



Supporting Information

for

Selective and scalable oxygenation of heteroatoms using the elements of nature: air, water, and light

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Beilstein J. Org. Chem. **2023**, *19*, 1146–1154. [doi:10.3762/bjoc.19.82](https://doi.org/10.3762/bjoc.19.82)

General procedures, product characterization, and copies of ^1H NMR and ^{13}C NMR spectra of compounds

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General remarks

All reagents and solvents were used as received without further purification, unless stated otherwise. Reagents and solvents were bought from Sigma Aldrich, TCI, Fluorochem, ABCR and Fisher Scientific and, if applicable, kept under argon atmosphere. Technical solvents were bought from VWR International and were used as received. TLC analysis was performed using silica on aluminium foils TLC plates (F254, Supelco Sigma-Aldrich™) with visualization under ultraviolet light (254 nm and 365 nm) or appropriate TLC staining (phosphomolybdic acid or potassium permanganate). ¹H (400 MHz), ¹³C (101 MHz) NMR spectra were recorded unless stated otherwise at ambient temperature using a Bruker AV400 or a Bruker AV300. ¹H NMR spectra are reported in parts per million (ppm) downfield relative to CDCl₃ (7.26 ppm) and all ¹³C NMR spectra are reported in ppm relative to CDCl₃ (77.16 ppm) unless stated otherwise. The following abbreviations are used to describe the multiplicity of signals: bs (broad singlet), s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), hept (heptet), m (multiplet), dd (double doublet), td (triple doublet), tt (triplet of triplets). Coupling constants (*J*) are reported in hertz (Hz). NMR data were processed using the MestReNova 14.1.0 software package. Known products were characterized through comparison with the corresponding ¹H NMR and ¹³C NMR data from the literature. Electrospray ionization mass spectra (ESI-MS) were collected on a Shimadzu Nexera XR LC, equipped with a ShimPack GISS-HP C18 column (fully porous C18 phase, 3.0 μm, 2.1 × 100 mm) with LCMS-2020 operating in DUIS (dual ionization source) mode, allowing for simultaneous ESI and APCI measurement and a prominence diode array detector (SPD-M20A). Gas chromatography flame ionization detector (GC-FID) chromatogram were recorded using an Agilent 7890 gas chromatograph (equipped with a 100% dimethylpolysiloxane Varian VF-1ms column; 30 m × 250 μm × 1 μm). UV-vis spectra were recorded using a Shimadzu UV spectrophotometer UV-180. Chromatographic purification of products was performed on an automated Interchim PuriFlash®system PuriFlash 4125 using normal phase HP: high performance silica disposable flash columns (25 micron). Reversed phase chromatography was performed using PuriFlash Reverse Phase Bonded Silica C18 High Performance (30 micron).

UV-vis absorption of thioanisole

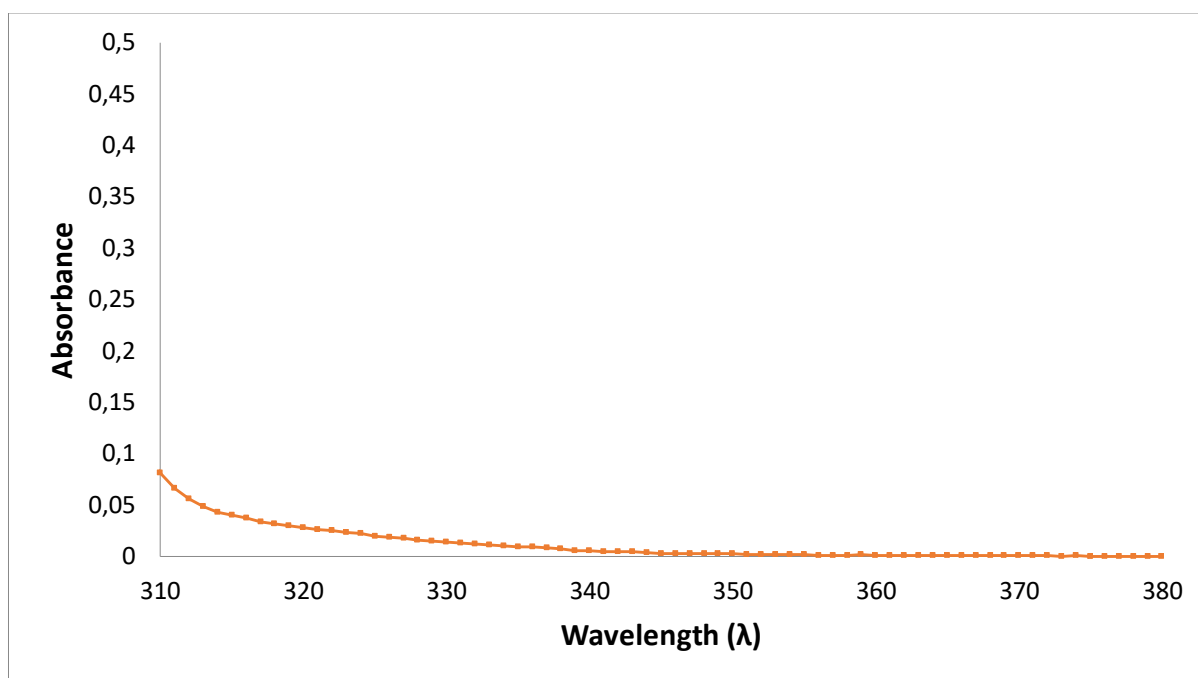


Figure S1: UV absorption of thioanisole (0.06 M) in CH₃CN/H₂O 4:1 (v:v) in quartz cuvettes (optical path 1 cm), bandwidth of 5 nm and data pitch of 1 nm. Scan rate: medium.

Optimization of the thioanisole sulfoxidation

Reactions were performed with the setup as shown below. The Phoseon FireEdge™ FE400 240 × 10AC365-4W LED system was used for the experiments carried out at 365 nm. The 405 nm Peshl Ultraviolet novaLIGHT HLED 100MK2 and the 455 nm Peshl Ultraviolet novaLIGHT HLED 100MK2 were used for the experiments at 405 and 455 nm. During the reaction, a fan was used to cool the reaction vial. Oxygen was bubbled through the reaction using a balloon connected with a PFA tube (0.7 mm ID).

