

Electrochemical synthesis of sulfonamides from arenesulfonohydrazides or sodium *p*-methylbenzenesulfinate and amines

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General information

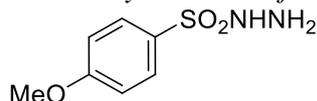
Electrolysis was carried out using manufactured in Russia DC regulated power supply MEGEON 30305 (voltage capability 0-30 V, current capability 0-5 A, voltage stability $\leq 0.2\%$, current stability $\leq 0.8\%$, current limitation and short-circuit protection). Carbon plate (3.4 cm \times 2.2 cm \times 0.3 cm) was employed as anode and iron plate (3.4 cm \times 2.2 cm \times 0.1 cm) was employed as cathode. A glassy wide-necked tube was applied as undivided electrochemical cell. NMR spectra were recorded on a commercial instrument (300.13 MHz for ^1H , 75.48 MHz for ^{13}C) in CDCl_3 and DMSO-d_6 . Mass-spectra were recorded on a commercial quadrupole mass spectrometer (direct input, electron impact ionization, ionization energy 70 eV). The TLC analyses were carried out on standard silica-gel chromatography plates. The melting points were determined on a Kofler hot-stage apparatus.

Preparation of arenesulfonhydrazides

Arenesulfonhydrazides **1b-f** were prepared according to the literature procedure.¹ A tetrahydrofuran (20 ml) solution containing corresponding arenesulfonyl chloride (5 g, 17-25 mmol) was cooled in an ice-water bath to 5 °C. A 64% w/w water solution of hydrazine hydrate (8.5-12.5 g; 0.17-0.25 mol) was slowly added while stirring. Following this addition, the reaction mixture was stirred at 5 °C for 30 min, and then the tetrahydrofuran was evaporated at reduced pressure (10-20 torr) and room temperature. The white solid product was dried on air overnight to give pure products.

Analytical data for arenesulfonhydrazides 1b-f

4-Methoxybenzenesulfonhydrazide, **1b**²



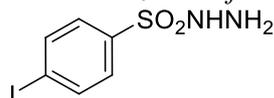
Yield 91%

Colourless crystals, mp 107-109°C (lit.² mp = 105-110°C)

^1H NMR (DMSO-d_6), δ : 3.83 (s, 3 H), 3.92 (br s, 2 H), 7.12 (d, $J = 8.8$ Hz, 2 H), 7.74 (d, $J = 8.8$, 2 H), 8.21 (s, 1 H).

^{13}C NMR (DMSO-d_6), δ : 55.7, 114.2, 129.5, 129.8, 162.4.

4-Iodobenzenesulfonhydrazide, **1c**³



Yield 88%

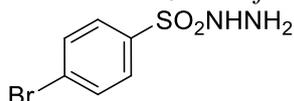
Colourless crystals, mp 173-175°C (lit.³ mp = 162°C)

^1H NMR (DMSO-d_6), δ : 4.16 (br s, 2 H), 7.56 (d, $J = 8.4$ Hz, 2 H), 7.98 (d, $J = 8.9$ Hz, 2 H), 8.46 (s, 1 H).

^{13}C NMR (DMSO-d_6), δ : 100.7, 129.4, 137.8, 137.9.

Calculated for $\text{C}_6\text{H}_7\text{IN}_2\text{O}_2\text{S}$ C: 24.17 %, H: 2.37 %, I: 42.57 %, N: 9.40 %, S: 10.76 %. Found C: 24.23 %, H: 2.45 %, I: 42.18 %, N: 9.21 %, S: 10.57 %.

4-Bromobenzenesulfonohydrazide, 1d⁴



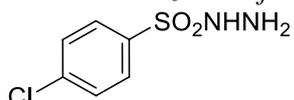
Yield 89%

Colourless crystals, mp 114-116°C (lit.⁴ mp = 113-114°C)

¹H NMR (DMSO-d₆), δ: 4.20 (br s, 2 H), 7.73 (d, *J* = 8.6 Hz, 2 H), 7.80 (d, *J* = 8.6 Hz, 2 H), 8.50 (s, 1 H).

¹³C NMR (DMSO-d₆), δ: 126.5, 129.7, 132.0, 137.5.

4-Chlorobenzenesulfonohydrazide, 1e⁵



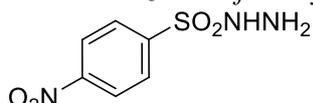
Yield 88%

Colourless crystals, mp 112-114°C (lit.⁵ mp = 112-114°C)

¹H NMR (DMSO-d₆), δ: 4.13 (br s, 2 H), 7.65 (d, *J* = 8.8 Hz, 2 H), 7.79 (d, *J* = 8.8 Hz, 2 H), 8.47 (s, 1 H).

¹³C NMR (DMSO-d₆), δ: 129.2, 129.6, 137.2, 137.5.

4-Nitrobenzenesulfonohydrazide, 1f²



Yield 85%

Yellow crystals, mp 150-152°C (lit.² mp = 152-157°C)

¹H NMR (DMSO-d₆): δ 4.34 (br s, 2 H), 8.05 (d, *J* = 8.9 Hz, 2 H), 8.42 (d, *J* = 8.9 Hz, 2 H), 8.75 (s, 1 H).

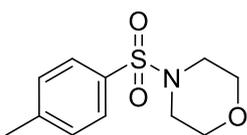
¹³C NMR (DMSO-d₆): δ 124.2, 129.2, 144.2, 149.7.

General procedure for Table 1. Electrochemical synthesis of sulfonamide 3aa from *p*-toluenesulfonohydrazide 1a and morpholine 2a.

An undivided cell was equipped with a carbon plate anode (7.5 cm²) and a Fe plate cathode (7.5 cm²) and connected to a DC regulated power supply. The solution of morpholine **2a** (1.61-4.83 mmol) in 30 ml (MeCN:H₂O = 1:1), *p*-toluenesulfonohydrazide **1a** (300 mg, 1.61 mmol) and supporting electrolyte KI, NaI, NH₄I, KBr, NaBr, NH₄Br, NH₄Cl (0.8 mmol) were added to the cell. The mixture was electrolyzed using constant current conditions (35-40 mA/cm²) at temperature 25-30°C under magnetic stirring. Then the solvent was removed under reduced pressure (10-20 torr). The residue was diluted with EtOAc (50 ml) and washed with brine (2×8 ml) and water (2×8 ml), dried over Na₂SO₄, and concentrated under reduced pressure (10-20 torr). The residue was purified by recrystallization from ethanol.

Analytical data for sulfonamides 3aa-fa

4-(*p*-Methylbenzenesulfonyl)morpholine, 3aa⁶



Yield 95%

Colourless crystals, mp 144.5-146°C (lit.⁶ mp = 145-147°C)

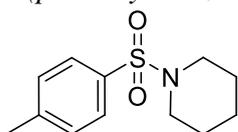
R_f = 0.35 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ: 2.42 (s, 3H), 2.94-2.97 (m, 4H), 3.69-3.72 (m, 4H), 7.32 (d, *J* = 8.1 Hz, 2 H), 7.61 (d, *J* = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ: 21.4, 45.9, 66.0, 127.8, 129.7, 132.1, 143.9.

m/z 241 (M⁺, 48%), 198 (14), 155 (31), 106 (11), 91 (48), 86 (100), 65 (28), 56 (66), 42 (11), 29 (18), 28 (33).

*1-(p-Methylbenzenesulfonyl)piperidine, 3ab*⁷



Yield 93%

Colourless crystals, mp 95-96°C (lit.⁷ mp = 95-97°C)

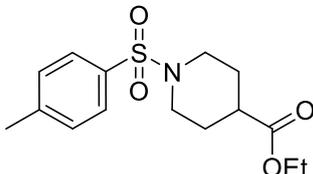
R_f = 0.54 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ: 1.34-1.40 (m, 2H), 1.57-1.65 (m, 4H), 2.40 (s, 3H), 2.94 (t, *J* = 5.2 Hz, 4H), 7.29 (d, *J* = 8.1 Hz, 2 H), 7.61 (d, *J* = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ: 21.4, 23.5, 25.1, 46.9, 127.6, 129.5, 133.3, 143.2.

m/z 239 (M⁺, 100%), 238 (67), 155 (31), 91 (52), 84 (81), 83 (31), 65 (15), 55 (12), 42 (18).

*Ethyl 1-(p-methylbenzenesulfonyl)piperidine-4-carboxylate, 3ac*⁸



Yield 98%

Colourless crystals, mp 101.5-102.5°C

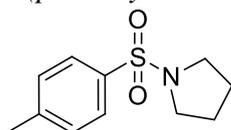
R_f = 0.42 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ: 1.19 (t, *J* = 5.2 Hz, 3H), 1.72-1.85 (m, 2H), 1.91-1.97 (m, 2H), 2.17-2.25 (m, 1H), 2.41-2.48 (m, 5H), 3.58-3.62 (m, 2H), 4.08 (q, *J* = 5.2 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2 H), 7.61 (d, *J* = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ: 14.1, 21.5, 27.4, 40.0, 45.4, 60.6, 127.6, 129.6, 133.1, 143.5, 173.8.

m/z 156 (M⁺-MeC₆H₄SO₂, 100%), 91 (26), 82 (50), 29 (10).

*1-(p-Methylbenzenesulfonyl)pyrrolidine, 3ad*⁹



Yield 94%

Colourless crystals, mp 115-116°C (lit.⁹ mp = 116°C)

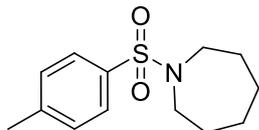
R_f = 0.48 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ: 1.70-1.74 (m, 4H), 2.40 (s, 3H), 3.18-3.23 (m, 4H), 7.29 (d, *J* = 8.1 Hz, 2 H), 7.69 (d, *J* = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ: 21.4, 25.1, 47.8, 127.5, 129.6, 133.9, 143.2.

m/z 225 (M^+ , 60%), 224 (24), 155 (11), 92 (32), 91 (92), 89 (14), 70 (100), 65 (15), 43 (11), 42 (46), 41 (18), 28 (16).

*1-(p-Methylbenzenesulfonyl)azepane, 3ae*¹⁰



Yield 90%

Colourless crystals, mp 70-72°C (lit.¹⁰ mp = 68-72°C)

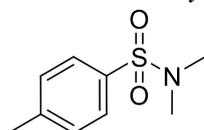
R_f = 0.57 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ : 1.59-1.72 (m, 8H), 2.42 (s, 3H), 3.24-3.28 (m, 4H), 7.29 (d, J = 8.1 Hz, 2 H), 7.68 (d, J = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ : 21.4, 26.8, 29.0, 48.1, 126.8, 129.5, 136.5, 142.8.

m/z 253 (M^+ , 47%), 155 (17), 98 (100), 92 (12), 91 (74), 69 (17), 68 (14), 65 (24), 55 (10), 44 (17), 42 (51), 41 (26).

*N,N,4-Trimethylbenzenesulfonamide, 3af*¹¹



Yield 90%

Colourless crystals, mp 76-77°C (lit.¹¹ mp = 79-81°C)

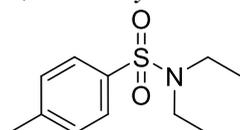
R_f = 0.42 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ : 2.41 (s, 3H), 2.66 (s, 6H), 7.31 (d, J = 8.1 Hz, 2 H), 7.63 (d, J = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ : 21.4, 37.9, 127.7, 129.5, 132.3, 143.4.

m/z 199 (M^+ , 55%), 155 (21), 92 (24), 91 (100), 65 (24), 44 (48), 43 (10), 42 (33), 39 (15).

*N,N-Diethyl-4-methylbenzenesulfonamide, 3ag*⁹



Yield 63%

Colourless crystals, mp 52.5-53.5°C (lit.⁹ mp = 48-49°C)

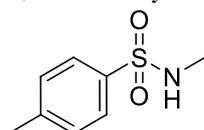
R_f = 0.61 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ : 1.13 (t, J = 7.3 Hz, 6 H), 2.43 (s, 3H), 3.23 (q, J = 7.3 Hz, 4 H), 7.29 (d, J = 8.1 Hz, 2 H), 7.70 (d, J = 8.1 Hz, 2 H).

¹³C NMR (CDCl₃), δ : 14.1, 21.4, 42.0, 127.0, 129.5, 137.4, 142.8.

m/z 227 (M^+ , 25%), 212 (100), 155 (84), 91 (96), 65 (24), 56 (12), 44 (10), 42 (16), 29 (16).

*N,4-Dimethylbenzenesulfonamide, 3ah*¹²



Yield 90%

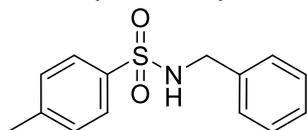
Colourless crystals, mp 70.5-72°C (lit.¹² mp = 71-72°C)

R_f = 0.26 (PE:EtOAc = 5:1)

¹H NMR (CDCl₃), δ : 2.41 (s, 3H), 2.61 (d, J = 5.1 Hz, 3 H), 4.68 (br s, 1H), 7.30 (d, J = 7.3 Hz, 2 H), 7.73 (d, J = 7.3 Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 21.4, 29.2, 127.2, 129.7, 135.7, 143.4.
 m/z 185 (M^+ , 39%), 155 (23), 121 (22), 92 (21), 91 (100), 65 (28), 39 (22), 30 (16).

