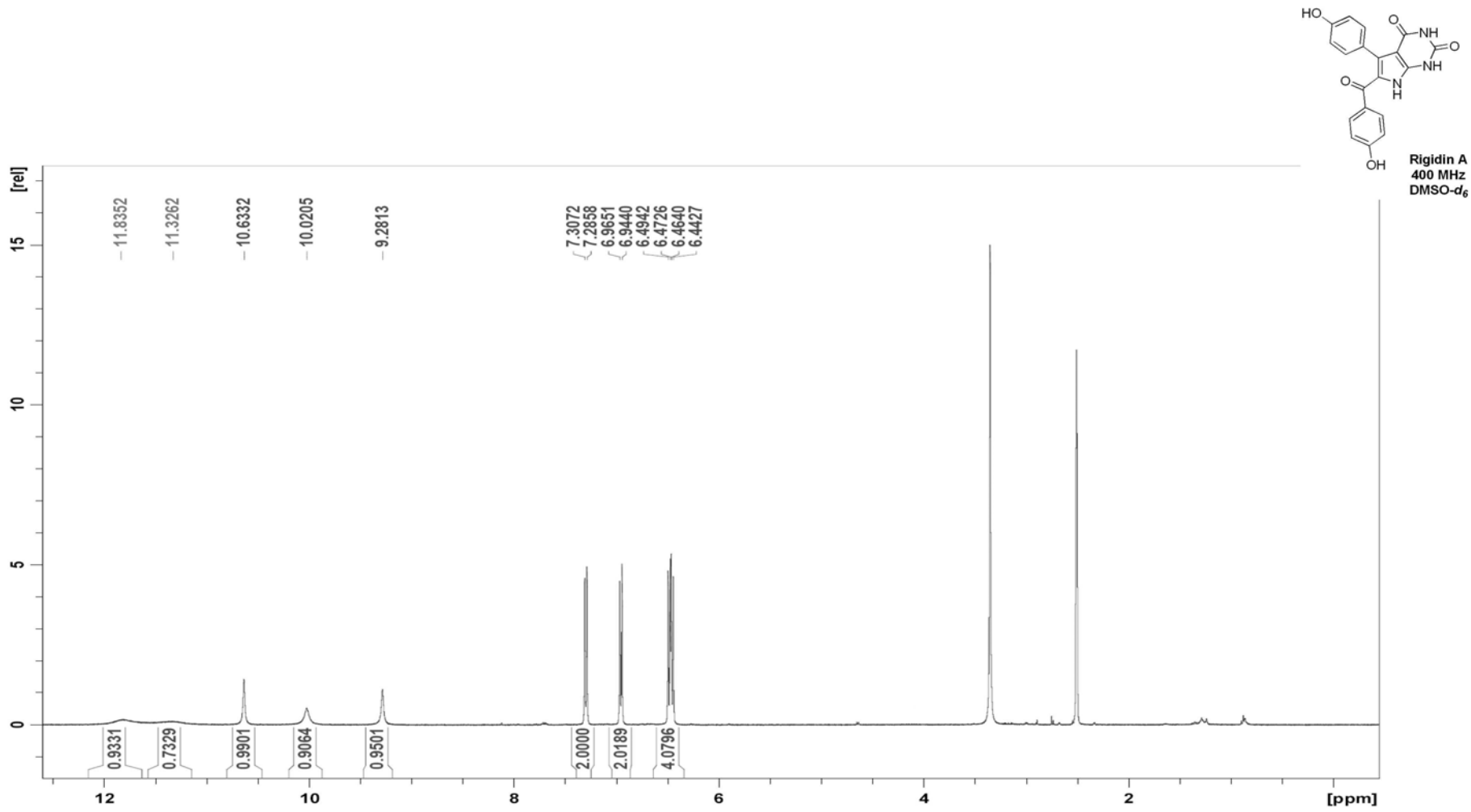

(Thousands)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
185.7380
160.1182
158.4539
151.7283
150.9268
149.1633
141.9491
137.7504
137.3992
132.8035
131.8493
128.8491
128.3071
128.1620
127.9864
127.8643
125.5893
125.4443
123.5358
114.1077
114.0009
99.1450
71.1509
70.2959
56.3409
X : parts per Million : 13C
Bn
O
NH
O
NH
N
H
O
O
Bn
C2
75 MHz
DMSO-d6

