Supporting Information

One-Pot Primary Aminomethylation of Aryl and Heteroaryl Halides with Sodium Phthalimidomethyltrifluoroborate

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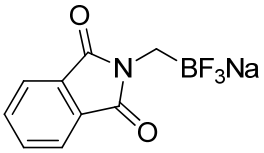
1. General

Nuclear magnetic resonance (^{1}H NMR (400 MHz), ^{13}C NMR (100MHz)) spectra were determined on a Varian Mercury plus 400 MHz or JEOL-ECA500. Chemical shifts for ^{1}H NMR are reported in parts per million down fields from tetramethylsilane (δ) as the internal standard and coupling constants are in hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t =triplet, q = quartet, m = multiplet, br = broad, br. s. = broad singlet. Chemical shifts for ^{13}C NMR were reported in ppm relative to the center line of a triplet at 77.16 ppm for deuteriochloroform and a septet at 39.52 ppm for hexadeuterodimethyl sulfoxide. ^{19}F and ^{11}B spectra were determined on a Avance 400 MHz. ^{19}F NMR chemical shifts were referenced to external trifluorotoluene (-67.73 ppm). ^{11}B NMR chemical shifts were referenced to external $BF_3 \cdot OEt_2$ (0.0 ppm).

Infrared (IR) spectra were recorded on a JASCO FT/IR-620 Spectrophotometer and were reported in wavenumbers (cm^{-1}). High resolution mass spectra (HRMS) were obtained on a Waters GCT Premier using electron ionization (EI) method or a ThermoFisherScientific Orbirtap using electro spray ionization (ESI) method. Analytical thin layer chromatography (TLC) was performed on Merck precoated analytical plates, 0.25 mm thick, silica gel 60 F254. Preparative TLC separations were performed on Merck analytical plates 0.50 mm thick precoated with silica gel 60 F254 or NH_2 F254s. Flash chromatography separations were performed on Merck silica gel 60 (0.040-0.063 mm, 230-400 mesh ASTM).

$Pd(OAc)_2$, $Pd(dba)_2$, and S-phos were purchased from Sigma-Aldrich and were used without further purification. Na_2CO_3 was purchased from Kanto Chemicals Co., Inc., and were used without further purification. All Substrates of Table **2** and **3** were purchased from Sigma-Aldrich or Tokyo Chemical Industry Co., Ltd., or Kanto Chemicals Co., Inc., and were used without further purification. 1,4-Dioxane dehydrate and Distilled water were purchased from Kanto Chemicals Co., Inc., and were used without further purification.

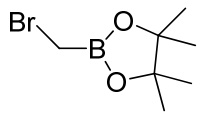
2. Preparation of sodium phthalimidomethyltrifluoroborate 1 (Scheme 1)



Preparation of sodium phthalimidomethyltrifluoroborate 1

Phthalimide (6.59 g, 44.8 mmol) was added to a mixture of sodium hydride (1.79 g, 44.8 mmol, 60% purity) and tetrahydrohuran (300 mL) at 0 ºC. After the reaction mixture was stirred at room temperature for 1 hour, 2-(bromomethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5.38 g, 22.4 mmol, 92% purity) was added at 0 ºC. Then, the reaction mixture was stirred at 60 ºC for 4 hours before cooling to room temperature. To the reaction mixture was added $NaHF_2$ (6.25 g, 101 mmol) at 0 ºC, and then distilled water (300 mL) was added dropwise to the stirring reaction mixture at the same temperature over 1 hour. The reaction mixture stirred at room temperature for 1 hour, concentrated under reduced pressure. The residue was azeotroped with toluene before drying in vacuo over 5 hours. The resulting solid was added acetone (300 mL) and methanol (30.0 mL), and then the reaction mixture was divided into white solid (a) and filtrate (b) by filtration. The white solid (a) was added methanol (100 mL) and acetone (100 mL), the mixture was filtered. The resulting filtrate was added ethyl acetate (50.0 mL), and then added heptane (ca. 150 mL) until the appearacce of the solid. The resulting solid was filtered and dried under reduced pressure to obtain the 1st crop of sodium phthalimidomethyltrifluoroborate **1** as a white solid (1.19 g, 4.74 mmol, 21.2%). The filtrate (b) was added ethyl acetate (100 mL), and then filtered. Ethyl acetate (50.0 mL) was added to the filtrate until the appearacce of the solid, the mixture was divided into solid (c) and filtrate (d) by filtration. The solid (c) was dried under reduced pressure to obtain the 2nd crop of sodium phthalimidomethyltrifluoroborate **1** (826 mg, 3.29 mmol, 17.7%). Ethyl acetate (100 mL) was added to the resulting filtrate (d) until the appearacce of the solid, the mixture was divided into solid (e) and filtrate (f) by filtration. The solid (e) was dried under reduced pressure to obtain the 3rd crop of sodium phthalimidomethyltrifluoroborate **1** (588 mg, 2.34 mmol, 10.5%). The analytical data was consistent with each other among 1st crop, 2nd crop, and 3rd crop of sodium phthalimidomethyltrifluoroborate **1**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.72 (s, 4H), 2.56 (q, J=5.12 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.3, 133.5, 132.4, 122.1; ^{19}F NMR (376.5 MHz, DMSO-d_6) δ -147.0; ^{11}B NMR (128.4 MHz, DMSO-d_6) δ 3.1; IR (ATR, cm^{-1}) 1772, 1706, 1465, 1435, 1401, 1319, 1278, 1188, 1086, 956, 769, 722; HRMS (ESI-) calcd for $C_9H_6BF_3NO_2^-$ $(M-H)^-$ 228.0448, found 228.0438.



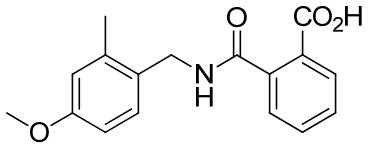
Preparation of 2-(bromomethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane[1]

To a mixture of triisopropyl borate (20.0 g, 110 mmol), dibromomethane (8.60 mL, 120 mmol) and tetrahydrofuran (150 mL) was added *n*-butyllithium (2.6M n-hexane solution, 39 mL 100 mmol) at -78 ºC (external temperature) over 1.5 hours. The reaction mixture was stirred at the same temperature for 1.5 hours, and then the reaction mixture was stirred at room temperature for 2 hours. After the mixture was cooled at 0 ºC (external temperature), to the reaction mixture was added methanesulfonic acid (6.50 mL, 100 mmol), and then the reaction mixture was stirred at room temperature for 1 hour. After the mixture was cooled at 0 ºC (external temperature), to the reaction mixture was added pinacol (12.0 g, 100 mmol), and then the reaction mixture was stirred at room temperature for 1 hour. After the solvent was concentrated, the resulting residue was distilled under reduce pressure (74-76 ºC, 8 mmHg), to obtain the title compound (16.0 g, 72.4 mmol, 72.4%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 2.57 (s, 2H), 1.27 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 84.6, 24.7; IR (ATR, cm^{-1}) 2979, 1415, 1372, 1336, 1272, 1214, 1135, 1055, 967, 886, 845, 673; HRMS (EI+) calcd for $C_7H_{14}BBrO_2$ $(M)^+$ 220.0270, found 220.0315.

3. General experimental procedure for Suzuki-Miyaura cross-coupling reactions with borate 1 (Table1)

A Biotage microwave vial was charged with 2-bromo-4-methoxytoluene or 4-chloro-3-methylanisole, $Pd(OAc)_2$ or $Pd(dba)_2$, S-phos, base, sodium phthalimidomethyltrifluoroborate **1**, 1,4-dioxane/distilled water (2/1). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours. The reaction mixture was cooled to room temperature. The reaction mixture was added water and chloroform. Organic layer was concentrated under reduced pressure, then added dibenzyl ether (δ 4.6 (s, 4H) in $CDCl_3$) as the internal standard, and yields of compounds **2–4** were determined by ^{1}HNMR ($CDCl_3$). Aqueous layer was added 1N-HCl aq. and chloroform, then extracted with chloroform (x 3). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was added dibenzyl ether (δ 4.5 (s, 4H) in DMSO-d_6) or triphenyl methane (δ 5.5 (s, 1H) in DMSO-d_6) as the internal standard, and yield of compound **5** was determined by ^{1}HNMR (DMSO-d_6).

4. Experimental procedure for preparing compounds 6a–6e (Table 2)



Preparation of 2-{[(4-methoxy-2-methylphenyl)methyl]carbamoyl}benzoic acid (6a) from 4-chloro-3-methylanisole

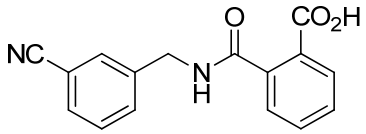
A Biotage microwave vial was charged with 4-chloro-3-methylanisole (31.3 mg, 0.200 mmol), $Pd(OAc)_2$ (2.24 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was washed with diethyl ether/heptane solution to afford **6a** (51.0 mg, 85.1%).

^{1}H NMR (400 MHz, DMSO-*d6*) δ 12.93 (br. s., 1H), 8.63 (t, *J*=5.49 Hz, 1H), 7.74 (d, *J*=7.68 Hz, 1H), 7.36-7.62 (m, 3H), 7.25 (d, *J*=8.42 Hz, 1H), 6.61-6.79, (m, 2H), 4.31 (d, *J*=5.86 Hz, 2H), 3.70 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.3, 168.1, 158.2, 138.5, 137.2, 131.1, 131.0, 129.3, 129.1, 129.1, 129.0, 127.8, 115.4, 110.8, 55.0, 40.3, 19.0; IR (ATR, cm^{-1}) 3337, 1698, 1650, 1577, 1531, 1420, 1299, 1252; HRMS (ESI+) calcd for $C_{17}H_{18}NO_4$ $(M+H)^+$ 300.1230, found 300.1228.

Preparation of 2-{[(4-methoxy-2-methylphenyl)methyl]carbamoyl}benzoic acid (6a) from 2-bromo-4-methoxyltoluene

A Biotage microwave vial was charged with 2-bromo-4-methoxytoluene (40.2 mg, 0.200 mmol), $Pd(dba)_2$ (5.75 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform (x 3). Organic layers

were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was washed with diethyl ether/heptane solution to afford **6b** (54.1 mg, 90.3%).

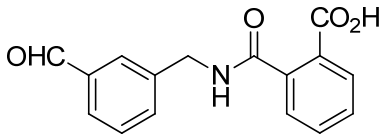


Preparation of 2-{[(3-cyanophenyl)methyl]carbamoyl}benzoic acid (6b)

A Biotage microwave vial was charged with 3-chloro-benzonitrile (27.5 mg, 0.200 mmol), $Pd(OAc)_2$ (2.25 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was washed with ethyl acetate and diethyl ether/heptane solution to afford **6b** (47.1 mg, 84.0%).

Commercially available compound: CAS [1156122-53-0]

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.94 (br. s., 1H), 7.66-7.89 (m, 4H), 7.39-7.62 (m, 4H), 4.47 (d, J=5.85 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.9, 168.0, 141.2, 138.2, 132.2, 131.3, 130.9, 130.7, 130.6, 129.4, 129.4, 129.3, 127.8, 119.0, 111.2, 41.9; HRMS (ESI+) calcd for $C_{16}H_{17}N_2O_3$ $(M+H)^+$ 281.0921, found 281.0914.

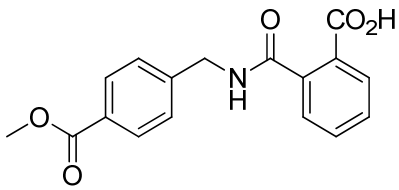


Preparation of 2-{[(3-formylphenyl)methyl]carbamoyl}benzoic acid (6c)

A Biotage microwave vial was charged with 3-chloro-benzaldehyde (21.7 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225 mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq.

HCl (1 N) and chloroform, and then extracted with chloroform (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was washed with ethyl acetate and diethyl ether/heptane solution to afford **6c** (36.5 mg, 85.9%).

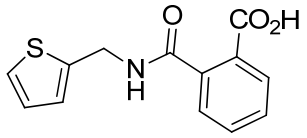
^{1}H NMR (490 MHz, DMSO-d_6) δ 10.03 (br. s., 1H), 8.99 (t, J=6.0 Hz, 1H), 7.93 (br, 1H), 7.74-7.81 (m, 3H), 7.49-7.61 (m, 4H), 4.54 (d, J=6.0 Hz, 2H); ^{13}C NMR (123 MHz, DMSO-d_6) δ 193.2, 168.8, 168.1, 140.8, 138.2, 136.3, 133.5, 131.2, 131.0, 129.3, 129.3, 129.1, 128.4, 127.8, 127.7, 42.2; IR (ATR, cm^{-1}) 3318, 1698, 1650, 1579, 1534, 1419, 1304, 778, 701; HRMS (ESI+) calcd for $C_{16}H_{14}NO_4$ $(M+H)^+$ 284.0917, found 284.0915.



Preparation of 2-({[4-(methoxycarbonyl)phenyl]methyl}carbamoyl)benzoic acid (6d)

A Biotage microwave vial was charged with methyl 4-chlorobenzoate (34.1 mg, 0.200 mmol), $Pd(OAc)_2$ (2.25 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was washed with ethyl acetate and diethyl ether/heptane solution to afford **6d** (54.5 mg, 87.1%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.96 (t, J=5.86 Hz, 1H), 7.91 (d, J=8.05 Hz, 2H), 7.73-7.79 (m, 1H), 7.43-7.62 (m, 5H), 4.50 (d, J=5.86 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.8, 168.1, 166.2, 145.3, 138.1, 131.2, 131.1, 129.3, 129.2, 129.1, 128.0, 127.7, 127.4, 52.1, 42.3; IR (ATR, cm^{-1}) 3315, 1701, 1649, 1533, 1418, 1218, 1109, 750; HRMS (ESI+) calcd for $C_{17}H_{16}NO_5$ $(M+H)^+$ 314.1023, found 314.1023.

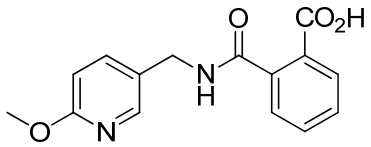


Preparation of 2-[(thiophen-2-ylmethyl)carbamoyl]benzoic acid (6e)

A Biotage microwave vial was charged with methyl 2-bromothiophene (16.3 mg, 0.100 mmol), $Pd(dba)_2$ (2.88 mg, 0.010 mmol), S-phos (4.92 mg, 0.012 mmol), Na_2CO_3 (47.7 mg, 0.450 mmol), sodium phthalimidomethyltrifluoroborate **1** (37.6 mg, 0.150 mmol), 1,4-dioxane (444 μL), distilled water (222 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform/tetrahydrofuran = 5/1 (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (silica gel, ethyl acetate/methanol/acetic acid = 100/10/1) to afford **6e** (16.3 mg, 62.3 %).

Commercially available compound: CAS [332361-08-7]

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.95 (br. s., 1H), 8.94 (t, *J*=5.86 Hz, 1H), 7.75 (dd, *J*=1.28, 7.50 Hz, 1H), 7.46-7.60 (m, 2H), 7.35-7.42 (m, 2H), 7.04 (dd, *J*=0.91, 3.48 Hz, 1H), 6.95 (dd, *J*=3.29, 5.12 Hz, 1H), 4.57 (d, *J*=5.86 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.3, 168.0, 142.4, 138.1, 131.2, 131.0, 129.4, 129.2, 127.7, 126.7, 125.3, 124.9, 37.7; HRMS (ESI+) calcd for $C_{13}H_{12}NO_3S$ $(M)^+$ 262.0520, found 262.0519.



Preparation of 2-{[(6-methoxypyridin-3-yl)methyl]carbamoyl}benzoic acid (6f)

A Biotage microwave vial was charged with methyl 5-chloro-2-methoxypyridine (28.8 mg, 0.200 mmol), $Pd(OAc)_2$ (2.25 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. HCl (1 N) and chloroform, and then extracted with chloroform/tetrahydrofuran = 5/1 (x

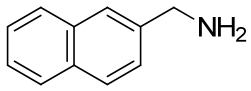
3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (silica gel, ethyl acetate/methanol/acetic acid = 100/10/1) to afford **6f** (24.2 mg, 42.2 %).

Commercially available compound: CAS [1178036-30-0]

^{1}H NMR (400 MHz, DMSO-*d6*) δ 12.96 (br. s., 1H), 8.80 (t, *J*=5.67 Hz, 1H), 8.14 (s, 1H), 7.74 (dd, *J*=8.05, 17.20 Hz, 2H), 7.47-7.60 (m, 2H), 7.42 (d, *J*=7.32 Hz, 1H), 6.76 (d, *J*=8.42 Hz, 1H), 4.36 (d, *J*=5.85 Hz, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 168.0, 162.7, 145.6, 138.9, 138.4, 131.3, 130.8, 129.2, 129.2, 128.0, 127.7, 110.1, 53.1, 39.6; HRMS (ESI+) calcd for $C_{15}H_{15}N_2O_4$ $(M+H)^+$ 267.1026, found 267.1026.

5. Experimental procedure for one-pot aminomethylation of aryl and heteroaryl halides, triflates, mesylates, and tosylates (Table 4 and 5)

One-pot primary aminomethylation of aryl and heteroaryl halides and triflates (Table 4)



Preparation of naphthalen-2-ylmethanamine (7a) from 2-chloronaphthalene[2]

A Biotage microwave vial was charged with 2-chloronaphthalene (24.4 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (20.1 mg, 85.2 %).

Commercially available compound: CAS [118-31-0]
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J*=8.05 Hz, 3H), 7.75 (s, 1H), 7.39-7.51 (m, 3H), 4.03 (s, 2H), 1.56 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.9, 133.7, 132.6, 128.3, 127.8, 127.8, 126.2, 125.9, 125.6, 125.2, 46.8; HRMS (EI+) calcd for $C_{11}H_{11}N$ $(M)^+$ 157.0886, found 157.0965.

Preparation of naphthalen-2-ylmethanamine (7a) from 2-bromonaphthalene

A Biotage microwave vial was charged with 2-bromonaphthalene (42.7 mg, 0.150 mmol), $Pd(dba)_2$ (4.31 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (18.7 mg, 79.3 %).

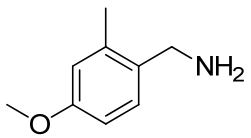
Preparation of naphthalen-2-ylmethanamine (7a) from 2-iodonaphthalene

A Biotage microwave vial was charged with 2-iodonaphthalene (38.1 mg, 0.150 mmol, 95% purity), $Pd(dba)_2$ (4.31 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate,

filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (12.4 mg, 52.6 %).

Preparation of naphthalen-2-ylmethanamine (7a) from 2-naphthyl trifluoromethanesulfonate

A Biotage microwave vial was charged with 2-naphthyl trifluoromethanesulfonate (41.4 mg, 0.150 mmol), $Pd(dba)_2$ (4.31 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 15 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (17.5 mg, 74.2 %).



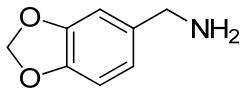
Preparation of (4-methoxy-2-methylphenyl)methanamine (7b)[3]

A Biotage microwave vial was charged with 4-chloro-3-methylanisole (31.3 mg, 0.200 mmol), $Pd(OAc)_2$ (2.25 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (93.5 μL, 1.400 mmol) and 1-propanol (888 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting

aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7b** (25.6 mg, 84.6 %).

Commercially available compound: CAS [21883-14-7]

^{1}H NMR (400 MHz, $CDCl_3$) δ7.19 (d, *J*=9.15 Hz, 1H), 6.67-6.76 (m, 2H), 3.79 (s, 2H), 3.78 (s, 3H), 2.33 (s, 3H), 1.51 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.5, 137.1, 133.7, 128.6, 116.2, 111.1, 55.3, 43.7, 19.2; HRMS (EI+) calcd for $C_9H_{13}NO$ $(M)^+$ 151.0992, found 151.1050.

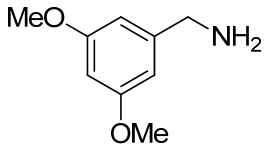


Preparation of 2H-1,3-benzodioxol-5-ylmethanamine (7c)[4]

A Biotage microwave vial was charged with 5-chloro-1,3-benzodioxole (23.5 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7c** (20.2 mg, 89.1 %).

Commercially available compound: CAS [2620-50-0]

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79 (s, 1H), 6.70-6.76 (m, 2H), 5.91 (s, 2H), 3.74 (s, 2H), 1.52 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 146.4, 137.5, 120.1, 108.2, 107.8, 100.9, 46.4; HRMS (EI+) calcd for $C_8H_9NO_2$ $(M)^+$ 151.0628, found 151.0704.

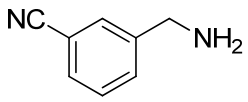


Preparation of (3,5-dimethoxyphenyl)methanamine (7d)[5]

A Biotage microwave vial was charged with 3,5-dimethoxychlorobenzene (25.9 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7d** (22.8 mg, 90.9 %).

Commercially available compound: CAS [34967-24-3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.47 (d, *J*=2.56 Hz, 2H), 6.35 (t, *J*=2.38 Hz, 1H), 3.81 (s, 2H), 3.79 (s, 6H), 1.58 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 146.0, 104.9, 98.8, 55.4, 46.7; HRMS (EI+) calcd for $C_9H_{13}NO_2$ $(M)^+$ 167.0941, found 167.1048.



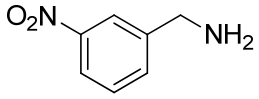
Preparation of 3-(aminomethyl)benzonitrile (7e)[6]

A Biotage microwave vial was charged with 3,5-dimethoxychlorobenzene (21.0 mg, 0.150 mmol, 98% purity), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was

stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7e** (16.1 mg, 81.2 %).

Commercially available compound: CAS [10406-24-3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.56 (dd, *J*=7.68, 18.66 Hz, 2H), 7.40-7.48 (m, 1H), 3.93 (s, 2H), 1.56 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 131.7, 130.7, 130.5, 129.3, 119.0, 112.4, 45.6.; HRMS (EI+) calcd for $C_8H_8N_2$ $(M)^+$ 132.0687, found 132.0758.



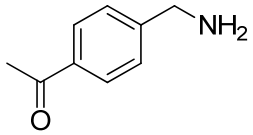
Preparation of (3-nitrophenyl)methanamine (7f)[7]

A Biotage microwave vial was charged with 3-nitorochlorobenzene (23.6 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7f** (18.9 mg, 82.8 %).

Commercially available compound: CAS [7409-18-9]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (s, 1H), 8.10 (dd, *J*=1.28, 8.23 Hz, 1H), 7.68 (d, *J*=7.68 Hz, 1H), 7.51 (t, *J*=7.87 Hz, 1H), 4.01 (s, 2H), 1.59 (s, 2H); ^{13}C NMR (100 MHz,

$CDCl_3$) δ 148.5, 145.3, 133.4, 129.4, 122.0, 121.9, 45.7.; HRMS (EI+) calcd for $C_7H_8N_2O_2$ $(M)^+$ 152.0580, found 152.0576.

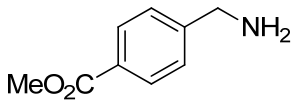


Preparation of 1-[4-(aminomethyl)phenyl]ethan-1-one (7g)

A Biotage microwave vial was charged with 4'-chlorobenzene (31.9 mg, 0.200 mmol, >97% purity), $Pd(OAc)_2$ (2.25 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (93.5 μL, 1.400 mmol) and 1-propanol (888 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7g** (22.4 mg, 75.1 %).

Commercially available compound: CAS [87171-25-3]

^{1}H NMR (490 MHz, $CDCl_3$) δ 7.94 (d, *J*=7.8 Hz, 2H), 7.42 (d, *J*=8.3 Hz, 2H), 3.95 (s, 2H), 2.60 (s, 3H), 1.48 (br. s., 2H); ^{13}C NMR (123 MHz, $CDCl_3$) δ 197.9, 148.8, 135.9, 128.7, 127.2, 46.2, 26.7.; HRMS (ESI+) calcd for $C_9H_{12}NO$ $(M)^+$ 150.0913, found 150.0913.



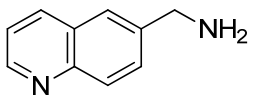
Preparation of methyl 4-(aminomethyl)benzoate (7h)[8]

After isolation of 2-({[4-(methoxycarbonyl)phenyl]methyl}carbamoyl)benzoic acid (**6d**) from methyl 4-chlorobenzoate (0.2 mmol) , a Biotage microwave vial was charged with 2-({[4-(methoxycarbonyl)phenyl]methyl}carbamoyl)benzoic acid, ethylenediamine (93.5 μL, 1.400 mmol) and *t*-butanol (1.5 mL). The test tube was

sealed with a cap, and the reaction mixture was stirred under reflux for 24 hours before cooling to room temperature. The reaction mixture was filtered, concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7h** (23.0 mg, 69.5 %).

Commercially available compound: CAS [18469-52-8]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J*=8.05 Hz, 2H), 7.39 (d, *J*=8.05 Hz, 2H), 3.94 (s, 2H), 3.91 (s, 3H), 1.54 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 148.5, 130.0, 128.8, 127.0, 52.2, 46.3; HRMS (EI+) calcd for $C_9H_{11}NO_2$ $(M)^+$ 165.0790, found 165.0791.



Preparation of quinolin-6-ylmethanamine (7i)[9] from 6-chloroquinoline

A Biotage microwave vial was charged with 6-chloroquinoline (24.5 mg, 0.150 mmol), $Pd(OAc)_2$ (1.68 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225 mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol/ = 15/1) to afford **7i** (19.1 mg, 80.5 %).

Commercially available compound: CAS [99071-54-2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (dd, *J*=1.65, 4.21 Hz, 1H), 8.02-8.14 (m, 2H), 7.71 (s, 1H), 7.65 (dd, *J*=2.01, 8.60 Hz, 1H), 7.36 (dd, *J*=4.21,
8.23 Hz, 1H), 4.04 (s, 2H), 1.69 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 147.5, 141.4, 135.8, 129.5, 129.4, 128.2, 124.7, 121.2, 46.2; HRMS (EI+) calcd for $C_{10}H_{10}N_2$ $(M)^+$ 158.0838, found 158.0881.

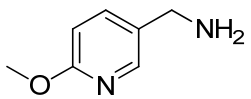
Preparation of quinolin-6-ylmethanamine (7i) from 6-bromoquinoline

A Biotage microwave vial was charged with 6-bromoquinoline (31.2 mg, 0.150 mmol), $Pd(dba)_2$ (4.31 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5

mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225 mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol/ = 15/1) to afford **7i** (18.6 mg, 78.4 %).

Preparation of quinolin-6-ylmethanamine (7i) from quinolin-6-yl trifluoromethanesulfonate

A Biotage microwave vial was charged with quinolin-6-yl trifluoromethanesulfonate (42.9 mg, 0.150 mmol, 97% purity), $Pd(dba)_2$ (4.31 mg, 0.008 mmol), S-phos (7.39 mg, 0.018 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225 mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol/ = 15/1) to afford **7i** (18.1 mg, 76.3 %).



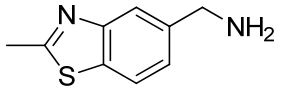
Preparation of (2-methyl-1,3-benzothiazol-5-yl)methanamine (7j)

To a solution of 5-Chloro-2-methoxypyridine (86.1 mg, 0.600 mmol), $Pd(OAc)_2$ (13.5 mg, 0.060 mmol), S-phos (59.1 mg, 0.144 mmol), Na_2CO_3 (286 mg, 2.700 mmol), sodium phthalimidomethyltrifluoroborate **1** (226 mg, 0.900 mmol), 1,4-dioxane (4.0 mL), distilled water (2.0 mL) was stirred under reflux for 48 hours before cooling to

room temperature. The reaction mixture was added ethylenediamine (281 μL, 4.20 mmol) and 1-propanol (4.0 mL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol/ = 15/1) to afford **7i** (50.6 mg, 61.0 %).

Commercially available compound: CAS [262295-96-5]

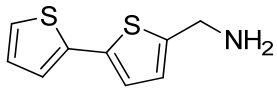
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J*=2.20 Hz, 1H), 7.57 (dd, *J*=2.20, 8.42 Hz, 1H), 6.73 (d, *J*=8.78 Hz, 1H), 3.93 (s, 3H), 3.81 (s, 2H), 1.50 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 138.4, 131.3, 131.3, 110.9, 53.5, 43.5; HRMS (EI+) calcd for $C_7H_{10}N_2O$ $(M)^+$ 138.0788, found 138.0796.



Preparation of (2-methyl-1,3-benzothiazol-5-yl)methanamine (7k)

A Biotage microwave vial was charged with 5-chloro-2-methyl-1,3-benzothiazole (18.4 mg, 0.100 mmol), $Pd(OAc)_2$ (1.12 mg, 0.005 mmol), S-phos (4.93 mg, 0.012 mmol), Na_2CO_3 (47.7 mg, 0.450 mmol), sodium phthalimidomethyltrifluoroborate **1** (37.6 mg, 0.150 mmol), 1,4-dioxane (444 μL), distilled water (222 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (46.7 μL, 0.700 mmol) and 1-propanol (444 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol/ = 20/1) to afford **7k** (14.8 mg, 83.0 %).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J*=0.73 Hz, 1H), 7.77 (d, *J*=8.05 Hz, 1H), 7.32 (dd, *J*=1.46, 8.05 Hz, 1H), 4.00 (s, 2H), 2.83 (s, 3H), 1.63 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.5, 153.9, 141.8, 134.0, 124.4, 121.5, 120.6, 46.5, 20.3; IR (ATR, cm^{-1}) 3283, 2918, 1568, 1521, 1453, 1422, 1373, 1328, 1300, 1171, 890, 806; HRMS (EI+) calcd for $C_9H_{10}N_2S$ $(M)^+$ 178.0559, found 178.0625.



Preparation of [5-(thiophen-2-yl)thiophen-2-yl]methanamine (7l)[10]

A Biotage microwave vial was charged with 2-bromo-5-(thiophen-2-yl)thiophene (49.0 mg, 0.200 mmol), $Pd(dba)_2$ (5.75 mg, 0.010 mmol), S-phos (9.85 mg, 0.024 mmol), Na_2CO_3 (95.4 mg, 0.900 mmol), sodium phthalimidomethyltrifluoroborate **1** (75.3 mg, 0.300 mmol), 1,4-dioxane (888 μL), distilled water (444 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 36 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (93.5 μL, 1.400 mmol) and 1-propanol (888 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7l** (22.4 mg, 57.3 %).

Commercially available compound: CAS [4380-96-5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (dd, *J*=0.73, 5.12 Hz, 1H), 7.10-7.15 (m, 1H), 6.95-7.03 (m, 2H), 6.80 (d, *J*=3.29 Hz, 1H), 4.02 (s, 2H), 1.63 (br. s., 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.8, 137.8, 136.1, 127.9, 124.3, 124.2, 123.5, 123.5, 41.7.; HRMS (EI+) calcd for $C_9H_9NS_2$ $(M)^+$ 180.0786, found 180.0822.

One-pot primary aminomethylation of aryl and heteroaryl mesylates and tosylates (Table 5)

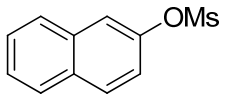
Preparation of naphthalen-2-ylmethanamine (7a) from naphthalene-2-yl methanesulfonate

A Biotage microwave vial was charged with naphthalene-2-yl methanesulfonate (33.3 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine

(70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (18.9 mg, 80.1 %).

Preparation of naphthalen-2-ylmethanamine (7a) from naphthalen-2-yl 4-methylbenzene-1-sulfonate

A Biotage microwave vial was charged with naphthalen-2-yl 4-methylbenzene-1-sulfonate (44.8 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7a** (20.1 mg, 85.2 %).



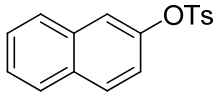
Preparation of naphthalene-2-yl methanesulfonate[1]

To a solution of 2-naphtol (3.00 g, 20.8 mmol) and pyridine (10.0 mL) in dichloromethane (20.0 mL) was added methanesulfonyl chloride (2.10 mL, 27.0 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with saturated aqueous ammonium chloride, and the

resulting mixture was extracted with ethyl acetate. The combined organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column chromatography (heptane/ethyl acetate = 4/1 to 2/1) to afford naphthalene-2-yl methanesulfonate (4.10 g, 89.1%).

Commercially available compound: CAS [10290-91-2]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.92 (m, 3H), 7.76 (d, *J*=2.20 Hz, 1H), 7.49-7.58 (m, 2H), 7.41 (ddd, *J*=1.10, 2.38, 8.97 Hz, 1H), 3.18 (d, *J*=1.10 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 133.7, 132.2, 130.4, 128.0, 128.0, 127.3, 126.7, 120.9, 119.6, 37.5; HRMS (EI+) calcd for $C_{11}H_{10}O_3S$ $(M)^+$ 222.0351, found 222.0370.



Preparation of naphthalene-2-yl 4-methylbenzenesulfonate[1]

To a solution of 2-naphtol (3.00 g, 20.8 mmol) and pyridine (10.0 mL) in CH_2Cl_2 (20.0 mL) was added p-toluenesulfonyl chloride (5.20 g, 27.3 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with saturated aqueous ammonium chloride, and the resulting mixture was extracted with ethyl acetate. The combined organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column chromatography (heptane/ethyl acetate = 4/1 to 2/1) to afford naphthalene-2-yl 4-methylbenzenesulfonate (3.20 g, 51.6%).

Commercially available compound: CAS [7385-85-5]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77-7.84 (m, 1H), 7.70-7.77 (m, 4H), 7.44-7.51 (m, 3H), 7.29 (d, *J*=8.05 Hz, 2H), 7.09 (dd, *J*=2.38, 8.97 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.3, 145.5, 133.6, 132.6, 132.0, 129.9, 129.9, 128.7, 128.0, 127.9, 127.0, 126.5, 121.3, 120.1, 21.9; HRMS (EI+) calcd for $C_{17}H_{14}O_3S$ $(M)^+$ 298.0664, found 298.0612.

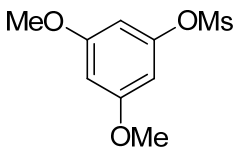
Preparation of (3,5-dimethoxyphenyl)methanamine (7d) from 3,5-dimethoxyphenyl methanesulfonate

A Biotage microwave vial was charged with 3,5-dimethoxyphenyl methanesulfonate (34.8 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours

before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7d** (17.5 mg, 70.0 %).

Preparation of (3,5-dimethoxyphenyl)methanamine (7d) from 3,5-dimethoxyphenyl 4-methylbenzene-1-sulfonate

A Biotage microwave vial was charged with 3,5-dimethoxyphenyl 4-methylbenzene-1-sulfonate (46.3 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7d** (20.1 mg, 80.1 %).

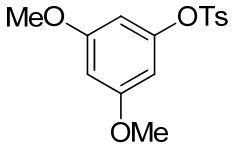


Preparation of 3,5-dimethoxyphenyl methanesulfonate

To a solution of 3,5-dimethoxyphenol (1.50 g, 9.54 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added methanesulfonyl chloride (964 mL, 12.4 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The

reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column chromatography (heptane/ethyl acetate = 2/1 to 3/2) to afford 3,5-dimethoxyphenyl methanesulfonate (2.14 g, 96.8%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.44 (d, *J*=2.20 Hz, 2H), 6.38-6.42 (m, 1H), 3.78 (s, 6H), 3.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.5, 150.8, 100.6, 99.5, 55.7, 37.4; HRMS (EI+) calcd for $C_9H_{12}O_5S$ $(M)^+$ 232.0400, found 232.0484.



Preparation of 3,5-dimethoxyphenyl 4-methylbenzene-1-sulfonate[11]

To a solution of 3,5-dimethoxyphenol (1.50 g, 9.54 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added p-toluenesulfonyl chloride (4.40 g, 22.9 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by NH silica gel column chromatography (heptane/ethyl acetate = 4/1 to 2/1) to afford 3,5-dimethoxyphenyl 4-methylbenzene-1-sulfonate (2.65 g, 88.3%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J*=8.42 Hz, 2H), 7.31 (d, *J*=8.05 Hz, 2H), 6.32 (t, *J*=2.20 Hz, 1H), 6.14 (d, *J*=2.20 Hz, 2H), 3.68 (s, 7H),
2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 151.1, 145.5, 132.6, 129.8, 128.7, 100.9, 99.5, 55.6, 21.8; HRMS (ESI+) calcd for $C_{15}H_{17}O_5S$ $(M)^+$ 309.0791, found 309.0781.

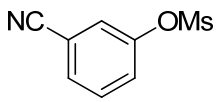
Preparation of 3-(aminomethyl)benzonitrile (7e) from 3-cyanophenyl methanesulfonate

A Biotage microwave vial was charged with 3-cyanophenyl methanesulfonate (29.6 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050

mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7e** (6.94 mg, 35.0 %).

Preparation of 3-(aminomethyl)benzonitrile (7e) from 3-cyanophenyl 4-methylbenzene-1-sulfonate

A Biotage microwave vial was charged with 3-cyanophenyl 4-methylbenzene-1-sulfonate (41.0 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added aq. HCl (2 N) and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added aq. NaOH (2 N) and chloroform, then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by NH silica gel column chromatography (ethyl acetate/methanol = 20/1) to afford **7e** (8.92 mg, 45.0 %).

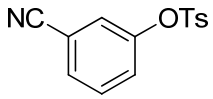


Preparation of 3-cyanophenyl methanesulfonate[12]

To a solution of 3-hydroxybenzonitrile (1.20 g, 10.1 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added methanesulfonyl chloride (1.60 mL, 20.2 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with

ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column chromatography (heptane/ethyl acetate = 2/1) to afford 3-cyanophenyl methanesulfonate (1.92 g, 97.0%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62-7.68 (m, 1H), 7.53-7.61 (m, 3H), 3.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 131.2, 131.2, 127.2, 125.9, 117.4, 114.3, 38.1; HRMS (EI+) calcd for $C_8H_7NO_3S$ $(M)^+$ 197.0141, found 197.0194.



Preparation of 3-cyanophenyl 4-methylbenzene-1-sulfonate[12]

To a solution of 3-hydroxybenzonitrile (1.20 g, 10.1 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added p-toluenesulfonyl chloride (3.80 g, 20.2 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by NH silica gel column chromatography (heptane/ethyl acetate = 4/1 to 3/1) to afford 3-cyanophenyl 4-methylbenzene-1-sulfonate (1.95 g, 70.8%).

Commercially available compound: CAS [49584-07-8]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J*=8.42 Hz, 2H), 7.53-7.61 (m, 1H), 7.45 (t, *J*=7.87 Hz, 1H), 7.36 (d, *J*=8.42 Hz, 2H), 7.28-7.33 (m, 1H), 7.24-7.27 (m, 1H), 2.48 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.7, 146.3, 131.7, 130.9, 130.9, 130.2, 128.5, 127.5, 126.1, 117.4, 113.8, 21.9; HRMS (EI+) calcd for $C_{14}H_{11}NO_3S$ $(M)^+$ 273.0454, found 273.0456.

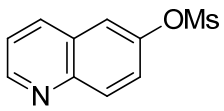
Preparation of quinolin-6-ylmethanamine from 6-chloroquinoline (7i) from quinolin-6-yl methanesulfonate

A Biotage microwave vial was charged with quinolin-6-yl methanesulfonate (33.5 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux

for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol = 15/1) to afford **7i** (16.9 mg, 71.2 %).

Preparation of quinolin-6-ylmethanamine from 6-chloroquinoline (7g) from quinolin-6-yl 4-methylbenzene-1-sulfonate

A Biotage microwave vial was charged with quinolin-6-yl 4-methylbenzene-1-sulfonate (44.9 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol = 15/1) to afford **7i** (17.2 mg, 72.5 %).

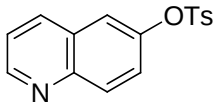


Preparation of quinolin-6-yl methanesulfonate[13]

To a solution of quinolin-6-ol (1.00 g, 6.89 mmol) and pyridine (2.00 mL) in dichloromethane (20.0 mL) was added methanesulfonyl chloride (1.10 mL, 13.8 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column chromatography (heptane/ethyl acetate = 2/3 to 1/2) to afford quinolin-6-yl methanesulfonate (1.20 g, 65.0%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.96 (dd, *J*=1.65, 4.21 Hz, 1H), 8.14-8.21 (m, 2H), 7.78

(d, *J*=2.56 Hz, 1H), 7.63 (dd, *J*=2.56, 9.15 Hz, 1H), 7.47 (dd, *J*=4.21, 8.23 Hz, 1H), 3.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 146.8, 146.8, 136.1, 132.1, 128.6, 124.4, 122.2, 119.5, 37.8; HRMS (EI+) calcd for $C_{10}H_9NO_3S$ $(M)^+$ 223.0298, found 223.0342.



