Base-Mediated Intermolecular sp^2 C-H Bond Arylation via Benzyne Intermediates

SUPPORTING INFORMATION

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Experimental Section

General considerations: Reactions were performed in 2-dram vials using screw caps with 17 mm hole and white silicone septum with white teflon face (from SUPELCO). Column chromatography was performed on 60Å silica gel (Sorbent Technologies). Purification by preparative HPLC was performed on a Shimadzu Prominence LC (LC-20AB) equipped with a SPD-20A UV-Vis detector and a Varian Dynamax (250 mm x 21.4 mm) column. GC-MS analyses were performed on a Shimadzu GCMS-QP5000 chromatograph equipped with a Restek column (Rtx-XLB, 30 m x 0.25 mm I.D.). The ^{1}H, ^{19}F and ^{13}C NMR were recorded on JEOL EC-400 or JEOL EC-500 spectrometers using residual solvent peak as a reference. α,α,α-Trifluorotoluene (neat, δ = -62.3ppm) was employed as an external standard in ^{19}F NMR spectra. Elemental analyses were performed by Atlantic Microlab Inc. of Norcross, GA. IR spectra were obtained on a ThermoNicolet Avatar 370 FT-IR instrument. Analytical thin layer chromatography was performed on silica gel IB-F (Baker-flex) by J. T. Baker. All procedures were performed under argon atmosphere unless otherwise noted.

Materials. The following starting materials were obtained from commercial sources and were used without further purification: benzothiophene, benzothiazole, 1-methylindole, 1-phenylpyrrole, 3-methoxypyrazine, 3-methoxypyridine, 2-chloroanisole, 2-chlorobenzotrifluoride, 3-fluorobenzoic acid, *N*-methylbenzimidazole, 2-*n*-butylthiophene, 2-*n*-butylfuran, 1,3-dimethoxybenzene, N-methylimidazole, 3-trifluoromethylanisole, 3-chloro-*N*,*N*-dimethylaniline, 2-chloro-*N*,*N*-dimethylaniline, 2,2,6,6-tetramethylpiperidine (TMPH), 9-chlorophenanthrene, 3,5-dimethoxychlorobenzene, 3-chloro-4-methoxytoluene, 4-*tert*-butylchlorobenzene, 2,3-benzo[b]furan, and *tert*-butyl-3-bromo-benzoate. *tert*-Butyl-3-fluorobenzoate was synthesized from 3-fluorobenzoic acid.[1]

TMPLi, Cy_2NLi, LDA 1M in THF: A 25 mL oven-fried flask equipped with a magnetic stirring bar and a septum was evacuated and backfilled with argon 5 times. TMPH (2,2,6,6-tetramethylpiperidine, 2.32 g, 16.5 mmol) or Cy_2NH (dicyclohexylamine, 2.98 g, 16.5 mmol) or diisopropylamine (1.67 g, 16.5 mmol) was added, followed by anhydrous THF to give 15 mL of solution. The mixture was cooled to -73 ^{o}C and stirred for 5 minutes. *n*-BuLi (2.5 M in hexanes, 6 mL, 15 mmol) was then added dropwise. The reaction mixture was stirred for 30 minutes at -73 ^{o}C followed by warming to 0^{o}C and stirring for additional 30 minutes. After that, the reaction mixture was vacuumed to give 15 mL of solution.

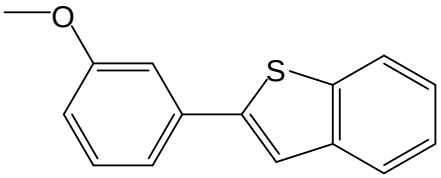
TMPLi 1 M in pentane: A 50 mL oven-fried flask equipped with magnetic stirring bar and a septum was evacuated and backfilled with argon 5 times. TMPH (2,2,6,6-tetramethylpiperidine, 4.64 g, 33.0 mmol) was added, followed by anhydrous pentane to give 30 mL of solution (marked the flask at the desired level). The mixture was cooled to -73 ^{o}C and stirred for 5 minutes. *n*-BuLi (2.5 M in hexanes, 12 mL, 30 mmol) was added dropwise and reaction mixture was stirred for 30 minutes at -73 ^{o}C, warmed up to room temperature and stirred overnight. The reaction mixture was vacuumed to give 30 mL (at the marked level) of TMPLi suspension.

The following cooling baths were used to attain the desired temperatures. Temperature control is very important to obtain reproducible yields. Temperature was monitored continuously and typically remained within 2 ^{o}C of the reported value.

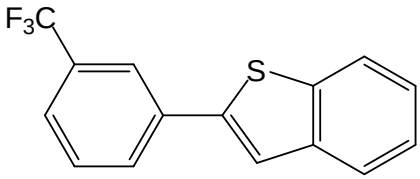
Diethylene glycol + CO_2 : -13 ^{o}C
Acetonitrile + CO_2 : -46 ^{o}C
Chloroform + CO_2 : -63 ^{o}C
Acetone + CO_2 : -73 ^{o}C
m-Toluidine + CO_2 : -35 ^{o}C

General procedure:

A 2 dram vial equipped with a magnetic stir bar was charged with arene or heterocycle (0.5 mmol) and ArCl (1.3 – 2.5 equiv). The vial was flushed with argon and capped. To this mixture was added the appropriate base solution or suspension (2.5-4.5 equiv) at the specified reaction temperature by injecting through the septum via syringe. If TMPLi in pentane was used, the base suspension was stirred vigorously while being withdrawn by syringe. The vial was flushed with argon (20 seconds) and then stirred at specified temperature for indicated time. Unless otherwise stated, reaction mixture was quenched with anhydrous MeOH (1.0 mL), evacuated to 1.0 mL and subjected to column chromatography on silica gel in hexane followed by appropriate solvent to elute the products. After concentrating the fractions containing the product, the residue was dried under reduced pressure.

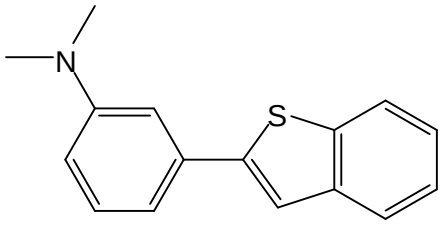


2-(3-Methoxyphenyl)benzothiophene (Entry 1, Table 1): 2-Chloroanisole (192 mg, 1.35 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2.1 mL), rt, 1h. After column chromatography (hexanes/CH_2Cl_2 65/35), 94 mg (78 %) of white solid was obtained. R_f = 0.46 (hexanes/ CH_2Cl_2 60/40). This compound is known.[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.84 (m, 1H), 7.80-7.78 (m, 1H), 7.60 (s, 1H), 7.39-7.31 (m, 4H), 7.29-7.27 (m, 1H), 6.91 (td, *J* = 7.0 Hz, 2.3 Hz, 1H), 3.88 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 160.1, 144.2, 140.7, 139.6, 135.7, 130.1, 124.7, 124.5, 123.7, 122.4, 119.8, 119.2, 113.9, 112.2, 55.5.



2-(3-Trifluoromethylphenyl)benzothiophene (Entry 2, Table 1): 2-Chlorobenzotrifluoride (180 mg, 1 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1.8 mL, 1.8 mmol), 0 ^{o}C, 4 hrs. After column chromatography (hexanes/CH_2Cl_2 80/20), 84 mg (61 %) of light yellow solid was obtained. R_f = 0.57 (hexanes/CH_2Cl_2 80/20), mp 108.5-109.5 ^{o}C (from hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.87-7.83 (m, 2H), 7.80 (m, 1H), 7.61-7.51 (m, 3H), 7.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 142.4, 140.5, 139.7, 135.2, 131.5 (q, J_{C-F} = 32 Hz), 129.7, 129.6, 125.0, 124.9, 124.8 (q, J_{C-F} = 3.7 Hz), 124.1 (q, J_{C-F} = 272 Hz), 124.0, 123.2 (q, J_{C-F} = 3.7 Hz), 122.5, 120.7. FT-IR (neat, cm^{-1}) υ 1326. 1297, 1179, 800, 754. Anal calcd for $C_{15}H_9F_3S$ (278.04g/mol): C, 64.74; H, 3.26; Found. C, 64.69; H, 3.23.

Note: Less than 5 % of isomeric 2-(2-trifluoromethylphenyl)benzothiophene was observed by GC.



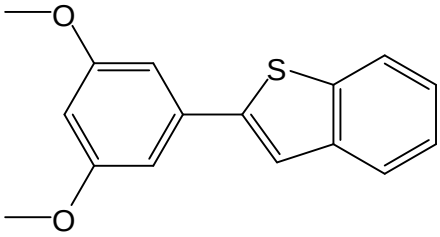
3-Benzothien-2-yl-N,N-dimethylbenzenamine (Entries 3 and 4, Table 1):

2-Chloro-*N*,*N*-dimethylaniline (195 mg, 1.25 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), rt, 2 hrs. After column chromatography (hexanes/CH_2Cl_2 20/80), 97 mg (77 %) of a light brown solid was obtained.

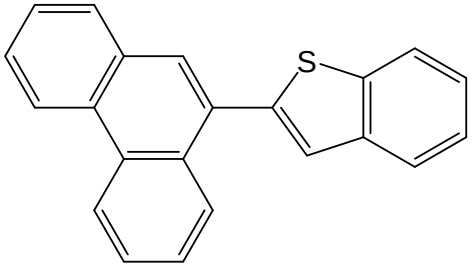
3-Chloro-*N*,*N*-dimethylaniline (195 mg, 1.25 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), rt, 2 hrs. After column chromatography (hexanes/CH_2Cl_2 20/80), 102 mg (81 %) of a light brown solid was obtained.

R_f = 0.58 (hexanes/EtOAc 70/30), mp 77-77.5 ^oC (from hexanes). ^1H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* =8.0 Hz, 1H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.57 (s, 1H), 7.39-7.31 (m, 3H), 7.12 (d, *J* = 8.1 Hz, 1H), 7.08 (t, *J* = 2.2 Hz, 1H), 6.76 (dd, *J* = 8.1 Hz, 2.2 Hz, 1H), 3.04 (s, 6H) . ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 151.0, 145.5, 140.8, 139.6, 135.1, 129.7, 124.5, 124.2, 123.6, 122.4, 119.4, 115.2, 112.7, 110.6, 40.7. FT-IR (neat, cm^{-1}) υ 1698, 1500, 1439, 823, 760, 754, 725, 680. Anal calcd for $C_{16}H_{15}NS$ (253.09g/mol): C, 75.85; H, 5.97, N, 5.53; Found. C, 75.81; H, 5.90; N, 5.35.

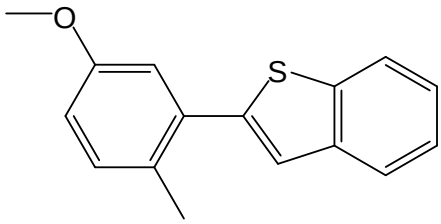
Note: Less than 3% of 2-benzothien-2-yl-N,N-dimethylbenzenamine was detected by GC.



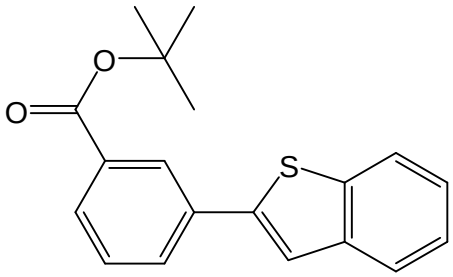
2-(3,5-Dimethoxyphenyl)benzothiophene (Entry 5, Table 1): 3,5-Dimethoxy-1-chlorobenzene (216 mg, 1.25 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), -73 ^oC, 12 hrs. After column chromatography (hexanes/CH_2Cl_2 40/60), 84 mg (62 %) of a light yellow solid was obtained. R_f = 0.39 (hexanes/CH_2Cl_2 50/50). This compound is known.[2] ^1H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 7.9 Hz, 1.0 Hz, 1H), 7.78 (dd, *J* = 7.9 Hz, 1.0 Hz, 1H), 7.54 (s, 1H), 7.38-7.30 (m, 2H), 6.88 (d, *J* = 2.3 Hz, 2H), 6.48 (t, *J* = 2.3 Hz, 1H), 3.86 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 161.2, 144.2, 140.6, 139.5, 136.2, 124.6, 124.5, 123.7, 122.4, 120.0, 104.9, 100.4, 55.6.



2-(9-Phenanthrenyl)benzothiophene (Entry 6, Table 1): 9-Chlorophenanthrene (265 mg, 1.25 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), 0 ^{o}C, 3 hrs followed by 1 h at rt. After column chromatography (hexanes/CH_2Cl_2 90/10), 116 mg (75 %) of a light yellow solid was obtained. R_f = 0.25 (hexanes/CH_2Cl_2 90/10), mp 146.5-148 ^{o}C (from hexanes).This compound is unknown. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.8 (d, *J* = 8.3 Hz, 1H), 8.72 (d, *J* = 8.3 Hz, 1H), 8.33 (dd, *J* = 8.2 Hz, 1.0 Hz, 1H), 7.95 (s, 1H), 7.94-7.87 (m, 3H), 7.73-7.60 (m, 4H), 7.51 (s, 1H), 7.46-7.38 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 142.2, 140.4, 140.3, 131.2, 131.1, 130.9, 130.7, 130.4, 129.6, 129.0, 127.4, 127.1, 127.0, 126.9, 126.7, 124.6, 124.5, 124.4, 123.7, 123.1, 122.7, 122.3. FT-IR (neat, cm^{-1}) υ 1705, 1298, 1158. Anal calcd for $C_{22}H_{14}S$ (310.08 g/mol): C, 85.12; H, 4.55; Found. C, 84.98; H, 4.48.



