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Ammonium iodide-mediated electrosynthesis of unsymmetrical thiosulfonates from arenesulfonohydrazides and thiols

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Table of Contents

General Information

NMR spectra were registered on Bruker Avance II 300 MHz instrument. Chemical shifts were measured relative to solvent peaks as an internal standard set to δ 7.25 and δ 77.0 ($CDCl_3$), δ 2.50 and δ 39.51 (DMSO-d_6). The TLC analyses were carried out on standard silica-gel chromatography plates. The melting points were determined on a Kofler hot-stage apparatus. Chromatography was performed on silica gel (60-200 mesh).

p-Toluenesulfonohydrazide (**1a**), thiophenol (**2a**), 4-methylbenzenethiol (**2b**), 4-chlorobenzenethiol (**2c**), 2-methylpropane-1-thiol (**2d**), butane-1-thiol (**2e**), 4-methoxybenzenesulfonyl chloride, 4-fluorobenzenesulfonyl chloride, 4-chlorobenzenesulfonyl chloride, 4-bromobenzenesulfonyl chloride, 4-iodobenzenesulfonyl chloride, 4-nitrobenzenesulfonyl chloride, naphthalene-2-sulfonyl chloride, 2,4,6-trimethylbenzenesulfonyl chloride, tetrabutylammonium perchlorate, KI, NH_4I, TBAI, NH_4Br, NH_4Cl, Na_2SO_4, THF, dioxane, EtOH, DMSO,petroleum ether (PE, 40/70), ethyl acetate (EA) were used as purchased. 4-Methoxybenzenesulfonohydrazide (**1b**), 4-fluorobenzenesulfonohydrazide (**1c**), 4-chlorobenzenesulfonohydrazide (**1d**), 4-bromobenzenesulfonohydrazide (**1e**), 4-iodobenzenesulfonohydrazide (**1f**), 4-nitrobenzenesulfonohydrazide (**1g**), naphthalene-2-sulfonohydrazide (**1h**), 2,4,6-trimethylbenzenesulfonohydrazide (**1i**) were synthesized according to the literature.[S1]

Cyclic voltammetry (CV) was implemented on an IPC-Pro computer-assisted potentiostat manufactured by Econix (scan rate error 1.0%, potential setting 0.25 mV). The experiments were performed in a 10ml five-necked glass conic electrochemical cell with a water jacket for thermostating. CV curves were recorded using a three-electrode scheme. The working electrode was a disc glassy-carbon electrode (d = 1.7 mm). A platinum wire served as an auxiliary electrode. A saturated calomel electrode was used as the reference electrode and was linked to the solution by a bridge with a porous ceramic diaphragm filled with background electrolyte. The tested solutions were thermostated at 25±0.5 °C. In a typical case, 5 ml solution was used and the depolarizer concentration was 3 mmol·dm^{-3}. The working electrode was polished before recording each CV curve.

General procedure 1.

Optimization of the reaction conditions for synthesis of 3a from sulfonohydrazide 1a and thiophenol 2a (see Table 1). An undivided cell was equipped with a graphite plate anode (5 cm^2) and a stainless steel plate cathode (5 cm^2) and connected to a DC regulated power supply. The solution of *p*-toluenesulfonohydrazide**1a** (1-3 mmol,186-558 mg), thiophenol**2a** (1-3 mmol,110-330 mg) and supporting electrolyte KI, NH_4I, TBAI, NH_4Br, NH_4Cl (0.5-3 mmol) in H_2O-THF (1:1), H_2O-dioxane (1:1), EtOH-THF (1:1), EtOH or DMSO (30 ml each) was electrolyzed at constant current (50 mA·cm^{-2}) at 30 °C under magnetic stirring. During the electrosynthesis the reaction mixture is heated, that is why external cooling is needed.Then the electrodes were washed with EtOH (2×30 ml). The solvent from combined organic phases was removed under reduced pressure. The residue was diluted with EtOAc (50 ml) and washed with saturated aqueous solution of $Na_2S_2O_3$ (8 ml), brine (2×8 ml) and water (2×8 ml), dried over Na_2SO_4, concentrated under reduced pressure and dried at 10-20 Torr. The desired product **3a** was isolated by chromatography on SiO_2 with elution using PE-EA in a linear gradient of the latter from 5 to 20 vol %.

General procedure 2.

Synthesis of thiosulfonates 3a-m (see Scheme 1). An undivided cell was equipped with a graphite plate anode (5 cm^2) and a stainless steel plate cathode (5 cm^2) and connected to a DC regulated power supply. The solution of sulfonyl hydrazide **1a-i** (1 mmol), thiol **2a-e** (2

mmol) and supporting electrolyte NH_4I (1 mmol) in THF-H_2O mixture (1:1, 30 ml) was electrolyzed at constant current (50 mA·cm^{-2}) at 30 °C under magnetic stirring. During the electrosynthesis the reaction mixture is heated, that is why external cooling is needed. Then the electrodes were washed with EtOH (2×30 ml). The solvent from combined organic phases was removed under reduced pressure. The residue was diluted with EtOAc (50 ml) and washed with saturated aqueous solution of $Na_2S_2O_3$ (8 ml), brine (2×8 ml) and water (2×8 ml), dried over Na_2SO_4, concentrated under reduced pressure and dried at 10-20 Torr. The products **3a-m** were isolated by chromatography on SiO_2 with elution using PE-EA in a linear gradient of the latter from 5 to 20 vol %.

Scale up experiment for electrosynthesis of 3a. An undivided cell was equipped with a graphite plate anode (10 cm^2) and a stainless steel plate cathode (10 cm^2) and connected to a DC regulated power supply. The solution of *p*-toluenesulfonohydrazide **1a** (10 mmol, 1.86g), thiophenol **2a** (20 mmol, 2.2g) and supporting electrolyte NH_4I (10 mmol) in H_2O-THF mixture (1:1, 150 ml) was electrolyzed at constant current (50 mA·cm^{-2}) at 30 °C under magnetic stirring. During the electrosynthesis the mixture is heated, and external cooling is needed. Then electrodes were washed with EtOH (2×50 ml). The solvent from combined organic phases was removed under reduced pressure. The residue was diluted with EtOAc (250 ml) and washed with saturated aqueous solution of $Na_2S_2O_3$ (30 ml), brine (2×30 ml) and water (2×30 ml), dried over Na_2SO_4, concentrated under reduced pressure and dried at 10-20 Torr. The desired product **3a** was isolated in 64% yield (6.4 mmol, 1962 mg) by chromatography on SiO_2 with elution using PE-EA in a linear gradient of the latter from 5 to 20 vol %.

Analytical data for thiosulfonates 3a-m.

S-Phenyl 4-methylbenzenesulfonothioate **(3a)**. [S2]White solid, m.p. = 70.5-72.5°C. Yield 70%. R_f = 0.24 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 2.40 (s, 3H), 7.19 (d, J = 8.1 Hz, 2H), 7.30-7.37 (m, 4H), 7.42-7.48 (m, 3H). ^{13}C NMR ($CDCl_3$), δ: 21.6, 127.5, 128.0, 128.8, 129.3, 131.2, 136.5, 140.3, 144.7.

S-Phenyl 4-methoxybenzenesulfonothioate **(3b)**. [S2]White solid, m.p. = 48.8-50.8°C. Yield 61%. R_f = 0.38 (TLC, PE:EA, 5:1). ^{1}H NMR ($CDCl_3$), δ: 3.84 (s, 3H), 6.83 (d, J = 8.9 Hz, 2H), 7.29-7.36 (m, 4H), 7.42-7.48 (m, 3H).^{13}C NMR ($CDCl_3$), δ: 55.7, 113.8, 128.1, 129.3, 129.8, 131.2, 134.8, 136.5, 163.6.