Preparation of quinolin-6-yl 4-methylbenzene-1-sulfonate[14]

To a solution of quinolin-6-ol (1.00 g, 6.89 mmol) and pyridine (2.00 mL) in dichloromethane (20.0 mL) was added p-toluenesulfonyl chloride (2.60 g, 13.8 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by NH silica gel column chromatography (heptane/ethyl acetate = 3/2 to 1/1) to afford quinolin-6-yl 4-methylbenzene-1-sulfonate (1.73 g, 84.0%).

Commercially available compound: CAS [426265-40-9]

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.92 (dd, *J*=1.65, 4.21 Hz, 1H), 8.09 (dd, *J*=1.10, 8.42 Hz, 1H), 8.01 (d, *J*=9.15 Hz, 1H), 7.73 (d, *J*=8.42 Hz, 2H), 7.55 (d, *J*=2.56 Hz, 1H), 7.42 (dd, *J*=4.21, 8.23 Hz, 1H), 7.22-7.34 (m, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 147.3, 146.7, 145.8, 136.1, 132.2, 131.5, 130.0, 128.6, 128.4, 124.7, 122.0, 120.1, 21.8; HRMS (ESI+) calcd for $C_{16}H_{14}NO_3S$ $(M)^+$ 300.0689, found 300.0680.

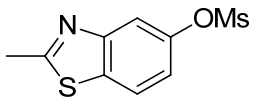
Preparation of (2-methyl-1,3-benzothiazol-5-yl)methanamine (7k) from 2-methyl-1,3-benzothiazol-5-yl methanesulfonate

A Biotage microwave vial was charged with 2-methyl-1,3-benzothiazol-5-yl methanesulfonate (36.5 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced

pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol = 15/1) to afford **7k** (18.7 mg, 69.9 %).

Preparation of (2-methyl-1,3-benzothiazol-5-yl)methanamine (7h) from 2-methyl-1,3-benzothiazol-5-yl 4-methylbenzene-1-sulfonate

A Biotage microwave vial was charged with 2-methyl-1,3-benzothiazol-5-yl 4-methylbenzene-1-sulfonate (47.9 mg, 0.150 mmol), $Pd(OAc)_2$ (2.47 mg, 0.011 mmol), S-phos (11.1 mg, 0.027 mmol), Na_2CO_3 (71.5 mg, 0.675 mmol), sodium phthalimidomethyltrifluoroborate **1** (56.5 mg, 0.225mmol), 1,4-dioxane (666 μL), distilled water (333 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added ethylenediamine (70.1 μL, 1.050 mmol) and 1-propanol (666 μL), and then the reaction mixture was stirred under reflux for 24 hours. The reaction mixture was cooled to room temperature, and then filtered, concentrated under reduced pressure. The residue was added chloroform and water, and then extracted with chloroform (x 2). Organic layers were combined and dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl acetate/methanol = 15/1) to afford **7k** (20.0 mg, 74.9 %).

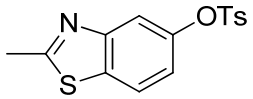


Preparation of 2-methyl-1,3-benzothiazol-5-yl methanesulfonate[13]

To a solution of 2-methyl-1,3-benzothiazol-5-ol (1.50 g, 10.0 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added methanesulfonyl chloride (1.60 mL, 20.0 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by NH silica gel column chromatography (heptane/ethyl acetate = 2/1 to 1/1) to afford 2-methyl-1,3-benzothiazol-5-yl methanesulfonate (2.14 g, 88.1%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74-7.94 (m, 2H), 7.33 (dd, *J*=2.20, 8.78 Hz, 1H), 3.19

(s, 3H), 2.85 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 154.1, 147.7, 134.7, 122.4, 119.3, 115.8, 37.4, 20.4; HRMS (EI+) calcd for $C_9H_9NO_3S_2$ $(M)^+$ 243.0018, found 243.0034



Preparation of 2-methyl-1,3-benzothiazol-5-yl 4-methylbenzene-1-sulfonate[15]

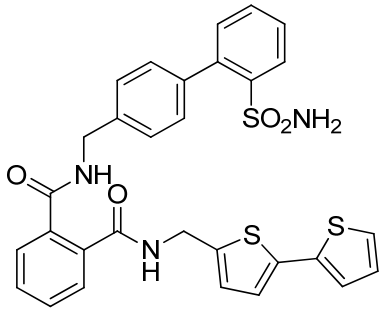
To a solution of 2-methyl-1,3-benzothiazol-5-ol (1.20 g, 8.00 mmol) and pyridine (3.00 mL) in dichloromethane (20.0 mL) was added p-toluenesulfonyl chloride (3.10 g, 16.0 mmol) at 0 ºC. The reaction mixture was stirring at room temperature for 12 hours. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by NH silica gel column chromatography (heptane/ethyl acetate = 4/1 to 2/1) to afford 2-methyl-1,3-benzothiazol-5-yl 4-methylbenzene-1-sulfonate (1.64 g, 64.3%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66-7.80 (m, 3H), 7.45 (d, *J*=2.56 Hz, 1H), 7.30 (d, *J*=8.42 Hz, 2H), 7.11 (dd, *J*=2.38, 8.60 Hz, 1H), 2.81 (s, 3H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 153.8, 148.2, 145.6, 134.4, 132.2, 130.0, 128.6, 122.0, 119.9, 116.1, 21.9, 20.4; HRMS (ESI+) calcd for $C_{15}H_{14}NO_3S_2$ $(M)^+$ 320.0410, found 320.0400.

6.Experimental procedure for preparing compounds 9 and 11

Preparation of 2-(4-chlorophenyl)benzene-1-sulfonamide (9)[16]

To a solution of 2-bromobenzene-1-sulfonamide (94.4 mg, 0.40 mmol), $Pd(P\textit{t}\text{-}Bu_3)_2$ (10.2 mg, 0.02 mmol), 4-chlorobenzene boronic acid (187.6 mg, 1.20 mmol), Cs_2CO_3 (260.8 mg, 0.80 mmol), 1,4-dioxane (4.00 mL), and distilled water (2.0 mL) was stirred under reflux for 15 h before cooling to room temperature. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate. The organic extracts were washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residual oil was purified by silica gel column

chromatography (heptane/ethyl acetate = 2/1 to 1/1) to afford 2-(4-chlorophenyl)benzene-1-sulfonamide (212 mg).
^{1}H NMR (400 MHz, DMSO-*d6*) δ 8.02 (dd, *J*=1.46, 7.68 Hz, 1H), 7.54-7.64 (m, 2H), 7.40-7.47 (m, 2H), 7.35-7.40 (m, 2H), 7.28-7.33 (m, 3H); ^{13}C NMR (100 MHz, DMSO-*d6*) δ 142.3, 138.8, 138.6, 132.3, 131.6, 131.1, 131.1, 128.0, 127.6, 127.3; HRMS (EI+) calcd for $C_{12}H_{10}ClNO_2S$ $(M+NH_4)^+$ 267.0115, found 267.0112.



Preparation of 1-N-{[4-(2-sulfamoylphenyl)phenyl]methyl}-2-N-{[5-(thiophen-2-yl)thiophen-2-yl]methyl}benzene-1,2-dicarboxamide (11)[10]

A Biotage microwave vial was charged with 2-(4-chlorophenyl)benzene-1-sulfonamide (20.6 mg, 0.077 mmol), $Pd(OAc)_2$ (0.86 mg, 0.004 mmol), S-phos (3.78 mg, 0.009 mmol), Na_2CO_3 (36.7 mg, 0.347 mmol), sodium phthalimidomethyltrifluoroborate **1** (29.0 mg, 0.116 mmol), 1,4-dioxane (444 μL), distilled water (222 μL). The test tube was sealed with a cap, and the reaction mixture was stirred under reflux for 48 hours before cooling to room temperature. The reaction mixture was added water and chloroform, and then aqueous layer was washed with chloroform (x 2). Resulting aqueous layer was added 1N-HCl aq. and chloroform, and then extracted with chloroform/tetrahydrofuran = 5/1 (x 3). Organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The crude mixture was used in the next step without further purification.

The crude mixture was added **7j** (18.0 mg, 0.092 mmol), EDC•HCl (22.2 mg, 0.116 mmol), HOBt•H_2O (17.5 mg, 0.116 mmol), *i*-Pr_2NEt (26.8 μL, 0.154 mmol), and tetrahydrofuran (2.0 mL). The reaction mixture was stirred at room temperature for 10 h. The reaction mixture was quenched with water, and the resulting mixture was extracted with ethyl acetate/tetrahydrofuran (x 2). The organic layers were combined and washed with brine, dried over magnesium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by preparative TLC (NH silica gel, ethyl

acetate/methanol = 10/1) to afford **11** (24.0 mg, 53.1 %).
^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 8.97 (t, *J*=5.86 Hz, 1H), 8.86 (t, *J*=5.86 Hz, 1H), 7.99-8.05 (m, 1H), 7.48-7.60 (m, 6H), 7.45 (d, *J*=5.12 Hz, 1H), 7.33-7.39 (m, 4H), 7.25-7.30 (m, 1H), 7.21 (d, *J*=3.66 Hz, 1H), 7.16 (br. s., 2H), 7.11 (d, *J*=3.66 Hz, 1H), 7.04 (dd, *J*=3.84, 4.94 Hz, 1H), 7.00 (d, *J*=3.66 Hz, 1H), 4.53 (d, *J*=5.49 Hz, 2H), 4.47 (d, *J*=6.22 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-*d*$_6$) δ 168.1, 168.1, 142.2, 141.9, 139.8, 138.5, 138.4, 136.7, 136.4, 135.9, 135.4, 132.5, 131.4, 129.6, 129.4, 129.1, 129.1, 128.3, 127.7, 127.5, 127.3, 126.4, 126.4, 125.1, 123.6, 123.4, 42.2, 37.9; HRMS (ESI+) calcd for $C_{30}H26N3O_4S$ $(M+NH_4)^+$ 605.1340, found 605.1345.

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7. Spectra for Compounds

2012/04/11 12:51:14

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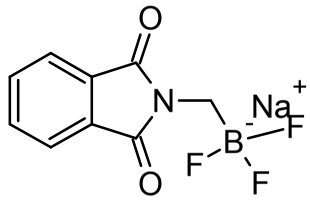
Receiver Gain	20.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20
Temperature (degree C)	AMBIENT TEMPERATURE								

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 7.72 (s, 4H), 2.56 (q, *J*=5.12 Hz, 2H)



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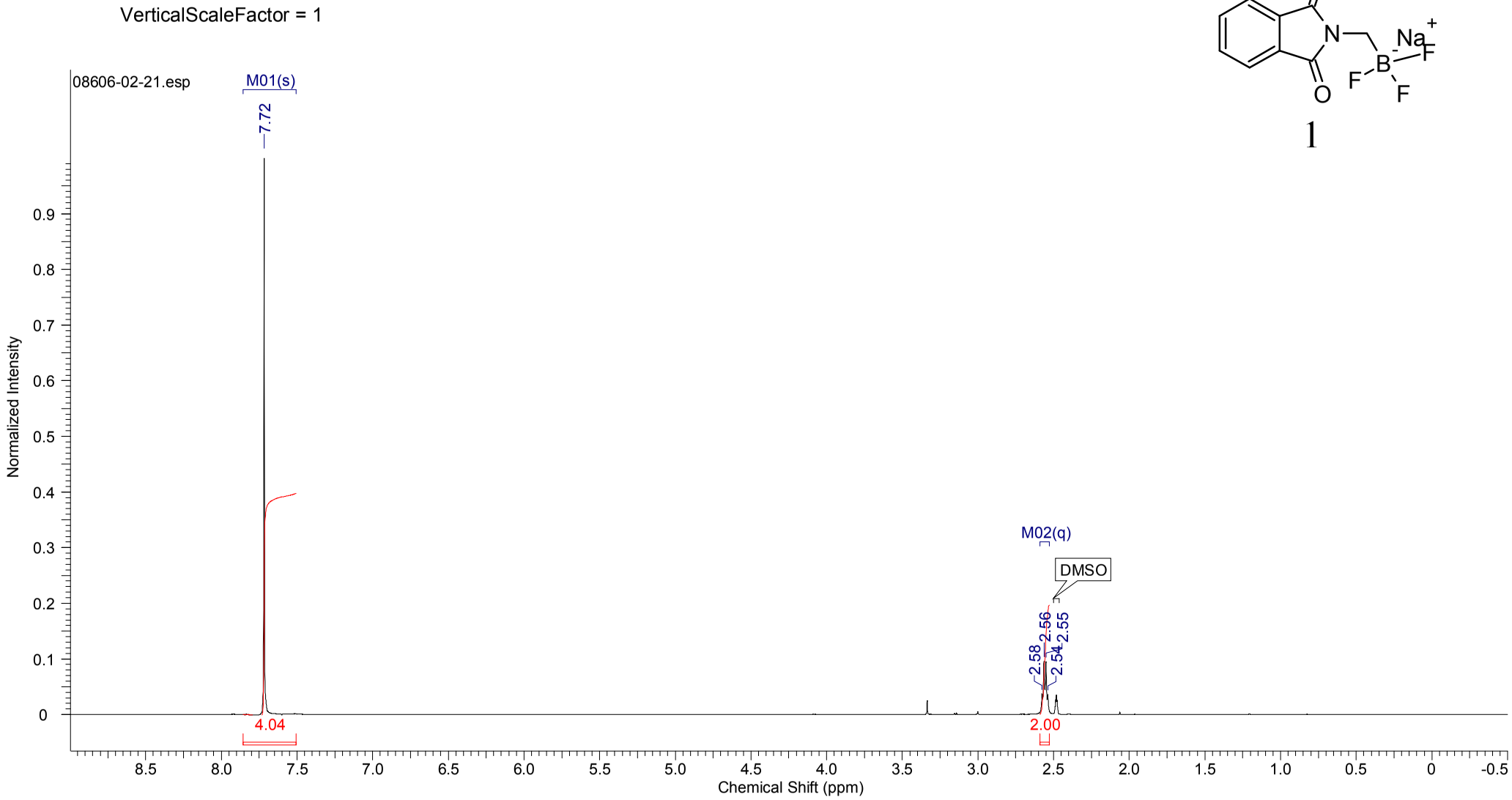

13C OBSERVE

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Ambient temperature
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288 repetitions
OBSERVE C13, 100.5611422 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
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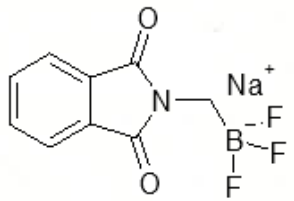
Total time 1 hr, 51 min, 37 sec



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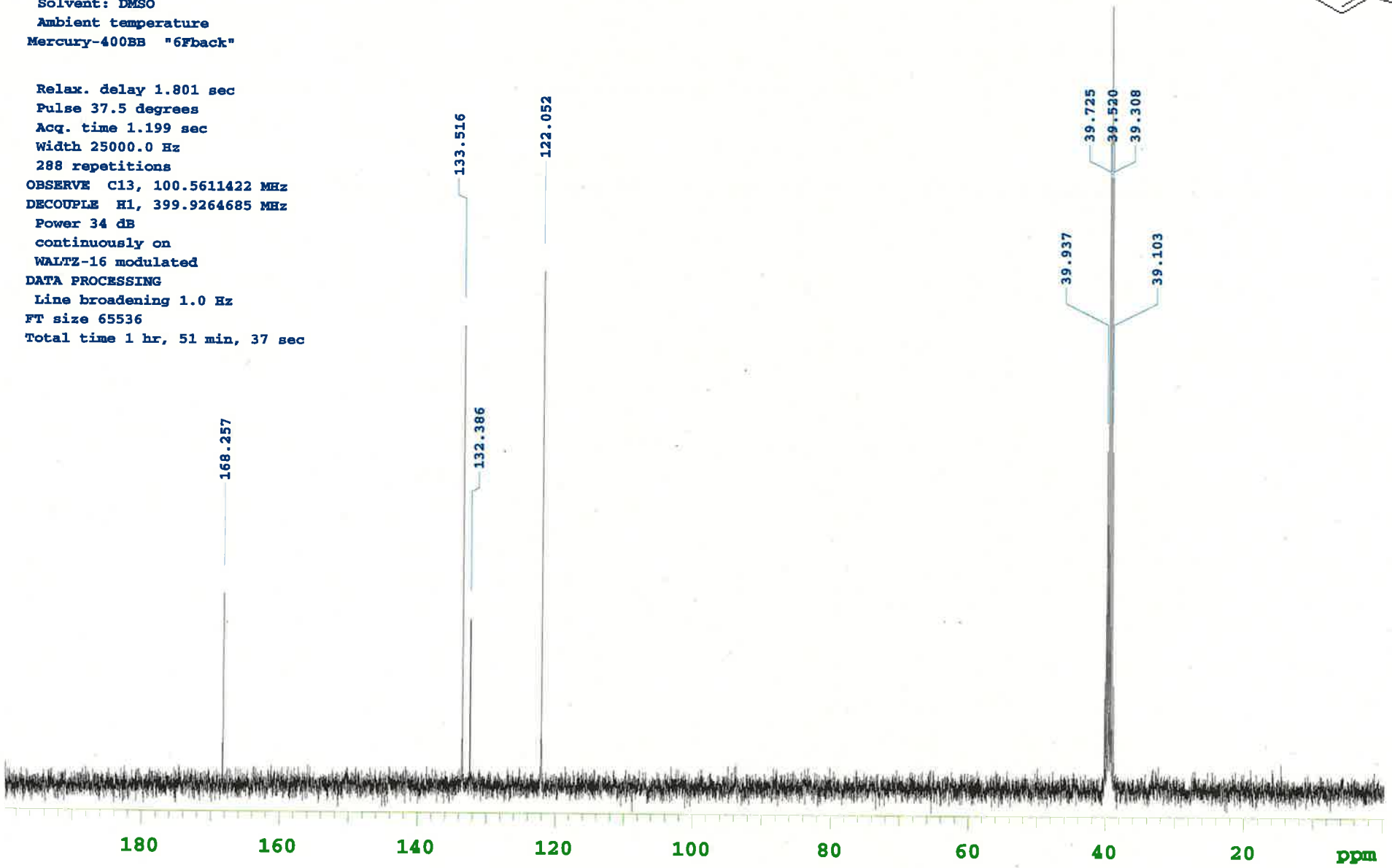

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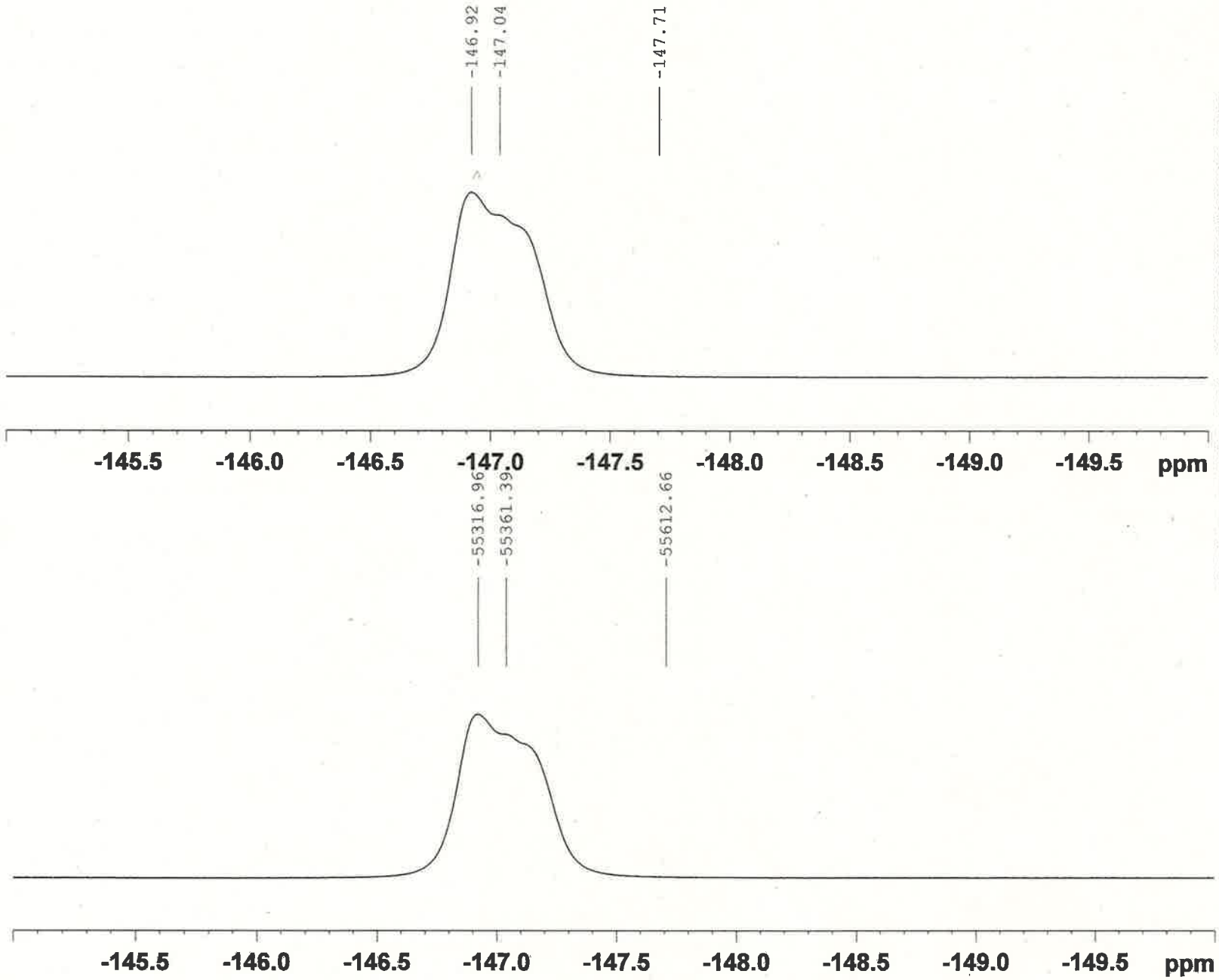

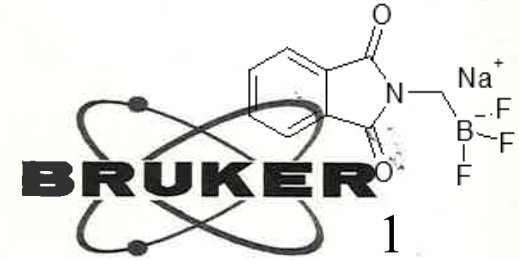

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DE                           6.00 usec
TE                          303.0 K
D1                     1.00000000 sec
d11                    0.03000000 sec
d12                    0.00002000 sec
TD0                              1

======== CHANNEL f1 ========
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PL1                           -5.00 dB
SFO1                    376.4607160 MHz

======== CHANNEL f2 ========
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PCPD2                        80.00 usec
PL12                         17.50 dB
PL2                          -0.70 dB
SFO2                    400.1316000 MHz

F2 - Processing parameters
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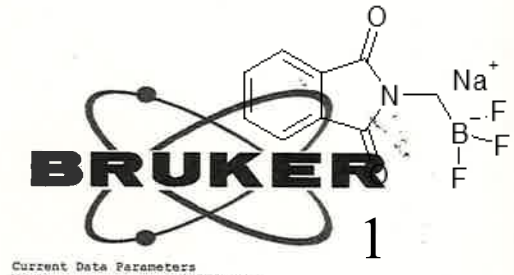

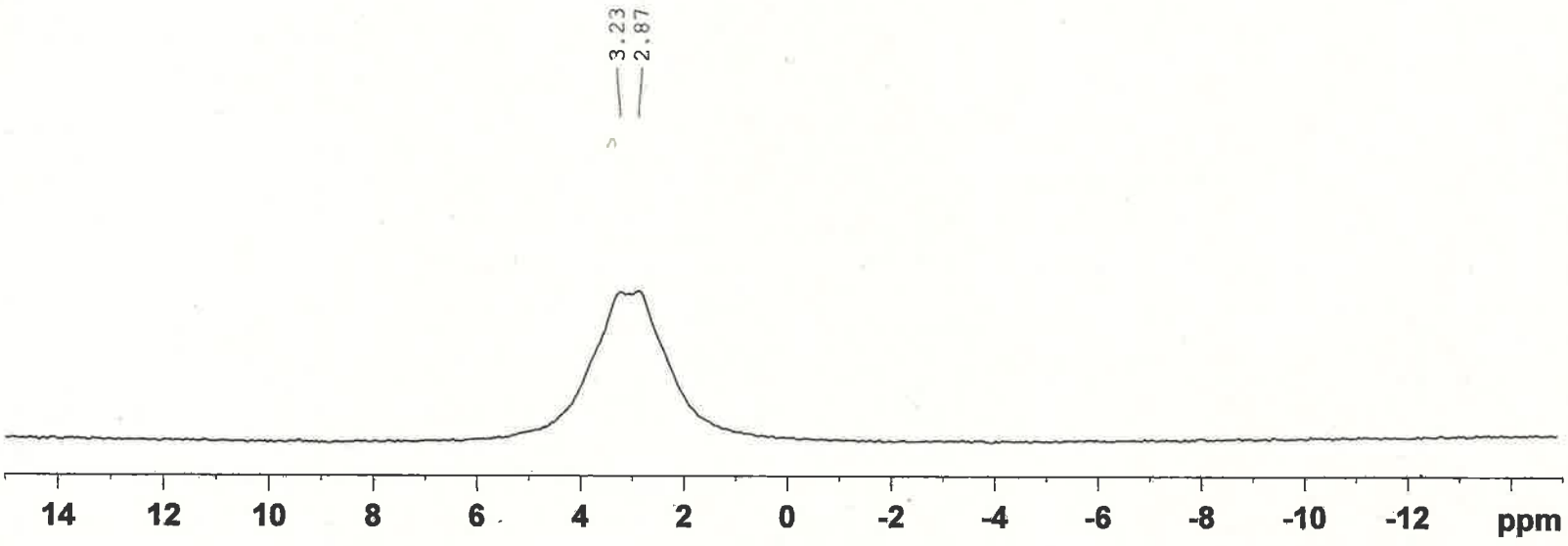


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d13                   0.00000400 sec
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F2 - Processing parameters
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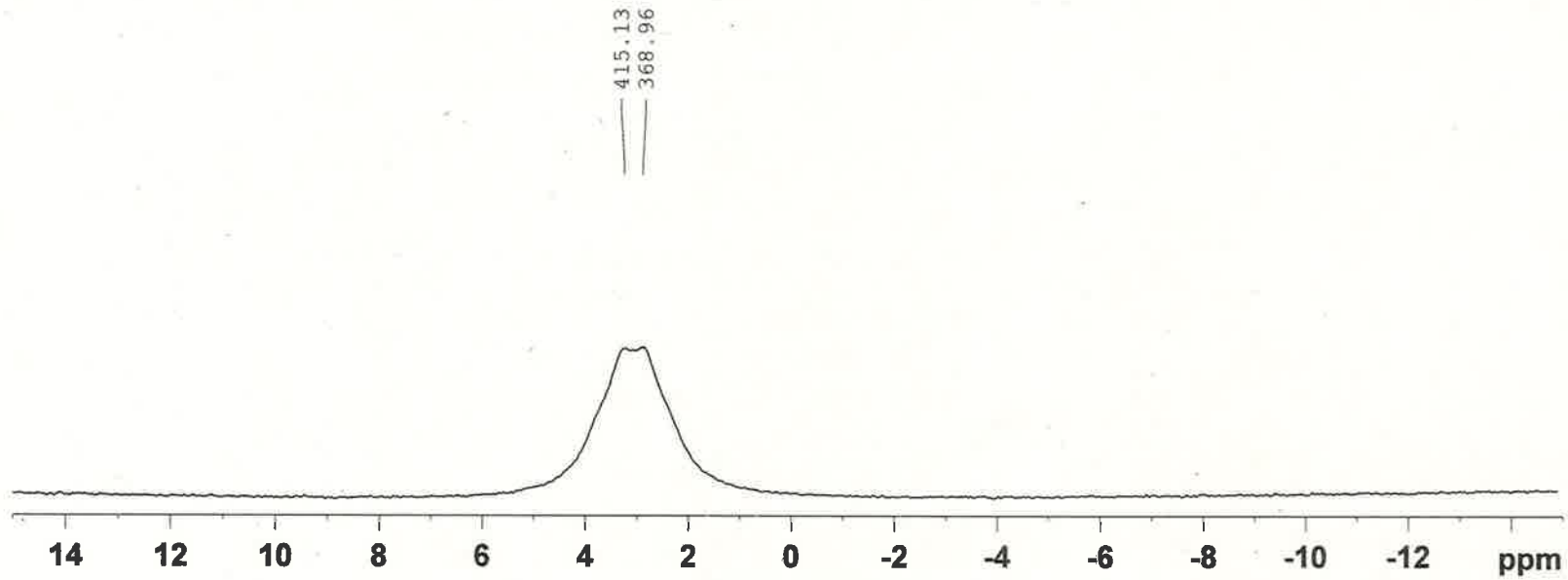

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Formula $C_7H_{14}BBrO_2$	FW 220.8999

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Spectrum Offset (Hz)	2247.6414	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

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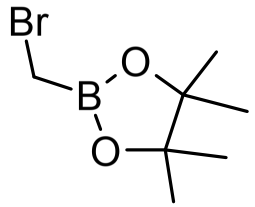

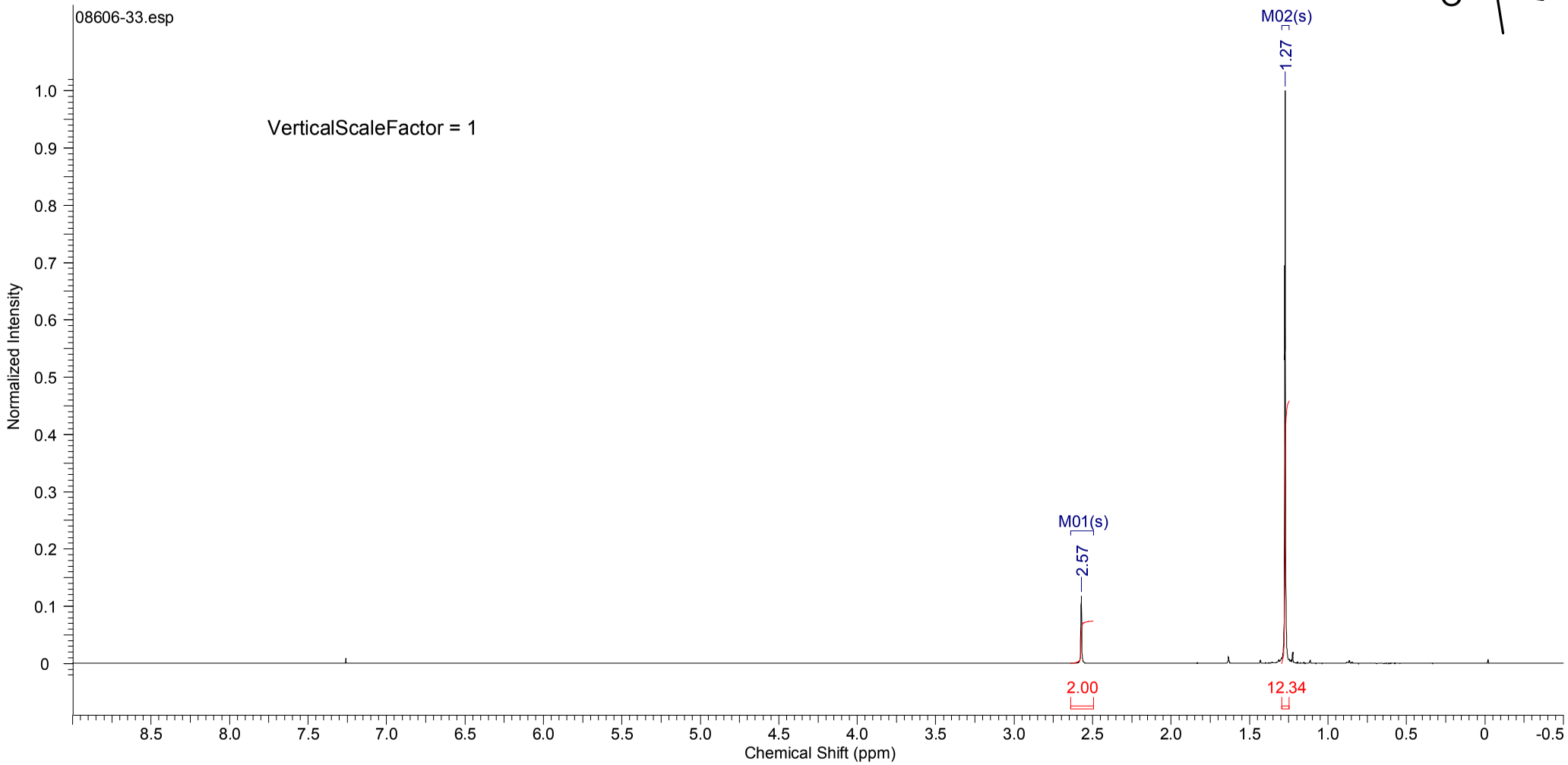

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DECOUPLE H1, 399.9245689 MHz
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DATA PROCESSING
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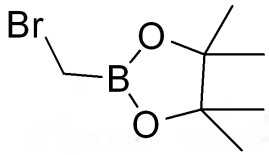
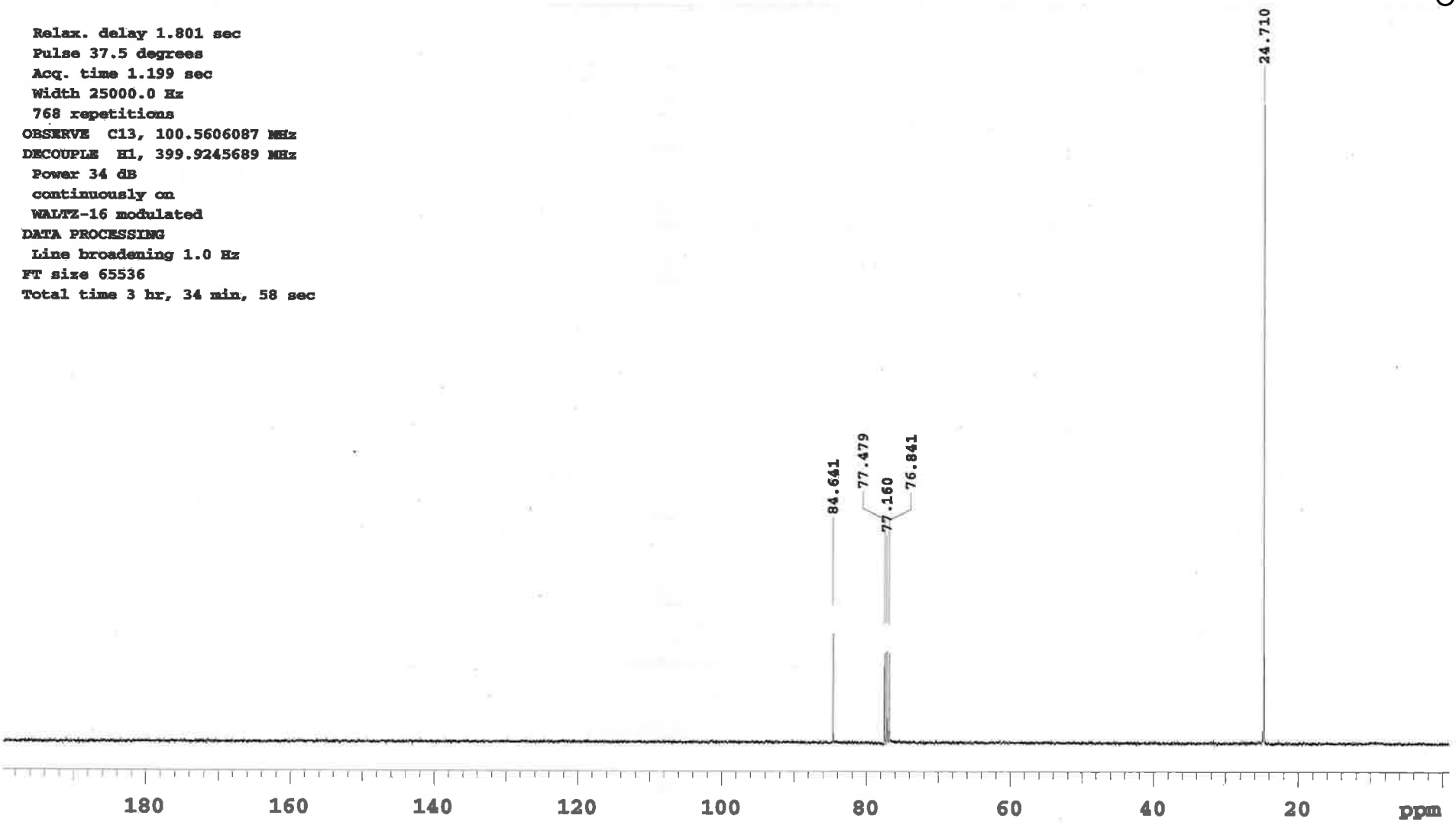
Total time 3 hr, 34 min, 58 sec





2012/04/11 9:11:13

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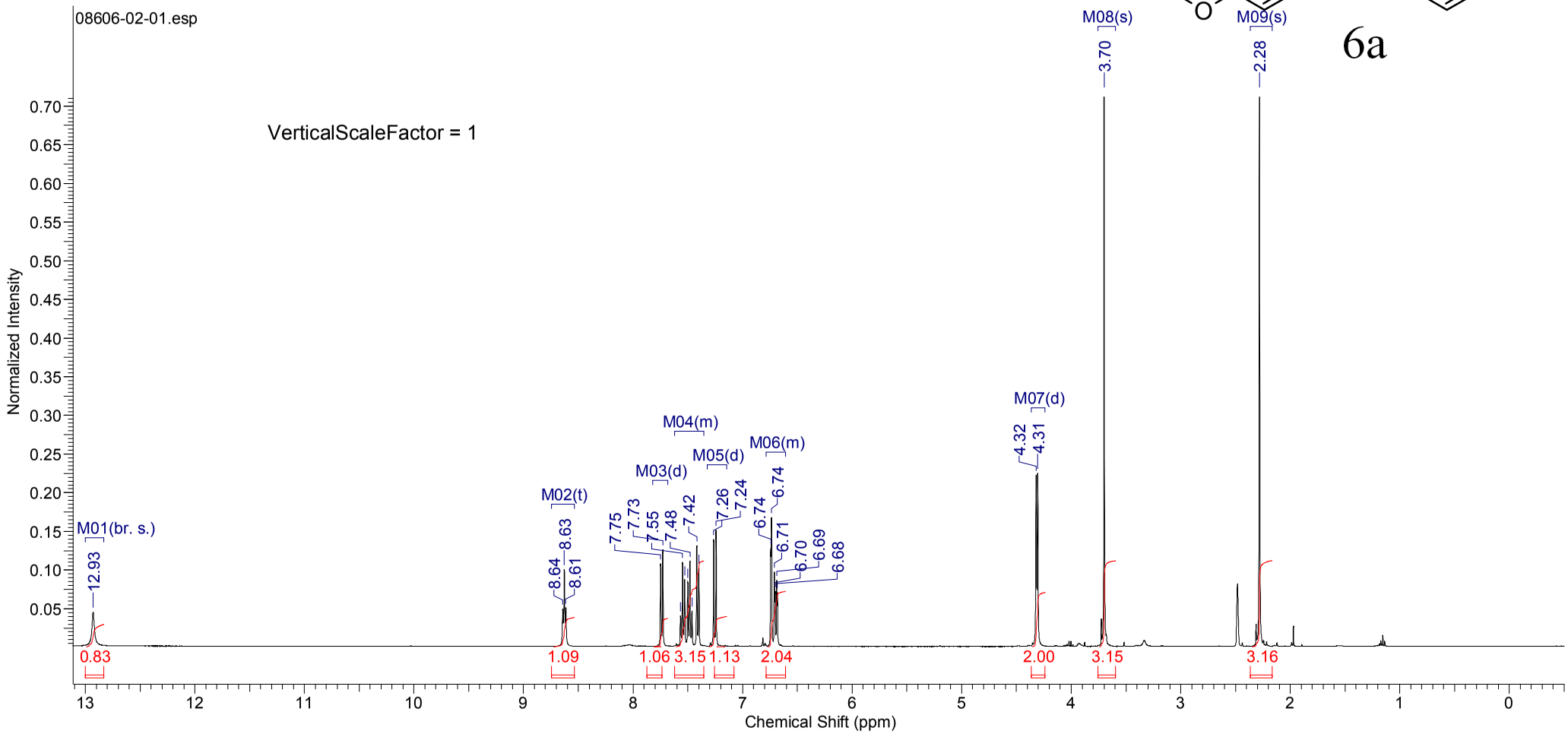
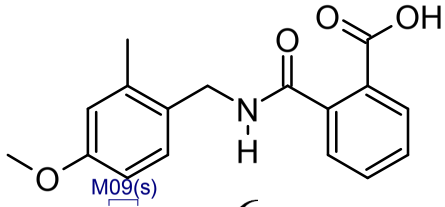
Receiver Gain	16.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type** STANDARD	**Sweep Width (Hz)** 5995.20
Temperature (degree C)	AMBIENT TEMPERATURE						