2-(5-Methoxy-2-methylphenyl)benzothiophene (Entry 7, Table 1): 3-Chloro-4-methoxytoluene (195 mg, 1.25 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), rt, 4 hrs. After column chromatography (hexanes/CH_2Cl_2 70/30), 92 mg (73 %) of a light yellow solid was obtained. R_f = 0.43 (hexanes/CH_2Cl_2 65/35), mp 57.0-58 ^{o}C (from ether). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.40-7.32 (m, 2H), 7.27 (s, 1H), 7.23 (d, *J* = 8.5 Hz, 1H), 7.04 (d, *J* = 2.8 Hz, 1H), 6.87 (dd, *J* = 8.5 Hz, 2.8 Hz, 1H), 3.83 (s, 3H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.6, 143.5, 140.2, 140.1, 135.1, 131.8, 128.5, 124.5, 124.2, 123.6, 123.1, 122.1, 115.9, 114.2, 55.5, 20.3. FT-IR (neat, cm^{-1}) υ 1605, 1500, 1250, 1032. Anal calcd for $C_{16}H_{14}OS$ (254.08g/mol): C, 75.55; H, 5.55; Found. C, 75.04; H, 5.48.



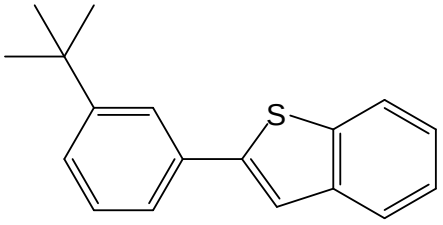
***tert*-Butyl-(3-benzothien-2-yl)benzoate(Entry 8, Table 1):**

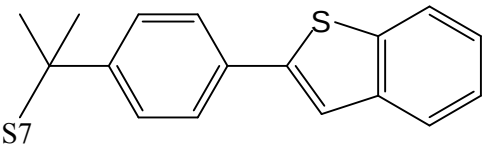
tert-Butyl-3-bromobenzoate (257 mg, 1 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), -73 ^{o}C while adding and then warm up to -46 ^{o}C by keeping vial inside the acetone-dry ice bath and not adding dry ice (2 hours); then move to acetonitrile-dry ice bath, and hold at -46 ^{o}C for 8 hrs; then move to ice bath to warm up to 0 ^{o}C and keep at that temperature for 30 minutes. After column chromatography (hexanes/Et_2O 90/10), 77 mg (50 %) of a yellow solid was obtained.

tert-Butyl-3-fluorobenzoate (192 mg, 1 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in THF (1 M, 2 mL), -73 ^{o}C; then slowly warm up to -35 ^{o}C (2 hrs) and keep at that temperature for 8 hrs, then move to ice bath to warm the reaction mixture to 0 ^{o}C and keep at that temperature for 30 minutes. After column chromatography (hexanes/Et_2O 90/10), 93 mg (60 %) of yellow solid was obtained.

R_f = 0.52 (hexanes/Et_2O 90/10), mp 135.8-137.1 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (t, *J* = 1.9 Hz, 1H), 7.96 (td, *J* = 7.8 Hz, 1.4 Hz, 1H), 7.86-7.82 (m, 2H), 7.79 (dd, *J* = 7.8 Hz, 1.9 Hz, 1H), 7.61 (s, 1H), 7.47 (t, *J* = 7.8 Hz, 1H), 7.38-7.31 (m, 2H), 1.64 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 165.5, 143.3, 140.7, 139.7, 134.5, 132.8, 130.3, 129.1, 129.0, 127.4, 124.8, 124.7, 123.8, 122.4, 120.2, 81.5, 28.3. FT-IR (neat, cm^{-1}) υ 1707, 1300, 1160. Anal calcd for $C_{19}H_{18}O_2S$ (310.10 g/mol): C, 73.52; H, 5.84; Found. C, 73.23; H, 5.72.

Note: The above two reactions were quenched with H_2O (15 mL), followed by addition of Et_2O (15 mL). The organic phase was then washed with brine (15 mL) and the aqueous layers were extracted with Et_2O (2x10 mL). The combined organic phase was then dried over $MgSO_4$, and concentrated under vacuum. The residue was then dissolved in minimum amount of dichloromethane and subjected to the flash chromatography on silica gel. After concentrating the fraction containing the product, the residue was dried over reduced pressure to yield pure product.

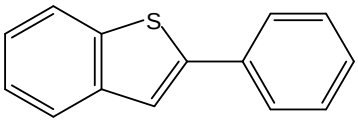




2-(3-*tert*-Butylphenyl)benzothiophene and 2-(4-*tert*-butylphenyl)benzothiophene (Entry 9, Table 1): 4-*tert*-butylchlorobenzene (135 mg, 0.8 mmol), benzothiophene (67 mg, 0.5 mmol), TMPLi in pentane (1 M, 1.5 mL) followed by anhydrous THF (0.38 mL), rt, 1.5 hrs. After column chromatography (hexanes/CH_2Cl_2 90/10), 113 mg (85 %) of a white solid was obtained. The isomer ratio was determined to be 1/1.2 *m/p* by GC analysis. The isomers were separated by fractional crystallization from hexanes. p-Isomer was crystallized and collected by vacuum filtration at 0 ^oC. Rest of the liquid was cooled down to -20 ^oC. p-Isomer and some m-isomer were collected. Pure m-isomer was isolated from the liquid phase. The following amounts were obtained: *p*-isomer (white solid): 51 mg, *m*-isomer (colorless liquid): 41 mg; additionally, 17 mg of isomer mixture was recovered.

p-Isomer is known.[2] R_f = 0.27 (hexanes). ^1H NMR (400 MHz, $CDCl_3$) δ7.81 (d, *J* = 8.1 Hz, 1H), 7.75 (d, *J* = 8.1 Hz, 1H), 7.65 (dt, *J* = 8.2 Hz, 2.0 Hz, 2H), 7.50 (s, 1H), 7.45 (dt, *J* = 8.2 Hz, 2.0 Hz, 2H), 7.36-7.27 (m, 2H), 1.36 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 151.6, 144.4, 140.9, 139.5, 131.6, 126.3, 126.0, 124.5, 124.2, 123.5, 122.3, 119.0, 35.0, 31.3.

m-Isomer: R_f = 0.39 (hexanes). ^1H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.8 Hz, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.75-7.73 (m, 1H), 7.56-7.53 (m, 2H), 7.41-7.29 (m, 4H), 1.40 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 152.0, 145.0, 140.8, 139.6, 134.1, 128.8, 125.6, 124.6, 124.3, 123.9, 123.7, 123.6, 122.4, 119.4, 34.9, 31.4. FT-IR (neat, cm^{-1}) υ 1296. Anal calcd for $C_{18}H_{18}S$ (266.11 g/mol): C, 81.15; H, 6.81; Found. C, 80.91; H, 6.71.



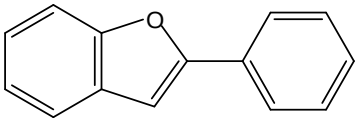
2- Phenylbenzothiophene (Entry 1, Table 2) :

Benzothiophene (67 mg, 0.5 mmol), PhCl (84 mg, 0.75 mmol), TMPLi in pentane (1 M, 1.4 mL) followed by anhydrous THF (0.35 mL), rt, 30 minutes. After column chromatography (90/10 hexanes/CH_2Cl_2), 90 mg (86 %) of a light yellow solid was obtained.

Benzothiophene (67 mg, 0.5 mmol), PhCl (112.5 mg, 1 mmol), Cy_2NLi in THF (1 M, 2.0 mL), 0 ^oC, 3 hours. After column chromatography (hexanes), 75 mg (72 %) of a light yellow solid was obtained.

Benzothiophene (67 mg, 0.5 mmol), PhF (120 mg, 1.25 mmol), TMPLi in THF (1 M, 2.0 mL), rt, 10 minutes. After column chromatography (90/10 hexanes/DCM), 89 mg (85 %) of a light yellow solid was obtained.

R_f = 0.35 (hexanes/CH_2Cl_2 95/5). This compound is known.[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.8 Hz, 1H), 7.77 (dd, *J* = 7.8 Hz, 1.3 Hz, 1H), 7.73 (dd, *J* = 7.8 Hz, 1.3 Hz, 2H), 7.56 (s, 1H), 7.43 (dt, *J* = 7.8 Hz, 1.3 Hz, 2H), 7.38-7.30 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 144.3, 140.8, 139.6, 134.4, 129.1, 128.4, 126.6, 124.6, 124.4, 123.7, 122.4, 119.6.



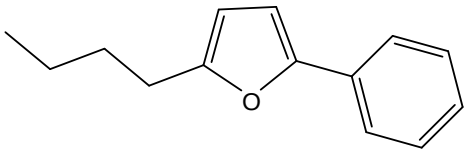
2-Phenylbenzofuran (Entry 2, Table 2):

Benzofuran (59 mg, 0.5 mmol), PhCl (84 mg, 0.75 mmol), TMPLi in pentane (1 M, 1.4 mL) followed by anhydrous THF (0.07 mL), rt, 2.5 hours. After column chromatography (hexanes/CH_2Cl_2 85/15), 79 mg (81%) of a white solid was obtained.

Benzofuran (59 mg, 0.5 mmol), PhCl (112.5 mg, 1 mmol), LDA in THF (1 M, 1.7 mL), 0 ^oC, 4 hours. After column chromatography (hexanes/ CH_2Cl_2 90/10), 72 mg (75 %) of a white solid was obtained.

R_f = 0.40 (hexanes/CH_2Cl_2 95/5). This compound is known.[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dt, *J* = 7.3 Hz, 1.3 Hz, 2H), 7.60 (dd, *J* = 7.9 Hz, 1.3 Hz, 1H), 7.55 (dd, *J* = 7.9 Hz, 1.1 Hz, 1H), 7.47 (dt, *J* = 7.9

Hz, 1.8 Hz, 2H), 7.37 (tt, *J* = 7.9 Hz, 1.1 Hz, 1H), 7.23-7.33 (m, 2H), 7.04(s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.0, 155.0, 130.6, 129.3, 128.9, 128.7, 125.0, 124.4, 123.0, 121.1, 111.3, 101.4.

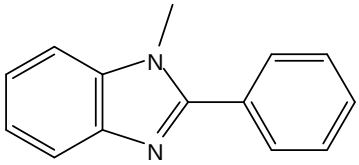


2-*n*-Butyl-5-phenylfuran (Entry 3, Table 2):

2-*n*-Butylfuran (62 mg, 0.5 mmol), PhCl (84 mg, 0.75 mmol), TMPLi in pentane (1 M, 1.4 mL) followed by anhydrous THF (0.35 mL), rt, 30 minutes. After column chromatography (hexanes/CH_2Cl_2 95/5), 80 mg (80 %) of a colorless oil was obtained.

2-*n*-Butylfuran (62 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), Cy_2NLi in THF (1 M, 1.7 mL), 0 ^{o}C, 4 hours. After column chromatography (hexanes), 68 mg (68 %) of a colorless oil was obtained.

R_f = 0.53 (hexanes/CH_2Cl_2 95/5). This compound is known.[4] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.62 (m, 2H), 7.36 (t, *J* = 7.8 Hz, 2H), 7.21 (tt, *J* = 7.8 Hz, 1.6 Hz, 1H), 6.55 (d, *J* = 3.3 Hz, 1H), 6.06 (d, *J* = 3.3 Hz, 1H), 2.69 (t, *J* = 7.7 Hz, 2H), 1.68 (pentet, *J* = 7.7 Hz, 2H), 1.42 (s, *J* = 7.7 Hz, 2H), 0.96 (t, *J* = 7.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 156.6, 152.1, 131.3, 128.7, 126.8, 123.4, 106.9, 105.7, 30.3, 28.0, 22.4, 13.9.

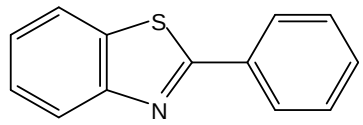


2-Phenyl-1-methylbenzimidazole (Entry 4, Table 2):

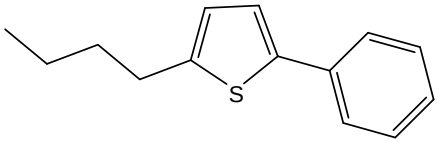
1-Methylbenzimidazole (66 mg, 0.5 mmol), PhCl (84 mg, 0.75 mmol), TMPLi in pentane (1 M, 1.4 mmol) followed by anhydrous THF (0.15 mL), rt, 1 hour. After column chromatography (hexanes/EtOAc 55/45), 95 mg (91%) of a brown solid was obtained.

1-Methylbenzimidazole (66 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), Cy_2NLi in THF (1 M, 2.0 mmol), 0 ^{o}C, 3 hours. After column chromatography (hexanes/EtOAc 55/45), 84 mg (81 %) of a brown solid was obtained.

R_f = 0.62 (hexanes/EtOAc 50/50). This compound is known.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.81 (m, 1H), 7.77-7.74 (m, 2H), 7.54-7.49 (m, 3H), 7.40-7.37 (m, 1H), 7.34-7.30 (m, 2H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 153.9, 143.0, 136.7, 130.3, 129.8, 129.6, 128.8, 122.9, 122.5, 119.9, 109.7, 31.8.



2-Phenylbenzothiazole (Entry 5, Table 2): Benzothiazole (67 mg, 0.5 mmol), PhCl (112.5 mg, 1 mmol), TMPLi in THF (1 M, 1.8 mL), -46 ^{o}C, 12 hours. After column chromatography (hexanes/CH_2Cl_2 55/45), 76 mg (72 %) of a light yellow solid was obtained. R_f = 0.48 (hexanes/EtOAc : 80/20). This compound is known.[5] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11-8.07 (m, 3H), 7.90 (dt, *J* = 7.7 Hz, 0.9 Hz, 1H), 7.51-7.47 (m, 4H), 7.38 (dt, *J* = 7.7 Hz, 0.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 168.2, 154.2, 135.2, 133.7, 131.1, 129.1, 127.7, 126.4, 125.3, 123.3, 121.7.

