

SUPPORTING INFORMATION

**Copper-Catalyzed Carboxylation of Aryl Iodides
with Carbon Dioxide**

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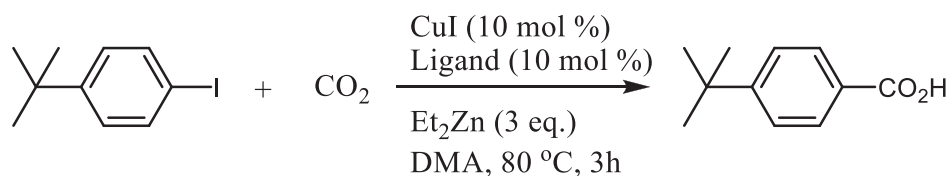
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Experimental Section

General considerations. Reactions were performed in 4-dram vials with PTFE/Liner caps (from VWR or Chemglass). Flash chromatography was performed on 60Å silica gel (Sorbent Technologies). GC analyses were performed on a Shimadzu GC-2010 chromatograph equipped with a Restek column (Rtx®-5, 15m, 0.25 mm I.D.). The ^1H , ^{19}F and ^{13}C NMR spectra were recorded on a JEOL ECX-400 or ECA-500 spectrometers using TMS as a reference. *For fluorine-containing compounds, ^{13}C - ^{19}F couplings were assigned where possible; otherwise list of peaks is reported.* The GC yields were calculated based on an internal standard (dodecane). TEM was performed on JEOL 2000 FX microscope equipped with energy dispersive spectrometer, and operated at 200 kV.

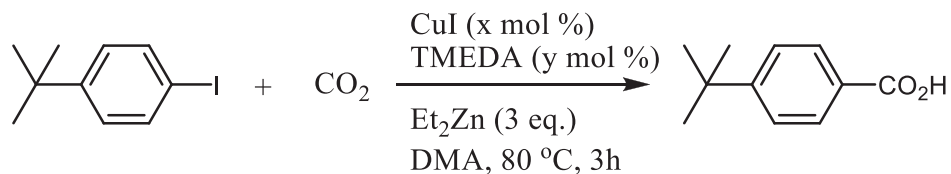
Materials. The following starting materials were obtained from commercial sources and were used without further purification: 1-tert-butyl-4-iodobenzene, 5-iodo-m-xylene, 2-iodothiophene, 1-chloro-4-iodobenzene, 1-bromo-4-iodobenzene, 1-bromo-3-iodobenzene, ethyl 4-iodobenzoate, 1-fluoro-4-iodobenzene, 1-fluoro-3-iodobenzene, and 3-iodobenzotrifluoride were bought from Oakwood. Iodobenzene, 1-iodonaphthalene, 4-iodoanisole, and copper (I) iodide were obtained from Acros. 2-Iodotoluene, 3-iodoanisole, *N,N'*-dimethylethylenediamine (DMEDA), and 4-iodophenol were purchased from Aldrich. 2-Iodo-1,3,5-trimethylbenzene, 5-iodoindole, and *N,N,N',N'*-tetramethylethylenediamine (TMEDA) were from Alfa Aesar. 4-Iodo-benzophenone was obtained from Matrix Scientific. Anhydrous dimethyl sulfoxide (DMSO), and *N,N*-dimethylacetamide (DMA) were bought from Sigma-Aldrich. Diethyl zinc was from Strem.

Optimization of ligands for carboxylation of 4-tert-Butyl iodobenzene: GC yields were used to determine the suitable ligands for the reaction. Method for determination of GC yields is given in the General procedure (see below). The results were presented in the Table S1.

Table S1. Ligand screen

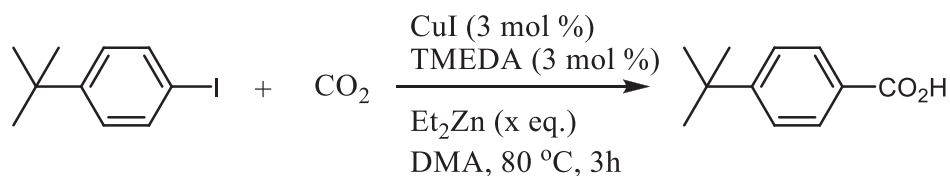
Ligand	GC Yield (%)
Ph ₃ P	44
Cy ₃ P	43
DIPHOS	23
1,10-Phen	73
BathoPhen	56
TMEDA	79
DMEDA	82
2,2-Dipy	70
trans-1,2-diamino cyclohexane	67

Optimization of ligand and catalyst amounts for carboxylation of 4-tert-butyl iodobenzene: The results for the optimization are presented in Table S2.

Table S2. Ligand and catalyst amount screen

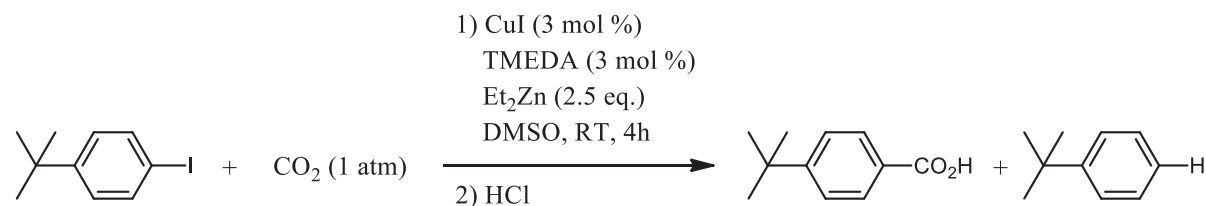
% mol Cu	% mol Ligand	GC Yield (%)
0	0	<2
0	10	<2
10	0	46
10	10	75
5	5	78
5	10	76
3	3	83

Optimization of diethyl zinc amount for carboxylation of 4-tert-butyl iodobenzene: Table S3 shows optimization of diethyl zinc amount.

Table S3. Diethyl zinc amount screen

Eq. of Et ₂ Zn	GC Yield (%)
0	<2
1.0	30
1.5	42
2.0	47
2.5	83
3.0	84
4.0	85

Control experiments: The control reactions are presented in Table S4. All the yields were determined by GC.

Table S4. Control experiments

Reaction conditions	ArI (%)	Ar-CO ₂ H (%)	Ar-H (%)
Standard	< 2	82	16
Without CO ₂	18	<2	79
Without CO ₂ , 1eq. CuI	16	<2	82
Without CO ₂ , without CuI	97	<2	<2
Using Cu(0) instead of CuI	98	<2	<2

Cu(0) is nano Cu powder (40-60 nm particle size) purchased from Sigma-Aldrich.

Propanoic acid and 4-ethyl-t-butyl benzene were not observed in the reaction mixture by GC and GC-MS.

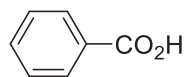
General procedure for Carboxylation of Aryl Iodides with CO₂. Outside the glovebox a 4-dram vial (20 mL) equipped with a magnetic stir bar was charged with TMEDA (3 mol%)

or DMEDA (3 mol%), CuI (3 mol %), and aryl iodide substrate. The vial was flushed with nitrogen, capped and placed inside glovebox. To this mixture was first added Et₂Zn (2.5 equiv) followed by solvent (4 mL; DMSO or DMA). **Caution:** *keeping the correct order of chemical addition is essential for achieving good yields. If the order of addition is changed, yields will be lower.* The sealed vial was then taken out of the glovebox and dry CO₂ gas was introduced to the reaction via needle. The vial was flushed with CO₂ for 30 seconds then stirred at room temperature for five minutes, warmed up, and heated for the indicated time. **Caution:** *attention should be paid for making sure that the CO₂ gas is continuously introduced into reaction vial during the course of reaction.* After the completion of the reaction, the mixture was cooled to room temperature, carefully quenched with aq. HCl (2 N) until pH = 1 to hydrolyze the resulting carboxylate, followed by addition of brine (3 mL). The mixture was extracted with AcOEt (4 x 20 mL). The combined organic extracts were dried over anhydrous MgSO₄, concentrated, and diluted with 3 mL toluene (3 mL) followed by column chromatography on silica gel in hexanes followed by appropriate solvent to elute the products. After concentrating the fractions containing the product, the residue was dried under reduced pressure to yield pure product.

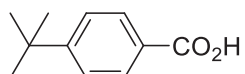
For determination of GC yields, response factors for 4-tert-butylbenzoic acid with respect to internal standard dodecane were determined as follows.

Mixtures of dodecane (45 mg, 0.25 mmol) with different amounts of 4-tert-butylbenzoic acid (44.5 mg, 0.25 mmol; 89 mg, 0.5 mmol; 133.5 mg, 0.75 mmol; and 178 mg, 1 mmol) were prepared by using 7 mL of ethyl acetate as the solvent. These mixtures were analyzed by GC and four values of response factor were determined from the ratio of analyte and dodecane standard peak areas. Four values of this response factor were found to be similar and the final response factor used was the average of these values.

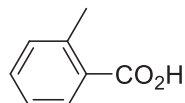
The reaction mixtures were acidified by aq. HCl (2 N) until pH=2 followed by addition of dodecane (45 mg, 0.25 mmol) as the internal standard. Mixtures were the diluted with ethyl acetate for GC analysis. The amounts of the products formed were calculated from the peak area of dodecane, product, and the response factor obtained previously. All GC yields are the average of at least two runs.



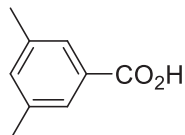
Benzoic acid (Entry 1, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), iodobenzene (204 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL), RT, 4h. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes), 107 mg (88%) of a white solid was obtained. This compound is known.¹ ¹H NMR (400 MHz, DMSO-d₆) δ 7.44-7.48 (m, 2H), 7.56-7.60 (m, 1H), 7.90-7.92 (m, 2H), 12.94 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆) δ 129.1, 129.8, 131.3, 133.4, 167.8.



4-tert-Butylbenzoic acid (Entry 2, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 4-tert-butyl iodobenzene (260 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL), RT, 5h. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes), 144 mg (81%) of a white solid was obtained. This compound is known.² ¹H NMR (500 MHz, DMSO-d₆) δ 1.25 (s, 9H), 7.47 (d, J = 8.5 Hz, 2H), 7.84 (d, J = 8.5, 2H), 12.77 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 31.4, 35.3, 125.9, 128.6, 129.7, 156.3, 167.8.

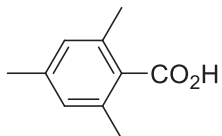


2-Methylbenzoic acid (Entry 3, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), DMEDA (2.7 mg, 0.03 mmol), 2-iodotoluene (218 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL), RT, 5h. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes), 117 mg (86%) of a white solid was obtained. This compound is known.¹ ¹H NMR (500 MHz, DMSO-d₆) δ 2.48 (s, 3H), 7.23-7.26 (m, 2H), 7.39-7.42 (m, 1H), 7.77-7.78 (m, 1H), 12.78 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 21.8, 126.4, 130.7, 130.9, 132.0, 132.2, 139.5, 169.2.

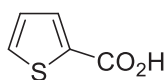


3,5-Dimethylbenzoic acid (Entry 4, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), DMEDA (2.7 mg, 0.03 mmol), 5-iodo-*m*-xylene (232 mg, 1 mmol), diethyl zinc (309 mg, 2.5

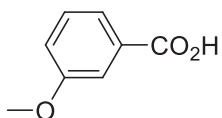
mmol), and DMSO (4 mL), RT, 5h. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes), 120 mg (80%) of a white solid was obtained. This compound is known.³ ¹H NMR (400 MHz, DMSO-d₆) δ 2.28 (s, 6H), 7.20 (s, 1H), 7.52 (s, 2H), 12.76 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆) δ 21.2, 127.5, 131.2, 134.7, 138.2, 168.0.



2,4,6-Trimethylbenzoic acid (Entry 5, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), DMEDA (2.7 mg, 0.03 mmol), 2-iodo-1,3,5-trimethylbenzene (246 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL), RT, 5h. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6, 1/1 and 6/4 ethyl acetate/hexanes), 100 mg (61%) of a white solid was obtained. This compound is known.² ¹H NMR (500 MHz, DMSO-d₆) δ 2.19 (s, 9H), 6.84 (s, 2H), 12.92 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 19.8, 21.2, 128.5, 133.0, 134.2, 138.5, 171.4.

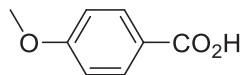


2-Thiophenecarboxylic acid (Entry 6, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 2-iodothiophene (210 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL), RT, 5 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 81 mg (63%) of a yellow solid was obtained. This compound is known.² ¹H NMR (500 MHz, DMSO-d₆) δ 7.15 (dd, J = 5.1, 4.0 Hz, 1H), 7.69 (dd, J=4.0, 1.4 Hz, 1H), 7.85 (dd, J=4.7, 1.2 Hz, 1H), 13.04 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 128.8, 133.7, 133.8, 135.2, 163.5.

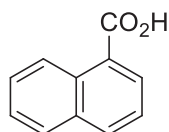


3-Methoxybenzoic acid (Entry 7, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), DMEDA (2.7 mg, 0.03 mmol), 3-iodoanisole (234 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 117 mg (77%) of a light yellow solid was obtained. This compound is known.⁴ ¹H NMR (500

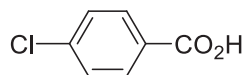
MHz, DMSO- d_6) δ 3.77 (s, 3H), 7.15 (dd, J = 8.0, 2.3 Hz, 1H), 7.36-7.40 (m, 2H), 7.50 (d, J = 8.0, 1H), 12.98 (s, 1H). ^{13}C NMR (125 MHz, DMSO- d_6) δ 55.8, 114.4, 119.4, 122.1, 130.2, 132.7, 159.7, 167.7.



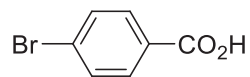
4-Methoxybenzoic acid (Entry 8, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 4-iodoanisole (234 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 91 mg (60%) of a white solid was obtained. This compound is known.¹ ^1H NMR (500 MHz, DMSO- d_6) δ 3.79 (s, 3H), 6.98 (d, J = 9.1, 2H), 7.86 (d, J = 9.1, 2H), 12.60 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 55.9, 114.3, 123.5, 131.9, 163.4, 167.5.



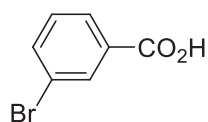
1-Naphthalenecarboxylic acid (Entry 9, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), DMEDA (2.7 mg, 0.03 mmol), 1-iodonaphthalene (254 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes), 117 mg (68%) of a white solid was obtained. This compound is known.³ ^1H NMR (500 MHz, DMSO- d_6) δ 7.54-7.57 (m, 2H), 7.59-7.63 (m, 1H), 7.99 (d, J =8.0 Hz, 1H), 8.11-8.14 (m, 2H), 8.83 (d, J =8.6 Hz, 1H), 13.13 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 125.4, 126.0, 126.5, 128.1, 128.2, 129.2, 130.4, 131.2, 133.5, 134.0, 169.2.



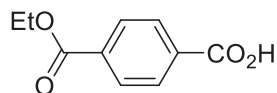
4-Chlorobenzoic acid (Entry 10, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 1-chloro-4-iodobenzene (238.5 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 95 mg (61%) of a white solid was obtained. This compound is known.⁴ ^1H NMR (500 MHz, DMSO- d_6) δ 7.54 (d, J = 8.6 Hz, 2H), 7.90 (d, J = 8.6, 2H), 13.17 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 129.3, 130.2, 131.7, 138.3, 167.0.



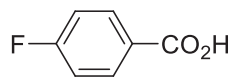
4-Bromo benzoic acid (Entry 11, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 1-bromo-4-iodobenzene (283 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, and 4/6 ethyl acetate/hexanes containing 0.5% acetic acid), 119 mg (59%) of a white solid was obtained. This compound is known.¹ ¹H NMR (500 MHz, DMSO-d₆) δ 7.66 (d, J = 8.3 Hz, 2H), 7.82 (d, J = 8.3, 2H), 13.16 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 127.4, 130.5, 131.8, 132.2, 167.2.



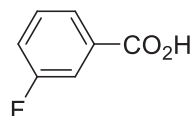
3-Bromobenzoic acid (Entry 12, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 1-bromo-3-iodobenzene (283 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, and 4/6 ethyl acetate/hexanes containing 0.5% acetic acid), 102 mg (51%) of a white solid was obtained. This compound is known.⁵ ¹H NMR (500 MHz, DMSO-d₆) δ 7.44 (t, J = 8.0 Hz, 1H), 7.80 (dd, J = 8.0, 1.7 Hz, 1H), 7.90 (dt, J = 8.0, 1.5, 1H), 8.00 (t, J = 1.7 Hz, 1H), 13.32 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 122.3, 128.8, 131.5, 132.3, 133.6, 136.1, 166.5.



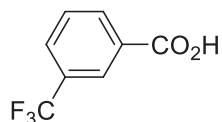
Monoethyl terephthalate (Entry 13, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), ethyl 4-iodobenzoate (276 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 120 mg (62%) of a white solid was obtained. This compound is known.¹ ¹H NMR (500 MHz, DMSO-d₆) δ 1.29 (t, J = 6.7, 3H), 4.29 (q, J = 6.7, 2H), 8.01 (s, 1H), 13.33 (s, 1H). ¹³C NMR (125 MHz, DMSO-d₆) δ 14.6, 61.7, 129.8, 130.1, 133.9, 135.3, 165.6, 167.1.



4-Fluorobenzoic acid (Entry 14, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 1-fluoro-4-iodobenzene (222 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 84.1 mg (60%) of a light yellow solid was obtained. This compound is known.² ¹H NMR (400 MHz, DMSO-d₆) δ 7.29 (t, J = 8.7 Hz, 2H), 7.96 (dd, J = 8.7, 5.6 Hz, 2H), 13.04 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆) δ 116.2 (d, J_{C-F} = 22.1 Hz), 127.9, 132.6 (d, J_{C-F} = 9.5 Hz), 165.4 (d, J_{C-F} = 250.6 Hz), 166.9.

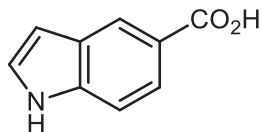


3-Fluorobenzoic acid (Entry 15, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 1-fluoro-3-iodobenzene (222 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMSO (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6 and 1/1 ethyl acetate/hexanes containing 0.5% acetic acid), 77 mg (55%) of a light yellow solid was obtained. This compound is known.⁶ ¹H NMR (500 MHz, DMSO-d₆) δ 7.46 (tdd, J = 8.5, 3.0, 1.1 Hz, 1H), 7.51-7.55 (m, 1H), 7.62 (dq, J = 9.7, 1.4 Hz, 1H), 7.75 (dt, J = 7.5, 1.1 Hz, 1H), 13.27 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆; C-F couplings assigned where possible; otherwise, list of signals) δ 116.3 (d, J_{C-F} = 22.1 Hz), 120.4 (d, J_{C-F} = 21.1 Hz), 125.9, 131.3, 131.4, 133.8, 161.3, 163.7, 166.7.

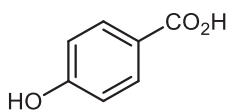


3-Trifluoromethylbenzoic acid (Entry 16, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 3-iodobenzotrifluoride (272 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7 and 4/6 ethyl acetate/hexanes containing 0.5% acetic acid), 106 mg (56%) of a white solid was obtained. This compound is known.³ ¹H NMR (500 MHz, DMSO-d₆) δ 7.73 (t, J = 7.5, 1H), 7.97 (t, J = 7.6, 1H), 8.14 (s, 1H), 8.19 (d, J = 7.5, 1H), 13.49 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆; C-F couplings assigned

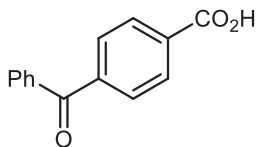
where possible; otherwise, list of signals) δ 124.2 (q, $J_{C-F} = 274.2$ Hz), 126.0, 129.4, 129.8, 130.1, 130.4, 132.4, 133.7, 166.5.



Indole-5-carboxylic acid (Entry 17, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 5-iodoindole (243 mg, 1 mmol), diethyl zinc (425 mg, 3.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6, 5/5, 6/4, 7/3 and 8/2 ethyl acetate/hexanes containing 0.5% acetic acid), 71 mg (44 %) of a dark brown solid was obtained. This compound is known.⁷ ^1H NMR (400 MHz, DMSO- d_6) δ 6.52-6.55 (m, 1H), 7.36-7.42 (m, 2H), 7.67 (dd, $J = 7.6, 1.4$, 1H), 8.20 (s, 1H), 11.40 (s, 1H), 12.37 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 103.0, 111.6, 121.9, 122.7, 123.3, 127.4, 127.7, 138.8, 168.9. Unreacted starting material was also recovered by column chromatography (96 mg, 40%).



4-Hydroxybenzoic acid (Entry 18, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 4-iodophenol (220 mg, 1 mmol), diethyl zinc (425 mg, 3.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6, 5/5, 6/4, 7/3 and 8/2 ethyl acetate/hexanes containing 0.5% acetic acid), 56 mg (40 %) of a brown solid was obtained. This compound is known.⁸ ^1H NMR (400 MHz, DMSO- d_6) δ 6.78 (d, $J = 7.3$, 2H), 7.74(d, $J = 7.3$, 2H), 10.19 (s, 1H), 12.37 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 115.6, 121.8, 132.0, 162.1, 167.7. Unreacted starting material was also recovered by column chromatography (132 mg, 60%).

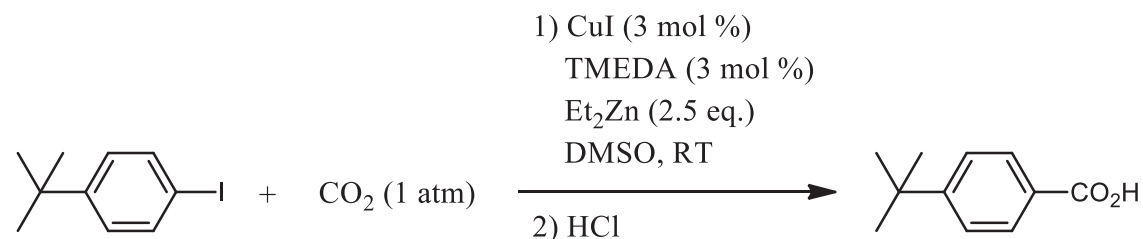


Benzophenone-4-carboxylic acid (Entry 19, Table 2): Copper(I) iodide (6.3 mg, 0.03 mmol), TMEDA (3.5 mg, 0.03 mmol), 4-iodobenzophenone (308 mg, 1 mmol), diethyl zinc (309 mg, 2.5 mmol), and DMA (4 mL) were stirred at 70 °C for 20 hours. After column chromatography (hexanes, 1/9 followed by 2/8, 3/7, 4/6, 5/5, and 6/4 ethyl acetate/hexanes

containing 0.5% acetic acid), 120.0 mg (53%) of a white solid was obtained. This compound is known.⁴ ¹H NMR (400 MHz, DMSO-d₆) δ 7.51-7.58 (m, 2H), 7.63-7.80 (m, 5H), 8.03-8.09 (m, 2H), 13.33 (s, 1H). ¹³C NMR (100 MHz, DMSO-d₆) δ 129.2, 129.9, 130.2, 130.3, 133.7, 134.6, 137.0, 141.0, 167.2, 195.9.

Mercury test. Outside the glovebox a 4-dram vial (20 mL) equipped with a magnetic stir bar was charged with TMEDA (3.5 mg, 0.03 mmol), CuI (6.3 mg, 0.03 mmol), and 4-tert-butyl iodobenzene (260 mg, 1 mmol). The vial was flushed with nitrogen, capped and placed inside glovebox. To this mixture was first added Et₂Zn (309 mg, 2.5 mmol) followed by anhydrous DMSO (4 mL). The sealed vial was then taken out of the glovebox and Hg (1204 mg, 6 mmol, 200 equiv relative to Cu or 3009 mg, 15 mmol, 500 equiv relative to Cu) was added. Subsequently, dry CO₂ gas was introduced into the reaction via needle. The vial was flushed with CO₂ for 30 seconds then stirred at room temperature for 3h. The reaction mixtures were acidified with aq. HCl (2N) until pH=2 followed by addition of dodecane (45 mg, 0.25 mmol) as the internal standard. Mixture was diluted with ethyl acetate for GC analysis. The yields of reaction are 24% and 10% for the samples with 200 equiv and 500 equiv Hg (with respect to Cu) instead of 79% in the absence of Hg.

Kinetic experiment



Reaction time (min)	GC Yield (%)
5	<2
10	<2
15	9
30	15
60	36
90	59
120	77

TEM Analysis. The sample was prepared according to the general procedure. After 1h, 0.1 ml and 0.05 ml of sample, separately, were taken out and diluted by 1 ml (Figure S1) and 5

ml (Figure S2) of methanol respectively. Five μL of these solutions were added to a TEM copper grid coated with ultrathin carbon film and allowed to dry. We analyzed samples by using JEOL 2000 FX microscope equipped with energy dispersive spectrometer, and operated at 200 kV.