S-Phenyl 4-fluorobenzenesulfonothioate **(3c)**. [S2]White solid, m.p. = 75.0-77.0°C. Yield 55%. R_f = 0.33 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.07 (t, 2H, J = 8.4 Hz), 7.31-7.36 (m, 4H), 7.45-7.50 (m, 1H), 7.53-7.58 (m, 2H).^{13}C NMR ($CDCl_3$), δ: 116.0 (d, J = 22.6 Hz), 127.7, 129.5, 130.4 (d, J = 10.0 Hz), 131.5, 136.5, 139.0 (d, J = 3.3 Hz), 165.5 (d, J = 256.6 Hz).

S-Phenyl 4-chlorobenzenesulfonothioate **(3d)**. [S2]White solid, m.p. = 77.5-79.5°C. Yield 46%. R_f = 0.38 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.32-7.38 (m, 6H), 7.46-7.51 (m, 3H).^{13}C NMR ($CDCl_3$), δ: 127.5, 128.9, 129.0, 129.6, 131.6, 136.5, 140.2, 141.4.

S-Phenyl 4-bromobenzenesulfonothioate **(3e)**. [S2]White solid, m.p. = 67.0-68.0°C. Yield 66%. R_f = 0.25 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.32-7.40 (m, 6H), 7.46-7.50 (m, 1H), 7.54 (d, J = 8.8 Hz, 2H). ^{13}C NMR ($CDCl_3$), δ: 127.5, 128.8, 128.9, 129.6, 131.6, 132.1, 136.5, 142.0.

S-Phenyl 4-iodobenzenesulfonothioate **(3f)**. White solid, m.p. = 77.5-79.0°C. Yield 57%. R_f = 0.21 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.26 (d, 2H, J = 8.5 Hz), 7.36-7.42 (m, 4H), 7.49-7.55 (m, 1H), 7.79 (d, 2H, J = 8.5 Hz).^{13}C NMR ($CDCl_3$), δ: 101.5, 127.5, 128.7, 129.6, 131.6, 136.5, 138.0, 142.7.

S-Phenyl 4-nitrobenzenesulfonothioate **(3g)**. [S2]Yellow solid, m.p. = 137.0-139.0°C. Yield 25%. R_f = 0.27 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.34-7.43 (m, 4H), 7.49-7.57 (m, 1H), 7.71 (d, J = 8.8 Hz, 2H), 8.25 (d, J = 8.8 Hz, 2H).^{13}C NMR ($CDCl_3$), δ: 124.1, 127.5, 128.7, 129.8, 132.0, 136.5, 147.9, 150.4.

S-Phenyl naphthalene-2-sulfonothioate **(3h)**. [S3]White solid, m.p. = 61.0-62.5°C. Yield 43%. R_f = 0.21 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 7.28-7.37 (m, 4H), 7.47 (t, J = 6.9 Hz, 1H), 7.61 (t, J = 7.3 Hz, 1H), 7.67-7.71 (m, 2H), 7.82 (d, J = 8.1 Hz, 1H), 7.93 (d, J = 8.8 Hz, 2H), 7.98 (s, 1H).^{13}C NMR ($CDCl_3$), δ: 122.3, 127.7, 127.9, 128.0, 129.2, 129.4, 129.4, 129.4, 131.4, 131.5, 135.1, 136.6, 139.6.

S-Phenyl 2,4,6-trimethylbenzenesulfonothioate **(3i)**. White solid, m.p. = 72.5-74.5°C. Yield 30%. R_f = 0.78 (TLC, PE:EA, 5:1). ^{1}H NMR ($CDCl_3$), δ: 2.29 (s, 3H), 2.31 (s, 6H), 6.85 (s, 2H), 7.29-7.33 (m, 4H), 7.42-7.47 (m, 1H).^{13}C NMR ($CDCl_3$), δ: 21.0, 22.6, 127.9, 129.2, 131.2, 131.7, 137.0, 140.1, 143.6.

S-*p*-Tolyl 4-methylbenzenesulfonothioate **(3j)**. [S2]White solid, m.p. = 70.5-72.5°C. Yield 68%. R_f = 0.62 (TLC, PE:EA, 5:1). ^{1}H NMR ($CDCl_3$), δ: 2.36 (s, 3H), 2.44 (s, 3H), 7.15 (d, J = 8.1 Hz, 2H), 7.24 (t, J = 8.1 Hz, 4H), 7.47 (d, J = 8.1 Hz, 2H).^{13}C NMR ($CDCl_3$), δ: 21.4, 21.6, 124.5, 127.5, 129.3, 130.2, 136.4, 140.4, 142.0, 144.6.

S-(4-Chlorophenyl) 4-methylbenzenesulfonothioate **(3k)**. [S2]White solid, m.p. = 80.5-82.5°C. Yield 80%. R_f = 0.22 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 2.45 (s, 3H), 7.24-7.32 (m, 6H), 7.48 (d, J = 8.1 Hz, 2H).^{13}C NMR ($CDCl_3$), δ: 21.6, 126.5, 127.6, 129.5, 129.6, 137.7, 138.1, 140.2, 145.0.

S-Isobutyl 4-methylbenzenesulfonothioate **(3l)**. Colorless liquid. Yield 35%. R_f = 0.46 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 0.89 (d, J = 6.6 Hz, 6H), 1.77-1.91 (m, 1H), 2.43 (s, 3H), 2.85 (d, J = 6.7 Hz, 2H), 7.31 (d, J = 8.1 Hz, 2H), 7.78 (d, J = 8.1 Hz, 2H).^{13}C NMR ($CDCl_3$), δ: 21.6, 21.6, 28.1, 44.3, 126.9, 129.7, 142.1, 144.5.

S-Butyl 4-methylbenzenesulfonothioate **(3m)**. [S4]Colorless liquid. Yield 55%. R_f = 0.46 (TLC, PE:EA, 10:1). ^{1}H NMR ($CDCl_3$), δ: 0.83 (t, J = 7.3 Hz, 3H), 1.25-1.37 (m, 2H), 1.51-1.61 (m, 2H), 2.43 (s, 3H), 2.96 (t, J = 7.3 Hz, 2H), 7.32 (d, J = 8.2 Hz, 2H), 7.79 (d, J = 8.2 Hz, 2H). ^{13}C NMR ($CDCl_3$), δ: 13.3, 21.6, 30.6, 35.6, 127.0, 129.8, 142.0, 144.6.

References

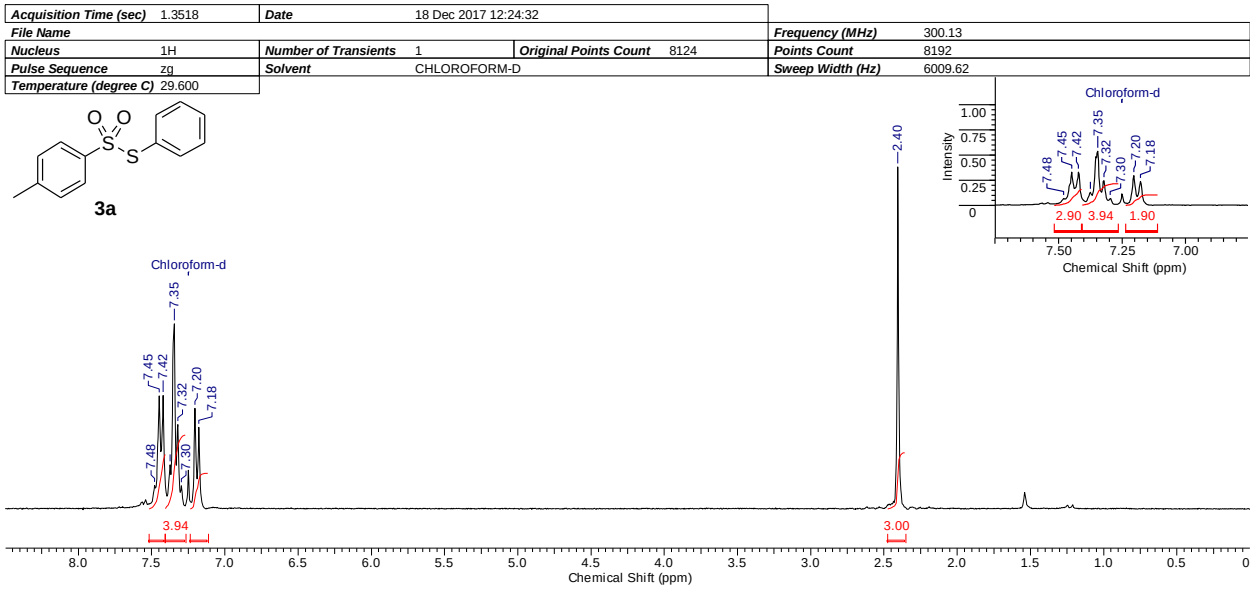
[S1] A. O. Terent'ev, O. M. Mulina, D. A. Pirgach, A. I. Ilovaisky, M. A. Syroeshkin, N. I. Kapustina and G. I. Nikishin, *Tetrahedron*,2017,**73**, 6871.