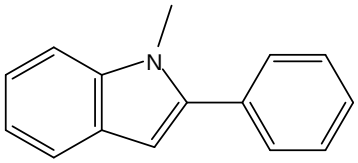


2-*n*-Butyl-5-phenylthiophene (Entry 6, Table 2):

2-*n*-Butylthiophene (70 mg, 0.5 mmol), PhCl (73 mg, 0.65 mmol), TMPLi in pentane (1 M, 1.4 mL) followed by anhydrous THF (0.15 mL), rt, 1 hour. After column chromatography (hexanes/CH_2Cl_2 95/5), 86 mg (80 %) of a colorless oil was obtained.

2-*n*-Butylthiophene (70 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), Cy_2NLi in THF (1 M, 1.7 mL), 0 ^{o}C, 4 hours. After column chromatography (hexanes), 80 mg (74 %) of a colorless oil was obtained.

R_f = 0.27 (hexanes). This compound is known.[6] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (dt, *J* = 8.0 Hz, 1.3 Hz, 2H), 7.34-7.38 (m, 2H), 7.25 (tt, *J* = 8.0 Hz, 1.3 Hz, 1H), 7.14 (d, *J* = 3.5 Hz, 1H), 6.76 (d, *J* = 3.5 Hz, 1H), 2.84 (t, *J* = 7.8 Hz, 2H), 1.71 (pt, *J* = 7.8 Hz, 2H), 1.43 (sextet, *J* = 7.8 Hz, 2H), 0.97 (t, *J* = 7.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 145.8, 141.7, 134.9, 128.9, 127.0, 125.6, 125.1, 122.8, 33.9, 30.1, 22.3, 14.0.

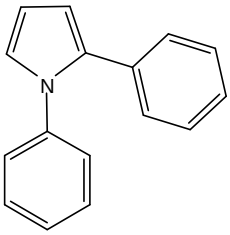


1-Methyl-2-phenylindole (Entry 7, Table 2):

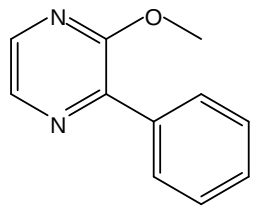
Procedure 1: A 2 dram vial equipped with a magnetic stir bar was charged with 1-methylindole (66 mg, 0.5 mmol). The vial was flushed with argon and capped. To this mixture was added TMPLi as a suspension in pentane (1 M, 1.7 mL), followed by anhydrous THF (0.2 mL) via syringe. The solution was stirred at rt for 30 minutes. PhCl (112 mg, 1 mmol) was dissolved in 9/1 pentane/THF mixture (0.6 mL) and added to the vial via syringe and reaction mixture was stirred for 1.5 hours. After column chromatography (hexanes/CH_2Cl_2 90/10), 93 mg (90 %) of a light brown solid was obtained.

Procedure 2 (following the general procedure): 1-Methylindole (66 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), TMPLi in pentane (1 M, 1.8 mL), 40 ^oC, 24 hours. After column chromatography (hexanes/CH_2Cl_2 90/10), 92 mg (89 %) of a light brown solid was obtained.

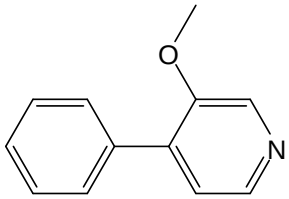
R_f = 0.48 (hexanes/CH_2Cl_2 85/15) . This compound is known.[7] ^1H NMR (400 MHz, $CDCl_3$) δ 7.68 (dt, *J* = 7.8 Hz, 0.9 Hz, 1H), 7.56-7.54 (m, 2H), 7.53-7.48 (m, 2H), 7.45-7.39 (m, 2H), 7.29 (dt, *J* = 7.1 Hz, 1.3 Hz, 1H), 7.18 (dt, *J* =7.1 Hz, 1.3 Hz, 1H), 6.6(s, 1H), 3.8 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 141.7, 138.4, 133.0, 129.5, 128.6, 128.0, 127.9, 121.8, 120.6, 120.0, 109.8, 101.8, 31.3.



1,2-Diphenylpyrrole (Entry 8, Table 2): 1-Phenylpyrrole (143 mg, 1 mmol), PhCl (56 mg, 0.5 mmol), TMPLi in pentane (1 M, 1.4 mL), 40 ^oC, 24 hours. After column chromatography (hexanes/CH_2Cl_2 95/5), 85 mg (78%) of a white solid was obtained. R_f = 0.56 (hexanes/CH_2Cl_2 90/10). This compound is known.[8] ^1H NMR (400 MHz, $CDCl_3$) δ7.35 (tq, *J* = 7.6 Hz, 1.4 Hz, 2H), 7.31-7.27 (m, 1H), 7.26-7.17 (m, 7H), 6.99 (dd, *J* = 7.6 Hz, 4.5 Hz, 1H), 6.47 (dd, *J* = 7.6 Hz, 4.5 Hz, 1H), 6.41 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 140.6, 133.9, 133.1, 129.1, 128.4, 128.2, 126.7, 125.9, 124.5, 110.8, 109.4.

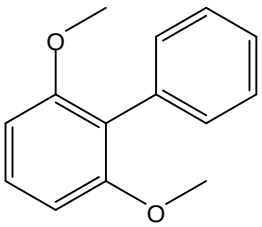


2-Methoxy-3-phenylpyrazine (Entry 9, Table 2): 2-Methoxypyrazine (55 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), TMPLi in THF (1 M, 1.8 mL), -46 ^{o}C, 12 hours. After column chromatography (hexanes/EtOAc 40/60), 47 mg (55 %) of a yellow solid was obtained. R_f = 0.54 (hexanes/EtOAc 30/70). This compound is known.[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 2.8 Hz, 1H), 8.05 (d, *J* = 2.8 Hz, 1H), 8.05-8.02 (m, 2H), 7.49-7.42 (m, 3H), 4.03 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 158.1, 143.5, 139.2, 136.5, 135.9, 129.3, 129.2, 128.3, 53.8.

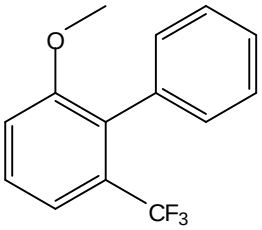


3-Methoxy-4-phenylpyridine (Entry 10, Table 2):

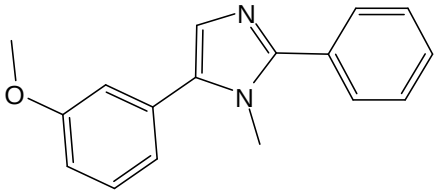
A 2 dram vial equipped with a magnetic stir bar was charged with 3-methoxypyridine (55 mg, 0.5 mmol), PhCl (112.5 mg, 1 mmol). The vial was flushed with argon, capped and placed into the cooling bath (-13 ^{o}C). To this mixture was slowly added TMPLi in pentane (1 M, 1.7 mL) followed by anhydrous THF (0.13 mL) by injecting through the septum via syringe.. The vial was flushed with argon for 20 seconds and then stirred for 12 hours at -13 °C. After column chromatography (hexanes/EtOAc 20/80), 66 mg (71 %) of a yellow solid was obtained. R_f = 0.32 (hexanes/EtOAc 20/80). This compound is known.[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36-8.30 (m, 2H), 7.58-7.55 (m, 2H), 7.45 – 7.37 (m, 3H), 7.24 (d, *J* = 5.3 Hz, 1H). 3.90 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 152.6, 143.0, 137.7, 135.8, 134.4, 129.3, 128.45, 128.4, 124.6, 56.4.



2,6-Dimethoxy-1,1'-biphenyl (Entry 11, Table 2): 1.3-Dimethoxybenzene (69 mg, 0.5 mmol), PhCl (112 mg, 1 mmol), TMPLi in pentane (1 M, 1.7 mL), 40 ^{o}C, 24 hours. After column chromatography (hexanes/CH_2Cl_2 50/50), 101 mg (95%) of a white solid was obtained. R_f = 0.29 (hexanes/CH_2Cl_2 60/40). This compound is known.[11] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.37 (m, 5H), 7.34 (t, *J* = 8.4 Hz, 1H), 6.71 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.8, 134.3, 131.1, 128.8, 127.8, 126.9, 119.6, 104.3, 56.1.1



2-Methoxy-6-trifluoromethyl-1,1'-biphenyl (Entry 12, Table 2): 3-Trifluoromethylanisole (88 mg, 0.5 mmol), PhCl (84 mg, 0.75 mmol), TMPLi in pentane (1 M, 1.5 mL), followed by anhydrous THF (0.1 mL), rt, 4h. After column chromatography (hexanes/CH_2Cl_2 80/20), 102 mg (81 %) of a white solid was obtained. R_f = 0.26 (hexanes/DCM 80/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.39 (m, 5H), 7.30-7.27 (m, 2H), 7.16 (d, *J* = 8.3 Hz, 1H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 157.9, 134.9, 130.4, 130.3 (q, J_{C-F} = 29.2 Hz), 130.1, 128.8, 127.7, 127.6, 124.05 (q, J_{C-F} = 280 Hz), 117.85 (q, J_{C-F} = 5.5 Hz), 114.3, 56.2.



2-(3-Methoxyphenyl)-1-methyl-5-phenylimidazole (Eq. 1)

Two-step method with isolation of intermediate: all reactions follow the general procedure.

Step 1: 1-Methylimidazole (41 mg, 0.5 mmol), PhCl (96 mg, 0.85 mmol), LDA (1 M, 1.6 mL), rt, 3 hours. After column chromatography (hexanes/EtOAc 20/80), 63 mg (80 %) of 1-methyl-2-phenylimidazole (light yellow liquid) was obtained.

Step 2: 1-Methyl-2-phenylimidazole (79 mg, 0.5 mmol), 2-chloroanisole (142.5 mg, 1.0 mmol), TMPLi in THF (1 M, 1.8 mL), rt, 2 hours. After column chromatography (MeOH/EtOAc 10/90), then HPLC (hexanes/EtOAc/MeOH 20/70/10), 94 mg (71 %) of yellowish liquid was obtained.

Reactions without isolation of intermediate: A 2 dram vial equipped with a magnetic stir bar was charged 1-methylimidazole (41 mg, 0.5 mmol), PhCl (96 mg, 0.85 mmol). The vial was flushed with argon and capped. To this mixture was slowly added LDA in THF (1 M, 1.6 mL) by injecting through the septum via syringe. The vial was flushed with argon (20 seconds) and then stirred at room temperature for 3 hours. The reaction mixture was quenched with anhydrous MeOH (0.6 mL) and evacuated to remove all solvent and diisopropylamine. 2-Chloroanisole (142.5 mg, 1.0 mmol) was then added to the vial containing the residue. The vial was flushed with argon and capped. To this mixture was slowly added TMPLi in THF (1 M, 1.8 mL) by injecting through the septum via syringe. The vial was flushed with argon again (20 seconds) and stirred at room temperature for another 2 hours. After column chromatography (MeOH/EtOAc 10/90), and then HPLC (hexanes/EtOAc/MeOH 20/70/10), 66 mg (50 %) of a yellowish liquid was obtained.

R_f = 0.18 (EtOAc). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.67 (m, 2H), 7.49-7.34 (m, 4H), 7.20 (s, 1H), 7.03 (d, *J* = 7.8 Hz, 1H), 6.99 – 6.97 (m, 1H), 6.92 (dd, *J* = 7.8 Hz, 2.4 Hz, 1H), 3.85 (s, 3H), 3.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, ppm) δ 159.9, 149.5, 135.4, 131.6, 130.9, 129.9, 128.9, 128.8, 128.7, 127.7, 121.1, 114.5, 113.3, 55.4, 33.9. FT-IR (neat, cm^{-1}) υ 1605, 1580, 1480. 1467, 1248, 1031. Anal calcd for $C_{17}H_{16}N_2O$ (264.13 g/mol): C, 77.25; H, 6.10; N, 10.60; Found. C, 77.08; H, 6.13; N, 10.88.