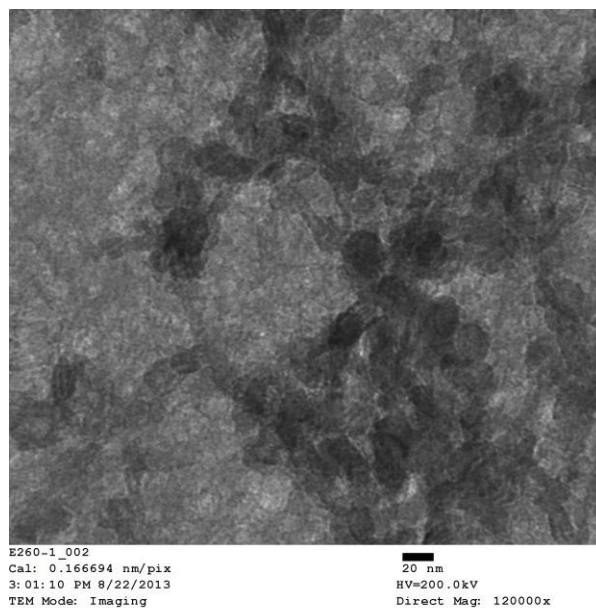


Figure S1

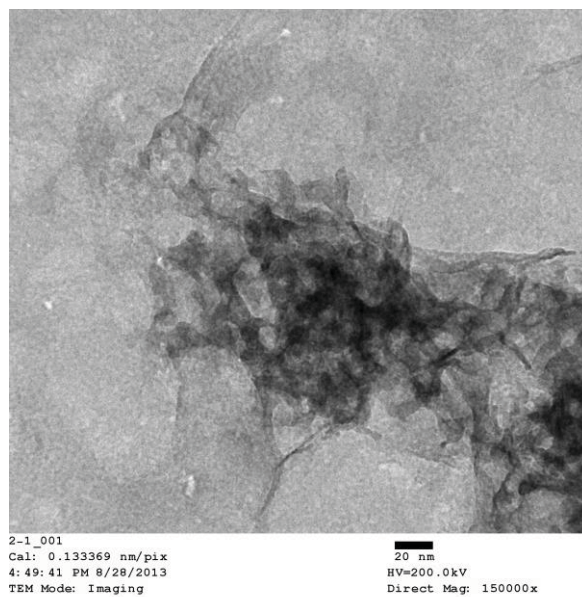


Figure S2

References

- (1) Kobayashi, K.; Kondo, Y. *Org. Lett.* **2009**, *11*, 2035.
- (2) Ohishi, T.; Nishiura, M.; Hou, Z. *Angew. Chem. Int. Ed. Eng.* **2008**, *47*, 5792.
- (3) Yang, D.; Yang, H.; Fu, H. *Chem. Commun.* **2011**, *47*, 2348.
- (4) Correa, A.; Martin, R. *J. Am. Chem. Soc.* **2009**, *131*, 15974.
- (5) Mukhopadhyay, C.; Datta, A. *Catal. Commun.* **2008**, *9*, 2588.
- (6) Shaikh, T. M.; Hong, F-E. *Adv. Synth. Catal.* **2011**, *353*, 1491.
- (7) Nirogi, R.V.S.; Kothmirkar, P.; Shinde, A. K.; Babu, J.; Sastry, K. R.; Dubey, P. K. *Der Pharma Chemica* **2011**, *3* (3), 221.
- (8) Zou, Y.-Q.; Chen, J.-R.; Liu, X.-P.; Lu, L.-Q.; Davis, R. L.; Jorgensen, K. A.; Xiao, W.-J. *Angew. Chem. Int. Ed. Eng.* **2012**, *51*, 784.

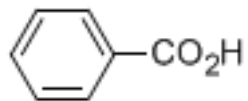
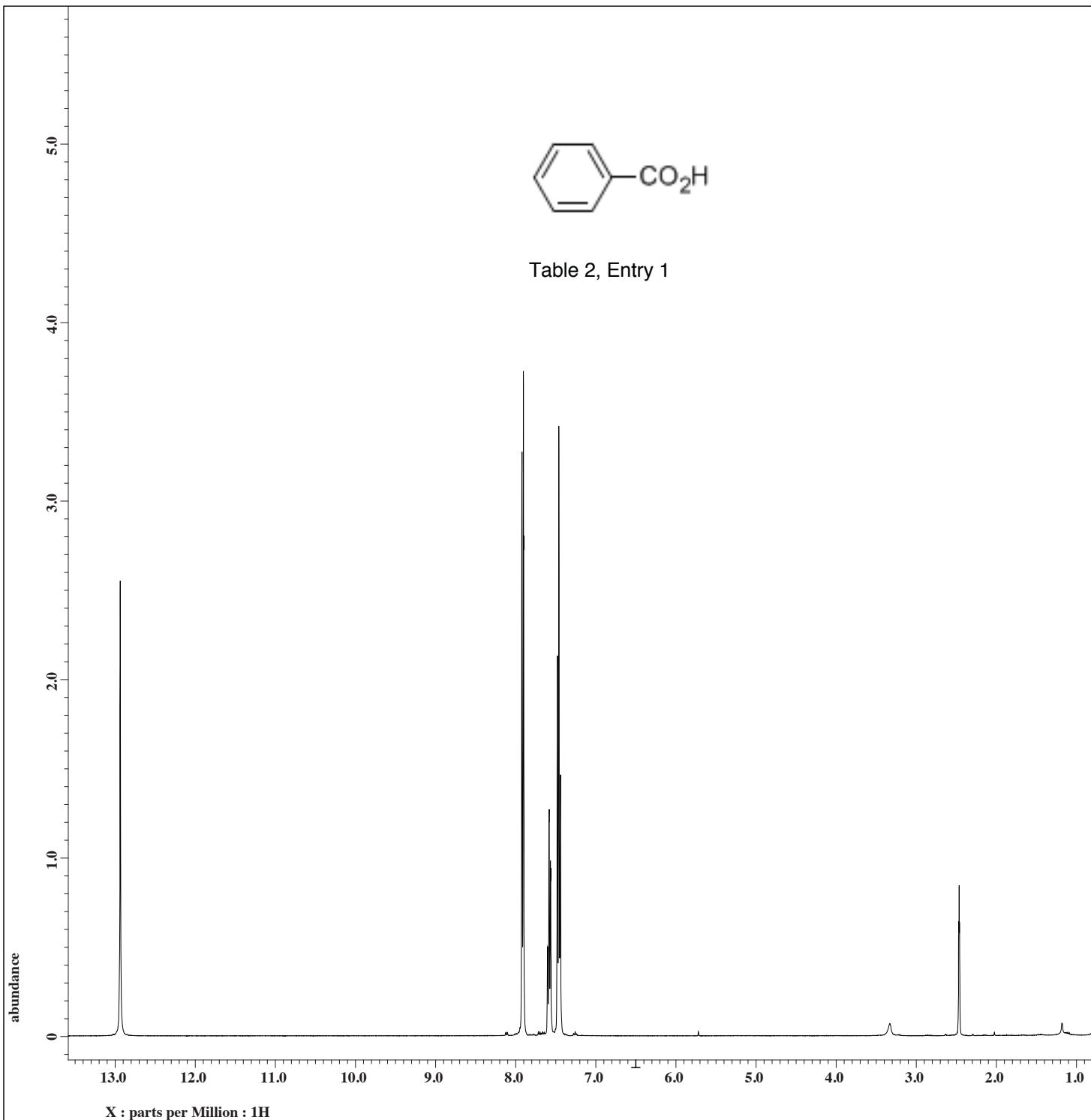


Table 2, Entry 1



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 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 15
 Solvent = DMSO-D6
 Creation_time = 6-APR-2012 15:36:17
 Revision_time = 22-MAY-2012 12:44:16
 Current_time = 22-MAY-2012 12:44:46

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 = 24
 Total_scans = 24

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 = 19.8[dc]

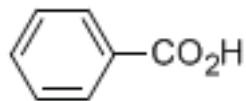
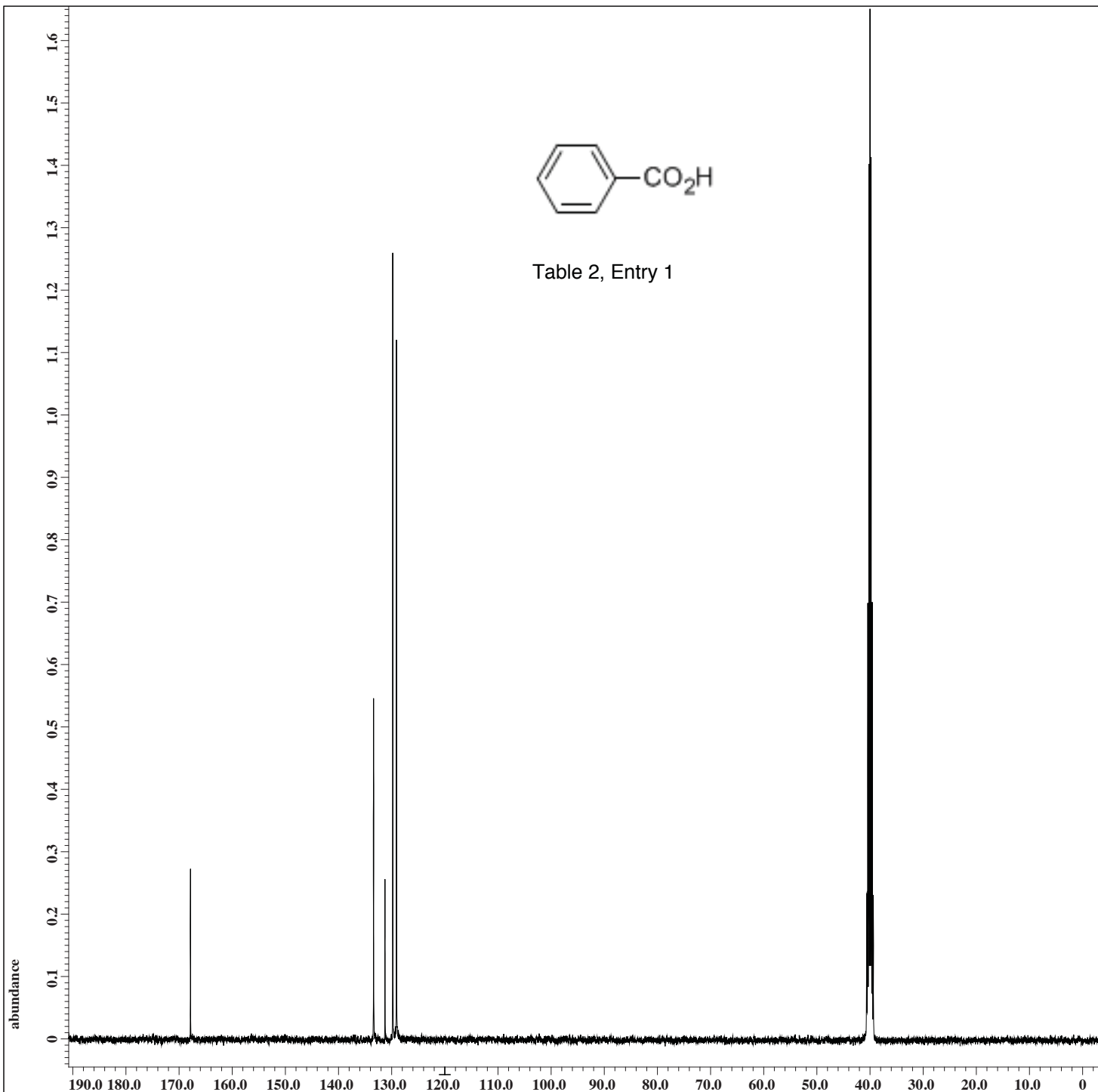


Table 2, Entry 1



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Experiment   = single_pulse_dec
Sample_id    = 15
Solvent      = DMSO-D6
Creation_time = 6-APR-2012 16:12:37
Revision_time = 22-MAY-2012 12:23:08
Current_time = 22-MAY-2012 12:27:21

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        = 1024
Total_scans  = 1024

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[degC]
    
```

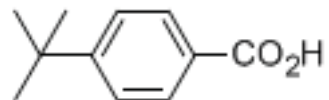
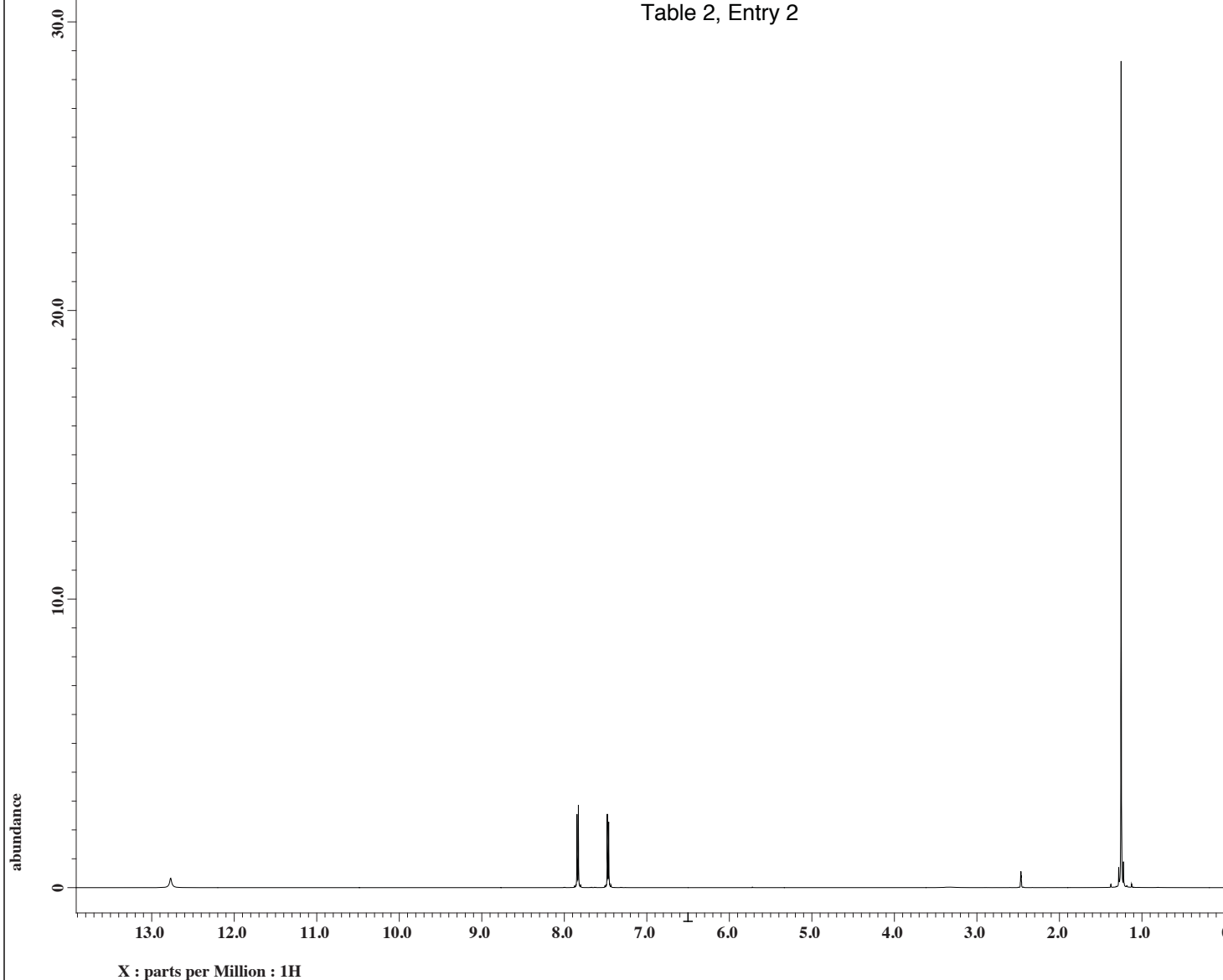


Table 2, Entry 2



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Filename      = 2-86-1-H-2.jdf
Author       = daugulis
Experiment    = single_pulse.ex2
Sample_id    = 16
Solvent      = DMSO-D6
Creation_time = 7-APR-2012 12:21:00
Revision_time = 22-MAY-2012 12:31:23
Current_time  = 22-MAY-2012 12:32:42
  
```

```

Comment      = single_pulse
Data_format   = 1D_COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = ECA500
  
```

```

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain      = 1H
X_freq       = 500.15991521[MHz]
X_offset     = 6.5[ppm]
X_points     = 16384
X_prescans   = 1
X_resolution = 0.57277737[Hz]
X_sweep      = 9.38438438[kHz]
Irr_domain   = 1H
Irr_freq     = 500.15991521[MHz]
Irr_offset   = 5.0[ppm]
Tri_domain   = 1H
Tri_freq     = 500.15991521[MHz]
Tri_offset   = 5.0[ppm]
Clipped     = FALSE
Mod_return   = 1
Scans       = 32
Total_scans  = 32
  
```

```

X_90_width   = 12.84[us]
X_acq_time   = 1.74587904[s]
X_angle      = 45[deg]
X_atn       = 3[dB]
X_pulse     = 6.42[us]
Irr_mode     = Off
Tri_mode     = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain   = 38
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get     = 19.9[dc]
  
```

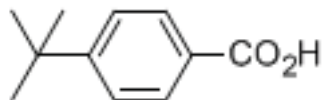
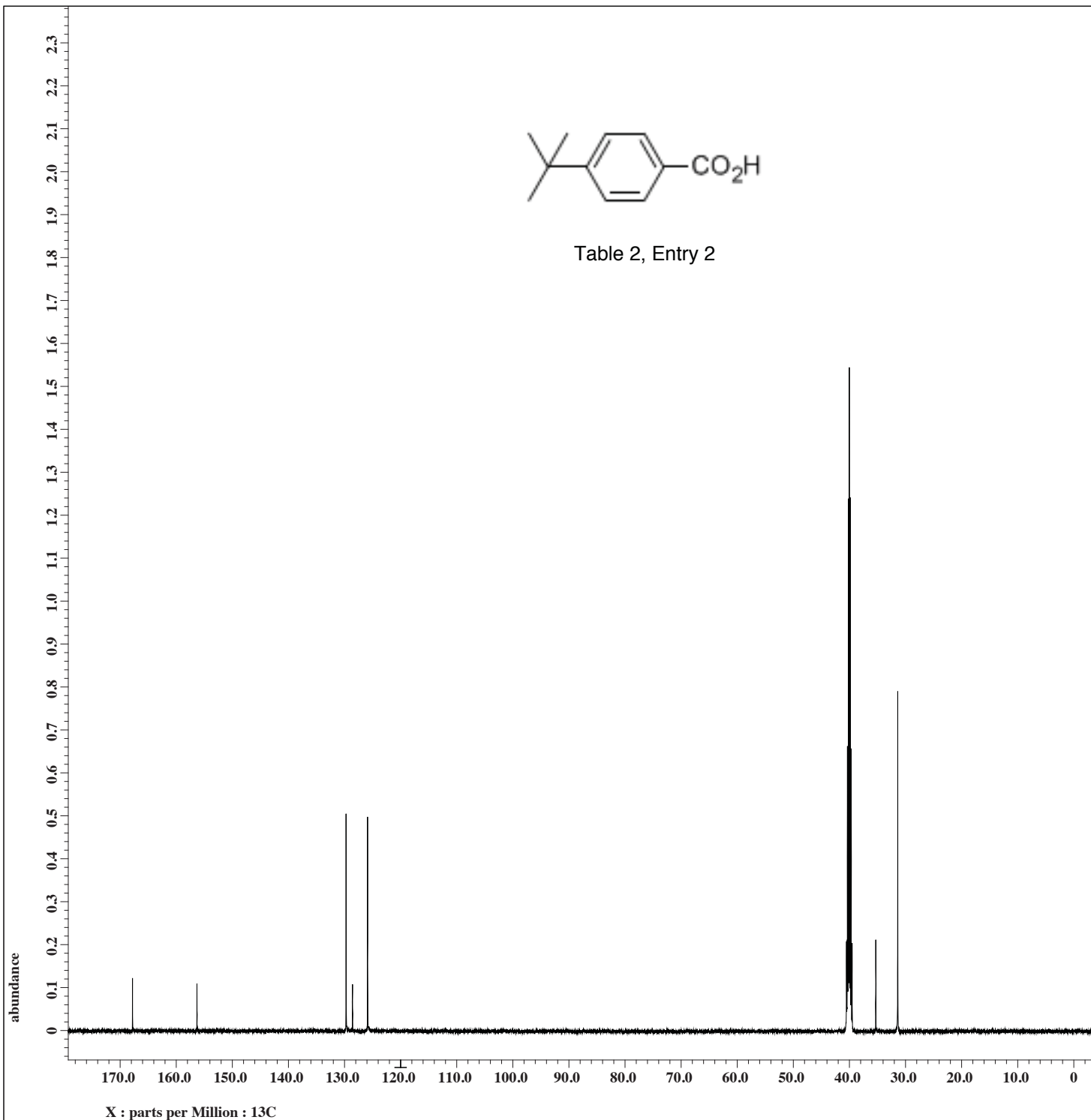


Table 2, Entry 2



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Filename      = 2-86-1-C-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 16
Solvent      = DMSO-D6
Creation_time = 7-APR-2012 13:08:30
Revision_time = 22-MAY-2012 12:33:09
Current_time  = 22-MAY-2012 12:33:56

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768[MHz]
X_offset      = 120[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521[MHz]
Irr_offset    = 5.0[ppm]
Clipped      = FALSE
Mod_return    = 1
Scans         = 1536
Total_scans   = 1536

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn        = 9[dB]
X_pulse      = 3.92333333[us]
Irr_atn_dec  = 19.5[dB]
Irr_atn_noe  = 19.5[dB]
Irr_noise    = WALTZ
Decoupling   = TRUE
Initial_wait = 1[s]
Noe          = TRUE
Noe_time     = 1[s]
Recvr_gain   = 58
Relaxation_delay = 1[s]
Repetition_time = 1.83361792[s]
Temp_get     = 20.6[dC]
    
```

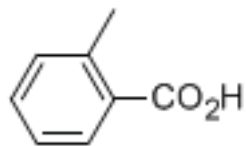
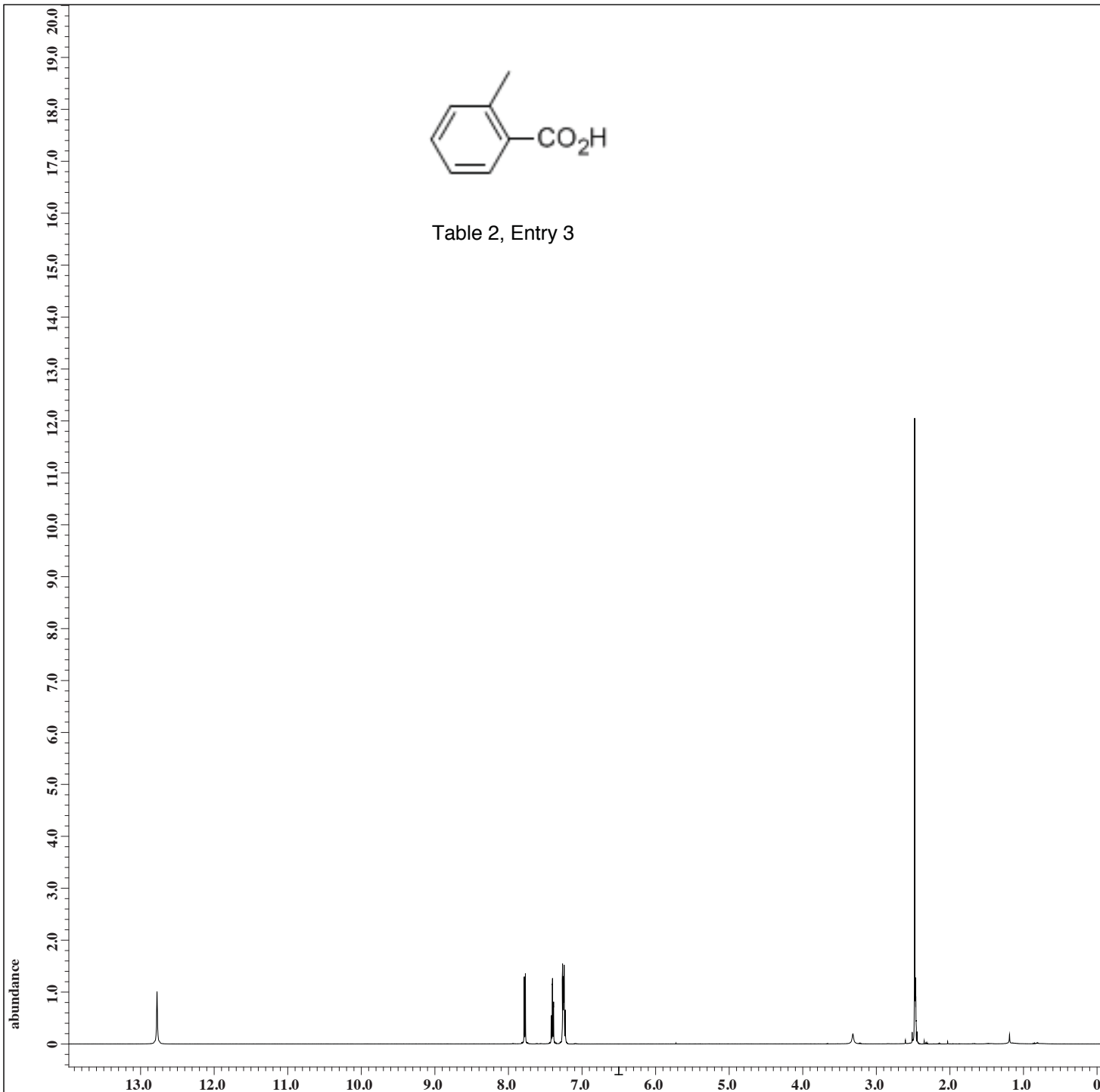


Table 2, Entry 3



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Author       = daugulis
Experiment   = single_pulse.ex2
Sample_id    = 12
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 18:23:18
Revision_time = 22-MAY-2012 12:35:30
Current_time  = 22-MAY-2012 12:36:57
  