N-Benzyl-4-methylbenzenesulfonamide, **3ai**¹³



Yield 56%

Yellow crystals, mp 105.5-107°C (lit.¹³ mp = 106-107°C)

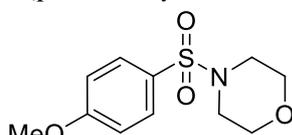
R_f = 0.44 (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 2.45 (s, 3H), 4.14 (s, 2H), 4.86 (br s, 1H), 7.20-7.33 (m, 7H), 7.77 (d, J = 8.0 Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 21.5, 47.2, 124.9, 127.2, 127.8, 128.6, 129.7, 136.3, 136.8, 143.5.

m/z 106 (M^+ - $\text{MeC}_6\text{H}_4\text{SO}_2$, 100%), 91 (43), 77 (12), 65 (12).

4-(*p*-Methoxybenzenesulfonyl)morpholine, **3ba**¹⁴



Yield 91%

Colourless crystals, mp 105-108°C (lit.¹⁴ mp = 112-114°C)

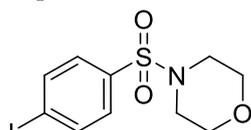
R_f = 0.19 (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 2.94-2.96 (m, 4H), 3.69-3.72 (m, 4H), 3.86 (s, 3H), 6.98 (d, J = 8.8 Hz, 2 H), 7.66 (d, J = 8.8 Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 45.9, 55.6, 66.0, 114.2, 126.6, 129.9, 163.2.

m/z 257 (M^+ , 100%), 171 (50), 122 (14), 107 (15), 92 (14), 86 (89), 77 (16), 56 (62), 28 (22).

4-(*p*-Iodobenzenesulfonyl)morpholine-1,4 **3ca**¹⁵



Yield 92%

Colourless crystals, mp 150.5-152°C (lit.¹⁵ mp = 149-151°C)

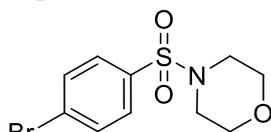
R_f = 0.40 (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 2.96-2.99 (m, 4H), 3.71-3.74 (m, 4H), 7.44 (d, J = 8.1 Hz, 2 H), 7.89 (d, J = 8.8 Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 45.9, 66.0, 100.6, 129.1, 134.8, 138.4.

m/z 353 (M^+ , 54%), 310 (12), 267 (18), 203 (26), 86 (100), 76 (33), 56 (74), 50 (15), 42 (15), 29 (12).

4-(*p*-Bromobenzenesulfonyl)morpholine-1,4, **3da**¹⁶



Yield 95%

Colourless crystals, mp 148.5-151°C (lit.¹⁶ mp = 151-153°C)

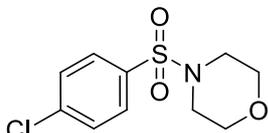
R_f = 0.42 (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 2.96-3.00 (m, 4H), 3.71-3.74 (m, 4H), 7.59 (d, $J = 8.1$ Hz, 2 H), 7.68 (d, $J = 8.8$ Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 45.9, 66.0, 128.2, 129.2, 132.4, 134.2.

m/z 307 (M^+ , 25%), 305 (25), 264 (10), 262 (10), 221 (17), 219 (17), 157 (29), 155 (32), 86 (93), 76 (21), 75 (18), 72 (12), 56 (100), 50 (13), 42 (22), 30 (17), 29 (26), 28 (40).

4-(*p*-Chlorobenzenesulfonyl)morpholine, **3ea**¹⁷



Yield 76%

Colourless crystals, mp 145-146°C (lit.¹⁷ mp = 146-147°C)

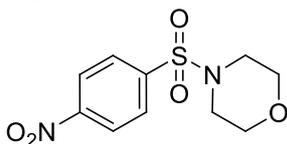
$R_f = 0.36$ (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 2.96-2.98 (m, 4H), 3.71-3.73 (m, 4H), 7.51 (d, $J = 8.8$ Hz, 2 H), 7.67 (d, $J = 8.8$ Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 45.8, 66.0, 129.1, 129.4, 133.6, 139.6.

m/z 261 (M^+ , 26%), 218 (11), 175 (21), 113 (12), 111 (40), 86 (92), 75 (28), 56 (100), 50 (10), 42 (22), 30 (16), 29 (26), 28 (40), 27 (12).

4-(*p*-Nitrobenzenesulfonyl)morpholine **3fa**¹⁶



Yield 73%

Yellow crystals, mp 160-161°C (lit.¹⁶ mp = 170-172°C)

$R_f = 0.26$ (PE:EtOAc = 5:1)

^1H NMR (CDCl_3), δ : 3.02-3.06 (m, 4H), 3.72-3.75 (m, 4H), 7.93 (d, $J = 8.8$ Hz, 2 H), 8.39 (d, $J = 8.8$ Hz, 2 H).

^{13}C NMR (CDCl_3), δ : 45.8, 66.0, 124.4, 128.9, 141.3, 150.3.

m/z 272 (M^+ , 100%), 241 (22), 229 (76), 186 (23), 122 (13), 86 (74), 76 (17), 56 (67), 30 (10).

General procedure for the optimization of synthesis of sulfonamide **3aa** from sodium *p*-methylbenzenesulfinate **4** and morpholine **2a**.

An undivided cell was equipped with a carbon plate anode (7.5 cm²) and a Fe plate cathode (7.5 cm²) and connected to a DC regulated power supply. The solution of morpholine **2a** (1.68-5.05 mmol) in 30 ml (MeCN:H₂O = 1:1), sodium *p*-methylbenzenesulfinate **4** (300 mg, 1.68 mmol) and supporting electrolyte KI, NH₄I, NH₄Br, NH₄Cl (0.84 mmol) were added to the cell. The mixture was electrolyzed with constant current (35-40 mA/cm²) at temperature 25-30°C under magnetic stirring. Then the solvent was removed under reduced pressure (10-20 torr). The residue was diluted with EtOAc (50 ml) and washed with brine (2×8 ml) and water (2×8 ml), dried over Na₂SO₄, and concentrated under reduced pressure (10-20 torr). The residue was purified by recrystallization from ethanol.

General procedure for the synthesis of sulfonamides 3aa-3ae from sodium *p*-methylbenzenesulfinate 4 and amines 2a-2e.

An undivided cell was equipped with a carbon plate anode (7.5 cm²) and a Fe plate cathode (7.5 cm²) and connected to a DC regulated power supply. The solution of corresponding amine 2 (2.53 mmol) in 30 ml (MECN:H₂O = 1:1), sodium *p*-methylbenzenesulfinate 4 (300 mg, 1.68 mmol) and supporting electrolyte NH₄Br (0.84 mmol) were added to the cell. The mixture was electrolyzed with constant current (35-40 mA/cm²) at temperature 25-30°C under magnetic stirring. Then the solvent was removed under reduced pressure (10-20 torr). The residue was diluted with EtOAc (50 ml) and washed with brine (2×8 ml) and water (2×8 ml), dried over Na₂SO₄, and concentrated under reduced pressure (10-20 torr). The residue was purified by recrystallization from ethanol.

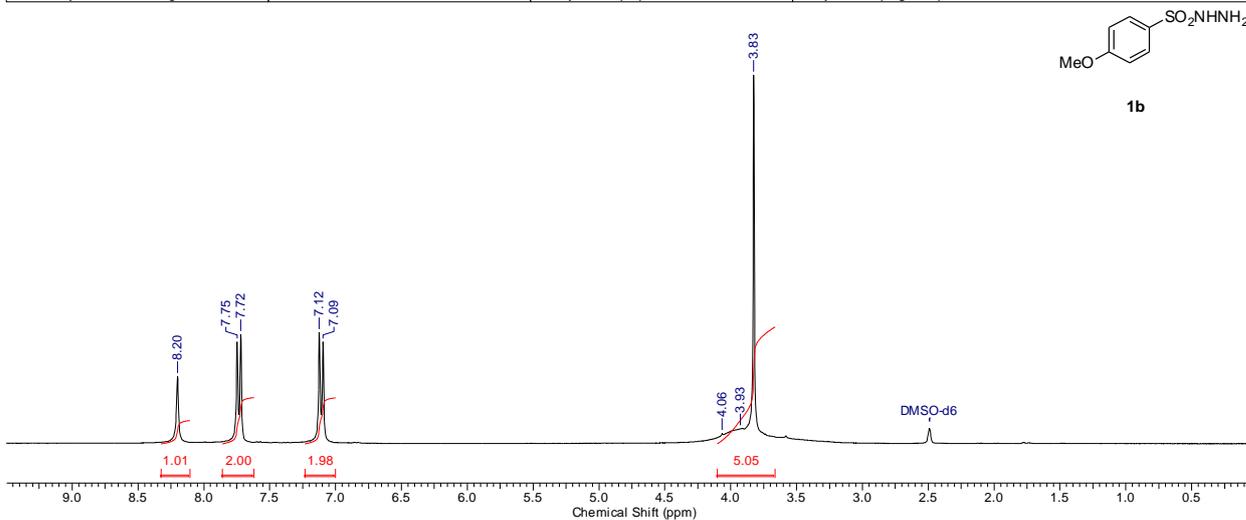
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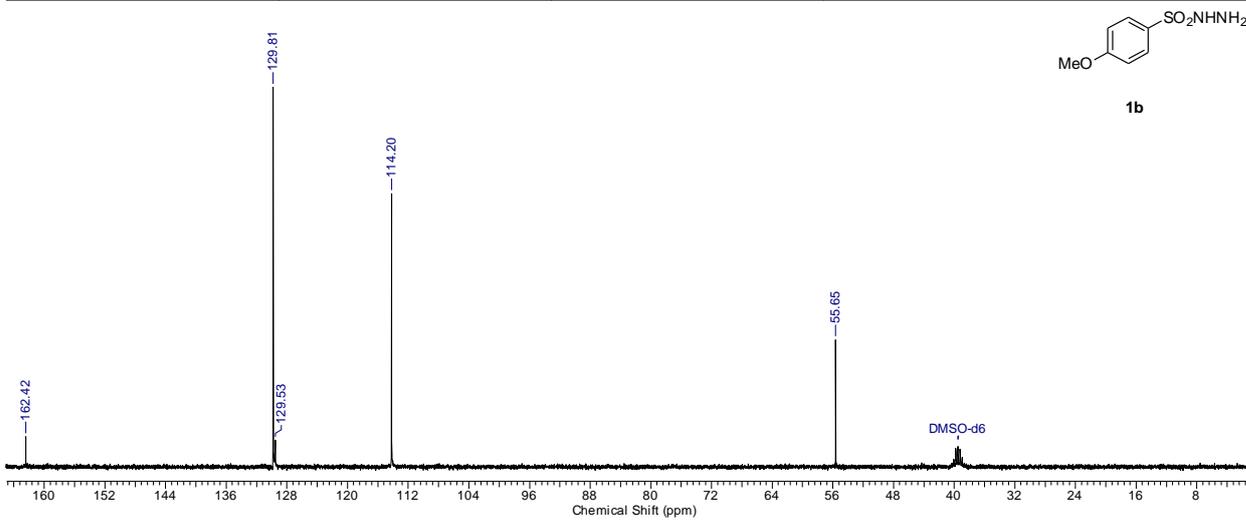
NMR Spectra of Synthesized Compounds

4-Methoxybenzenesulfonylhydrazide, **1b**

Acquisition Time (sec)	1.3521	Date	15 Jul 2015 12:48:00	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8116
Nucleus	¹ H	Solvent	DMSO-D6	Sweep Width (Hz)	6002.40
Pulse Sequence	zg			Temperature (degree C)	25.500

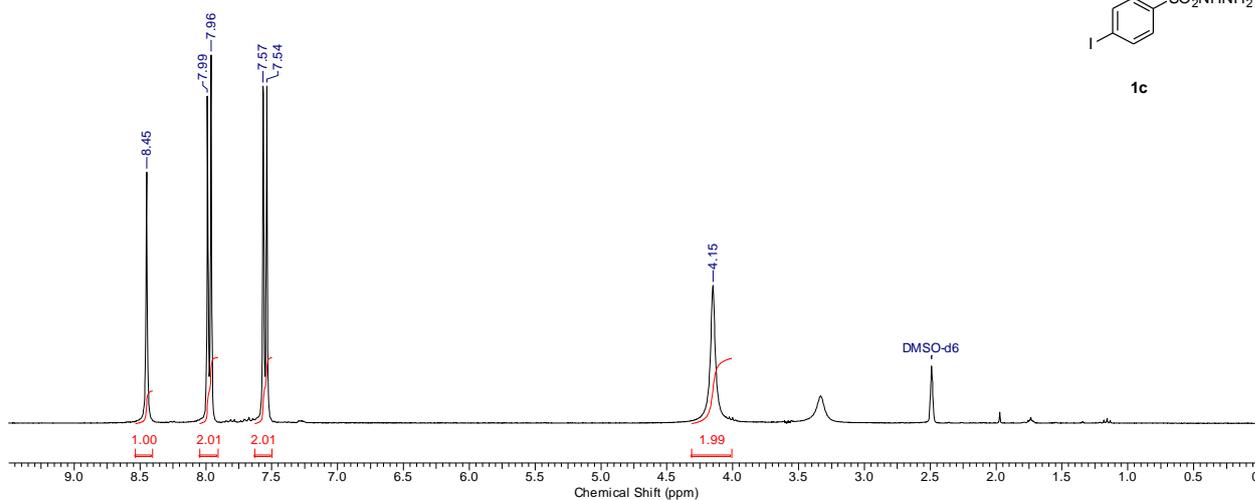


Acquisition Time (sec)	0.9002	Date	15 Jul 2015 12:52:16	Frequency (MHz)	75.48
File Name		Number of Transients	177	Original Points Count	16308
Nucleus	¹³ C	Solvent	DMSO-D6	Sweep Width (Hz)	18115.94
Pulse Sequence	zpgp60base			Temperature (degree C)	25.600