References

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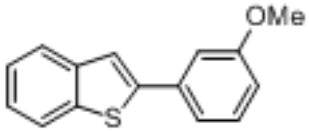

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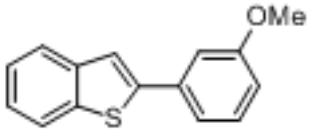

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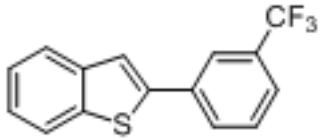

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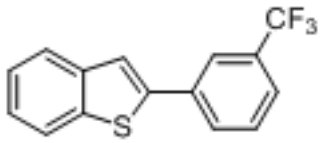

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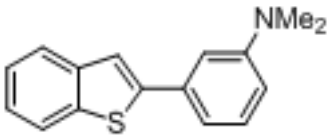
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.4[dC]
```



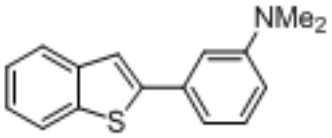
Entry 3 and Entry 4, Table 1


```
Filename          = 5-103-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 5-103-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 11:24:08
Revision_time     = 18-DEC-2010 15:07:45
Current_time      = 18-DEC-2010 15:08:05

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.5[dC]
```



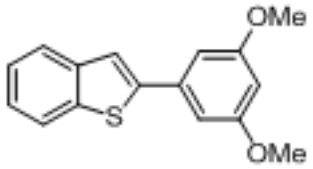
Entry 3 and Entry 4, Table 1


```
Filename          = 5-103-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 5-103-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 11:42:05
Revision_time     = 18-DEC-2010 15:08:11
Current_time      = 18-DEC-2010 15:08:30

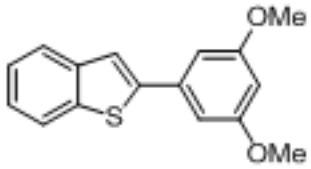
Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.9[dC]
```



Entry 5, Table 1



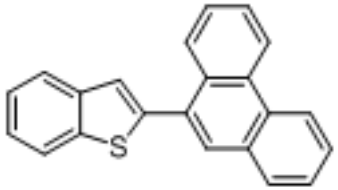
Entry 5, Table 1


```
Filename          = 2-79-6 c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 2-79-6-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 09:36:49
Revision_time     = 18-DEC-2010 14:42:55
Current_time      = 18-DEC-2010 14:43:28

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.1[dC]
```



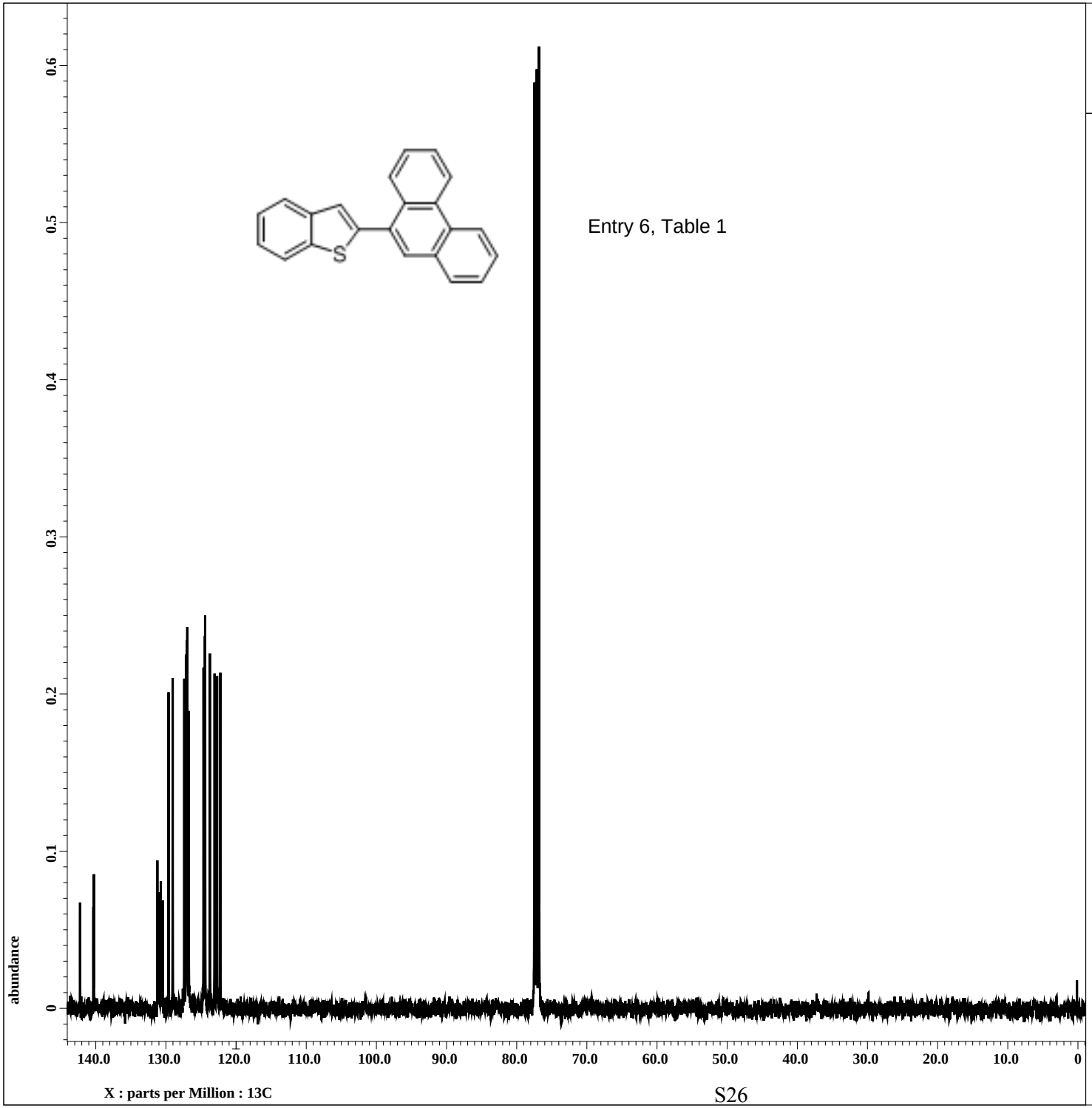
Entry 6, Table 1


```
Filename          = 3-153-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 3-153-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 20:15:50
Revision_time     = 18-DEC-2010 14:53:44
Current_time      = 18-DEC-2010 14:54:18

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 18.5[dC]
```

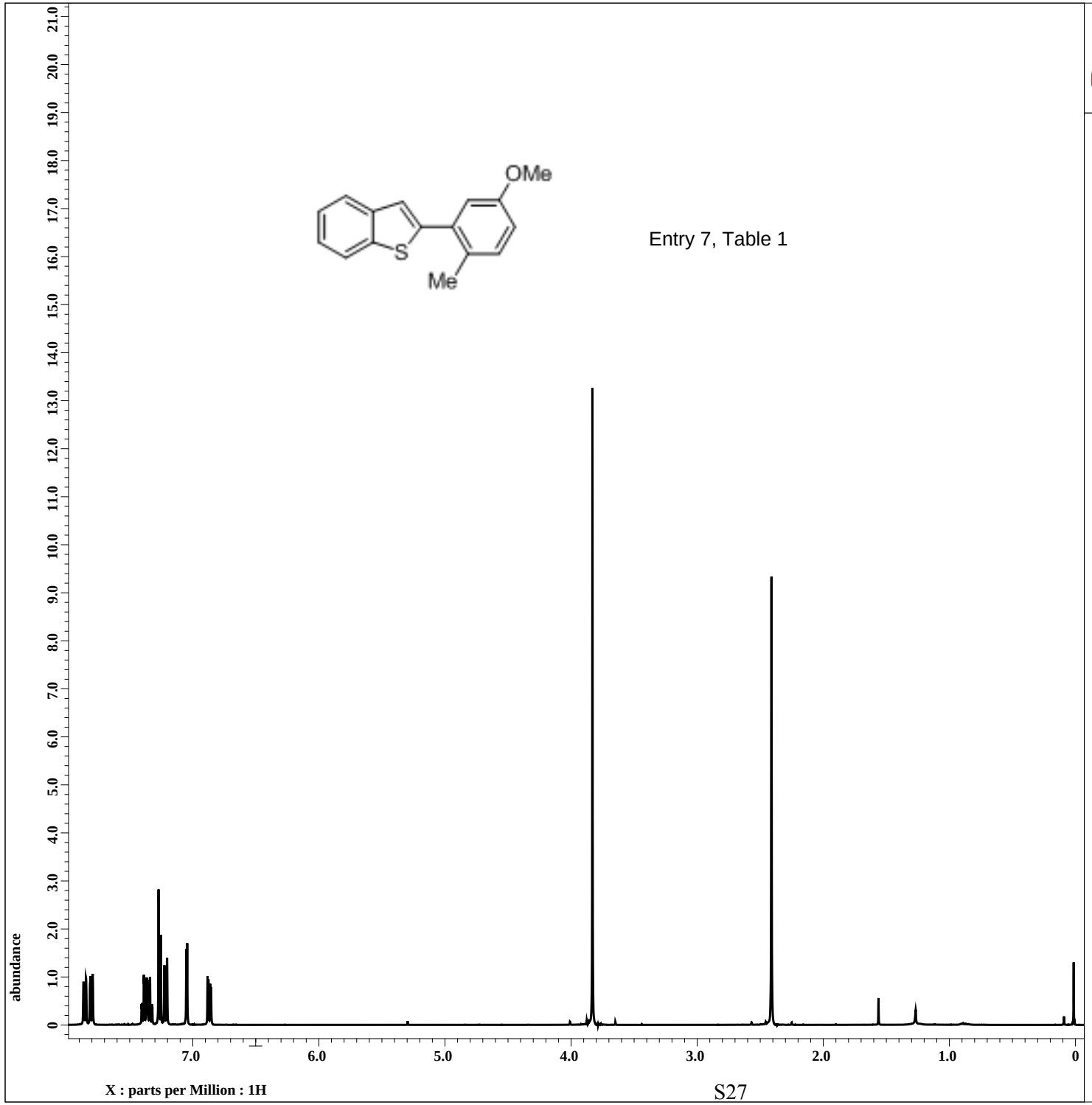



```
Filename          = 3-153-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 3-153-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 20:33:47
Revision_time     = 18-DEC-2010 14:54:25
Current_time      = 18-DEC-2010 14:55:00

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 18.9[dC]
```

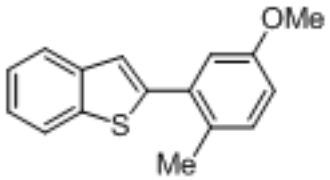



```
Filename          = 2-113-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 2-113-6-h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 10:47:50
Revision_time     = 18-DEC-2010 14:44:05
Current_time      = 18-DEC-2010 14:44:25

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 18.9[dC]
```



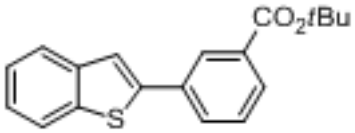
Entry 7, Table 1


```
Filename          = 2-113-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 2-113-6-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 11:06:13
Revision_time     = 18-DEC-2010 14:44:30
Current_time      = 18-DEC-2010 14:44:52

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.2[dC]
```



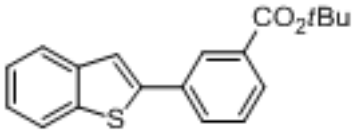
Entry 8, Table 1


```
Filename          = 2-7-7-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 2-7-7-h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 08:52:20
Revision_time     = 18-DEC-2010 14:40:46
Current_time      = 18-DEC-2010 14:41:15

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 18.8[dC]
```



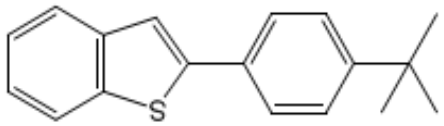
Entry 8, Table 1


```
Filename          = 2-7-7-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 2-7-7 c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 09:11:04
Revision_time     = 18-DEC-2010 14:41:33
Current_time      = 18-DEC-2010 14:41:57

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.4[dC]
```



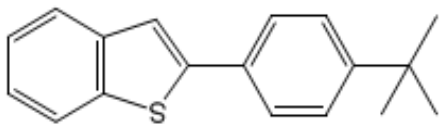
Entry 9, Table 1


```
Filename          = 1-107-6-1-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-107-6-1-h1
Solvent           = CHLOROFORM-D
Creation_time     =  1-DEC-2010 20:23:50
Revision_time     = 18-DEC-2010 14:35:57
Current_time      = 18-DEC-2010 14:36:29

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.8[dC]
```



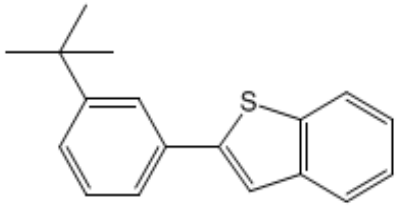
Entry 9, Table 1


```
Filename          = 1-107-6-1 c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-107-6-1-c13
Solvent           = CHLOROFORM-D
Creation_time     =  1-DEC-2010 21:17:44
Revision_time     = 18-DEC-2010 14:36:37
Current_time      = 18-DEC-2010 14:37:02

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1536
Total_scans       = 1536