```

```

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

```

Field_strength = 11.7473579[T] (500[MH]
X_acq_duration = 1.74587904[s]
X_domain       = 1H
X_freq         = 500.15991521[MHz]
X_offset       = 6.5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737[Hz]
X_sweep        = 9.38438438[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521[MHz]
Tri_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 24
Total_scans    = 24
  
```

```

X_90_width     = 12.84[us]
X_acq_time     = 1.74587904[s]
X_angle        = 45[deg]
X_atn          = 3[dB]
X_pulse        = 6.42[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 44
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get       = 20[dC]
  
```

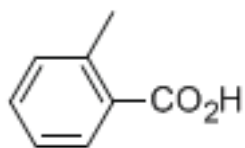
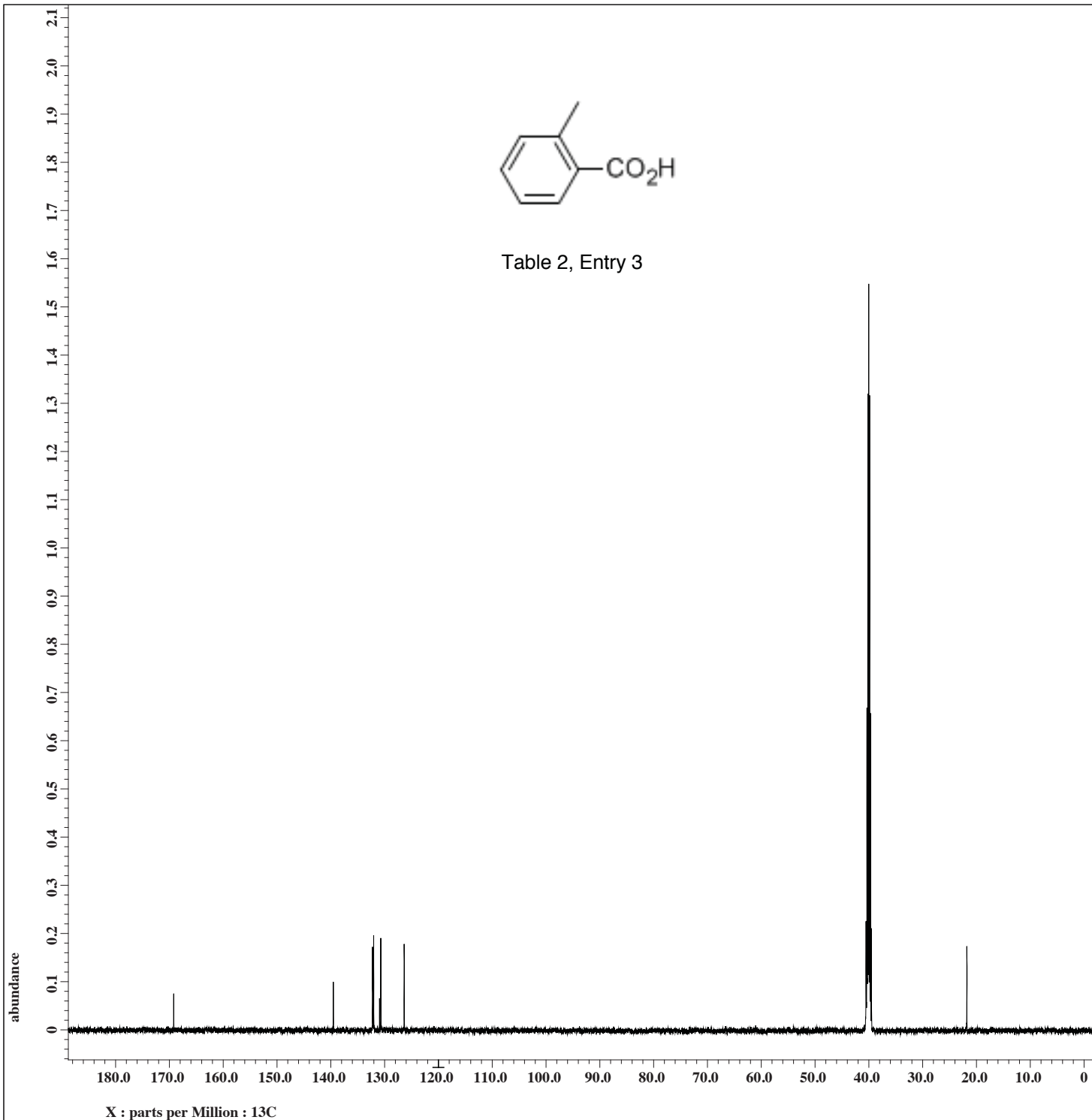


Table 2, Entry 3



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Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 1
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 18:39:41
Revision_time = 22-MAY-2012 12:37:35
Current_time = 22-MAY-2012 12:38:30

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768[MHz]
X_offset      = 120[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521[MHz]
Irr_offset    = 5.0[ppm]
Clipped      = FALSE
Mod_return    = 1
Scans         = 512
Total_scans   = 512

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.92333333[us]
Irr_atn_dec   = 19.5[dB]
Irr_atn_noe   = 19.5[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 1[s]
Recvr_gain    = 58
Relaxation_delay = 1[s]
Repetition_time = 1.83361792[s]
Temp_get      = 20.7[dC]
    
```

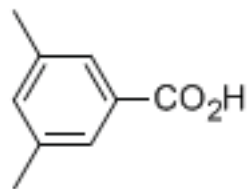
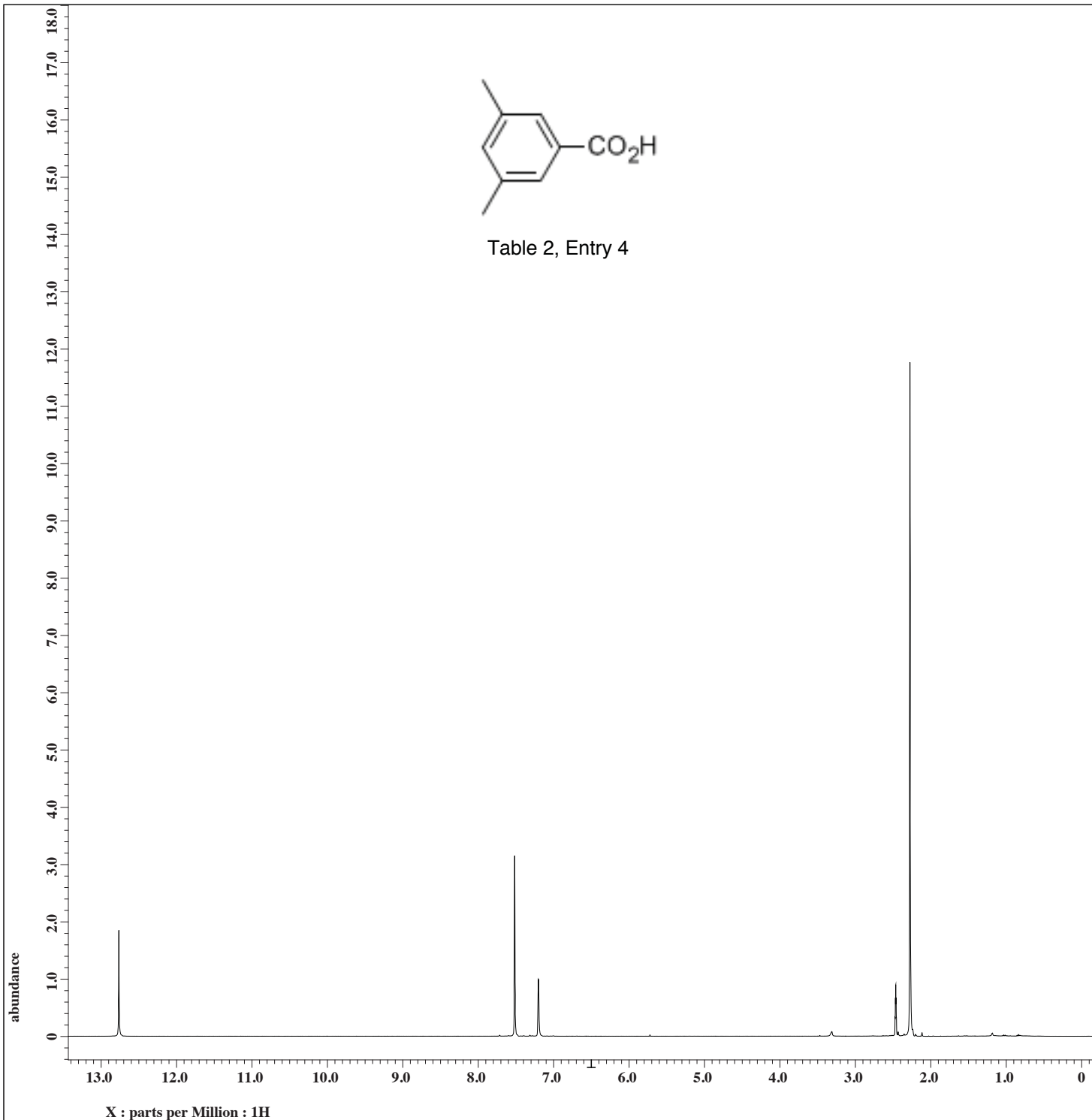


Table 2, Entry 4



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Author       = daugulis
Experiment   = single_pulse.ex2
Sample_id    = 14
Solvent      = DMSO-D6
Creation_time = 6-APR-2012 10:17:48
Revision_time = 22-MAY-2012 12:40:32
Current_time = 22-MAY-2012 12:41:44
  
```

```

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       = 24
Total_scans = 24
  
```

```

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   = 19.7[dc]
  
```

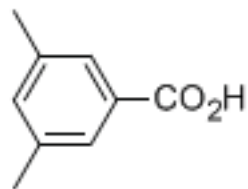
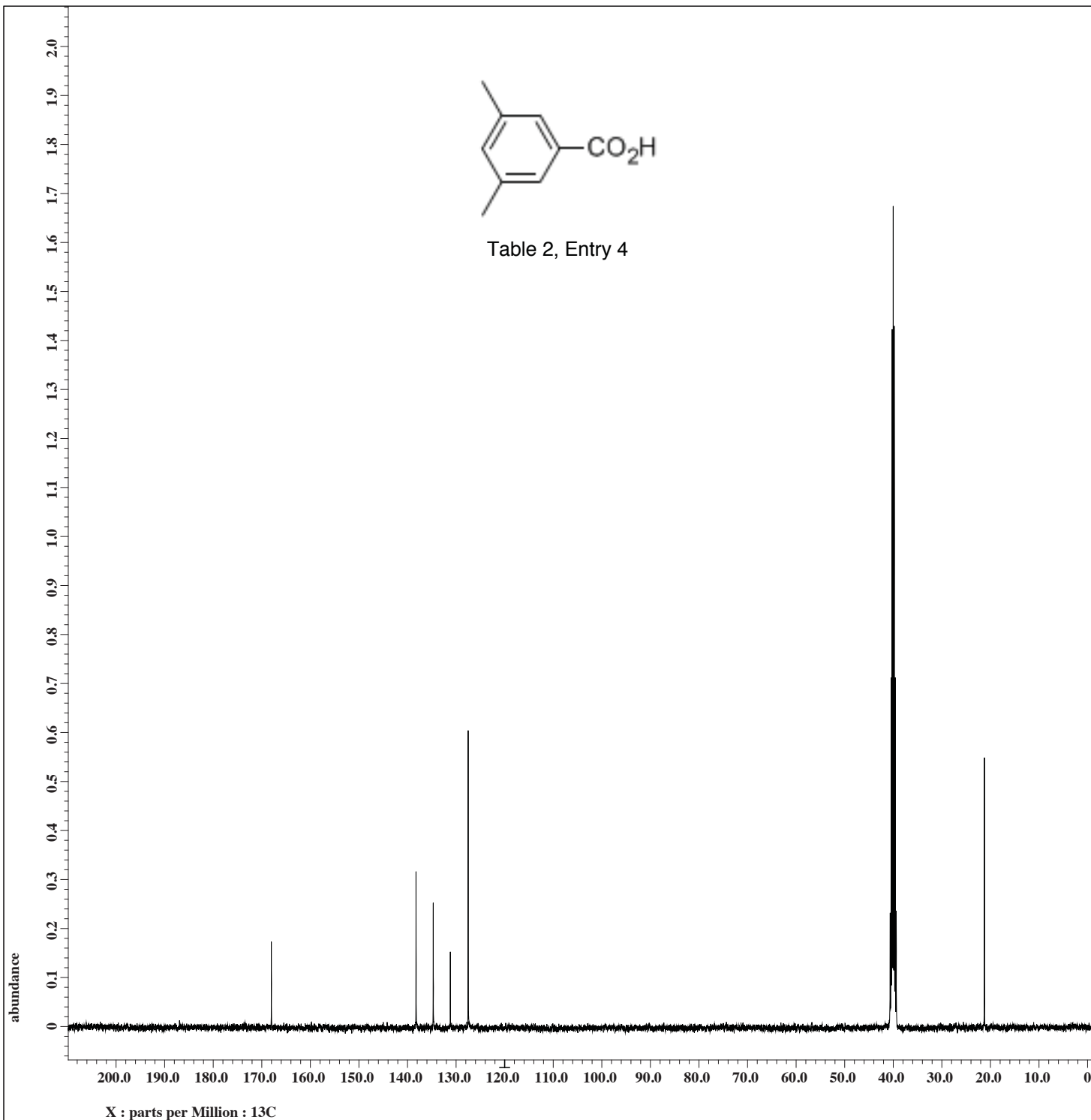


Table 2, Entry 4



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Filename      = 2-106-2-C-2.jdf
Author       = daugulis
Experiment    = single_pulse_dec
Sample_id    = 14
Solvent      = DMSO-D6
Creation_time = 6-APR-2012 10:41:13
Revision_time = 22-MAY-2012 12:42:08
Current_time  = 22-MAY-2012 12:43:28

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     = TRUE
Mod_return   = 1
Scans        = 672
Total_scans  = 672

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     = 20.1[dc]
    
```

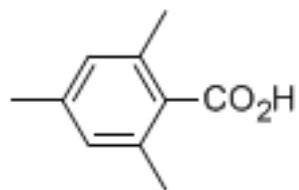
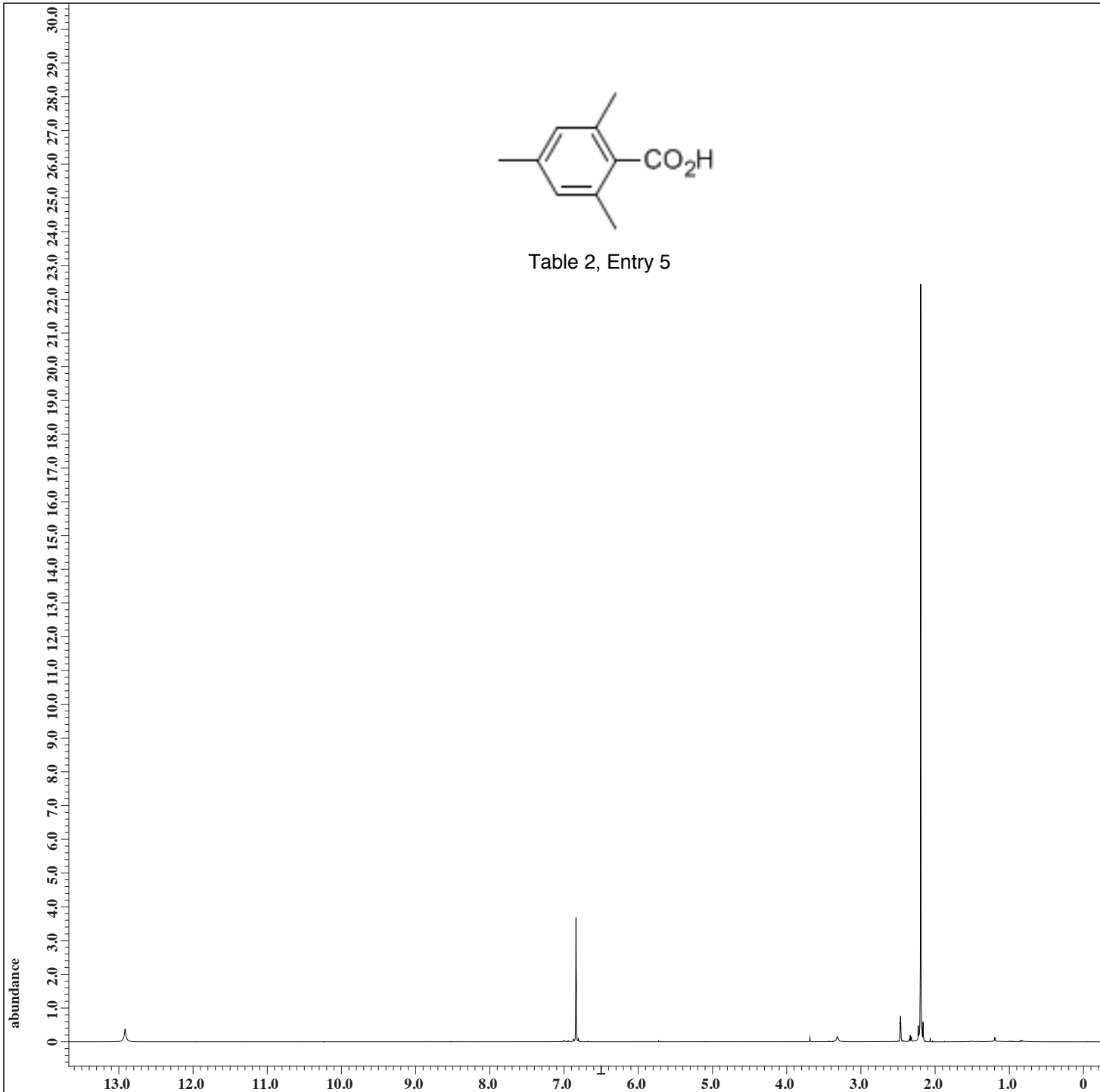


Table 2, Entry 5



```

Filename      = 2-105-1-H-2.jdf
Author       = daugulis
Experiment    = single_pulse.ex2
Sample_id    = 7
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 15:39:33
Revision_time = 22-MAY-2012 12:46:05
Current_time  = 22-MAY-2012 12:47:03
  
```

```

Comment      = single_pulse
Data_format  = 1D_COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = ECA500
  
```

```

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain       = 1H
X_freq         = 500.15991521[MHz]
X_offset       = 6.5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737[Hz]
X_sweep        = 9.38438438[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521[MHz]
Tri_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 32
Total_scans    = 32
  
```

```

X_90_width    = 12.84[us]
X_acq_time     = 1.74587904[s]
X_angle        = 45[deg]
X_atn          = 3[dB]
X_pulse        = 6.42[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 42
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get       = 20.1[dC]
  
```

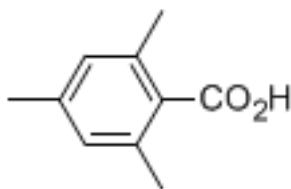
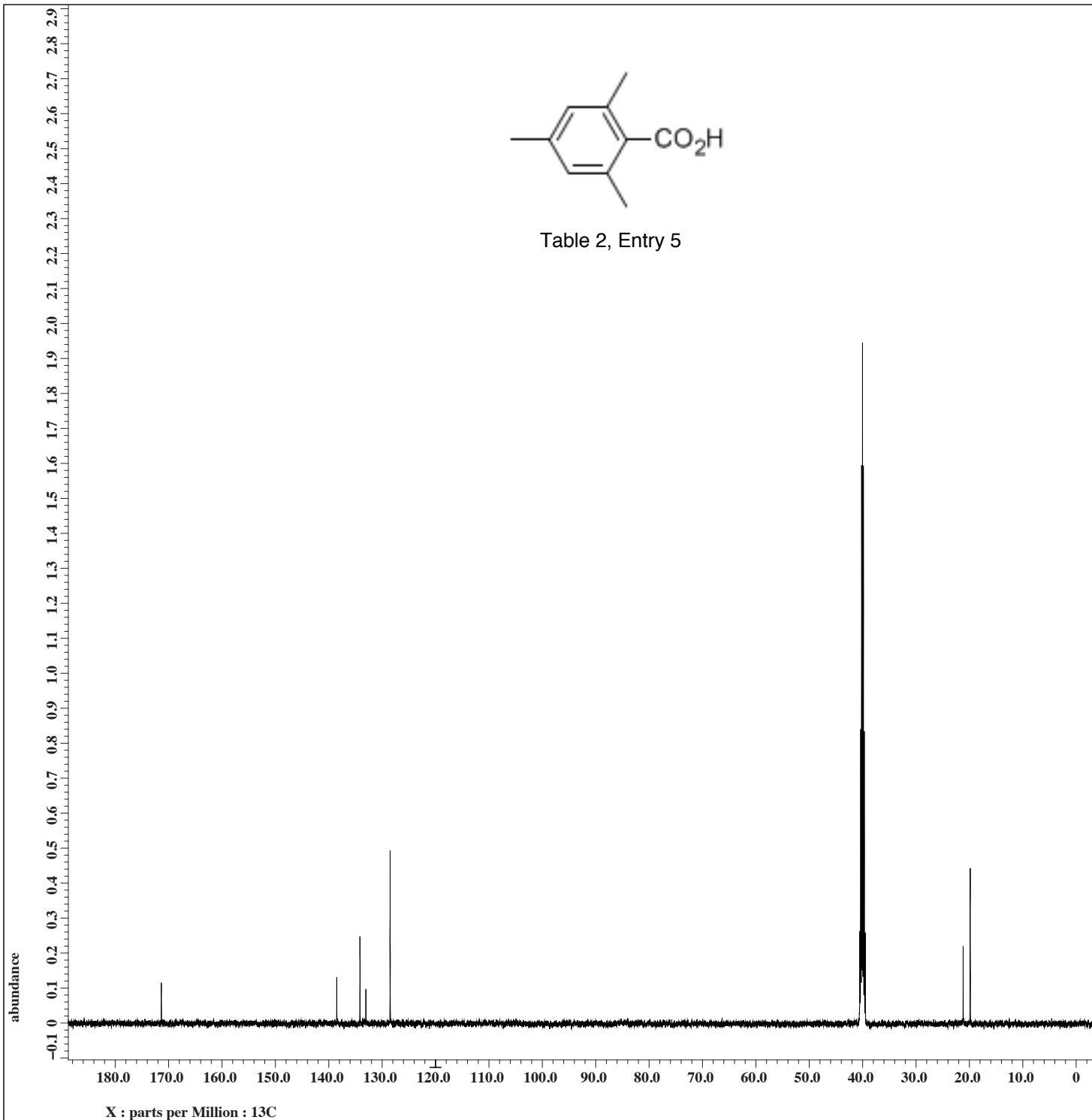


Table 2, Entry 5



```

Filename           = 2-105-1-C-4.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 7
Solvent           = DMSO-D6
Creation_time     = 5-APR-2012 15:50:40
Revision_time    = 22-MAY-2012 12:47:36
Current_time     = 22-MAY-2012 12:48:04

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

Field_strength   = 11.7473579[T] (500[MH
X_acq_duration   = 0.83361792[s]
X_domain         = 13C
X_freq           = 125.76529768[MHz]
X_offset         = 120[ppm]
X_points         = 32768
X_prescans       = 4
X_resolution     = 1.19959034[Hz]
X_sweep          = 39.3081761[kHz]
Irr_domain       = 1H
Irr_freq         = 500.15991521[MHz]
Irr_offset       = 5.0[ppm]
Clipped          = FALSE
Mod_return       = 1
Scans            = 335
Total_scans      = 335

X_90_width       = 11.77[us]
X_acq_time       = 0.83361792[s]
X_angle          = 30[deg]
X_atn            = 9[dB]
X_pulse         = 3.92333333[us]
Irr_atn_dec      = 19.5[dB]
Irr_atn_noe     = 19.5[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  = 1.83361792[s]
Temp_get         = 20.7[dc]
    
```