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.93 (br. s., 1H), 8.63 (t, *J*=5.49 Hz, 1H), 7.74 (d, *J*=7.68 Hz, 1H), 7.36-7.62 (m, 3H), 7.25 (d, *J*=8.42 Hz, 1H), 6.61-6.79 (m, 2H), 4.31 (d, *J*=5.86 Hz, 2H), 3.70 (s, 3H), 2.28 (s, 3H)





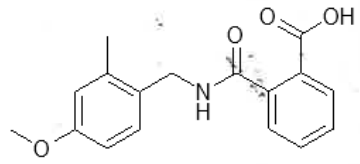
6a

13C OBSERVE

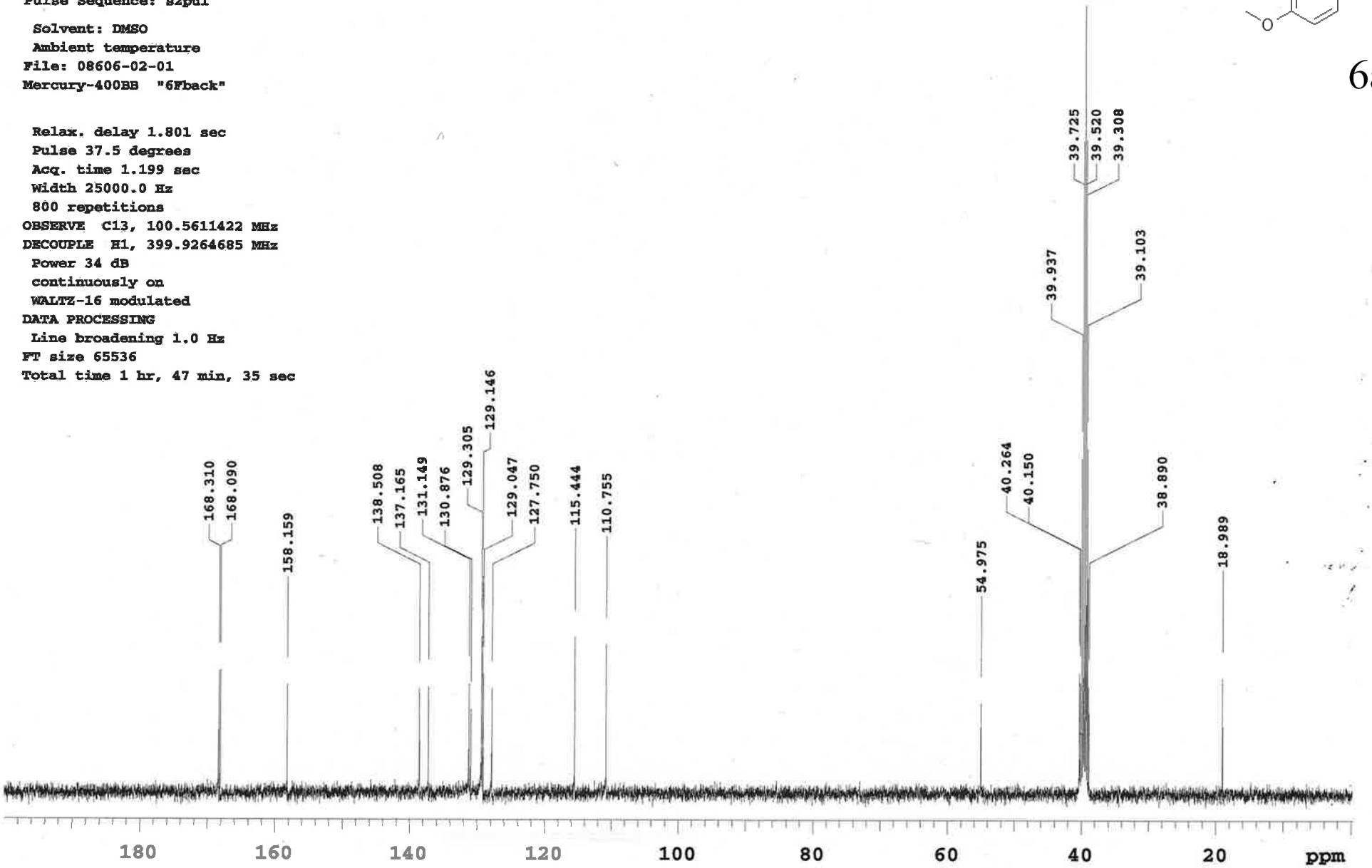
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 08606-02-01
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
800 repetitions
OBSERVE C13, 100.5611422 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



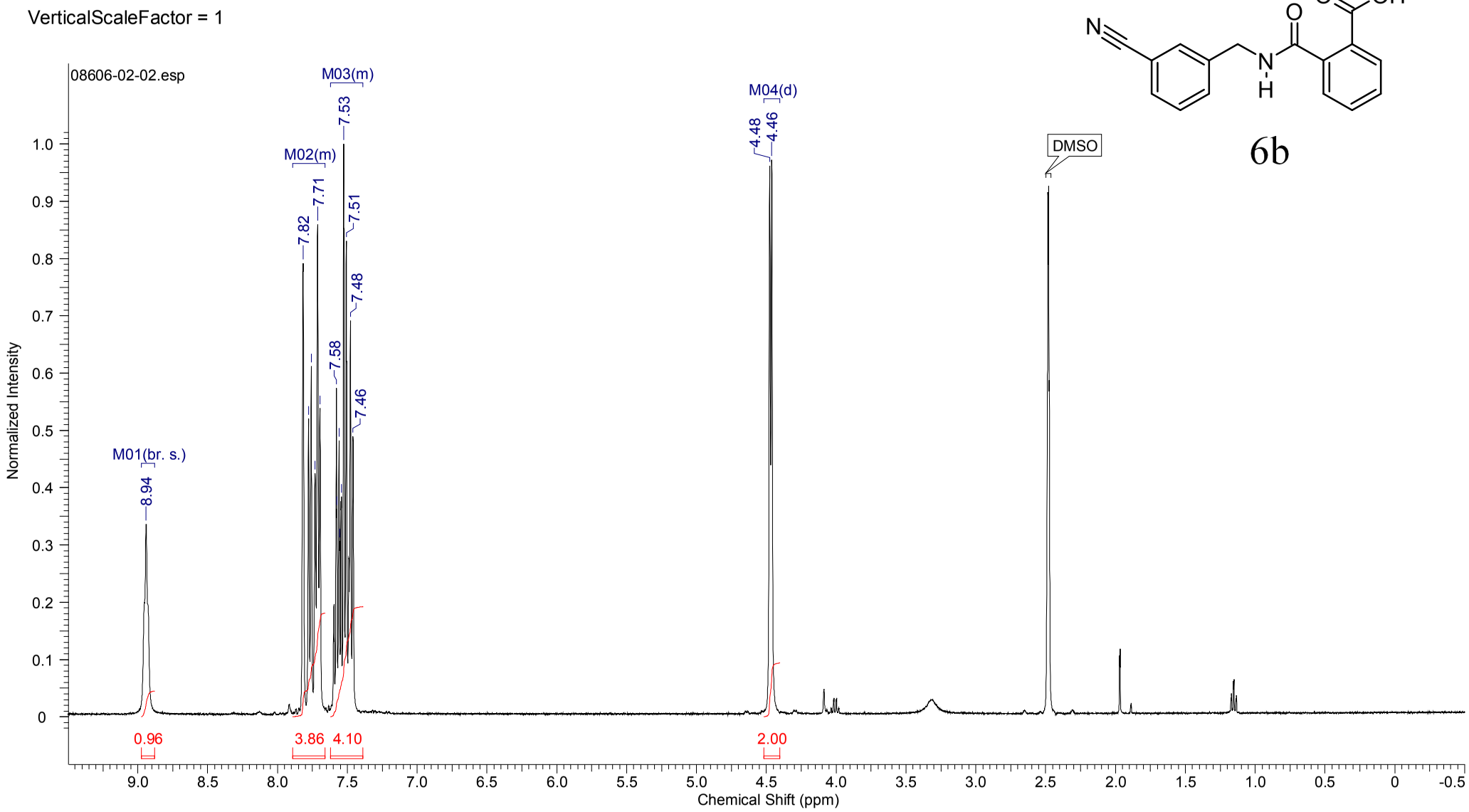
6a

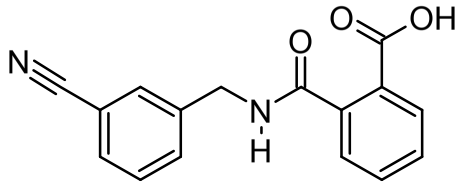


2012/04/11 14:19:31

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 26 2012		
Date Stamp	Feb 26 2012			**File Name**	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.93
Nucleus	1H	**Number of Transients**	16	**Original Points Count**	16379	**Points Count**	16384	**Pulse Sequence**	s2pul
Receiver Gain	20.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20
Temperature (degree C)	AMBIENT TEMPERATURE								

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.94 (br. s., 1H), 7.66-7.89 (m, 4H), 7.39-7.62 (m, 4H), 4.47 (d, *J*=5.85 Hz, 2H)





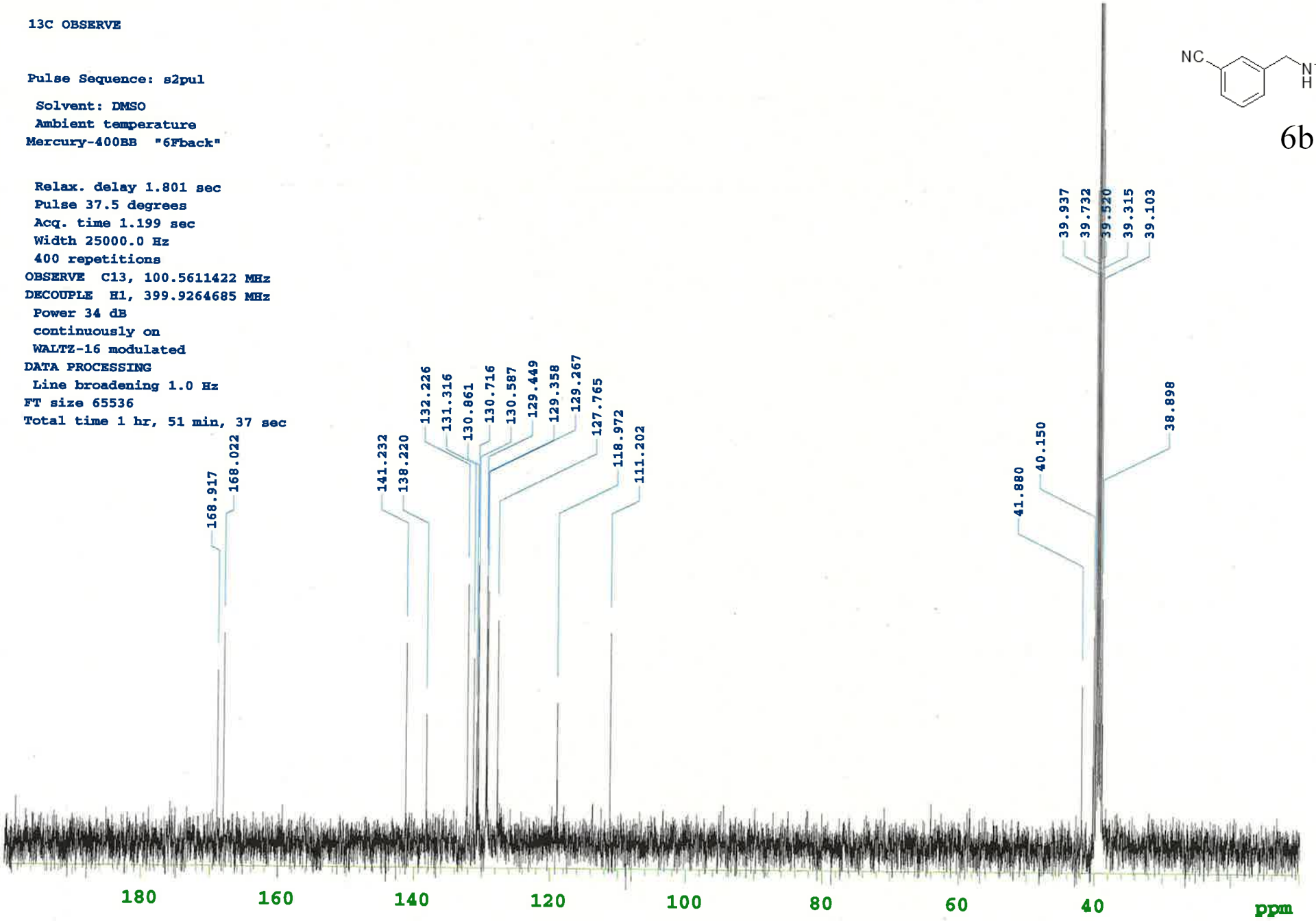
6b

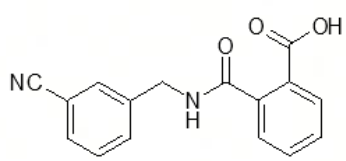
13C OBSERVE

Pulse Sequence: s2pul

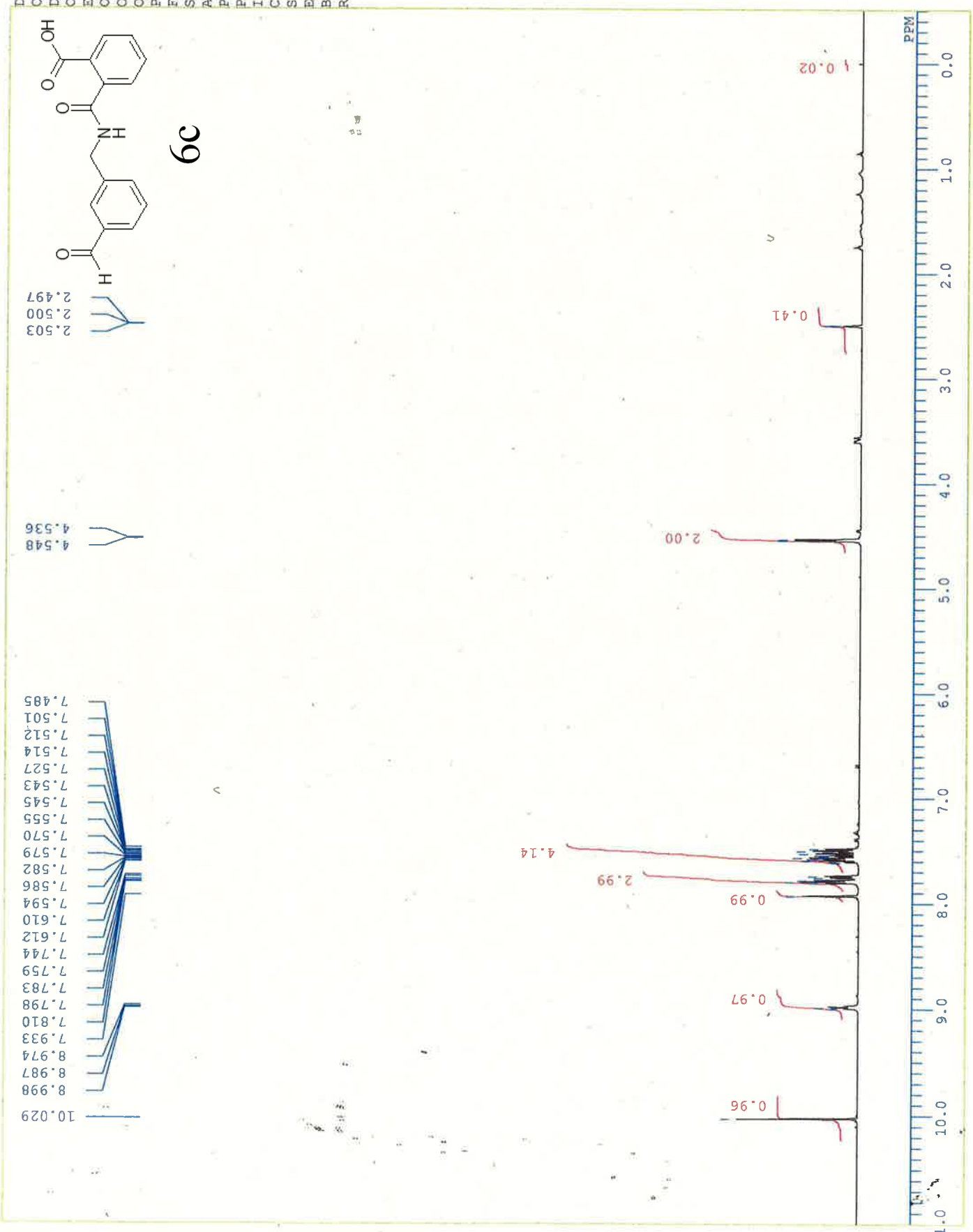
Solvent: DMSO
Ambient temperature
Mercury-400BB "6Fback"

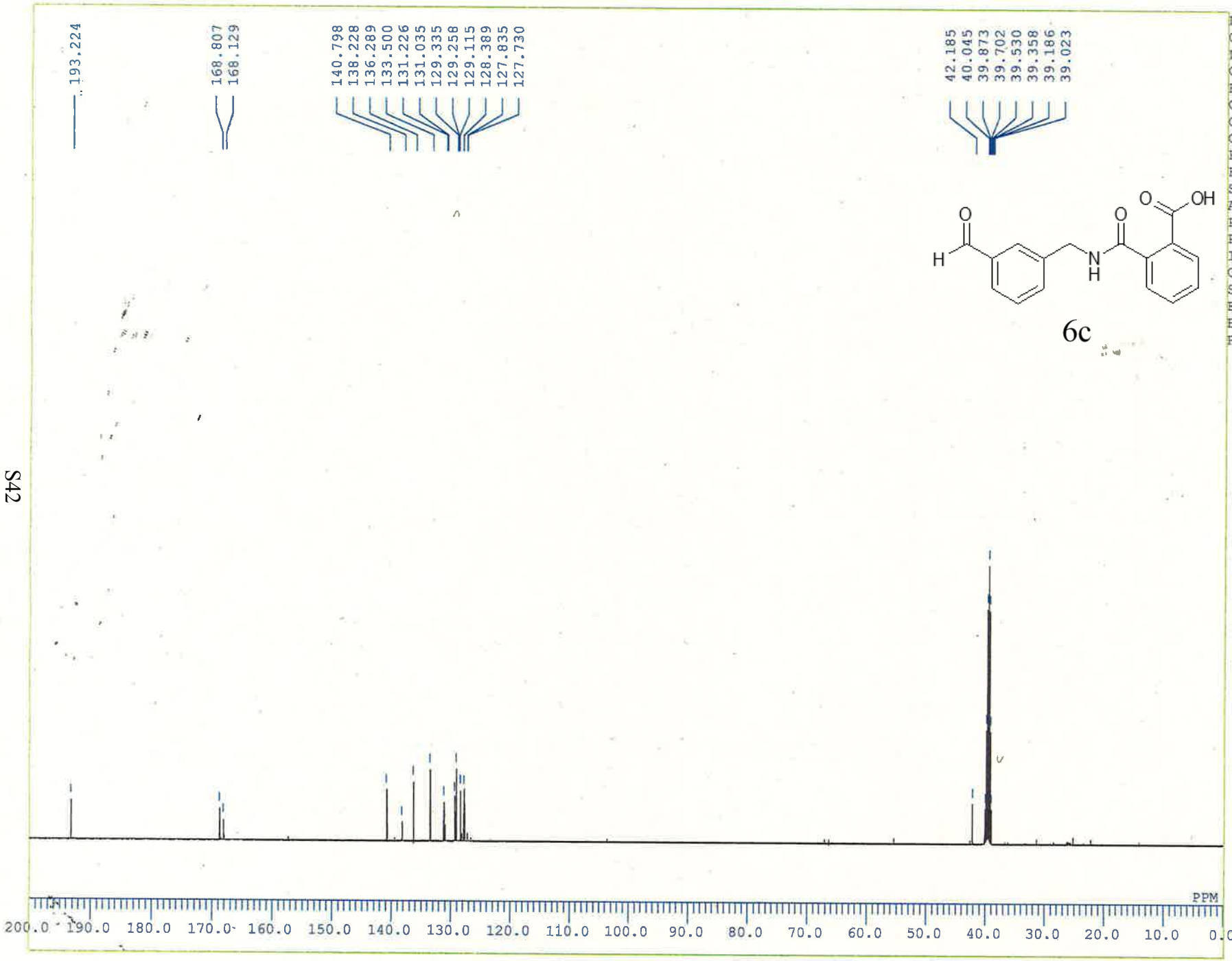
Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
400 repetitions
OBSERVE C13, 100.5611422 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 51 min, 37 sec





6b


DFILE NOM123245.1
COMNT single_pulse
DATIM 2012-03-23 22:48:14
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 490.15 MHz
OBSET 9.16 KHz
OBFIN 7.60 Hz
POINT 16384
FREQU 9191.18 Hz
SCANS 8
ACQTM 1.7826 sec
PD 3.0000 sec
PW1 8.55 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT DMSO
EXREF 2.50 ppm
BF 0.12 Hz
RGAIN 30
6c
PPM
0.02
0.41
2.00
4.14
2.99
0.99
0.97
0.96
2.503
2.500
2.497
4.548
4.536
10.029
8.998
8.987
8.974
7.933
7.810
7.798
7.783
7.759
7.744
7.612
7.610
7.594
7.586
7.582
7.579
7.570
7.555
7.545
7.543
7.527
7.514
7.512
7.501
7.485

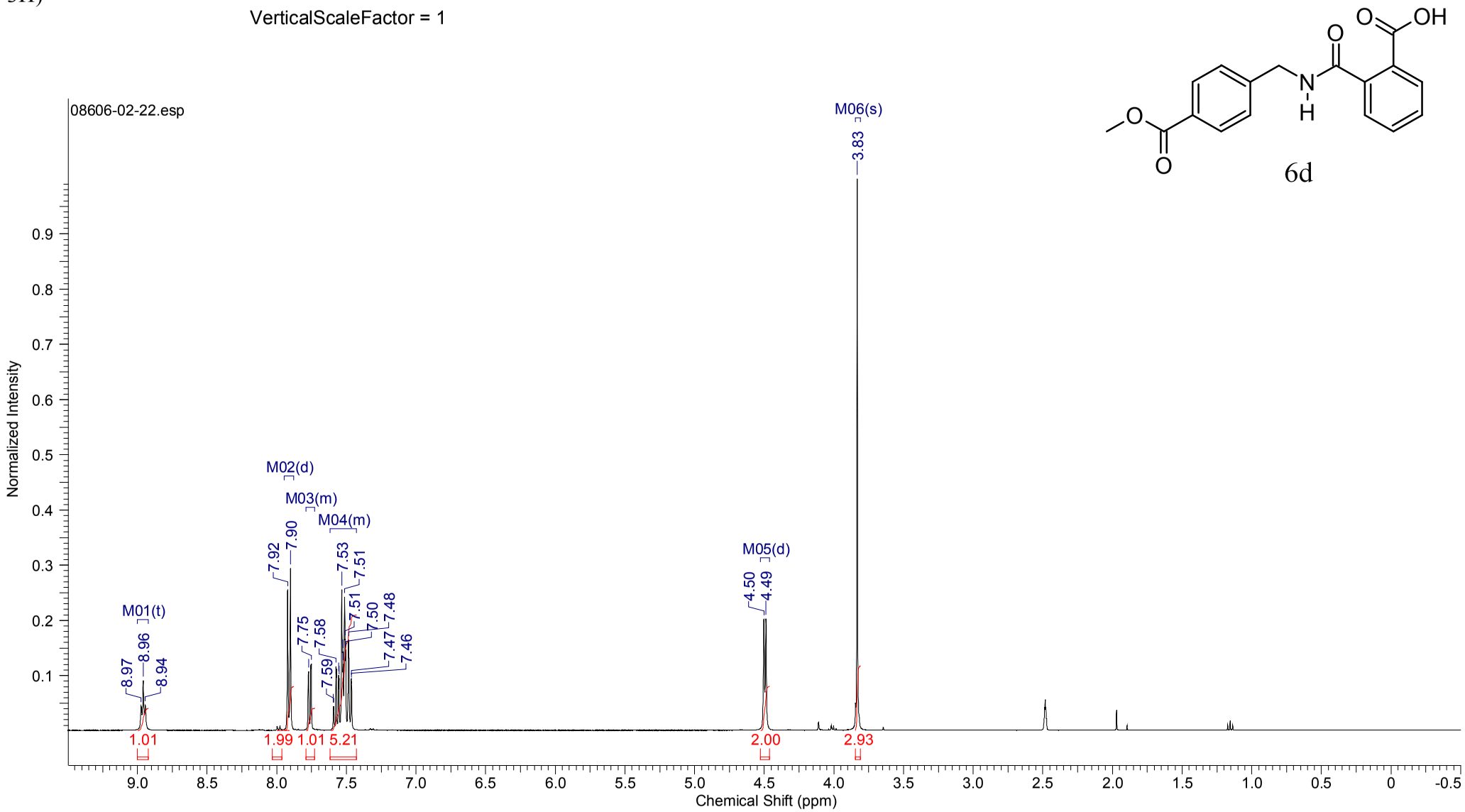


DFILE	NOM123246(C).1
COMNT	single pulse decoupl
DATIM	2012-03-24 06:45:43
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
POINT	32768
FREQU	38580.25 Hz
SCANS	10000
ACQTM	0.8493 sec
PD	2.0000 sec
PW1	3.20 usec
IRNUC	1H
CTEMP	25.0 c
SLVNT	DMSO
EXREF	39.52 ppm
BF	0.12 Hz
RGAIN	50

2012/04/11 12:47:18

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012		
Date Stamp	Mar 20 2012			**File Name**	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.93
Nucleus	1H	**Number of Transients**	16	**Original Points Count**	16379	**Points Count**	16384	**Pulse Sequence**	s2pul
Receiver Gain	12.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20
Temperature (degree C)	AMBIENT TEMPERATURE								

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.96 (t, *J*=5.86 Hz, 1H), 7.91 (d, *J*=8.05 Hz, 2H), 7.73-7.79 (m, 1H), 7.43-7.62 (m, 5H), 4.50 (d, *J*=5.86 Hz, 2H), 3.83 (s, 3H)



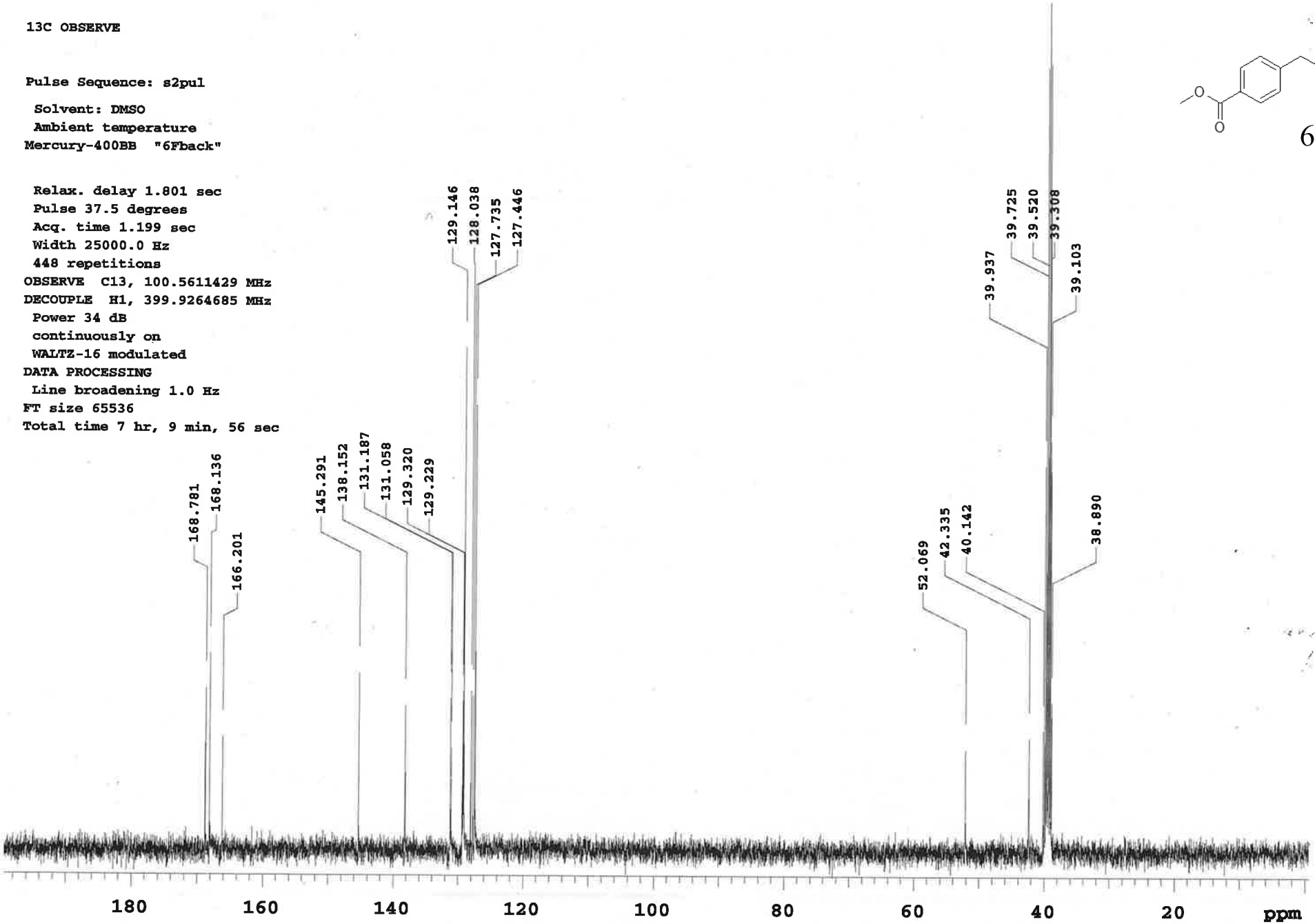
6d

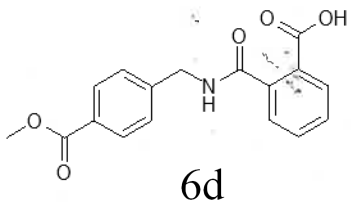
13C OBSERVE

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
448 repetitions
OBSERVE C13, 100.5611429 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 9 min, 56 sec





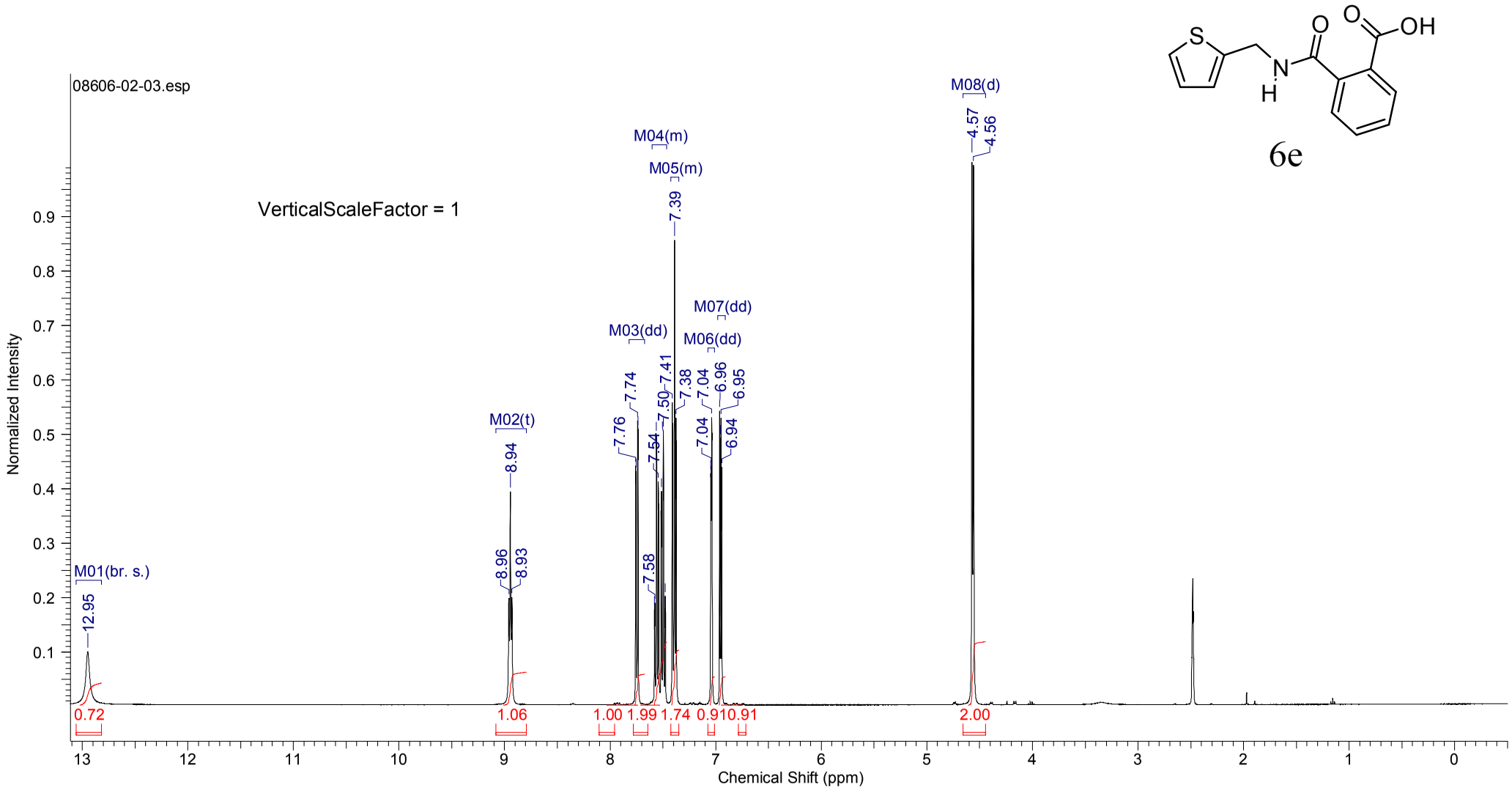
6d

2012/04/11 10:16:18

Formula $C_{13}H_{11}NO_3S$	FW 261.2963

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 26 2012
Date Stamp	Feb 26 2012			**File Name**	C:¥USR¥NMR¥FID		**Frequency (MHz)** 399.93
Nucleus	1H	**Number of Transients**	32	**Original Points Count**	16379	**Points Count** 16384	**Pulse Sequence** s2pul
Receiver Gain	16.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type** STANDARD	**Sweep Width (Hz)** 5995.20
Temperature (degree C)	AMBIENT TEMPERATURE						

^{1}H NMR (400 MHz, DMSO-d$_6$) δ 12.95 (br. s., 1H), 8.94 (t, *J*=5.86 Hz, 1H), 7.75 (dd, *J*=1.28, 7.50 Hz, 1H), 7.46-7.60 (m, 2H), 7.35-7.42 (m, 2H), 7.04 (dd, *J*=0.91, 3.48 Hz, 1H), 6.95 (dd, *J*=3.29, 5.12 Hz, 1H), 4.57 (d, *J*=5.86 Hz, 2H)



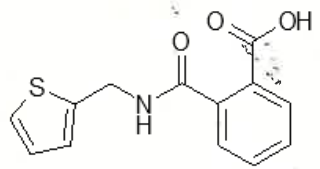
6e

13C OBSERVE

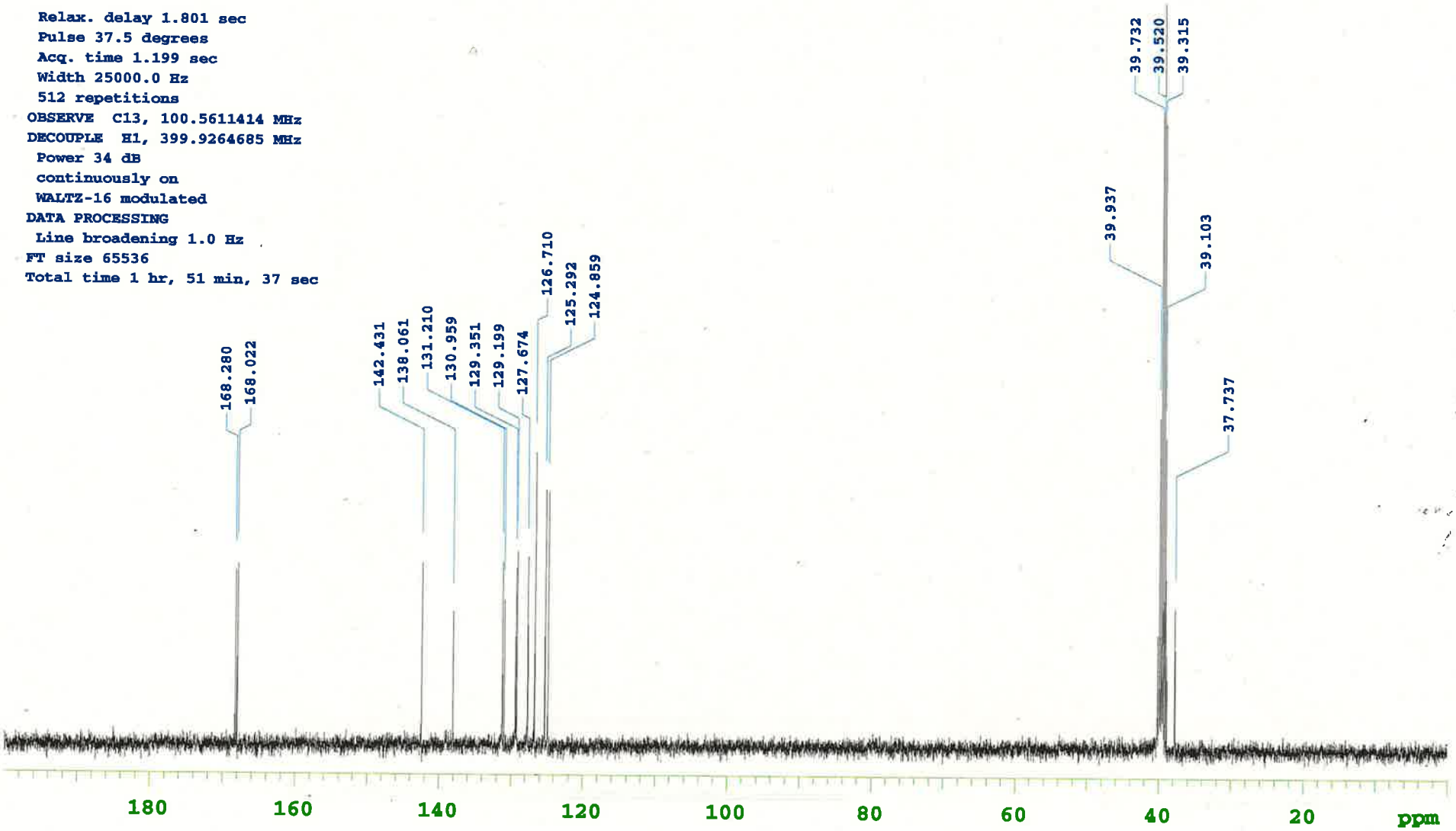
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
512 repetitions
OBSERVE C13, 100.5611414 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 51 min, 37 sec