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 20.2[dC]
```



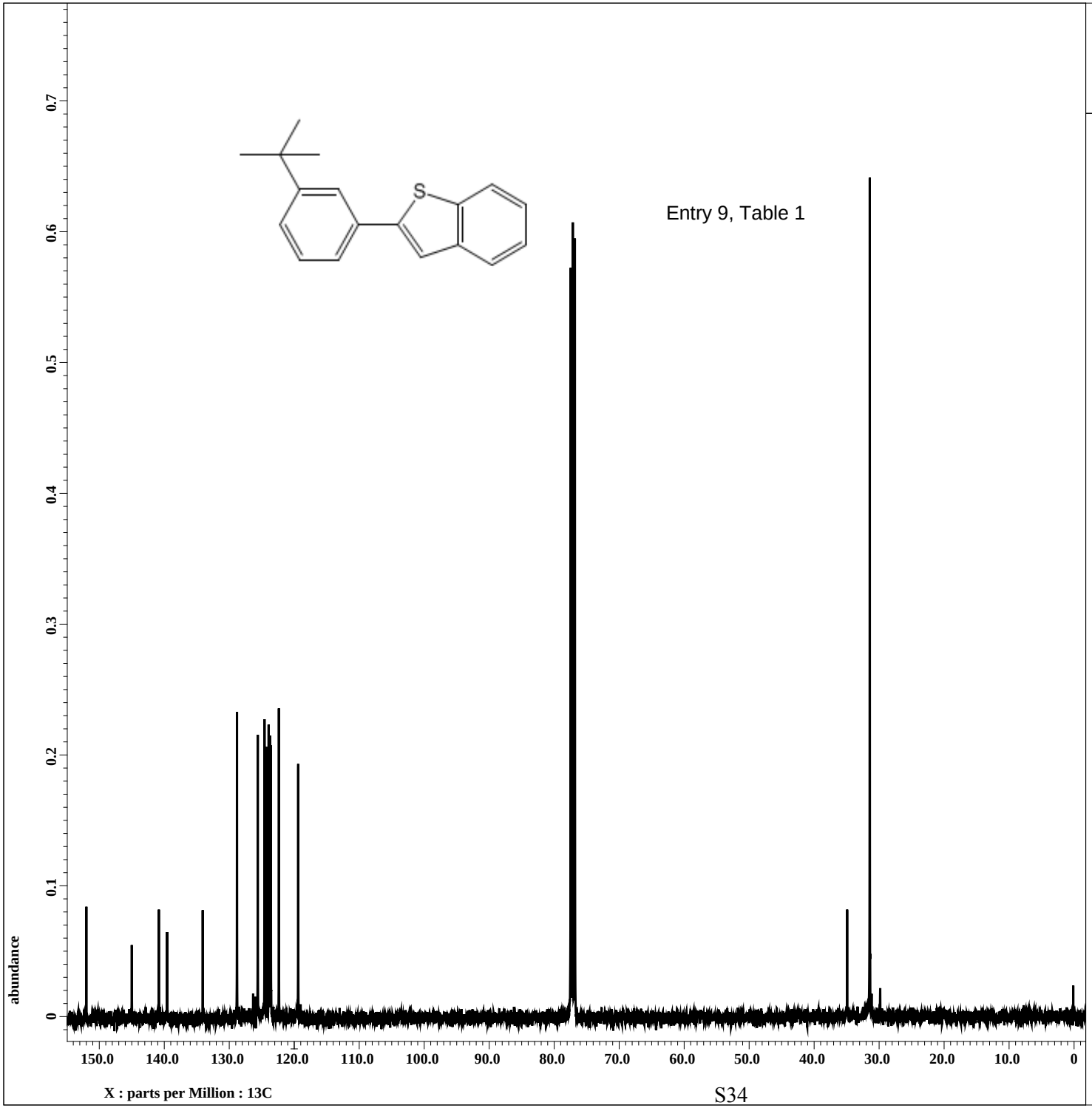
Entry 9, Table 1


```
Filename          = 1-107-6-2-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-107-6-2-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 17:25:48
Revision_time     = 18-DEC-2010 15:09:46
Current_time      = 18-DEC-2010 15:10:09

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 56
Total_scans       = 56

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.4[dC]
```




```
Filename          = 1-107-6-2-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-107-6-2-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 17:43:41
Revision_time     = 18-DEC-2010 15:10:15
Current_time      = 18-DEC-2010 15:10:32

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.6[dC]
```

Entry 1, Table 2


```
Filename          = 1-13-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-13-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 09:58:19
Revision_time     = 20-DEC-2010 15:12:59
Current_time      = 20-DEC-2010 15:13:28

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19[dC]
```

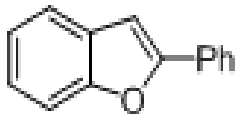
Entry 1, Table 2


```
Filename          = 1-13-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-13-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 10:16:36
Revision_time     = 18-DEC-2010 14:30:46
Current_time      = 18-DEC-2010 14:31:40

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.8[dC]
```



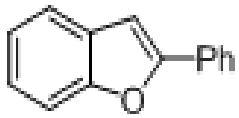
Entry 2, Table 2


```
Filename          = 4-11-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 4-11-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 09:29:01
Revision_time     = 18-DEC-2010 14:55:23
Current_time      = 18-DEC-2010 14:55:44

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.1[dC]
```



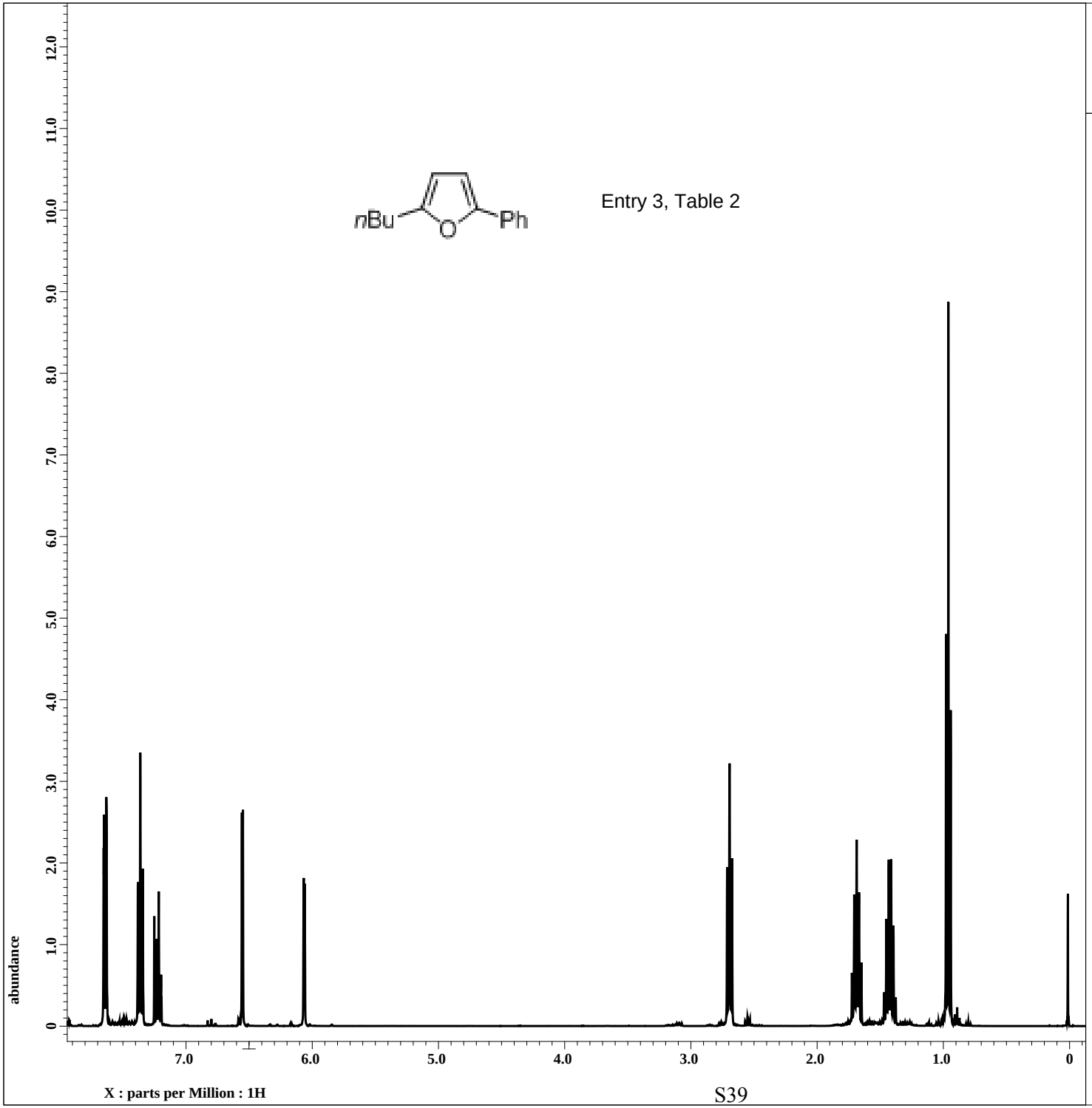
Entry 2, Table 2


```
Filename          = 4-11-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 4-11-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 09:46:57
Revision_time     = 18-DEC-2010 14:55:50
Current_time      = 18-DEC-2010 14:56:09

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.5[dC]
```

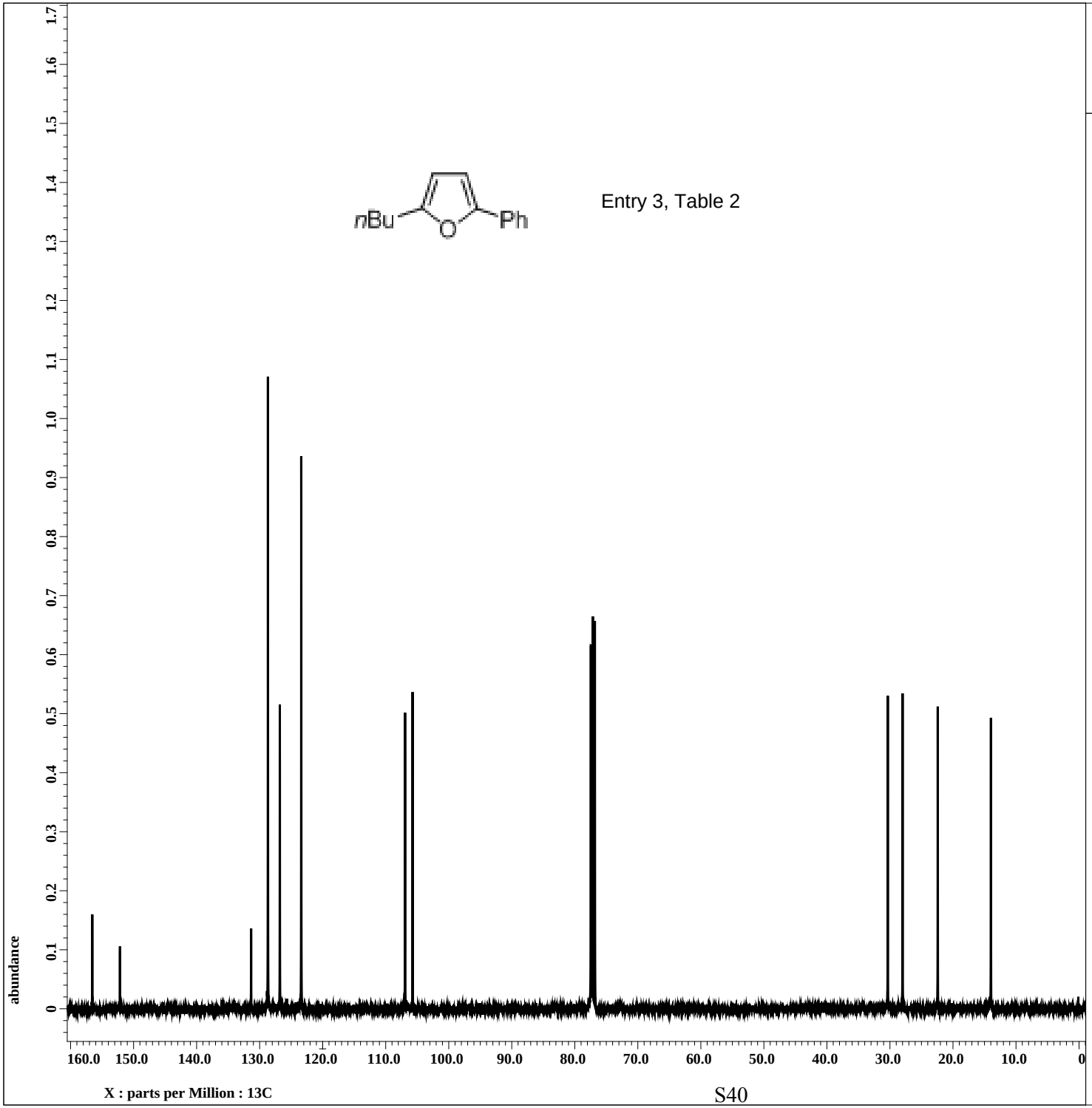



```
Filename          = 4-93-6-5-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 4-93-6-5-h1
Solvent           = CHLOROFORM-D
Creation_time     = 13-DEC-2010 14:57:28
Revision_time     = 18-DEC-2010 14:57:28
Current_time      = 18-DEC-2010 14:57:48

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.6[dC]
```

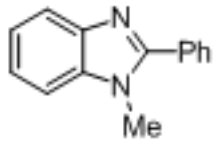


Filename = 4-93-6-5-c13-2.jdf
Author = daugulis
Experiment = single_pulse_dec
Sample_id = 4-93-6-5-c13
Solvent = CHLOROFORM-D
Creation_time = 13-DEC-2010 15:15:28
Revision_time = 18-DEC-2010 14:57:54
Current_time = 18-DEC-2010 14:58:17

Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.389766[T] (400[MHz]
X_acq_duration = 1.04333312[s]
X_domain = 13C
X_freq = 100.52530333[MHz]
X_offset = 120[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95846665[Hz]
X_sweep = 31.40703518[kHz]
Irr_domain = 1H
Irr_freq = 399.78219838[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 512
Total_scans = 512

X_90_width = 12.4525[us]
X_acq_time = 1.04333312[s]
X_angle = 30[deg]
X_atn = 6[dB]
X_pulse = 4.15083333[us]
Irr_atn_dec = 22[dB]
Irr_atn_noe = 22[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 1[s]
Recvr_gain = 60
Relaxation_delay = 1[s]
Repetition_time = 2.04333312[s]
Temp_get = 19.7[dC]



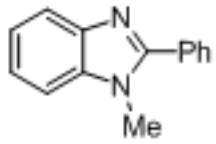
Entry 4, Table 2


```
Filename          = 3-93-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 3-93-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 08:54:14
Revision_time     = 18-DEC-2010 14:46:23
Current_time      = 18-DEC-2010 14:46:45

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.4[dC]
```



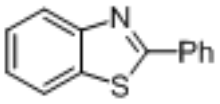
Entry 4, Table 2


```
Filename          = 3-93-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 3-93-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 09:13:56
Revision_time     = 18-DEC-2010 14:46:51
Current_time      = 18-DEC-2010 14:47:39

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 20[dC]
```



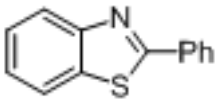
Entry 5, Table 2