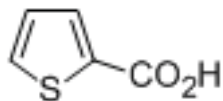
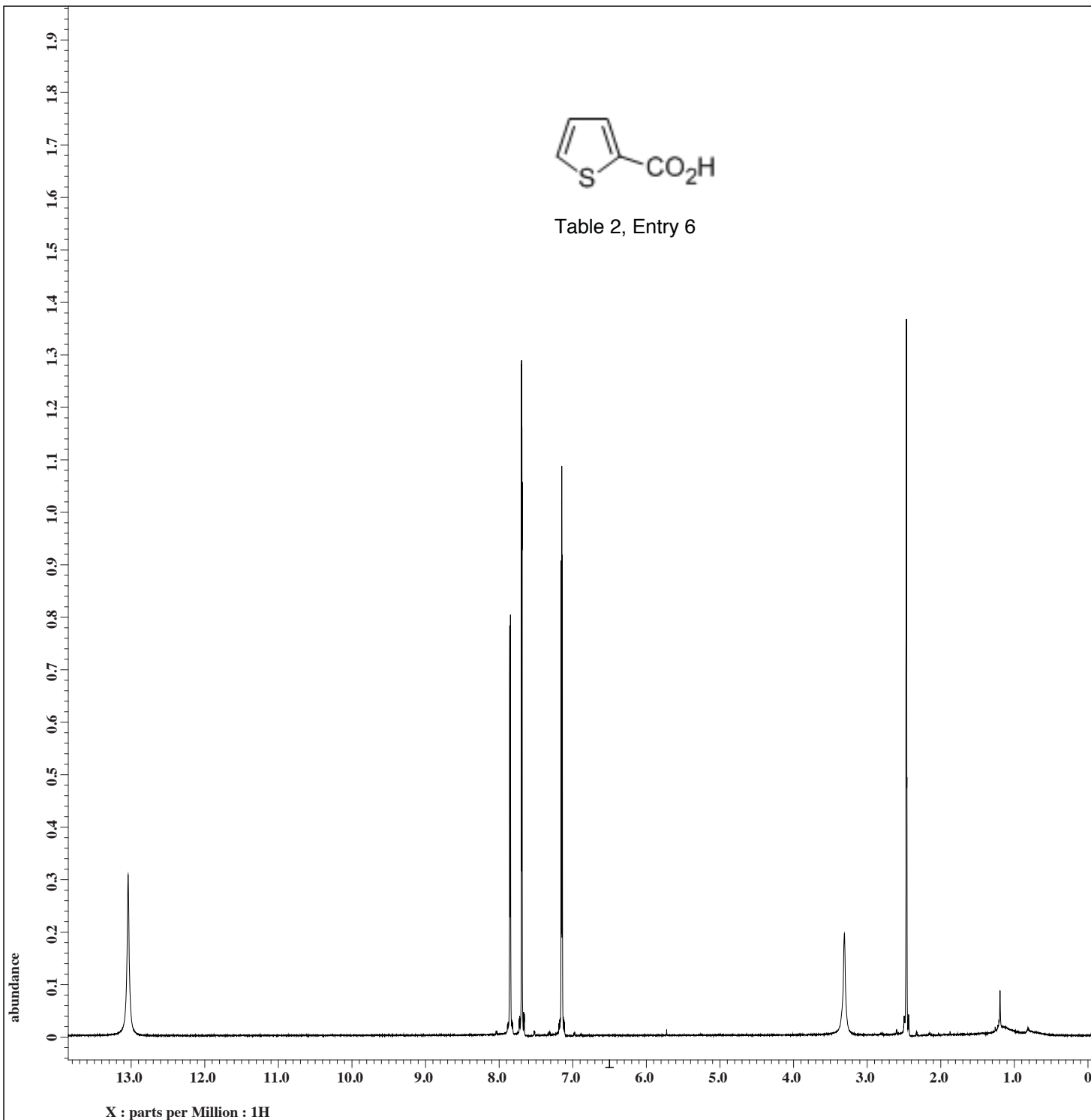


Table 2, Entry 6



Filename = 2-66-1-H-2.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 8
 Solvent = DMSO-D6
 Creation_time = 5-APR-2012 15:58:46
 Revision_time = 22-MAY-2012 12:50:03
 Current_time = 22-MAY-2012 12:50:32

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 32
 Total_scans = 32

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 46
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 20.1[dc]

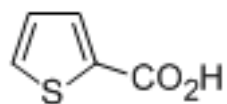
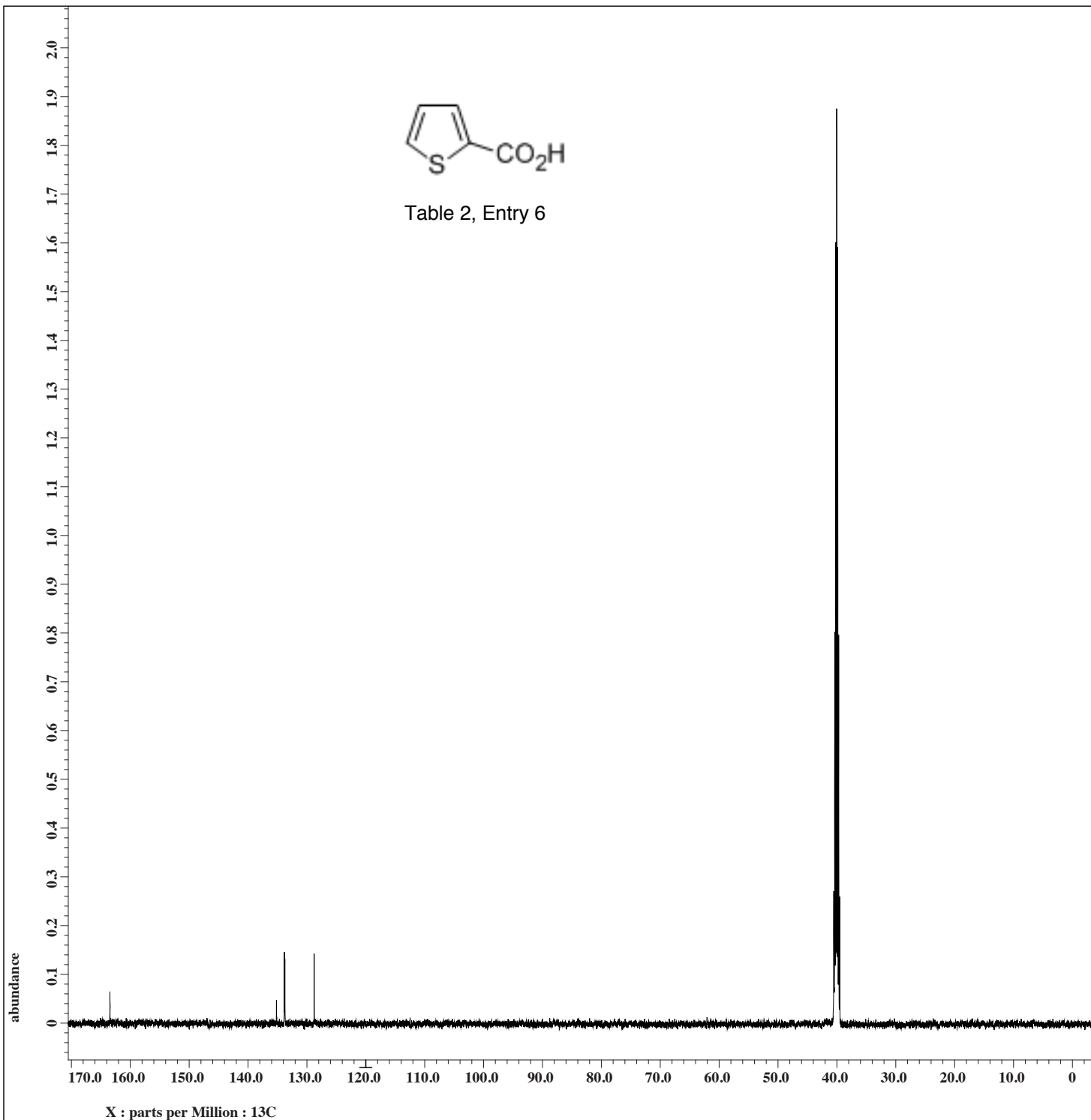


Table 2, Entry 6



```

Filename      = 2-66-1-C-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 8
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 16:13:47
Revision_time = 22-MAY-2012 12:48:45
Current_time  = 22-MAY-2012 12:49:40

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 120[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 472
Total_scans    = 472

X_90_width     = 11.77[us]
X_acq_time     = 0.83361792[s]
X_angle        = 30[deg]
X_atn          = 9[dB]
X_pulse        = 3.92333333[us]
Irr_atn_dec    = 19.5[dB]
Irr_atn_noe    = 19.5[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 = 1.83361792[s]
Temp_get       = 20.7[dC]
    
```

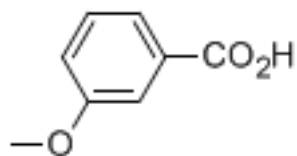
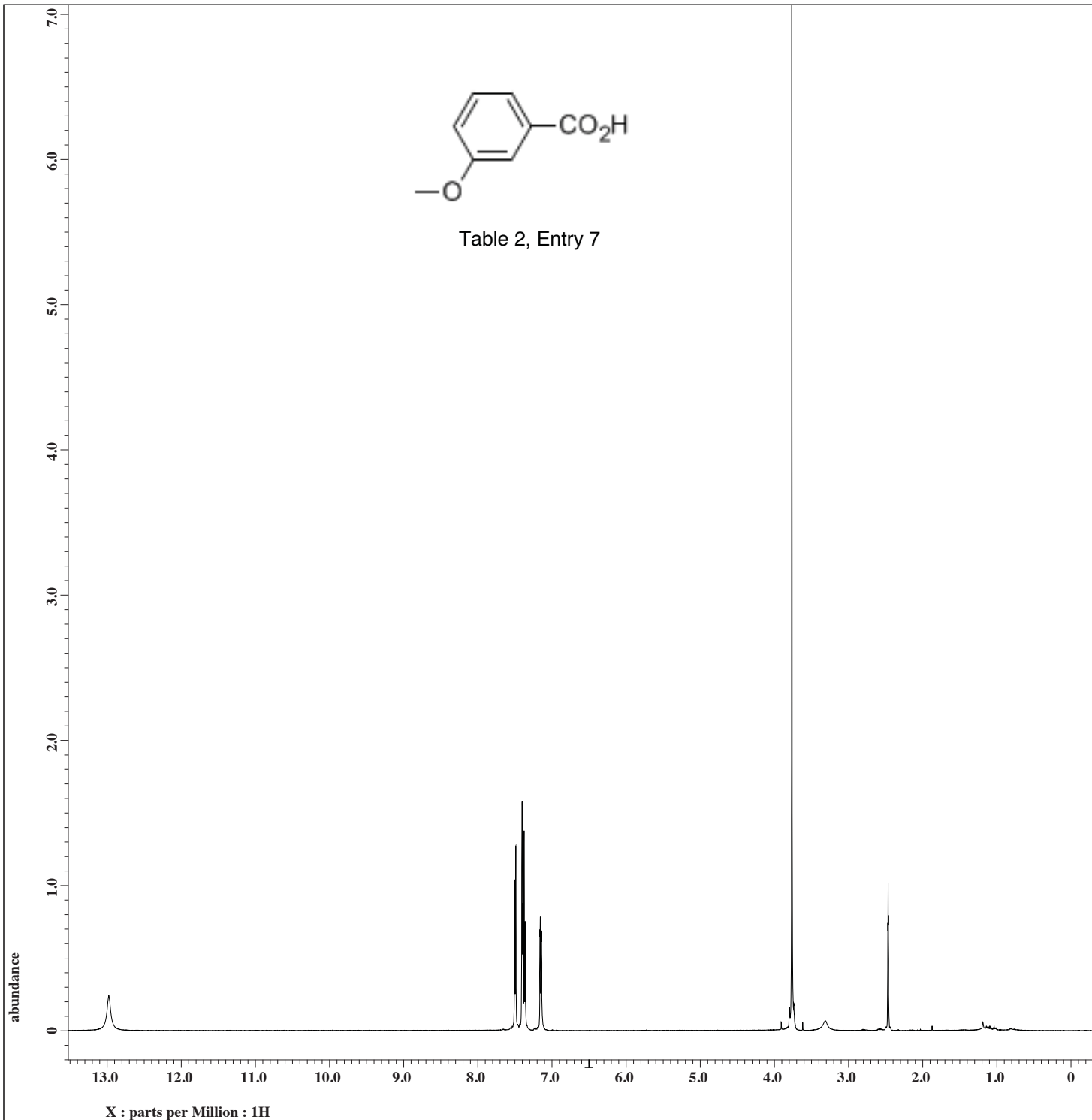


Table 2, Entry 7



Filename = 2-124-1-H-3.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 10
 Solvent = DMSO-D6
 Creation_time = 5-APR-2012 16:21:52
 Revision_time = 22-MAY-2012 14:27:53
 Current_time = 22-MAY-2012 14:28:37

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 24
 Total_scans = 24

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 44
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 20.1[dc]

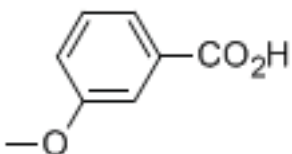
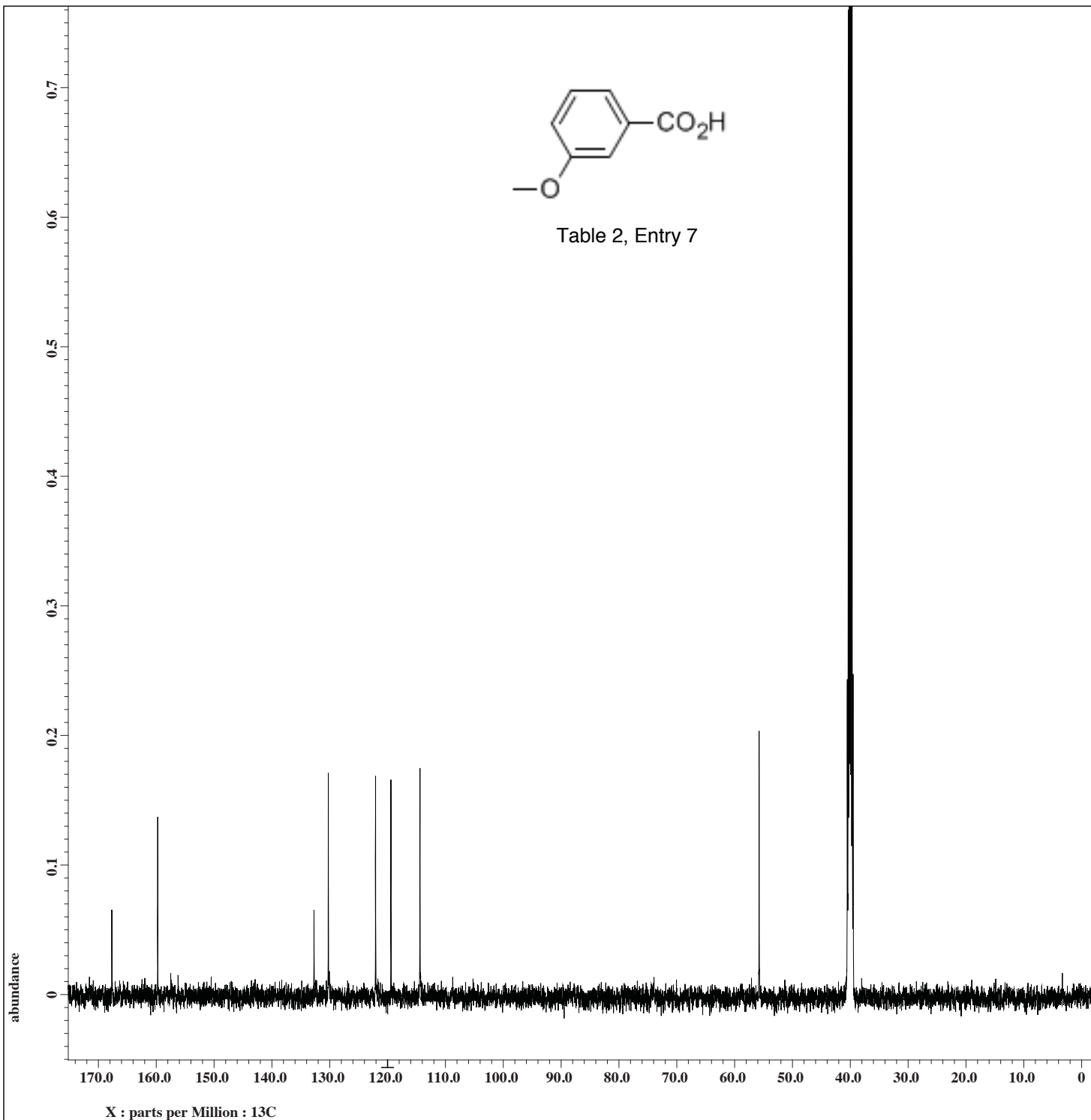


Table 2, Entry 7



```

Filename      = 2-124-1-C-2.jdf
Author       = daugulis
Experiment    = single_pulse_dec
Sample_id    = 1
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 16:32:47
Revision_time = 22-MAY-2012 15:57:09
Current_time  = 22-MAY-2012 15:58:09

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 120[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 339
Total_scans    = 339

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.92333333[us]
Irr_atn_dec   = 19.5[dB]
Irr_atn_noe   = 19.5[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 = 1.83361792[s]
Temp_get      = 20.7[dC]
  
```

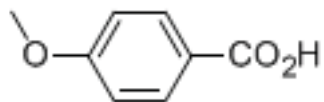
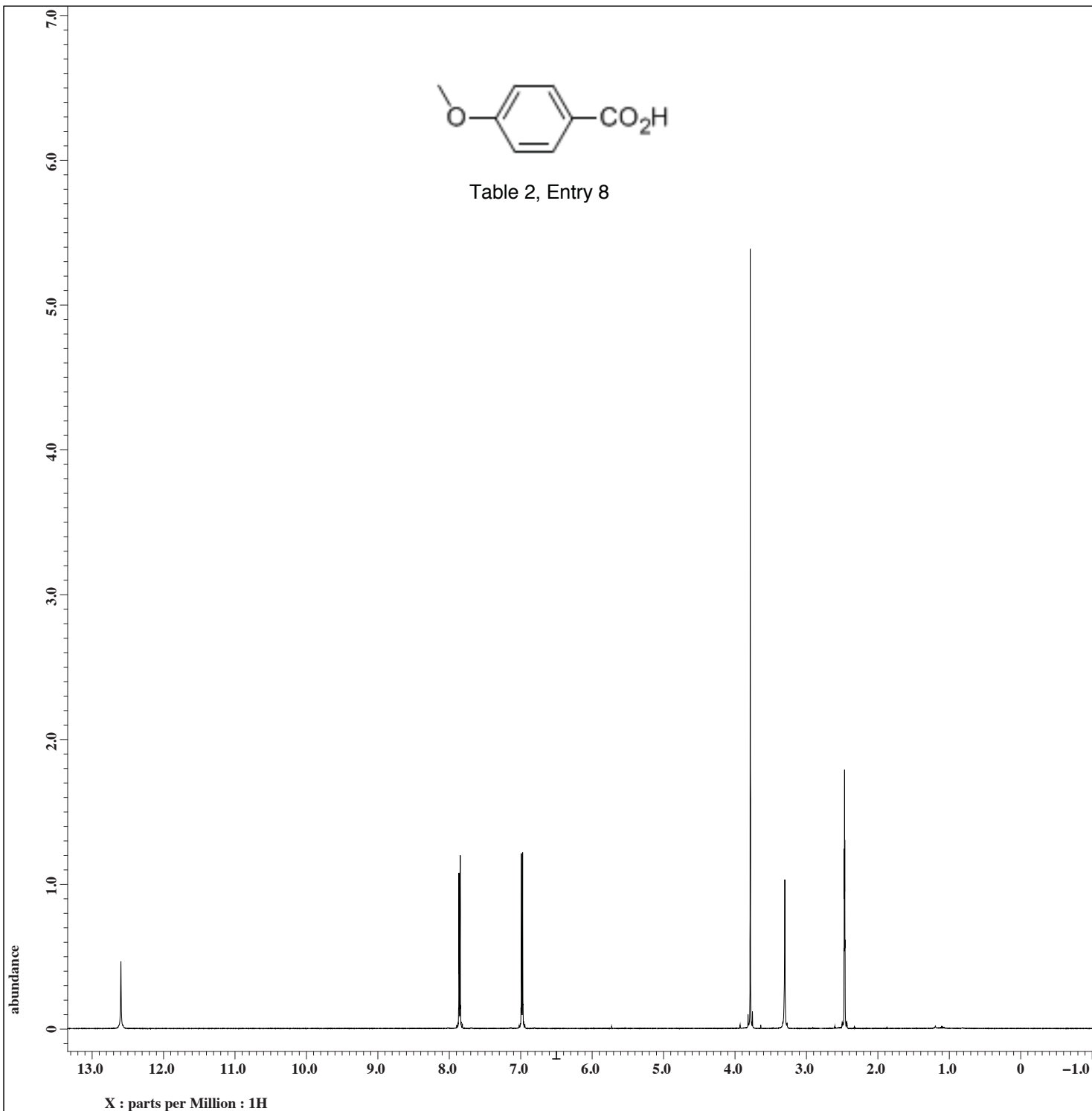


Table 2, Entry 8



```

Filename      = 2-103-2-H-3.jdf
Author       = daugulis
Experiment   = single_pulse.ex2
Sample_id    = 13
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 18:46:02
Revision_time = 22-MAY-2012 12:55:31
Current_time  = 22-MAY-2012 12:55:59

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain       = 1H
X_freq         = 500.15991521[MHz]
X_offset       = 6.5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737[Hz]
X_sweep        = 9.38438438[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521[MHz]
Tri_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 24
Total_scans    = 24

X_90_width    = 12.84[us]
X_acq_time     = 1.74587904[s]
X_angle       = 45[deg]
X_atn         = 3[dB]
X_pulse       = 6.42[us]
Irr_mode      = Off
Tri_mode      = Off
Dante_presat  = FALSE
Initial_wait  = 1[s]
Recvr_gain    = 48
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get      = 20.1[dc]
    
```

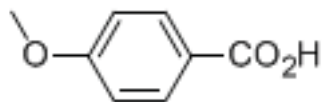
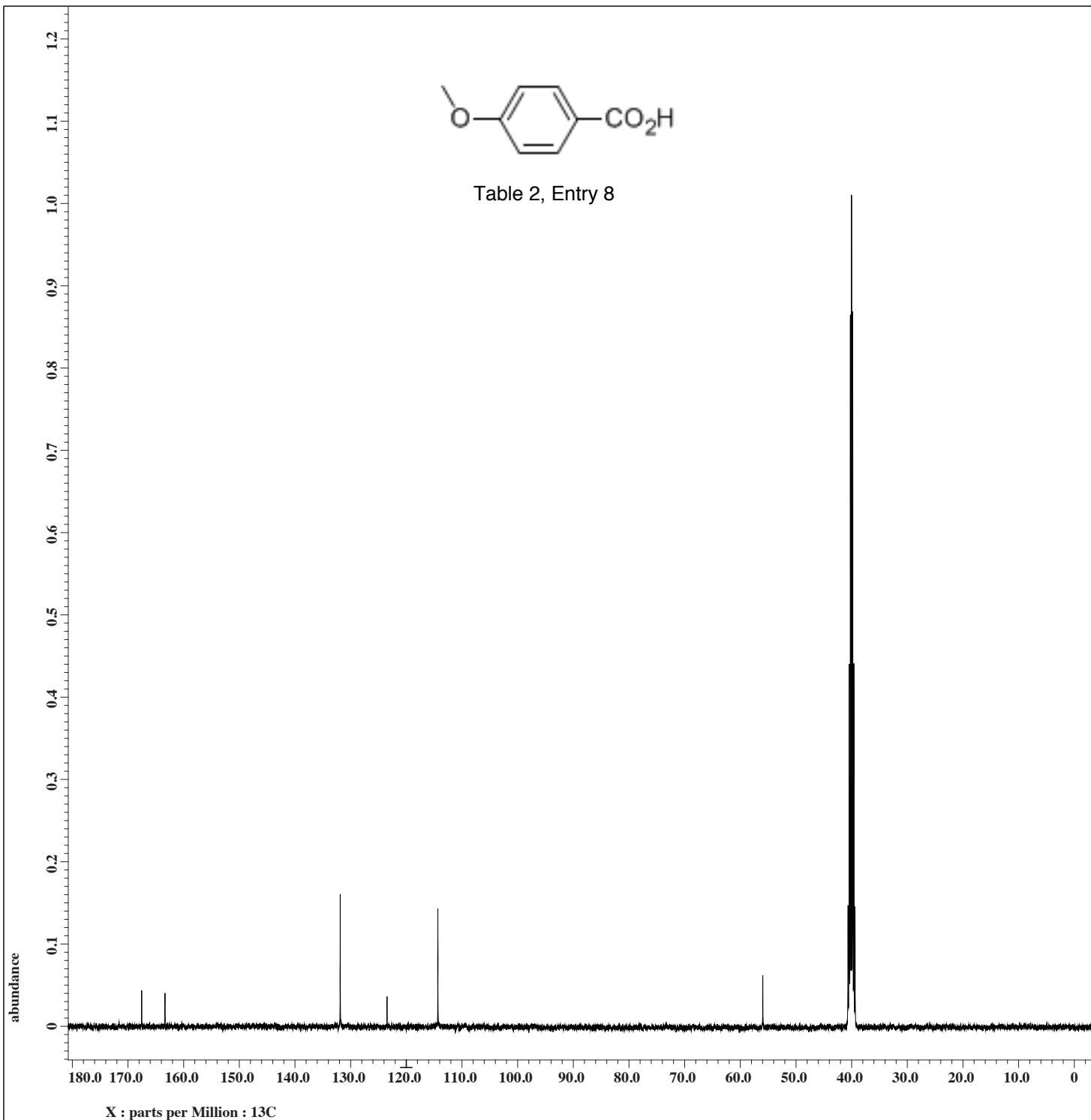


Table 2, Entry 8



```

Filename      = 2-103-2-C-1-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 13
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 21:04:02
Revision_time = 22-MAY-2012 12:56:32
Current_time  = 22-MAY-2012 12:56:58

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        = 1098
Total_scans  = 1098

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   = 56
Relaxation_delay = 1[s]
Repetition_time = 2.04333312[s]
Temp_get     = 20.1[dc]
  