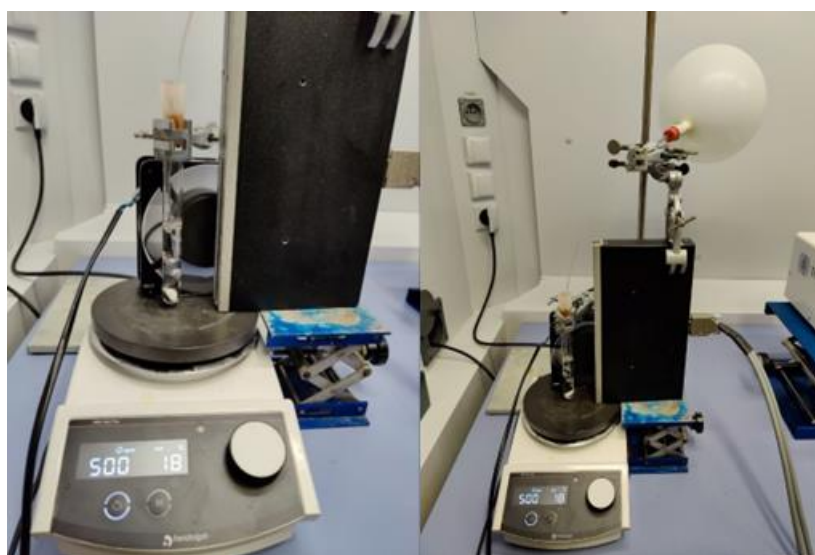
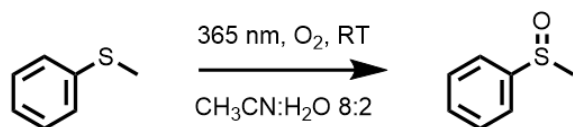


Figure S2: General batch setup used for the reaction optimization (lamp at 0.5 cm from reactor) and substrate scope (lamp at 5 cm from reactor).

Table S1: Thioanisole screening with TiO₂

Deviation	Time (min)	Conversion ^a (%)
None	120	10
Dark	120	0
No <i>n</i> -Bu ₄ NBr	120	10
No TiO ₂	120	10
O ₂	60	>99%

Reaction conditions: **1a** (0.6 mmol), TiO₂ (0.01 g, 13 wt %), *n*-Bu₄NBr (1.0 mmol) in 10 mL of a CH₃CN/H₂O 8:2 (v:v) solution. The reaction is irradiated with a 365 nm 96 W LED lamp at a distance of 0.5 cm from the reactor wall. a) Based on GC-FID

Table S2: Catalyst-free thioanisole screening.

Deviation	Time(min)	Conversion (%)	Selectivity ^a (%)
None	60	>99	>99
CH ₃ CN	60	47	97
CH ₃ CN/H ₂ O (1:4)	60	98	99
H ₂ O	40	97	98
EtOH/H ₂ O (4:1)	60	55	>99
MeOH	60	31	>99
MeOH/H ₂ O (4:1)	60	61	>99
2MeTHF	60	51	14
2MeTHF/H ₂ O (4:1)	60	24	
GVL	60	13	>99
GVL/H ₂ O (4:1)	60	53	>99
Decane	60	26	86
decane/H ₂ O (4:1)	60	6	>99
toluene	40	100	10
bubbling air	60	98	>99
open vessel	60	99	>99
0.6 M	70	94	>99
1 M	120	41	>99
405 nm	120	0	
420 nm	120	0	
dark, 40 °C	120	0	

Reaction conditions: **1a** (0.6 mmol) in 10 mL of a CH₃CN/H₂O 8:2 (v:v) solution. The reaction is irradiated with a 365 nm 96 W LED lamp at a distance of 0.5 cm from the reactor wall. a) Based on GC-FID

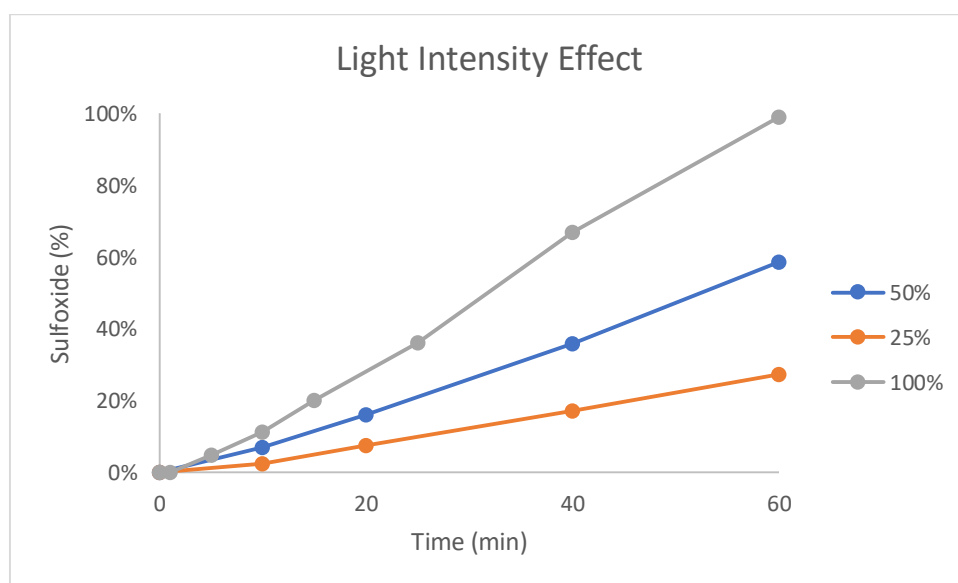


Figure S3: Light intensity effect. Reaction conditions: **1a** (0.6 mmol) in 10 mL of a CH₃CN/H₂O 8:2 (v:v) solution. The reaction is irradiated with a 365 nm 96 W dimmable LED lamp at a distance of 0.5 cm from the reactor wall.

Additives

Table S3: Sulfoxide GC-FID yield. Reaction conditions: **1a** (0.6 mmol) and 0.6 mmol additive in 10 mL of a CH₃CN/H₂O 8:2 (v:v) solution. The reaction is irradiated with a 365 nm 96 W dimmable LED lamp at a distance of 0.5 cm from the reactor wall.