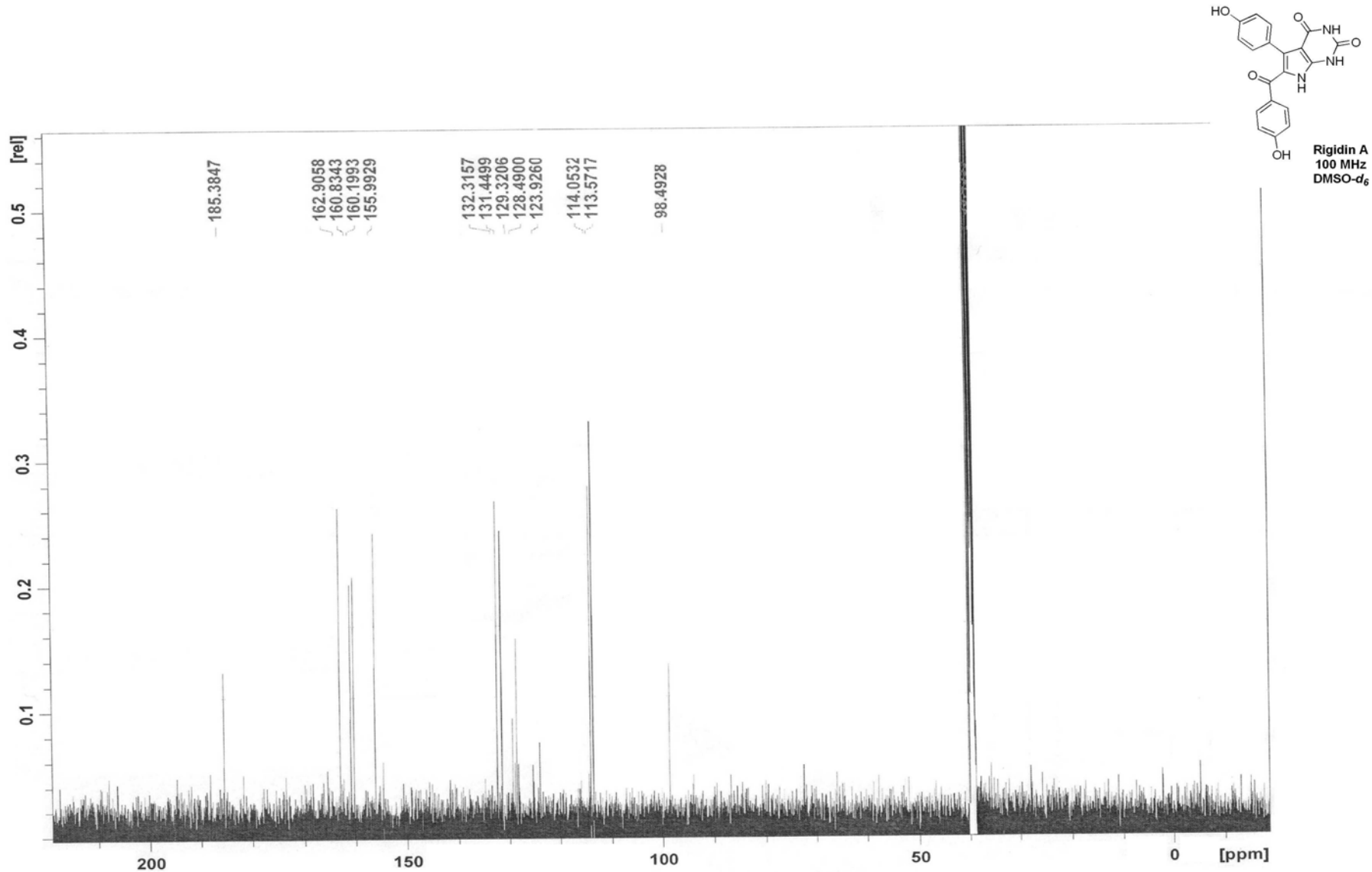

15.38
4.6
4
0.53
0.14
0.45
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
12.4708
11.9590
11.4773
11.3491
10.7018
7.3450
6.7361
6.6601
5.0247
4.9670
X : parts per Million : 1H
Bn
O
NH
N
H
C3
300 MHz
DMSO-d6