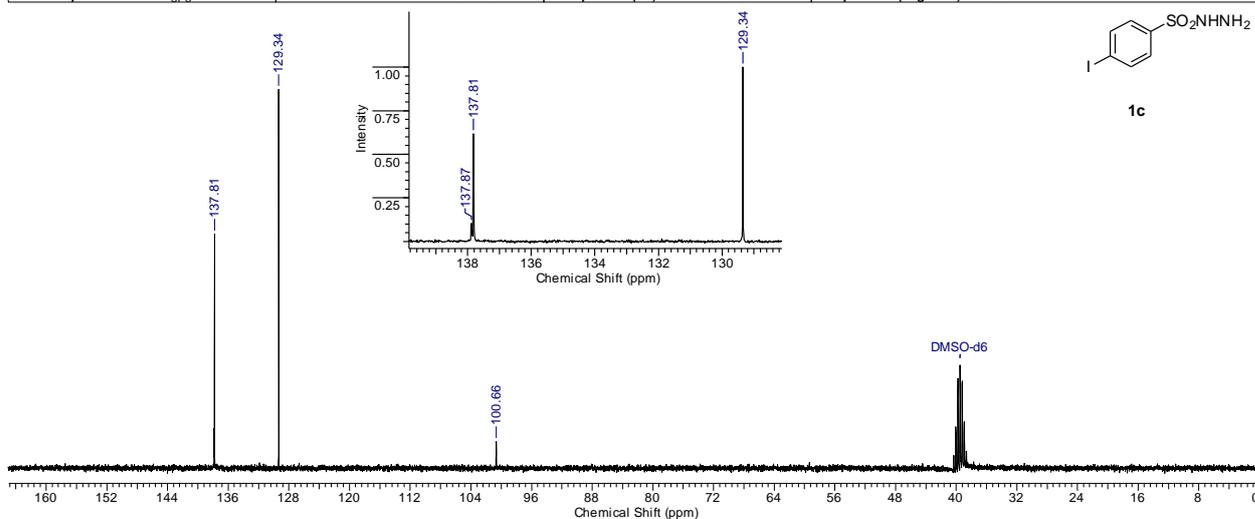


4-Iodobenzenesulfonohydrazide, **1c**

Acquisition Time (sec)	1.3521	Comment	Avance-300, 1H DMSO-d6	Date	20 Aug 2015 15:30:08	
File Name				Frequency (MHz)	300.13	
Nucleus	1H	Number of Transients	1	Original Points Count	8116	
Pulse Sequence	zg	Solvent	DMSO-D6	Points Count	8192	
			Sweep Width (Hz)	6002.40	Temperature (degree C)	27.000

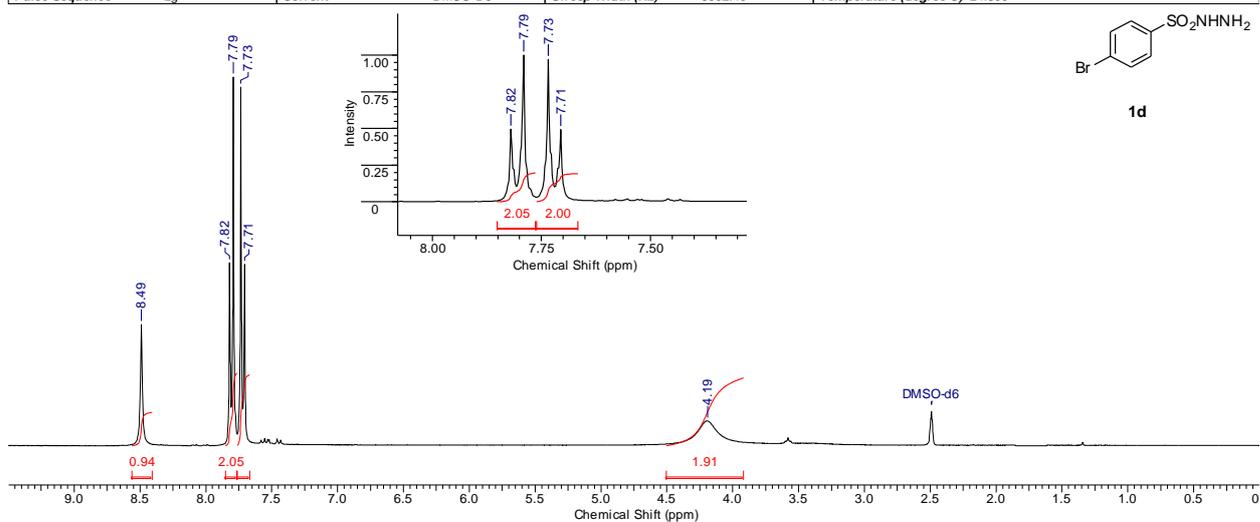


Acquisition Time (sec)	0.8643	Comment	c13,dms0	Date	24 Aug 2015 14:00:32	
File Name				Frequency (MHz)	75.48	
Nucleus	13C	Number of Transients	155	Original Points Count	16308	
Pulse Sequence	zgpg60base	Solvent	DMSO-D6	Points Count	16384	
			Sweep Width (Hz)	18867.92	Temperature (degree C)	25.100

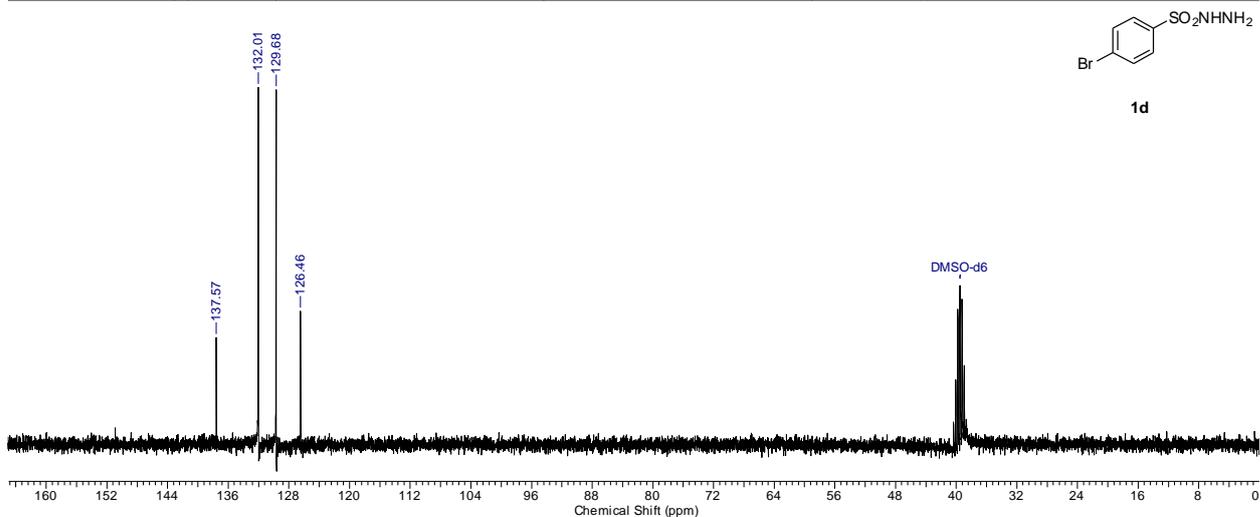


4-Bromobenzenesulfonylhydrazide, **1d**

Acquisition Time (sec)	1.3521	Comment	Avance-300, 1H DMSO-d6	Date	27 Aug 2015 10:40:00
File Name				Frequency (MHz)	300.13
Nucleus	1H	Number of Transients	1	Original Points Count	8116
Pulse Sequence	zg	Solvent	DMSO-D6	Sweep Width (Hz)	6002.40
				Points Count	8192
				Temperature (degree C)	24.300

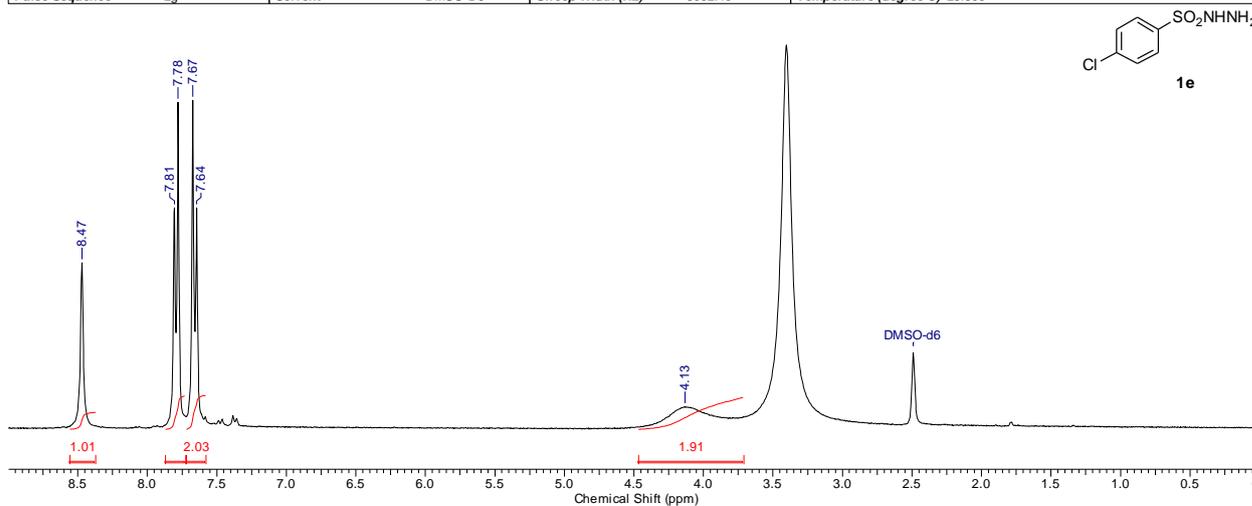


Acquisition Time (sec)	0.8643	Comment	c13.dms0	Date	05 Sep 2015 09:29:36
File Name				Frequency (MHz)	75.48
Nucleus	13C	Number of Transients	67	Original Points Count	16308
Pulse Sequence	zgpg60base	Solvent	DMSO-D6	Sweep Width (Hz)	18867.92
				Points Count	16384
				Temperature (degree C)	25.900

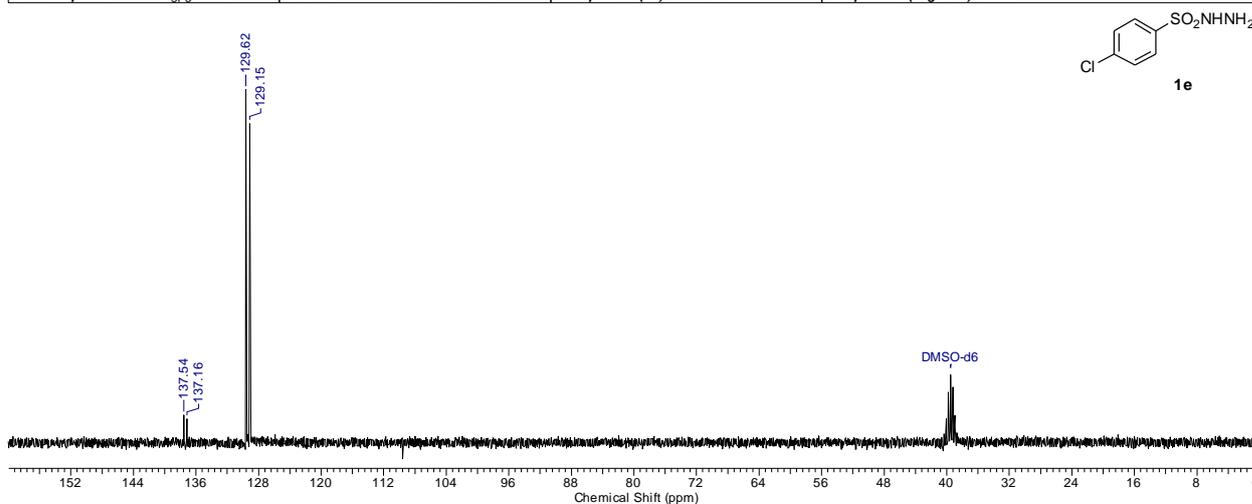


4-Chlorobenzenesulfonohydrazide, **1e**

Acquisition Time (sec)	1.3521	Date	13 Aug 2015 10:42:08	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8116
Nucleus	1H	Solvent	DMSO-D6	Sweep Width (Hz)	6002.40
Pulse Sequence	zg			Points Count	8192
				Temperature (degree C)	25.600