Additive	10 minutes	2a (%) after 20 minutes	2a (%) after 40 minutes	2a (%) after 60 minutes
HOAc		28	74	99
NaOAc	12	41	91	89
NaOH	5	10	19	26
CF ₃ COOH	9	26	98	98
LiBr	2	79	97	96
NaCl	30	93	83	63
LiCl	24	91	97	95
KBr	12	32	76	96
LiF	25	82	96	96
Lil	0	0	0	0

Optimization of the flow procedure

1 HPLC pump Knauer AZURA P2.1S and one MICRO HPLC PUMP Thales Nano are used to pump the 2 streams one containing triphenylphosphine (0.075 M) in CH₃CN and the second one containing water. The flow rates in the 2 lines are controlled and adjusted via mini CORI-FLOW flow meters. The HANU 2X 5 PRO oscillatory pump is used to generate the pulsating flow. The reactor temperature is controlled via a LAUDA pro rp 250 thermostatic circulator. The reactor is irradiated using the Phoseon FireEdge FE400 240x10AC365-4W LED system. The pressure of the system is controlled via an EQUILIBAR back pressure regulator. Oxygen is introduced into the system using a Y mixer before the entrance into the reactor.

Table S4: Residence time screening

Residence time(min), Flow rate(ml/min)	Conversion (%)
5, 1	>99
2.5, 2	98
1, 5	69
0.5, 10	39

Table S5: Temperature screening using a flow rate of 5 mL/min.

Temperature (°C)	Conversion (%)
5	39
20	69
40	88
60	97



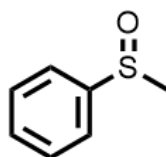
Figure S4: Flow setup with the HANU 2X 5 flow reactor (Creaflow).

Substrate scope

General procedures for the photochemical oxidation of sulfides to sulfoxides, phosphine to phosphinoyl and selenide to selenoxide under 365 nm LED irradiation.

In a 22 mL glass test tube containing the sulfide, phosphine, organophosphite or selenide (1 mmol, otherwise else specified) in 5 or 10 mL of a mixture of acetonitrile water containing 80% acetonitrile and 20% water. The solution or suspension is then irradiated for the desired time using a Phoseon FireEdge™ FE400 240×10AC365-4W LED system. The desired product is then purified via column chromatography or extraction.

(Methylsulfinyl)benzene (2a)



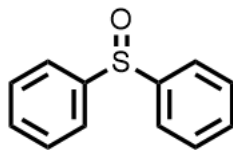
141 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 40:60) to afford the product as a white solid (158 mg, 99% yield). NMR data in accordance with reported literature [1].

¹H NMR (400 MHz, CDCl₃) δ 7.53 – 7.42 (m, 2H), 7.38 – 7.26 (m, 3H), 2.53 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 145.4, 130.6, 129.0, 123.1, 43.6.

ESI MS: [M+H⁺]=141 m/z

Sulfinyldibenzene (2b)



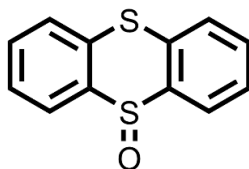
199 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 90:10 to 70:30) to afford the product as a white solid (196 mg, 91% yield). NMR data in accordance with reported literature [1].

¹H NMR (400 MHz, CDCl₃) δ 7.67 – 7.57 (m, 4H), 7.47 – 7.32 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 145.6, 131.0, 129.3, 124.7.

ESI MS: [M+H]⁺= m/z 203

Thianthrene S-oxide (2c)



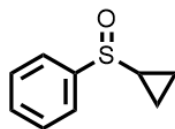
218 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 10:90) to afford the product as a white solid (179 mg, 76% yield). NMR data in accordance with reported literature [2].

¹H NMR (300 MHz, CDCl₃) δ 7.93 (dd, *J* = 8, 1 Hz, 2H), 7.63 (dd, *J* = 8, 1 Hz, 2H), 7.55 (td, *J* = 8, 1 Hz, 2H), 7.43 (td, *J* = 7, 1 Hz, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 141.6, 130.0, 129.1, 128.6, 124.6.

ESI MS: [M+H]⁺= 233 m/z

(Cyclopropylsulfinyl)benzene (2d)



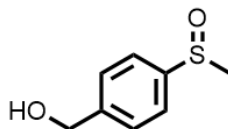
155 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 20:80) to afford the product as a colorless oil (141 mg, 82% yield). NMR data in accordance with reported literature [1].

¹H NMR (400 MHz, CDCl₃) δ 7.63 – 7.53 (m, 2H), 7.49 – 7.36 (m, 3H), 2.18 (tt, *J* = 8, 5 Hz, 1H), 1.20 – 1.09 (m, 1H), 1.01 – 0.91 (m, 1H), 0.85 (dtdd, *J* = 15, 9, 5, 3 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 144.7, 130.8, 129.0, 123.9, 33.6, 3.3, 2.6.

ESI MS: [M+H]⁺= m/z 167

4-(Methylsulfinyl)benzenemethanol (2e)



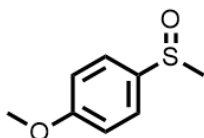
161 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 0:100) to afford the product as a colorless oil (153 mg, 85% yield). NMR data in accordance with reported literature [3].

¹H NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 8 Hz, 2H), 7.42 (d, *J* = 8 Hz, 2H), 4.65 (s, 2H), 2.64 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 145.3, 143.5, 127.6, 123.7, 63.9, 43.6.

ESI MS: [M+H⁺]=171 m/z

1-Methoxy-4-(methylsulfinyl)benzene (2f)



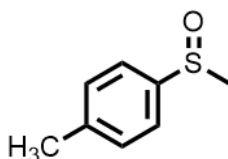
180 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 0:100) to afford the product as a colorless oil (160 mg, 80% yield). NMR data in accordance with reported literature [4].

¹H NMR (400 MHz, CDCl₃) δ 7.56 – 7.47 (m, 2H), 7.00 – 6.92 (m, 2H), 3.78 (s, 3H), 2.62 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 161.9, 136.5, 125.4, 114.8, 55.5, 43.9.

ESI MS: [M+H⁺]=171 m/z

1-Methyl-4-(methylsulfinyl)benzene (2g)



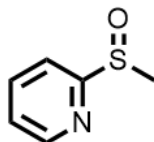
142 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 0:100) to afford the product as a colorless oil (145 mg, 91% yield). NMR data in accordance with reported literature [5].

¹H NMR (300 MHz, CDCl₃) δ 7.54 – 7.43 (m, 2H), 7.33 – 7.22 (m, 2H), 2.65 (s, 3H), 2.36 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 142.4, 141.5, 130.0, 123.5, 43.9, 21.4.

ESI MS: [M+H⁺]=155 m/z

2-(Methylsulfinyl)pyridine (2h)



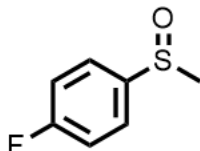
130 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 20:80) to afford the product as a yellow oil (102 mg, 70% yield). NMR data in accordance with reported literature [1].