```

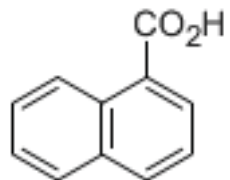
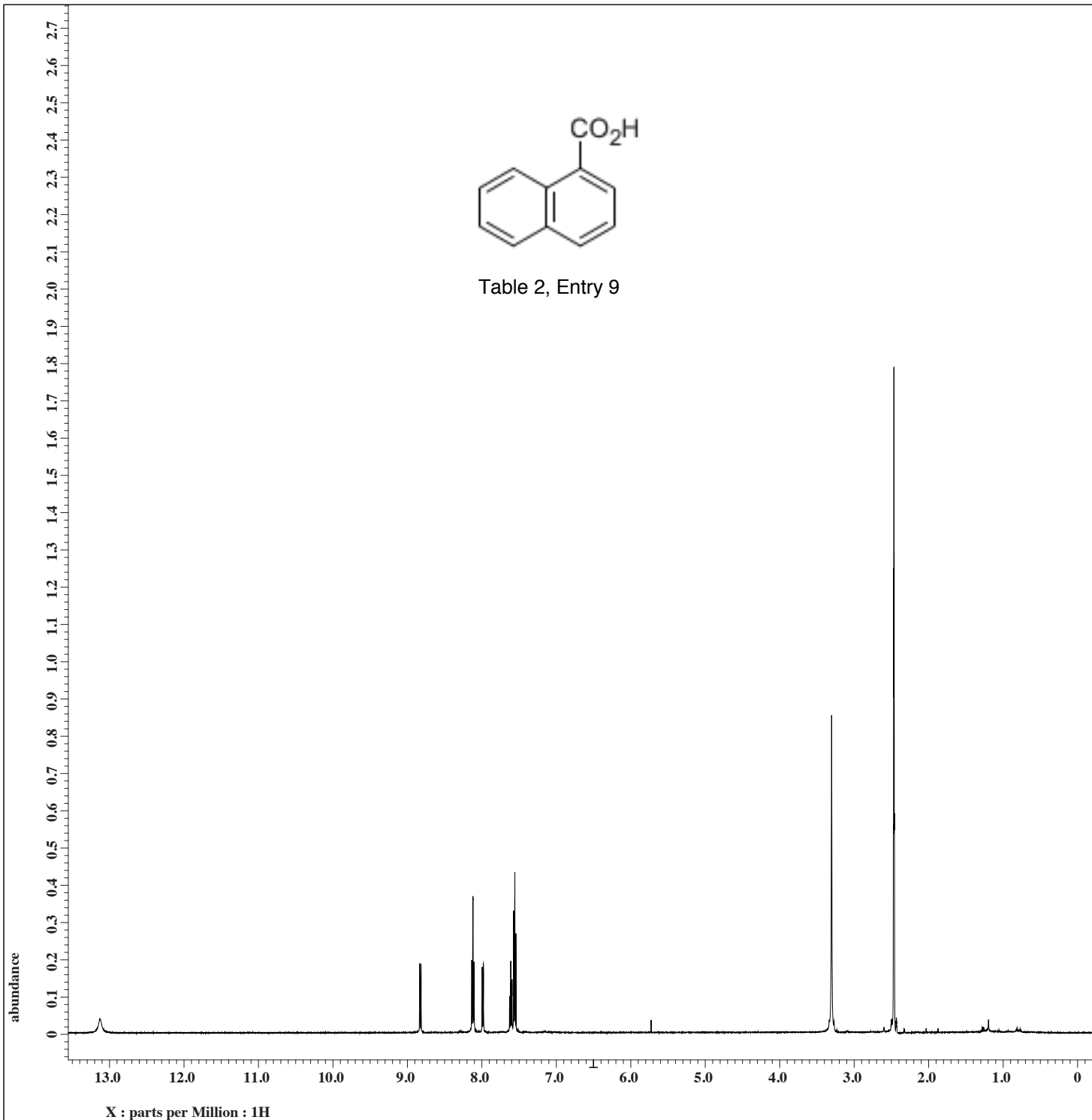


Table 2, Entry 9



Filename = 2-115-2-H-7.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = S#566318
 Solvent = DMSO-D6
 Creation_time = 7-APR-2012 14:49:08
 Revision_time = 22-MAY-2012 13:00:25
 Current_time = 22-MAY-2012 13:01:02

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 40
 Total_scans = 40

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 48
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 20[dC]

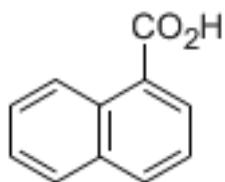
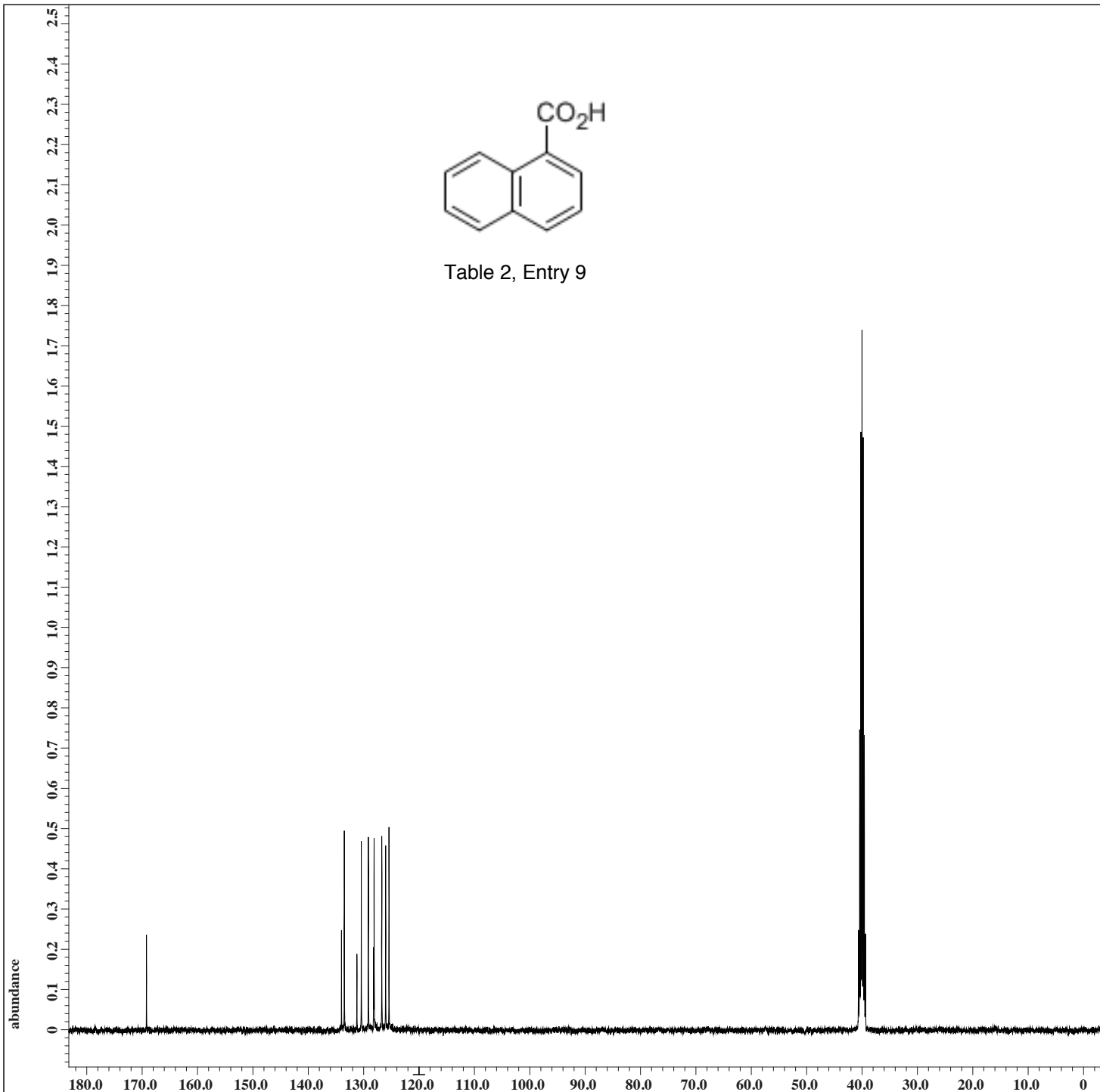


Table 2, Entry 9



X : parts per Million : 13C

```

Filename           = 2-115-2-C-3.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 11
Solvent           = DMSO-D6
Creation_time     = 5-APR-2012 18:30:12
Revision_time    = 22-MAY-2012 12:57:59
Current_time      = 22-MAY-2012 12:58:30

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         = TRUE
Mod_return       = 1
Scans            = 743
Total_scans      = 743

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         = 20.1[dc]
    
```

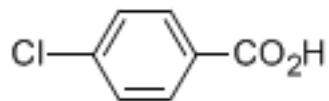
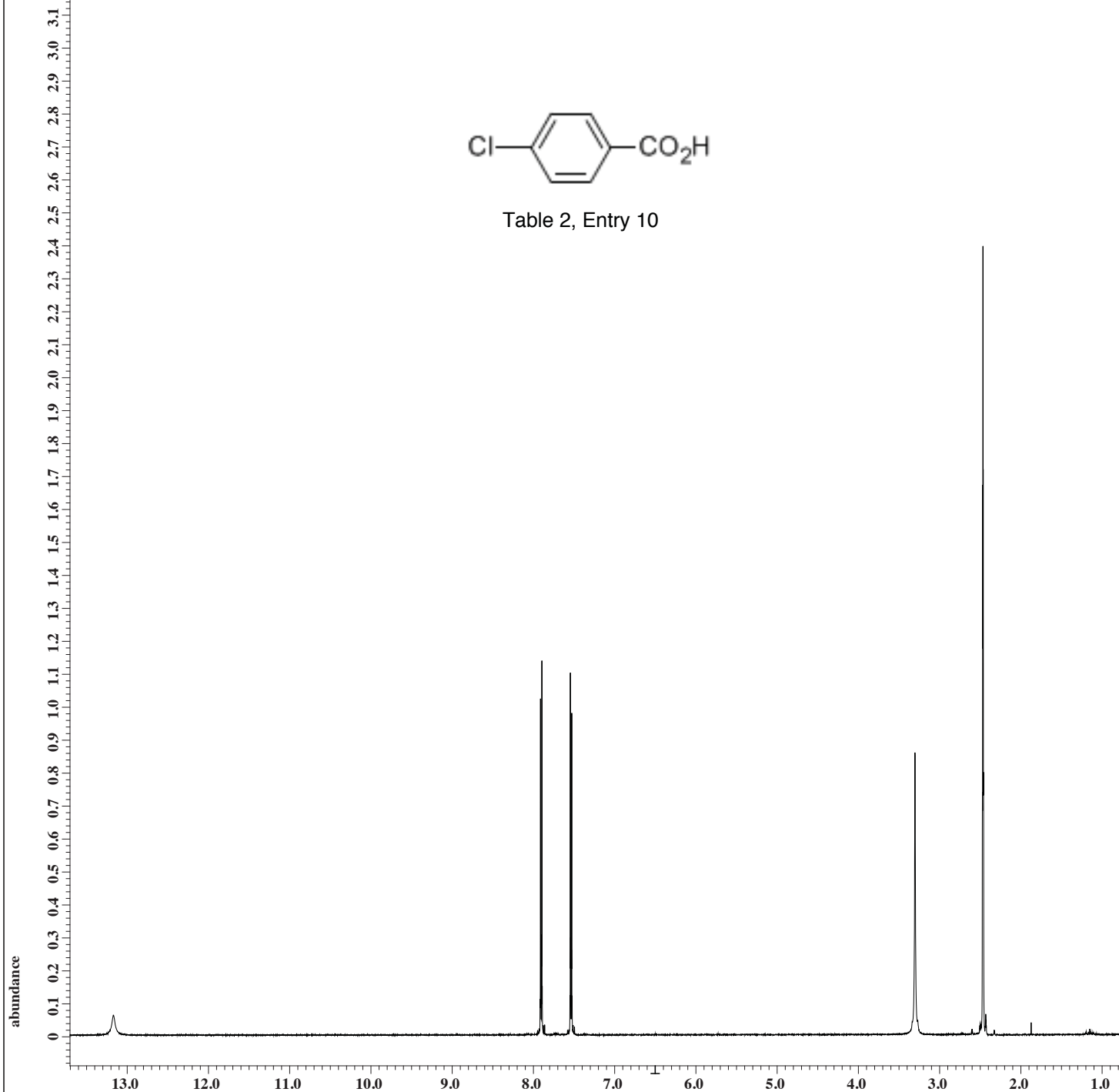


Table 2, Entry 10



Filename = 2-138-1-H-2.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = S#562058
 Solvent = DMSO-D6
 Creation_time = 7-APR-2012 14:41:57
 Revision_time = 22-MAY-2012 13:25:48
 Current_time = 22-MAY-2012 13:26:22

Comment = single_pulse
 Data_format = 1D_COMPLEX
 Dim_size = 13107
 Dim_title = 1H
 Dim_units = [ppm]
 Dimensions = X
 Site = ECA 500
 Spectrometer = ECA500
 Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 40
 Total_scans = 40
 X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 50
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 19.9[dc]

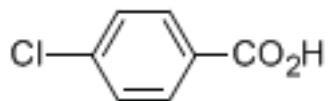
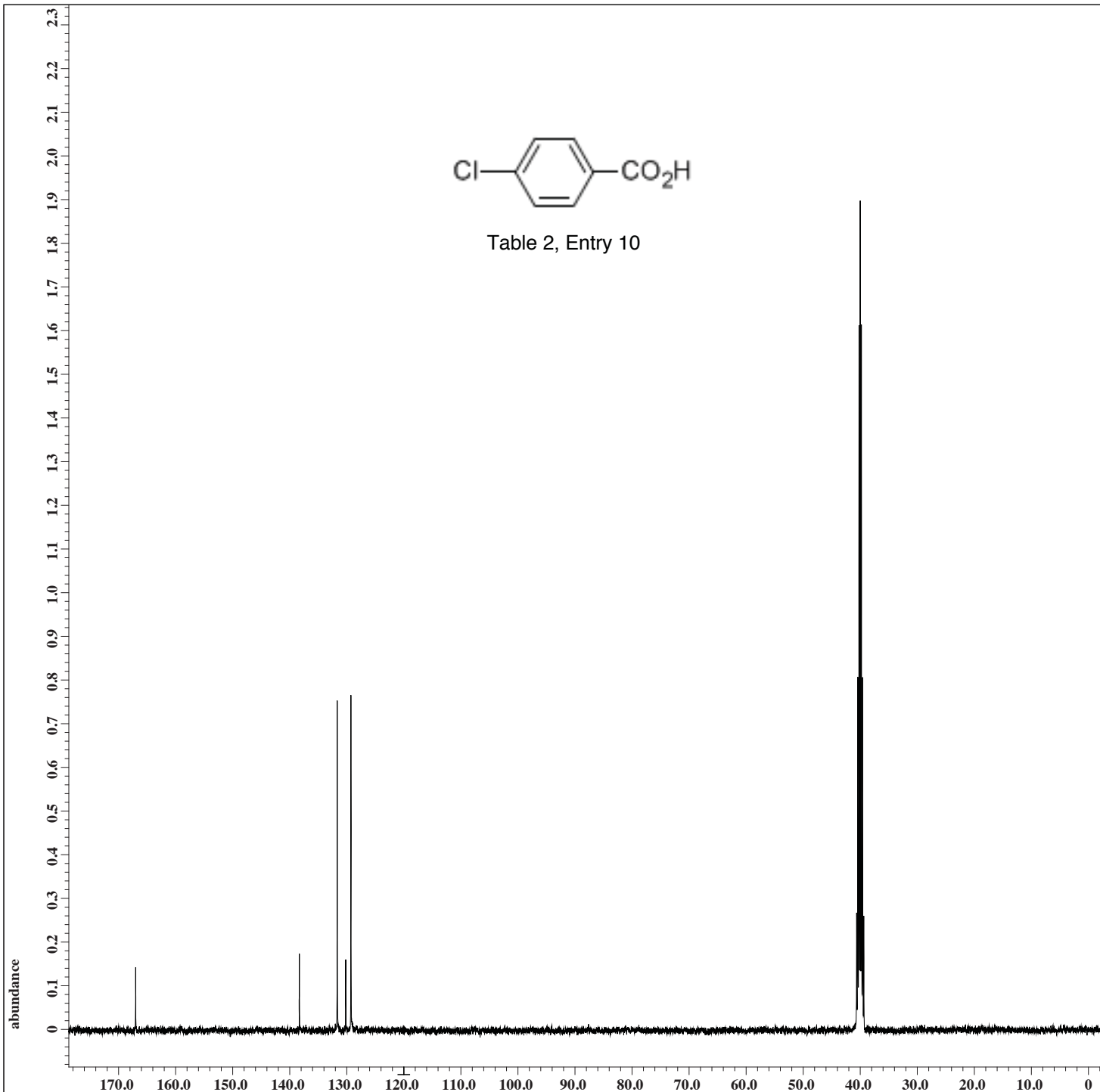


Table 2, Entry 10



```

Filename      = 2-145-1-C-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 4
Solvent      = DMSO-D6
Creation_time = 4-APR-2012 19:13:01
Revision_time = 22-MAY-2012 13:02:24
Current_time = 22-MAY-2012 13:03:02

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        = TRUE
Mod_return     = 1
Scans          = 530
Total_scans    = 530

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      = 20.1[degC]
  
```

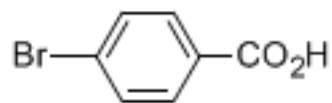
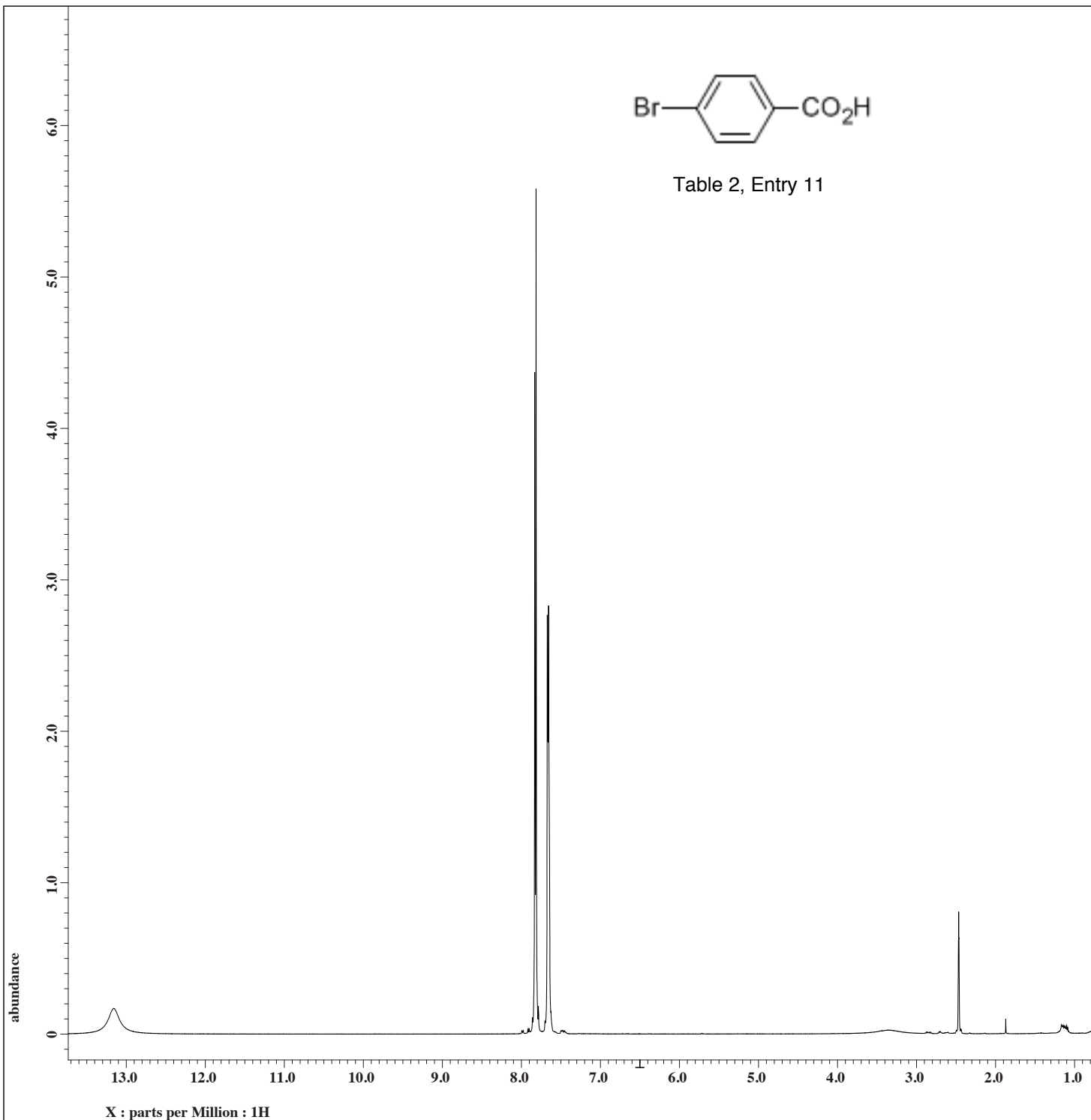


Table 2, Entry 11



Filename = 2-154-1-H-2.jdf
Author = daugulis
Experiment = single_pulse.ex2
Sample_id = 2
Solvent = DMSO-D6
Creation_time = 4-APR-2012 13:07:50
Revision_time = 22-MAY-2012 13:27:50
Current_time = 22-MAY-2012 13:28:28

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain = 1H
X_freq = 500.15991521[MHz]
X_offset = 6.5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.57277737[Hz]
X_sweep = 9.38438438[kHz]
Irr_domain = 1H
Irr_freq = 500.15991521[MHz]
Irr_offset = 5.0[ppm]
Tri_domain = 1H
Tri_freq = 500.15991521[MHz]
Tri_offset = 5.0[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 32
Total_scans = 32

X_90_width = 12.84[us]
X_acq_time = 1.74587904[s]
X_angle = 45[deg]
X_atn = 3[dB]
X_pulse = 6.42[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 42
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get = 19.9[dc]

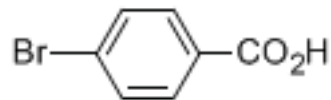
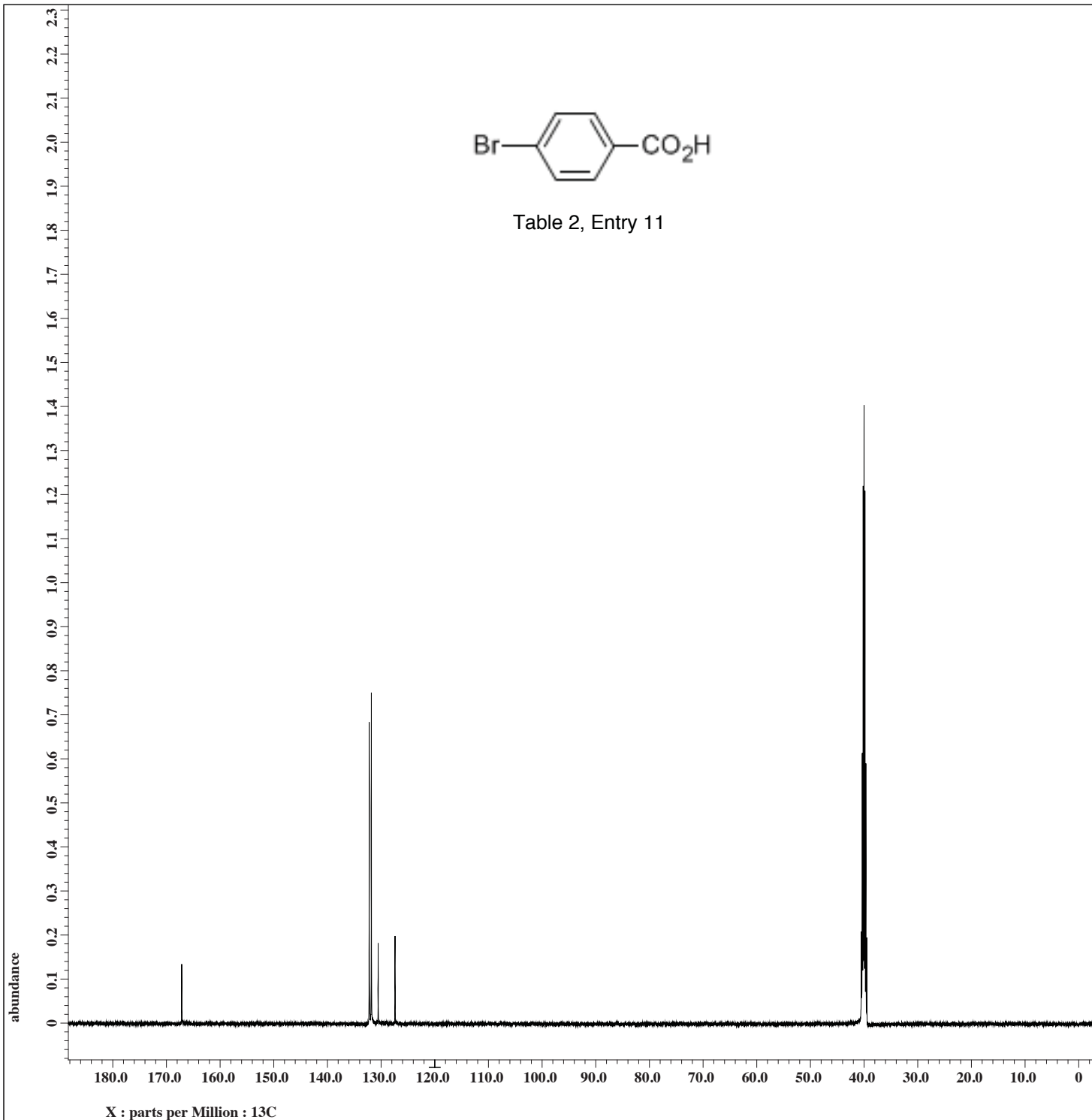