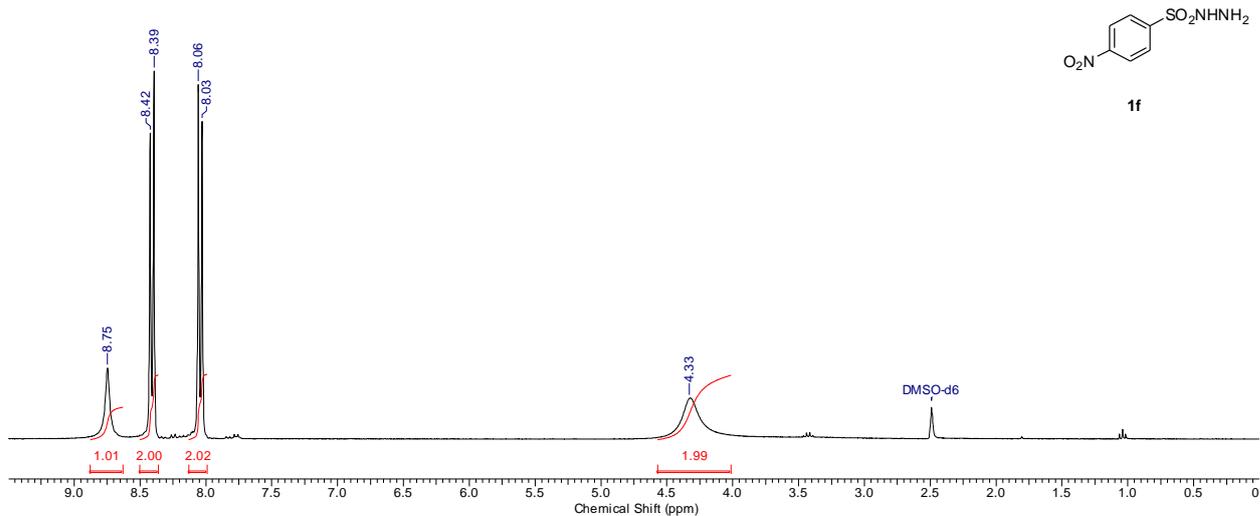


Acquisition Time (sec)	0.9002	Date	11 Aug 2015 14:26:08	Frequency (MHz)	75.48
File Name		Number of Transients	362	Original Points Count	16308
Nucleus	13C	Solvent	DMSO-D6	Sweep Width (Hz)	18115.94
Pulse Sequence	zgpg60base			Points Count	16384
				Temperature (degree C)	25.300

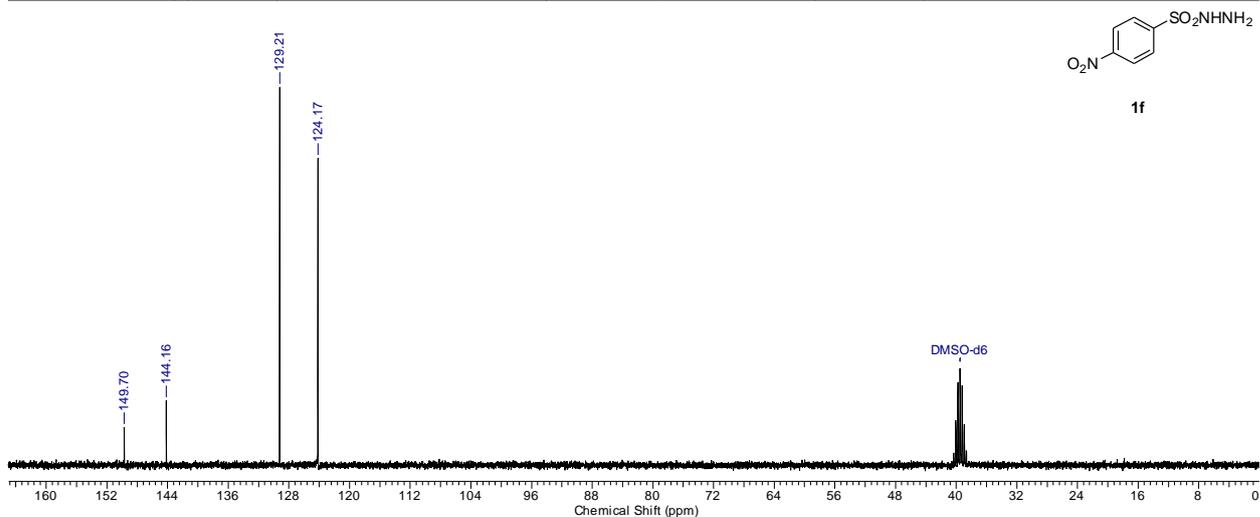


4-Nitrobenzenesulfonylhydrazide, **1f**

Acquisition Time (sec)	1.3521	Comment	Avance-300, 1H DMSO-d6	Date	20 Aug 2015 11:54:40	
File Name				Frequency (MHz)	300.13	
Nucleus	1H	Number of Transients	1	Original Points Count	8116	
Pulse Sequence	zg	Solvent	DMSO-D6	Points Count	8192	
			Sweep Width (Hz)	6002.40	Temperature (degree C)	26.200

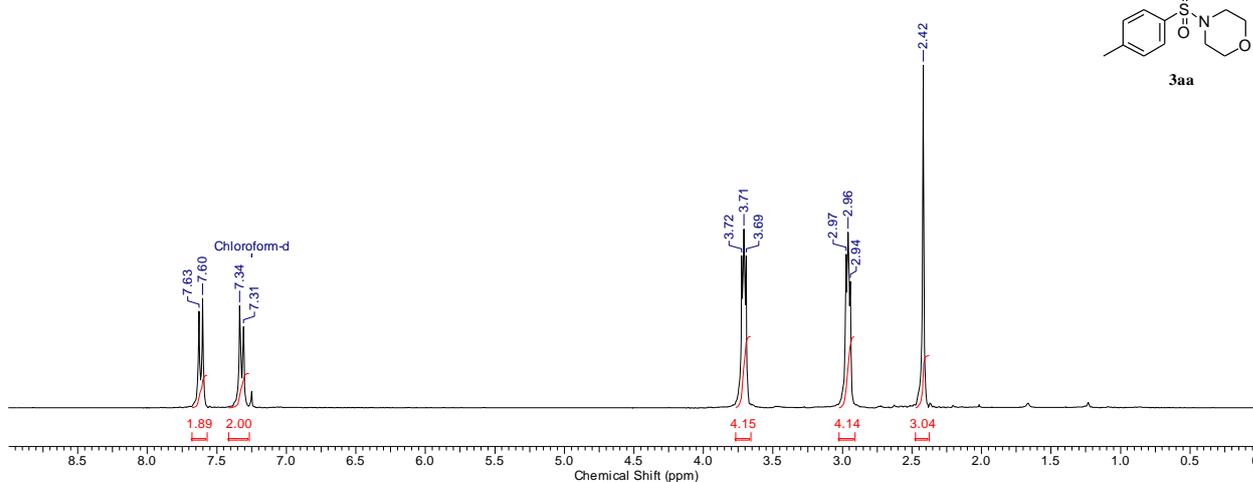


Acquisition Time (sec)	0.8643	Comment	c13,dms0	Date	21 Aug 2015 14:51:44	
File Name				Frequency (MHz)	75.48	
Nucleus	13C	Number of Transients	119	Original Points Count	16308	
Pulse Sequence	zpgp60base	Solvent	DMSO-D6	Points Count	16384	
			Sweep Width (Hz)	18867.92	Temperature (degree C)	28.200

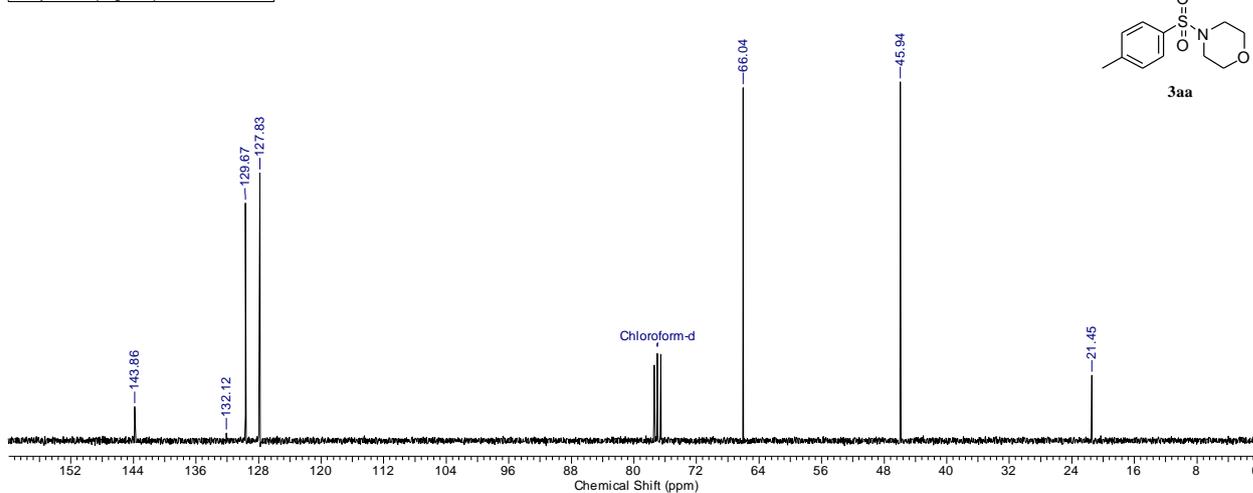


4-(*p*-Methylbenzenesulfonyl)-tetrahydrooxazine-1,4, **3aa**

Acquisition Time (sec)	1.3518	Date	18 Feb 2016 14:58:08		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	¹ H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg						
Temperature (degree C)	26.300						

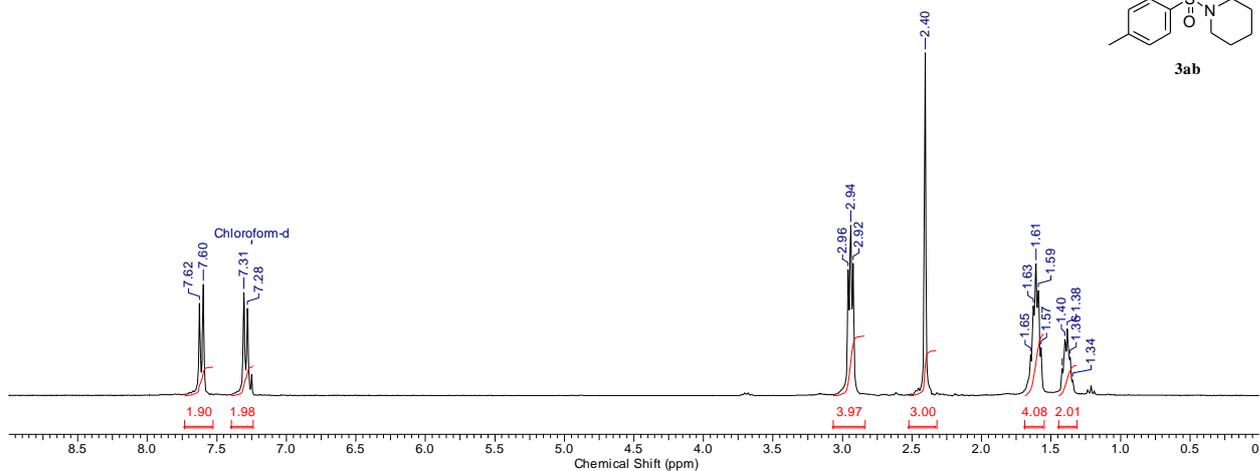


Acquisition Time (sec)	0.8680	Comment	adil.cdcl3.c13	Date	20 Feb 2016 15:45:04		
File Name		Number of Transients	183	Original Points Count	16316	Frequency (MHz)	75.48
Nucleus	¹³ C	Solvent	CHLOROFORM-D		Points Count	16384	
Pulse Sequence	zpgg60base				Sweep Width (Hz)	18796.99	
Temperature (degree C)	27.000						

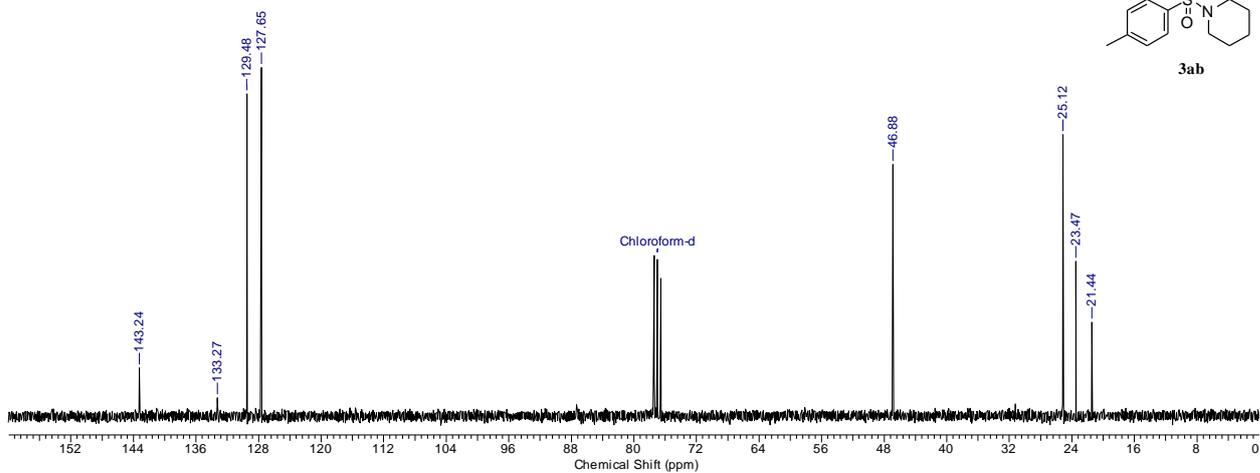


1-(p-Methylbenzenesulfonyl)-piperidine, **3ab**

Acquisition Time (sec)	1.3518	Date	29 Mar 2016 12:26:40	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	¹ H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)	6009.62		
Temperature (degree C)	25.300				