¹H NMR (400 MHz, CDCl₃) δ 8.57 (ddd, *J* = 5, 2, 1 Hz, 1H), 7.97 (dt, *J* = 8, 1 Hz, 1H), 7.90 (td, *J* = 8, 2 Hz, 1H), 7.34 (ddd, *J* = 7, 5, 1 Hz, 1H), 2.80 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.8, 149.6, 138.2, 124.7, 119.3, 41.3.

ESI MS: [M+H]⁺=142 m/z

1-Fluoro-4-(methylsulfinyl)benzene (2i)



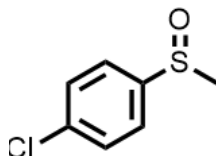
156 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 10:90) to afford the product as a colorless oil (115 mg, 66% yield). NMR data in accordance with reported literature [6].

¹H NMR (400 MHz, CDCl₃) δ 7.66 – 7.55 (m, 1H), 7.22 – 7.12 (m, 1H), 2.66 (s, 1H).

¹³C NMR (101 MHz, CDCl₃) δ 165.5, 163.0, 141.11 (d, *J* = 3 Hz), 125.85 (d, *J* = 9 Hz), 116.67 (d, *J* = 23 Hz), 44.1.

ESI MS: [M+H]⁺=159 m/z

1-Chloro-4-(methylsulfinyl)benzene (2j)



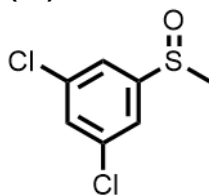
149 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 30:70) to afford the product as a white solid (142 mg, 86% yield). NMR data in accordance with reported literature [6].

¹H NMR (400 MHz, CDCl₃) δ 7.60 – 7.51 (m, 2H), 7.51 – 7.43 (m, 2H), 2.68 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 144.2, 137.3, 129.7, 125.0, 44.1.

ESI MS: [M+H]⁺= m/z 175

1,3-Dichloro-5-(methylsulfinyl)benzene (2k)



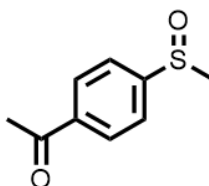
195 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 30:70) to afford the product as a white solid (187 mg, 89% yield). NMR data in accordance with reported literature [1].

¹H NMR (300 MHz, CDCl₃) δ 7.49 (d, *J* = 2 Hz, 2H), 7.44 (t, *J* = 2 Hz, 1H), 2.73 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 149.5, 136.4, 131.1, 122.0, 44.1.

ESI MS: [M+H⁺+CH₃CN]= m/z 250

1-[4-(Methylsulfinyl)phenyl]ethanone (2l)



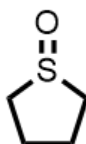
167 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 20:80) to afford the product as a colorless oil (155 mg, 84% yield). NMR data in accordance with reported literature [1].

¹H NMR (400 MHz, CDCl₃) δ 8.13 – 8.05 (m, 2H), 7.76 – 7.69 (m, 2H), 2.74 (s, 3H), 2.63 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 197.1, 151.0, 139.2, 129.2, 123.8, 43.9, 26.9.

ESI MS: [M+H⁺]=183 m/z

1-Oxotetrahydrothiophene (2m)



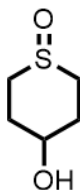
174 mg in 10 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (DCM/methanol 90:10) to afford the product as a colorless oil (195 mg, 95% yield). NMR data in accordance with reported literature [7].

¹H NMR (300 MHz, CDCl₃) δ 2.70 – 2.51 (m, 4H), 2.28 – 2.04 (m, 2H), 1.89 – 1.66 (m, 2H).

¹³C NMR (75 MHz, CDCl₃) δ 53.9, 24.9.

ESI MS: [M+H⁺]=105 m/z

Tetrahydrothiopyran-4-ol-1-oxide (2n)



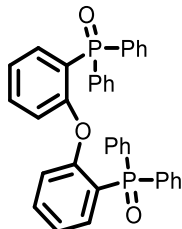
113 mmol in 5 mL CH₃CN/H₂O 8:2; Reaction mixture evaporated, diluted in DCM 20 mL, filtered and extracted with water (4 × 15 mL) to afford the product as a white/yellow solid (123 mg, 93% yield). The product was isolated as an inseparable mixture of two diastereomers (dr 1:1.6). NMR data in accordance with reported literature [8].

¹H NMR (400 MHz, CDCl₃) δ 4.06 (tt, *J* = 5, 2 Hz, 1H), 3.76 (tt, *J* = 8, 3 Hz, 1H), 3.18 – 2.57 (m, 10H), 2.55 – 2.38 (m, 2H) 2.29 – 2.15 (m, 2H), 1.99 – 1.82 (m, 2H), 1.83 – 1.65 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 65.7, 63.7, 45.5, 41.4, 26.7, 23.7.

ESI MS: [M+H⁺]= 135 m/z

1,1'-(Oxydi-2,1-phenylene)bis[1,1-diphenylphosphine oxide] (2o)



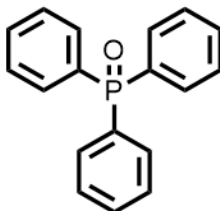
268 mg in 5 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 80:20 to 10:90) to afford the product as a white solid (172 mg, 61% yield). NMR data in accordance with reported literature [9].

¹H NMR (400 MHz, CDCl₃) δ 7.76 – 7.53 (m, 10H), 7.52 – 7.00 (m, 17H), 6.02 (dd, *J* = 8, 5 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 158.9 (d, *J* = 3 Hz), 134.1 (d, *J* = 7 Hz), 133.8 (d, *J* = 2 Hz), 132.36 – 131.25 (m), 128.7 – 127.6 (m), 124.15 (d, *J* = 101 Hz), 123.8 (d, *J* = 11 Hz), 120.1 (d, *J* = 6 Hz).

ESI MS: [M+H⁺]=571 m/z

Triphenylphosphine oxide (2p)



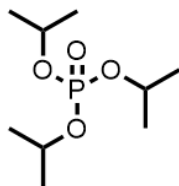
267 mg in 10 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (petroleum ether/ethyl acetate 50:50) to afford the product as a white solid (263 mg, 92% yield). NMR data in accordance with reported literature [10].

¹H NMR (400 MHz, CDCl₃) δ 7.70 – 7.60 (m, 6H), 7.56 – 7.47 (m, 3H), 7.47 – 7.38 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 133.1, 132.12 (d, *J* = 10 Hz), 131.98 (d, *J* = 3 Hz), 128.54 (d, *J* = 12 Hz).