Table 2, Entry 11



```

Filename      = 2-154-1-C-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 2
Solvent      = DMSO-D6
Creation_time = 4-APR-2012 13:55:38
Revision_time = 22-MAY-2012 13:05:23
Current_time = 22-MAY-2012 13:05:56

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768[MHz]
X_offset      = 120[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521[MHz]
Irr_offset    = 5.0[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 1536
Total_scans   = 1536

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.92333333[us]
Irr_atn_dec   = 19.5[dB]
Irr_atn_noe   = 19.5[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 1[s]
Recvr_gain    = 58
Relaxation_delay = 1[s]
Repetition_time = 1.83361792[s]
Temp_get      = 20.7[dc]
  
```

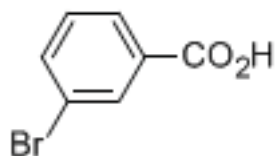
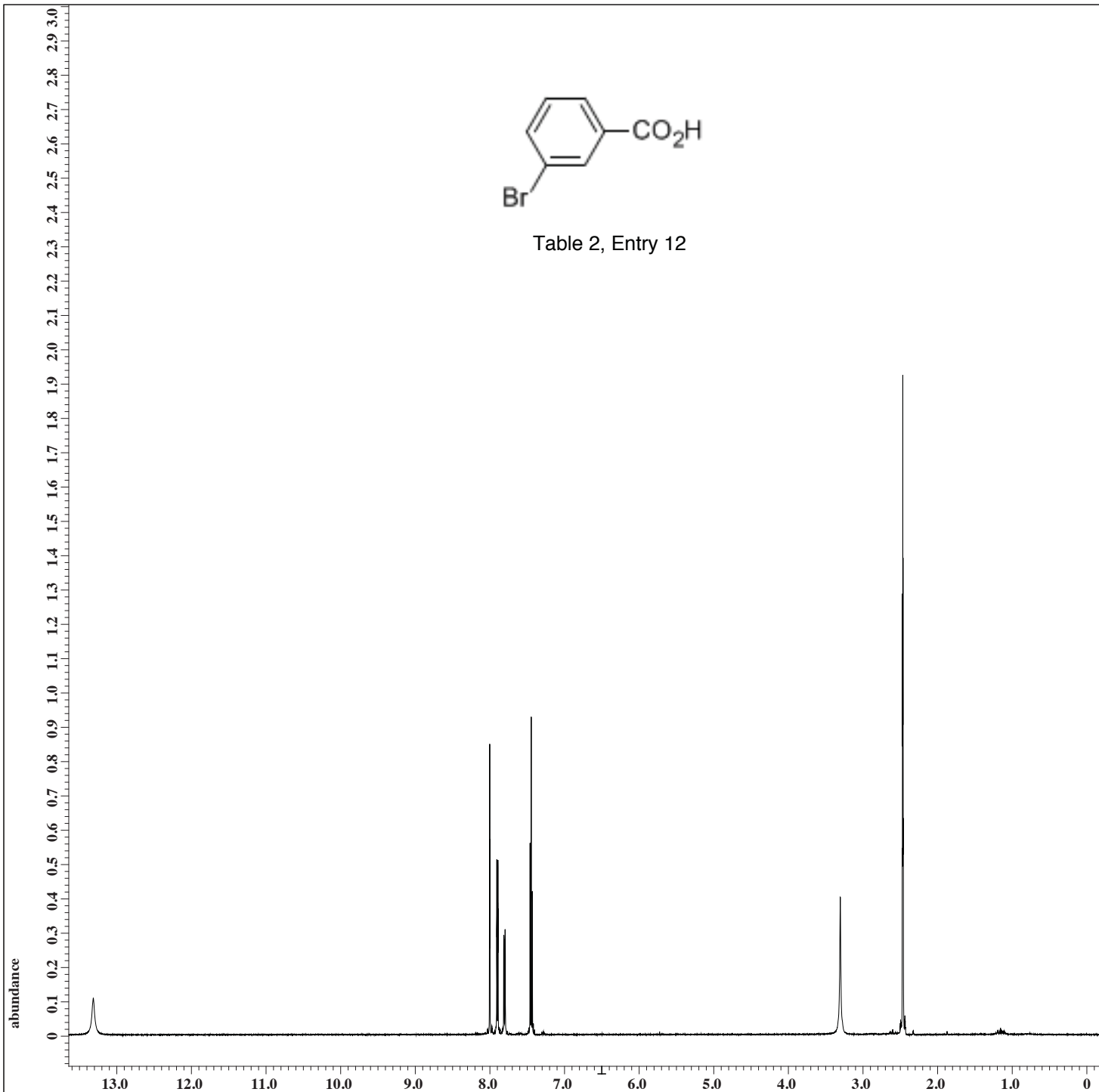


Table 2, Entry 12



Filename = 2-149-1-H-2.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 1
 Solvent = DMSO-D6
 Creation_time = 3-APR-2012 17:08:41
 Revision_time = 22-MAY-2012 13:29:15
 Current_time = 22-MAY-2012 13:30:07

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 32
 Total_scans = 32

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 48
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 19.9[dc]

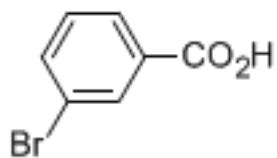
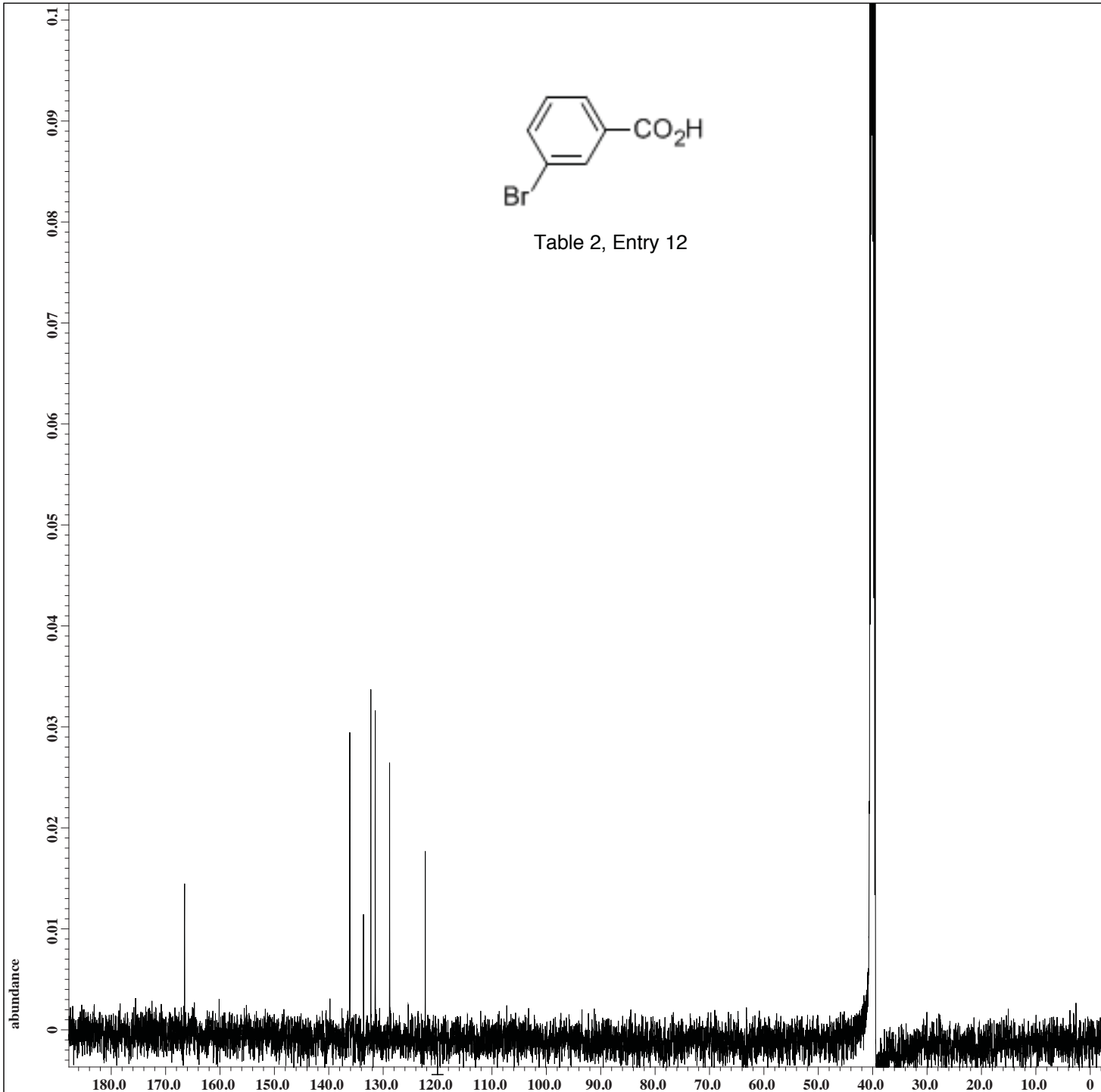


Table 2, Entry 12



X : parts per Million : 13C

```

Filename      = 2-149-1-C2-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 1
Solvent      = DMSO-D6
Creation_time = 3-APR-2012 20:55:55
Revision_time = 22-MAY-2012 13:08:31
Current_time = 22-MAY-2012 13:09:17

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq         = 125.76529768[MHz]
X_offset       = 120[ppm]
X_points       = 32768
X_prescans     = 4
X_resolution   = 1.19959034[Hz]
X_sweep        = 39.3081761[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 2048
Total_scans    = 2048

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.92333333[us]
Irr_atn_dec   = 19.5[dB]
Irr_atn_noe   = 19.5[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 1[s]
Recvr_gain    = 56
Relaxation_delay = 1[s]
Repetition_time = 1.83361792[s]
Temp_get      = 20.5[dc]
    
```

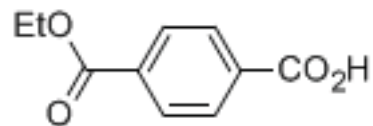
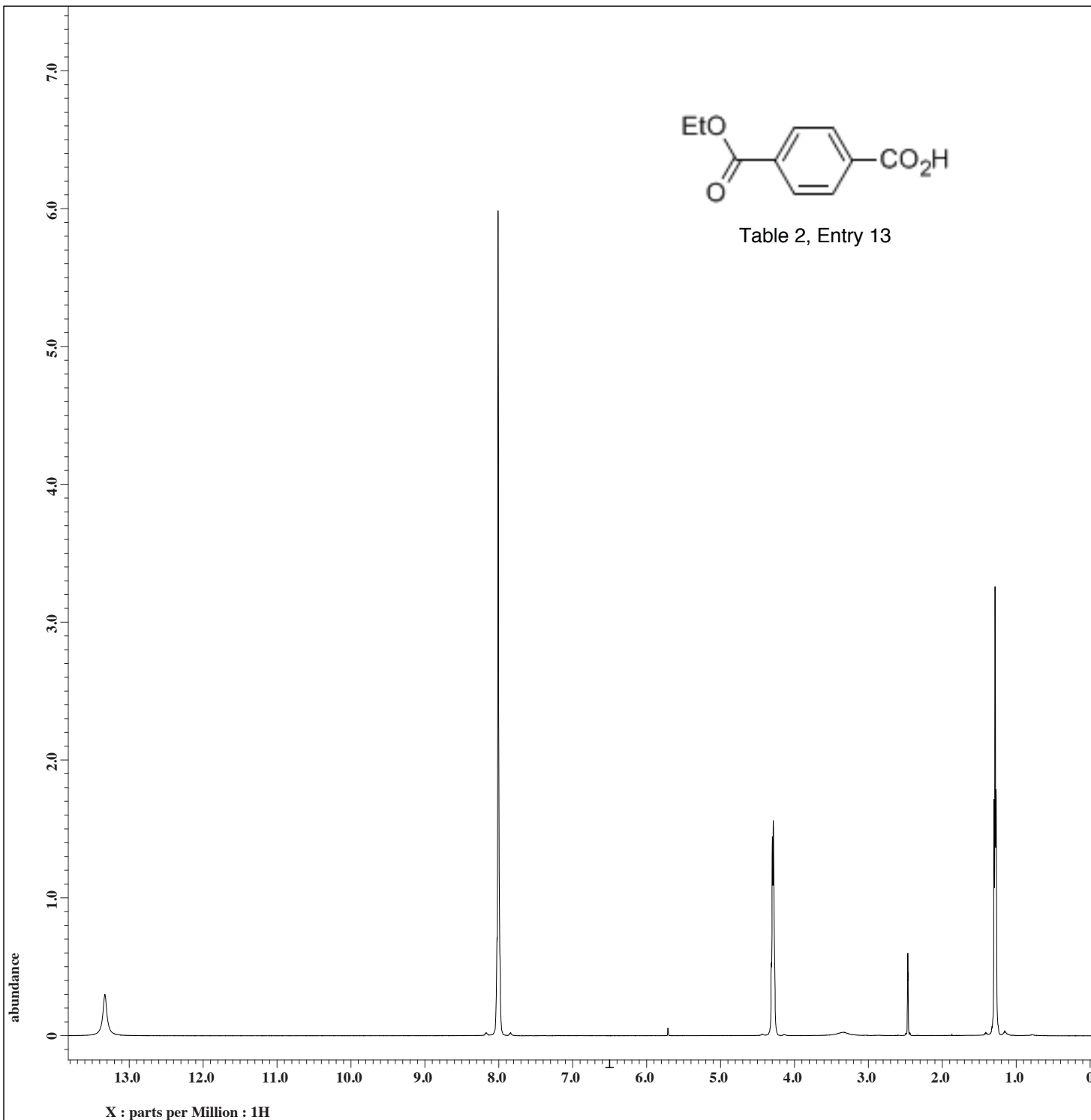


Table 2, Entry 13



Filename = 2-132-1-H-2.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 6
 Solvent = DMSO-D6
 Creation_time = 5-APR-2012 15:18:35
 Revision_time = 22-MAY-2012 13:30:42
 Current_time = 22-MAY-2012 13:31:15

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 32
 Total_scans = 32

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 38
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 20[dC]

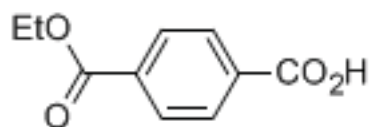
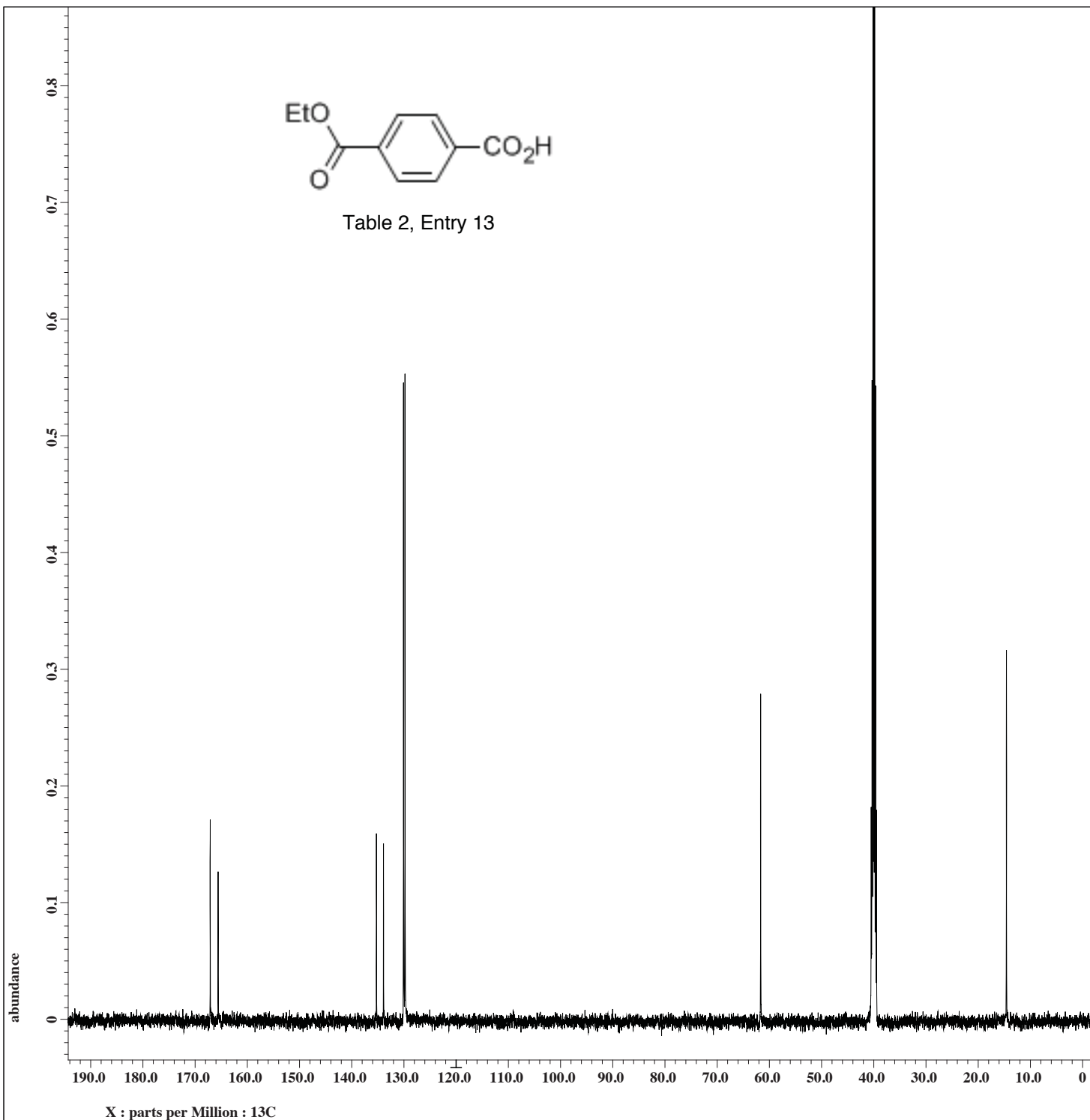


Table 2, Entry 13



```

Filename      = 2-132-1-C-2.jdf
Author       = daugulis
Experiment    = single_pulse_dec
Sample_id    = 6
Solvent      = DMSO-D6
Creation_time = 5-APR-2012 15:32:12
Revision_time = 22-MAY-2012 13:10:27
Current_time  = 22-MAY-2012 13:11:11

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

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 0.83361792[s]
X_domain       = 13C
X_freq        = 125.76529768[MHz]
X_offset      = 120[ppm]
X_points      = 32768
X_prescans    = 4
X_resolution  = 1.19959034[Hz]
X_sweep       = 39.3081761[kHz]
Irr_domain    = 1H
Irr_freq      = 500.15991521[MHz]
Irr_offset    = 5.0[ppm]
Clipped       = FALSE
Mod_return    = 1
Scans         = 421
Total_scans   = 421

X_90_width    = 11.77[us]
X_acq_time    = 0.83361792[s]
X_angle       = 30[deg]
X_atn         = 9[dB]
X_pulse       = 3.92333333[us]
Irr_atn_dec   = 19.5[dB]
Irr_atn_noe   = 19.5[dB]
Irr_noise     = WALTZ
Decoupling    = TRUE
Initial_wait  = 1[s]
Noe           = TRUE
Noe_time      = 1[s]
Recvr_gain    = 58
Relaxation_delay = 1[s]
Repetition_time = 1.83361792[s]
Temp_get      = 20.7[dC]
    
```

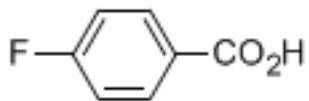
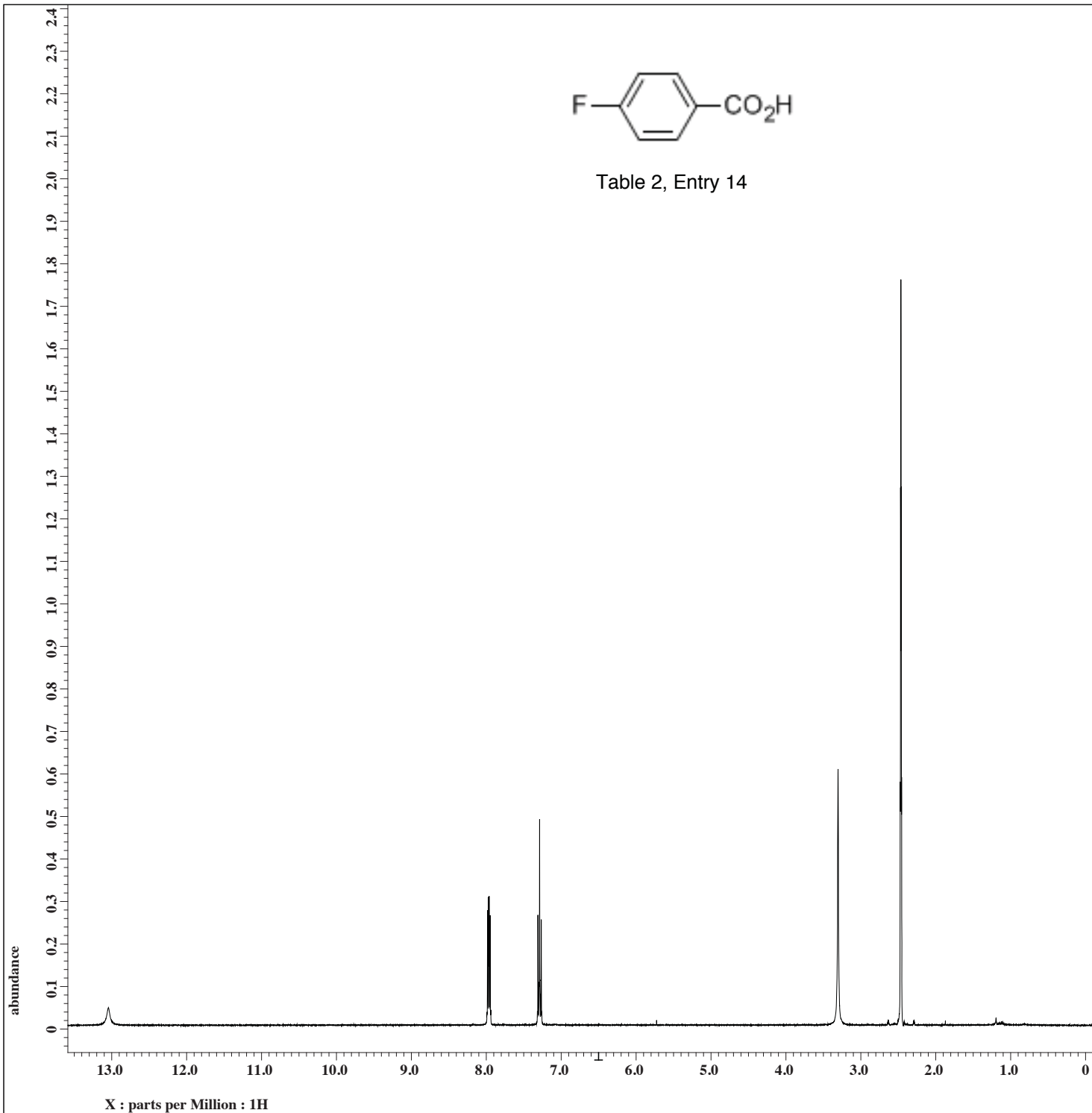


Table 2, Entry 14



Filename = 2-143-1-H-1-2.jdf
Author = daugulis
Experiment = single_pulse.ex2
Sample_id = 5
Solvent = DMSO-D6
Creation_time = 5-APR-2012 16:16:35
Revision_time = 22-MAY-2012 13:33:20
Current_time = 22-MAY-2012 13:34:10

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 = 42
Relaxation_delay = 1[s]
Repetition_time = 3.18365952[s]
Temp_get = 19.8[dc]