```
Filename          = 3-57-5-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 3-57-5-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 10:57:23
Revision_time     = 18-DEC-2010 14:45:04
Current_time      = 18-DEC-2010 14:45:32

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.5[dC]
```



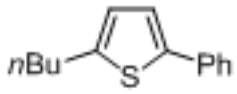
Entry 5, Table 2


```
Filename          = 3-57-5-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 3-57-5-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 11:15:19
Revision_time     = 18-DEC-2010 14:45:40
Current_time      = 18-DEC-2010 14:46:02

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.9[dC]
```



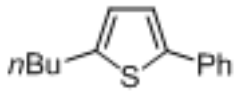
Entry 6, Table 2


```
Filename          = 1-103-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-103-6-h1
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 17:00:49
Revision_time     = 18-DEC-2010 15:11:05
Current_time      = 18-DEC-2010 15:11:28

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.3[dC]
```



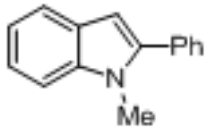
Entry 6, Table 2


```
Filename          = 1-103-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-103-6-c13
Solvent           = CHLOROFORM-D
Creation_time     =  6-DEC-2010 17:18:46
Revision_time     = 18-DEC-2010 15:11:34
Current_time      = 18-DEC-2010 15:11:50

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.6[dC]
```



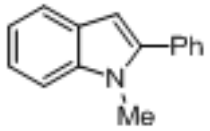
Entry 7, Table 2


```
Filename          = 1-151-l5-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-151-l5-h1
Solvent           = CHLOROFORM-D
Creation_time     = 29-SEP-2010 17:31:51
Revision_time     = 18-DEC-2010 14:38:54
Current_time      = 18-DEC-2010 14:39:56

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.3[dC]
```



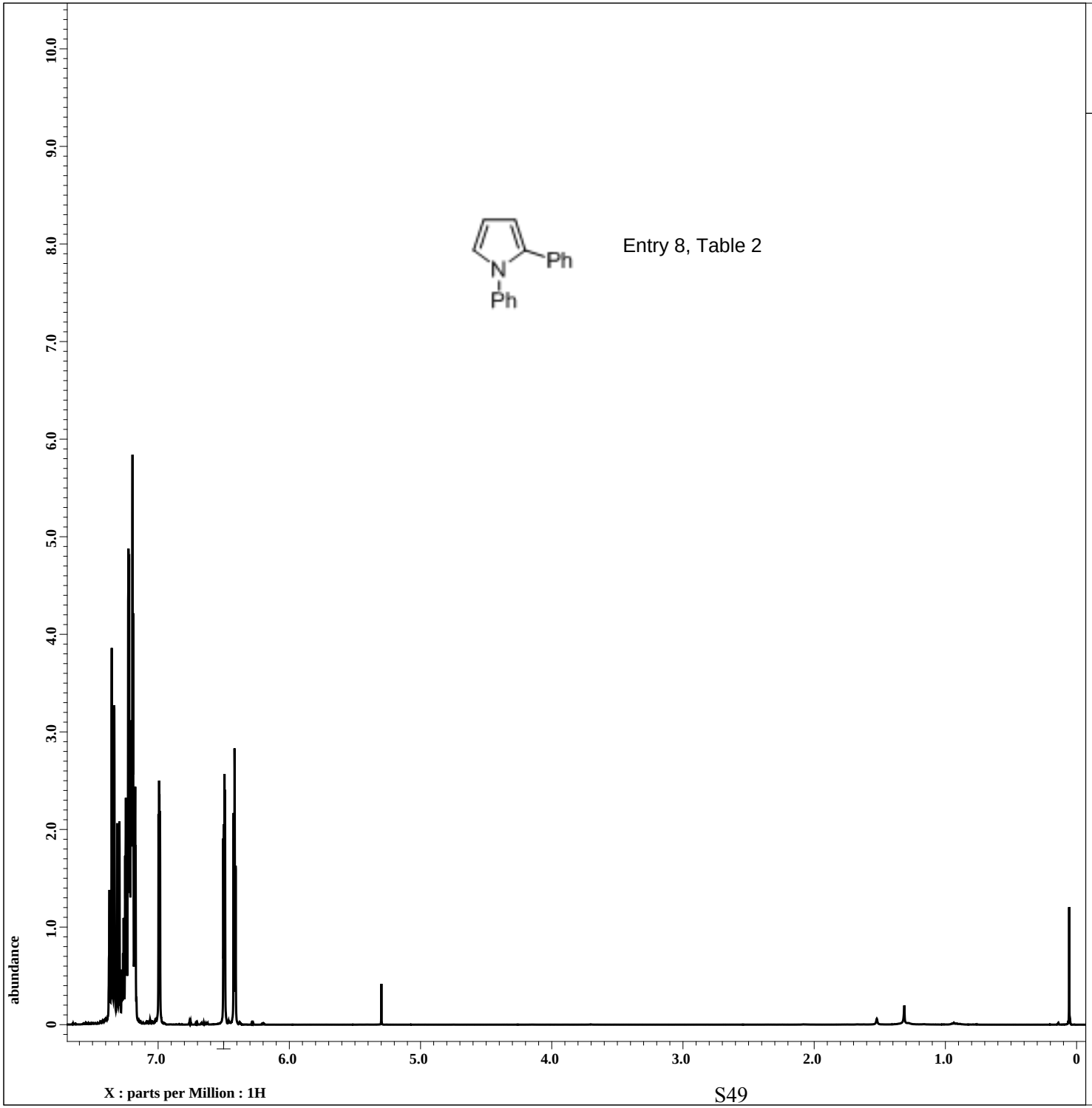
Entry 7, Table 2


```
Filename          = 1-151-l5-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-151-l5-c13
Solvent           = CHLOROFORM-D
Creation_time     = 29-SEP-2010 17:54:05
Revision_time     = 18-DEC-2010 14:40:02
Current_time      = 18-DEC-2010 14:40:23

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.6[dC]
```

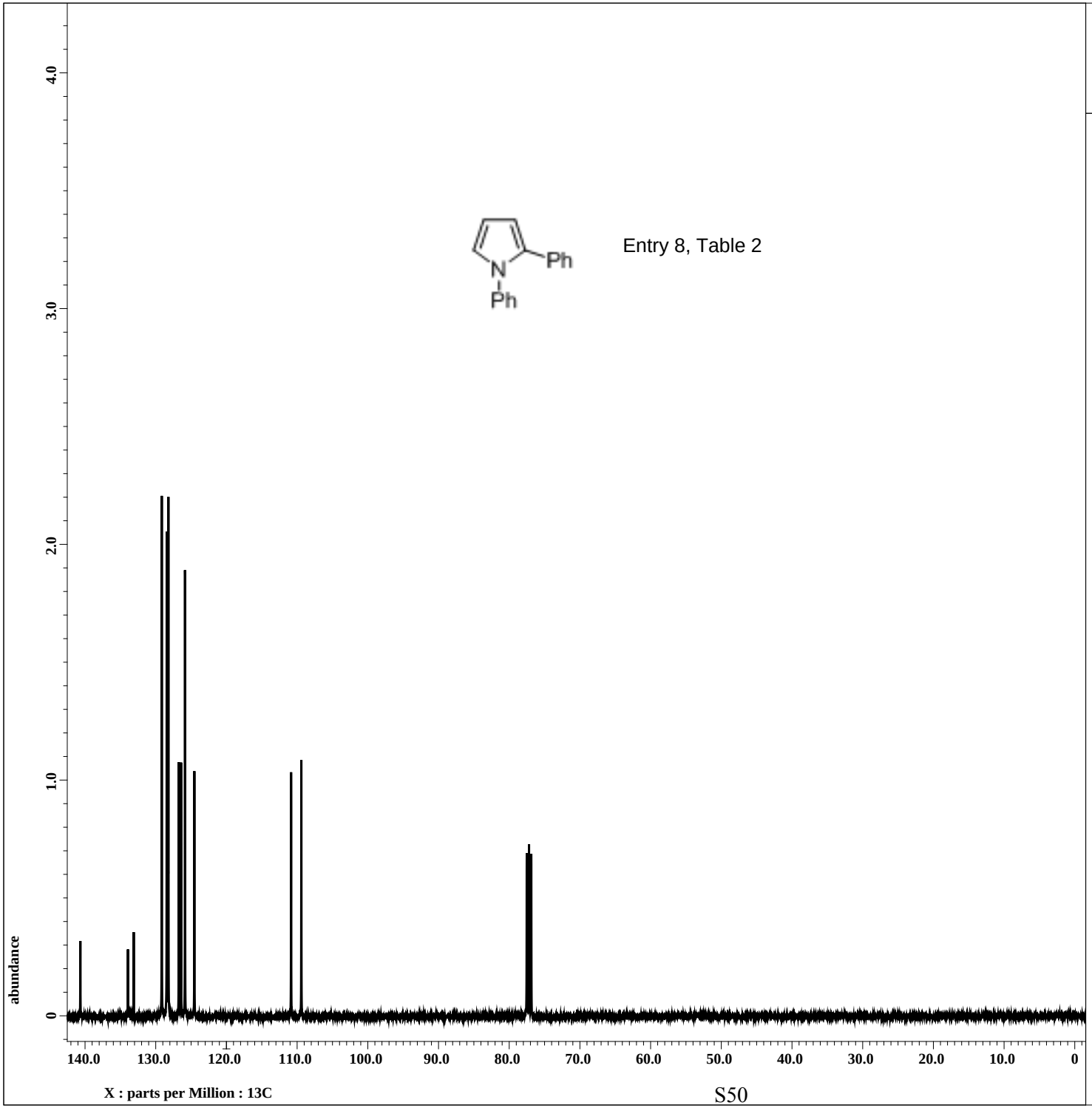



```
Filename          = 1-95-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-95-6 h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 12:25:34
Revision_time     = 18-DEC-2010 14:32:17
Current_time      = 18-DEC-2010 14:32:42

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 32
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19[dC]
```

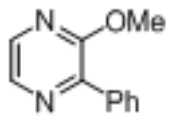



```
Filename          = 1-95-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-95-6-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 12:43:32
Revision_time     = 18-DEC-2010 14:33:08
Current_time      = 18-DEC-2010 14:33:30

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.4[dC]
```



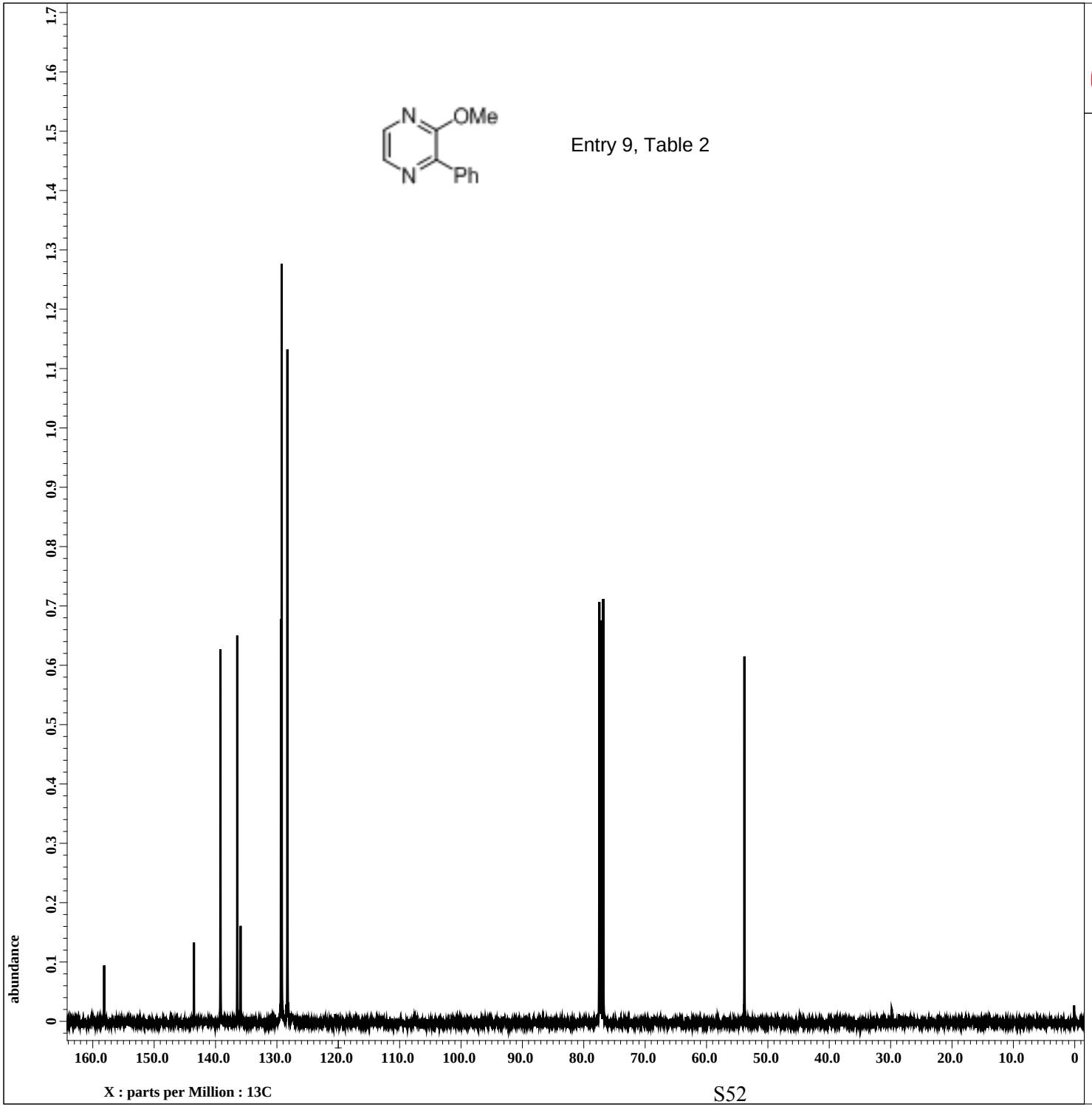
Entry 9, Table 2