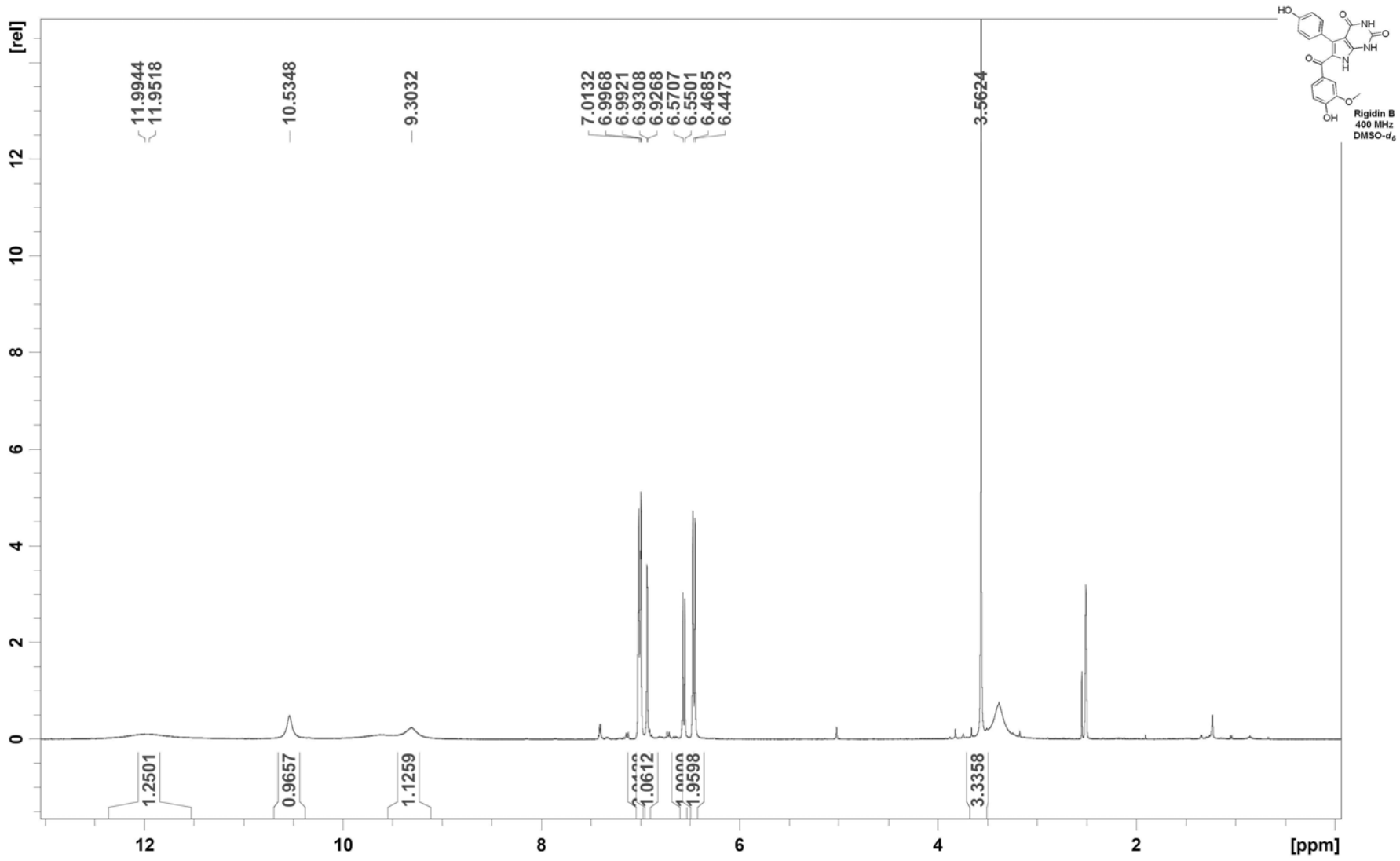

Bn
O
O
NH
O
NH
N
H
O
Bn
O
C3
75 MHz
DMSO-d6
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
185.8144
161.5228
160.3243
151.2627
137.6893
136.9946
131.6431
131.1698
129.1163
129.0018
128.9254
128.4827
128.3300
128.1315
125.7649
125.5359
124.3526
116.2071
114.2451
113.1000
98.9541
70.6394
70.0440
55.8599
X : parts per Million : 13C