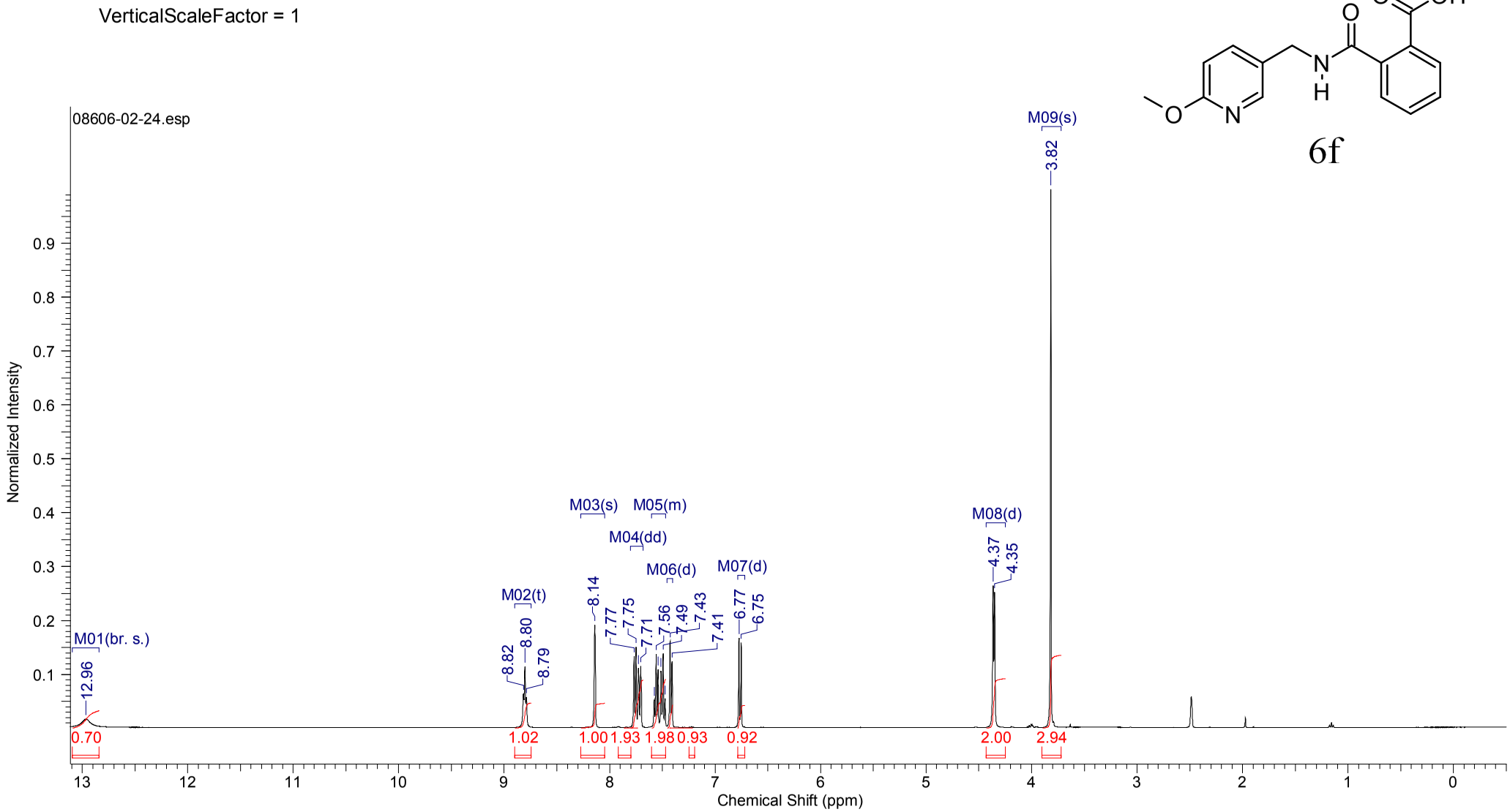
6e

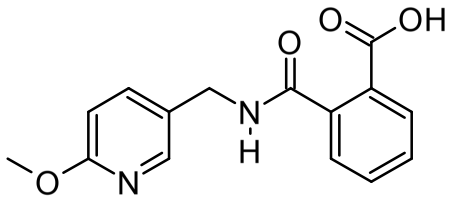


2012/04/11 12:58:54

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE		**Date**	Mar 20 2012	
Date Stamp	Mar 20 2012			**File Name**	C:¥USR¥NMR¥FID		**Frequency (MHz)** 399.93
Nucleus	1H	**Number of Transients**	32	**Original Points Count**	16379	**Points Count** 16384	**Pulse Sequence** s2pul
Receiver Gain	12.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type** STANDARD	**Sweep Width (Hz)** 5995.20
Temperature (degree C)	AMBIENT TEMPERATURE						

^{1}H NMR (400 MHz, DMSO-d_6) δ 12.96 (br. s., 1H), 8.80 (t, *J*=5.67 Hz, 1H), 8.14 (s, 1H), 7.74 (dd, *J*=8.05, 17.20 Hz, 2H), 7.47-7.60 (m, 2H), 7.42 (d, *J*=7.32 Hz, 1H), 6.76 (d, *J*=8.42 Hz, 1H), 4.36 (d, *J*=5.85 Hz, 2H), 3.82 (s, 3H)





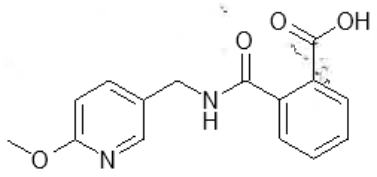
6f

13C OBSERVE

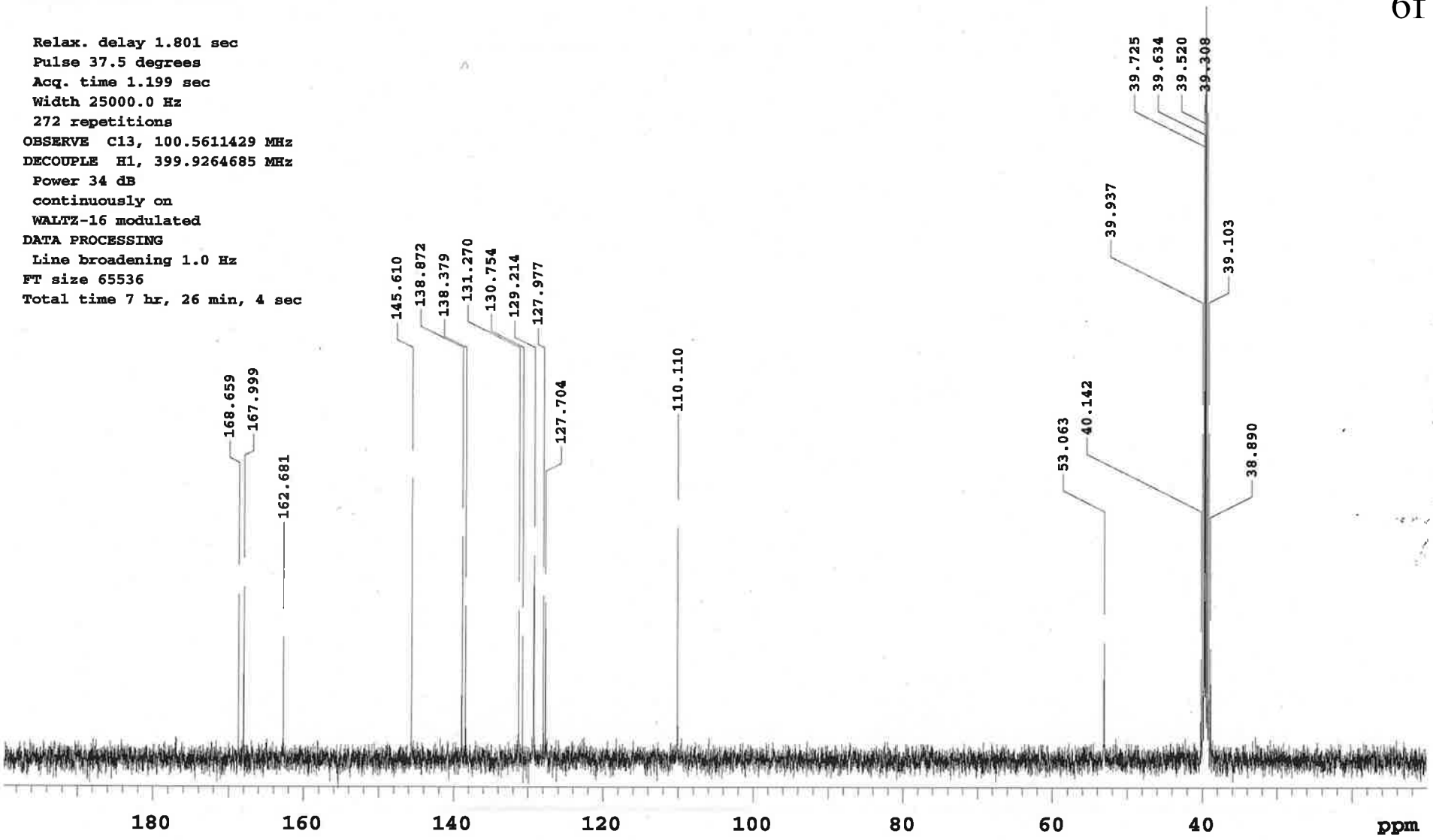
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
272 repetitions
OBSERVE C13, 100.5611429 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 4 sec



6f

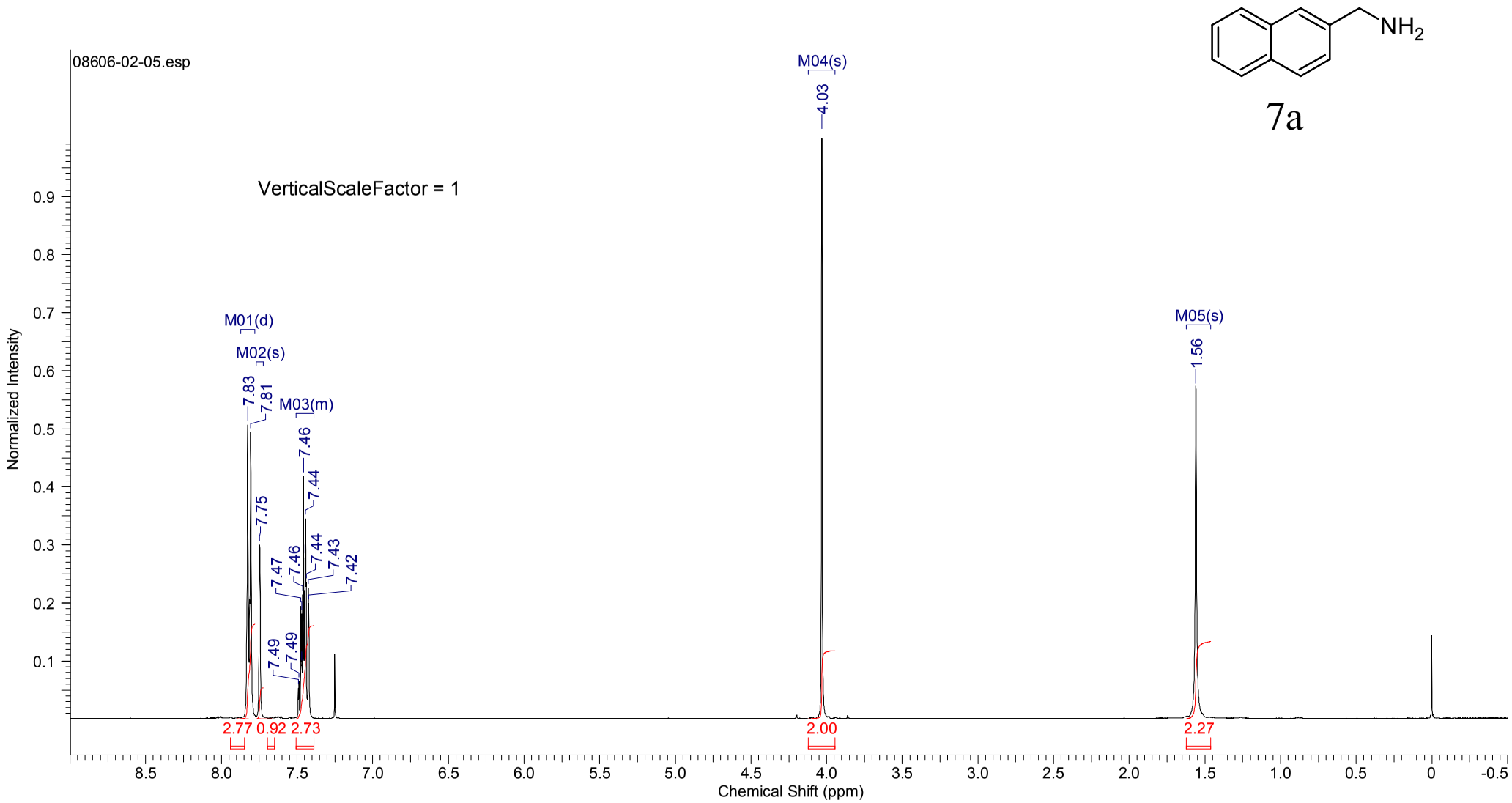


2012/04/11 10:32:12

Formula $C_{11}H_{11}N$	FW 157.2117

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	16
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	16.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2244.4961	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.82 (d, *J*=8.05 Hz, 3H), 7.75 (s, 1H), 7.39-7.51 (m, 3H), 4.03 (s, 2H), 1.56 (s, 2H)

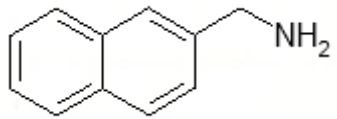


13C OBSERVE

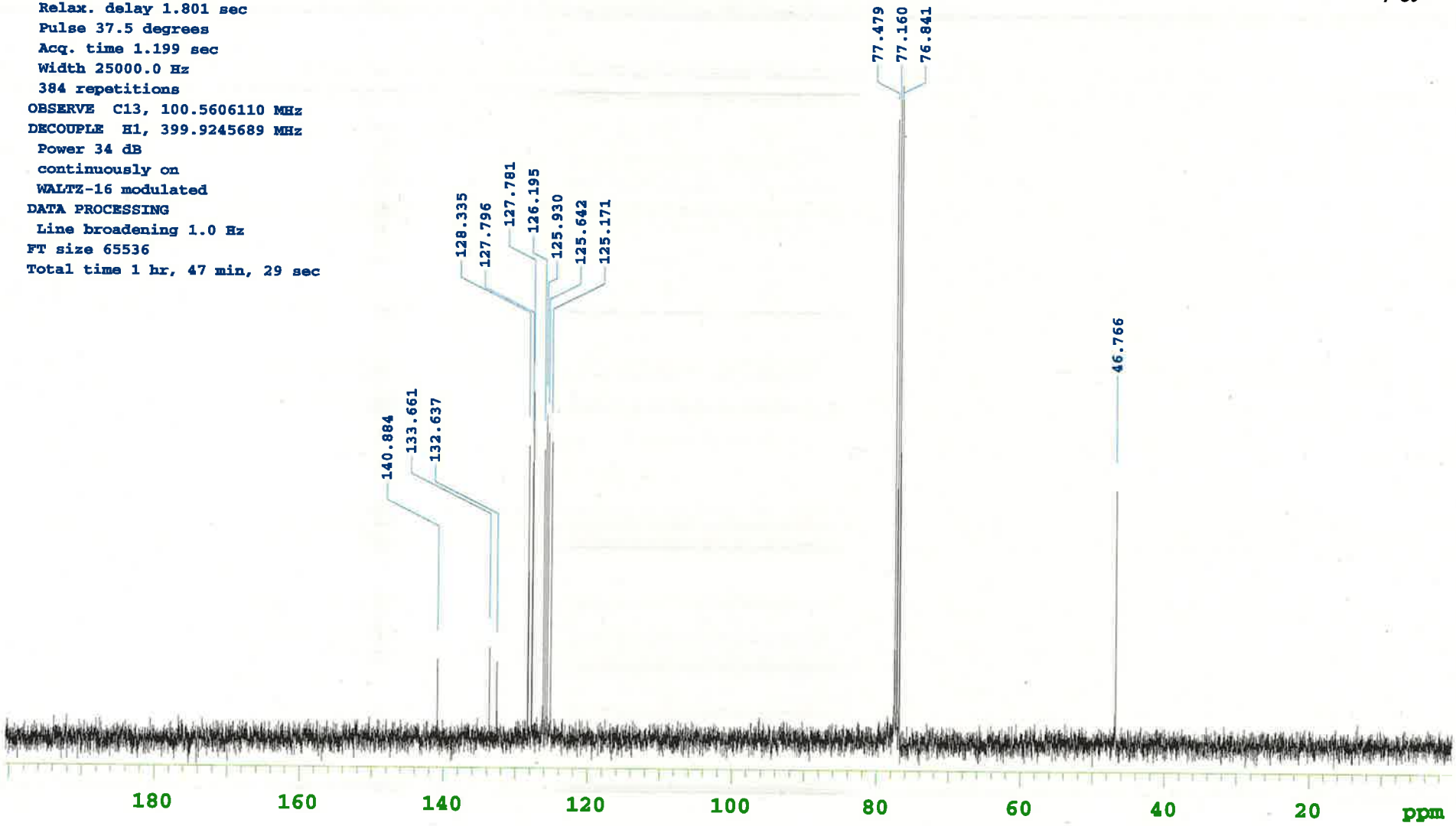
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
384 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 29 sec



7a

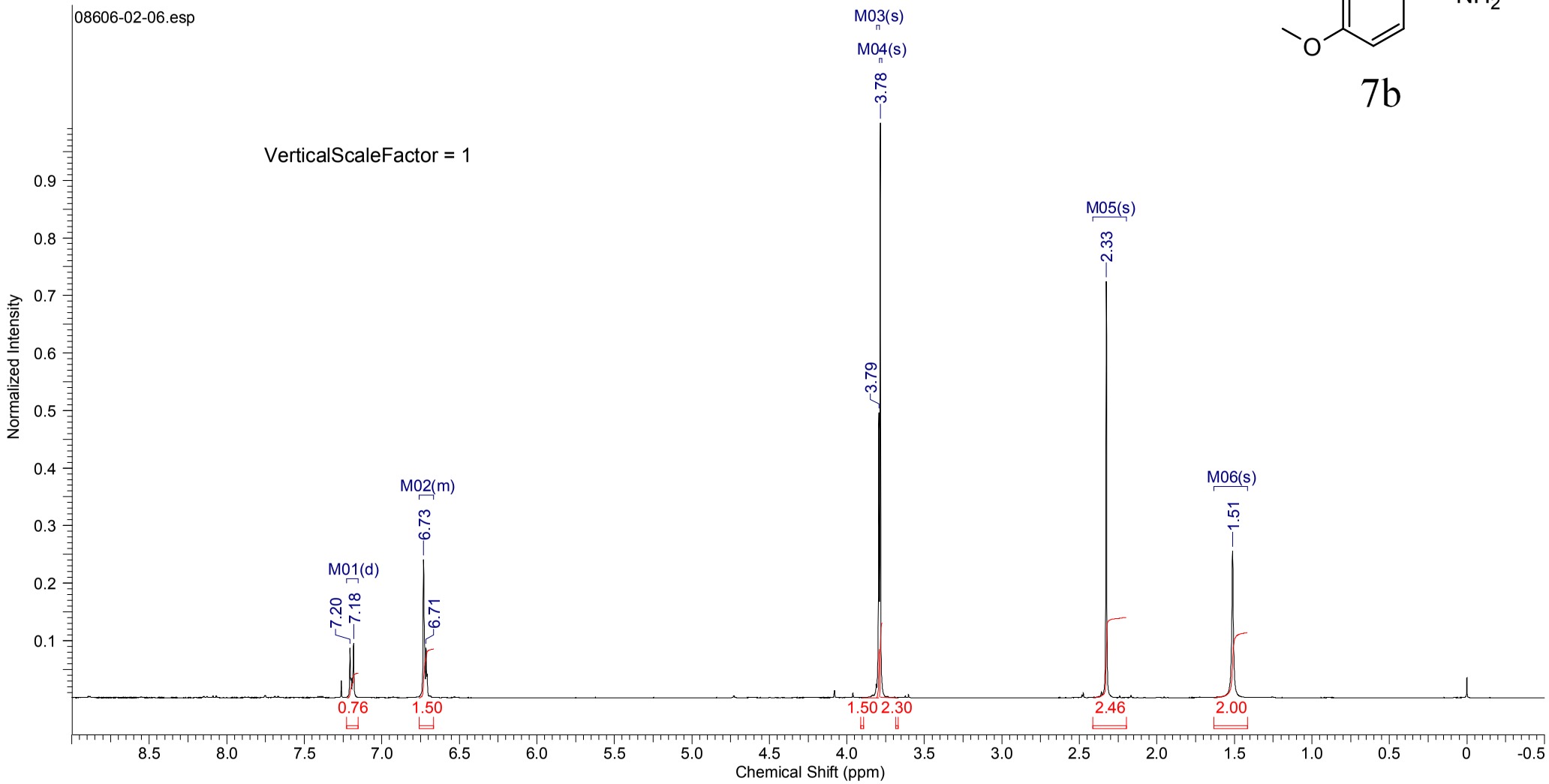


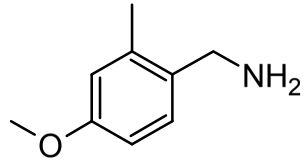
2012/04/11 10:50:11

Formula $C_9H_{13}NO$	FW 151.2056

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2248.5215	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.19 (d, *J*=9.15 Hz, 1H), 6.67-6.76 (m, 2H), 3.79 (s, 2H), 3.78 (s, 3H), 2.33 (s, 3H), 1.51 (s, 2H)





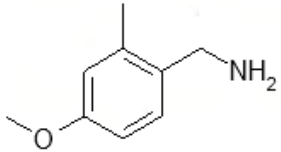
7b

13C OBSERVE

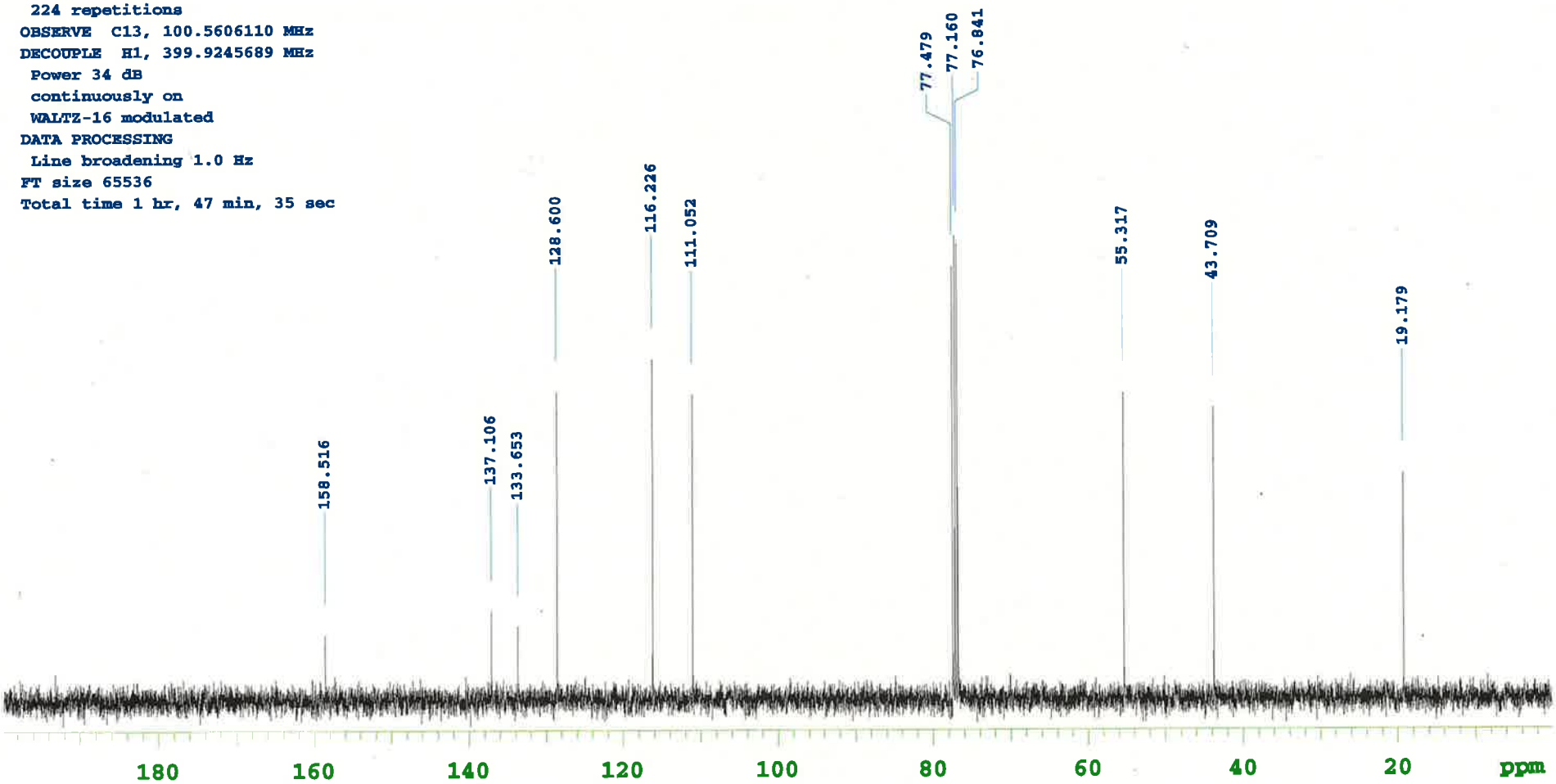
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
224 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



7b

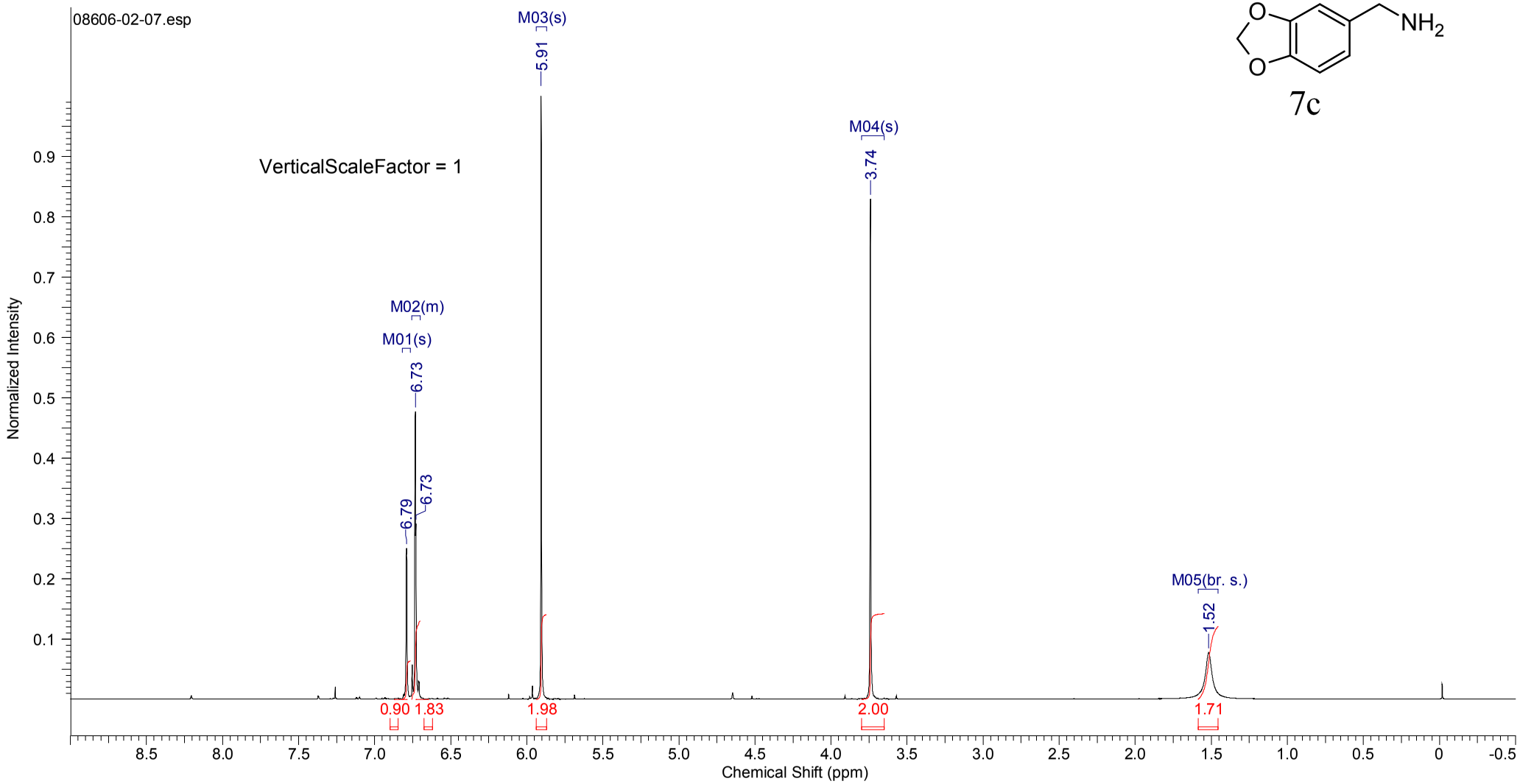


2012/04/11 10:46:48

Formula $C_8H_9NO_2$	FW 151.1626

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	16
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Solvent**	CHLOROFORM-d		
Spectrum Offset (Hz)	2247.6414	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 6.79 (s, 1H), 6.70-6.76 (m, 2H), 5.91 (s, 2H), 3.74 (s, 2H), 1.52 (br. s., 2H)

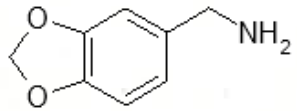


13C OBSERVE

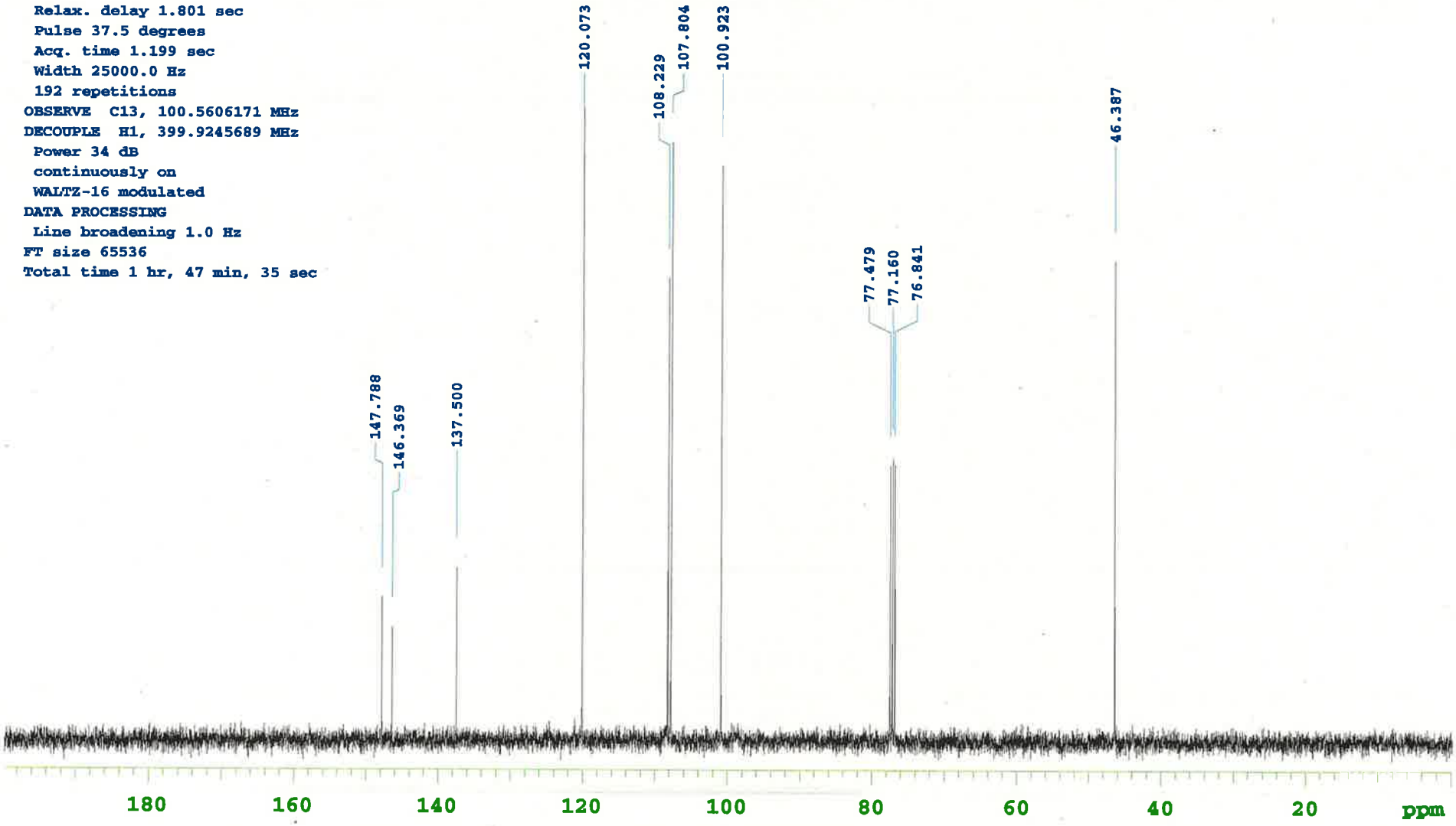
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.5606171 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



7c

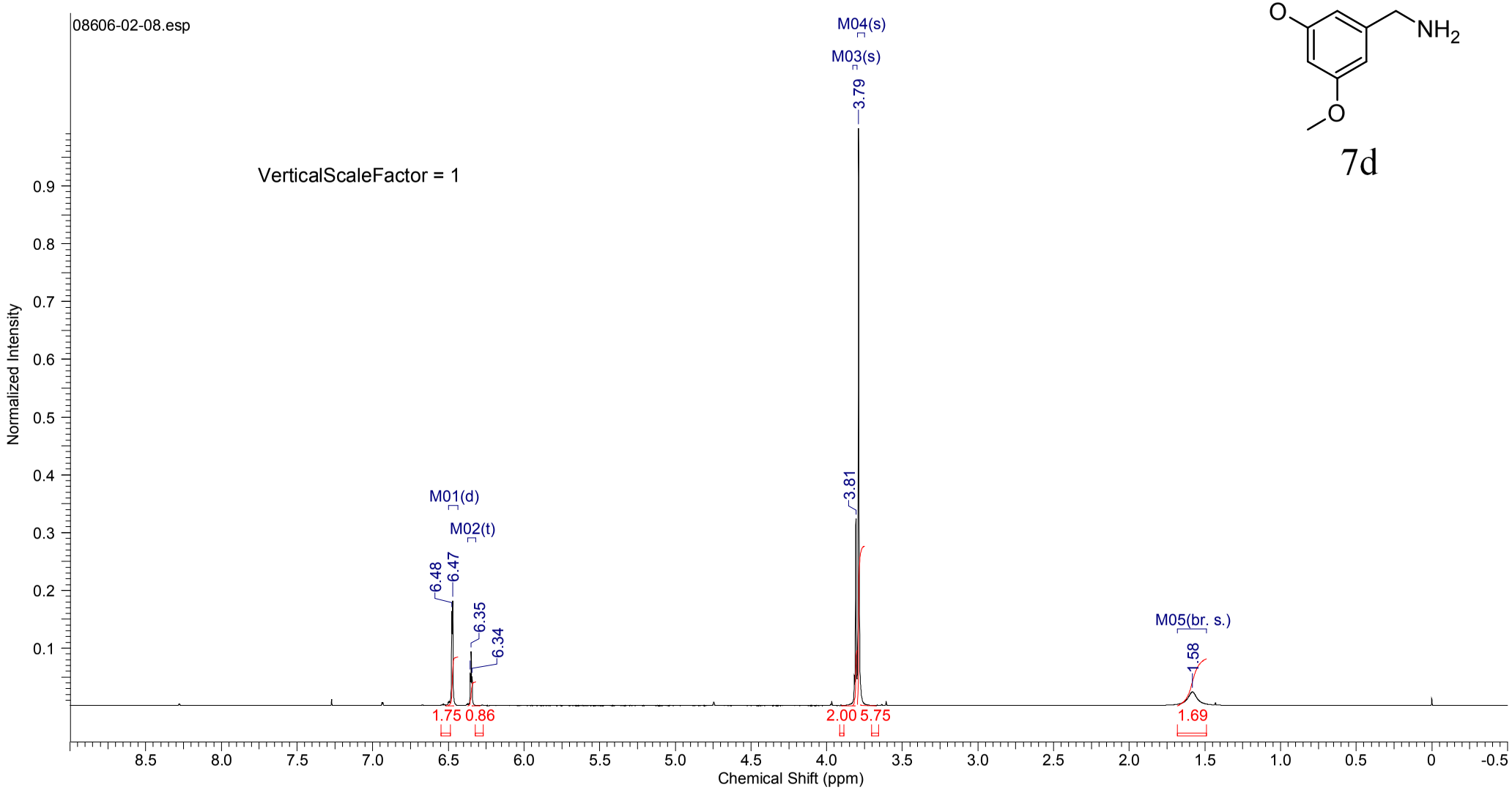


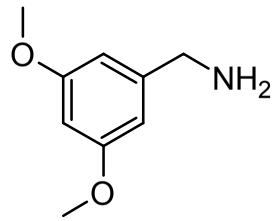
2012/04/11 10:55:30

Formula $C_9H_{13}NO_2$	FW 167.2050

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Solvent**	CHLOROFORM-d		
Spectrum Offset (Hz)	2252.5469	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 6.47 (d, *J*=2.56 Hz, 2H), 6.35 (t, *J*=2.38 Hz, 1H), 3.81 (s, 2H), 3.79 (s, 6H), 1.58 (br. s., 2H)





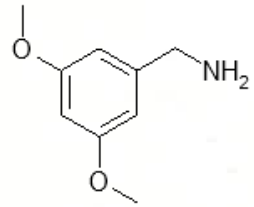
7d

13C OBSERVE

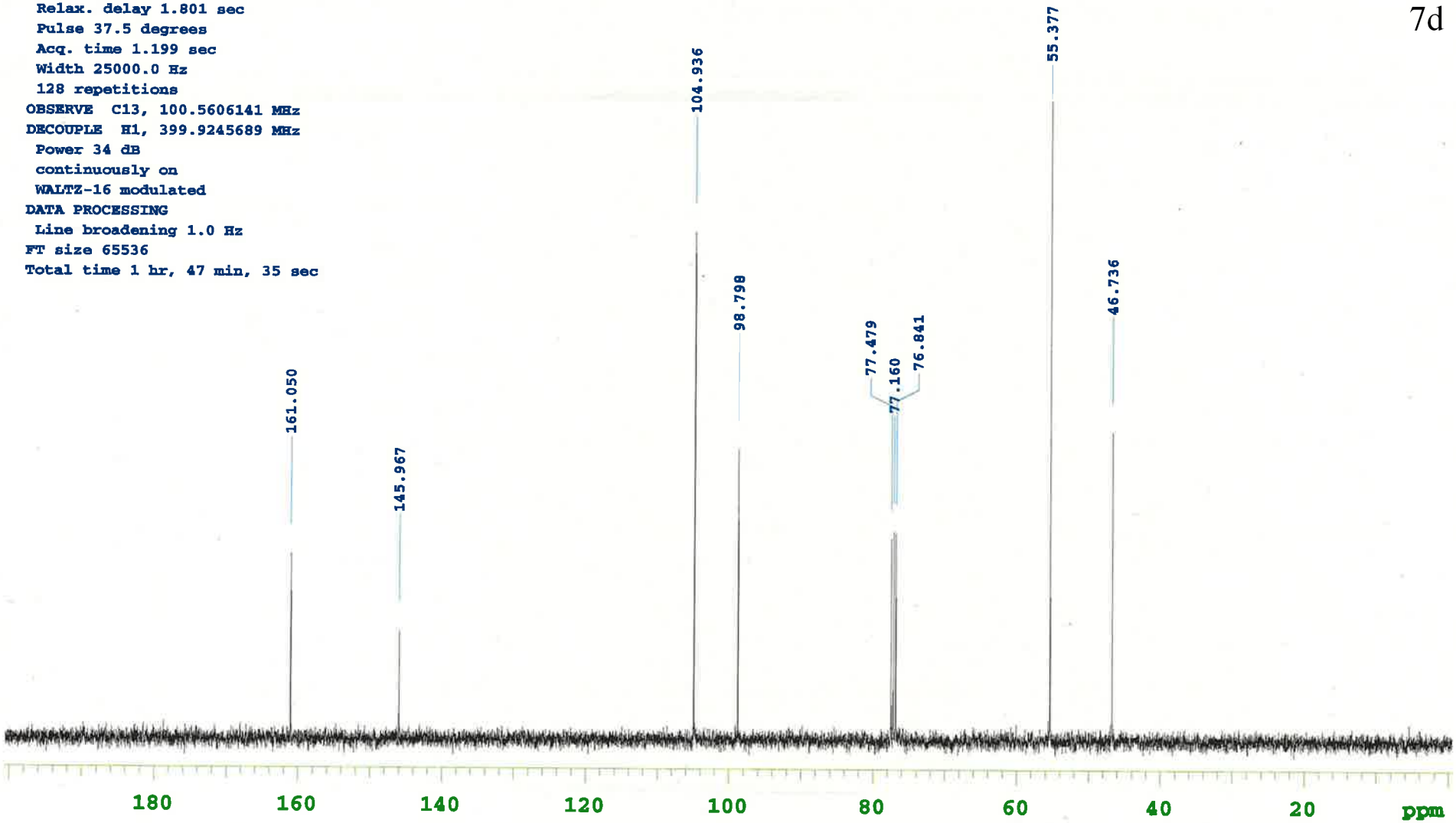
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
128 repetitions
OBSERVE C13, 100.5606141 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