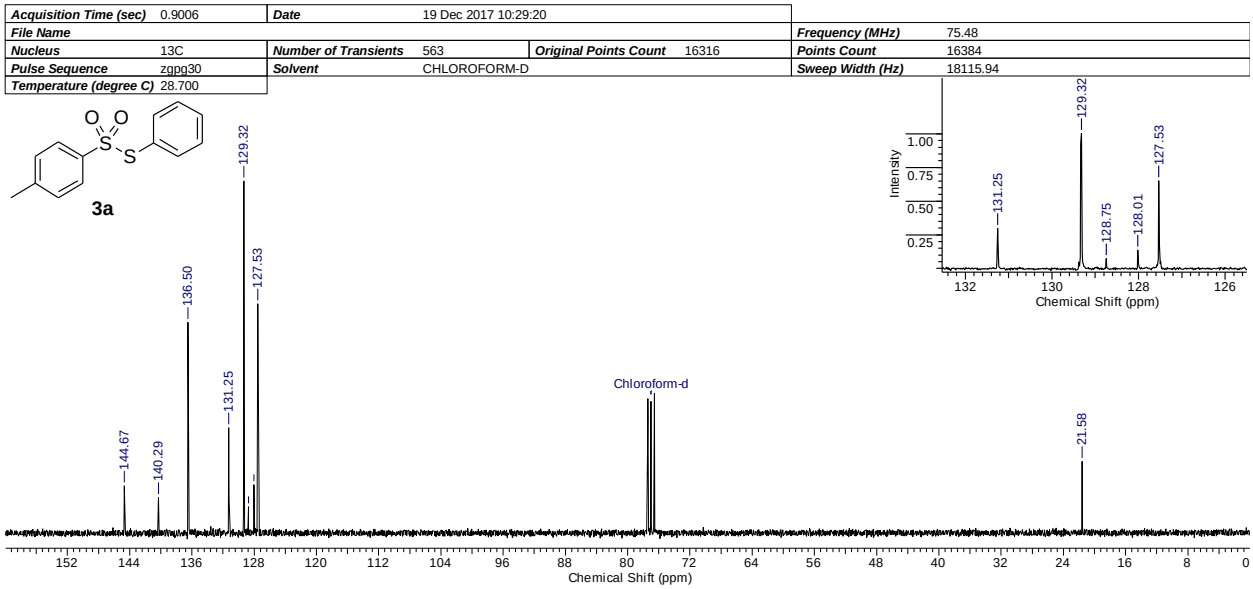
[S2] G. Y. Zhang, S. S. Lv, A. Shoberu and J. P. Zou, *J. Org. Chem.*,2017, **82**, 9801.

[S3] Z. Peng, X. Zheng, Y. Zhang, D. An and W. Dong, *Green Chem.*,2018, **20**, 1760.

[S4] N. Taniguchi, *J. Org. Chem.*,2015, **80**, 1764.

NMR Spectra of Synthesized Compounds





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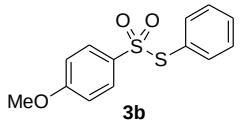

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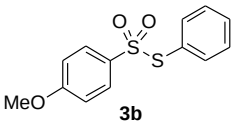

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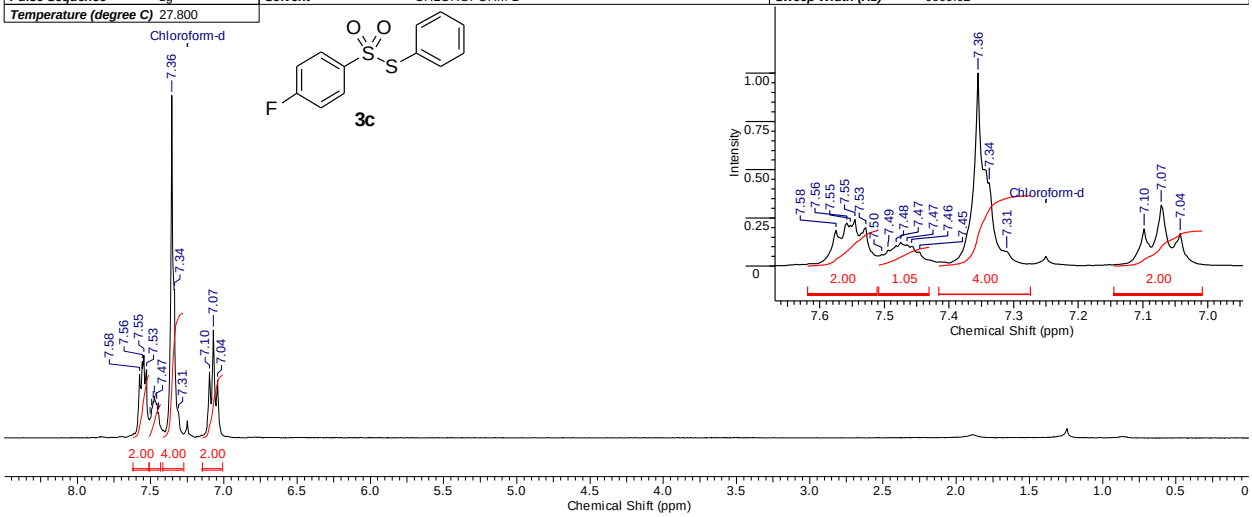

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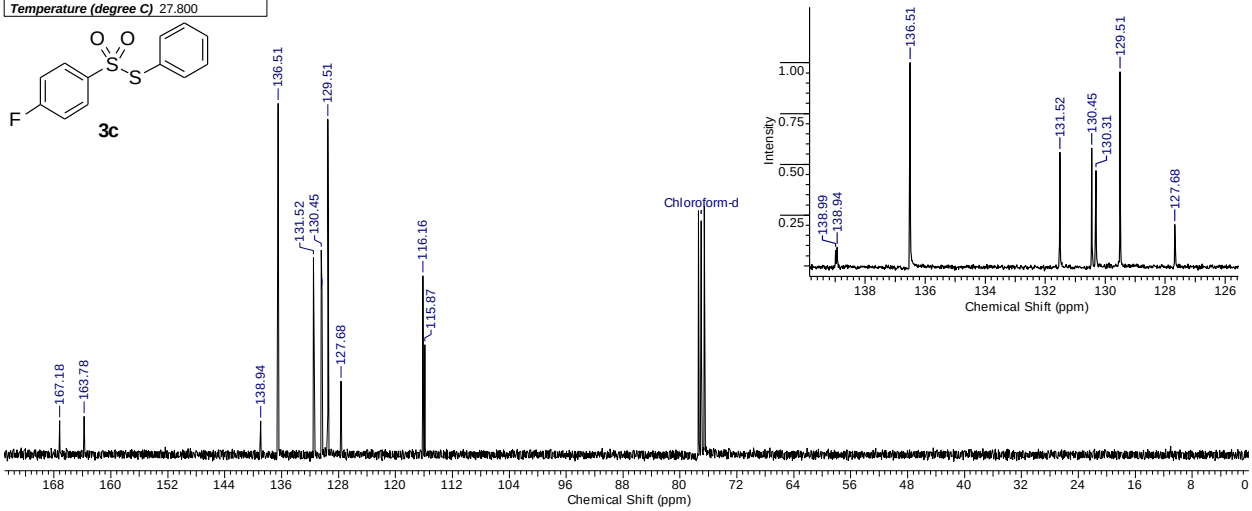