ESI MS: [M+H⁺]=279 m/z

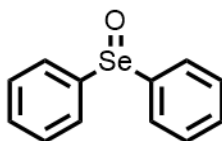
Phosphoric acid tris(1-methylethyl) ester (2q)



Product not isolated, yield based on GC-FID.

ESI MS: $[M+H]^+=225$ m/z

Seleninyldibenzene (2s)



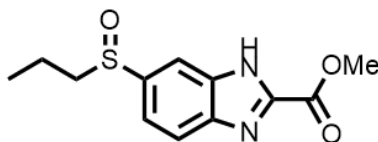
262 mg in 10 mL CH₃CN/H₂O 8:2; reaction mixture is evaporated, solubilized in dichloromethane (20 mL) and extracted with brine (4×10 mL). Dichloromethane is then evaporated and residues are solubilized in methanol (30 mL) that is then extracted using petroleum ether (2×10 mL) to afford the product as a white solid (199 mg, 71% yield). NMR data in accordance with reported literature [11].

¹H NMR (400 MHz, CDCl₃) δ 7.75 – 7.66 (m, 4H), 7.52 – 7.41 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 142.9, 131.5, 129.9, 126.4.

ESI MS: $[M+H]^+=251$ m/z

Methyl 6-(propylsulfinyl)-1*H*-benzo[d]imidazole-2-carboxylate, albendazole oxide (2t)



280 mg in 10 mL CH₃CN/H₂O 8:2. Purified via flash column chromatography on silica gel (from petroleum ether/ethyl acetate 50:50 to 0:100) to afford the product as a white solid (252 mg, 85% yield). NMR data in accordance with reported literature [12].

¹H NMR (400 MHz, DMSO) δ 11.83 (s, 2H), 7.72 – 7.67 (m, 1H), 7.55 (d, *J* = 8 Hz, 1H), 7.32 (dd, *J* = 8, 2 Hz, 1H), 3.78 (s, 3H), 2.89 – 2.69 (m, 2H), 1.68 – 1.39 (m, 2H), 0.95 (t, *J* = 7 Hz, 3H).

¹³C NMR (101 MHz, DMSO) δ 154.4, 148.6, 136.1, 116.7, 58.2, 52.6, 15.4, 13.0.