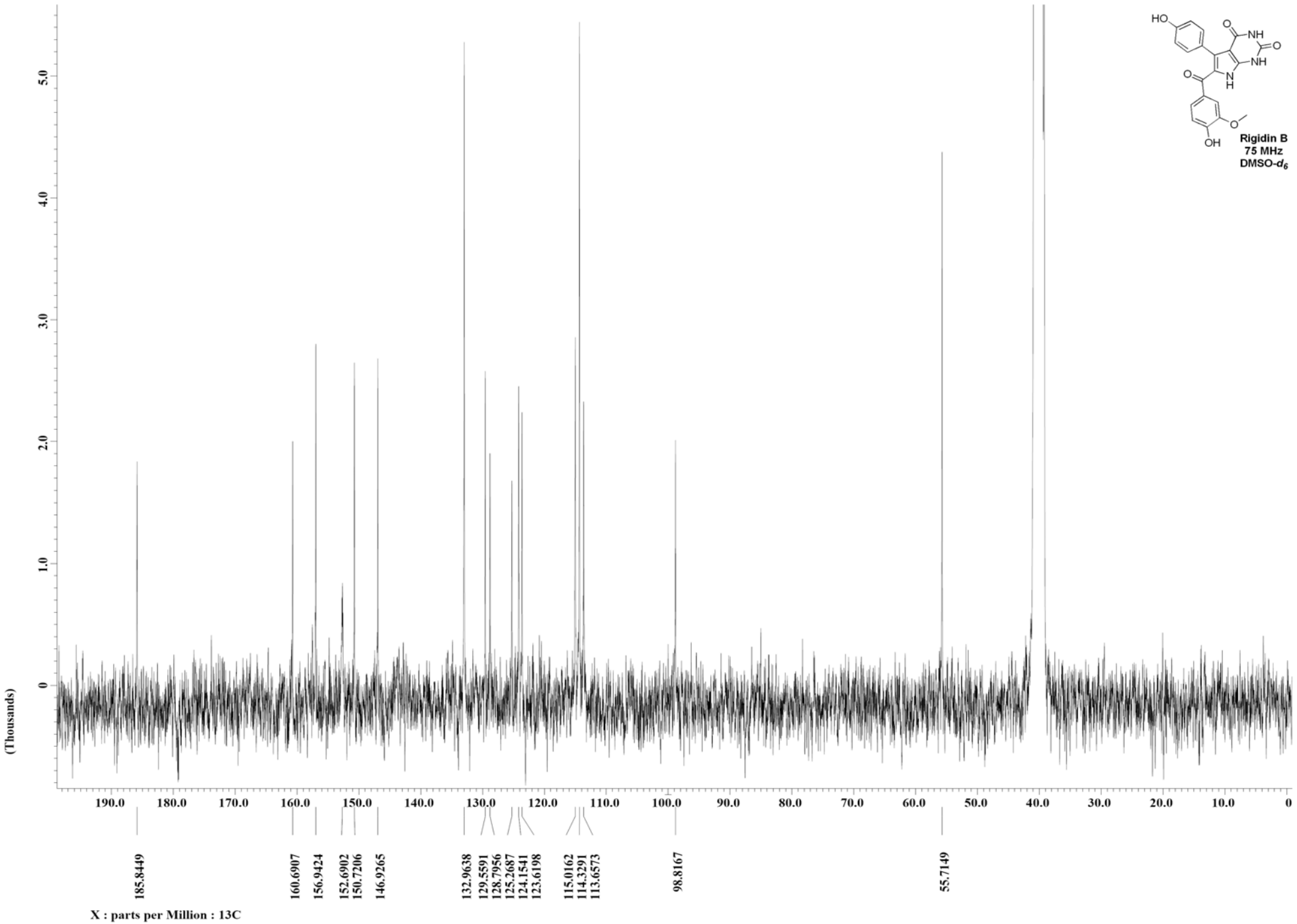

11.9587
11.2722
10.7296
7.3914
7.3710
7.3589
7.3379
7.3283
7.3199
7.3125
7.1242
7.1203
7.1034
7.0994
6.9317
6.9279
6.8797
6.8586
6.7837
6.7383
5.0351
4.9776
3.5485
3.5246
0.9709
0.9663
0.9665
10.1067
1.1184
4.9895
4.0000
6.4375
[rel]
[ppm]
C4
400 MHz
DMSO-d6