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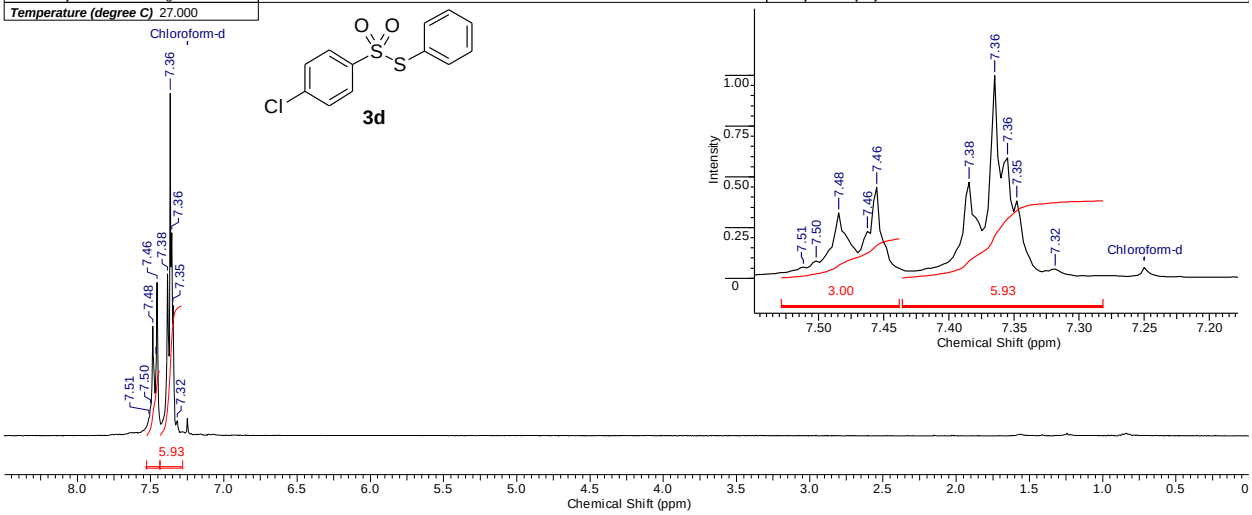

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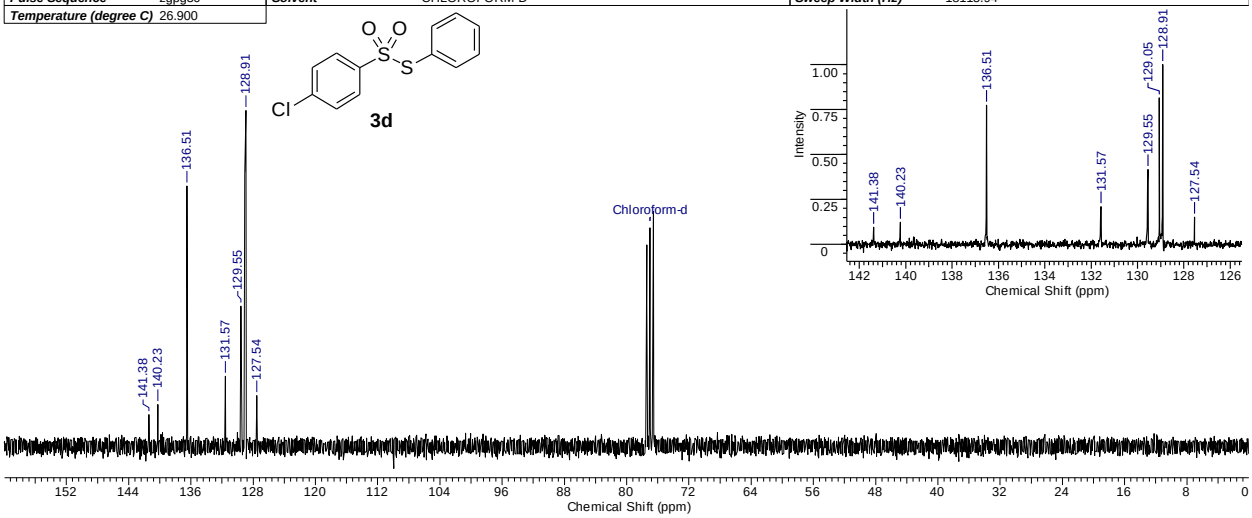

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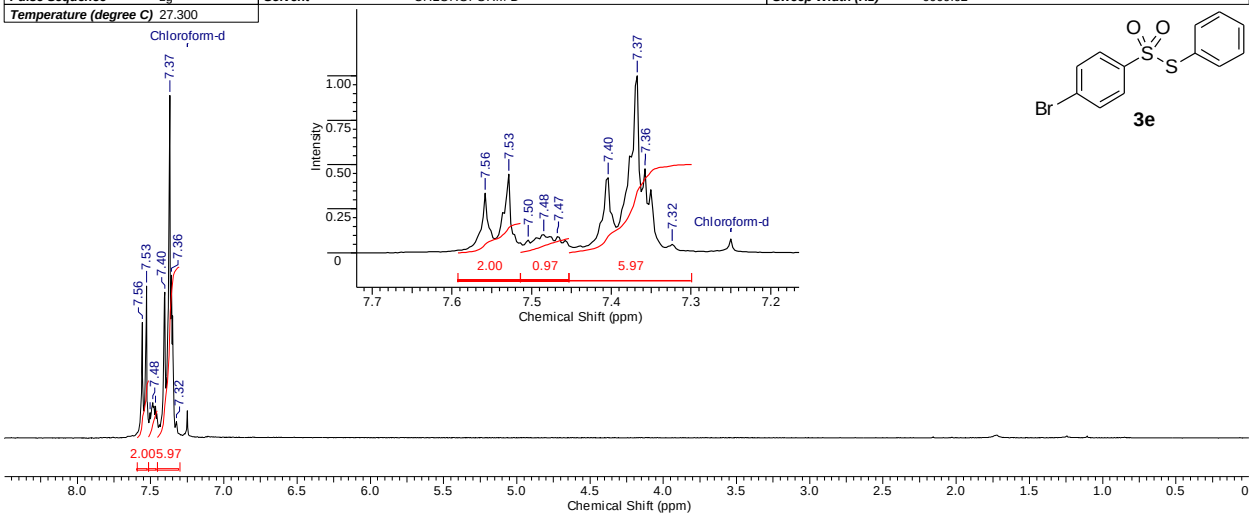

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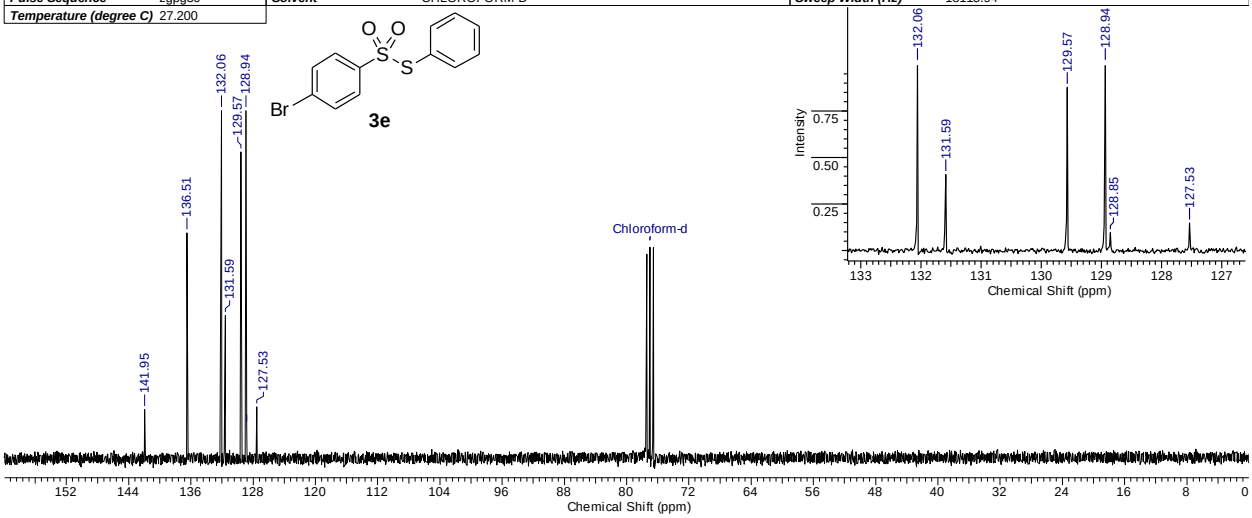