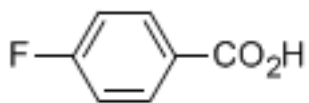
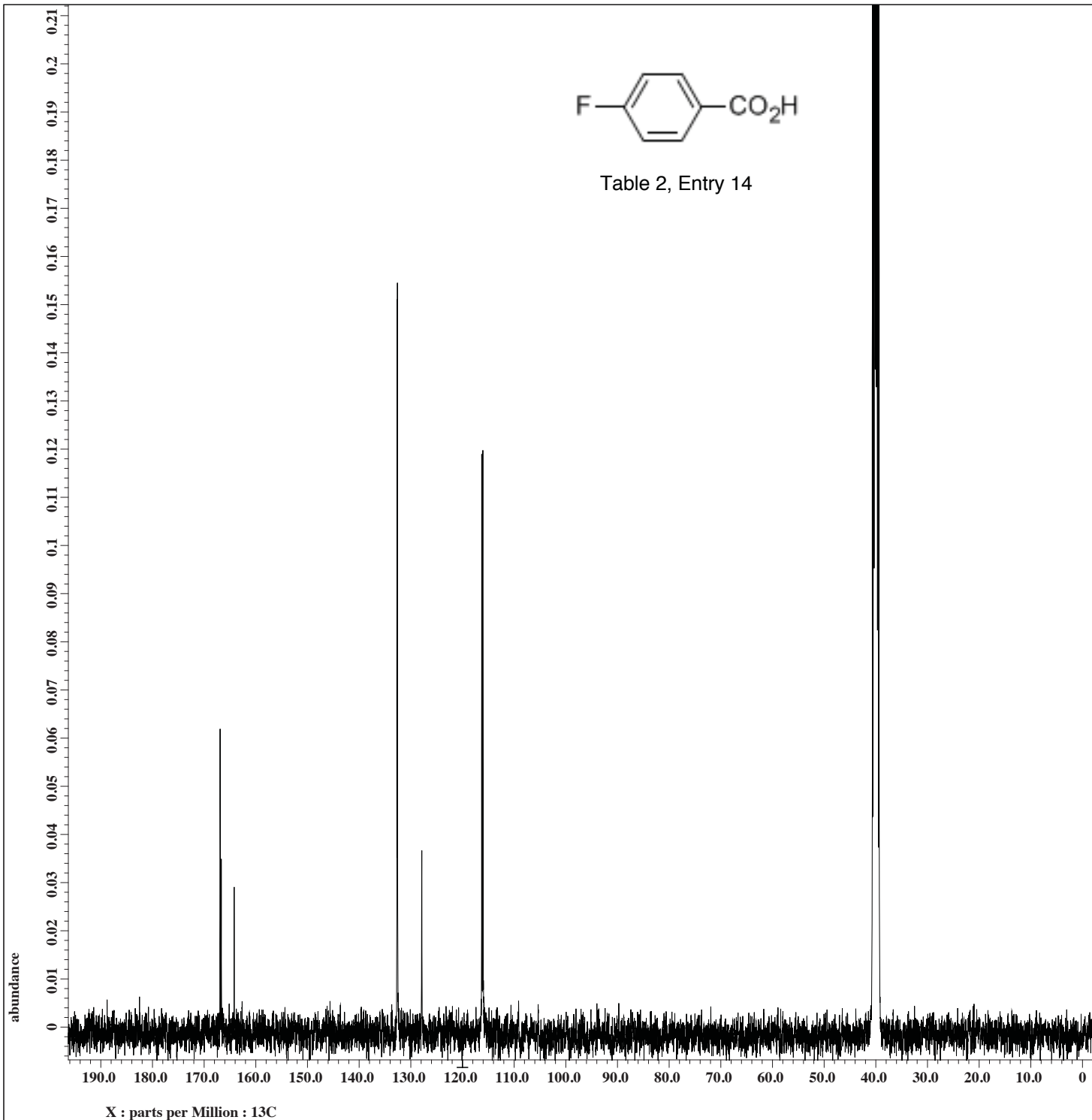


Table 2, Entry 14



```

Filename      = 2-143-1-C-2.jdf
Author       = daugulis
Experiment    = single_pulse_dec
Sample_id    = 5
Solvent      = DMSO-D6
Creation_time = 4-APR-2012 22:10:30
Revision_time = 22-MAY-2012 13:12:55
Current_time  = 22-MAY-2012 13:14:04

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        = 1903
Total_scans  = 1903

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]
  
```

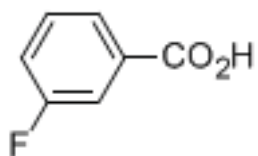
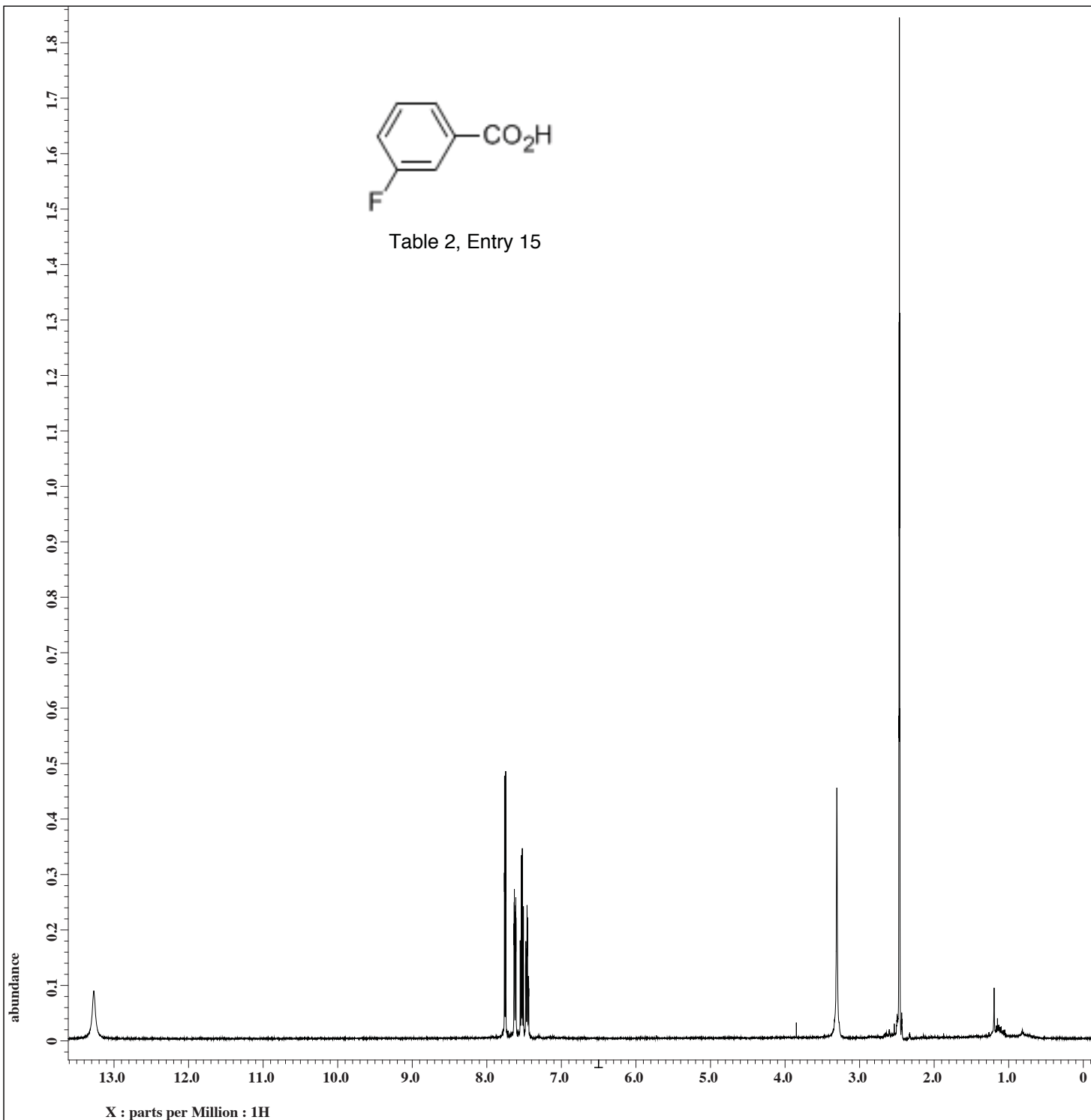


Table 2, Entry 15



```

Filename      = 2-120-1-H-2.jdf
Author       = daugulis
Experiment   = single_pulse.ex2
Sample_id    = S#558189
Solvent      = DMSO-D6
Creation_time = 7-APR-2012 14:35:14
Revision_time = 22-MAY-2012 13:35:57
Current_time = 22-MAY-2012 13:36:32
  
```

```

Comment      = single_pulse
Data_format  = 1D_COMPLEX
Dim_size     = 13107
Dim_title    = 1H
Dim_units    = [ppm]
Dimensions   = X
Site         = ECA 500
Spectrometer = ECA500
  
```

```

Field_strength = 11.7473579[T] (500[MH
X_acq_duration = 1.74587904[s]
X_domain       = 1H
X_freq         = 500.15991521[MHz]
X_offset       = 6.5[ppm]
X_points       = 16384
X_prescans     = 1
X_resolution   = 0.57277737[Hz]
X_sweep        = 9.38438438[kHz]
Irr_domain     = 1H
Irr_freq       = 500.15991521[MHz]
Irr_offset     = 5.0[ppm]
Tri_domain     = 1H
Tri_freq       = 500.15991521[MHz]
Tri_offset     = 5.0[ppm]
Clipped        = FALSE
Mod_return     = 1
Scans          = 32
Total_scans    = 32
  
```

```

X_90_width    = 12.84[us]
X_acq_time     = 1.74587904[s]
X_angle        = 45[deg]
X_atn          = 3[dB]
X_pulse        = 6.42[us]
Irr_mode       = Off
Tri_mode       = Off
Dante_presat   = FALSE
Initial_wait   = 1[s]
Recvr_gain     = 48
Relaxation_delay = 1[s]
Repetition_time = 2.74587904[s]
Temp_get       = 19.9[dc]
  
```

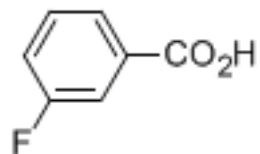
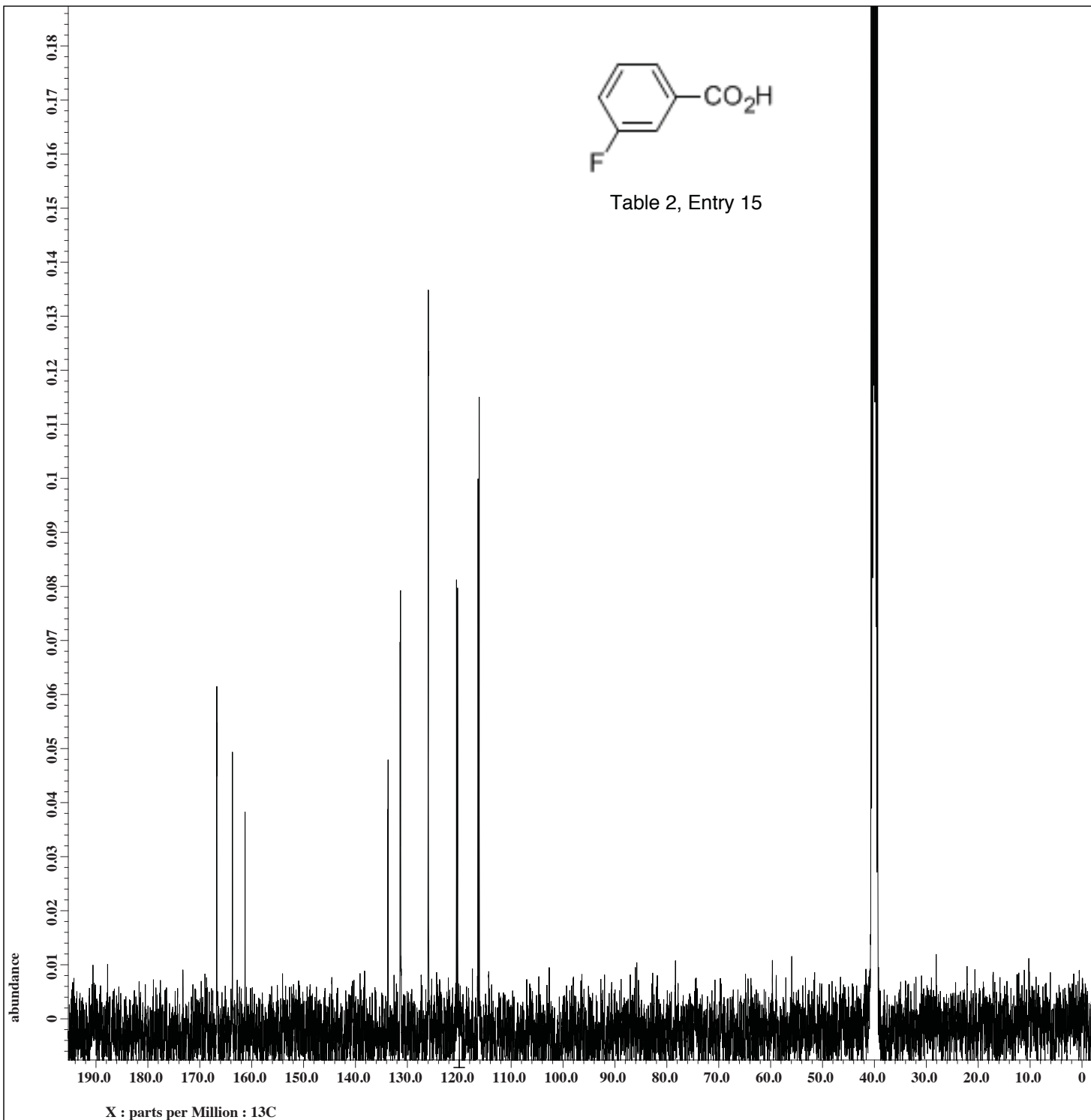


Table 2, Entry 15



```

Filename           = 2-120-1-C-2.jdf
Author            = daugulis
Experiment        = single_pulse_dec
Sample_id         = 9
Solvent           = DMSO-D6
Creation_time     = 5-APR-2012 17:06:19
Revision_time    = 22-MAY-2012 13:14:39
Current_time      = 22-MAY-2012 13:15:19

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         = 20.2[degC]
  
```

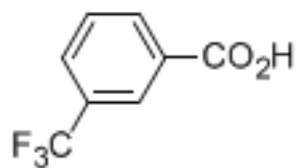
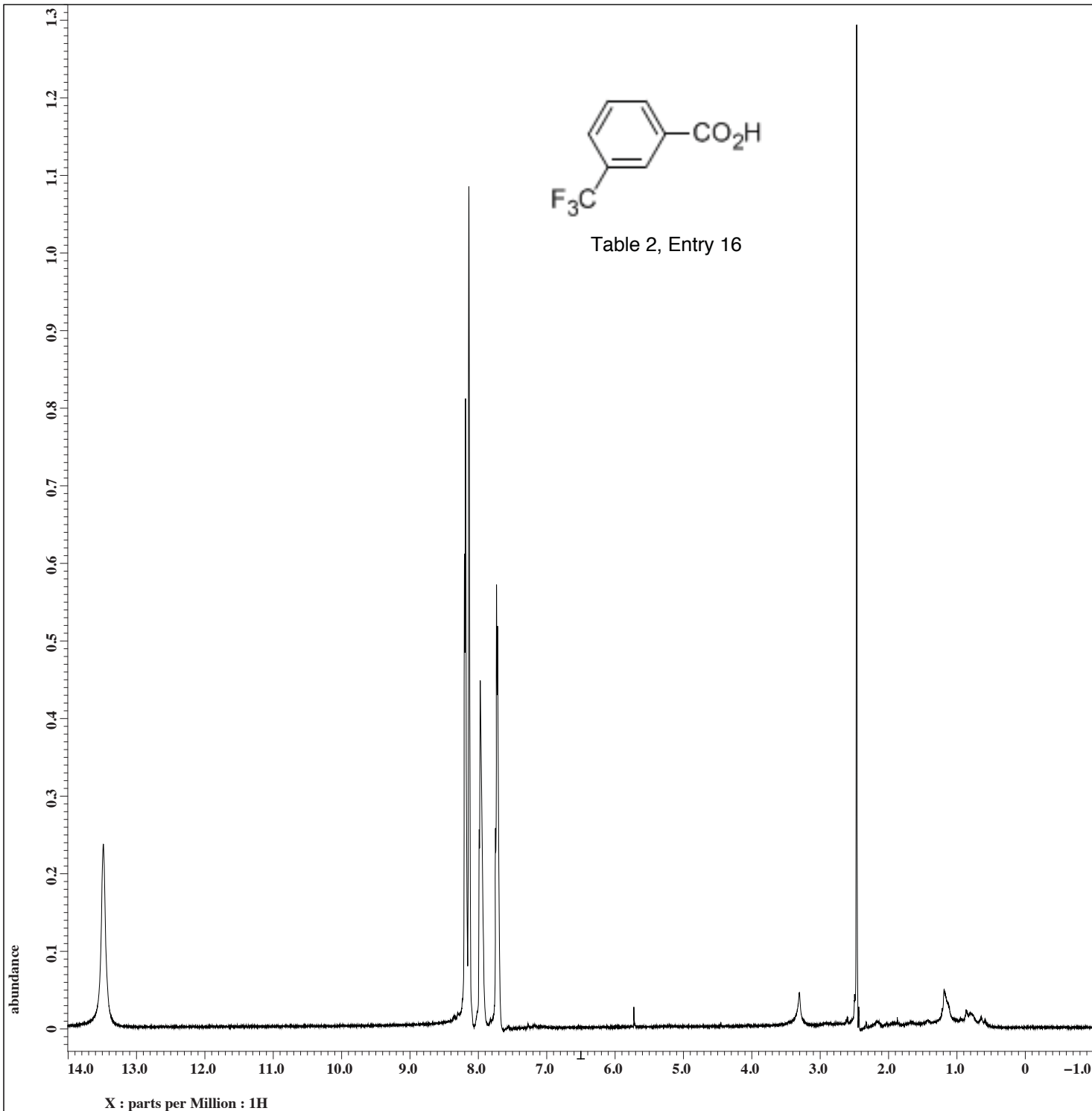


Table 2, Entry 16



Filename = 2-147-1-H-2.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 3
 Solvent = DMSO-D6
 Creation_time = 4-APR-2012 18:27:46
 Revision_time = 22-MAY-2012 13:37:21
 Current_time = 22-MAY-2012 13:37:50

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

Field_strength = 11.7473579[T] (500[MH
 X_acq_duration = 1.74587904[s]
 X_domain = 1H
 X_freq = 500.15991521[MHz]
 X_offset = 6.5[ppm]
 X_points = 16384
 X_prescans = 1
 X_resolution = 0.57277737[Hz]
 X_sweep = 9.38438438[kHz]
 Irr_domain = 1H
 Irr_freq = 500.15991521[MHz]
 Irr_offset = 5.0[ppm]
 Tri_domain = 1H
 Tri_freq = 500.15991521[MHz]
 Tri_offset = 5.0[ppm]
 Clipped = FALSE
 Mod_return = 1
 Scans = 24
 Total_scans = 24

X_90_width = 12.84[us]
 X_acq_time = 1.74587904[s]
 X_angle = 45[deg]
 X_atn = 3[dB]
 X_pulse = 6.42[us]
 Irr_mode = Off
 Tri_mode = Off
 Dante_presat = FALSE
 Initial_wait = 1[s]
 Recvr_gain = 46
 Relaxation_delay = 1[s]
 Repetition_time = 2.74587904[s]
 Temp_get = 20[dC]

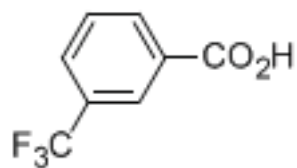
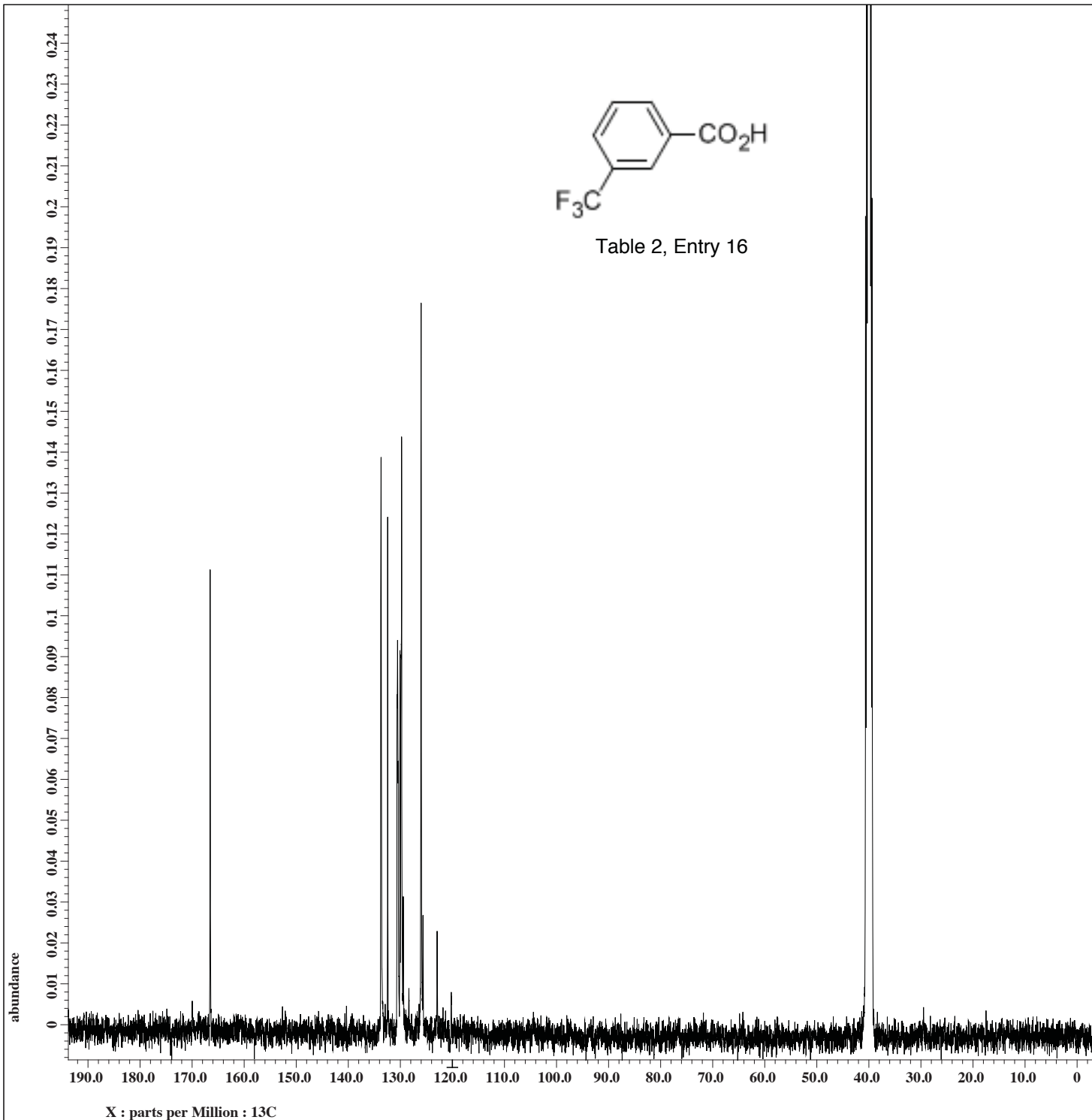


Table 2, Entry 16



```

Filename      = 2-147-1-C-2.jdf
Author       = daugulis
Experiment    = single_pulse_dec
Sample_id    = 3
Solvent      = DMSO-D6
Creation_time = 4-APR-2012 20:44:47
Revision_time = 22-MAY-2012 13:16:09
Current_time  = 22-MAY-2012 13:16:47

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      = TRUE
Mod_return   = 1
Scans        = 2560
Total_scans  = 2560

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]
    
```