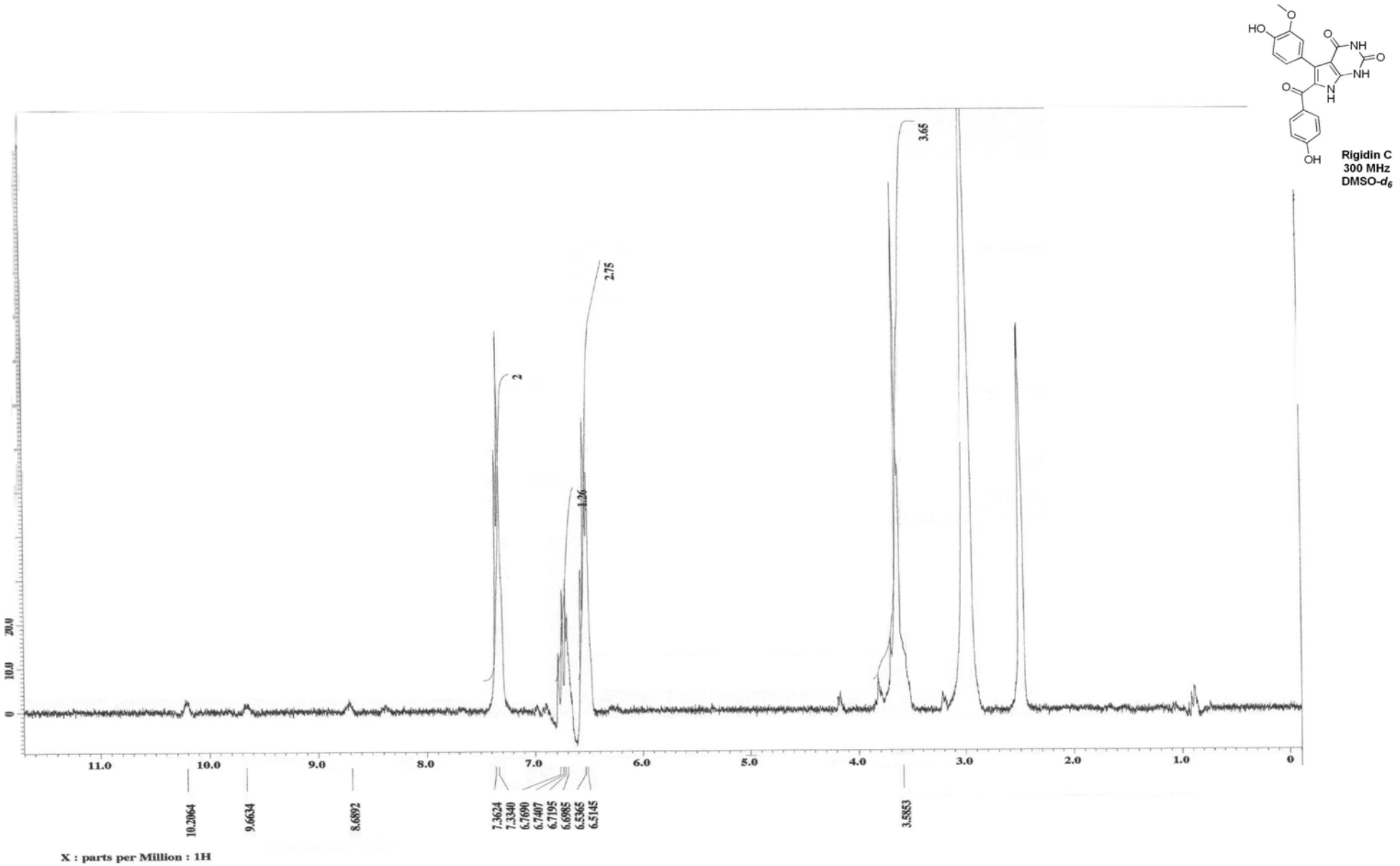

185.7436
160.2397
151.2491
151.1033
148.3769
148.1144
147.7200
141.9398
137.6144
137.0317
131.3512
128.9017
128.8555
128.8069
128.3557
128.1959
128.1366
128.0486
125.5909
125.5549
124.2745
123.2600
113.1531
112.7366
98.8102
70.7321
70.5297
55.7045
55.6541
[rel]
[ppm]
C4
100 MHz
DMSO-d6