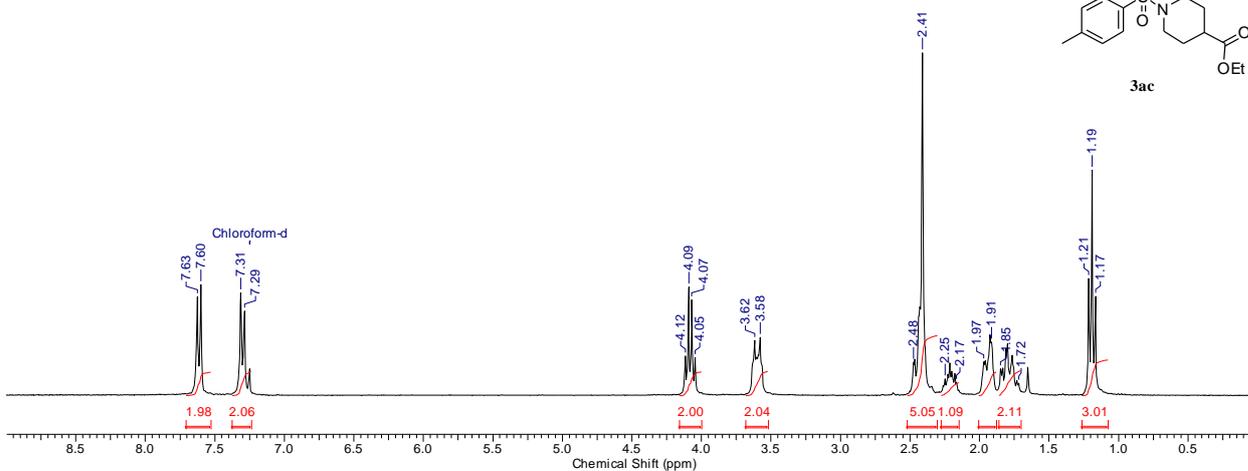


Acquisition Time (sec)	0.9006	Date	30 Mar 2016 11:12:00	Frequency (MHz)	75.48
File Name		Number of Transients	151	Original Points Count	16316
Nucleus	¹³ C	Solvent	CHLOROFORM-D	Points Count	16384
Pulse Sequence	zgpg30	Sweep Width (Hz)	18115.94		
Temperature (degree C)	25.700				

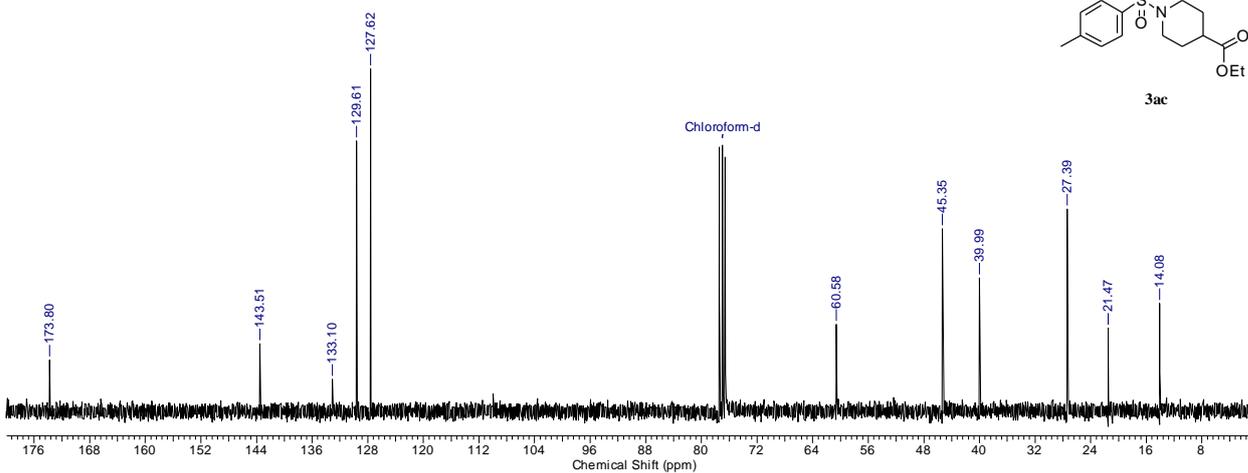


Ethyl 1-(p-methylbenzenesulfonyl)piperidine-4-carboxylate, **3ac**

Acquisition Time (sec)	1.3518	Date	10 May 2016 14:02:40	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	¹ H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)	6009.62		
Temperature (degree C)	22.900				

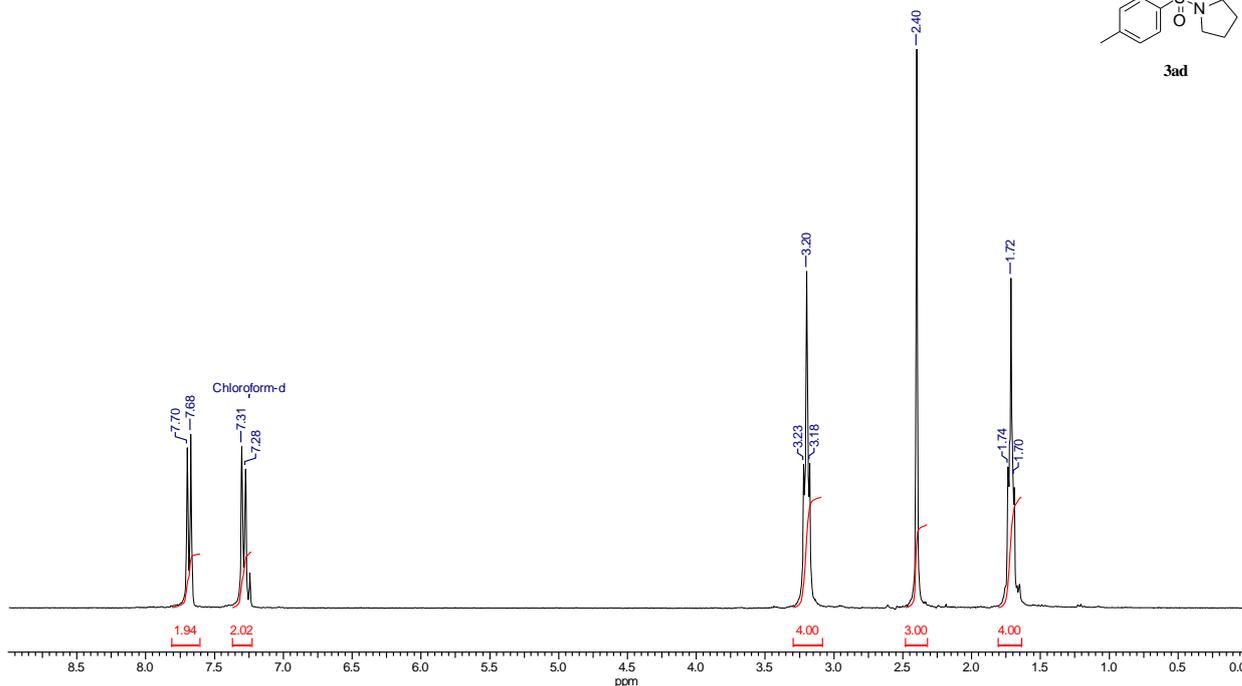
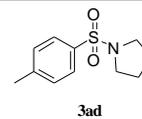


Acquisition Time (sec)	0.9006	Date	11 May 2016 16:59:44	Frequency (MHz)	75.48
File Name		Number of Transients	243	Original Points Count	16316
Nucleus	¹³ C	Solvent	CHLOROFORM-D	Points Count	16384
Pulse Sequence	zgpg30	Sweep Width (Hz)	18115.94		
Temperature (degree C)	24.000				

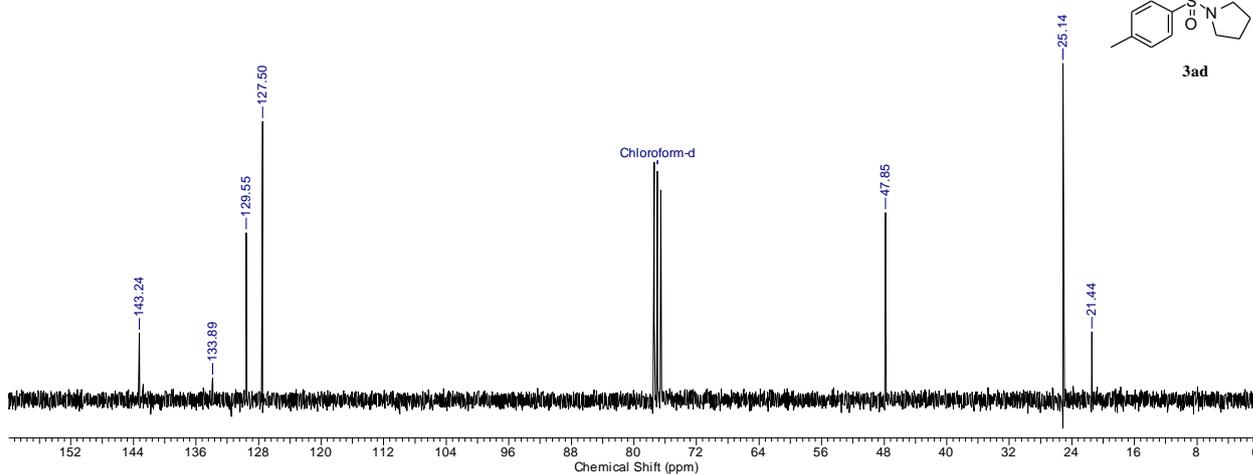
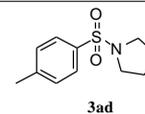


1-(p-Methylbenzenesulfonyl)-pyrrolidine, 3ad

Acquisition Time (sec)	0.6759	Date	29 Mar 2016 12:24:32		
File Name		Frequency (MHz)	300.13		
Nucleus	1H	Number of Transients	1	Original Points Count	8124
Pulse Sequence	zg	Solvent	CHLOROFORM-D		
Temperature (degree C)	25.400	Points Count	8192		
		Sweep Width (Hz)	6009.62		

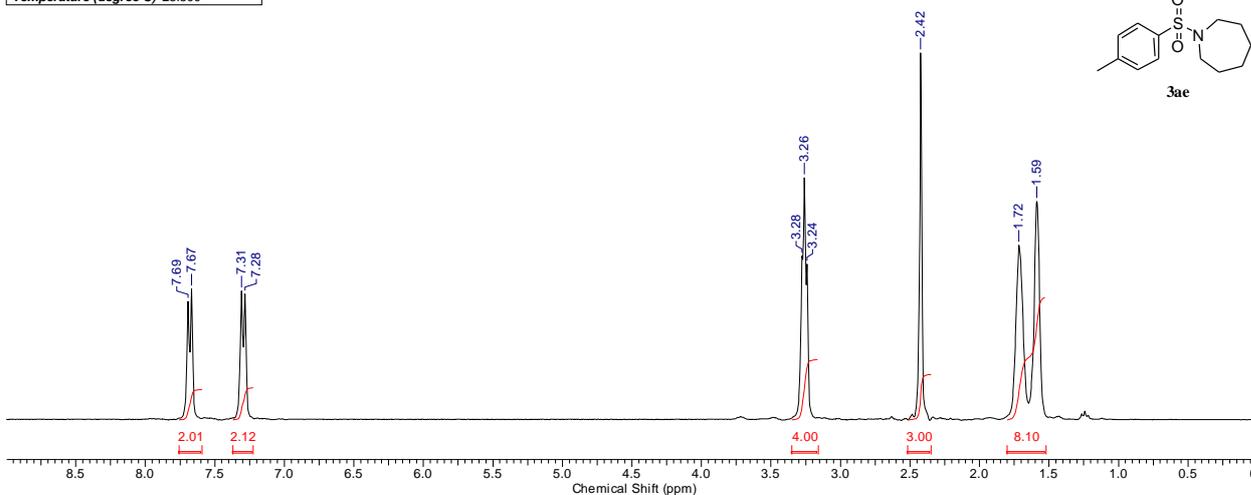


Acquisition Time (sec)	0.9006	Date	30 Mar 2016 11:01:20		
File Name		Frequency (MHz)	75.48		
Nucleus	13C	Number of Transients	102	Original Points Count	16316
Pulse Sequence	zpgq30	Solvent	CHLOROFORM-D		
Temperature (degree C)	25.200	Points Count	16384		
		Sweep Width (Hz)	18115.94		

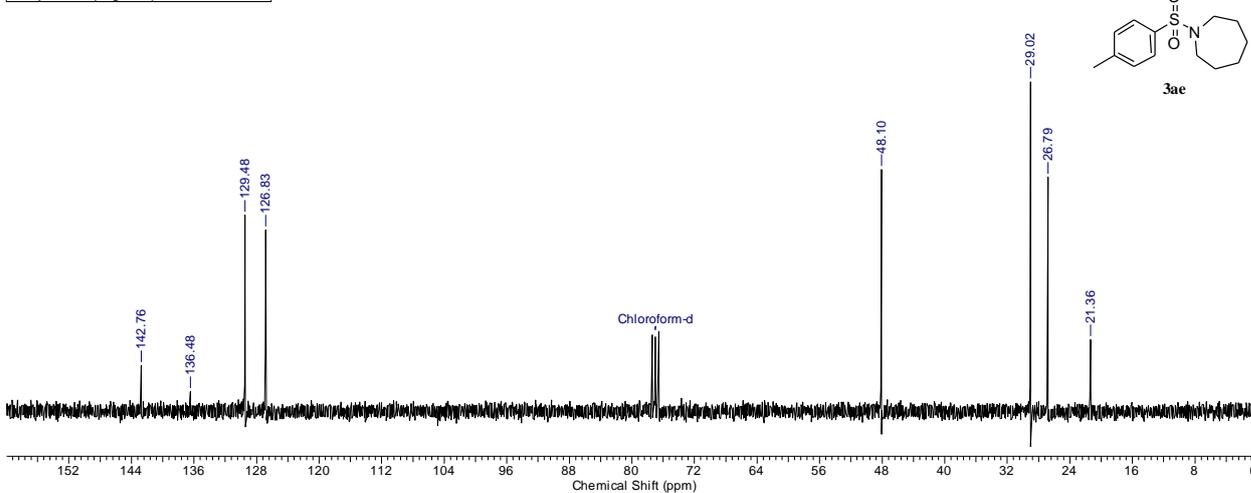


1-(p-Methylbenzenesulfonyl)-azepane, **3ae**

Acquisition Time (sec)	1.3518	Date	14 Apr 2016 16:27:44	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	1H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)			6009.62
Temperature (degree C)	23.500				