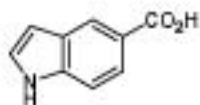
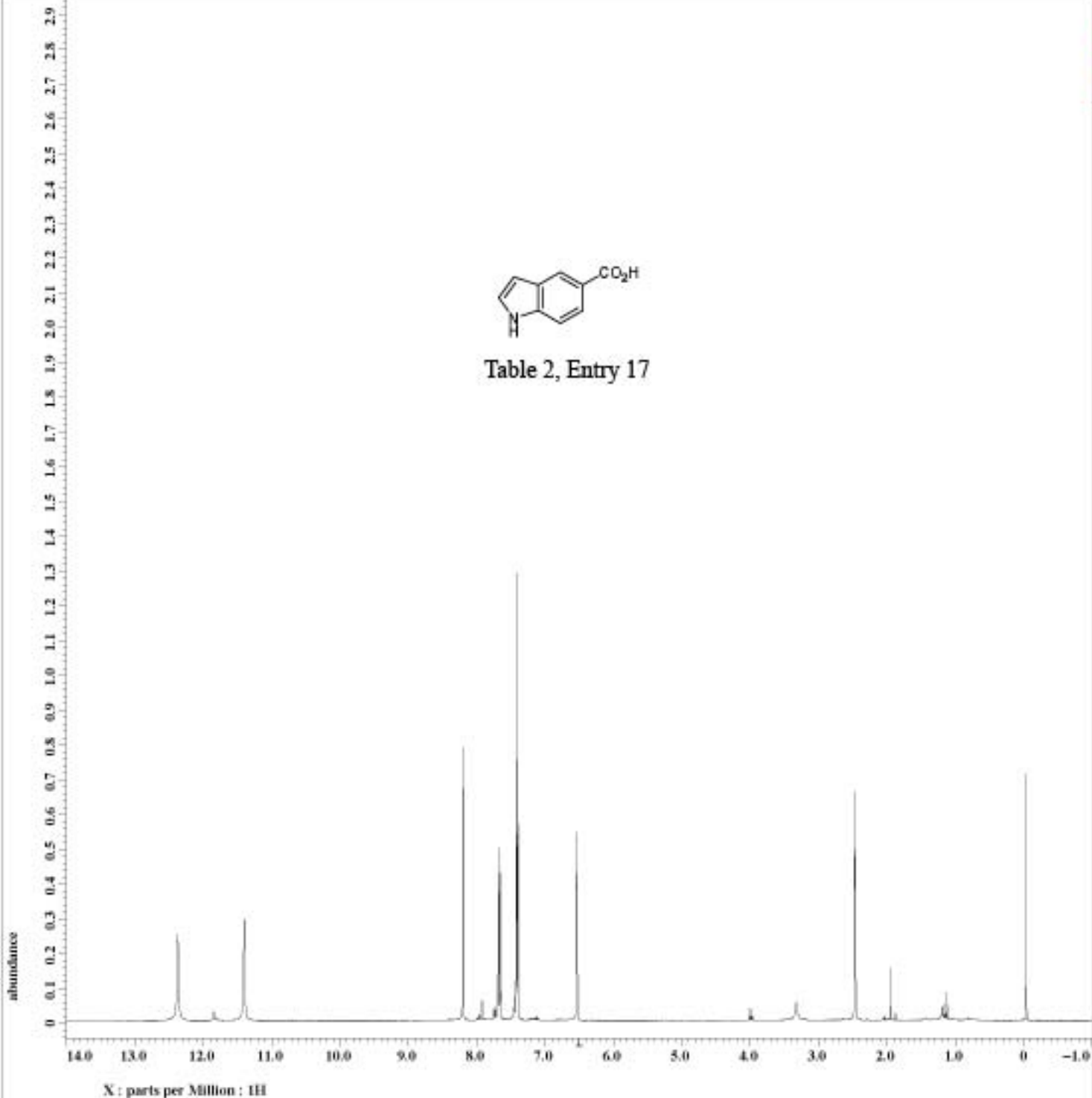


Table 2, Entry 17



```

Filename      = 3-37-1-N-2.jdf
Author       = daugulis
Experiment   = single_pulse.ex2
Sample id    = 3
Solvent      = CHLOROFORM-D
Creation time = 3-JUN-2013 19:20:58
Revision time = 11-JUN-2013 17:01:14
Current_time = 11-JUN-2013 17:02:16

Comment      = single_pulse
Data_format  = 1D COMPLEX
Bin_size     = 13107
Bin_title    = 1H
Bin_units    = [ppm]
Dimensions   = X
Site         = ECX 400P
Spectrometer = DELTA2_MMR

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         = 24
Total_scans   = 24

X_90_width    = 14[us]
X_acq_time     = 2.18365952[s]
X_angle       = 45[deg]
X_atn         = 3[dB]
X_pulse       = 7[us]
Irr_mode      = Off
Tri_mode      = Off
Dante_preset  = FALSE
Initial_wait  = 1[s]
Recvr_gain    = 36
Relaxation_delay = 1[s]
Repetition_time = 3.18365952[s]
Temp_get      = 408.4[4C]
    
```

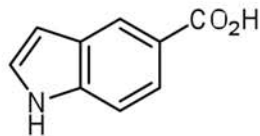
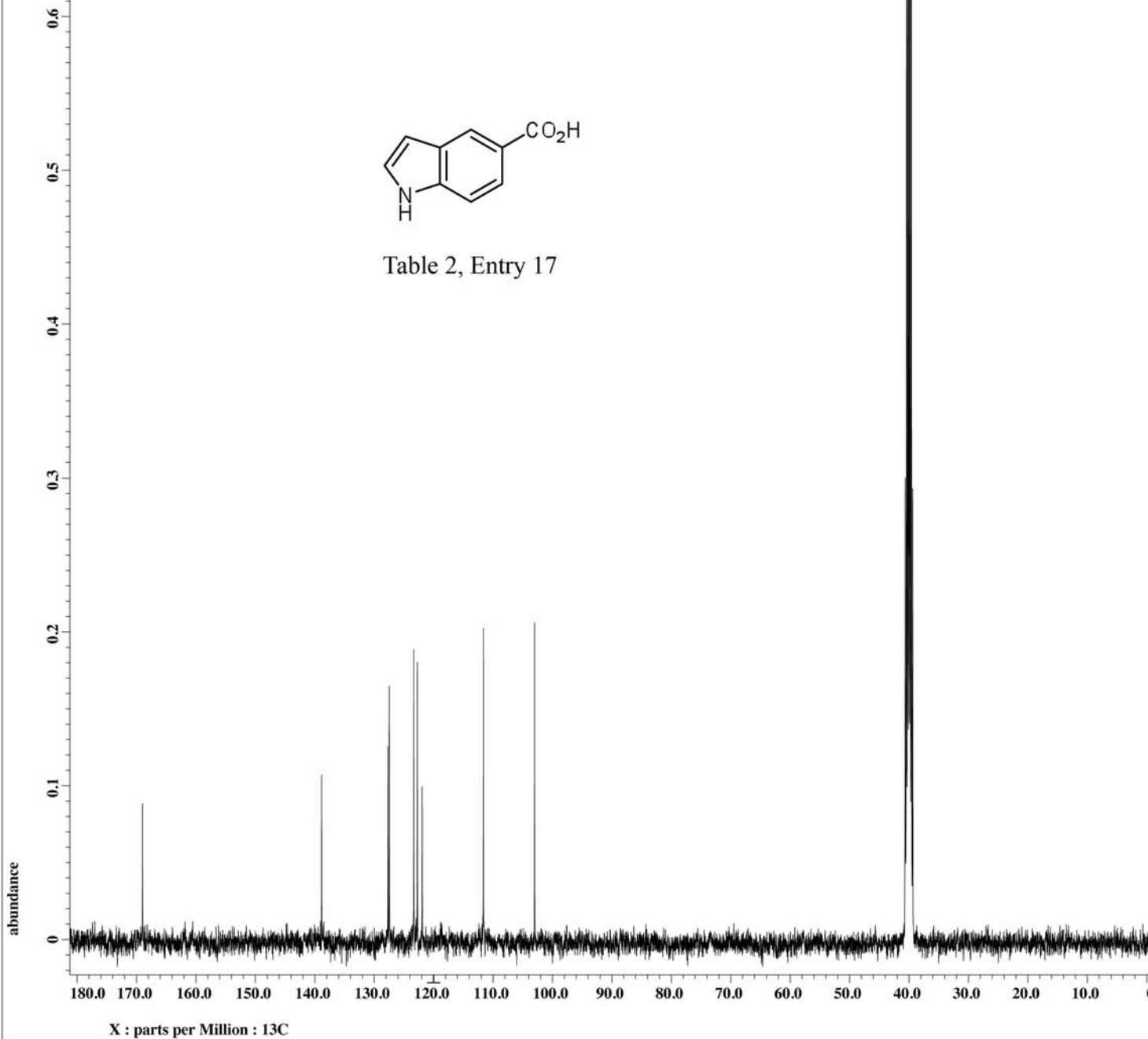


Table 2, Entry 17



Filename = 3-37-1-C-2.jdf
 Author = daugulis
 Experiment = single_pulse_dec
 Sample_id = 3
 Solvent = CHLOROFORM-D
 Creation_time = 3-JUN-2013 19:43:02
 Revision_time = 11-JUN-2013 17:05:46
 Current_time = 11-JUN-2013 17:06:58

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 = 627
 Total_scans = 627

X_90_width = 13[us]
 X_acq_time = 1.04333312[s]
 X_angle = 30[deg]
 X_atn = 6[dB]
 X_pulse = 4.33333333[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 = 408.6[dC]

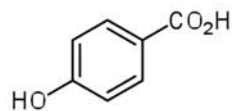
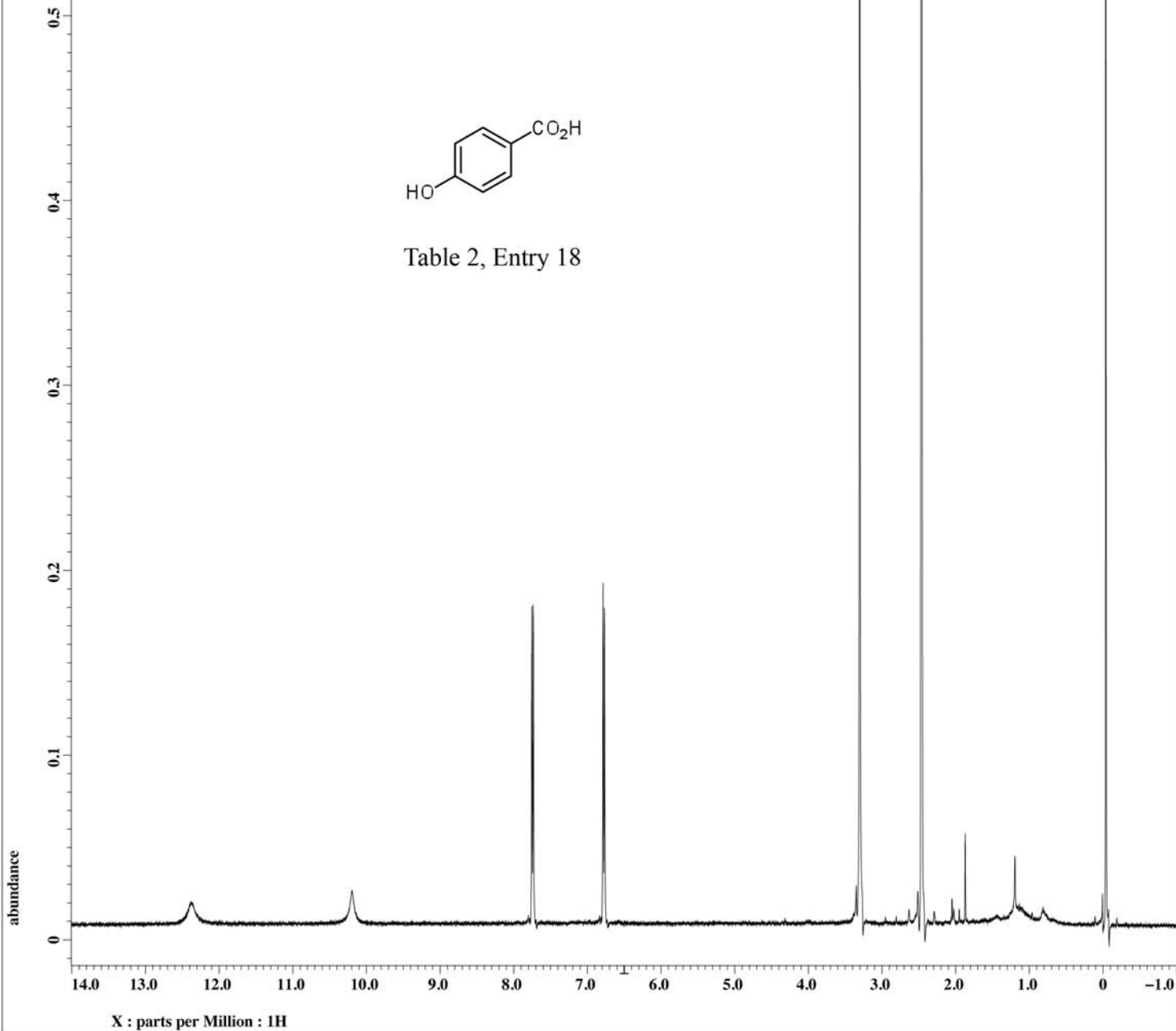


Table 2, Entry 18



Filename = 3-28-3-3.jdf
 Author = daugulis
 Experiment = single_pulse.ex2
 Sample_id = 1
 Solvent = DMSO-D6
 Creation_time = 12-SEP-2012 15:14:15
 Revision_time = 11-JUN-2013 17:07:23
 Current_time = 11-JUN-2013 17:09:16

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 = 24
 Total_scans = 24

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 = 42
 Relaxation_delay = 1[s]
 Repetition_time = 3.18365952[s]
 Temp_get = 20.1[dC]

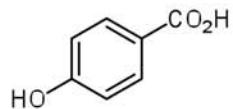
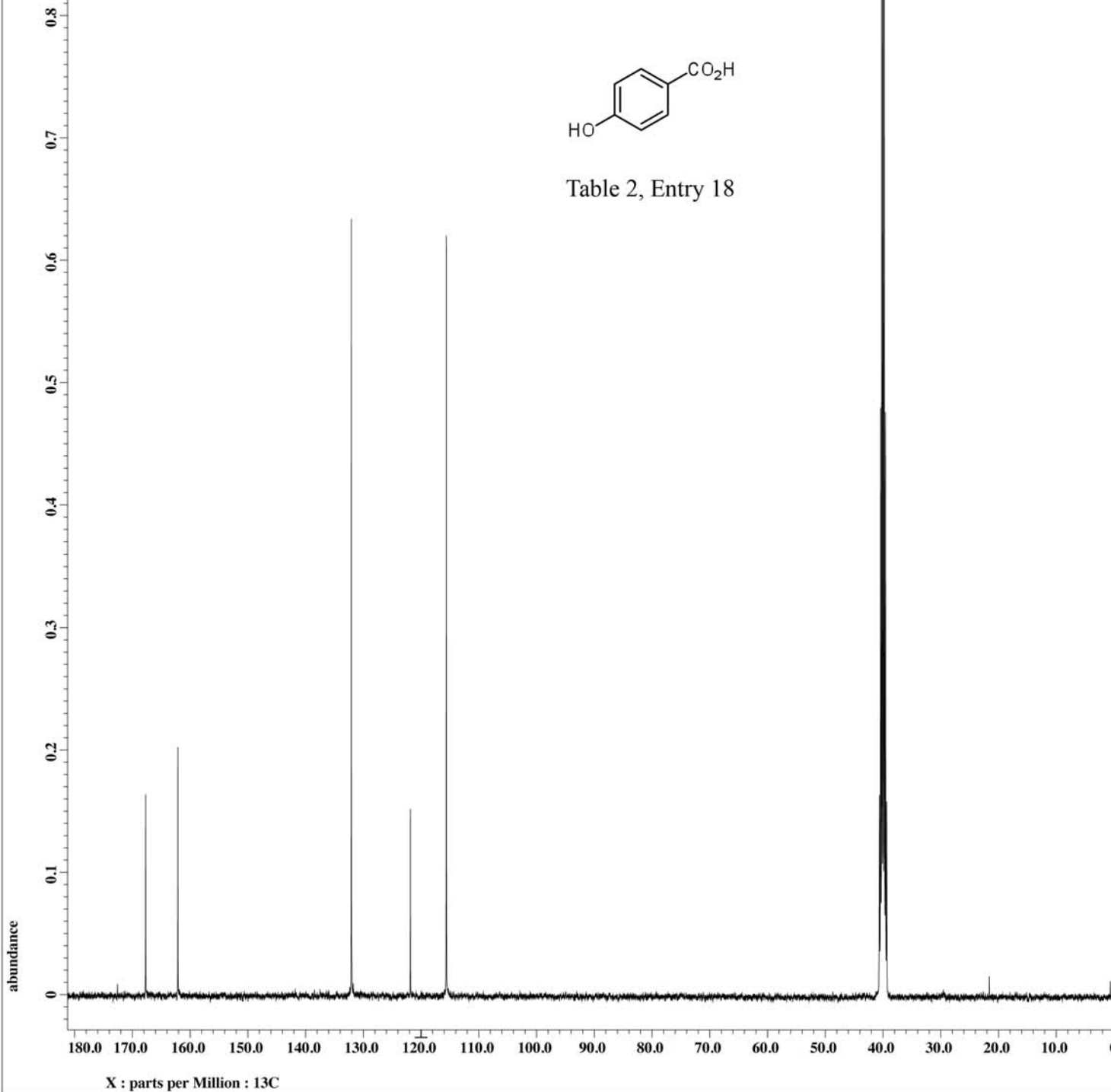


Table 2, Entry 18



```

Filename      = 3-28-3-C-1-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 5
Solvent      = DMSO-D6
Creation time = 15-SEP-2012 17:02:00
Revision_time = 11-JUN-2013 17:09:30
Current_time  = 11-JUN-2013 17:10:39

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       = TRUE
Mod_return    = 1
Scans         = 3100
Total_scans   = 3100

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      = 20.4[dC]
    
```

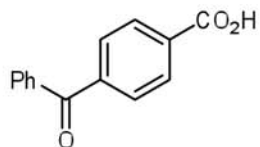
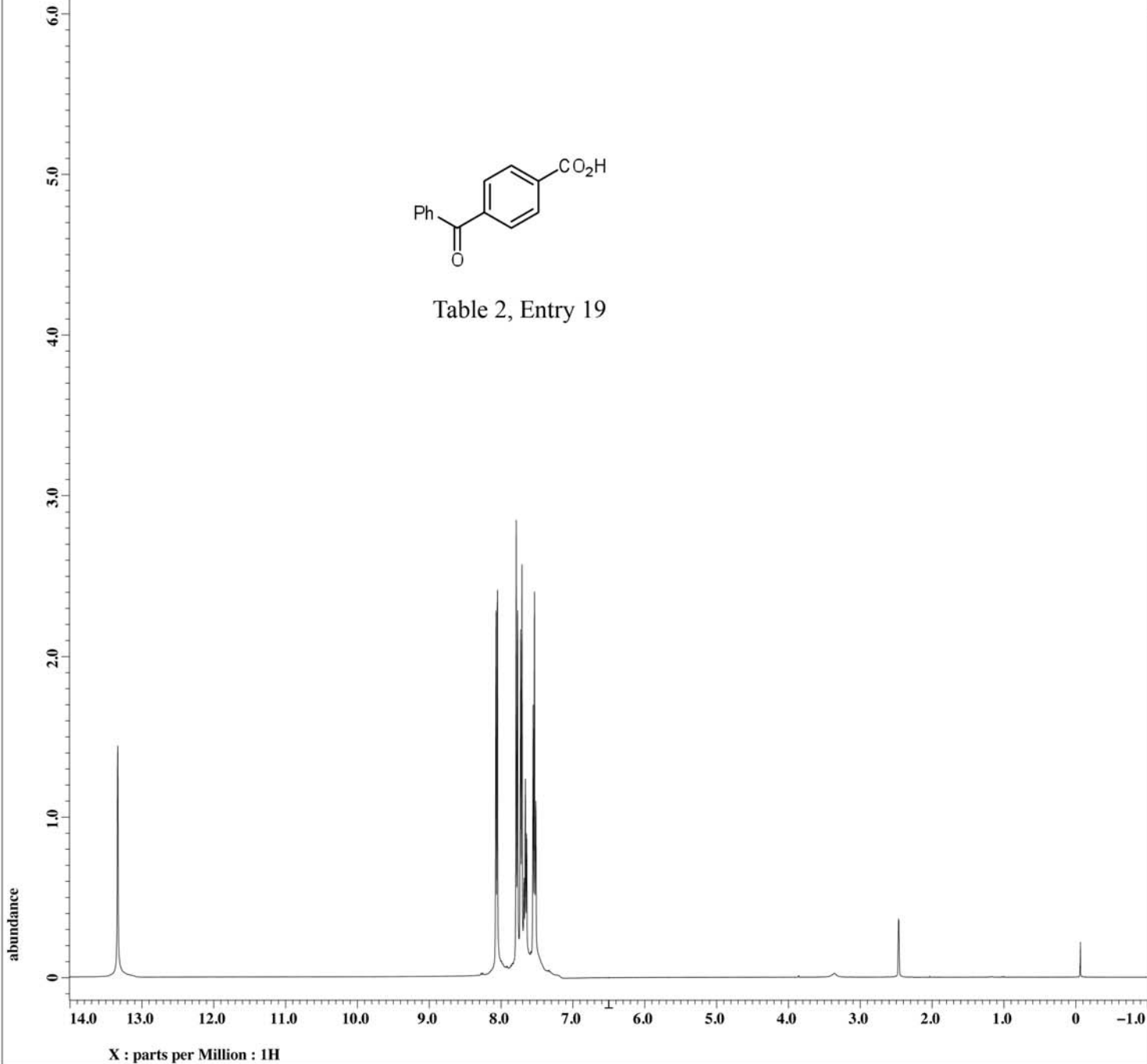


Table 2, Entry 19



```

Filename      = 3-43-2-H-2.jdf
Author       = daugulis
Experiment   = single_pulse.ex2
Sample_id    = 3
Solvent      = DMSO-D6
Creation_time = 4-JUN-2013 14:39:02
Revision_time = 11-JUN-2013 17:10:57
Current_time  = 11-JUN-2013 17:11:21

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         = 24
Total_scans   = 24

X_90_width    = 14[us]
X_acq_time    = 2.18365952[s]
X_angle       = 45[deg]
X_atn         = 3[dB]
X_pulse       = 7[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      = 408.7[dC]
    
```

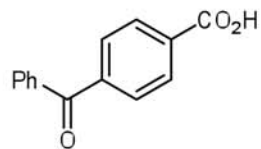
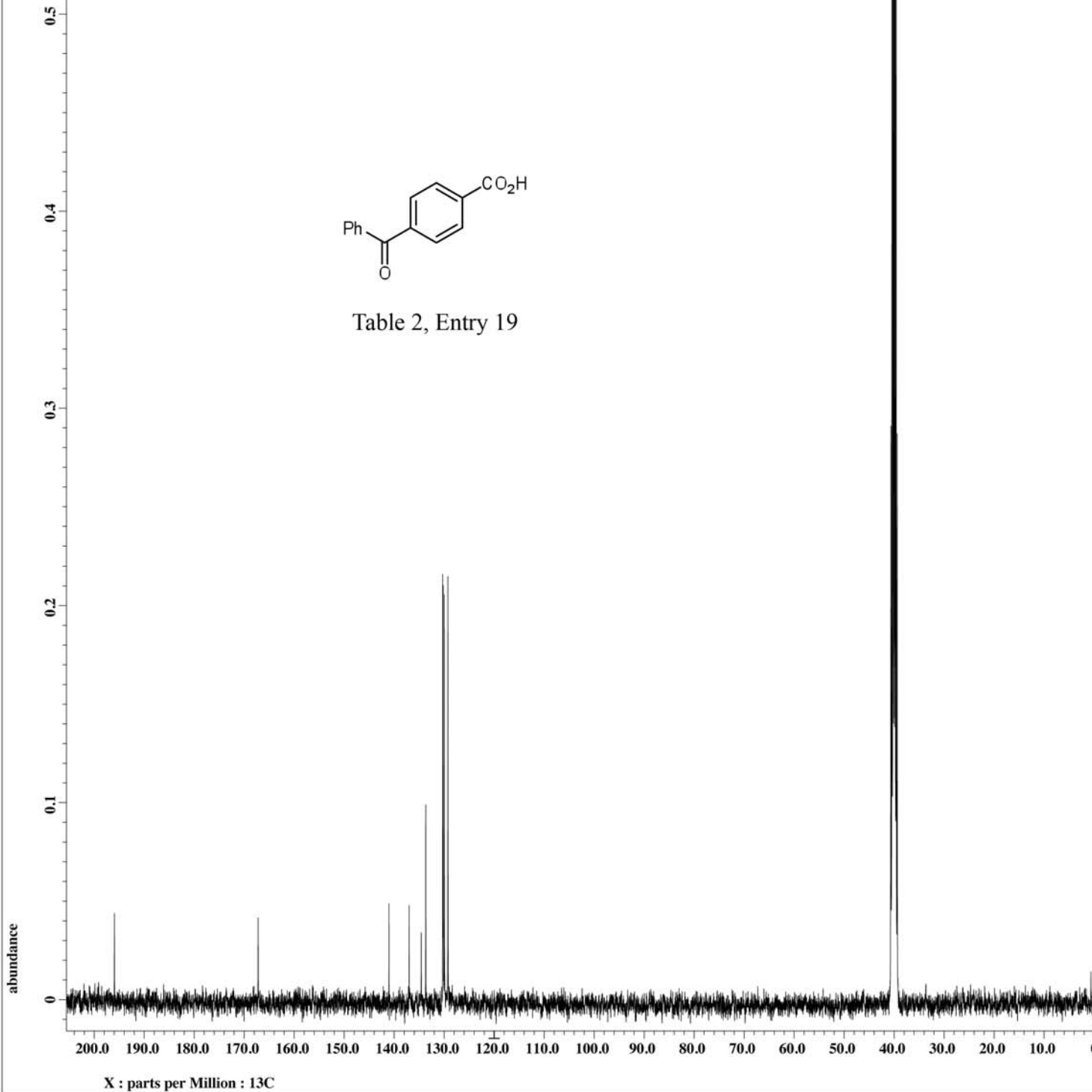


Table 2, Entry 19



```

Filename      = 3-43-1-C-2.jdf
Author       = daugulis
Experiment   = single_pulse_dec
Sample_id    = 3
Solvent      = DMSO-D6
Creation time = 3-JUN-2013 20:33:56
Revision_time = 11-JUN-2013 17:11:31
Current_time  = 11-JUN-2013 17:11:55

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         = 1152
Total_scans   = 1152

X_90_width    = 13[us]
X_acq_time    = 1.04333312[s]
X_angle       = 30[deg]
X_atn         = 6[dB]
X_pulse       = 4.33333333[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      = 408.4[dC]
    
```