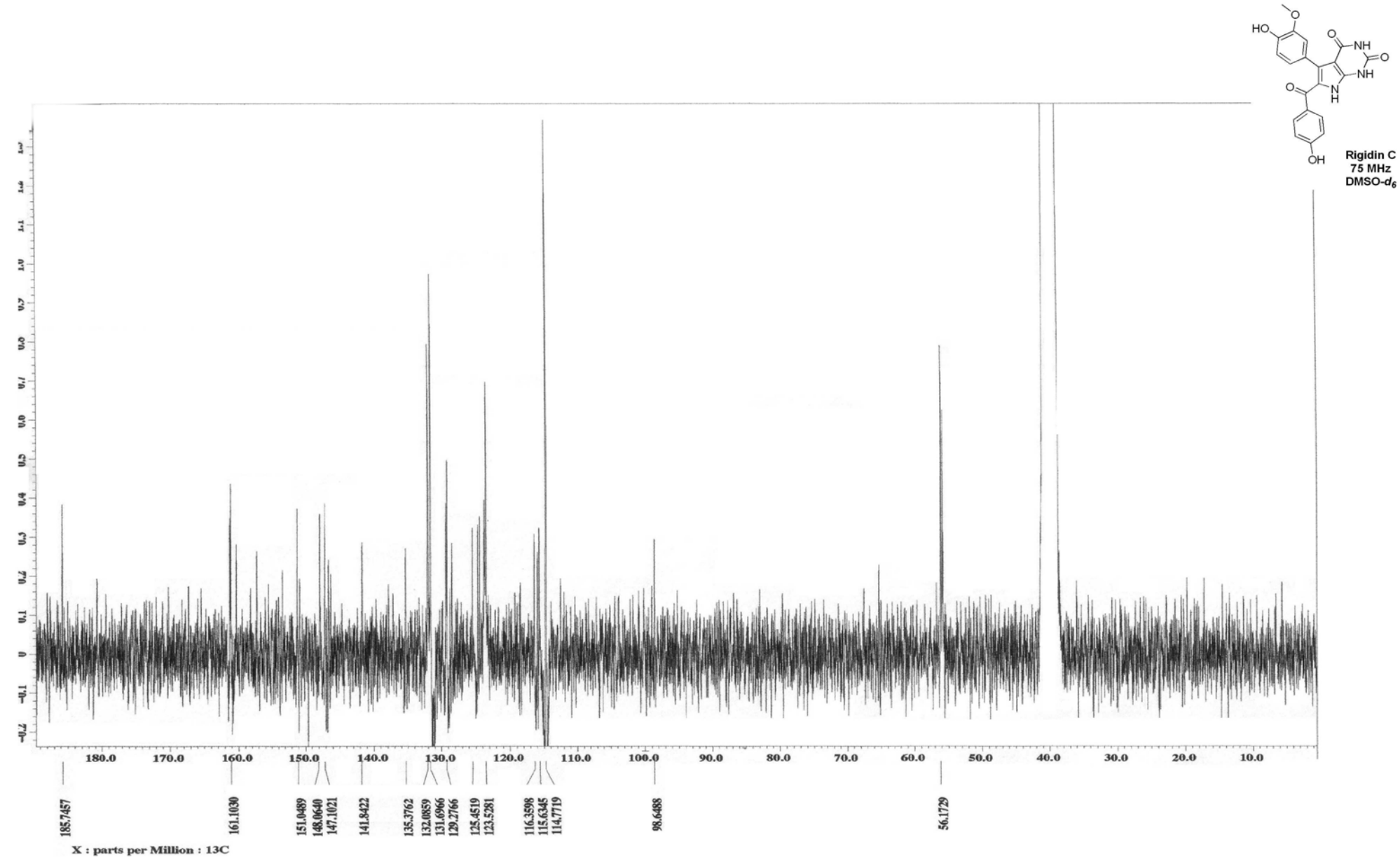

Rigidin A
400 MHz
DMSO-d_6
[rel]
11.8352
11.3262
10.6332
10.0205
9.2813
7.3072
7.2858
6.9651
6.9440
6.4942
6.4726
6.4640
6.4427
0.9331
0.7329
0.9901
0.9064
0.9501
2.0000
2.0189
4.0796
[ppm]