```
Filename          = 1-137-5 h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-137-5-h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 13:27:44
Revision_time     = 18-DEC-2010 14:37:33
Current_time      = 18-DEC-2010 14:38:08

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.1[dC]
```



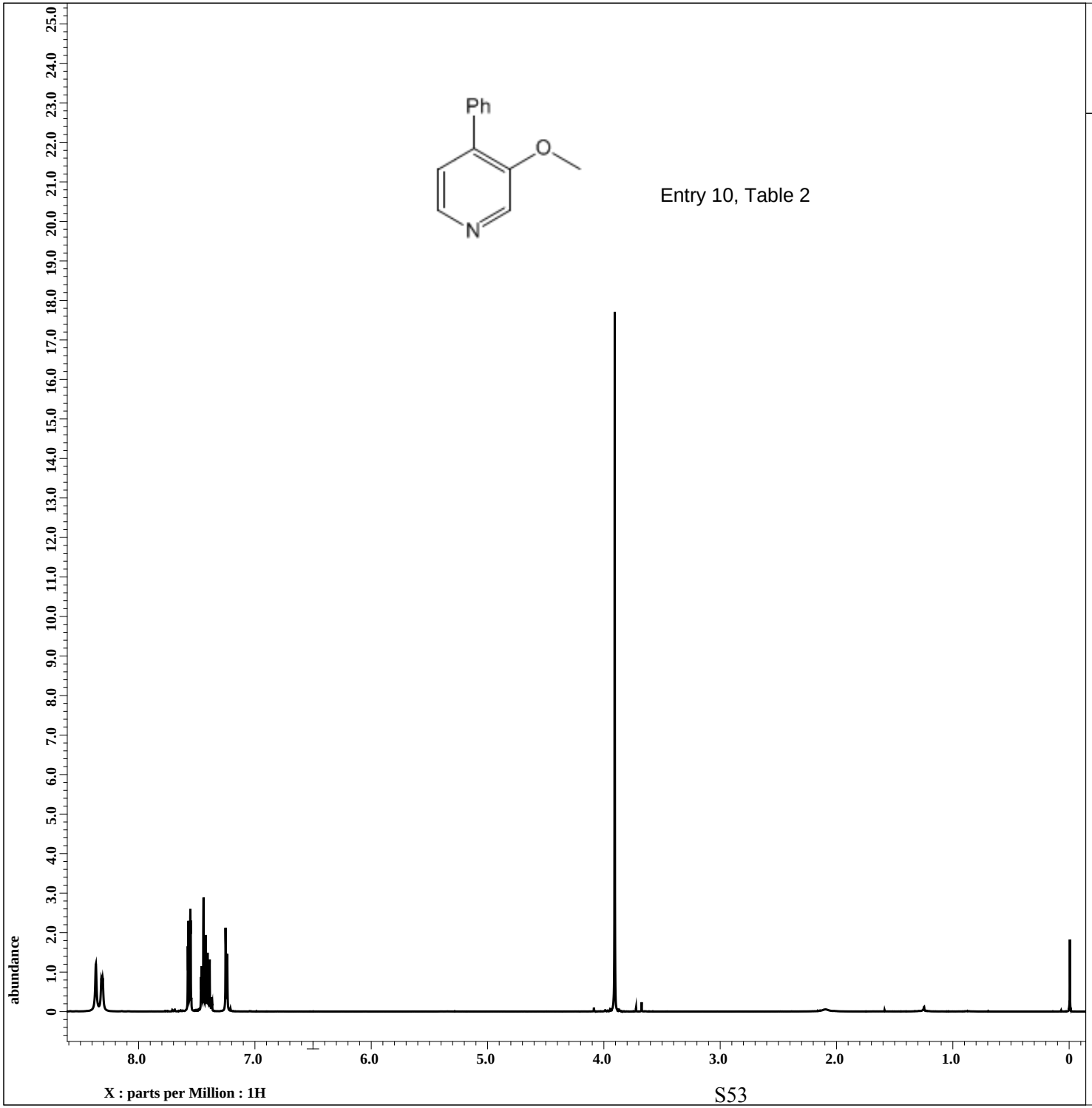
JEOL

```
Filename          = 1-137-5-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 1-137-5-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 13:45:37
Revision_time     = 18-DEC-2010 14:38:14
Current_time      = 18-DEC-2010 14:38:35

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.4[dC]
```

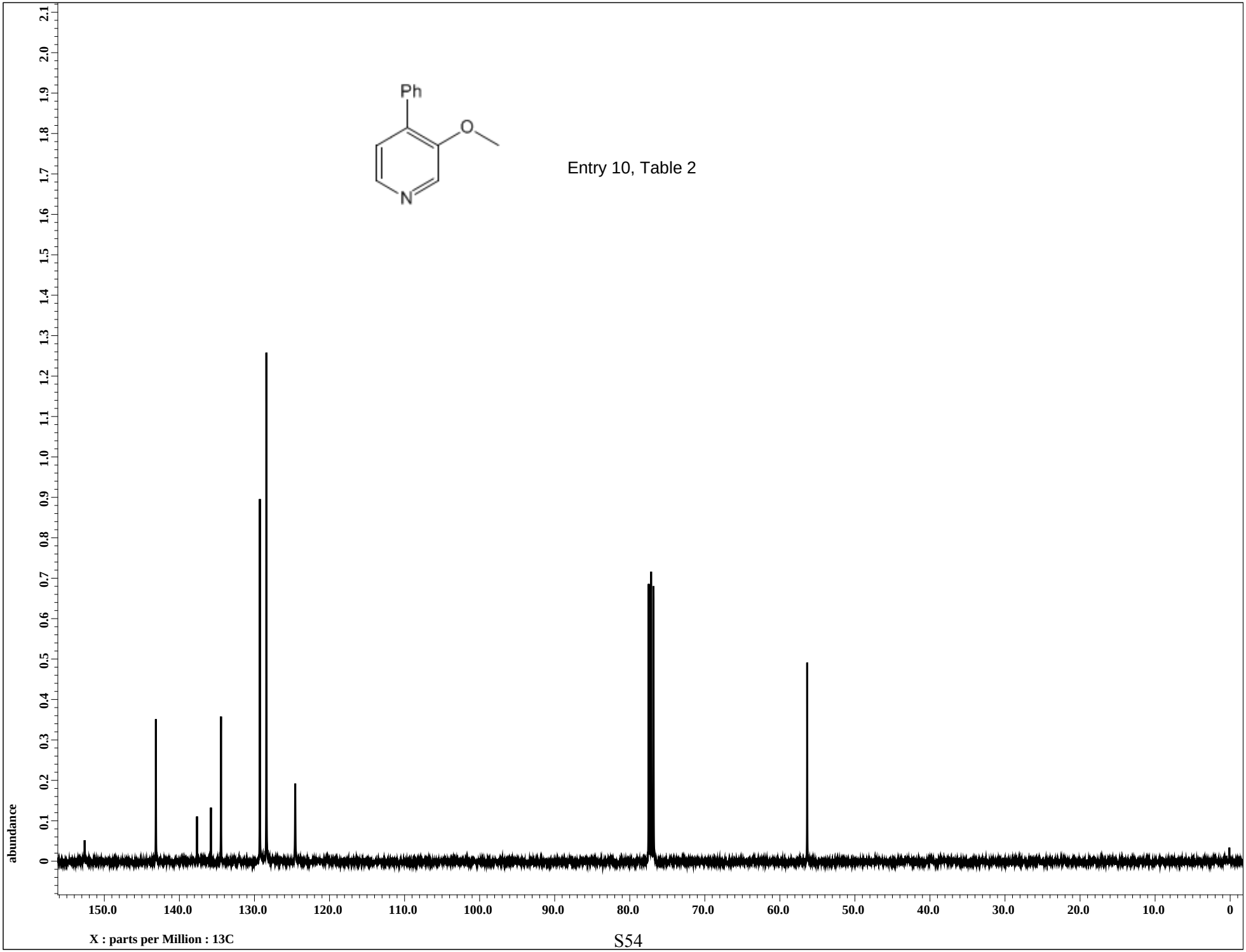


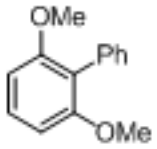

```
Filename          = 1-97-6-h1-2-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 1-97-6-h1-2
Solvent           = CHLOROFORM-D
Creation_time     = 24-NOV-2010 13:25:21
Revision_time     = 18-DEC-2010 14:33:52
Current_time      = 18-DEC-2010 14:34:28

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 36
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 18.9[dC]
```


Ph
O
N
Entry 10, Table 2
abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1.9
2.0
2.1
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : 13C



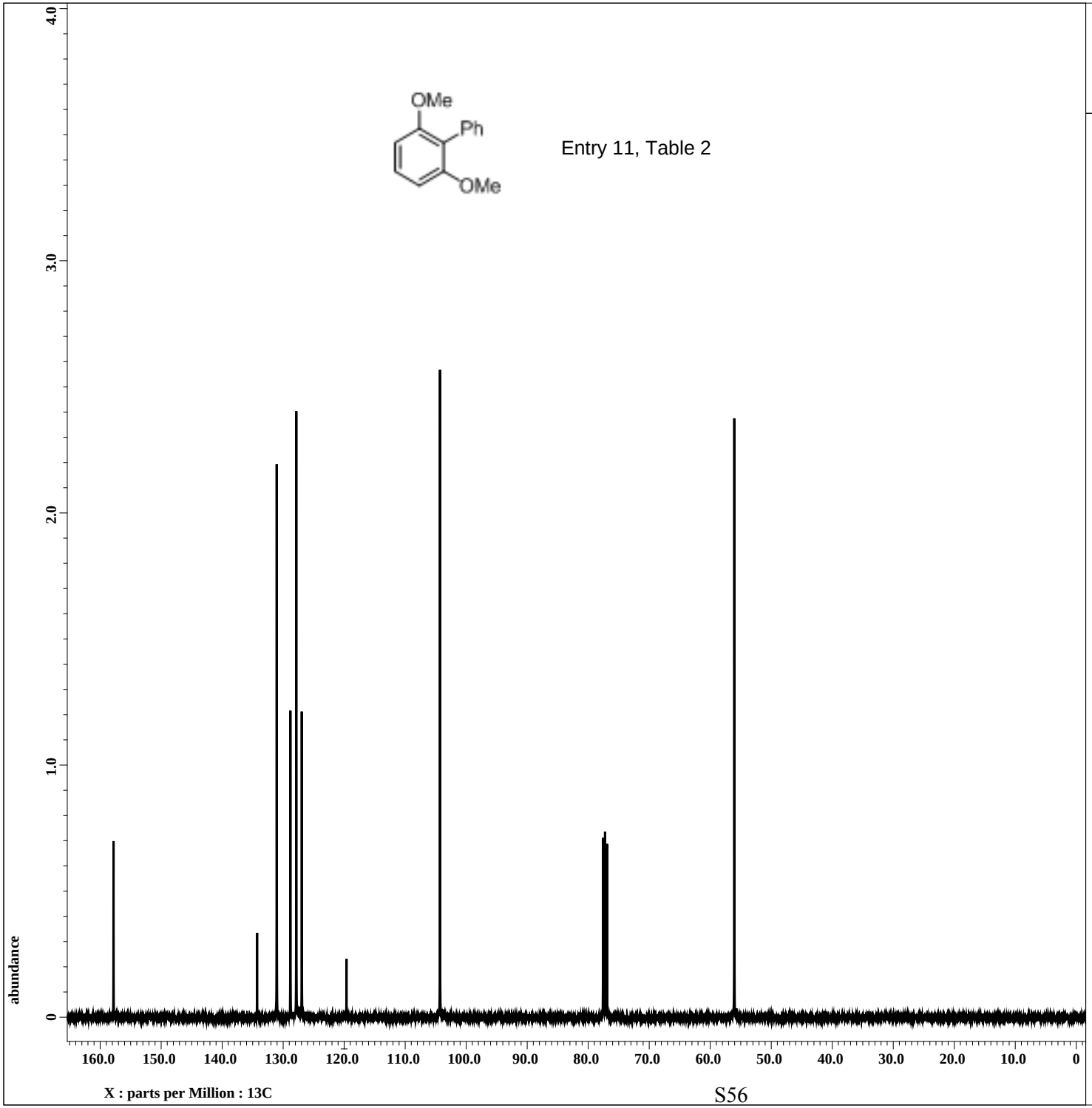
Entry 11, Table 2


```
Filename          = 4-101-6 h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 4-101-6-h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 10:21:11
Revision_time     = 18-DEC-2010 14:58:30
Current_time      = 18-DEC-2010 14:59:20