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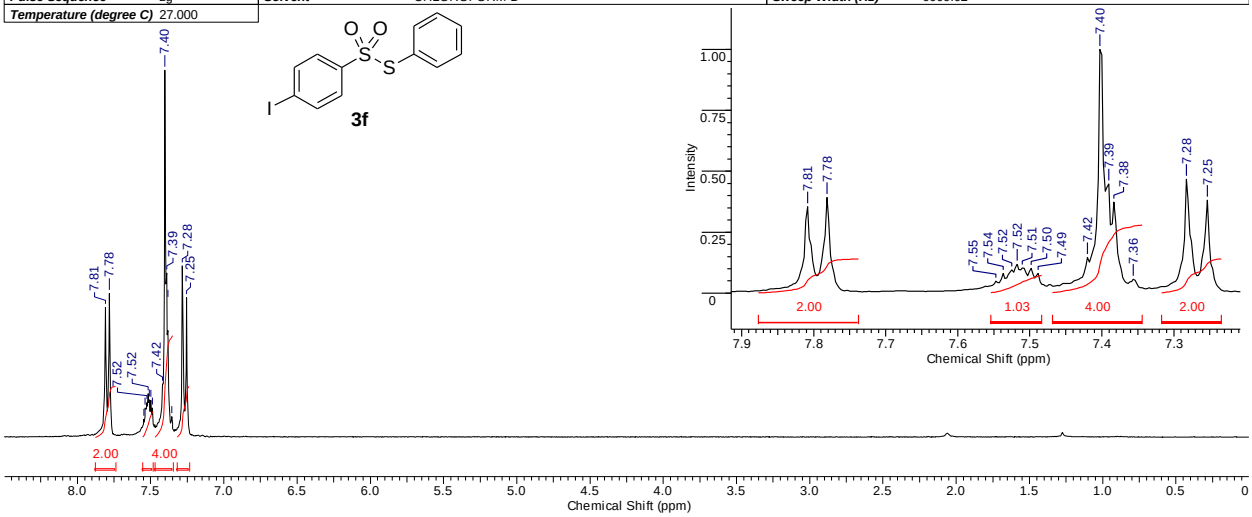

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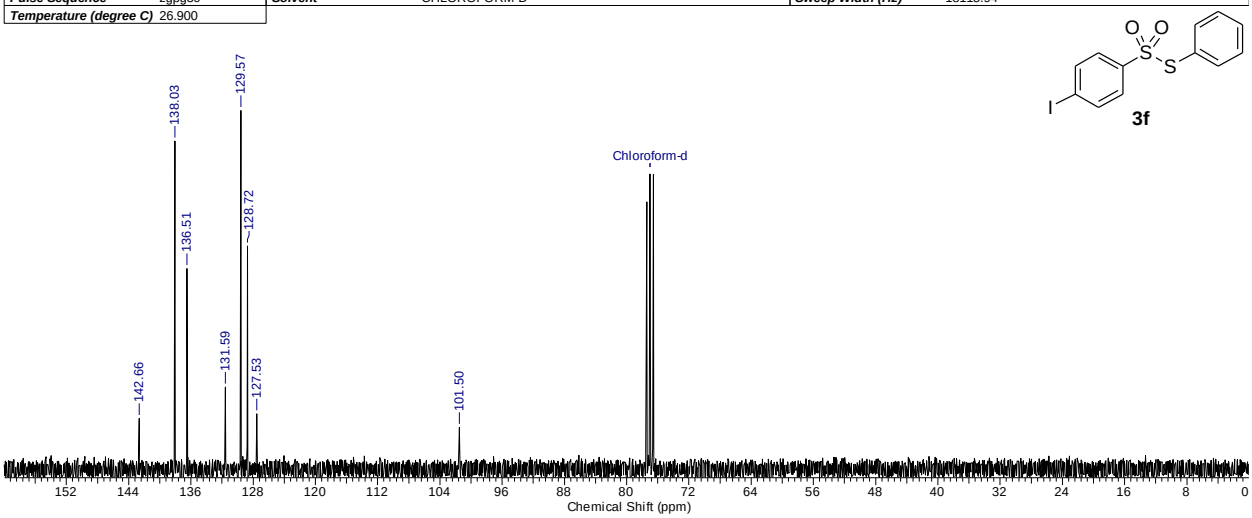

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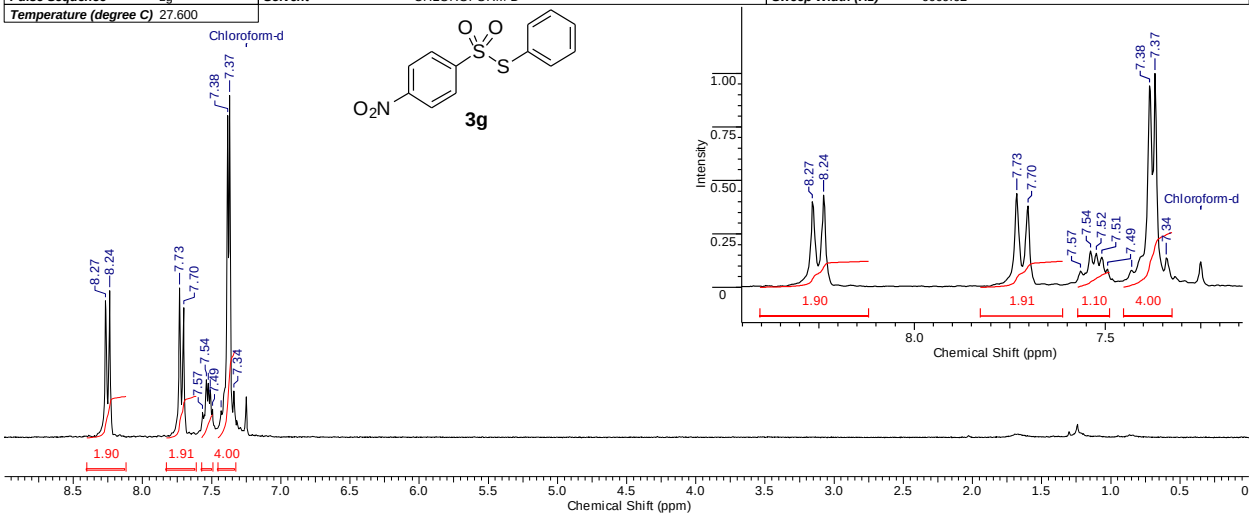

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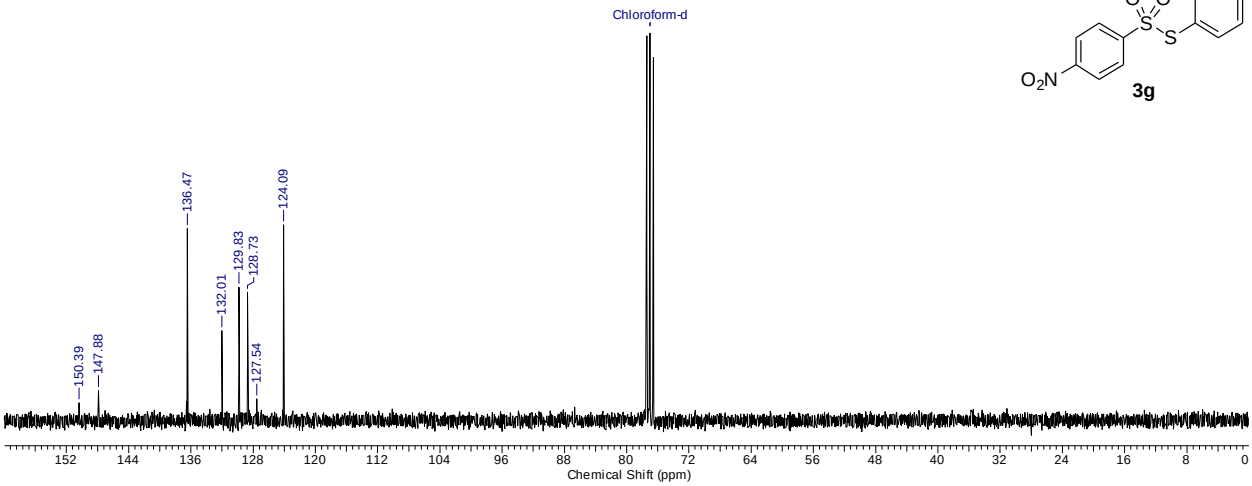