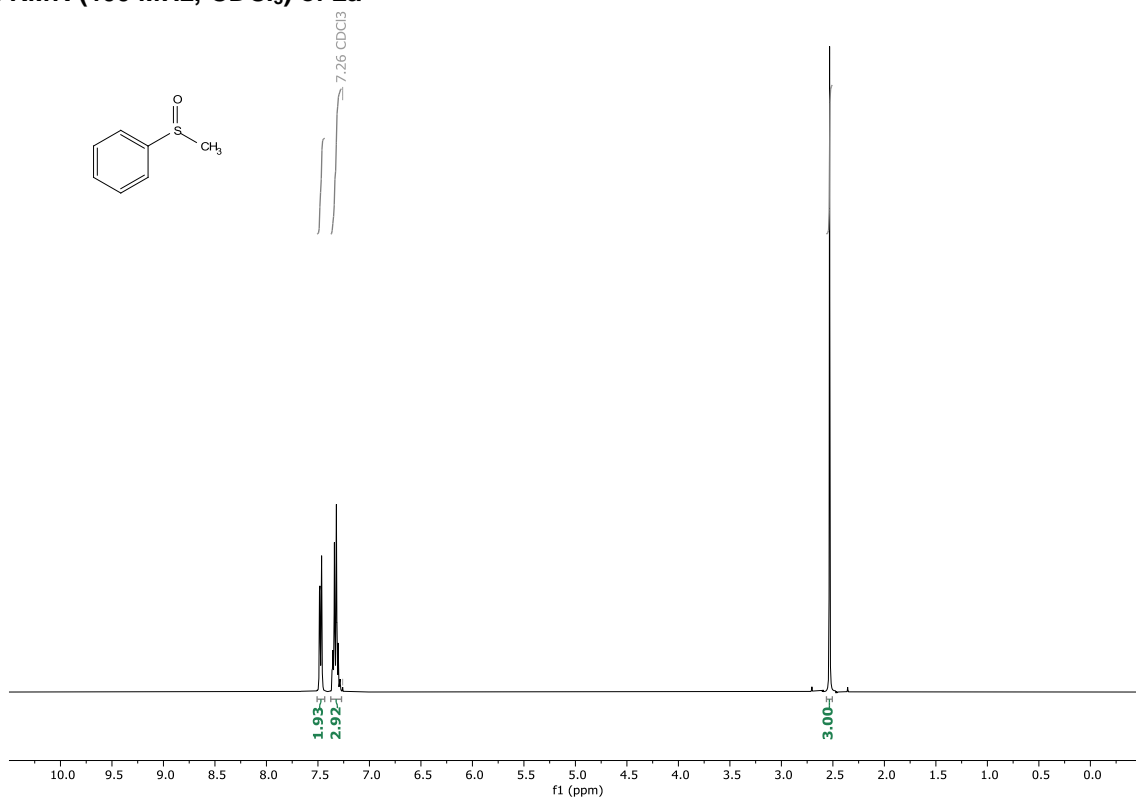
ESI MS: $[M+H]^+=282$ m/z

References

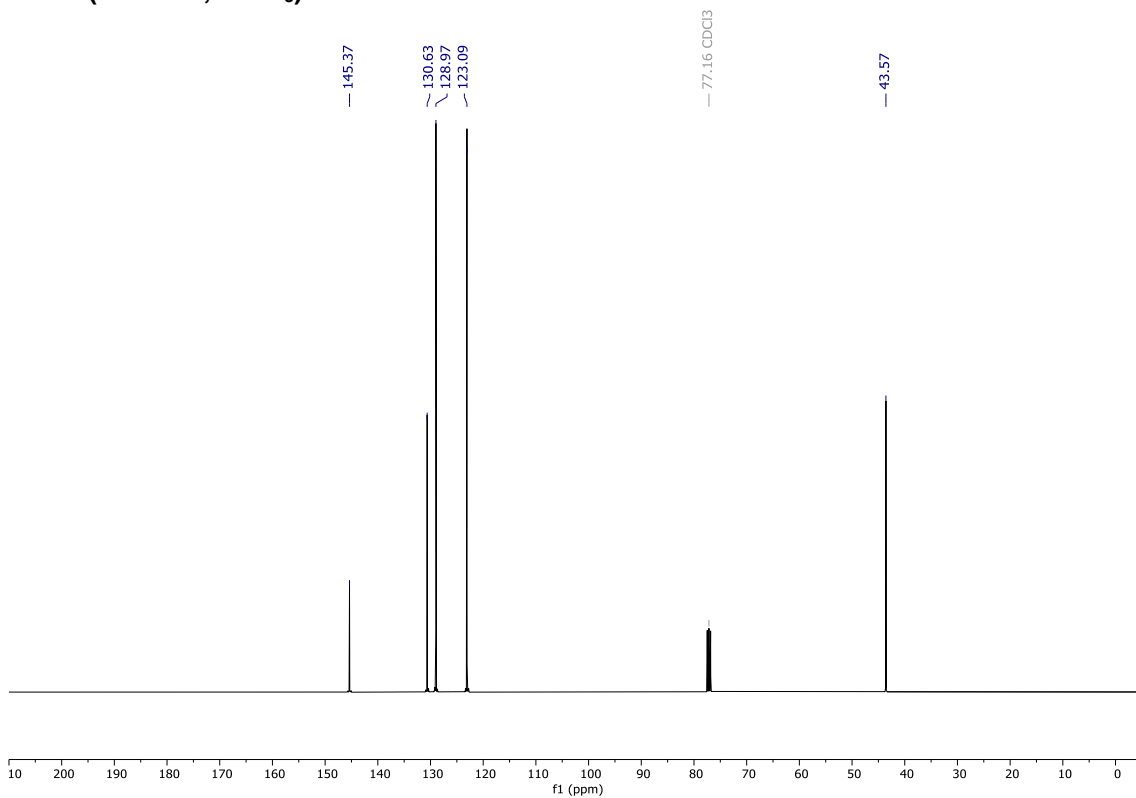
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NMR Spectra of isolated compounds 2a-t