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19[dC]
```

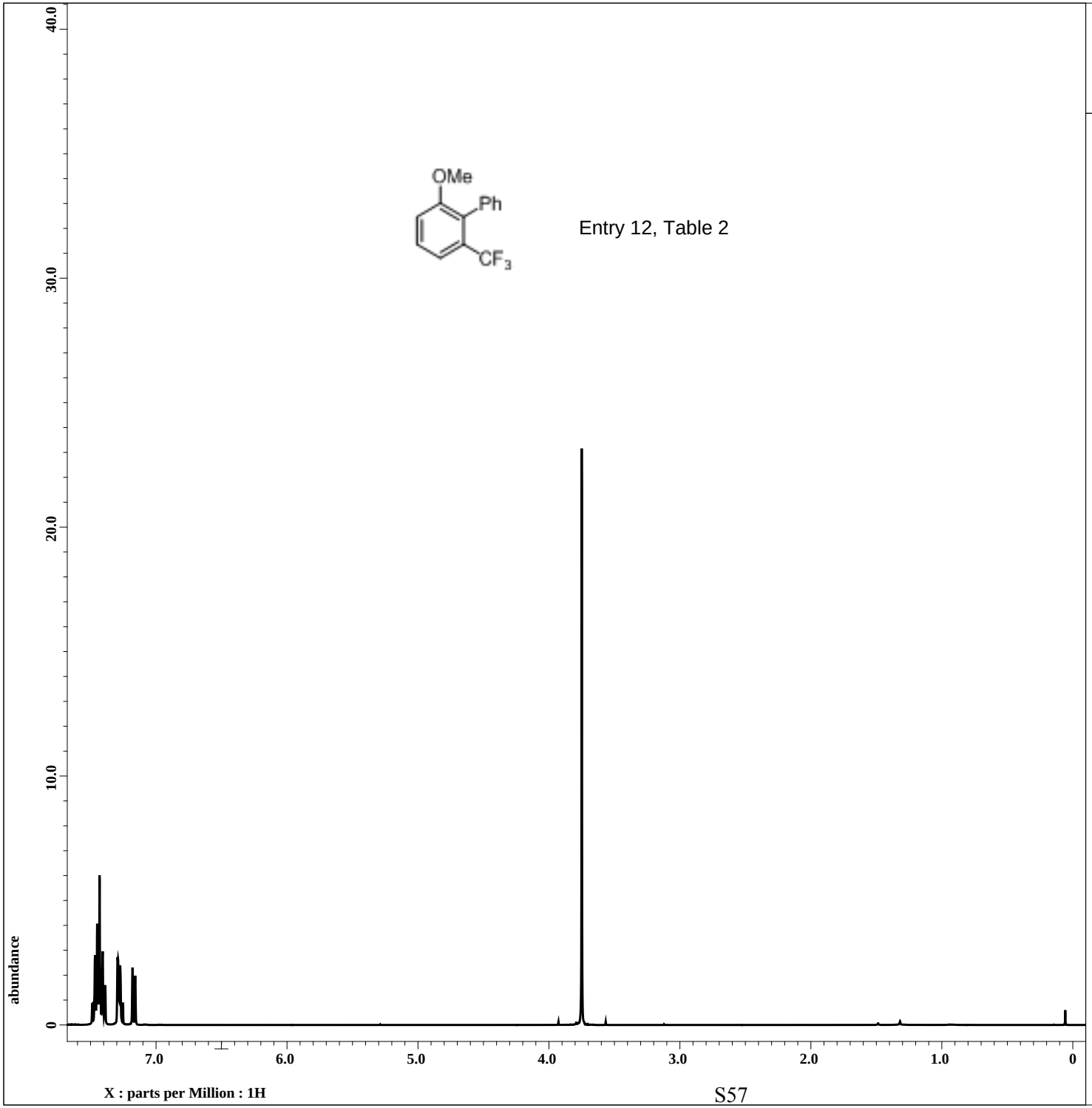



```
Filename          = 4-101-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 4-101-6-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 10:39:21
Revision_time     = 18-DEC-2010 14:59:27
Current_time      = 18-DEC-2010 14:59:46

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = TRUE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.3[dC]
```

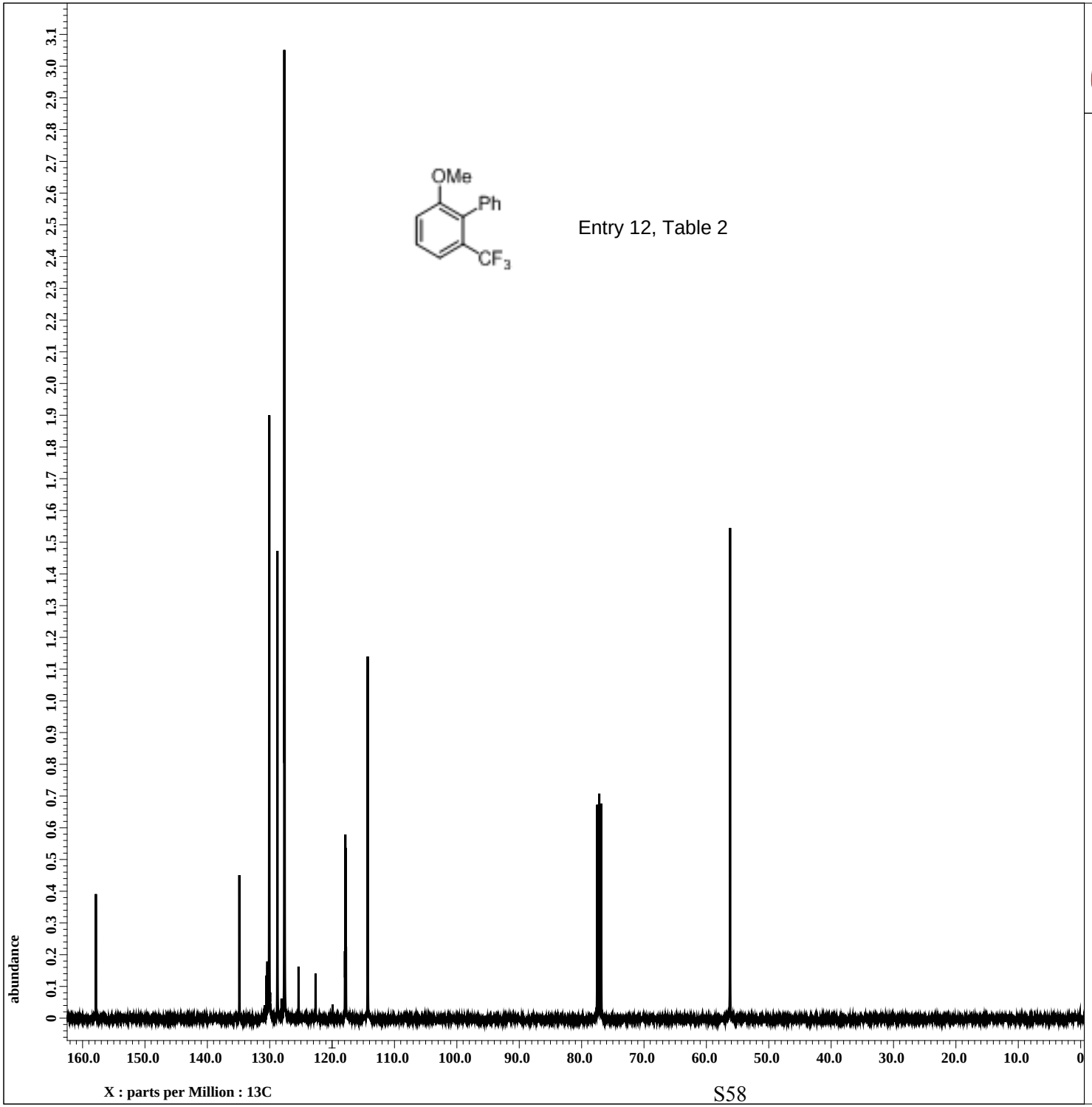



```
Filename          = 4-117-6-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 4-117-6-h1
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 09:54:26
Revision_time     = 18-DEC-2010 14:59:58
Current_time      = 18-DEC-2010 15:00:15

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 18.7[dC]
```

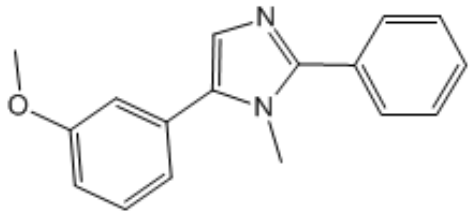



```
Filename          = 4-117-6-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 4-117-6-c13
Solvent           = CHLOROFORM-D
Creation_time     = 16-NOV-2010 10:12:47
Revision_time     = 18-DEC-2010 15:00:21
Current_time      = 18-DEC-2010 15:00:40

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.7[dC]
```



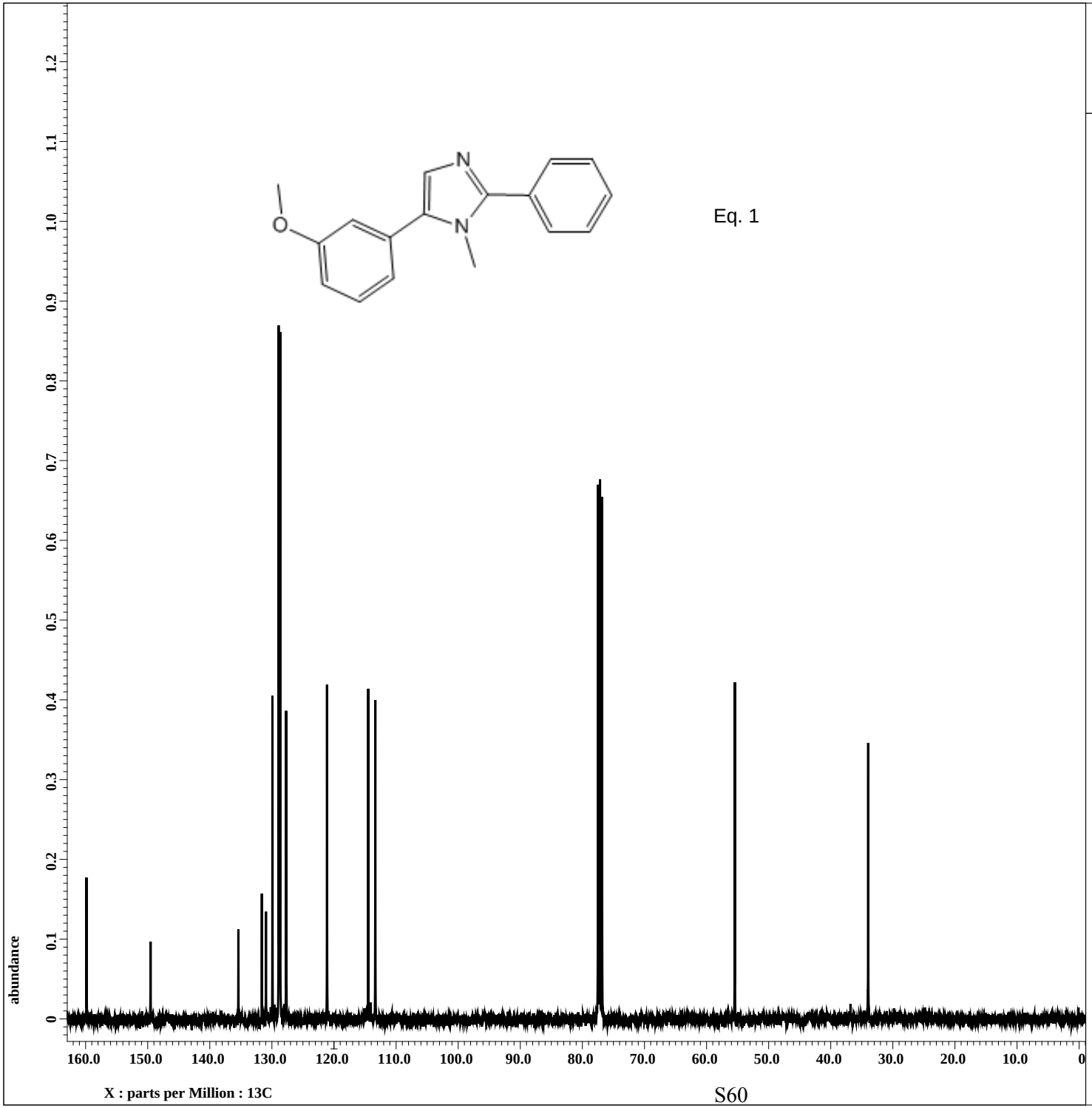
Eq. 1


```
Filename          = 3-99-7-h1-2.jdf
Author            = daugulis
Experiment        = single_pulse.ex2
Sample_id         = 3-99-7-h1
Solvent           = CHLOROFORM-D
Creation_time     = 18-DEC-2010 10:51:14
Revision_time     = 18-DEC-2010 14:47:56
Current_time      = 18-DEC-2010 14:48:17

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 2.18365952[s]
X_domain          = 1H
X_freq            = 399.78219838[MHz]
X_offset          = 6.5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45794685[Hz]
X_sweep           = 7.5030012[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 399.78219838[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 12.56[us]
X_acq_time        = 2.18365952[s]
X_angle           = 45[deg]
X_atn             = 3[dB]
X_pulse           = 6.28[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 1[s]
Repetition_time   = 3.18365952[s]
Temp_get          = 19.4[dC]
```




```
Filename          = 3-99-7-c13-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 3-99-7-c13
Solvent           = CHLOROFORM-D
Creation_time     = 18-DEC-2010 11:09:44
Revision_time     = 18-DEC-2010 14:48:23
Current_time      = 18-DEC-2010 14:48:47

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.389766[T] (400[MHz]
X_acq_duration    = 1.04333312[s]
X_domain          = 13C
X_freq            = 100.52530333[MHz]
X_offset          = 120[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95846665[Hz]
X_sweep           = 31.40703518[kHz]
Irr_domain        = 1H
Irr_freq          = 399.78219838[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 512
Total_scans       = 512

X_90_width        = 12.4525[us]
X_acq_time        = 1.04333312[s]
X_angle           = 30[deg]
X_atn             = 6[dB]
X_pulse           = 4.15083333[us]
Irr_atn_dec       = 22[dB]
Irr_atn_noe       = 22[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 1[s]
Recvr_gain        = 60
Relaxation_delay  = 1[s]
Repetition_time   = 2.04333312[s]
Temp_get          = 19.8[dC]
```