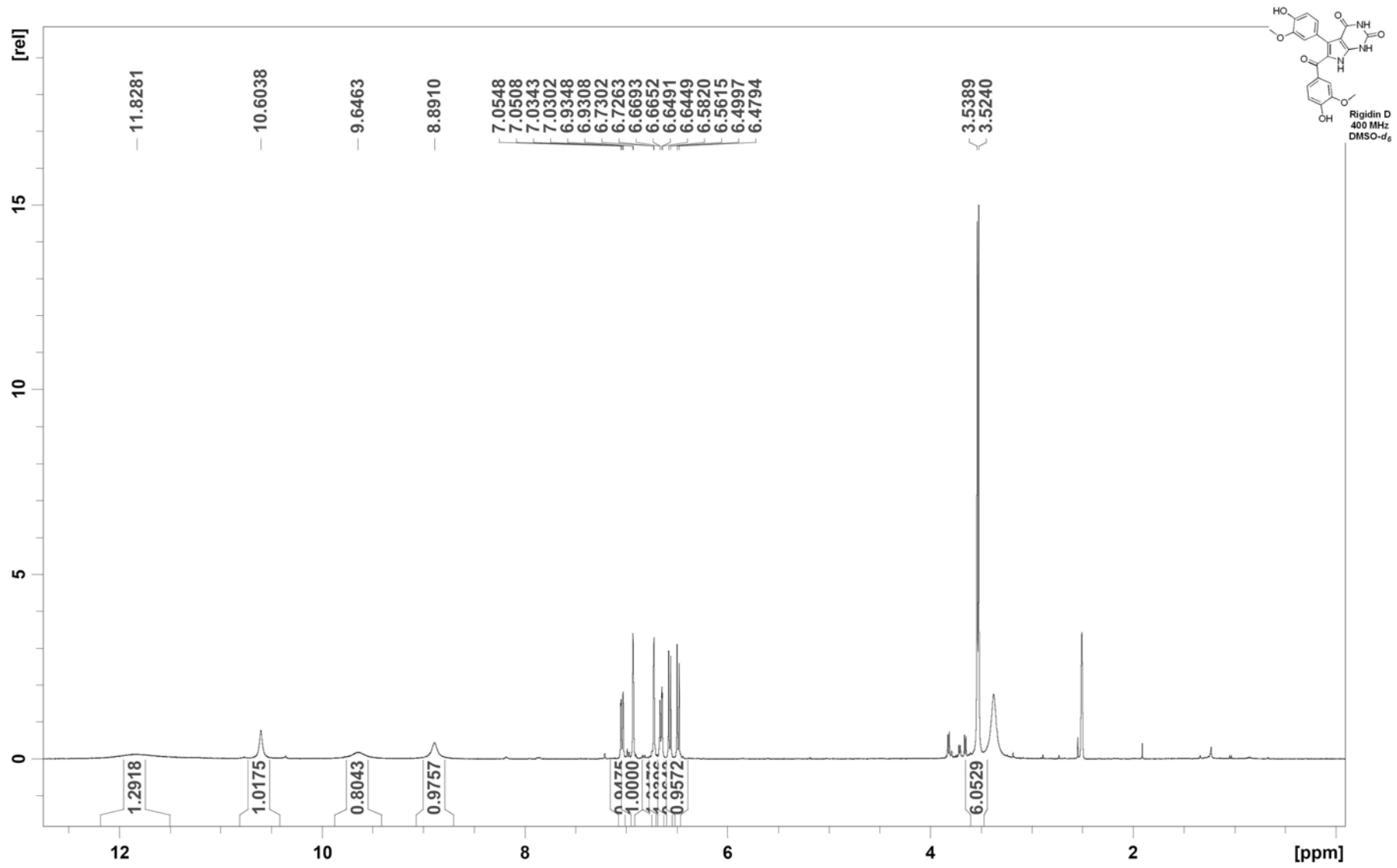

Rigidin A
100 MHz
DMSO-d_6
[rel]
0.5
0.4
0.3
0.2
0.1
185.3847
162.9058
160.8343
160.1993
155.9929
132.3157
131.4499
129.3206
128.4900
123.9260
114.0532
113.5717
98.4928
200
150
100
50
0
[ppm]