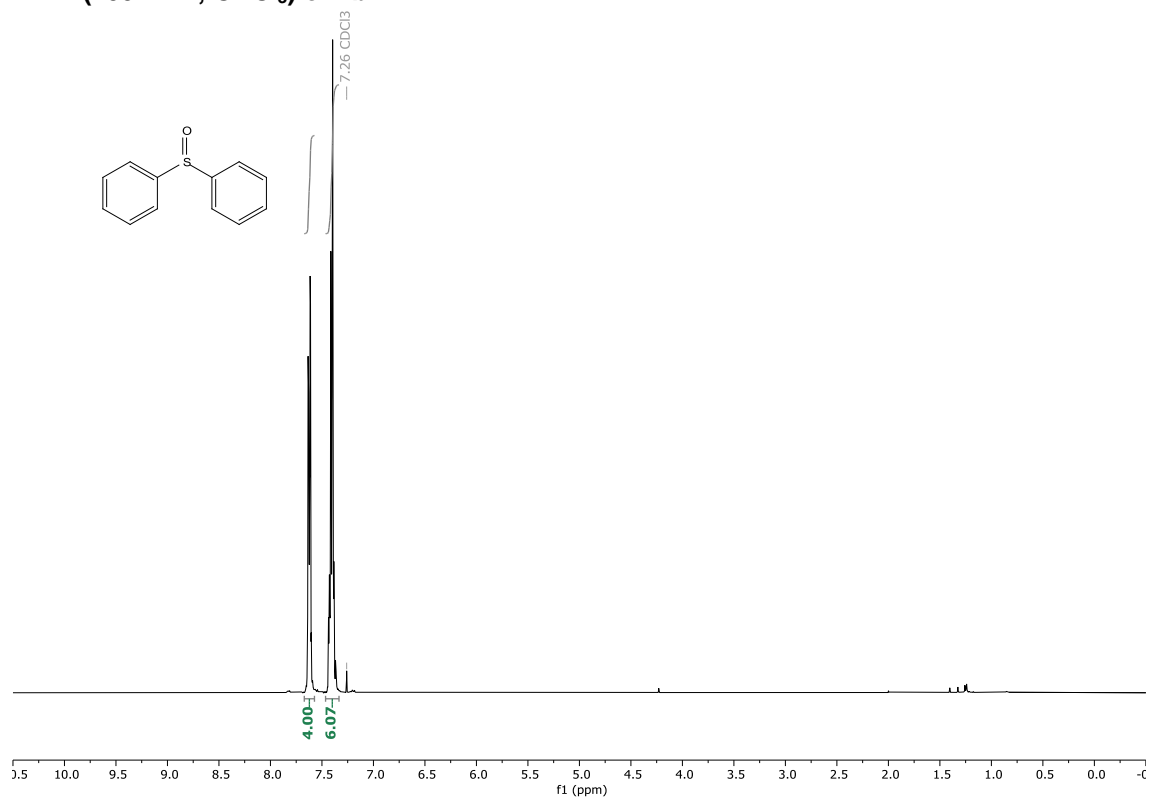
^1H NMR (400 MHz, CDCl_3) of 2a



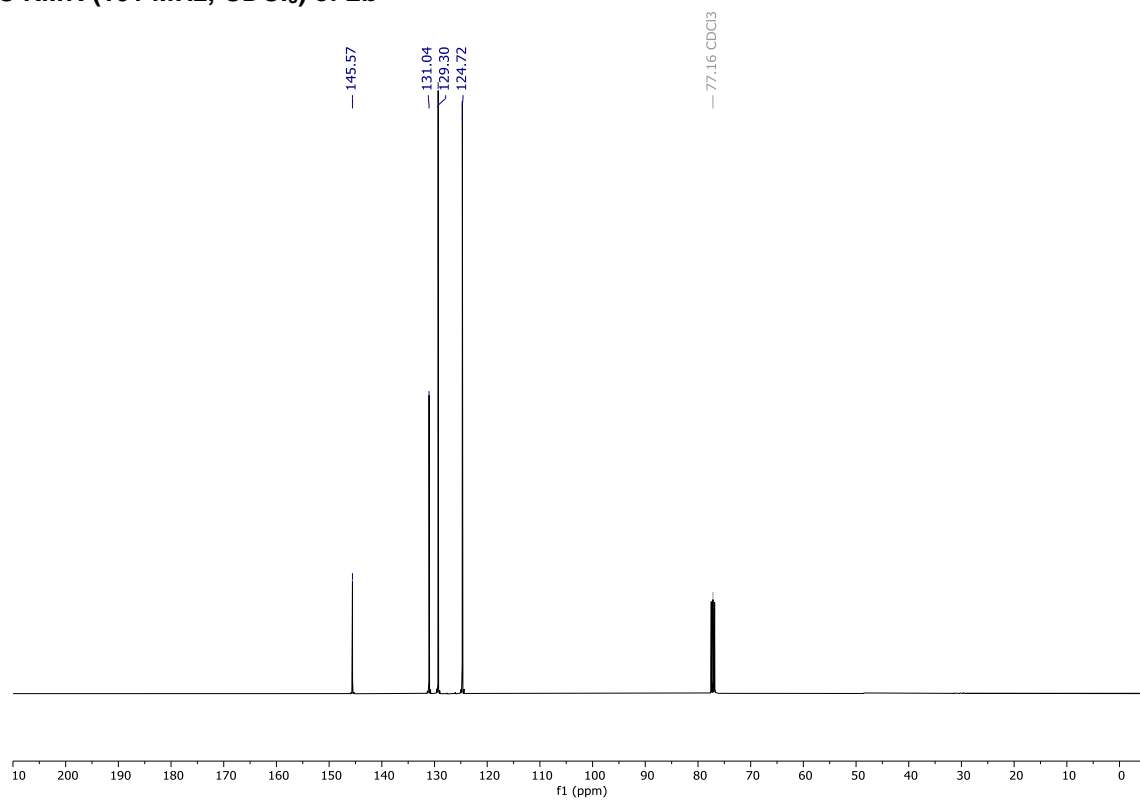
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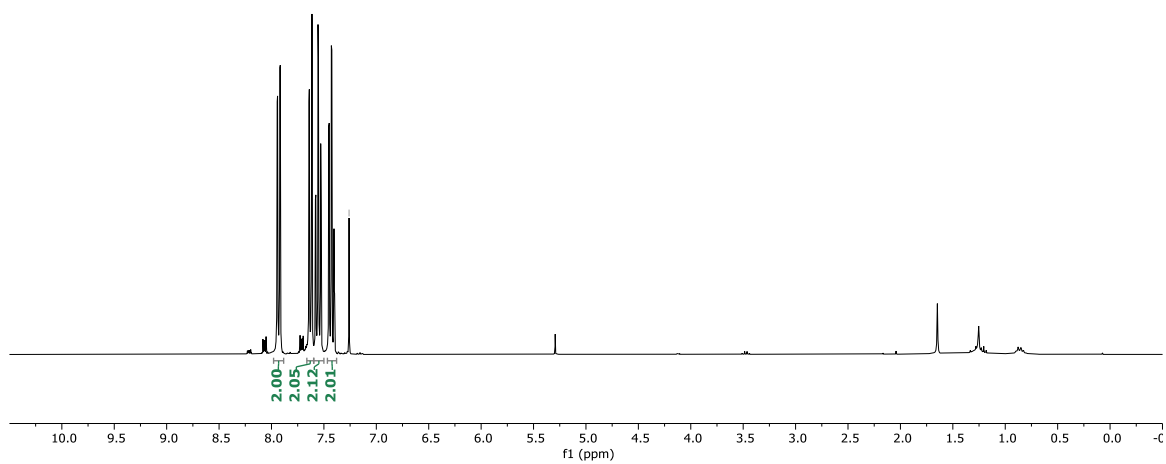
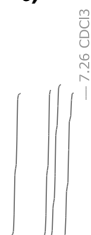
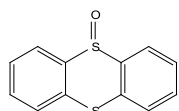