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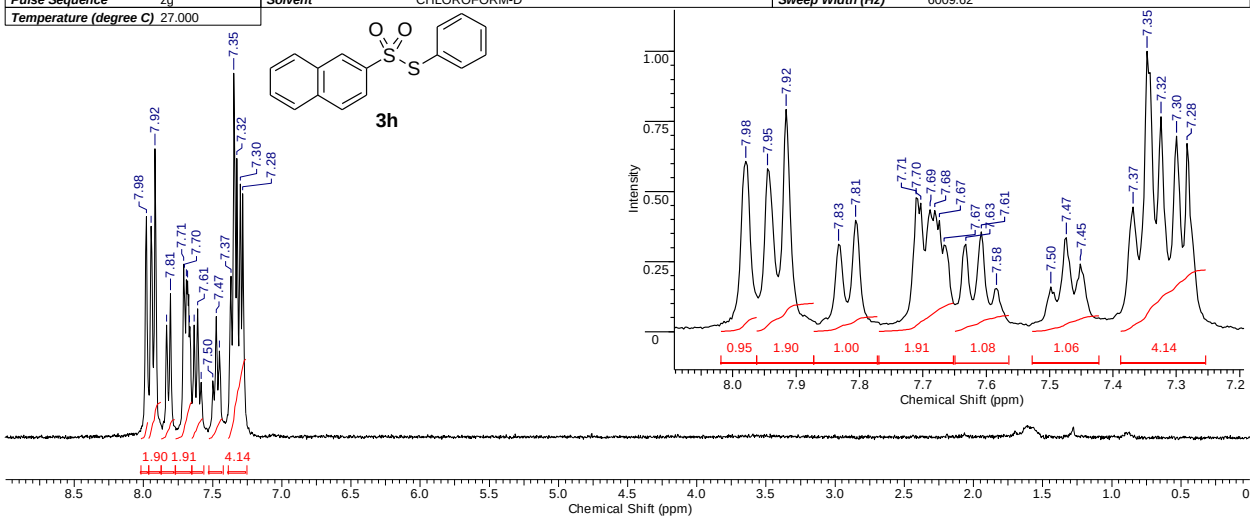

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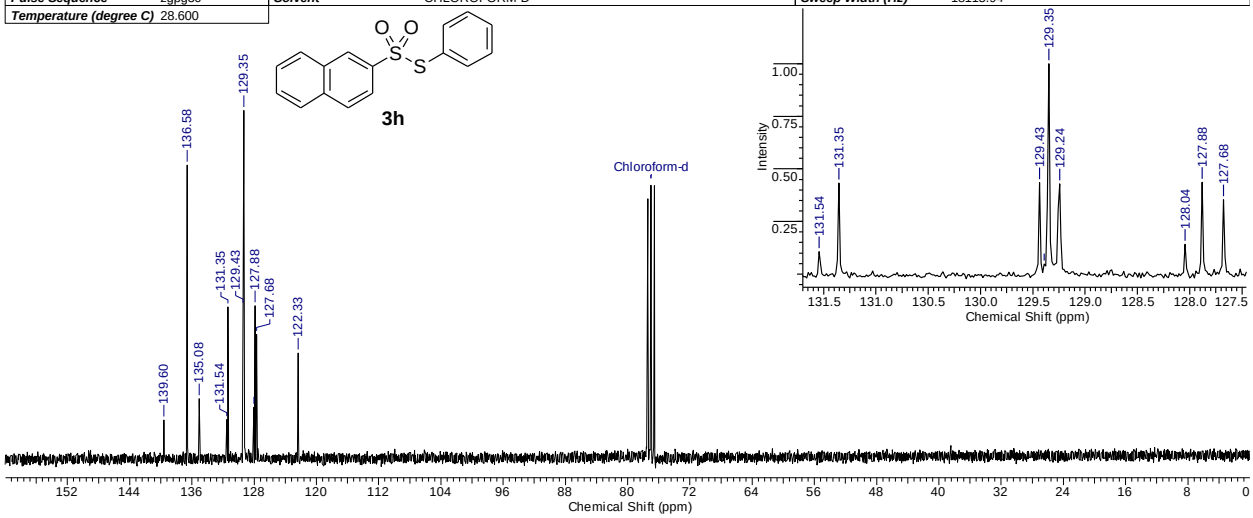
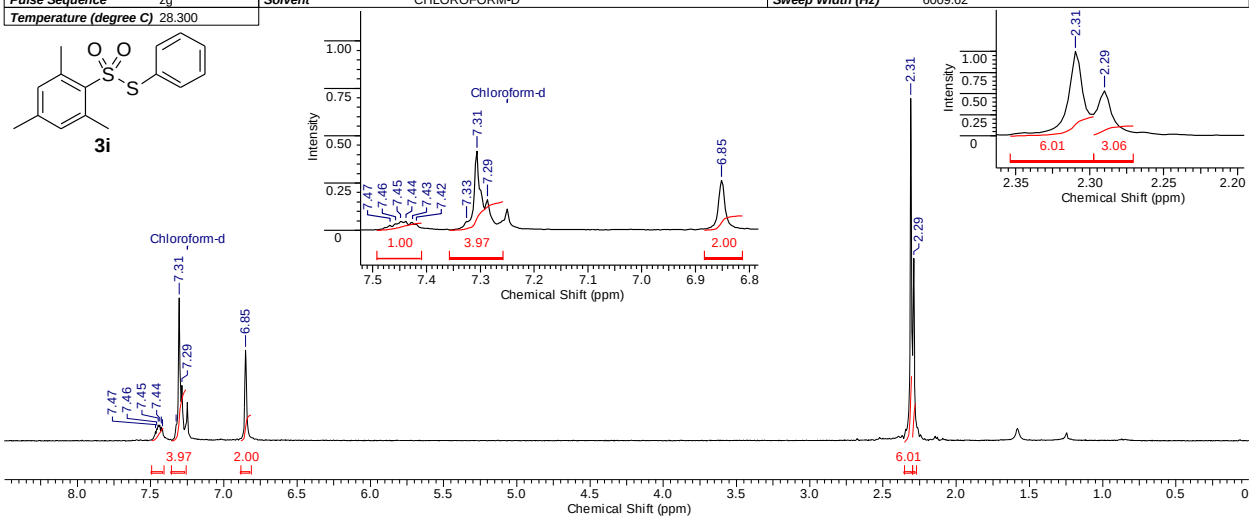
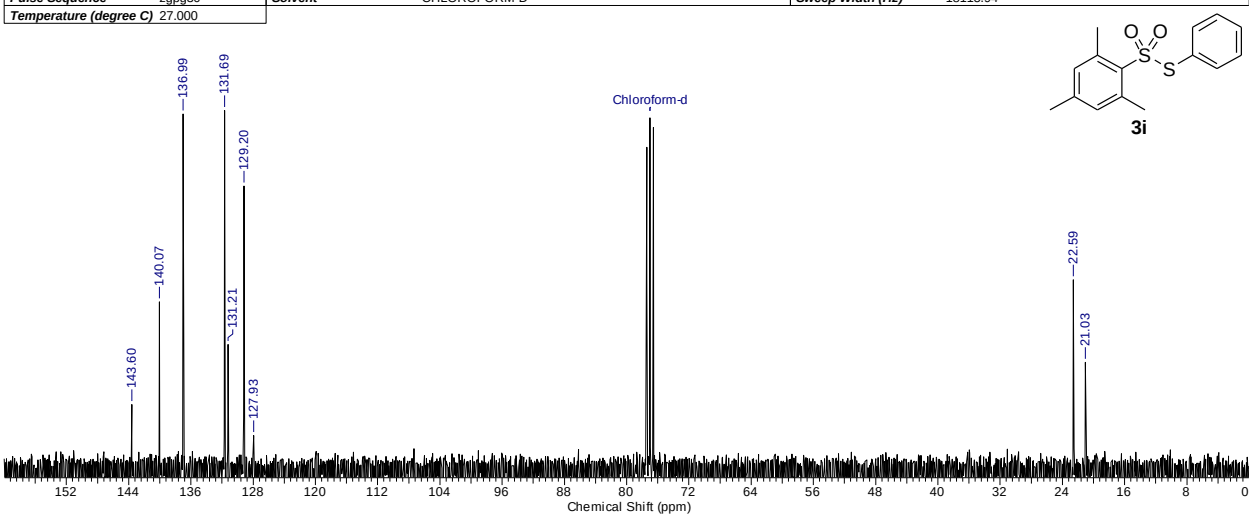
Pulse Sequence	zgpg30	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	18115.94
Temperature (degree C)	28.600						