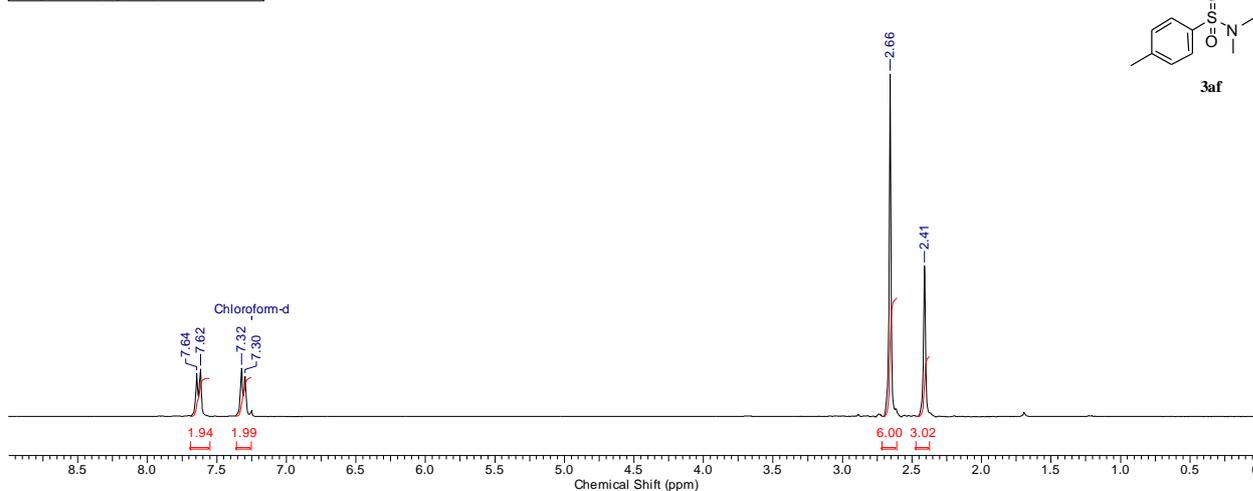


Acquisition Time (sec)	0.8680	Comment	adil.cdcl3.c13	Date	18 Apr 2016 08:55:28	Frequency (MHz)	75.48
File Name		Number of Transients	44	Original Points Count	16316	Points Count	16384
Nucleus	13C	Solvent	CHLOROFORM-D	Sweep Width (Hz)			18796.99
Pulse Sequence	zpgg60base						
Temperature (degree C)	23.400						

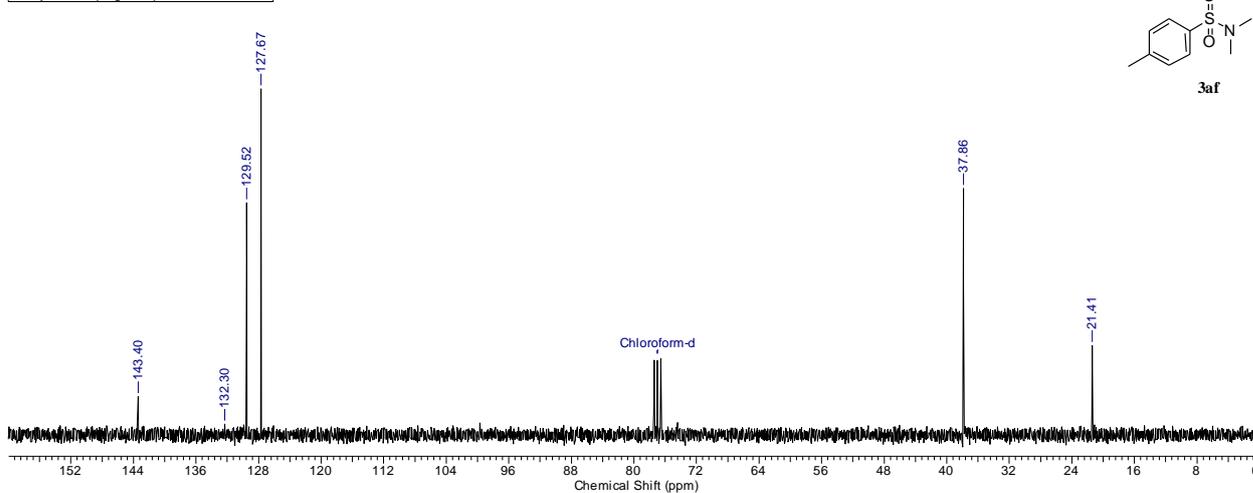


N,N,4-Trimethylbenzenesulfonamide, **3af**

Acquisition Time (sec)	1.3518	Date	14 Apr 2016 16:29:52	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	¹ H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)	6009.62		
Temperature (degree C)	23.500				

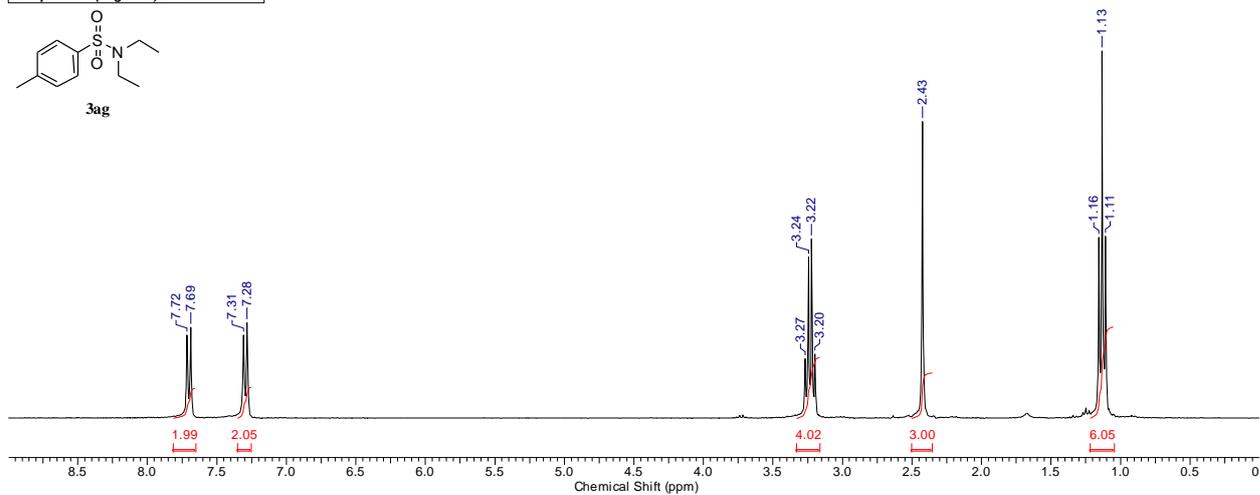
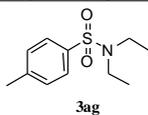


Acquisition Time (sec)	0.8680	Comment	adil.cdcl3.c13	Date	18 Apr 2016 08:51:12	Frequency (MHz)	75.48
File Name		Number of Transients	43	Original Points Count	16316	Points Count	16384
Nucleus	¹³ C	Solvent	CHLOROFORM-D	Sweep Width (Hz)	18796.99		
Pulse Sequence	zgpg90base						
Temperature (degree C)	23.200						

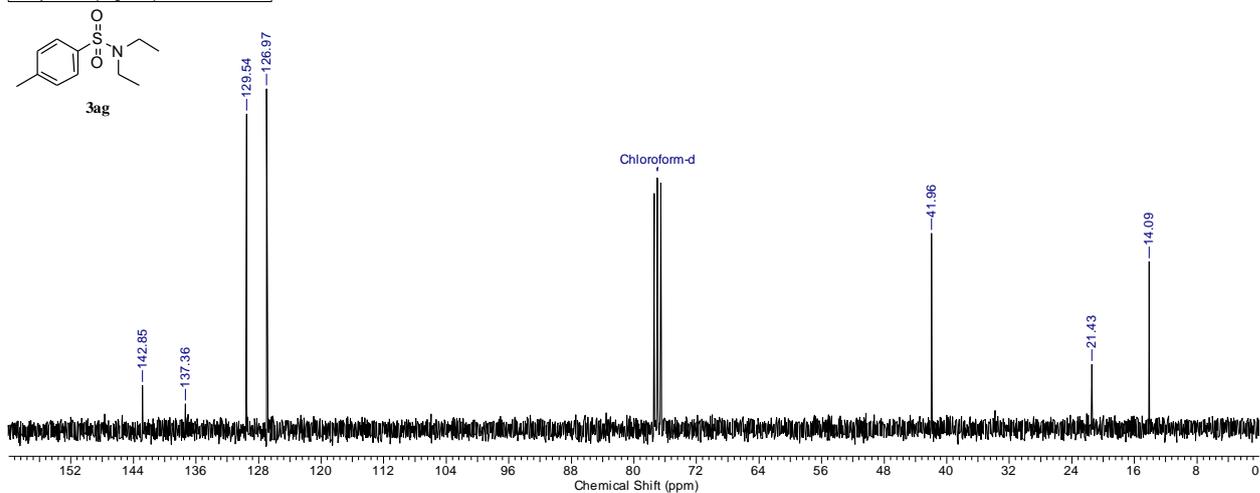
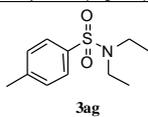


N,N-Diethyl-4-methylbenzenesulfonamide, **3ag**

Acquisition Time (sec)	1.3518	Date	05 May 2016 11:48:16		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	¹ H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg						
Temperature (degree C)	22.400						

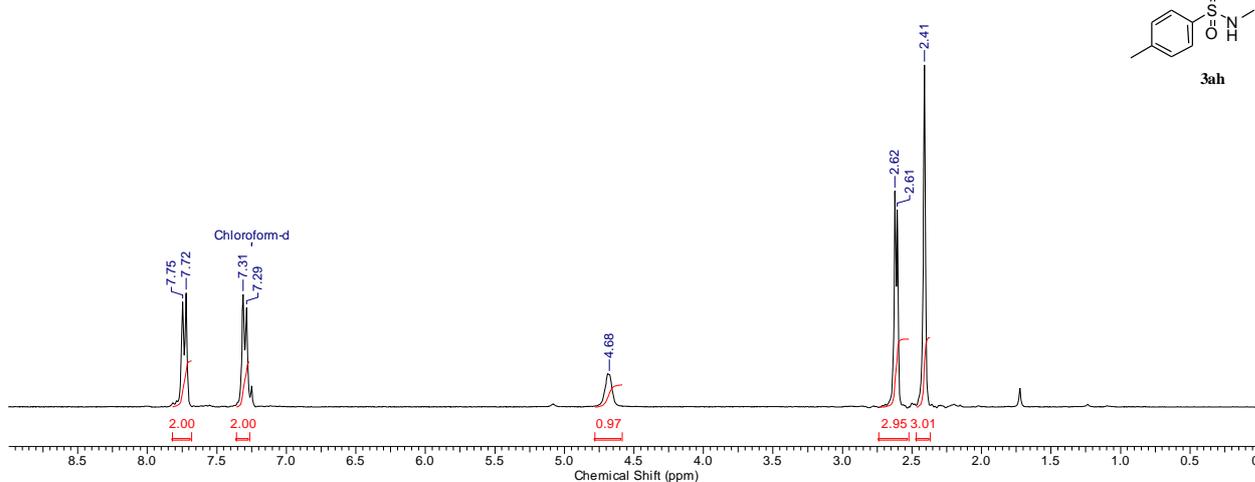


Acquisition Time (sec)	0.9006	Date	05 May 2016 15:19:28		Frequency (MHz)	75.48	
File Name		Number of Transients	123	Original Points Count	16316	Points Count	16384
Nucleus	¹³ C	Solvent	CHLOROFORM-D		Sweep Width (Hz)	18115.94	
Pulse Sequence	zgpg30						
Temperature (degree C)	23.100						

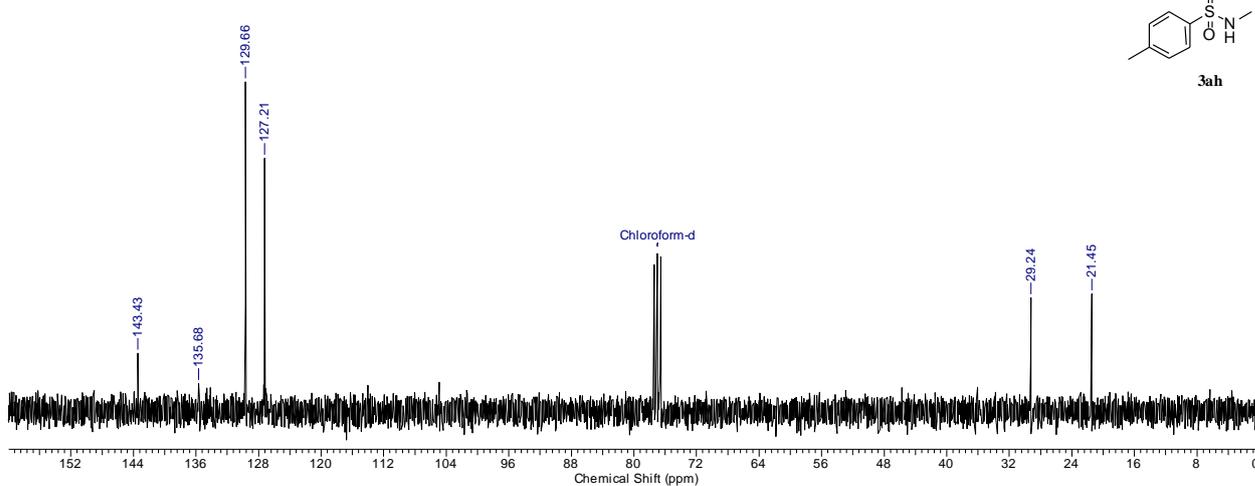


N,4-Dimethylbenzenesulfonamide, **3ah**

Acquisition Time (sec)	1.3518	Date	14 Apr 2016 16:32:00		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	¹ H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg						
Temperature (degree C)	23.400						