7d

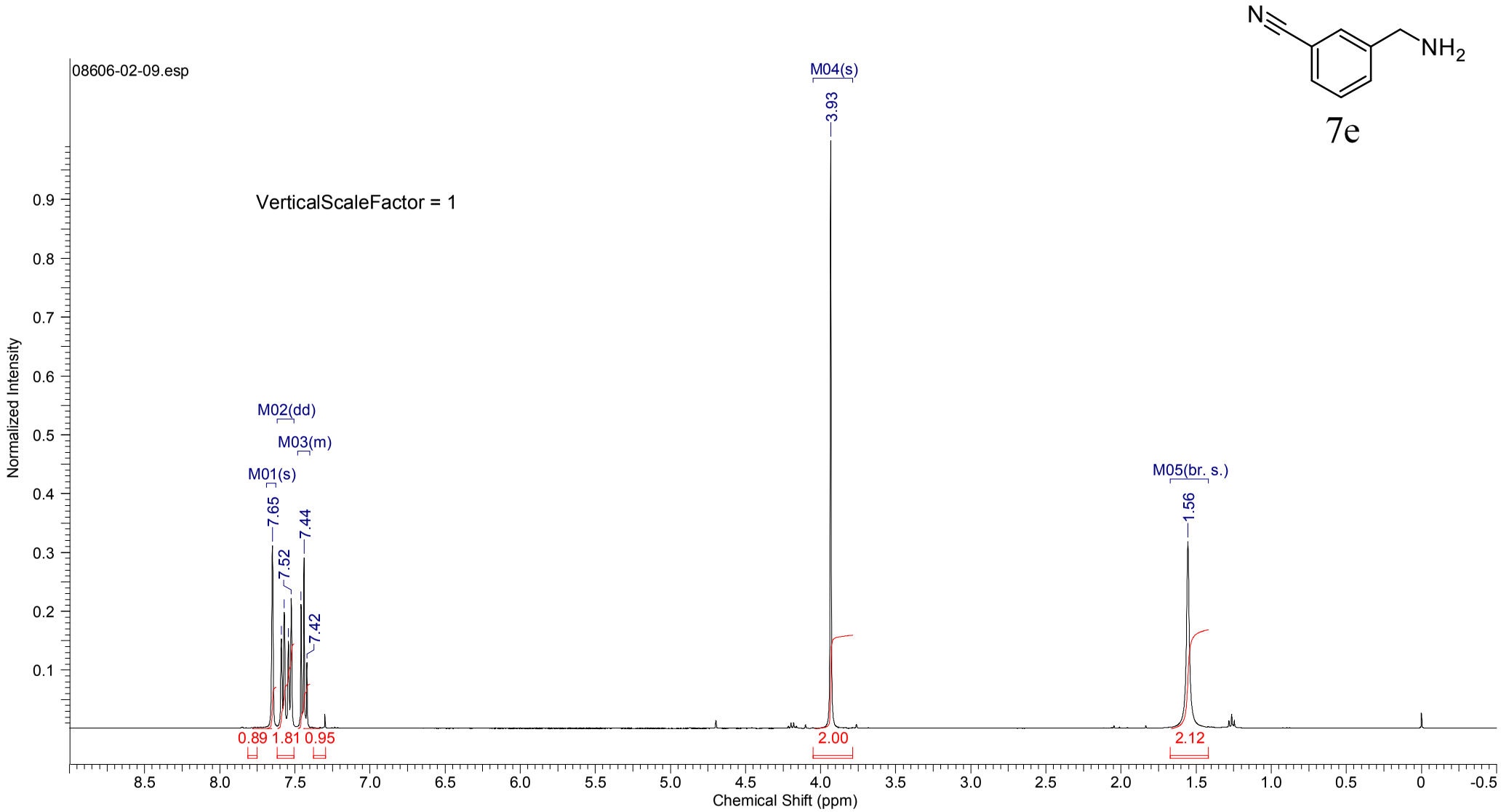


2012/04/11 10:58:33

Formula $C_8H_8N_2$	FW 132.1625

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	6.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2263.8911	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.65 (s, 1H), 7.56 (dd, *J*=7.68, 18.66 Hz, 2H), 7.40-7.48 (m, 1H), 3.93 (s, 2H), 1.56 (br. s., 2H)



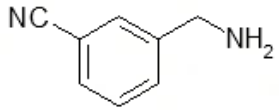
7e

13C OBSERVE

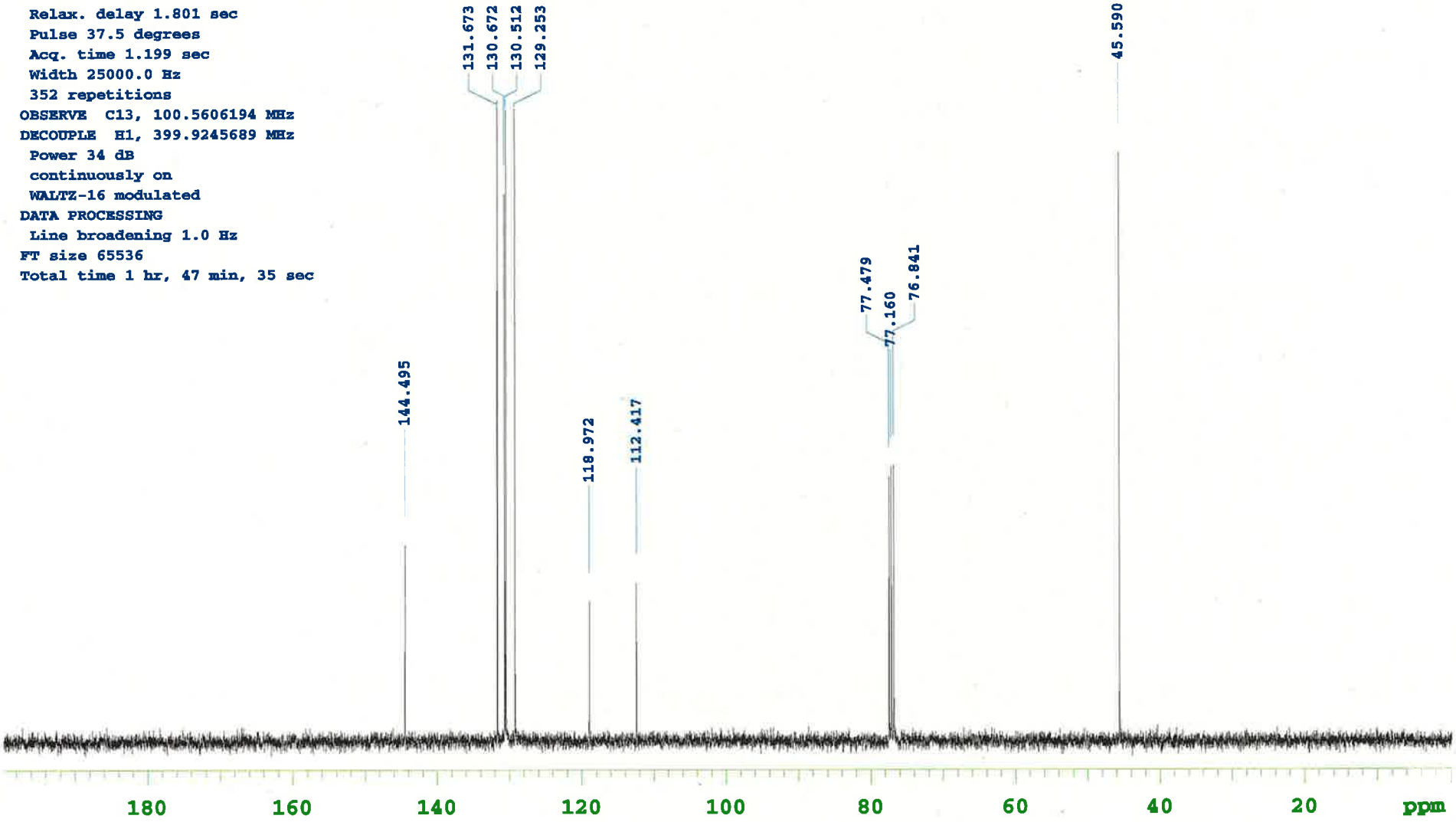
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 100.5606194 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



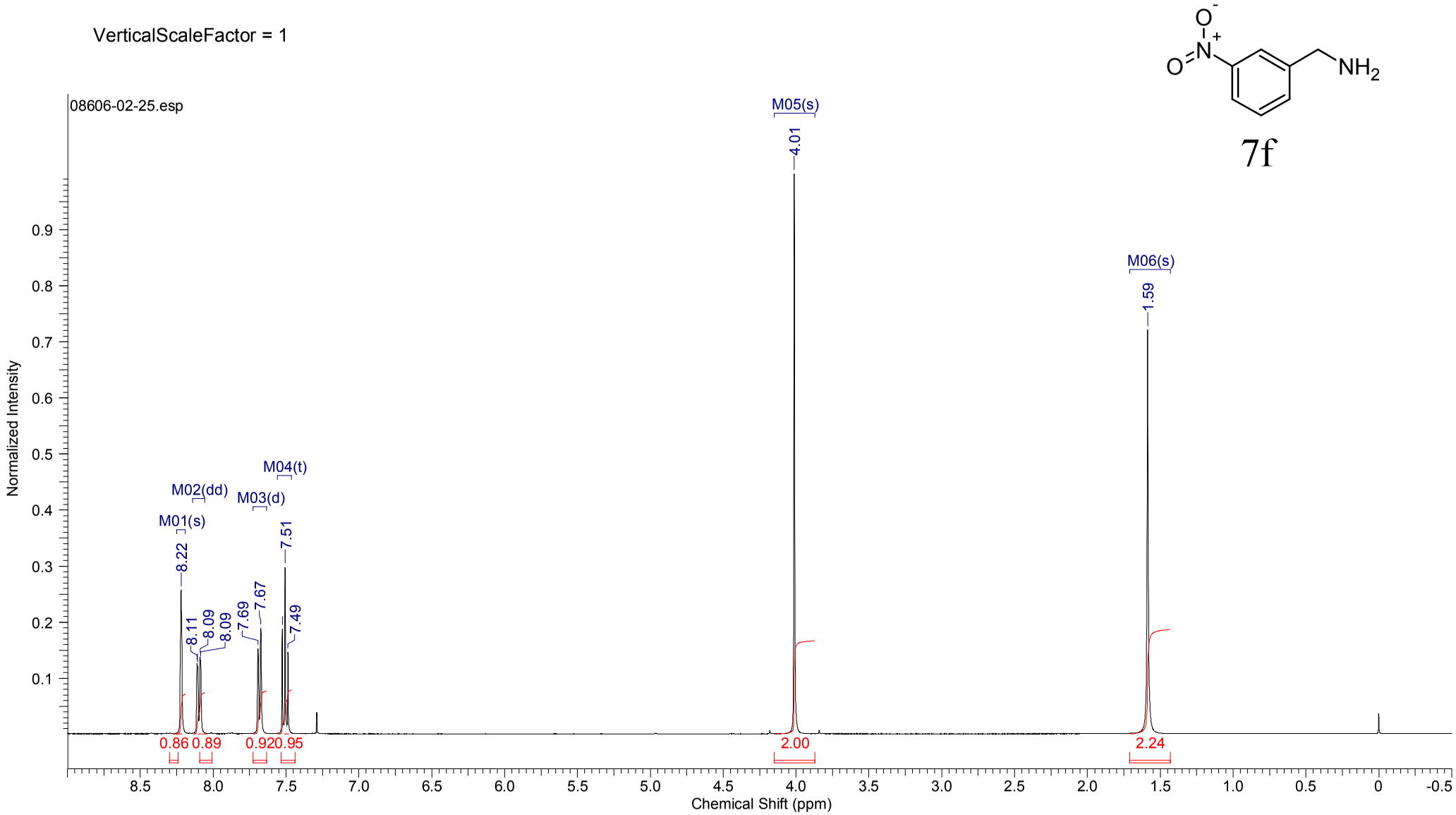
7e



2012/04/11 13:03:04

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012	**Date Stamp**	Mar 20 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2259.4998	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.22 (s, 1H), 8.10 (dd, *J*=1.28, 8.23 Hz, 1H), 7.68 (d, *J*=7.68 Hz, 1H), 7.51 (t, *J*=7.87 Hz, 1H), 4.01 (s, 2H), 1.59 (s, 2H)



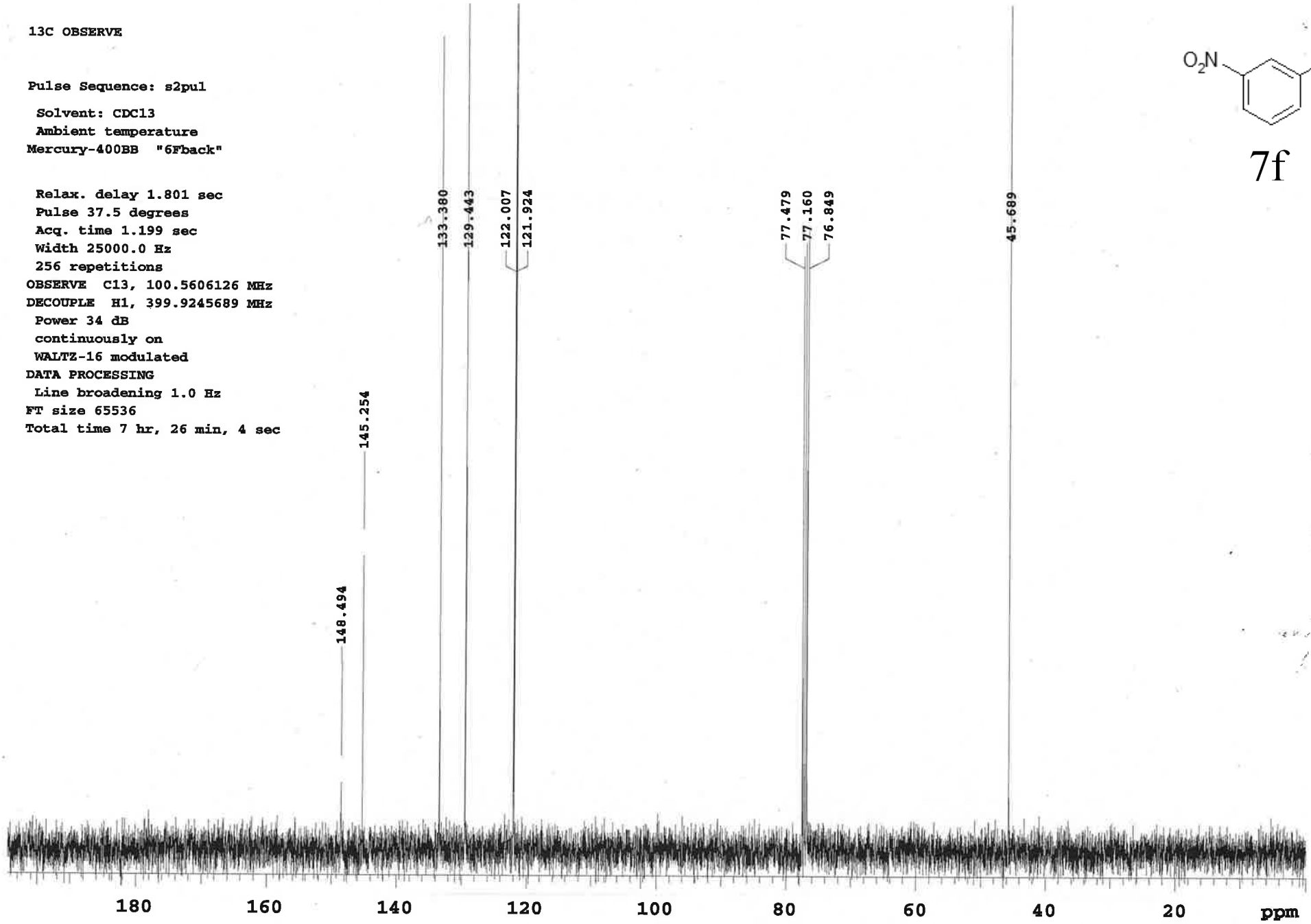
7f

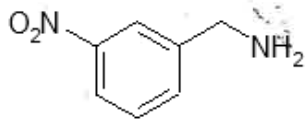
13C OBSERVE

Pulse Sequence: s2pul

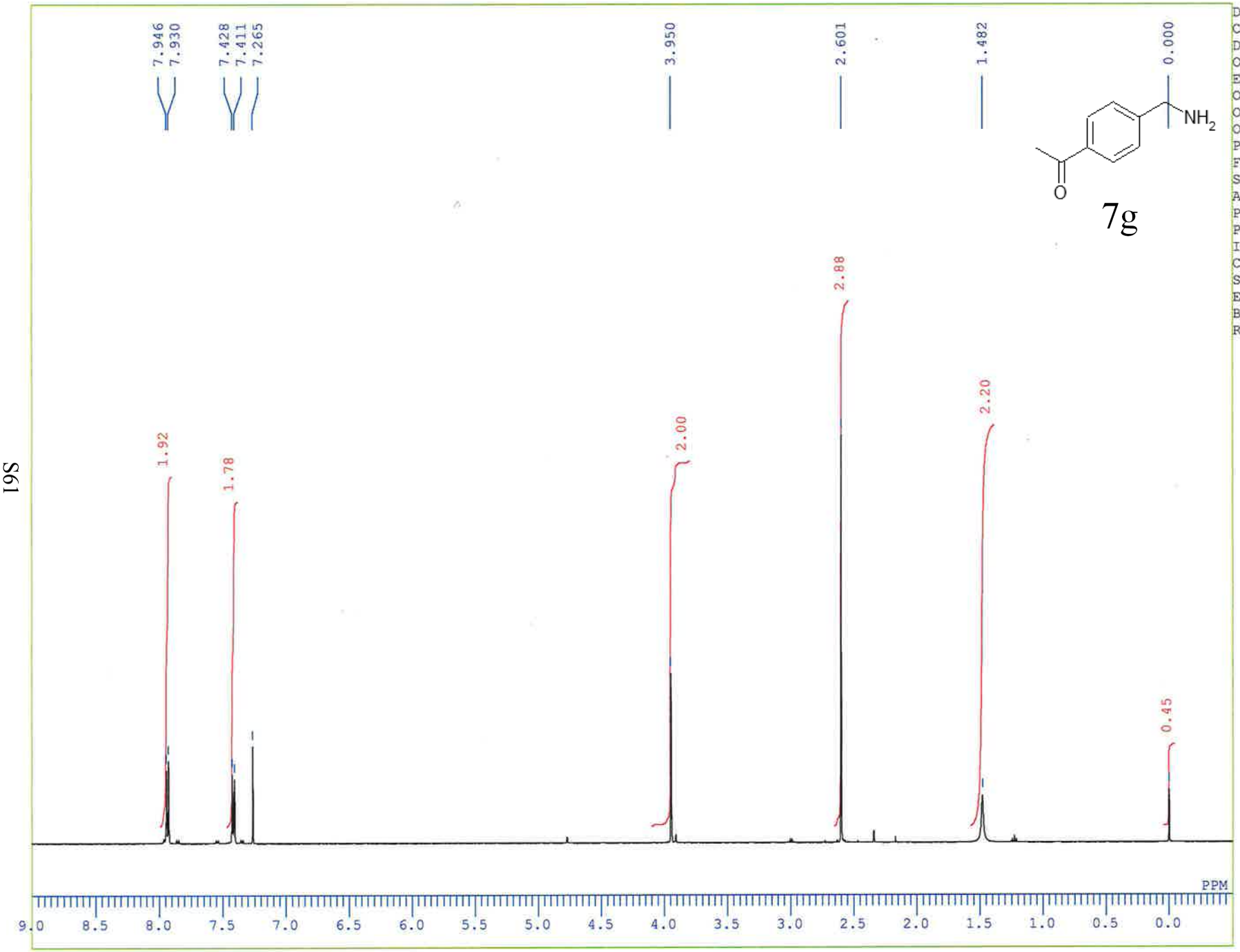
Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
256 repetitions
OBSERVE C13, 100.5606126 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 4 sec

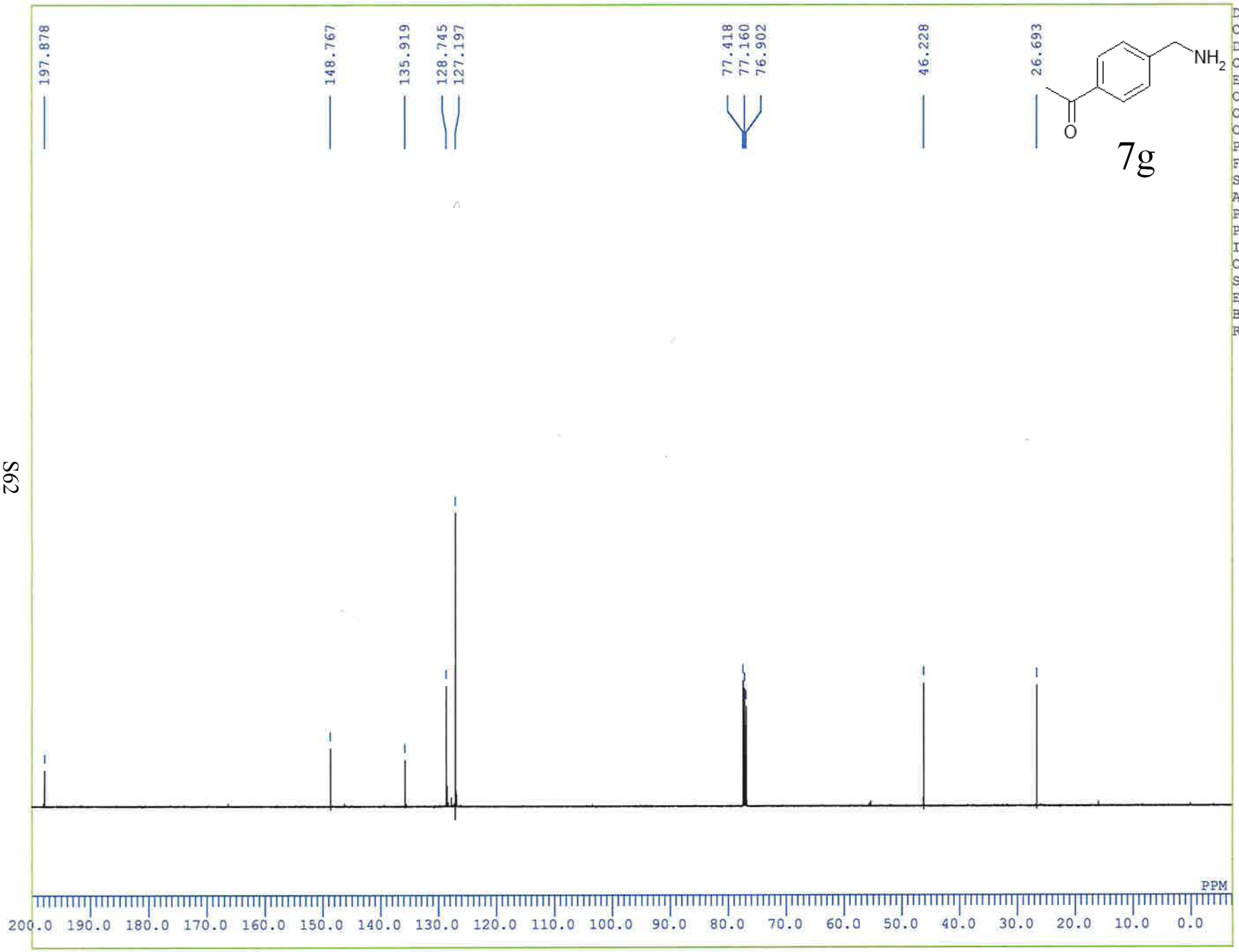




7f



DFILE	NOM123054.1
COMNT	single_pulse
DATIM	2012-03-05 14:34:31
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	490.15 MHz
OBSET	9.16 KHz
OBFIN	7.60 Hz
POINT	16384
FREQU	9191.18 Hz
SCANS	8
ACQTM	1.7826 sec
PD	3.0000 sec
PW1	8.55 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



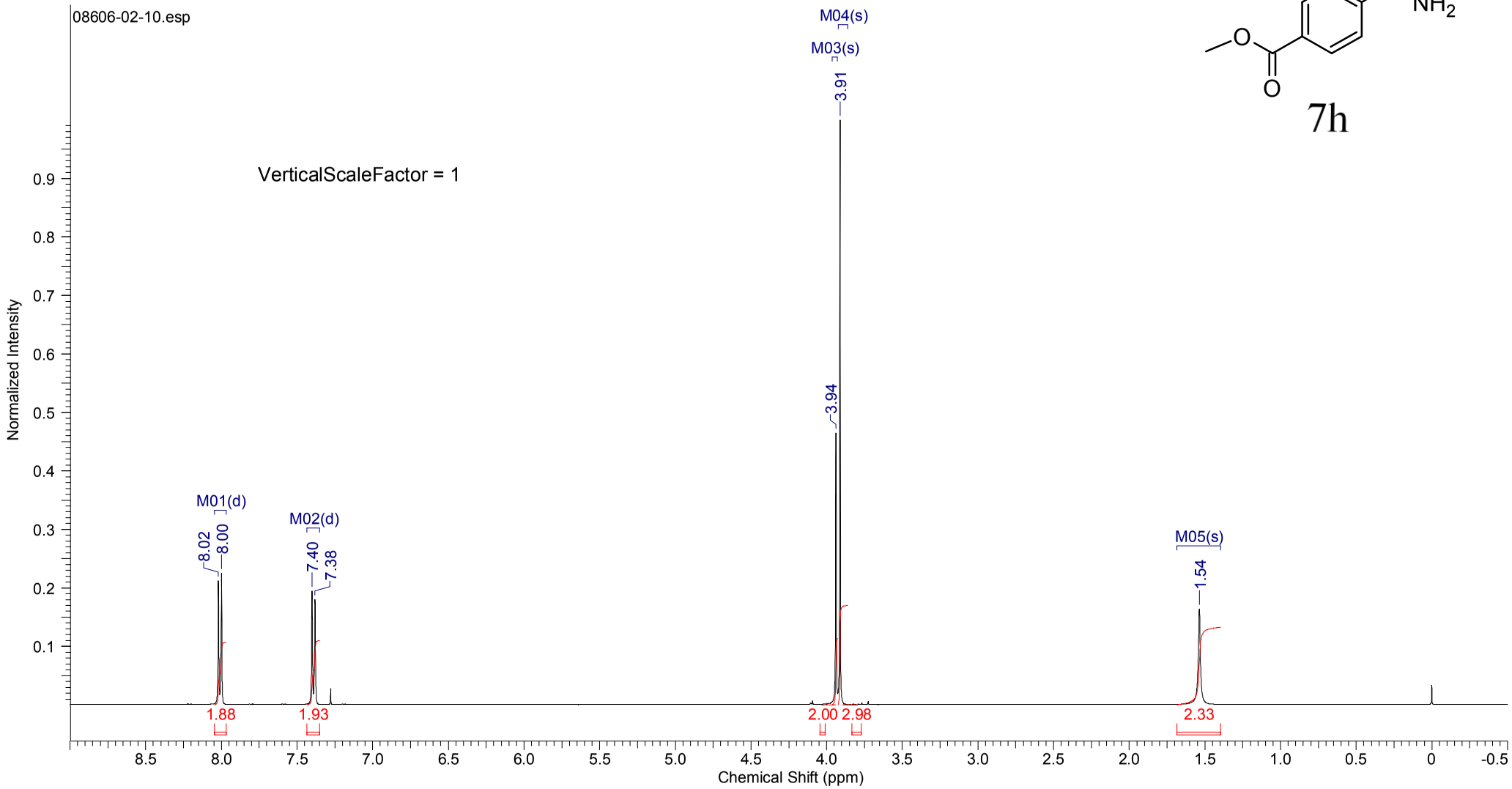
DFILE	NOM123247.1
COMNT	single pulse decoupl
DATIM	2012-03-24 09:24:25
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	123.26 MHz
OBSET	2.31 KHz
OBFIN	6.71 Hz
POINT	32768
FREQU	38580.25 Hz
SCANS	2048
ACQTM	0.8493 sec
PD	2.0000 sec
PW1	3.20 usec
IRNUC	1H
CTEMP	24.8 c
SLVNT	CDCL3
EXREF	77.16 ppm
BF	0.12 Hz
RGAIN	50

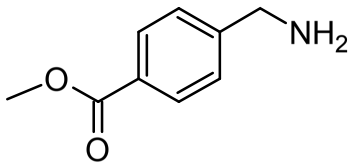
2012/04/11 11:03:38

Formula $C_9H_{11}NO_2$	FW 165.1891

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2255.1084	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.01 (d, *J*=8.05 Hz, 2H), 7.39 (d, *J*=8.05 Hz, 2H), 3.94 (s, 2H), 3.91 (s, 3H), 1.54 (s, 2H)





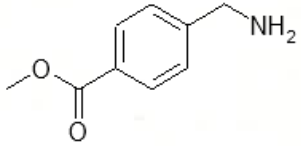
7h

13C OBSERVE

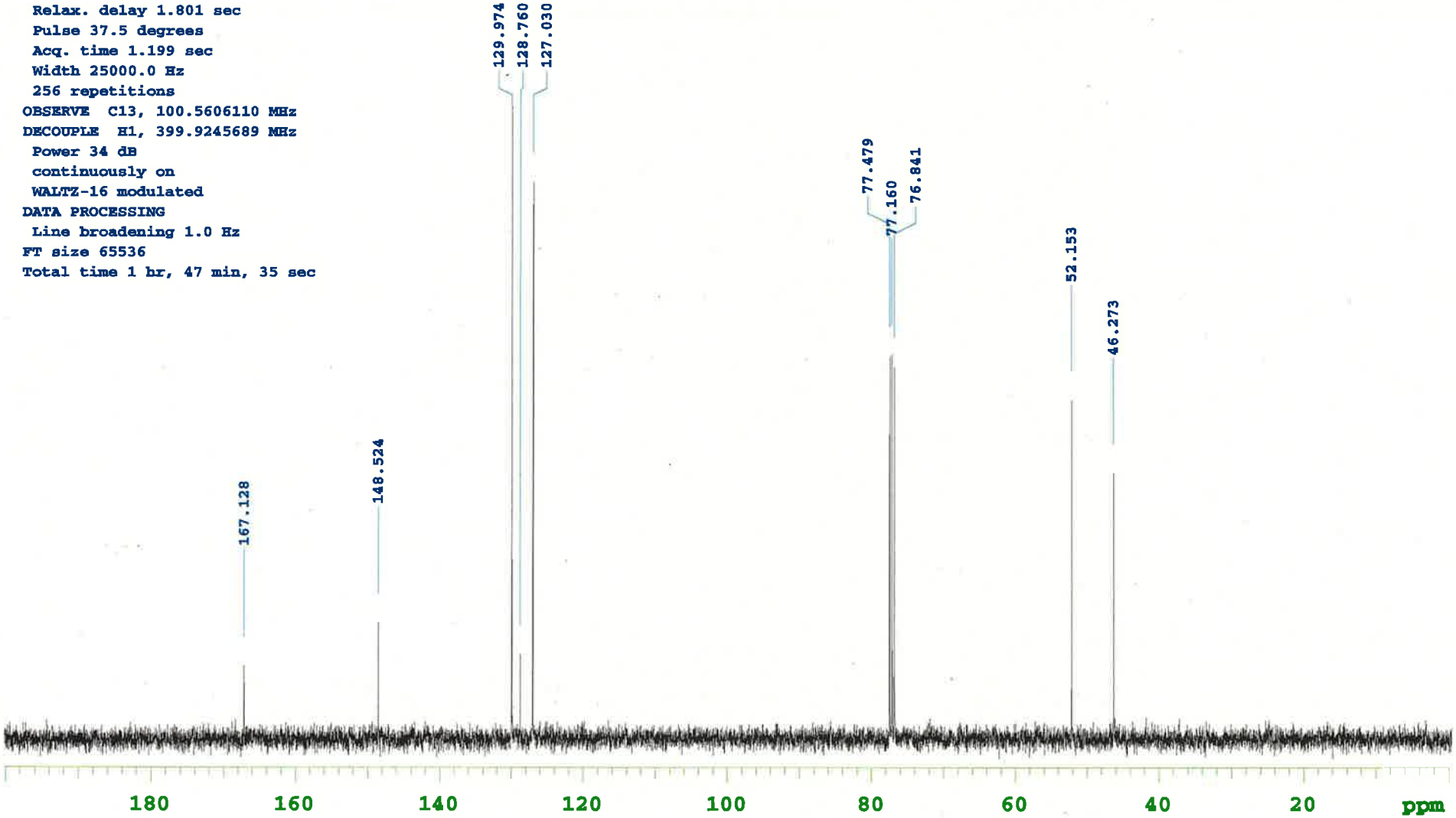
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
256 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



7h

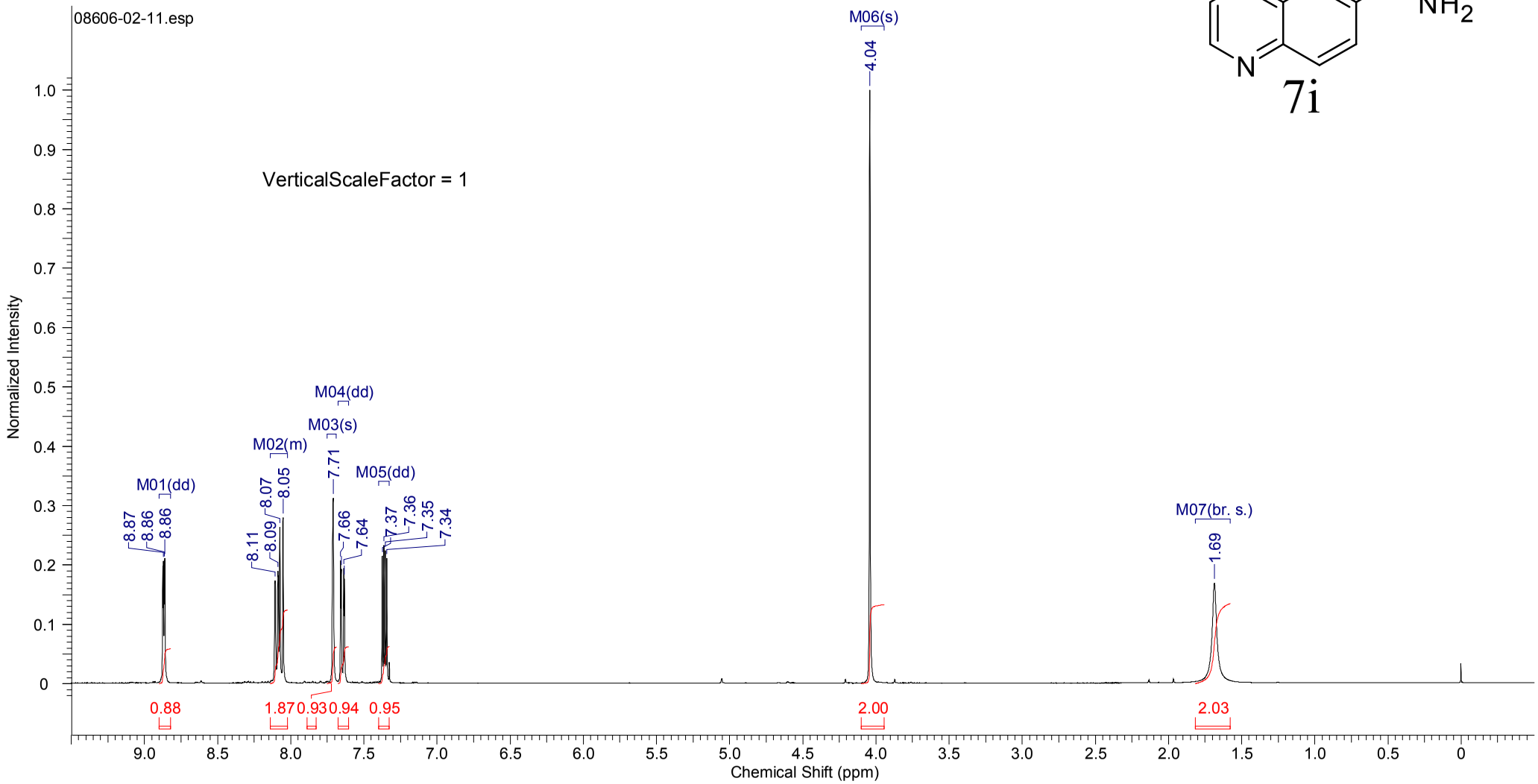


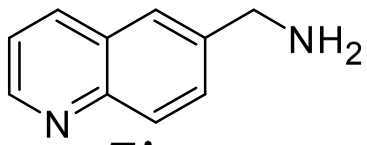
2012/04/11 11:07:46

Formula $C_{10}H_{10}N_2$	FW 158.1998

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Solvent**	CHLOROFORM-d		
Spectrum Offset (Hz)	2274.1375	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.87 (dd, *J*=1.65, 4.21 Hz, 1H), 8.02-8.14 (m, 2H), 7.71 (s, 1H), 7.65 (dd, *J*=2.01, 8.60 Hz, 1H), 7.36 (dd, *J*=4.21, 8.23 Hz, 1H), 4.04 (s, 2H), 1.69 (br. s., 2H)





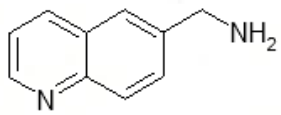
7i

13C OBSERVE

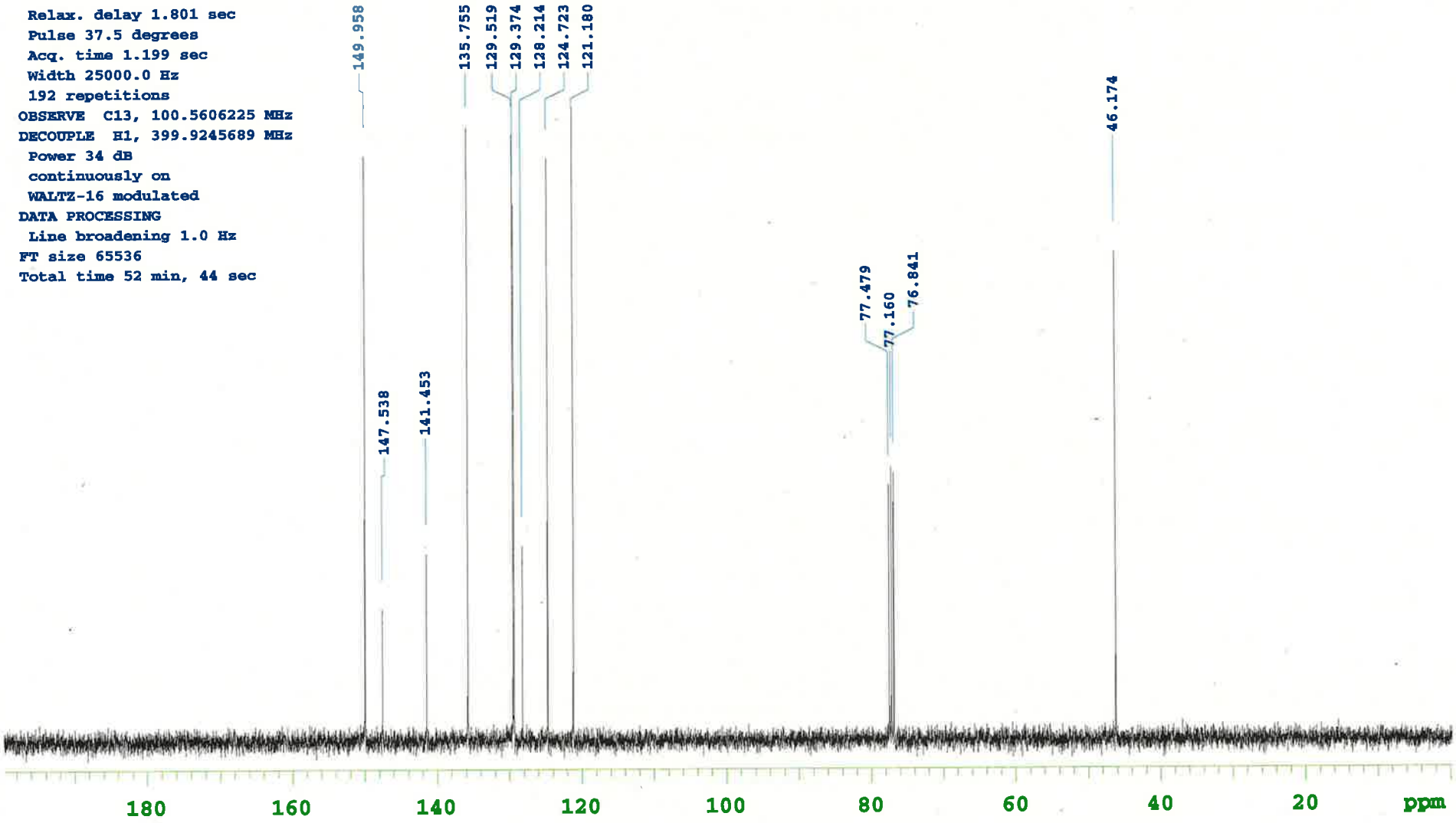
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.5606225 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 52 min, 44 sec