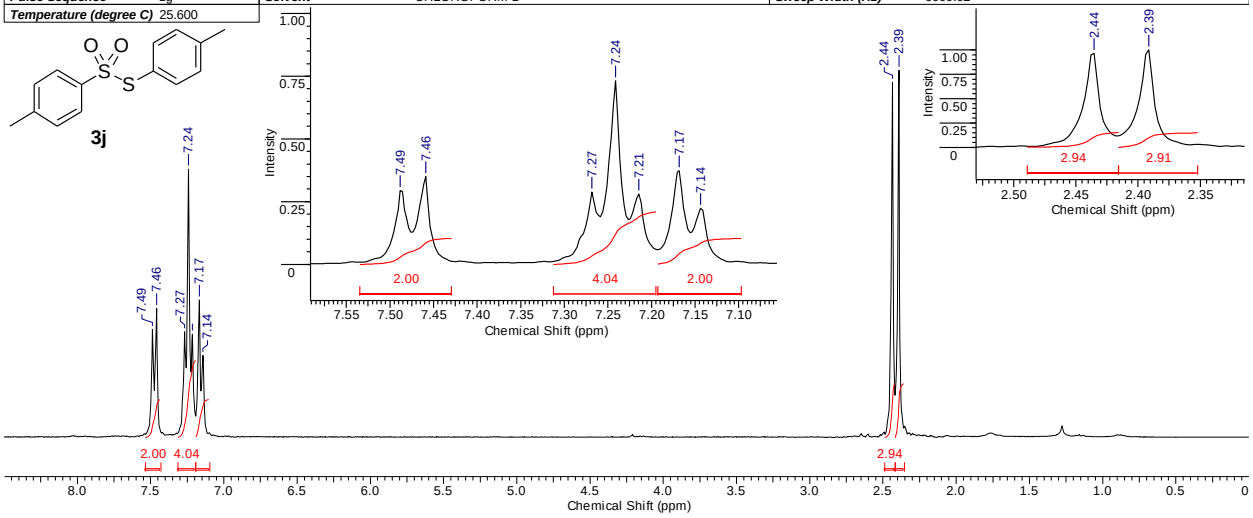
Acquisition Time (sec)	1.3518	**Date**	14 May 2018 16:12:48		
File Name				**Frequency (MHz)**	300.13
Nucleus	1H	**Number of Transients**	1	**Original Points Count**	8124	**Points Count**	8192
Pulse Sequence	zg	**Solvent**	CHLOROFORM-D	**Sweep Width (Hz)**	6009.62
Temperature (degree C)	28.300				



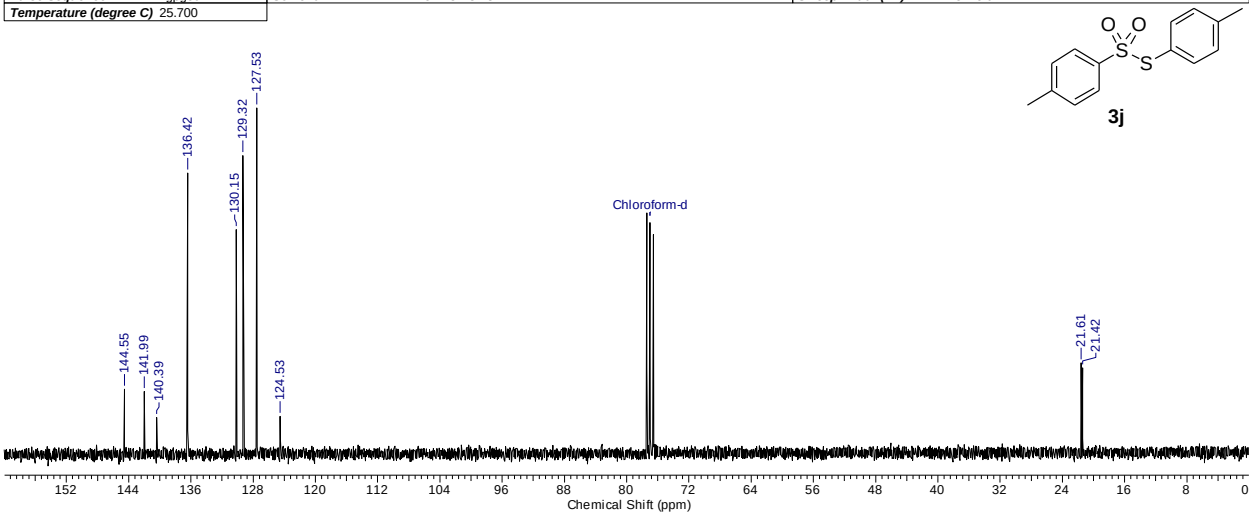
Acquisition Time (sec)	0.9006	**Date**	25 May 2018 09:14:40		
File Name				**Frequency (MHz)**	75.48
Nucleus	13C	**Number of Transients**	122	**Original Points Count**	16316	**Points Count**	16384
Pulse Sequence	zgpg30	**Solvent**	CHLOROFORM-D	**Sweep Width (Hz)**	18115.94
Temperature (degree C)	27.000				



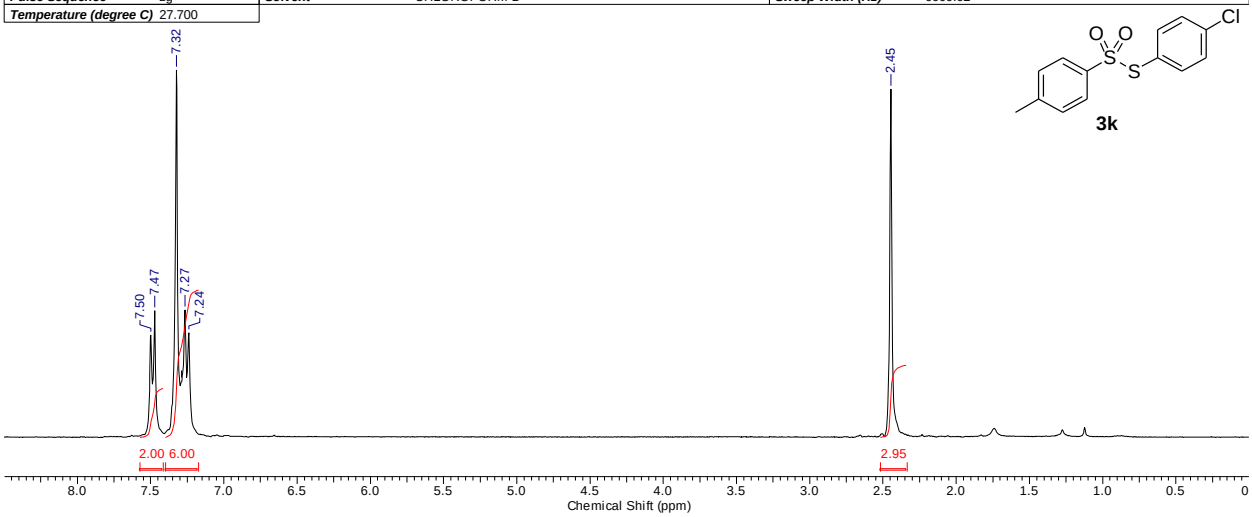
Acquisition Time (sec)	1.3518	**Date**	03 Jul 2018 12:54:24		
File Name				**Frequency (MHz)**	300.13
Nucleus	1H	**Number of Transients**	1	**Original Points Count**	8124	**Points Count**	8192
Pulse Sequence	zg	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	6009.62
Temperature (degree C)	25.600						