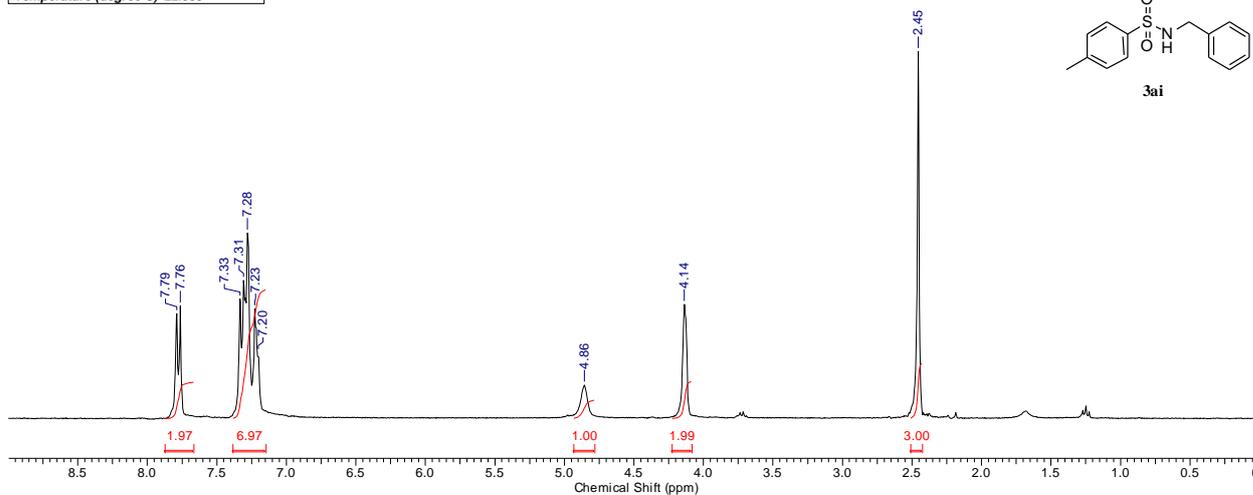


Acquisition Time (sec)	0.8680	Comment	adil.cdci3.c13	Date	18 Apr 2016 09:04:00		
File Name		Number of Transients	46	Original Points Count	16316	Frequency (MHz)	75.48
Nucleus	¹³ C	Solvent	CHLOROFORM-D		Points Count	16384	
Pulse Sequence	zpgg60base				Sweep Width (Hz)	18796.99	
Temperature (degree C)	23.300						

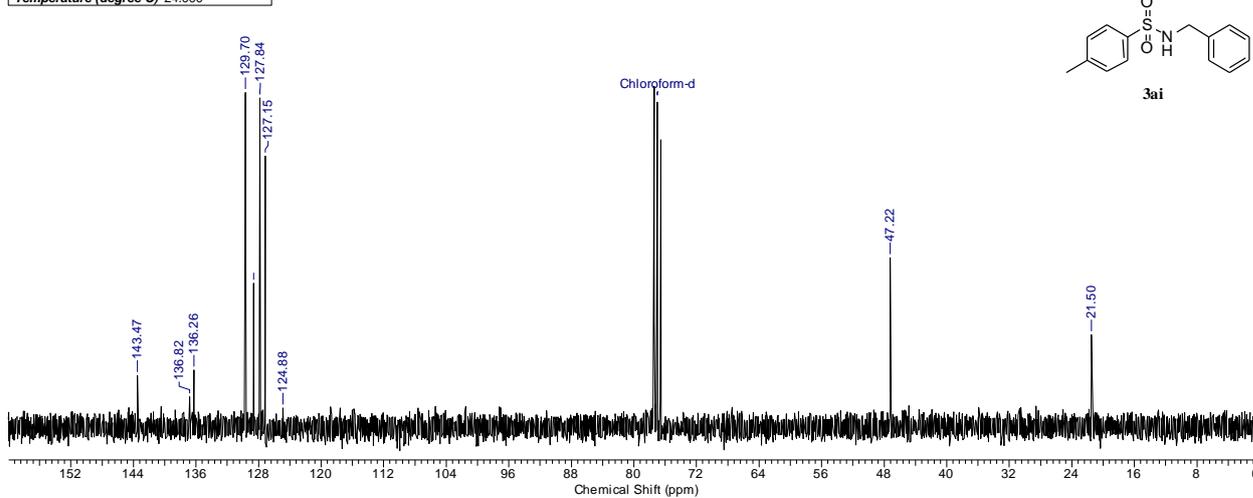


N-Benzyl-4-methylbenzenesulfonamide, **3ai**

Acquisition Time (sec)	1.3518	Date	10 May 2016 14:00:32	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	¹ H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)	6009.62		
Temperature (degree C)	22.900				

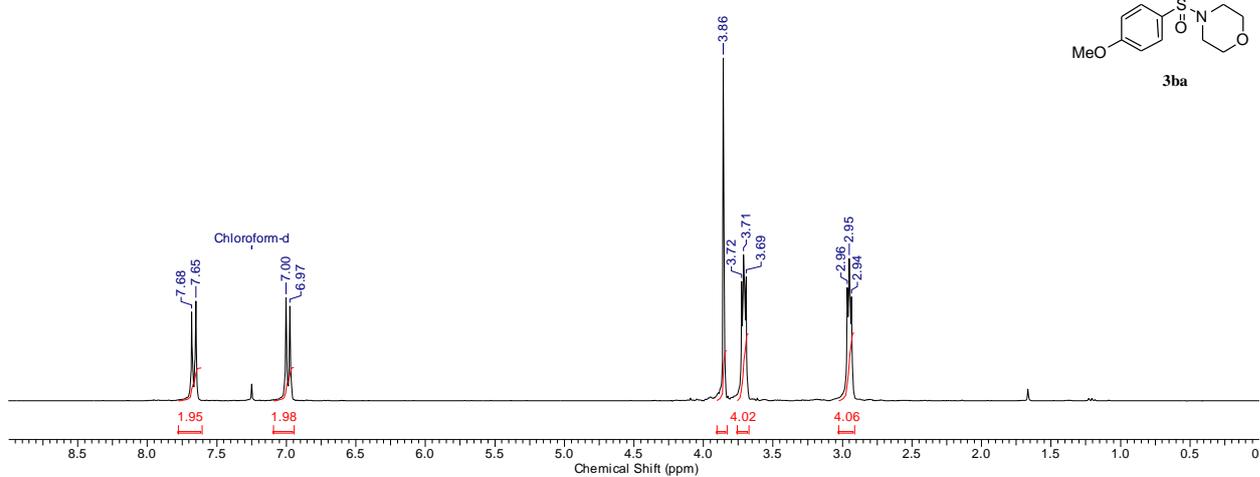


Acquisition Time (sec)	0.9006	Date	11 May 2016 17:08:16	Frequency (MHz)	75.48
File Name		Number of Transients	124	Original Points Count	16316
Nucleus	¹³ C	Solvent	CHLOROFORM-D	Points Count	16384
Pulse Sequence	zgpg30	Sweep Width (Hz)	18115.94		
Temperature (degree C)	24.000				

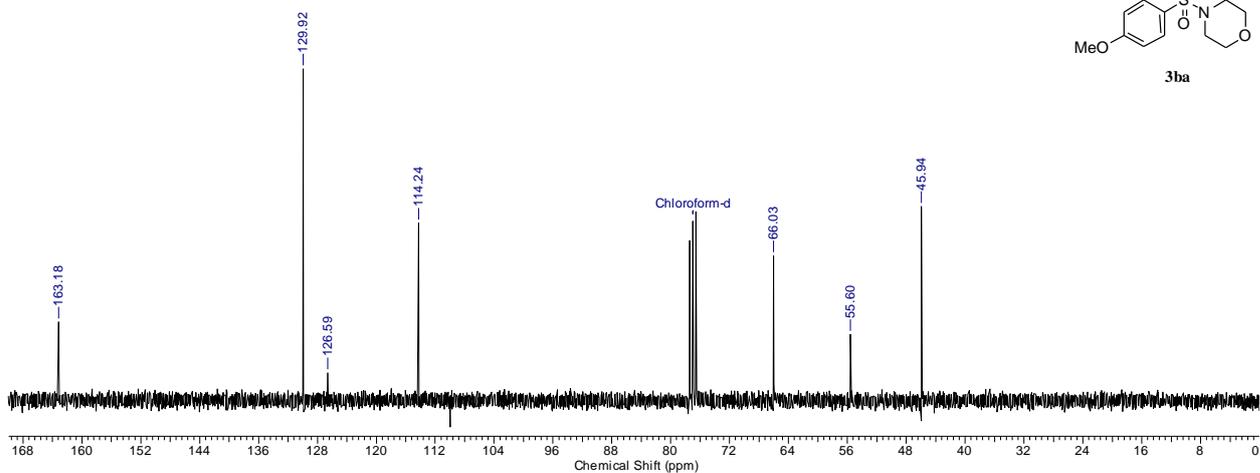


4-(*p*-Methoxybenzenesulfonyl)-tetrahydrooxazine-1,4, **3ba**

Acquisition Time (sec)	1.3518	Date	29 Mar 2016 12:24:32		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	¹ H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg	Temperature (degree C)	25.400				

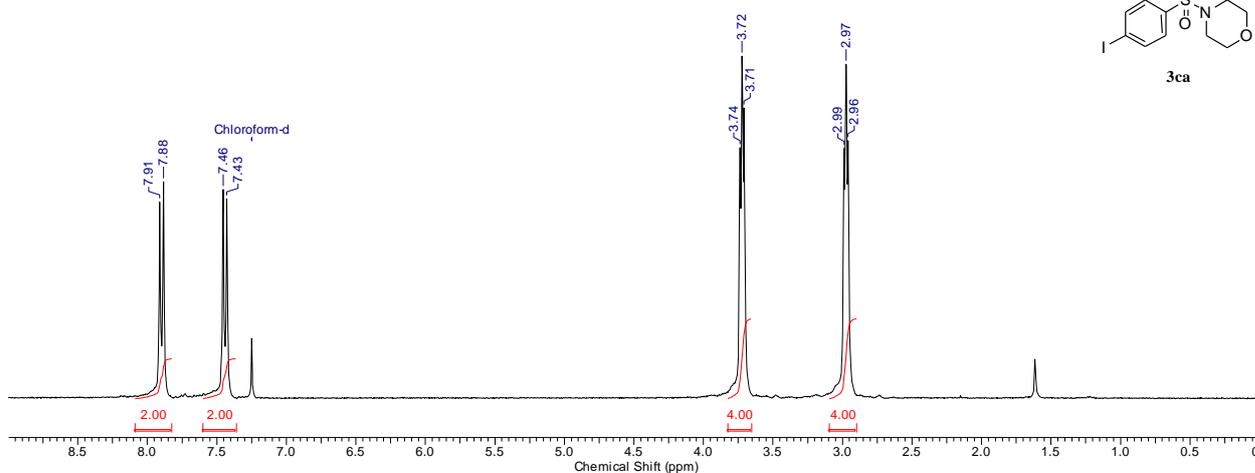


Acquisition Time (sec)	0.9006	Date	30 Mar 2016 11:18:24		Frequency (MHz)	75.48	
File Name		Number of Transients	72	Original Points Count	16316	Points Count	16384
Nucleus	¹³ C	Solvent	CHLOROFORM-D		Sweep Width (Hz)	18115.94	
Pulse Sequence	zgpg30	Temperature (degree C)	25.600				

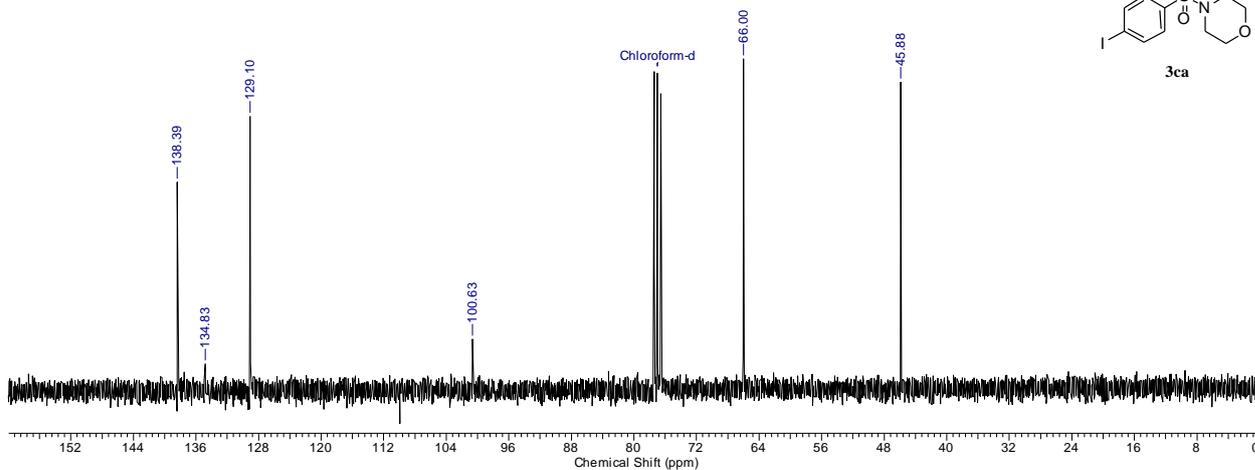


4-(p-Iodobenzenesulfonyl)-tetrahydrooxazine-1,4, **3ca**

Acquisition Time (sec)	1.3518	Date	05 May 2016 11:46:08		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	¹ H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg	Temperature (degree C)	22.400				