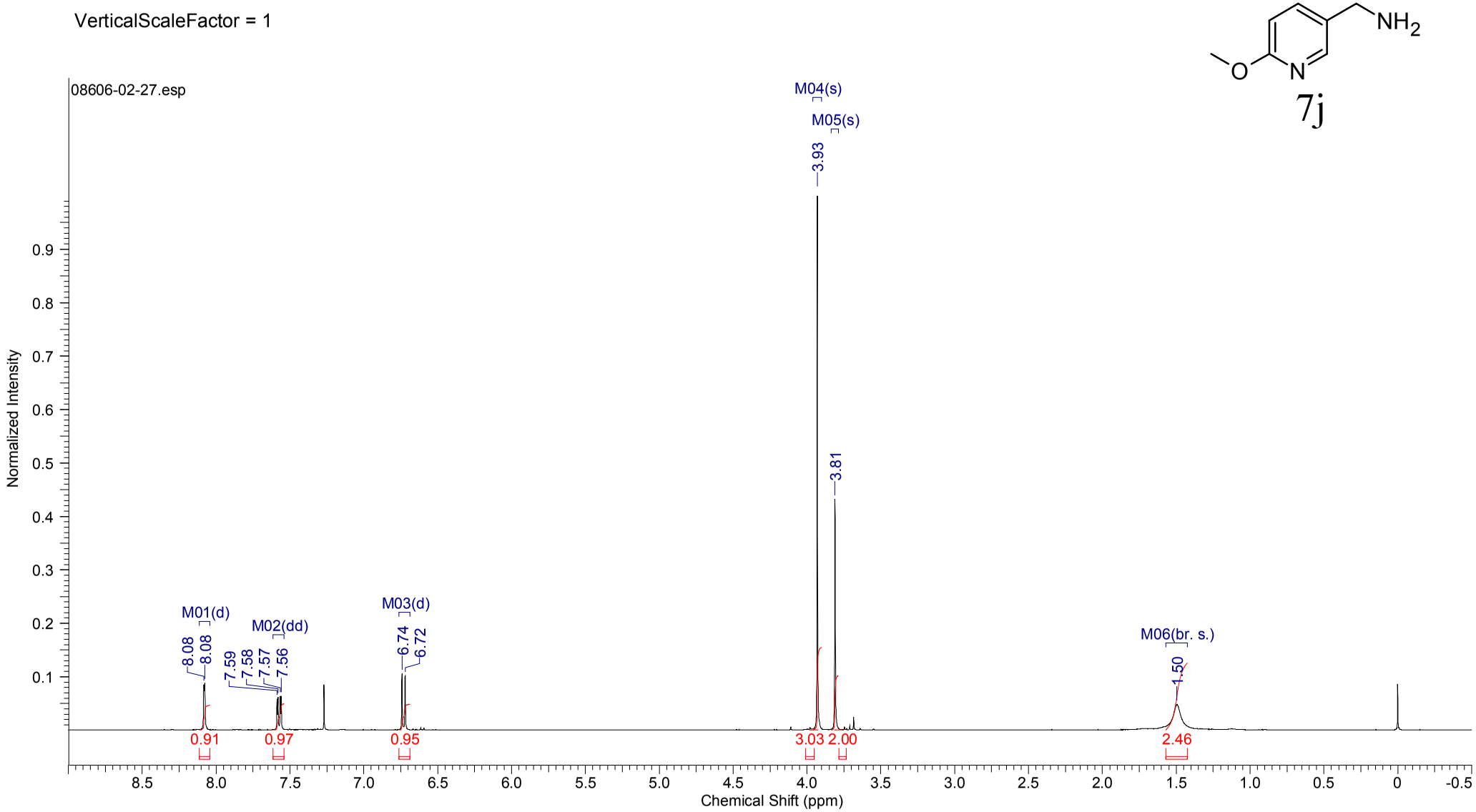
7i



2012/04/11 13:09:05

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012	**Date Stamp**	Mar 20 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	20.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2251.4490	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.08 (d, *J*=2.20 Hz, 1H), 7.57 (dd, *J*=2.20, 8.42 Hz, 1H), 6.73 (d, *J*=8.78 Hz, 1H), 3.93 (s, 3H), 3.81 (s, 2H), 1.50 (br. s., 2H)



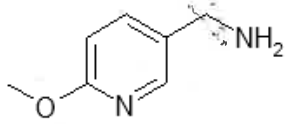
7j

13C OBSERVE

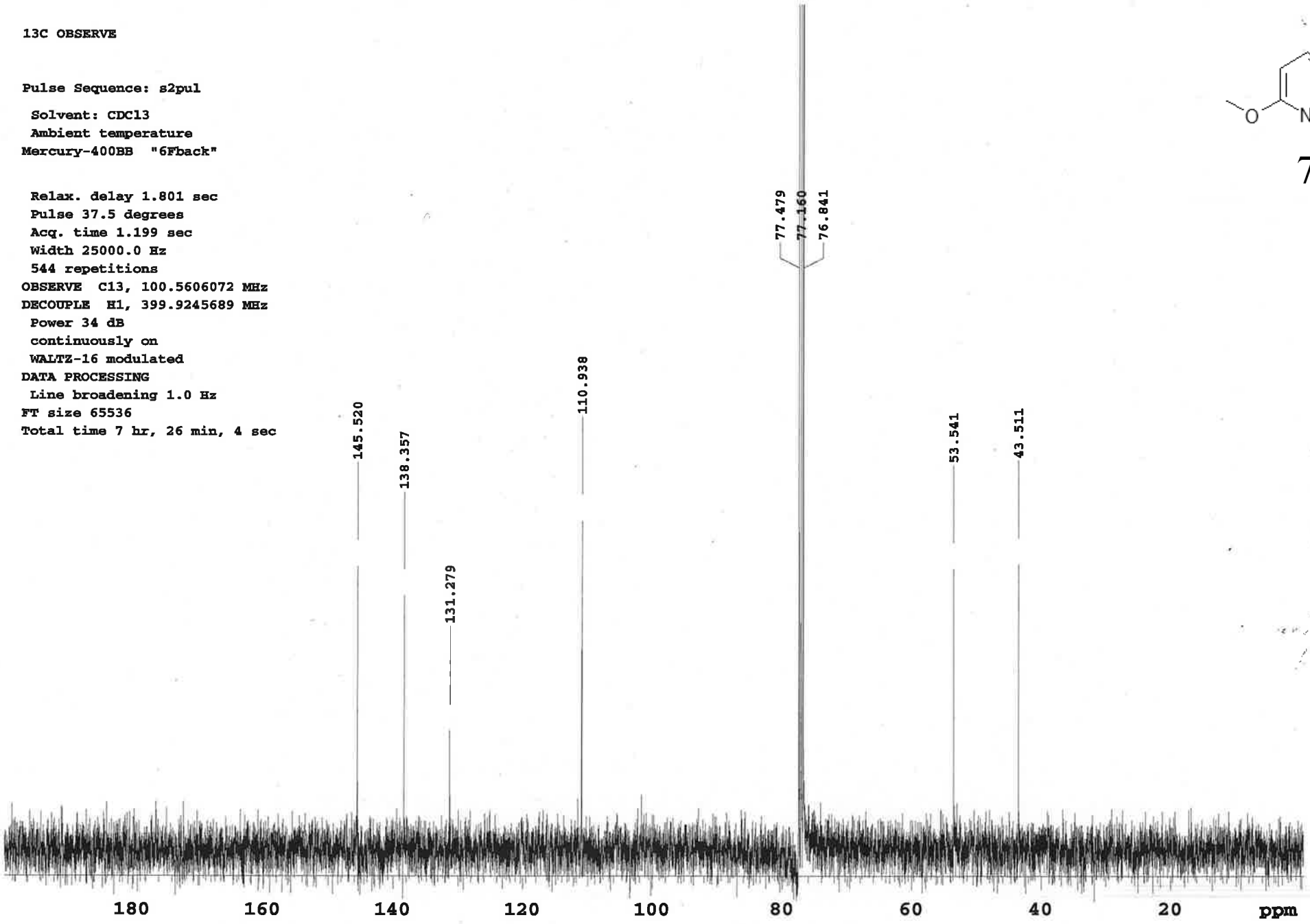
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
544 repetitions
OBSERVE C13, 100.5606072 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 4 sec



7j

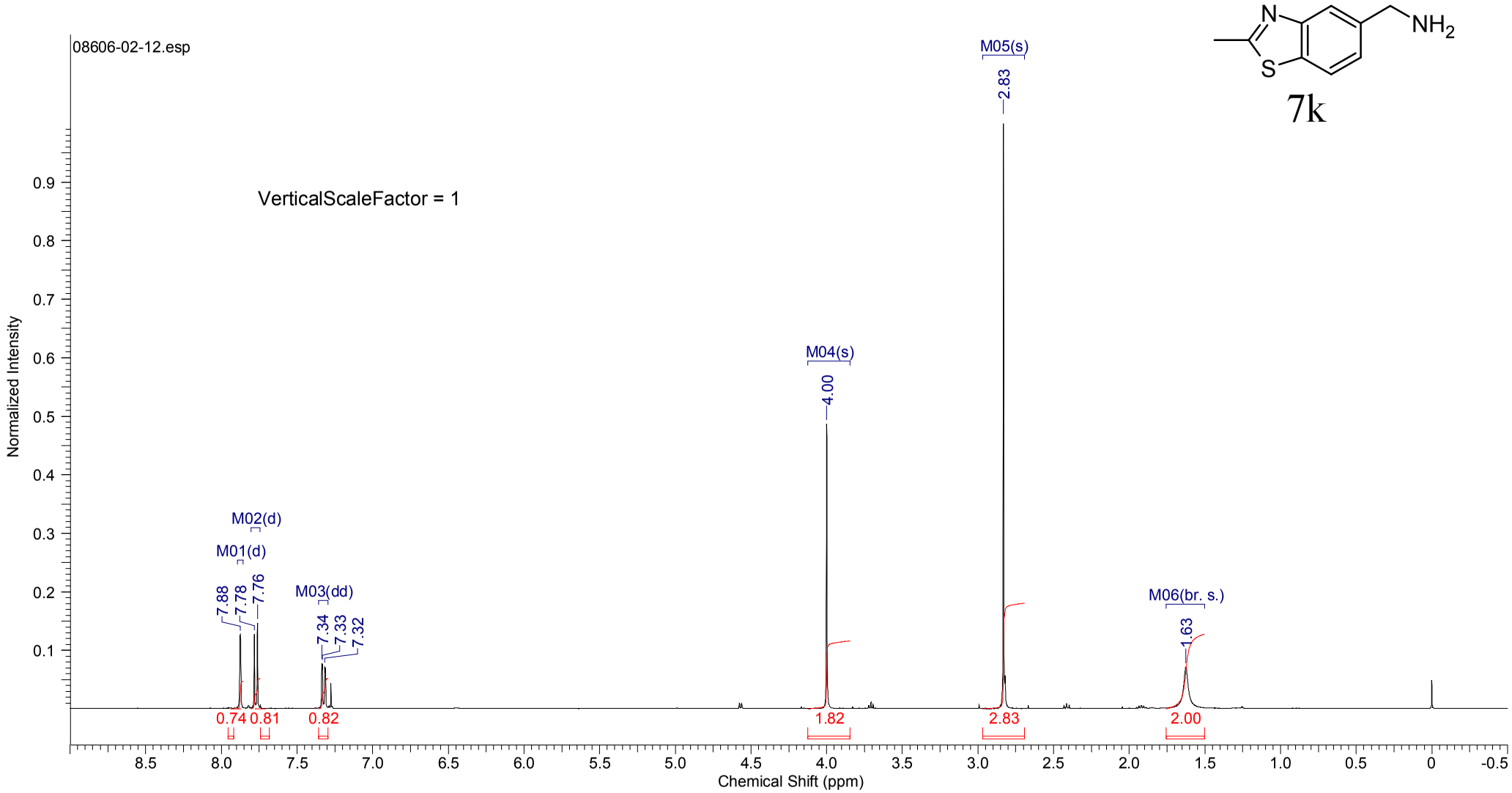


2012/04/11 11:13:28

Formula $C_9H_{10}N_2S$	FW 178.2541

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	12.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2254.3765	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.87 (d, *J*=0.73 Hz, 1H), 7.77 (d, *J*=8.05 Hz, 1H), 7.32 (dd, *J*=1.46, 8.05 Hz, 1H), 4.00 (s, 2H), 2.83 (s, 3H), 1.63 (br. s., 2H)



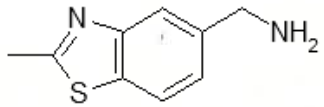
7k

13C OBSERVE

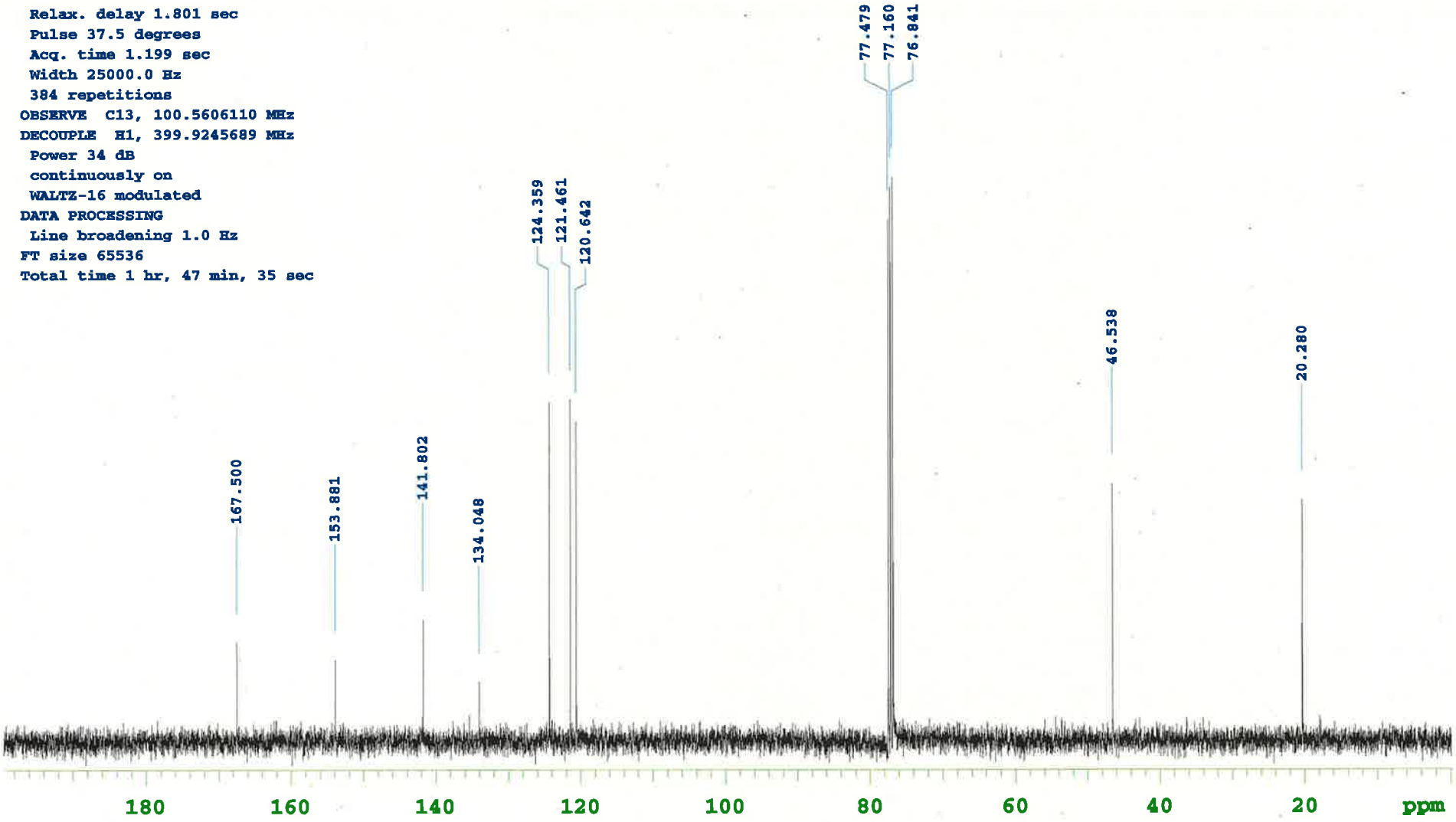
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
384 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



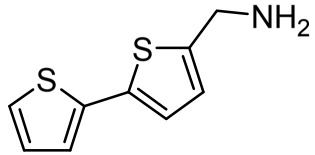
7k



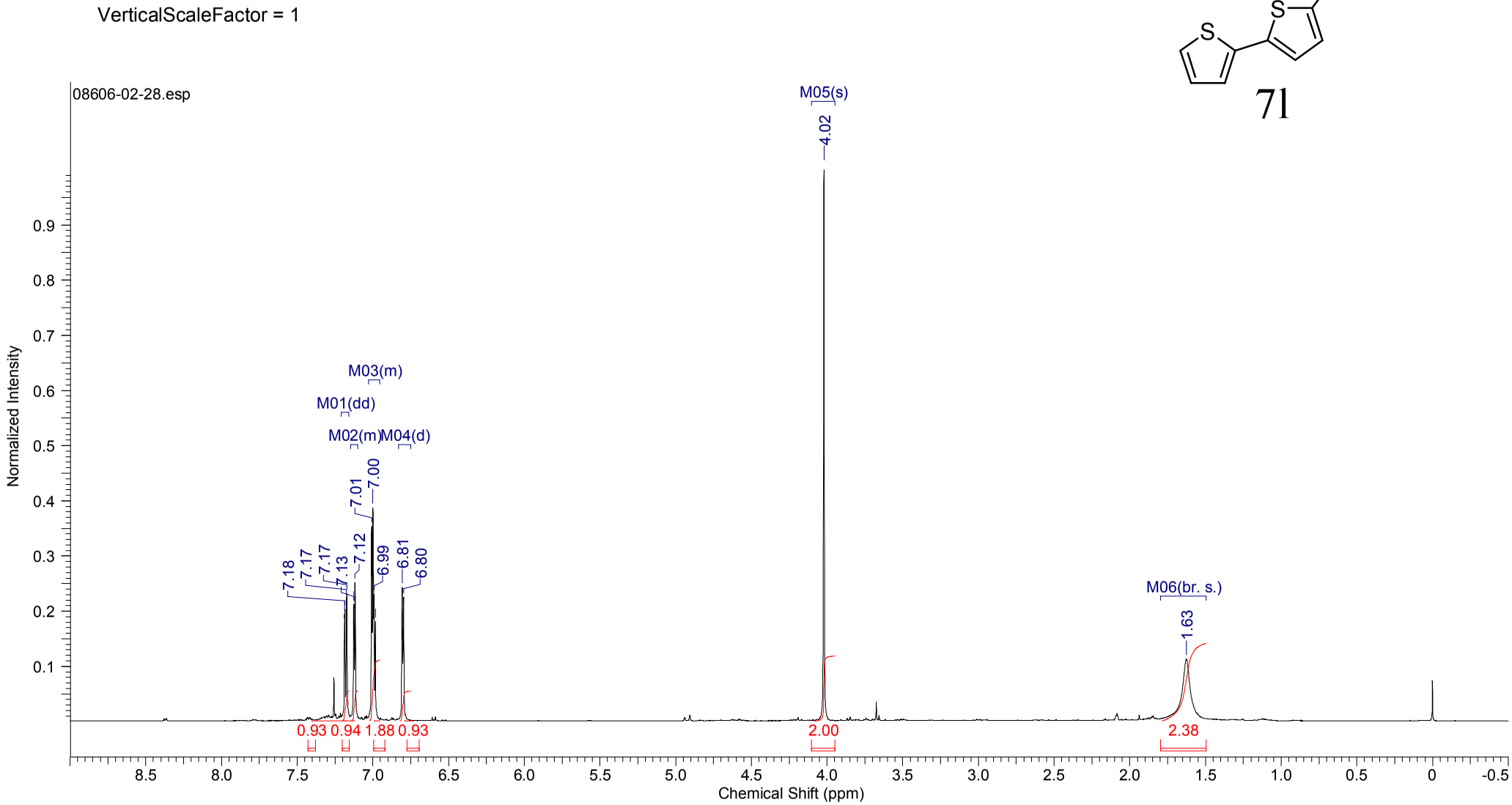
2012/04/11 13:15:01

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012	**Date Stamp**	Mar 20 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	14.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2246.6919	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.18 (dd, *J*=0.73, 5.12 Hz, 1H), 7.10-7.15 (m, 1H), 6.95-7.03 (m, 2H), 6.80 (d, *J*=3.29 Hz, 1H), 4.02 (s, 2H), 1.63 (br. s., 2H)



71

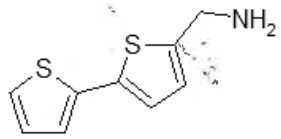


13C OBSERVE

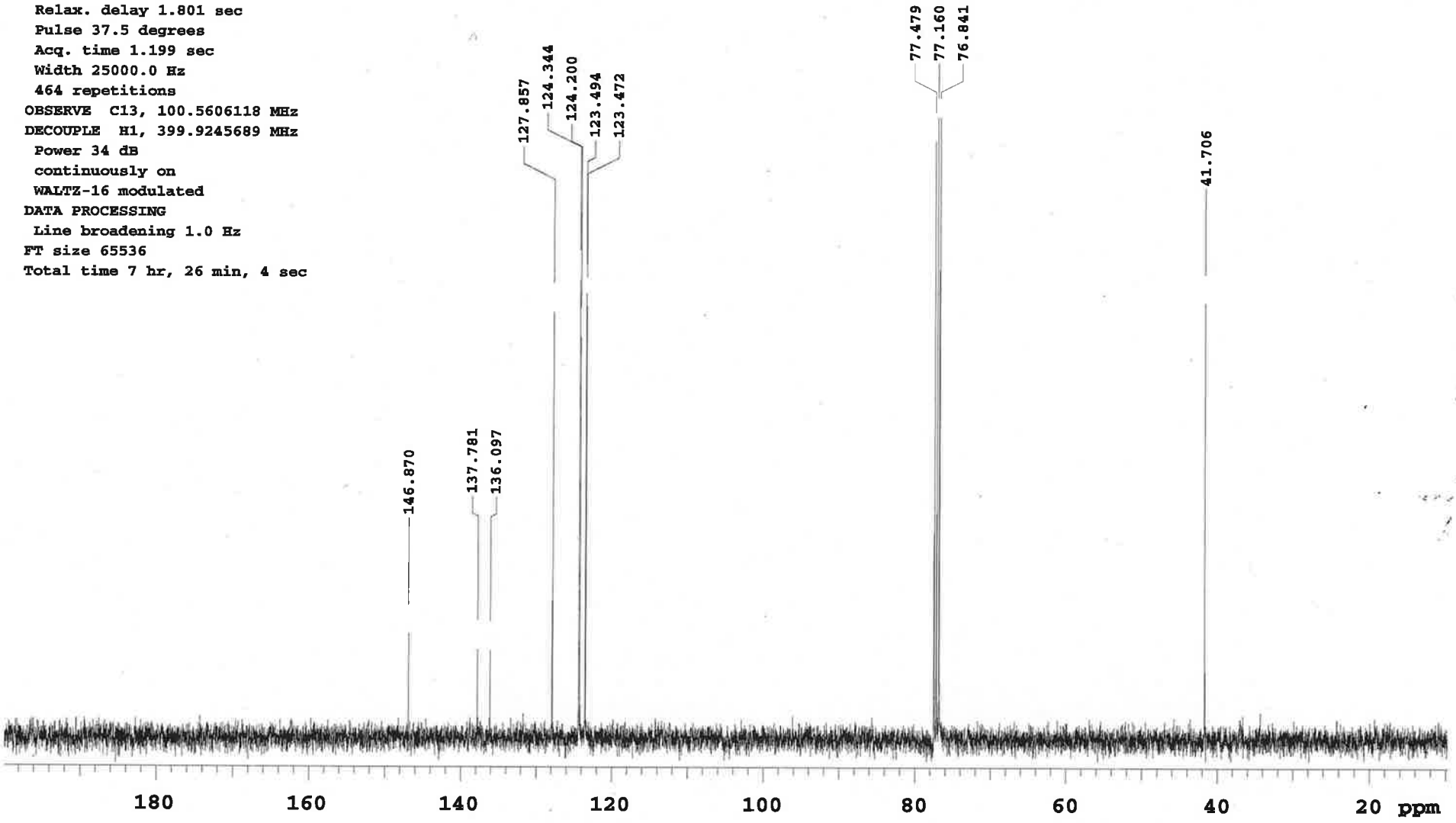
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
464 repetitions
OBSERVE C13, 100.5606118 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 26 min, 4 sec



71

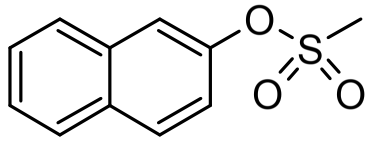


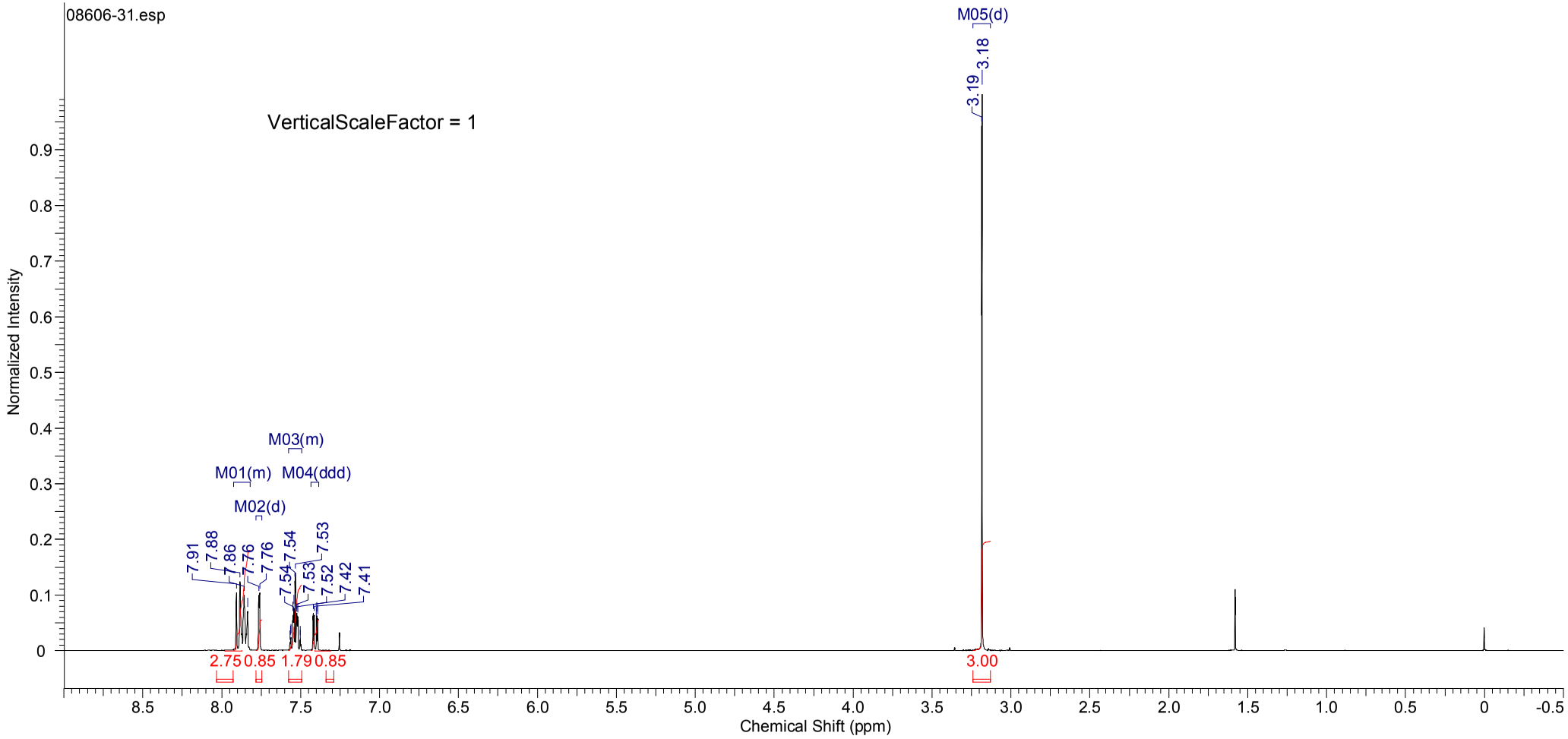
2011/11/05 12:25:17

Formula $C_{11}H_{10}O_3S$	FW 222.2603

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Oct 23 2011	**Date Stamp**	Oct 23 2011
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	14.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2246.3259	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.82-7.92 (m, 3H), 7.76 (d, *J*=2.20 Hz, 1H), 7.49-7.58 (m, 2H), 7.41 (ddd, *J*=1.10, 2.38, 8.97 Hz, 1H), 3.18 (d, *J*=1.10 Hz, 3H)



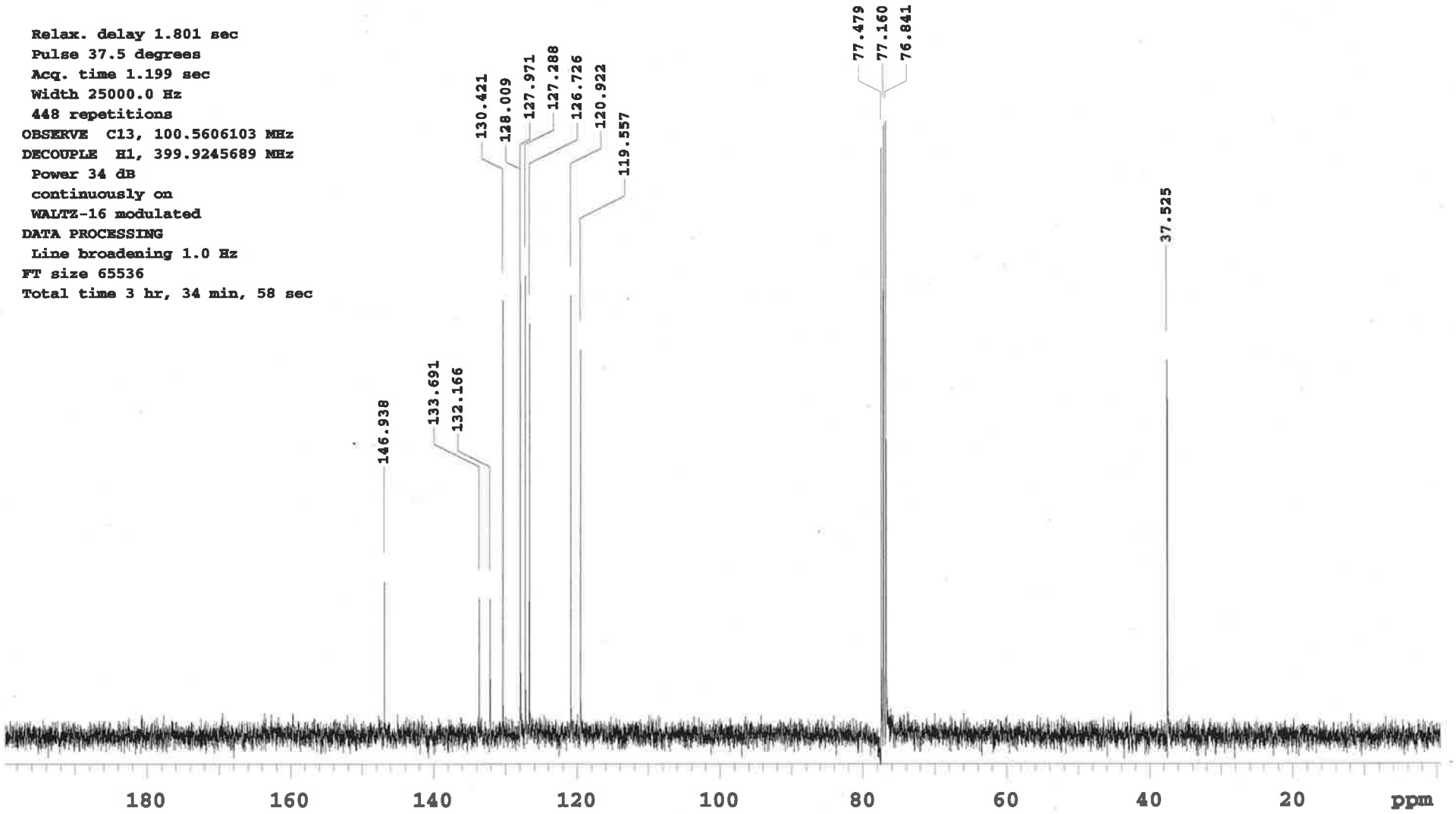


13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 08606-31
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
448 repetitions
OBSERVE C13, 100.5606103 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 34 min, 58 sec

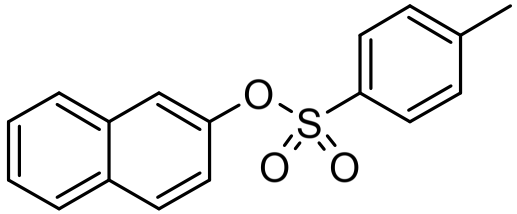


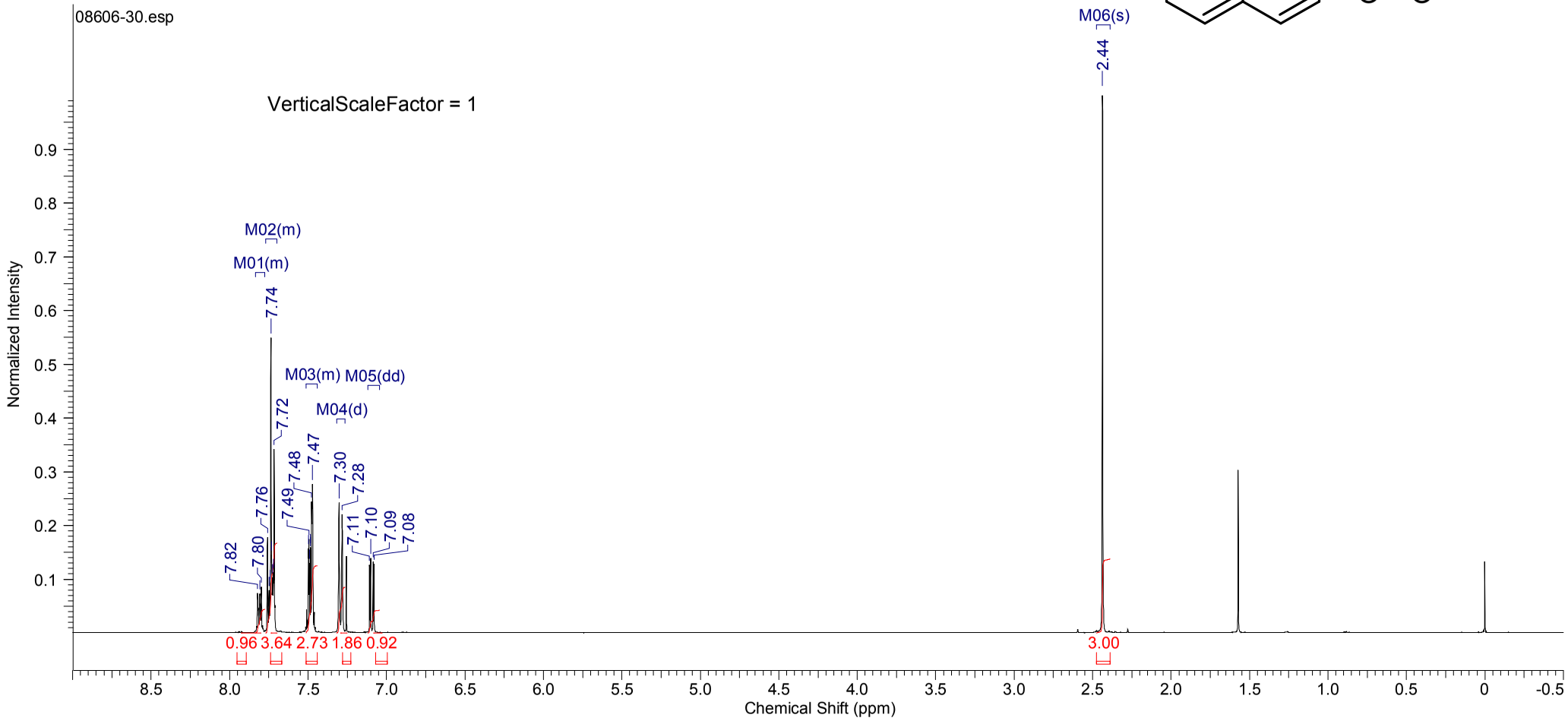
2011/11/05 12:22:40

Formula $C_{17}H_{14}O_3S$	FW 298.3563

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Oct 23 2011	**Date Stamp**	Oct 23 2011
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	14.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2246.6919	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.77-7.84 (m, 1H), 7.70-7.77 (m, 4H), 7.44-7.51 (m, 3H), 7.29 (d, *J*=8.05 Hz, 2H), 7.09 (dd, *J*=2.38, 8.97 Hz, 1H), 2.44 (s, 3H)



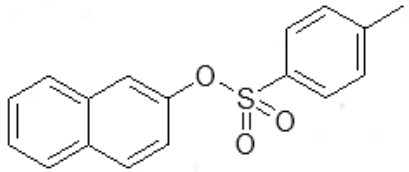


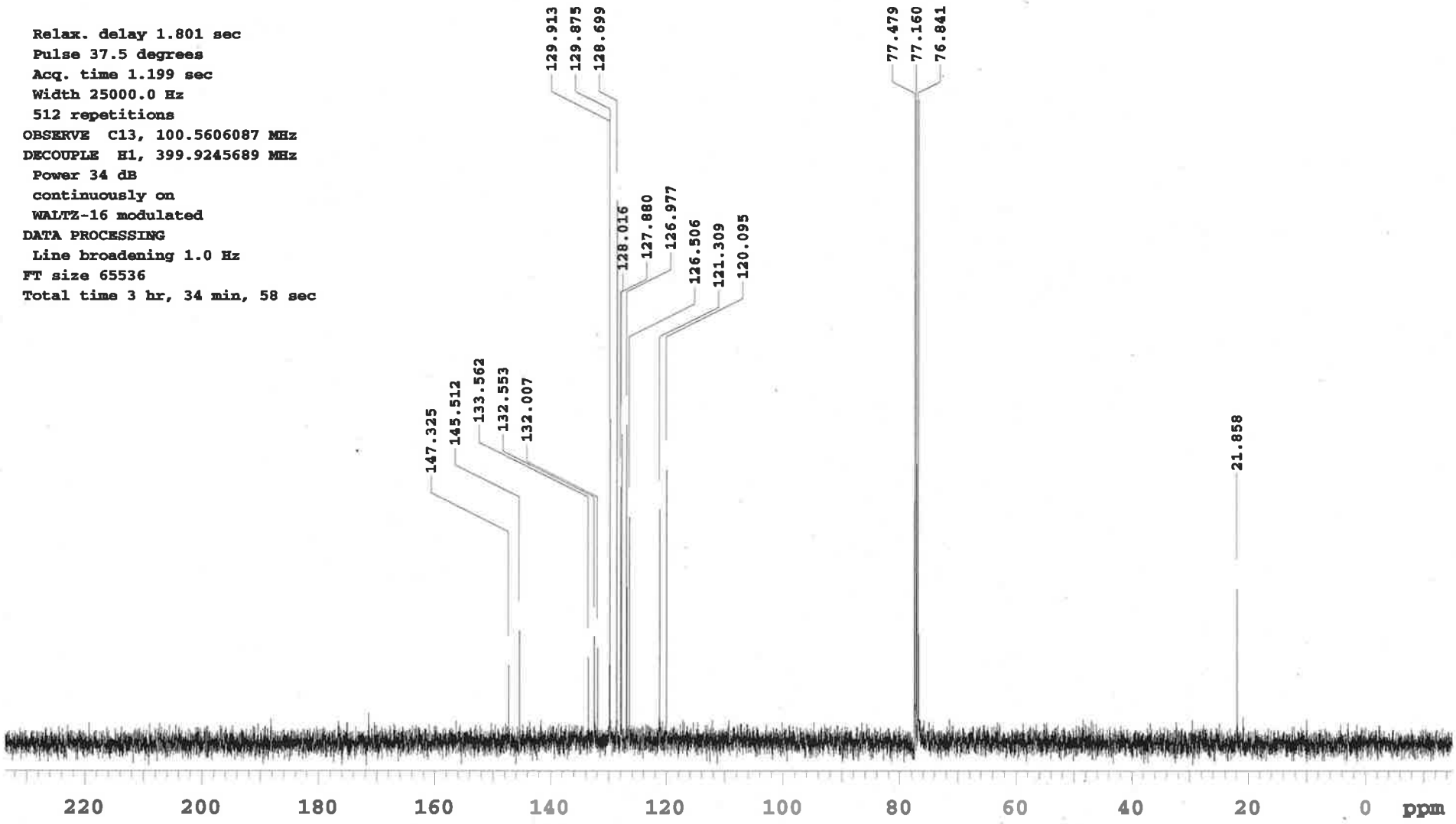
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 08606-30
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
512 repetitions
OBSERVE C13, 100.5606087 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 34 min, 58 sec