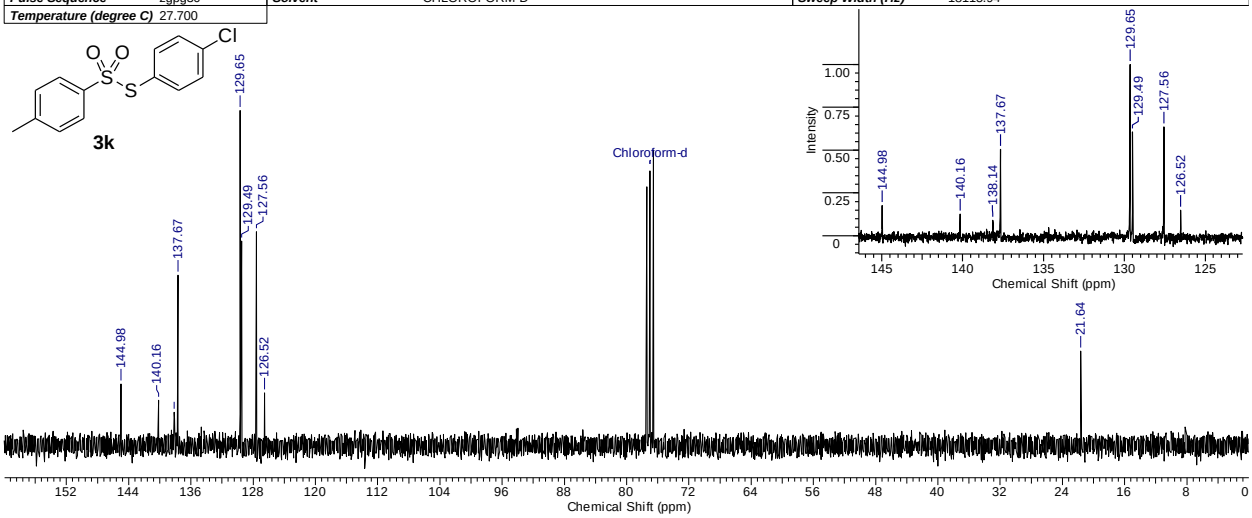
Acquisition Time (sec)	0.9006	**Date**	03 Jul 2018 12:54:24		
File Name				**Frequency (MHz)**	75.48
Nucleus	13C	**Number of Transients**	254	**Original Points Count**	16316	**Points Count**	16384
Pulse Sequence	zgpg30	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	18115.94
Temperature (degree C)	25.700						



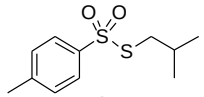
Acquisition Time (sec)	1.3518	**Date**	04 Jul 2018 09:14:40				
File Name						**Frequency (MHz)**	300.13
Nucleus	1H	**Number of Transients**	1	**Original Points Count**	8124	**Points Count**	8192
Pulse Sequence	zg	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	6009.62
Temperature (degree C)	27.700						



Acquisition Time (sec)	0.9006	**Date**	04 Jul 2018 09:16:48				
File Name						**Frequency (MHz)**	75.48
Nucleus	13C	**Number of Transients**	109	**Original Points Count**	16316	**Points Count**	16384
Pulse Sequence	zgpg30	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	18115.94
Temperature (degree C)	27.700						

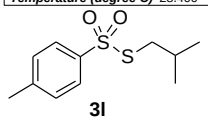


Acquisition Time (sec)	1.3518	***Date***	30 Jul 2018 09:04:00				
File Name						***Frequency (MHz)***	300.13
Nucleus	1H	***Number of Transients***	1	***Original Points Count***	8124	***Points Count***	8192
Pulse Sequence	zg	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	6009.62
Temperature (degree C)	28.400						



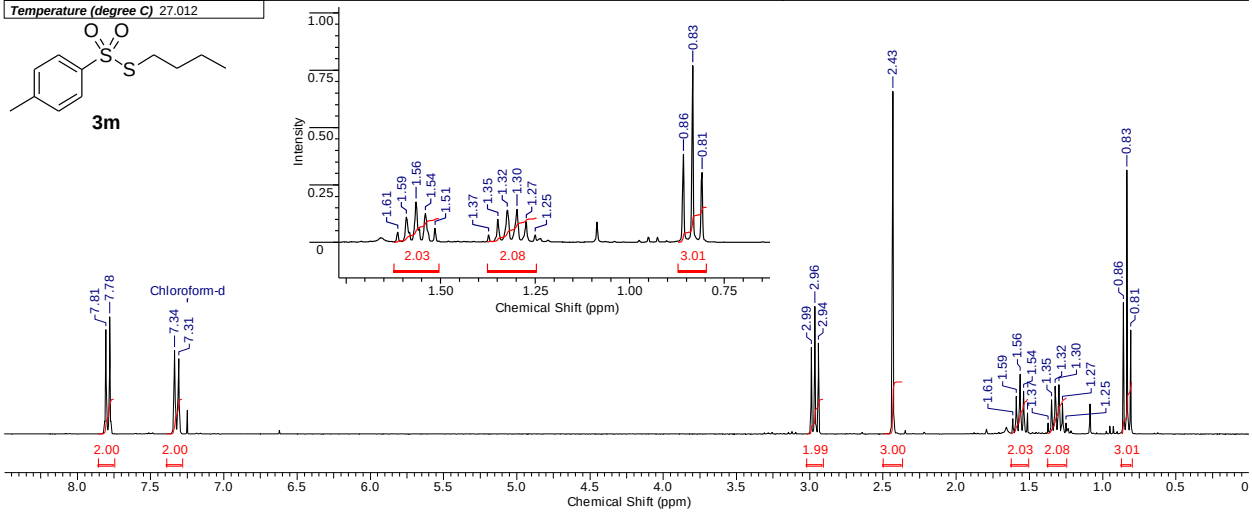
3l

Acquisition Time (sec)	0.9006	***Date***	30 Jul 2018 09:06:08				
File Name						***Frequency (MHz)***	75.48
Nucleus	13C	***Number of Transients***	294	***Original Points Count***	16316	***Points Count***	16384
Pulse Sequence	zgpg30	***Solvent***	CHLOROFORM-D			***Sweep Width (Hz)***	18115.94
Temperature (degree C)	28.400						



3l

Acquisition Time (sec)	2.7150	**Comment**		**Date**	04 Sep 2018 13:13:36
File Name				**Frequency (MHz)**	300.13
Nucleus	1H	**Number of Transients**	1	**Original Points Count**	16384	**Points Count**	16384
Pulse Sequence	zg	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	6009.62
Temperature (degree C)	27.012						



Acquisition Time (sec)	0.9050	**Comment**		**Date**	04 Sep 2018 13:13:36
File Name				**Frequency (MHz)**	75.48
Nucleus	13C	**Number of Transients**	55	**Original Points Count**	16384	**Points Count**	16384
Pulse Sequence	zgpg30	**Solvent**	CHLOROFORM-D			**Sweep Width (Hz)**	18028.85
Temperature (degree C)	27.055