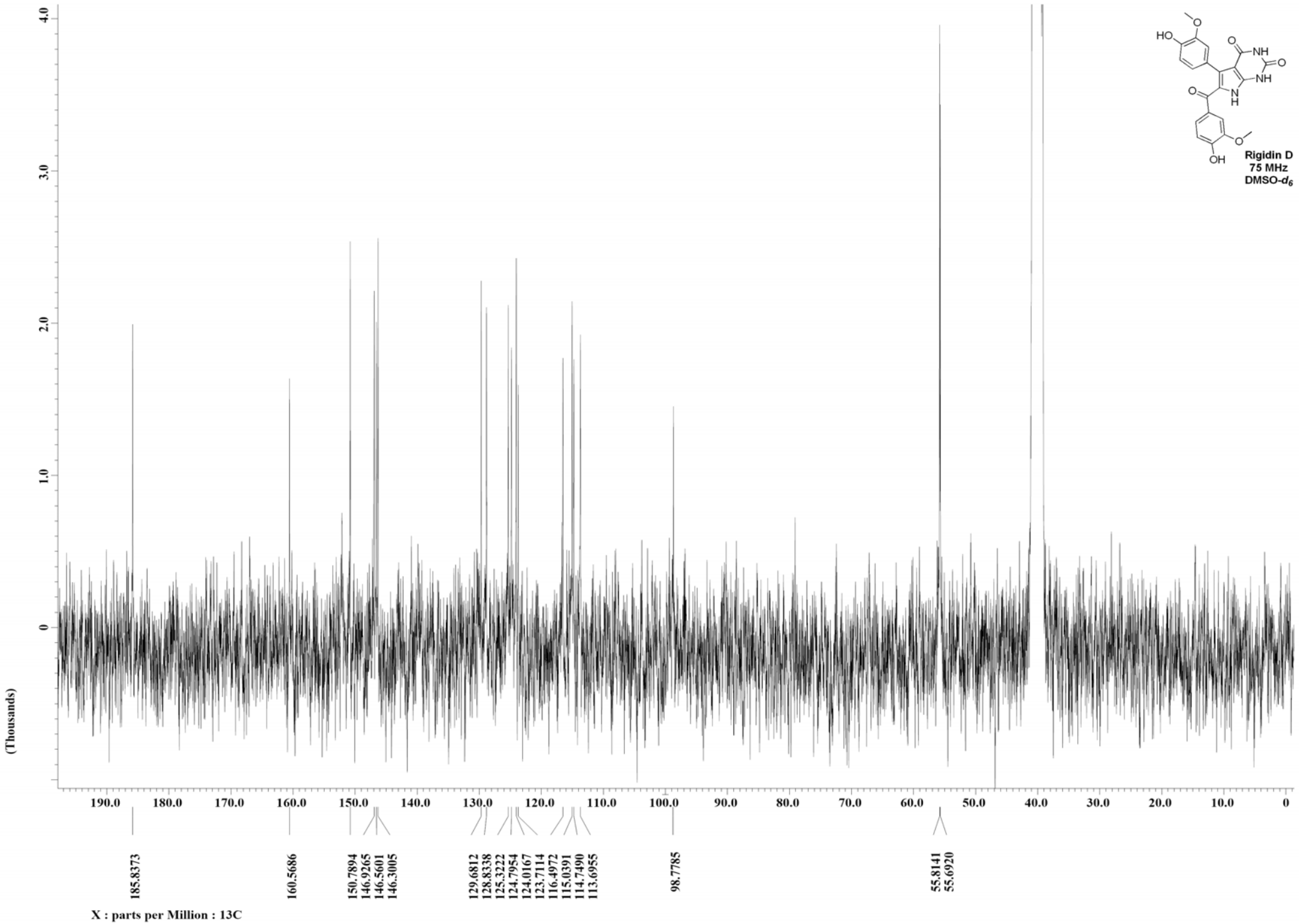

[rel]
11.9944
11.9518
10.5348
9.3032
7.0132
6.9968
6.9921
6.9308
6.9268
6.5707
6.5501
6.4685
6.4473
3.5624
1.2501
0.9657
1.1259
1.0612
1.9598
3.3358
[ppm]
Rigidin B
400 MHz
DMSO-d_6


Rigidin B
75 MHz
DMSO-d_6
(Thousands)
5.0
4.0
3.0
2.0
1.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
185.8449
160.6907
156.9424
152.6902
150.7206
146.9265
132.9638
129.5591
128.7956
125.2687
124.1541
123.6198
115.0162
114.3291
113.6573
98.8167
55.7149
X : parts per Million : 13C


Rigidin C
300 MHz
DMSO-d_6
3.65
2.75
1.26
2
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.2064
9.6634
8.6892
7.3624
7.3340
6.7690
6.7407
6.7195
6.6985
6.5365
6.5145
3.5853
X : parts per Million : 1H


Rigidin C
75 MHz
DMSO-d_6
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
185.7457
161.1030
151.0489
148.0640
147.1021
141.8422
135.3762
132.0859
131.6966
129.2766
125.4519
123.5281
116.3598
115.6345
114.7719
98.6488
56.1729
X : parts per Million : 13C


[rel]
11.8281
10.6038
9.6463
8.8910
7.0548
7.0508
7.0343
7.0302
6.9348
6.9308
6.7302
6.7263
6.6693
6.6652
6.6491
6.6449
6.5820
6.5615
6.4997
6.4794
3.5389
3.5240
Rigidin D
400 MHz
DMSO-d_6
1.2918
1.0175
0.8043
0.9757
1.0000
0.9572
6.0529
[ppm]


Rigidin D
75 MHz
DMSO-d_6
(Thousands)
4.0
3.0
2.0
1.0
0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
185.8373
160.5686
150.7894
146.9265
146.5601
146.3005
129.6812
128.8338
125.3222
124.7954
124.0167
123.7114
116.4972
115.0391
114.7490
113.6955
98.7785
55.8141
55.6920
X : parts per Million : 13C