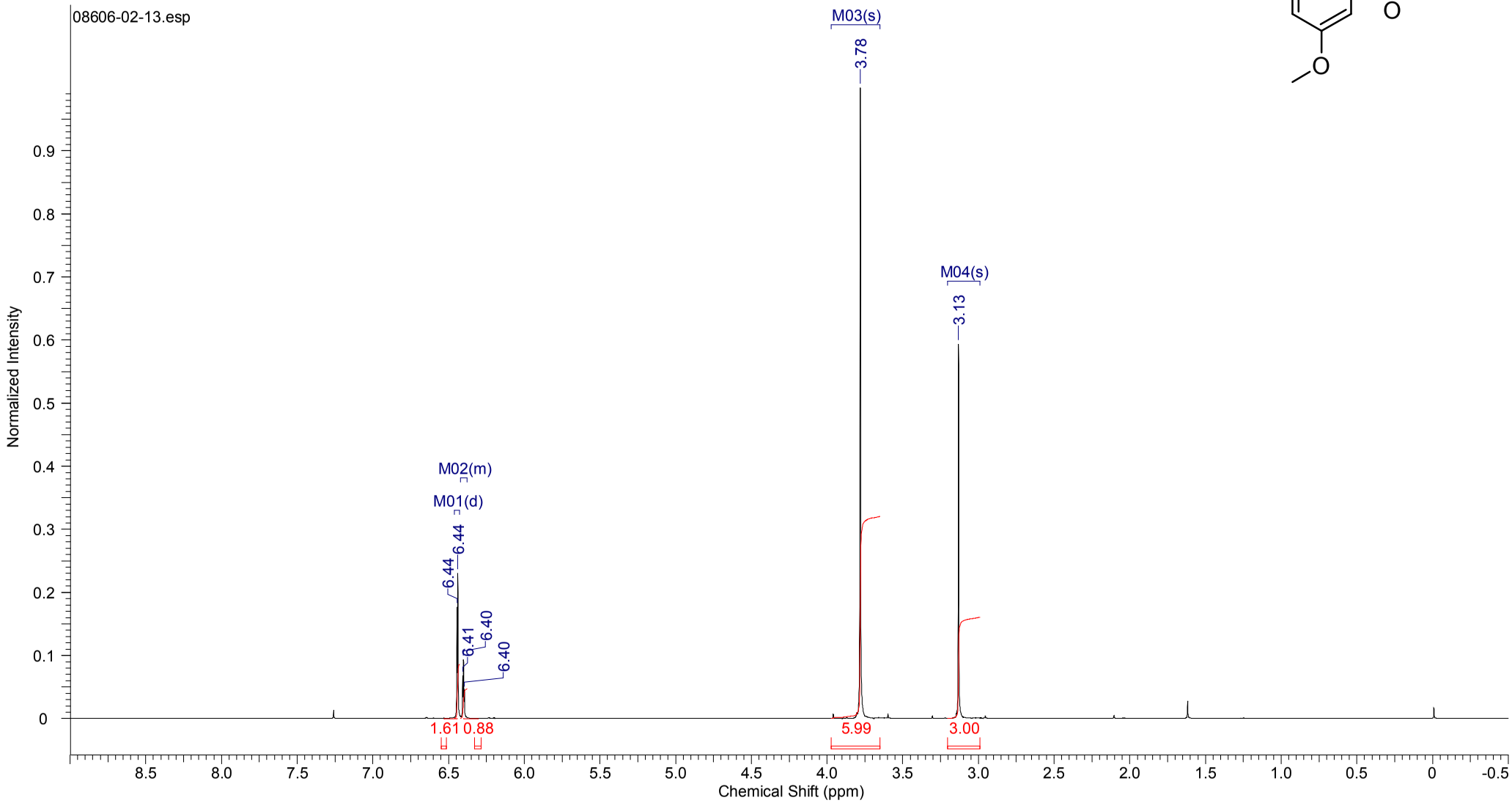


2012/04/11 11:17:39

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2247.6414	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 6.44 (d, *J*=2.20 Hz, 2H), 6.38-6.42 (m, 1H), 3.78 (s, 6H), 3.13 (s, 3H)

VerticalScaleFactor = 1

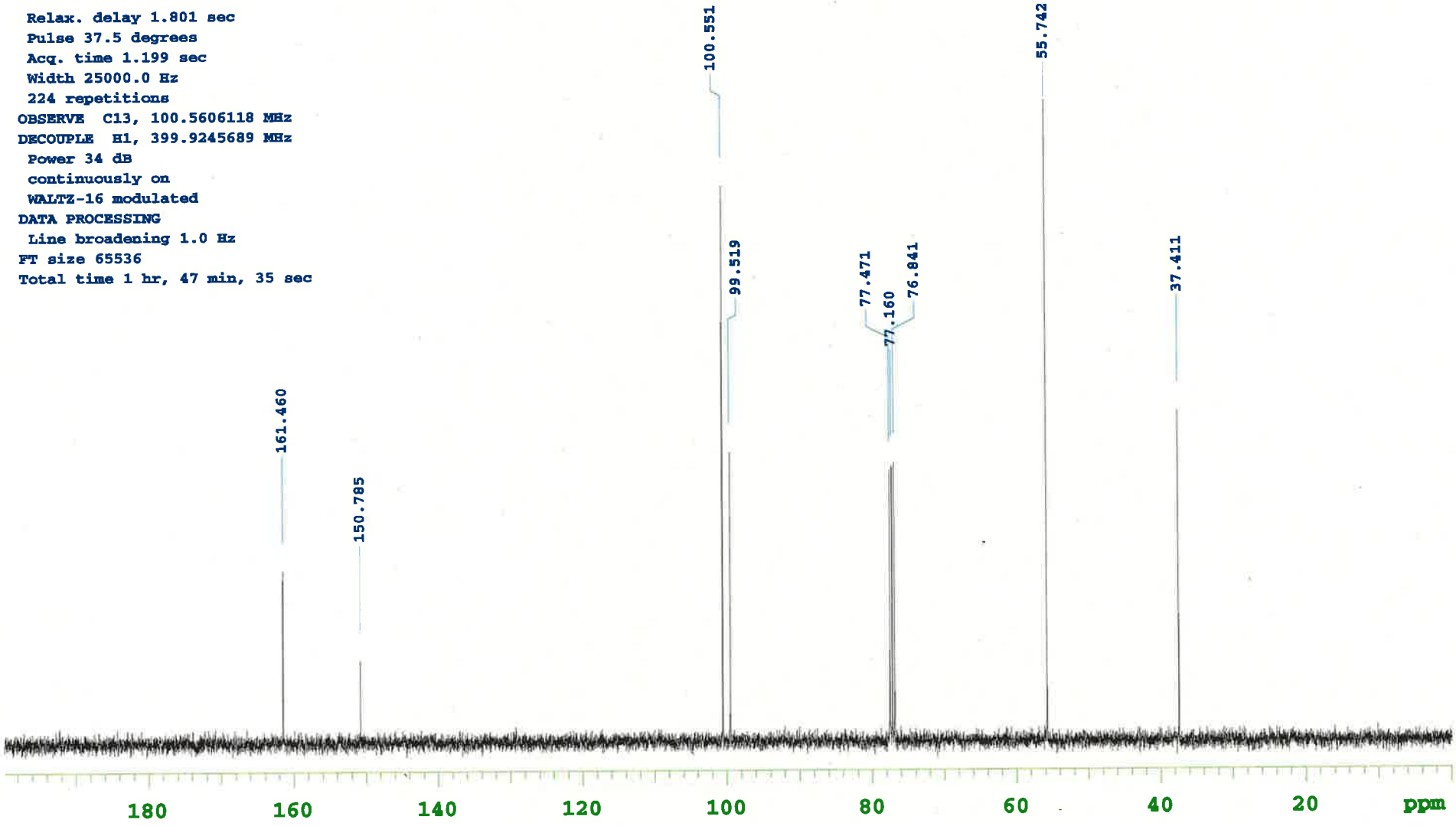


13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
224 repetitions
OBSERVE C13, 100.5606118 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec

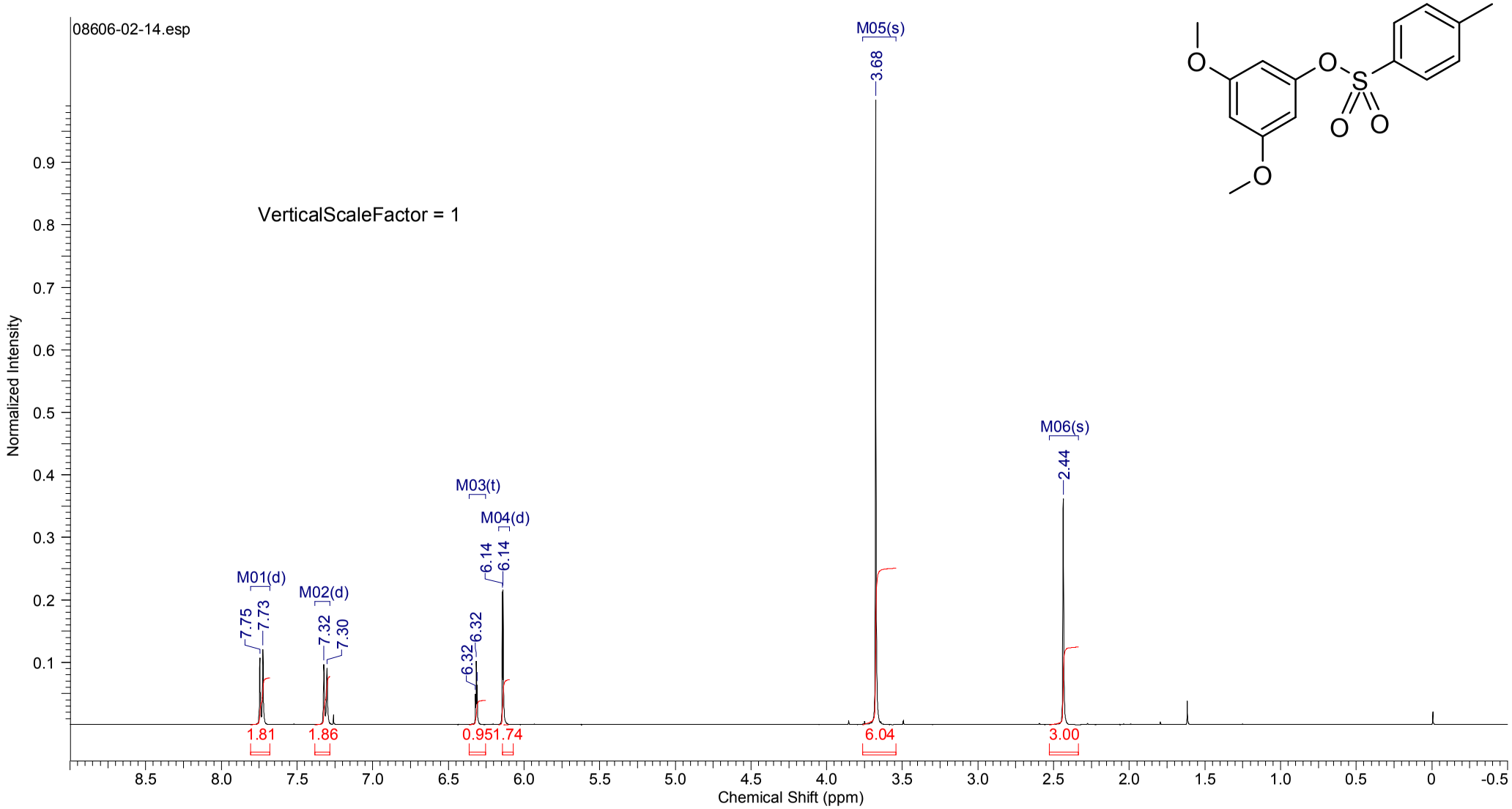


2012/05/11 14:45:25

Formula $C_{15}H_{16}O_5S$	FW 308.3495

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	6.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2247.6414	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.74 (d, *J*=8.42 Hz, 2H), 7.31 (d, *J*=8.05 Hz, 2H), 6.32 (t, *J*=2.20 Hz, 1H), 6.14 (d, *J*=2.20 Hz, 2H), 3.68 (s, 7H), 2.44 (s, 3H)

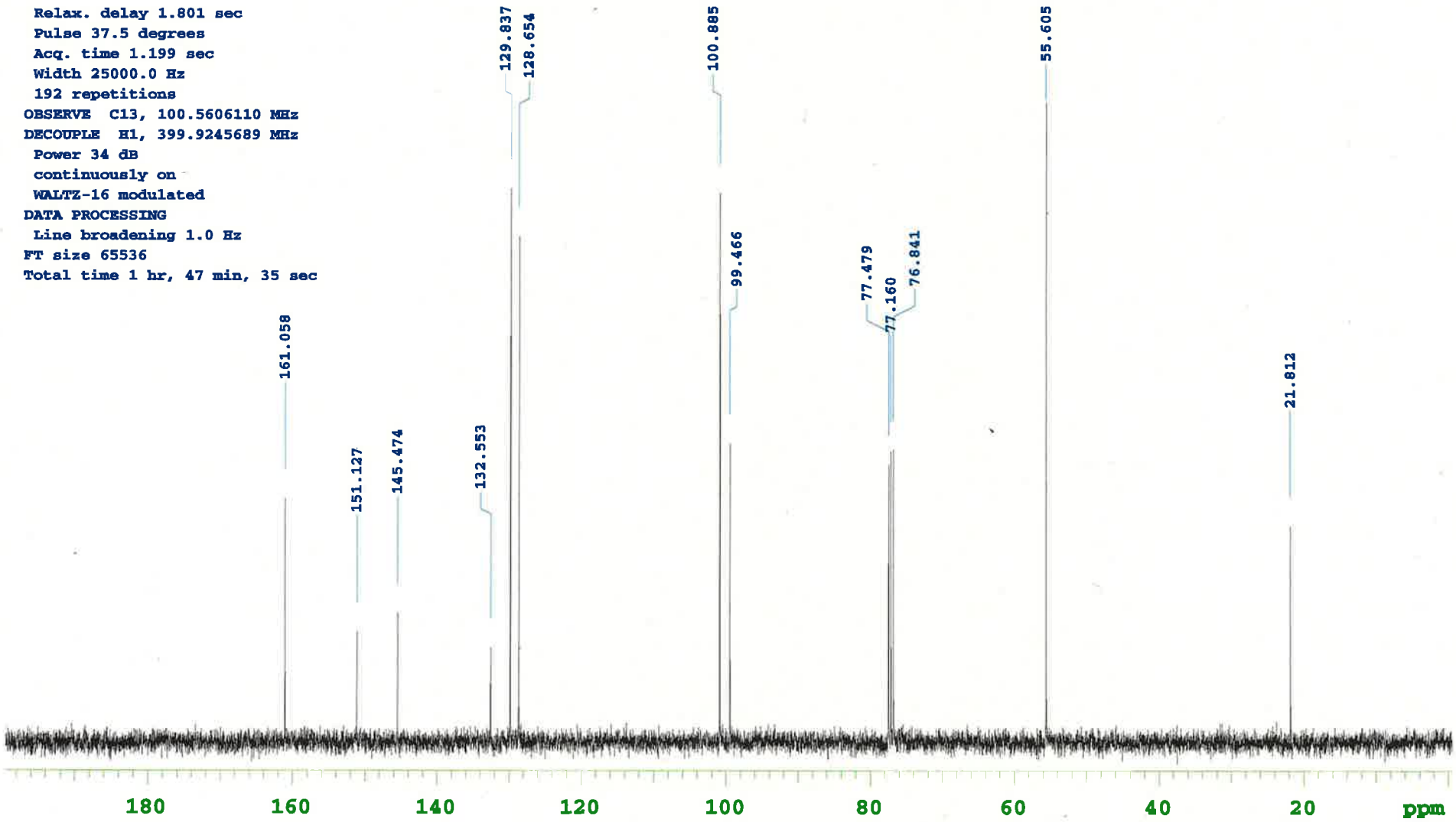


13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec

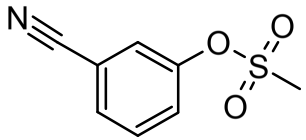


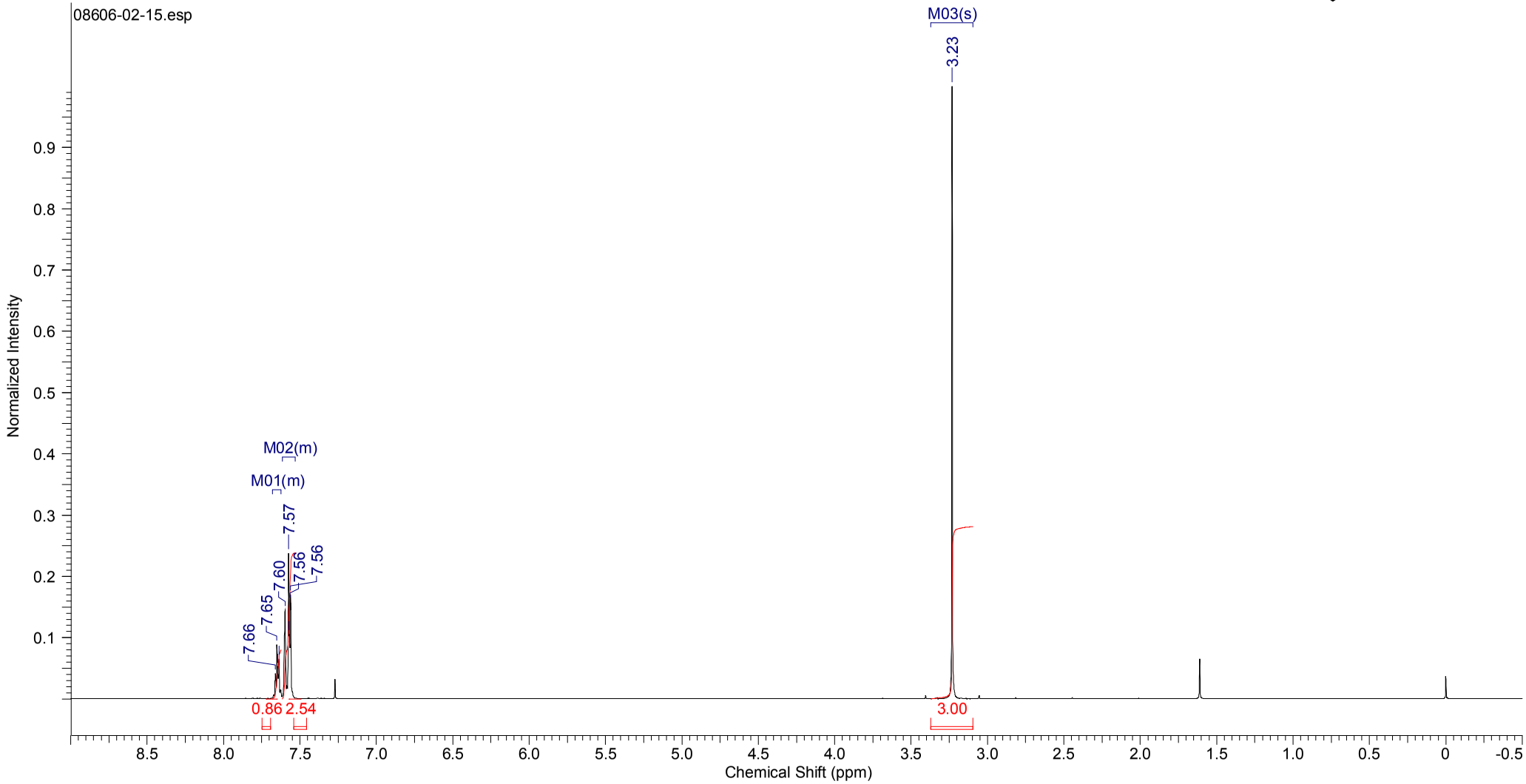
2012/04/11 11:27:44

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	16.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2252.1809	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.62-7.68 (m, 1H), 7.53-7.61 (m, 3H), 3.23 (s, 3H)

VerticalScaleFactor = 1



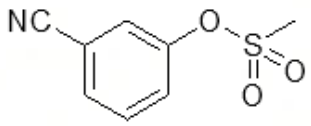


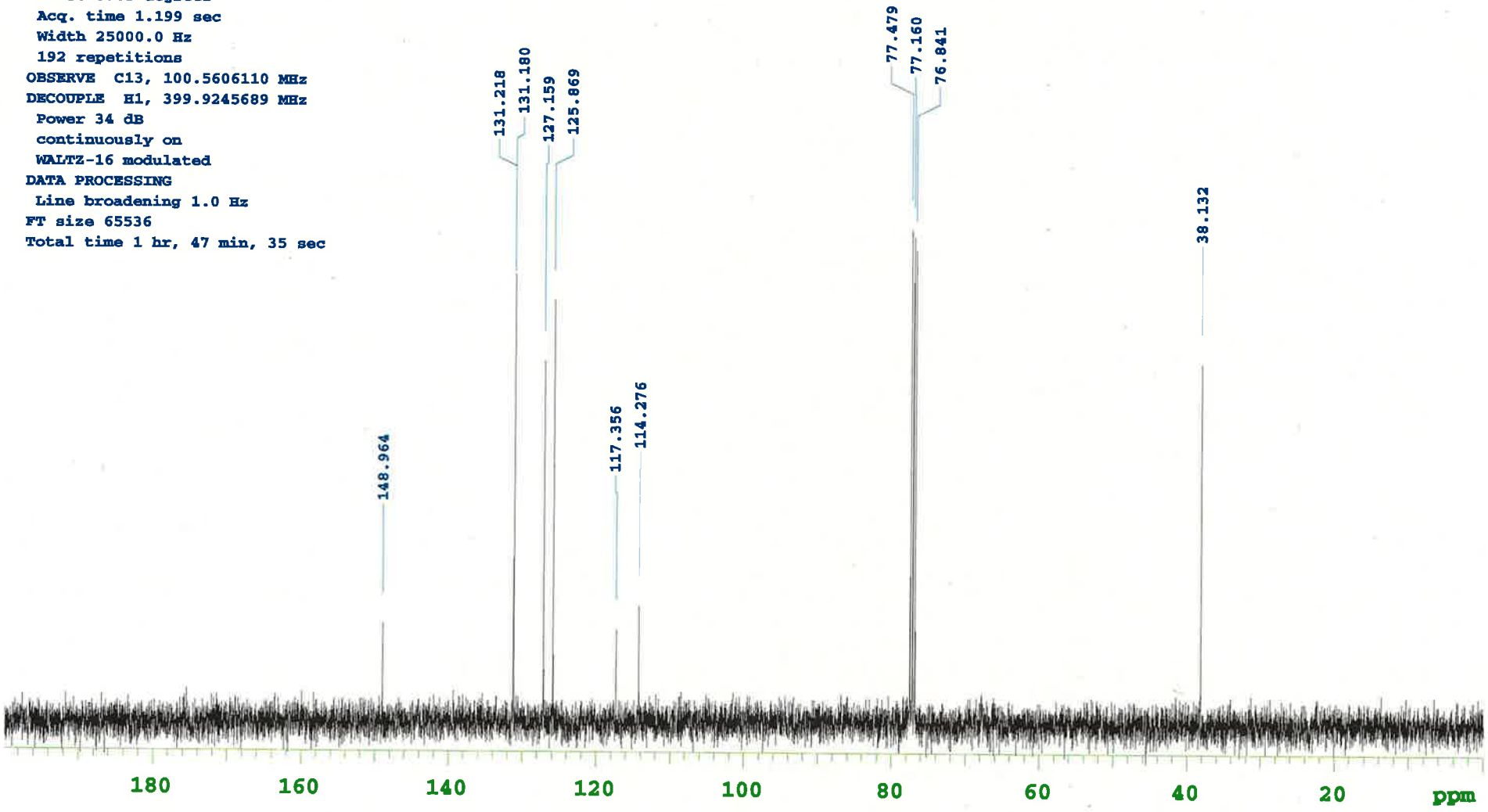
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



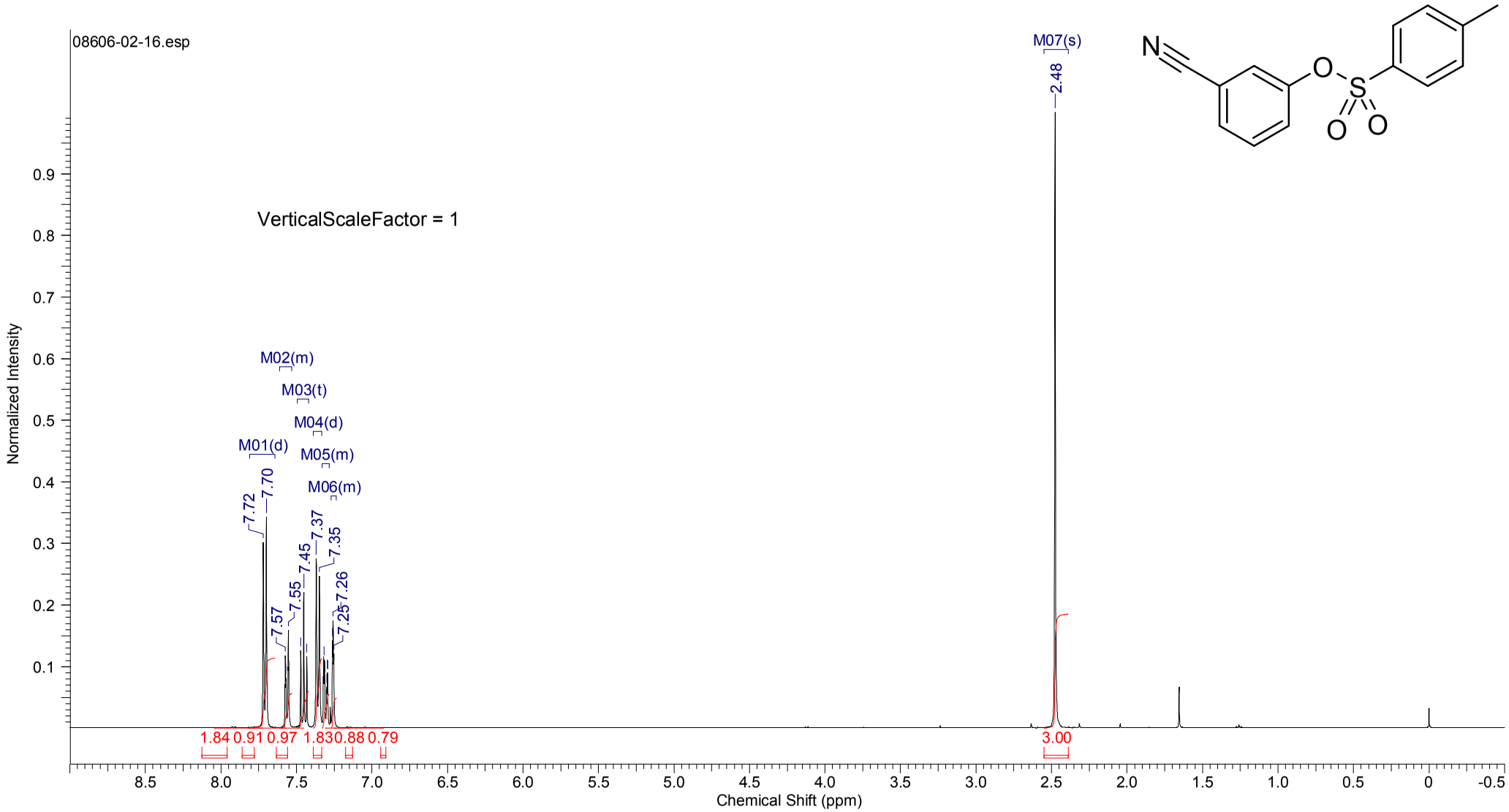


2012/05/11 14:37:59

Formula $C_{14}H_{11}NO_3S$	FW 273.3070

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	6.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2254.3765	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.71 (d, *J*=8.42 Hz, 2H), 7.53-7.61 (m, 1H), 7.45 (t, *J*=7.87 Hz, 1H), 7.36 (d, *J*=8.42 Hz, 2H), 7.28-7.33 (m, 1H), 7.24-7.27 (m, 1H), 2.48 (s, 3H)

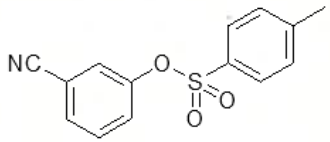


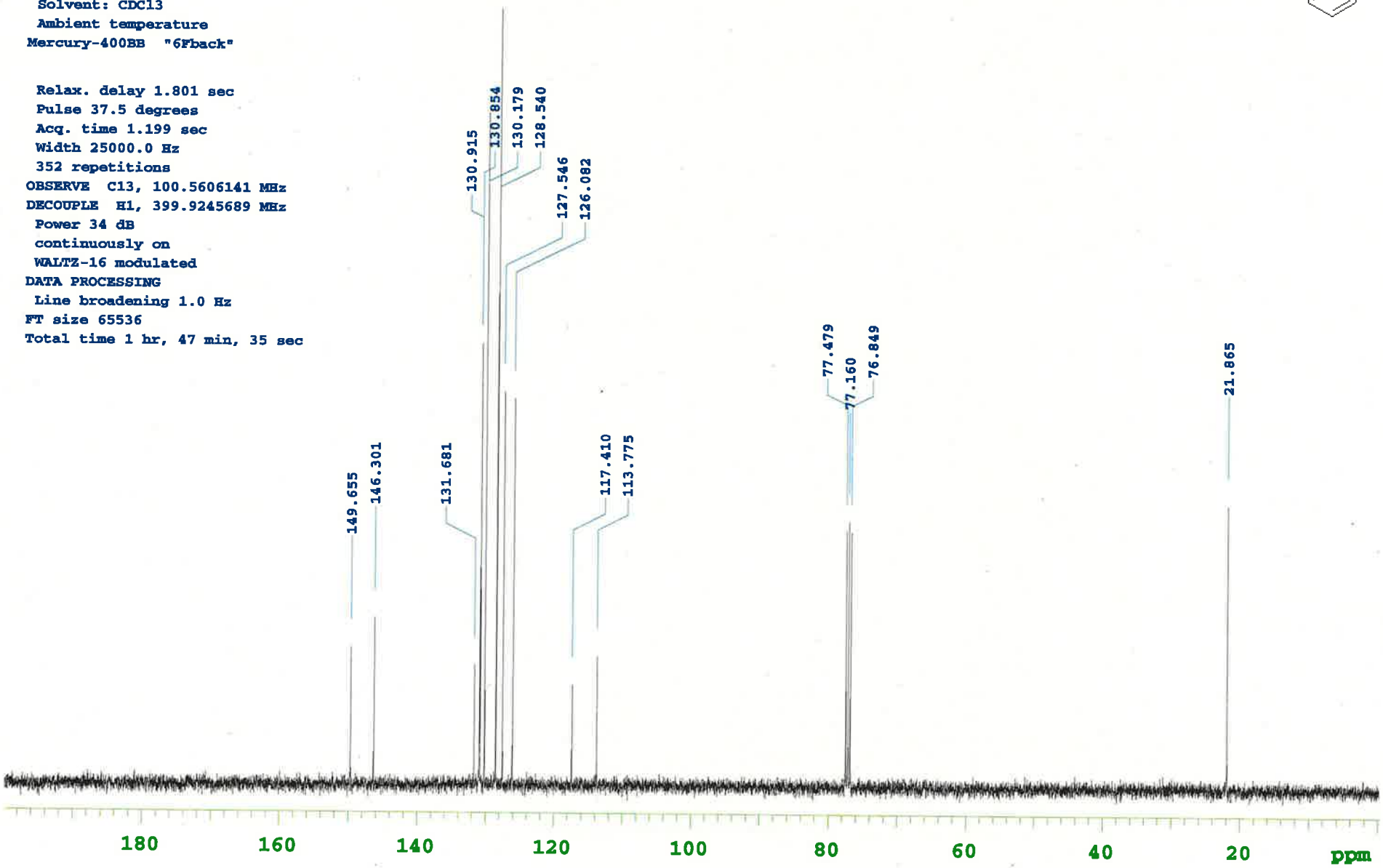
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 100.5606141 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



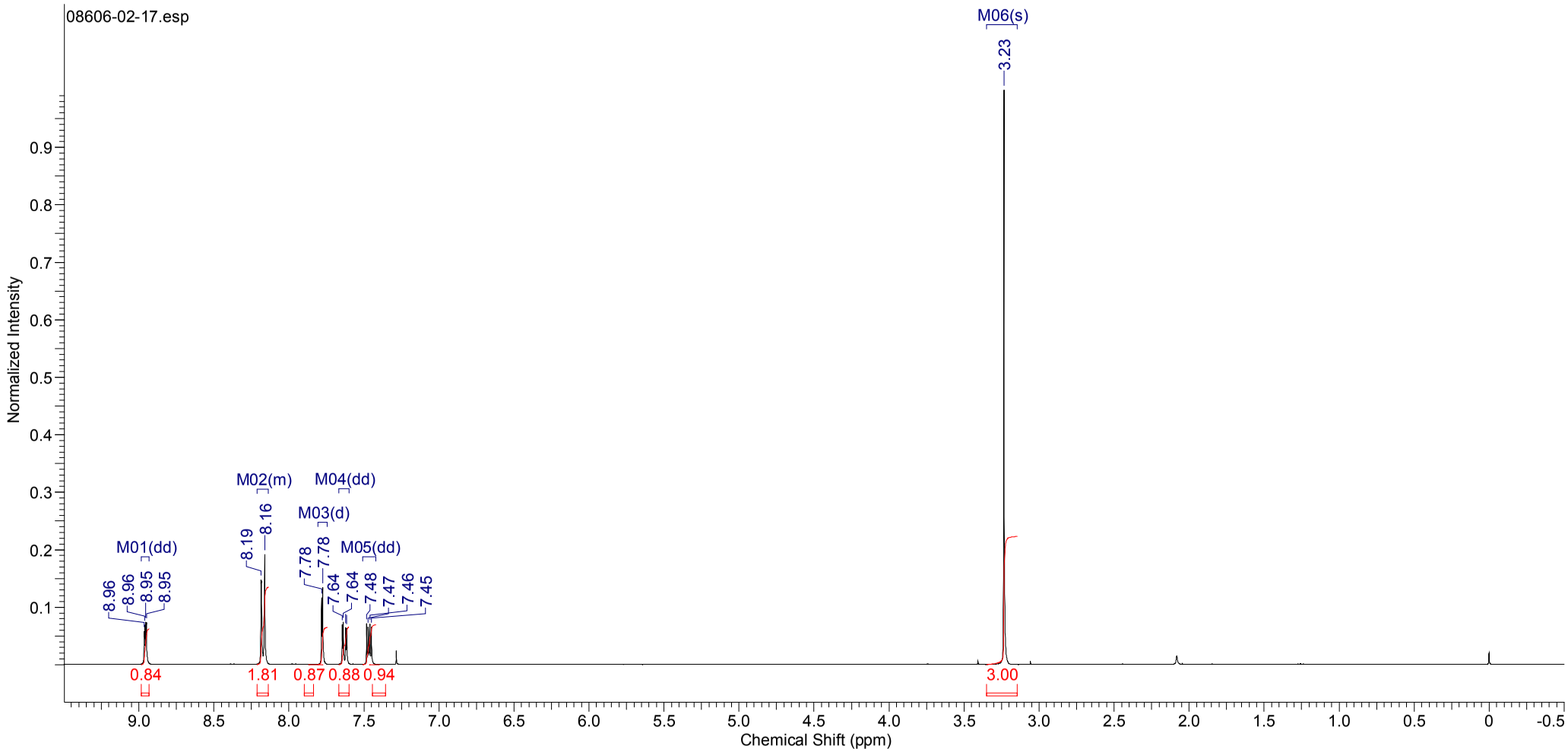


2012/04/11 11:33:51

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2257.6699	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.96 (dd, *J*=1.65, 4.21 Hz, 1H), 8.14-8.21 (m, 2H), 7.78 (d, *J*=2.56 Hz, 1H), 7.63 (dd, *J*=2.56, 9.15 Hz, 1H), 7.47 (dd, *J*=4.21, 8.23 Hz, 1H), 3.23 (s, 3H)

VerticalScaleFactor = 1

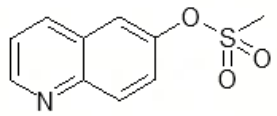


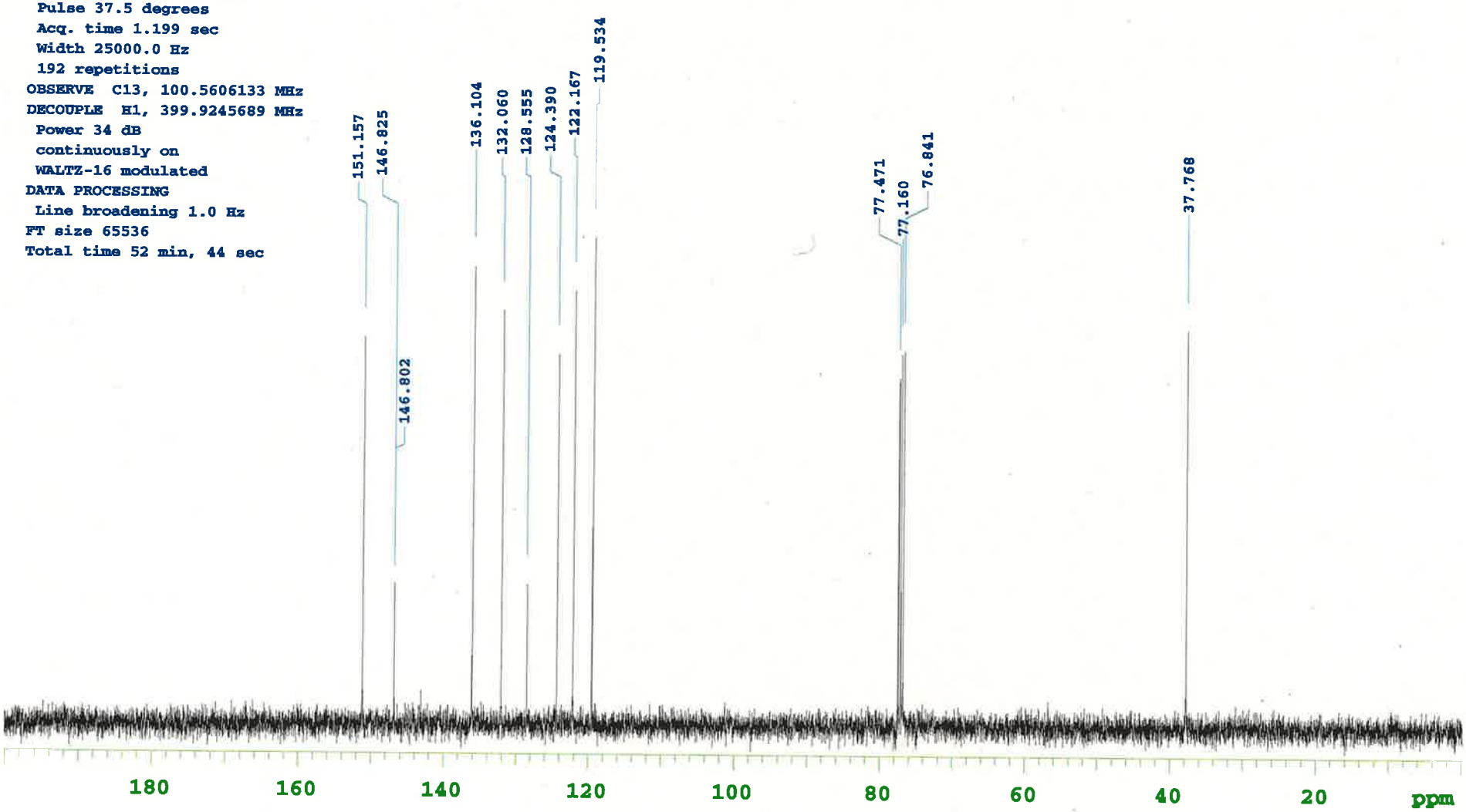
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
192 repetitions
OBSERVE C13, 100.5606133 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 52 min, 44 sec