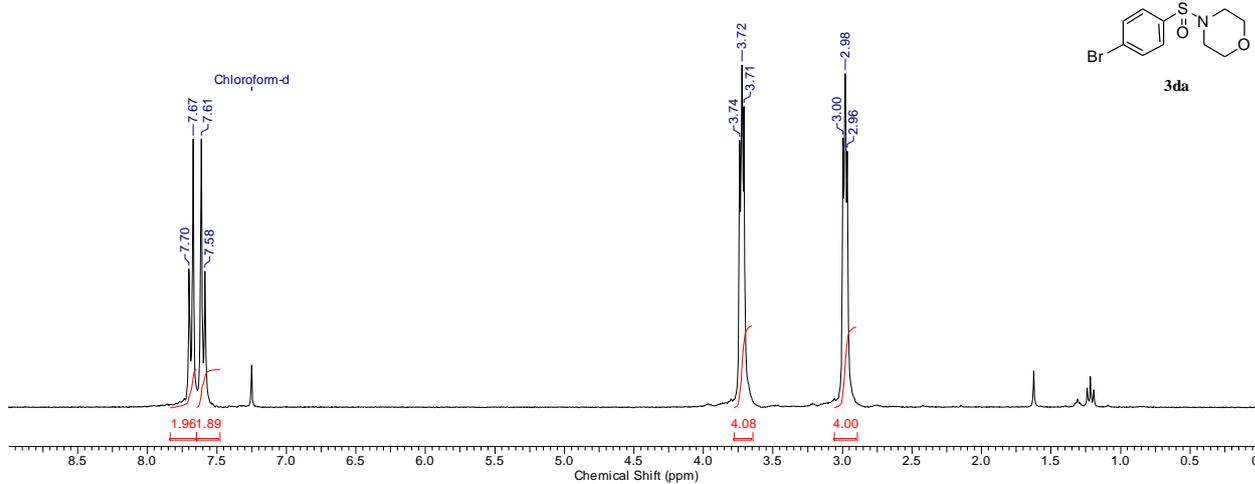


Acquisition Time (sec)	0.9006	Date	05 May 2016 15:13:04		Frequency (MHz)	75.48	
File Name		Number of Transients	138	Original Points Count	16316	Points Count	16384
Nucleus	¹³ C	Solvent	CHLOROFORM-D		Sweep Width (Hz)	18115.94	
Pulse Sequence	zgpg30	Temperature (degree C)	23.100				

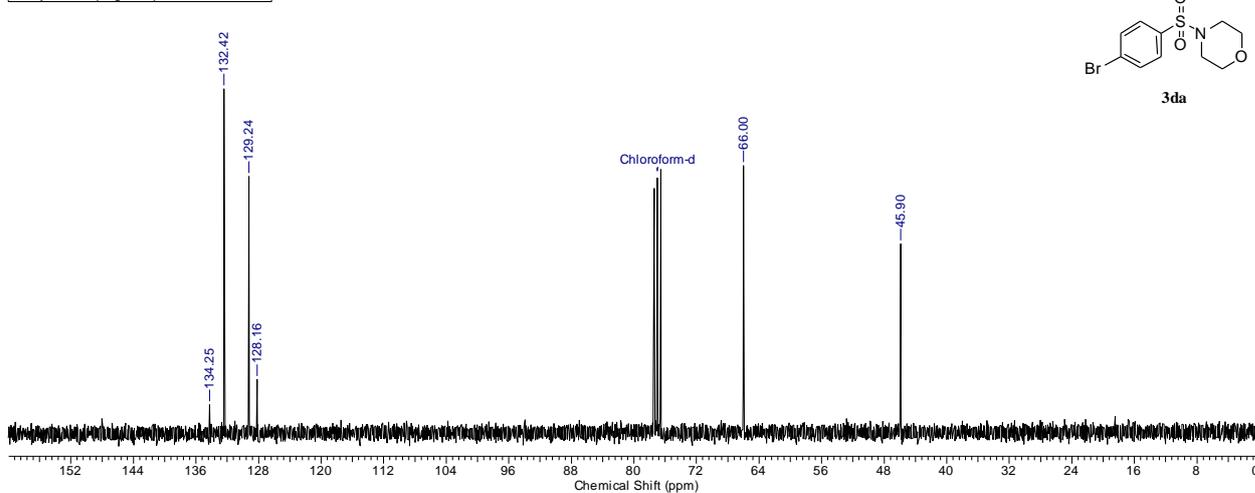


4-(p-Bromobenzenesulfonyl)-tetrahydrooxazine-1,4, 3da

Acquisition Time (sec)	1.3518	Date	29 Mar 2016 12:22:24		Frequency (MHz)	300.13	
File Name		Number of Transients	1	Original Points Count	8124	Points Count	8192
Nucleus	1H	Solvent	CHLOROFORM-D		Sweep Width (Hz)	6009.62	
Pulse Sequence	zg	Temperature (degree C)	25.500				

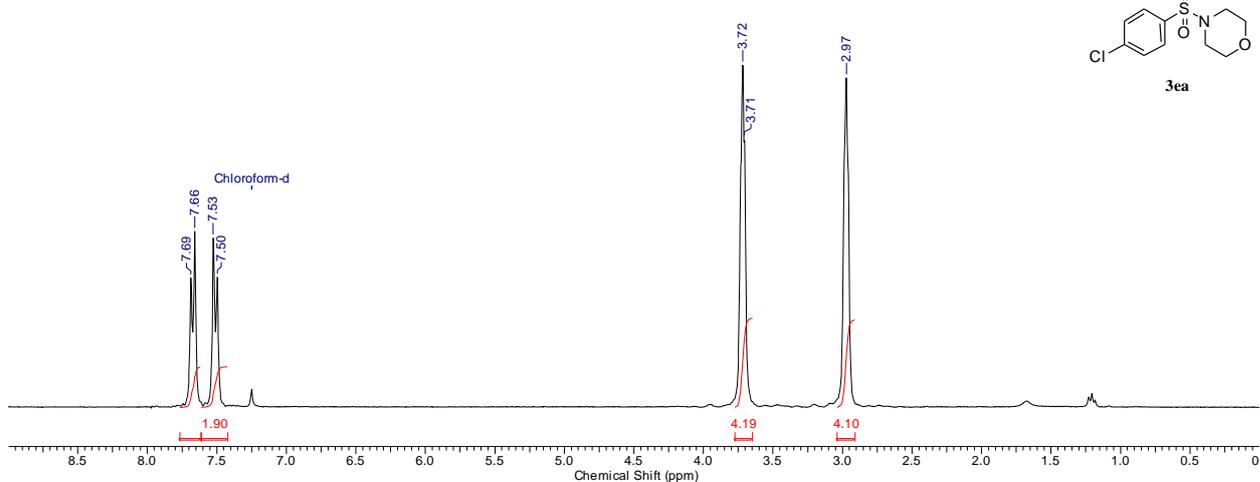


Acquisition Time (sec)	0.9006	Date	30 Mar 2016 11:05:36		Frequency (MHz)	75.48	
File Name		Number of Transients	191	Original Points Count	16316	Points Count	16384
Nucleus	13C	Solvent	CHLOROFORM-D		Sweep Width (Hz)	18115.94	
Pulse Sequence	zgpg30	Temperature (degree C)	25.500				

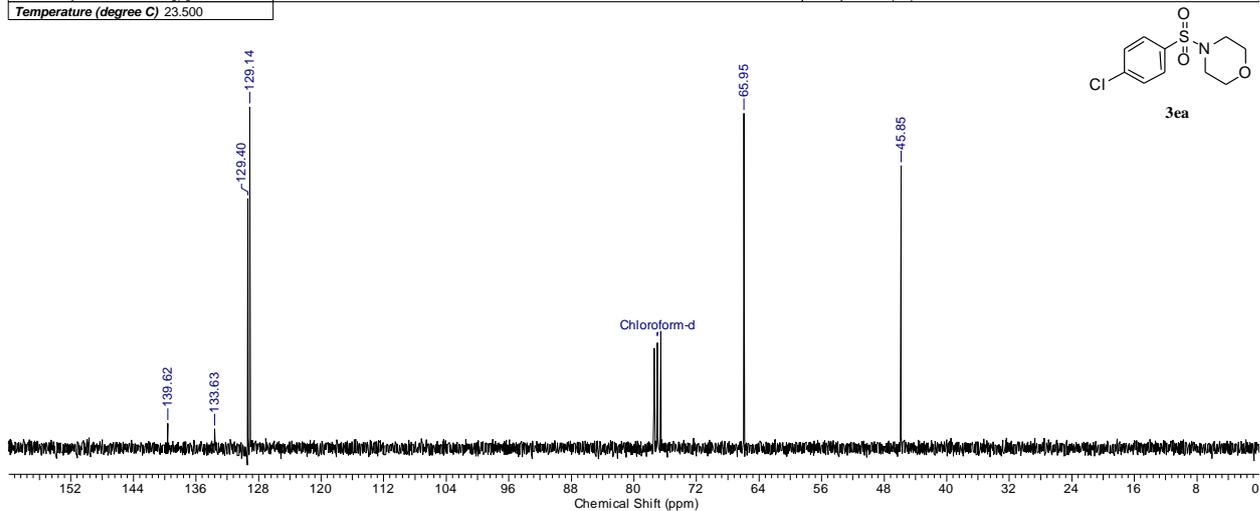


4-(p-Chlorobenzenesulfonyl)-tetrahydrooxazine-1,4, **3ea**

Acquisition Time (sec)	1.3518	Date	14 Apr 2016 16:38:24	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	1H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)			6009.62
Temperature (degree C)	23.300				

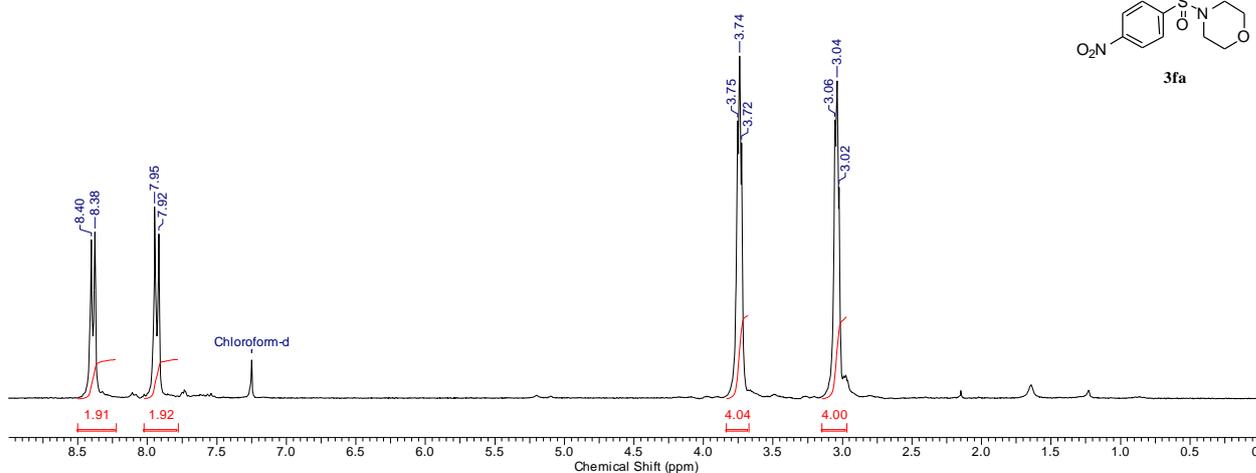


Acquisition Time (sec)	0.8680	Comment	adil.cdcl3.c13	Date	18 Apr 2016 08:59:44	Frequency (MHz)	75.48
File Name		Number of Transients	47	Original Points Count	16316	Points Count	16384
Nucleus	13C	Solvent	CHLOROFORM-D	Sweep Width (Hz)			18796.99
Pulse Sequence	zpgg50base						
Temperature (degree C)	23.500						



4-(p-Nitrobenzenesulfonyl)-tetrahydrooxazine-1,4, **3fa**

Acquisition Time (sec)	1.3518	Date	10 May 2016 14:02:40	Frequency (MHz)	300.13
File Name		Number of Transients	1	Original Points Count	8124
Nucleus	¹ H	Solvent	CHLOROFORM-D	Points Count	8192
Pulse Sequence	zg	Sweep Width (Hz)			6009.62
Temperature (degree C)	22.900				



Acquisition Time (sec)	0.9006	Date	11 May 2016 16:51:12	Frequency (MHz)	75.48
File Name		Number of Transients	176	Original Points Count	16316
Nucleus	¹³ C	Solvent	CHLOROFORM-D	Points Count	16384
Pulse Sequence	zgpg30	Sweep Width (Hz)			18115.94
Temperature (degree C)	23.900				