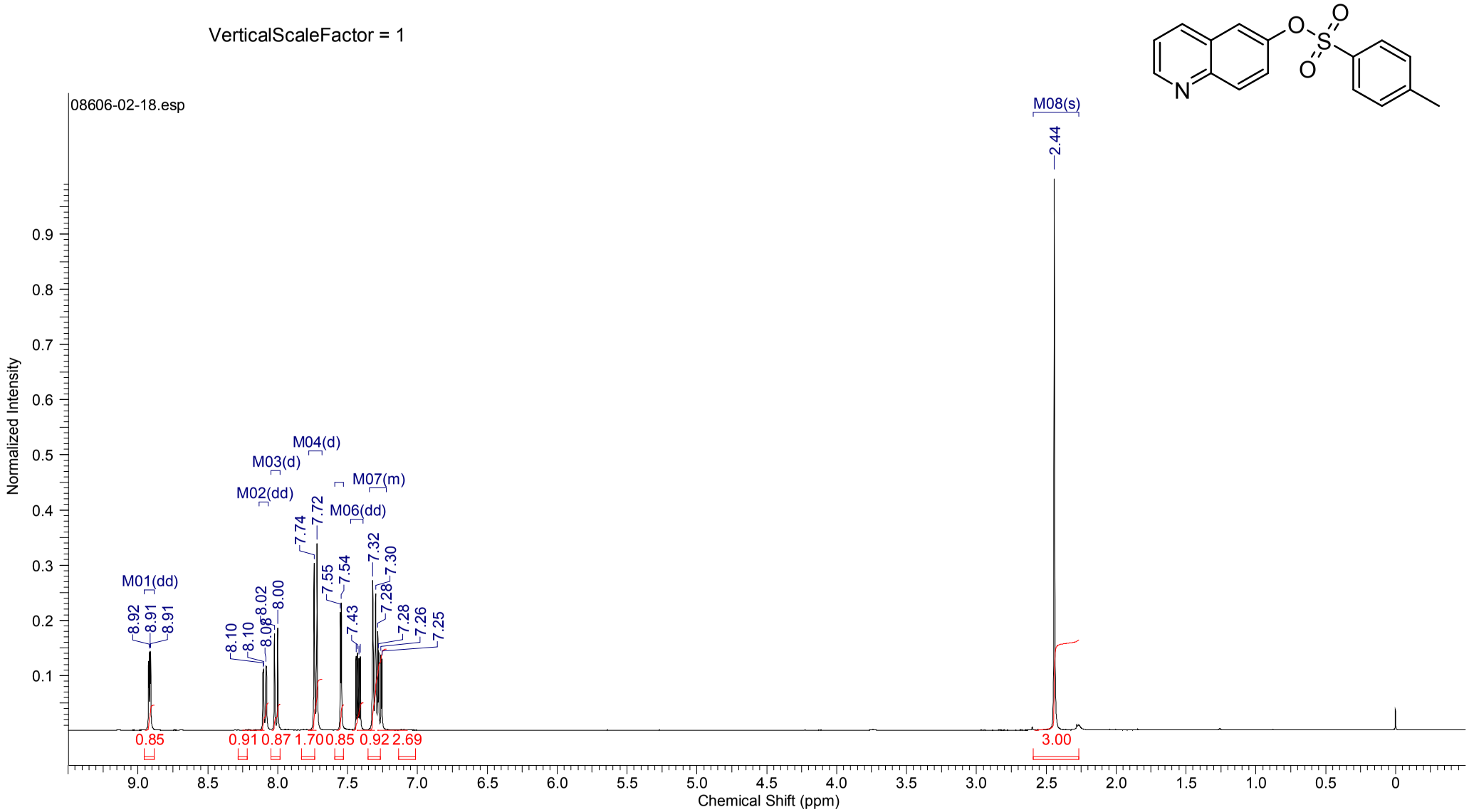




2012/04/11 11:37:46

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	6.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2257.3042	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 8.92 (dd, *J*=1.65, 4.21 Hz, 1H), 8.09 (dd, *J*=1.10, 8.42 Hz, 1H), 8.01 (d, *J*=9.15 Hz, 1H), 7.73 (d, *J*=8.42 Hz, 2H), 7.55 (d, *J*=2.56 Hz, 1H), 7.42 (dd, *J*=4.21, 8.23 Hz, 1H), 7.22-7.34 (m, 3H), 2.44 (s, 3H)

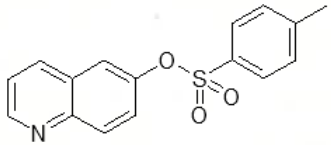


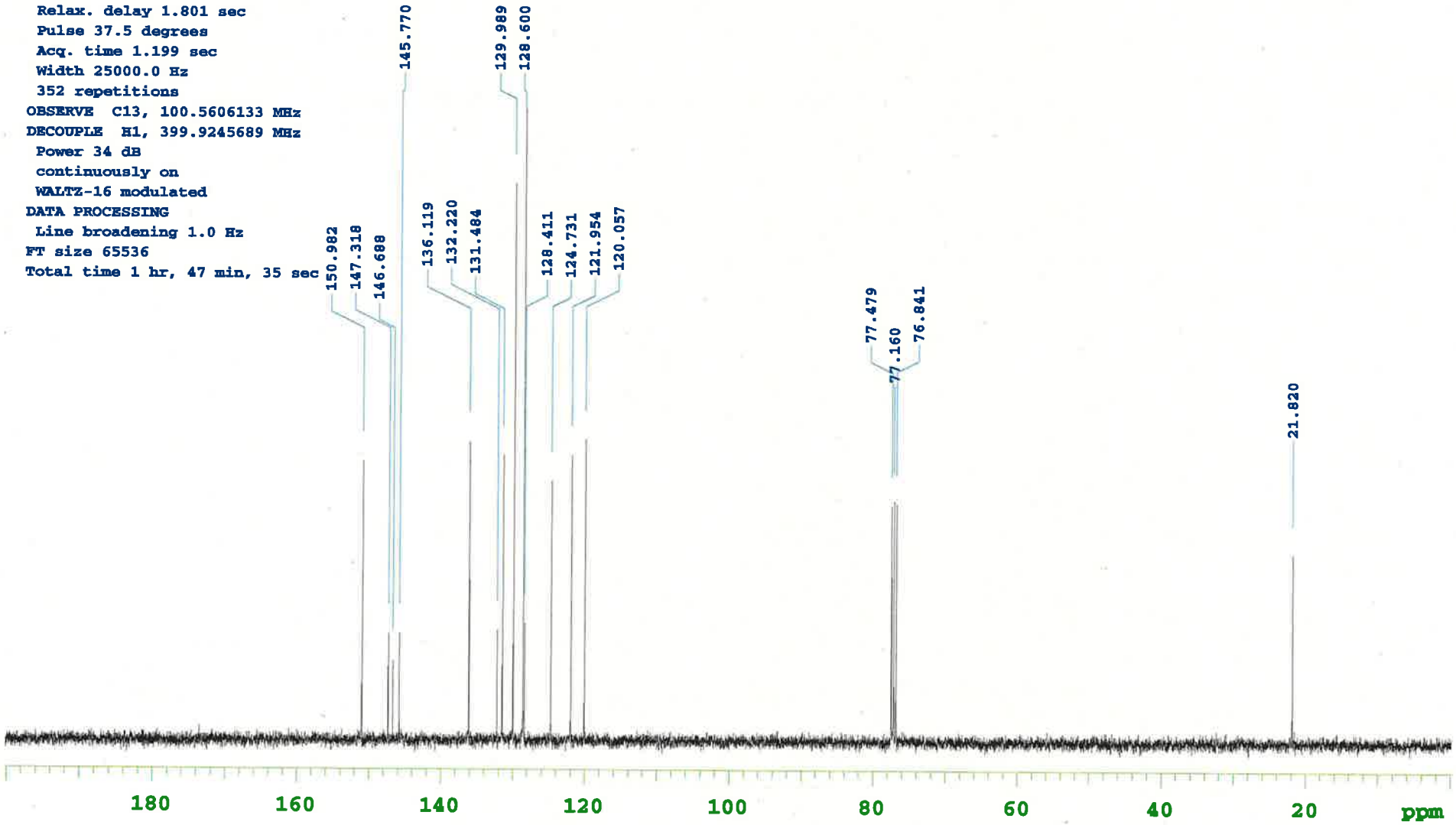
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
352 repetitions
OBSERVE C13, 100.5606133 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



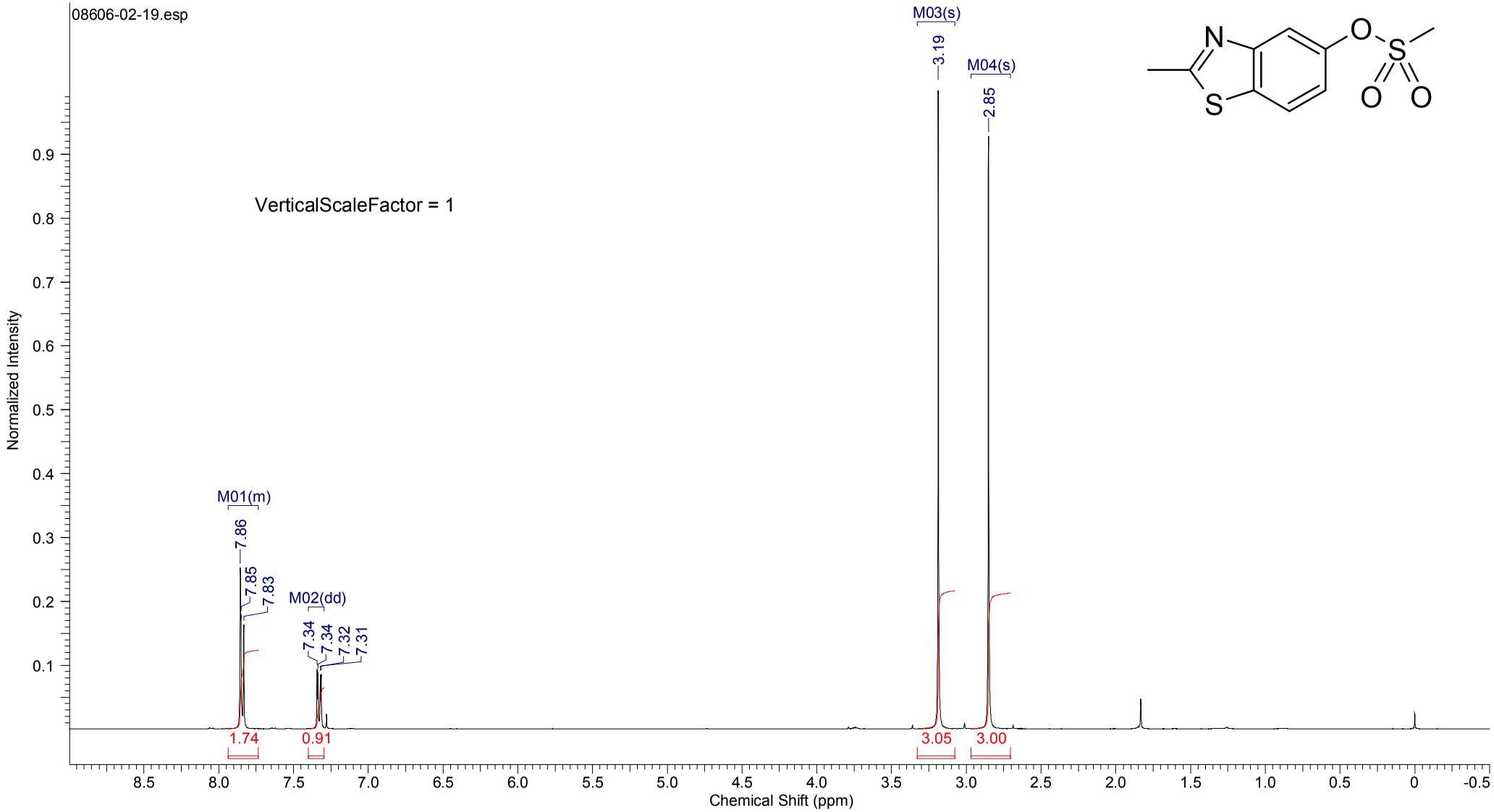


2012/05/11 14:29:46

Formula $C_9H_9NO_3S_2$	FW 243.3027

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	10.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2255.8403	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.74-7.94 (m, 2H), 7.33 (dd, *J*=2.20, 8.78 Hz, 1H), 3.19 (s, 3H), 2.85 (s, 3H)

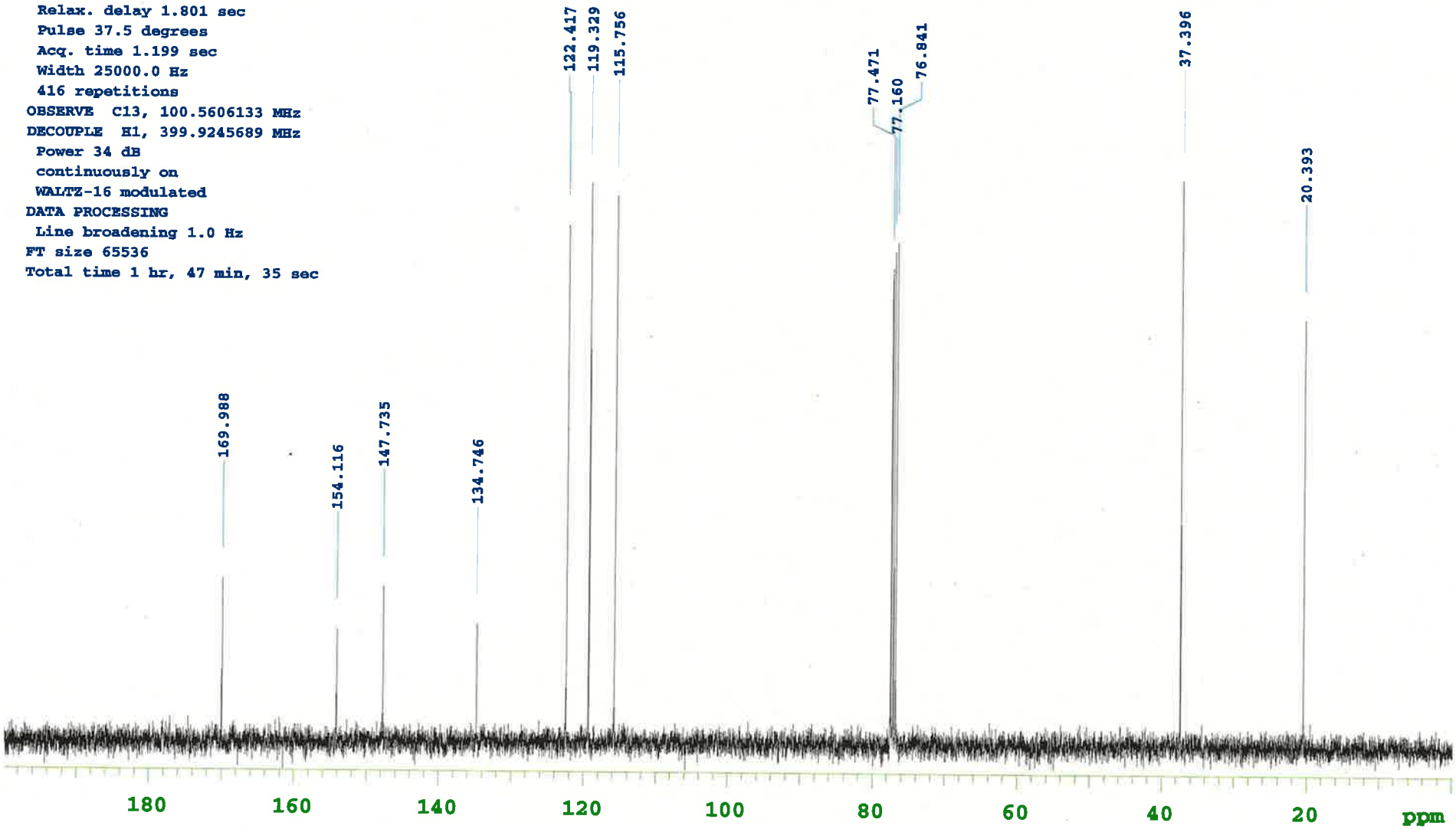


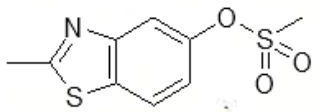
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
416 repetitions
OBSERVE C13, 100.5606133 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



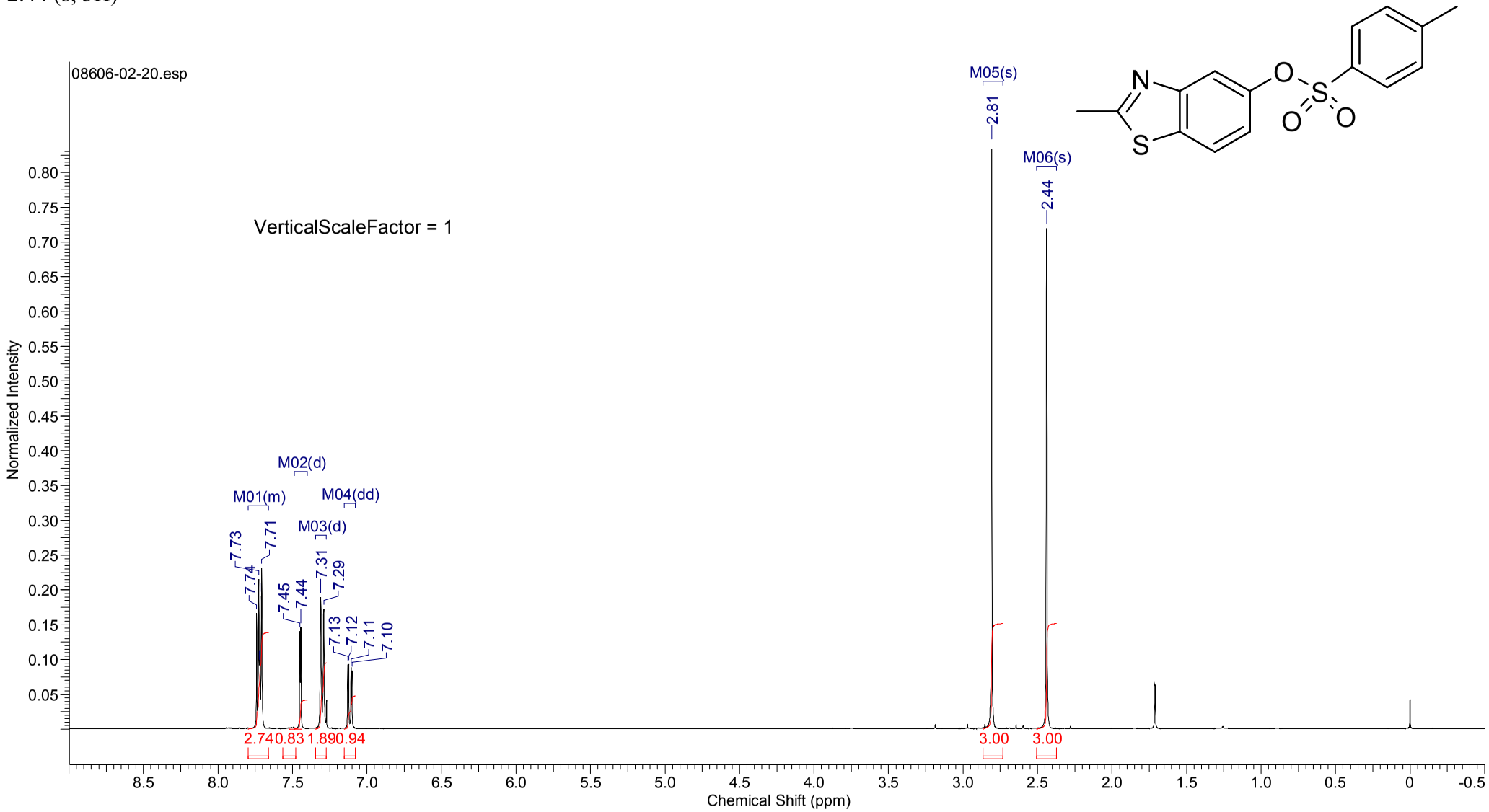


2012/05/11 14:14:31

Formula $C_{15}H_{13}NO_3S_2$	FW 319.3986

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Feb 25 2012	**Date Stamp**	Feb 25 2012
File Name	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.92	**Nucleus**	1H	**Number of Transients**	32
Original Points Count	16379	**Points Count**	16384	**Pulse Sequence**	s2pul	**Receiver Gain**	12.00	**Solvent**	CHLOROFORM-d
Spectrum Offset (Hz)	2252.9128	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20	**Temperature (degree C)**	AMBIENT TEMPERATURE		

^{1}H NMR (400 MHz, CHLOROFORM-d) δ 7.66-7.80 (m, 3H), 7.45 (d, *J*=2.56 Hz, 1H), 7.30 (d, *J*=8.42 Hz, 2H), 7.11 (dd, *J*=2.38, 8.60 Hz, 1H), 2.81 (s, 3H), 2.44 (s, 3H)

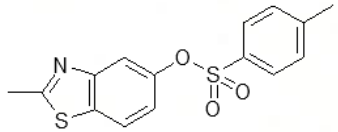


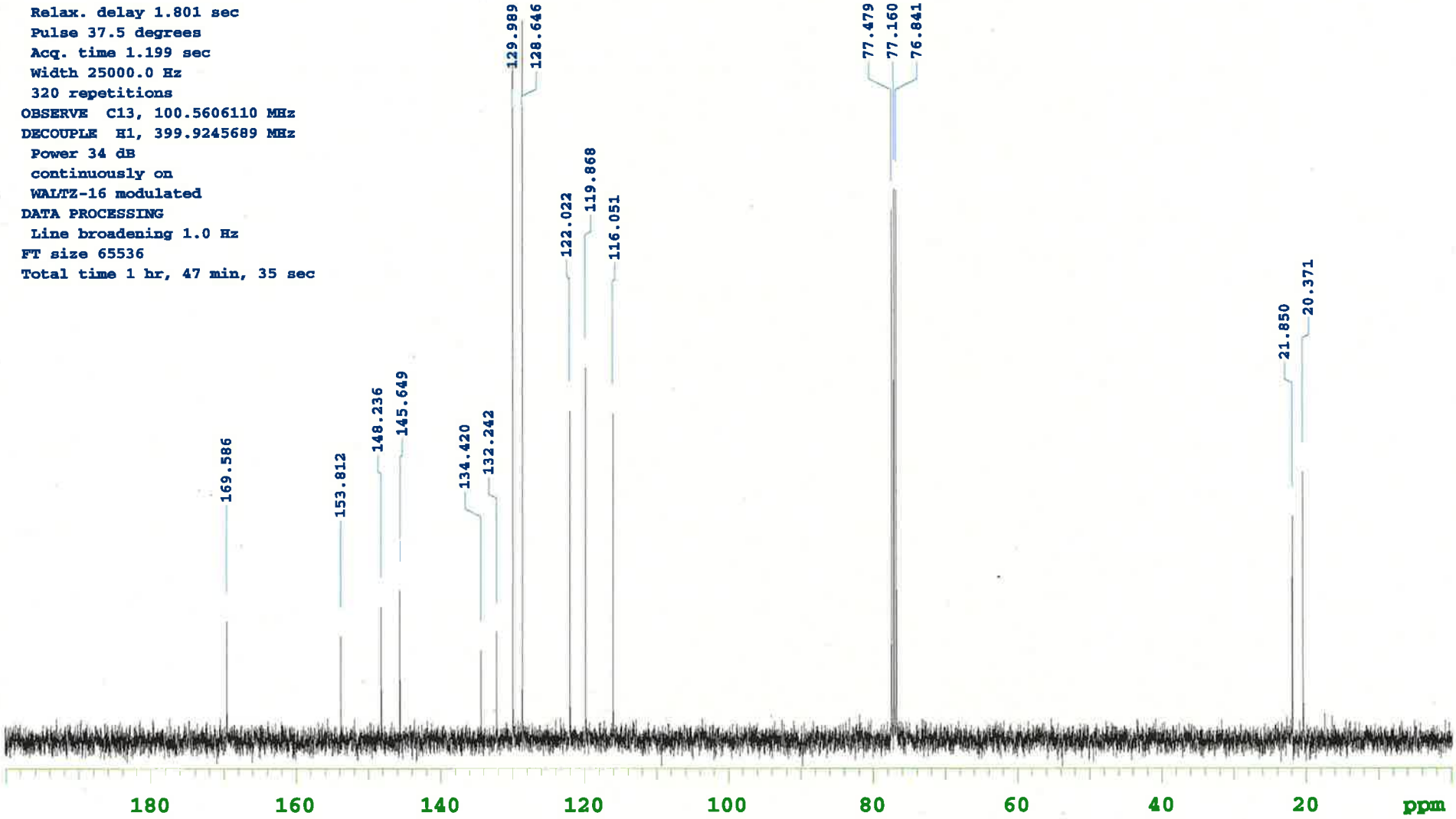
13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
320 repetitions
OBSERVE C13, 100.5606110 MHz
DECOUPLE H1, 399.9245689 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 47 min, 35 sec



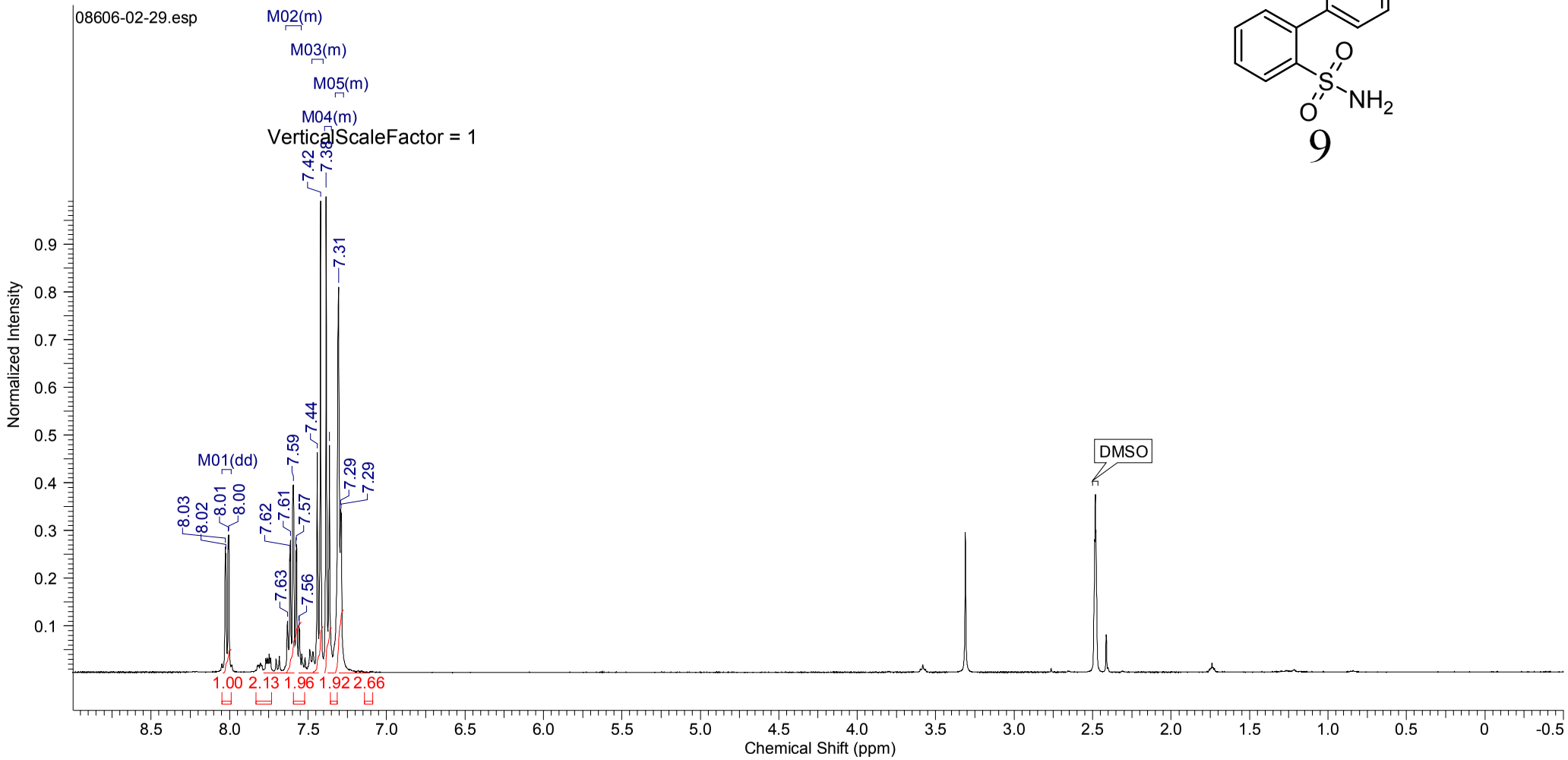


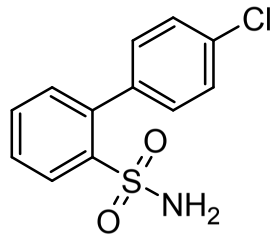
2012/04/11 13:44:03

Formula $C_{12}H_{10}ClNO_2S$		**FW**	267.7313

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012		
Date Stamp	Mar 20 2012			**File Name**	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.93
Nucleus	1H	**Number of Transients**	32	**Original Points Count**	16379	**Points Count**	16384	**Pulse Sequence**	s2pul
Receiver Gain	20.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20
Temperature (degree C)	AMBIENT TEMPERATURE								

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.02 (dd, *J*=1.46, 7.68 Hz, 1H), 7.54-7.64 (m, 2H), 7.40-7.47 (m, 2H), 7.35-7.40 (m, 2H), 7.28-7.33 (m, 3H)





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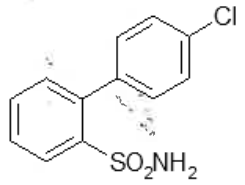
13C OBSERVE

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
448 repetitions
OBSERVE C13, 100.5611445 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 3 hr, 43 min, 2 sec



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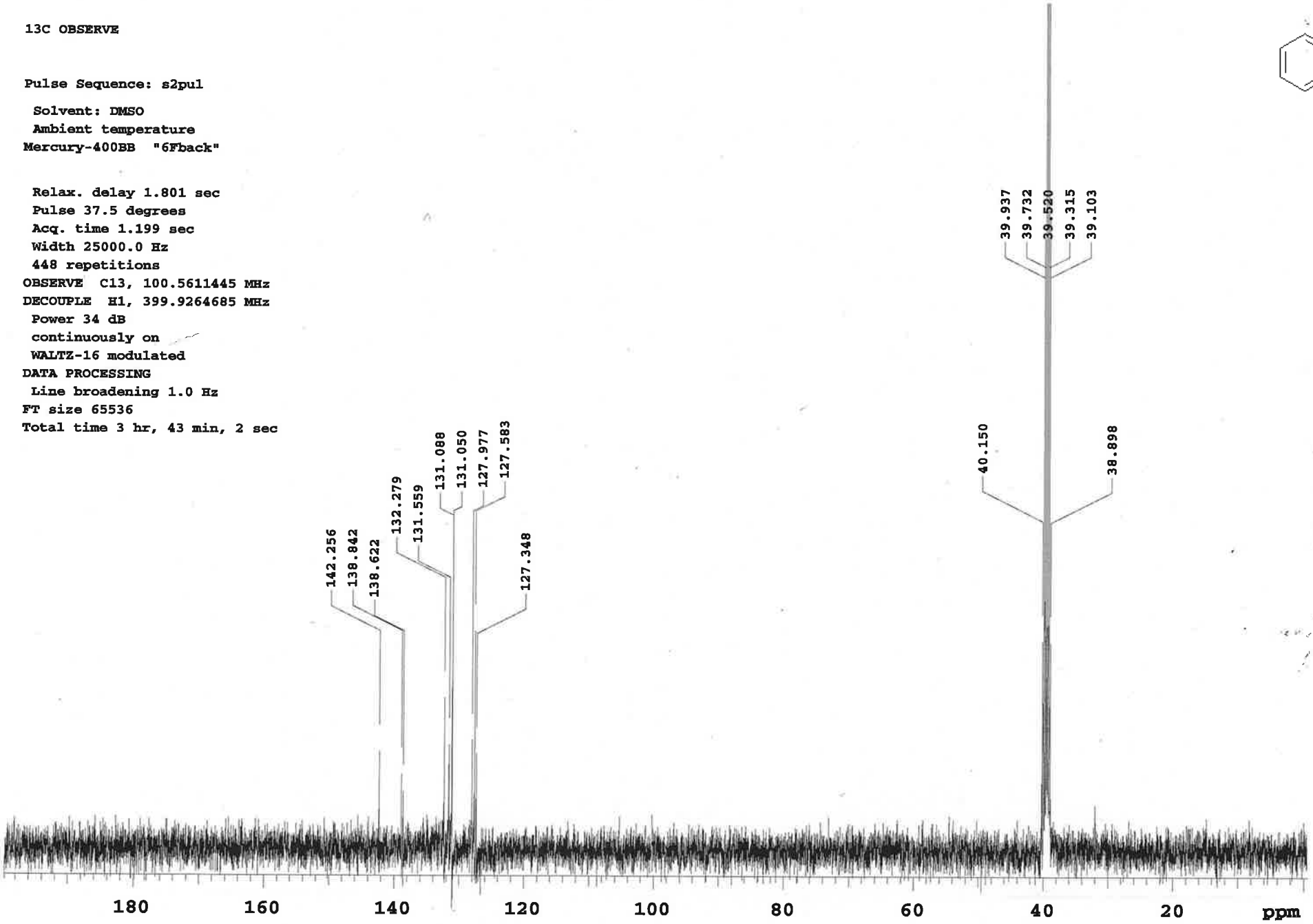
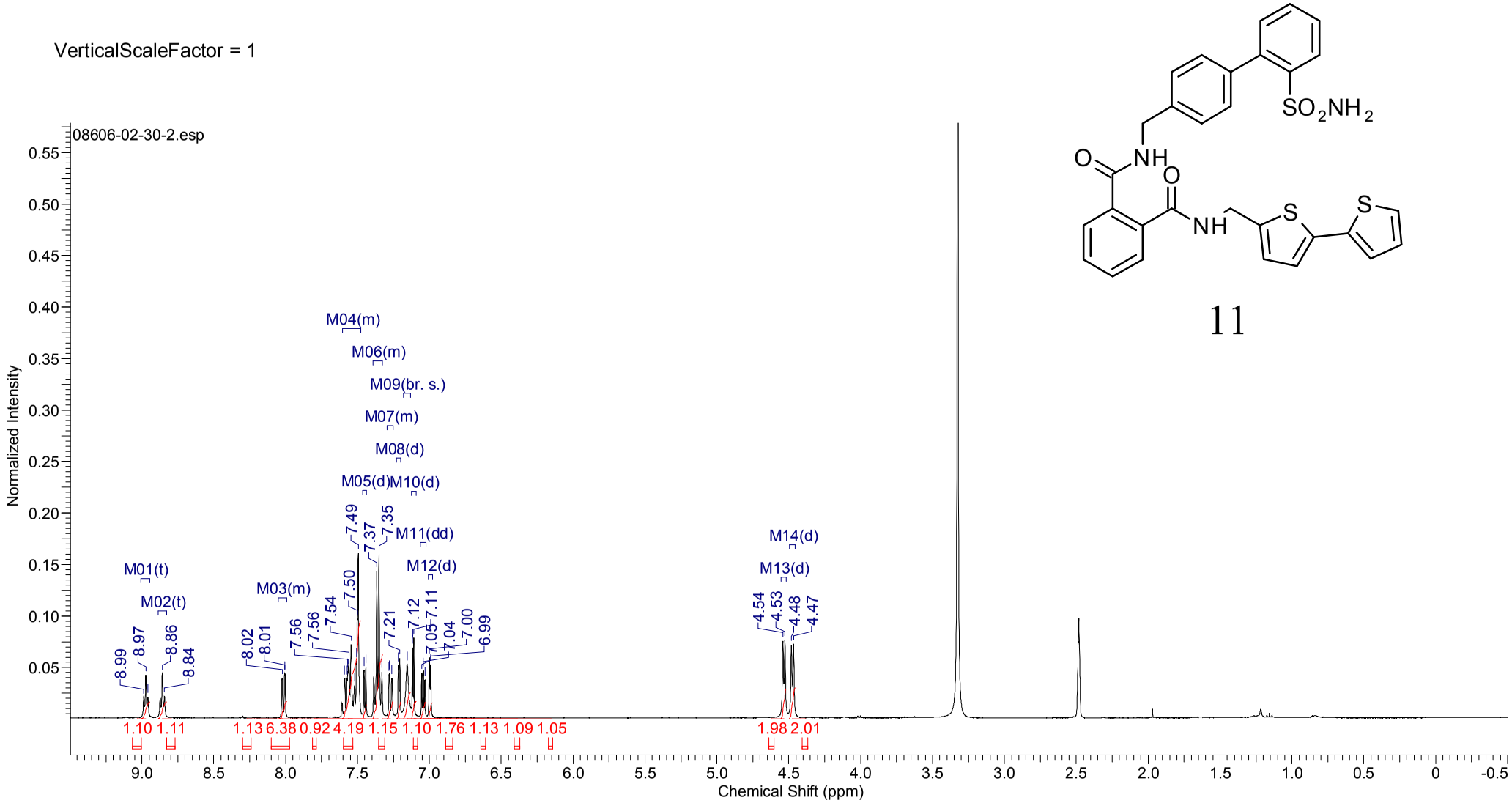


2012/04/11 13:53:51

Acquisition Time (sec)	2.7320	**Comment**	STANDARD 1H OBSERVE			**Date**	Mar 20 2012		
Date Stamp	Mar 20 2012			**File Name**	C:¥USR¥NMR¥FID			**Frequency (MHz)**	399.93
Nucleus	1H	**Number of Transients**	32	**Original Points Count**	16379	**Points Count**	16384	**Pulse Sequence**	s2pul
Receiver Gain	20.00	**Solvent**	DMSO-d6	**Spectrum Offset (Hz)**	2247.6611	**Spectrum Type**	STANDARD	**Sweep Width (Hz)**	5995.20
Temperature (degree C)	AMBIENT TEMPERATURE								

^{1}H NMR (400 MHz, DMSO-d_6) δ 8.97 (t, *J*=5.86 Hz, 1H), 8.86 (t, *J*=5.86 Hz, 1H), 7.99-8.05 (m, 1H), 7.48-7.60 (m, 6H), 7.45 (d, *J*=5.12 Hz, 1H), 7.33-7.39 (m, 4H), 7.25-7.30 (m, 1H), 7.21 (d, *J*=3.66 Hz, 1H), 7.16 (br. s., 2H), 7.11 (d, *J*=3.66 Hz, 1H), 7.04 (dd, *J*=3.84, 4.94 Hz, 1H), 7.00 (d, *J*=3.66 Hz, 1H), 4.53 (d, *J*=5.49 Hz, 2H), 4.47 (d, *J*=6.22 Hz, 2H)



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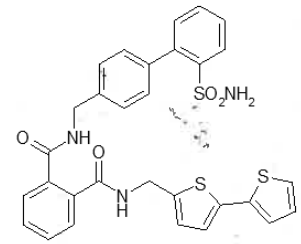
13C OBSERVE

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 08606-02-30
Mercury-400BB "6Fback"

Relax. delay 1.801 sec
Pulse 37.5 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
2512 repetitions
OBSERVE C13, 100.5611452 MHz
DECOUPLE H1, 399.9264685 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening
FT size 65536
Total time 7 hr, 26 min, 4 sec



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