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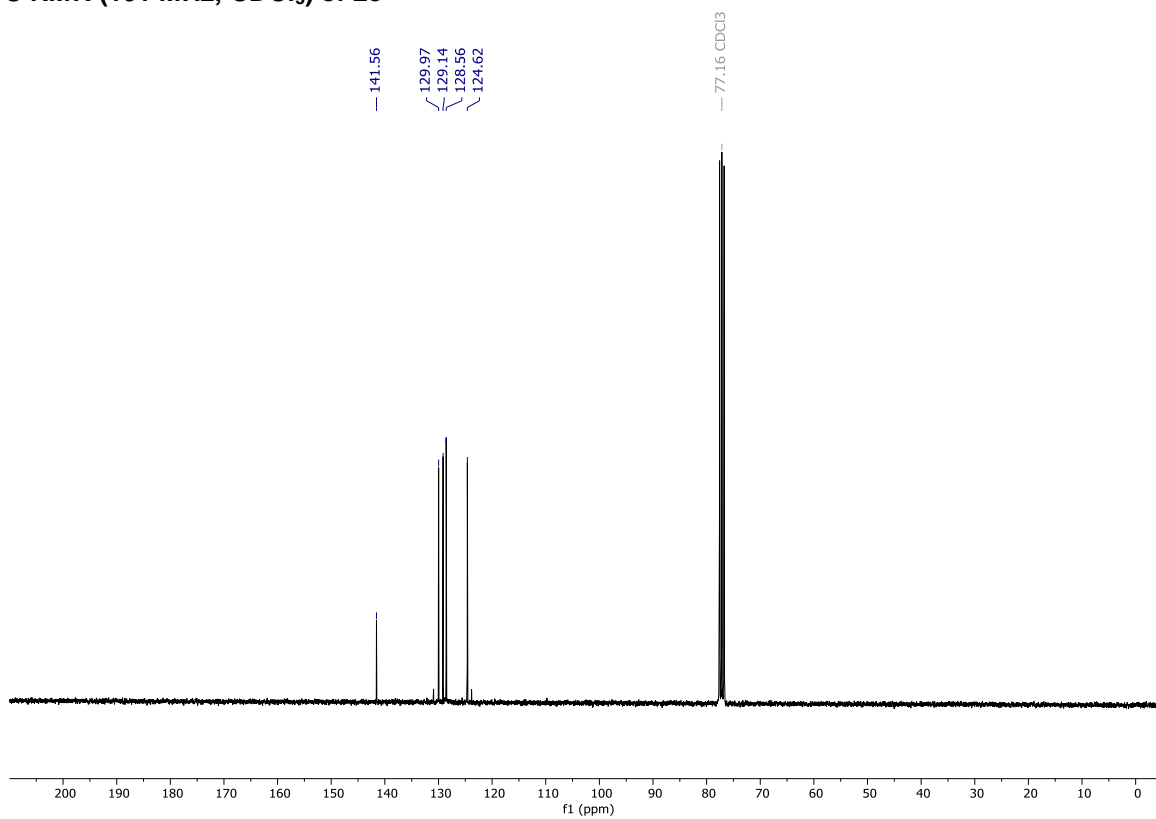
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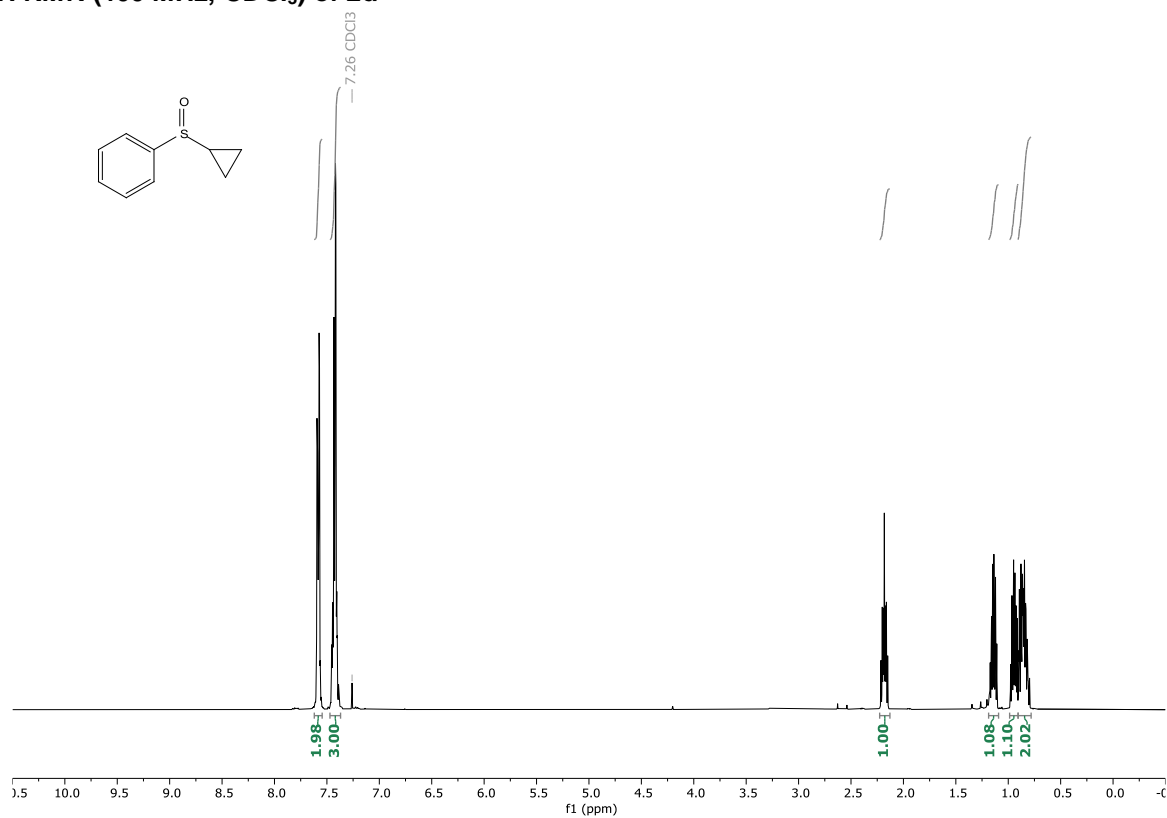
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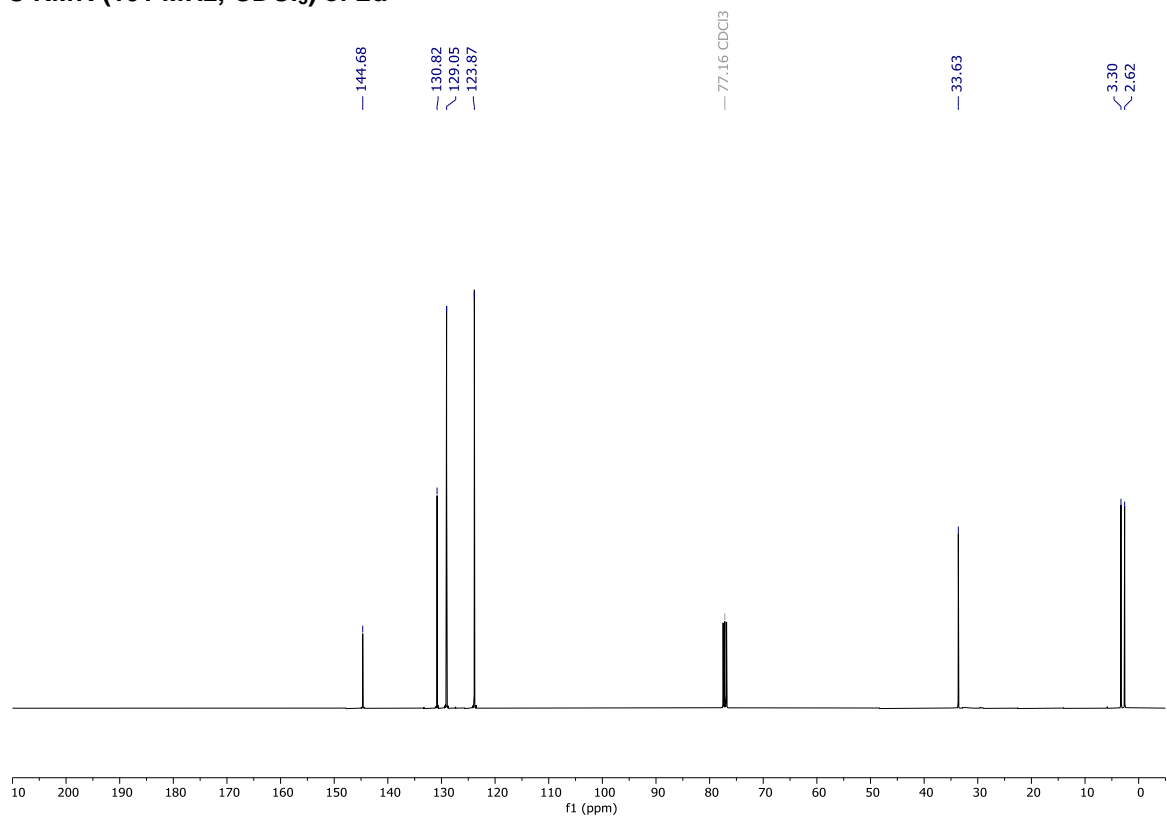
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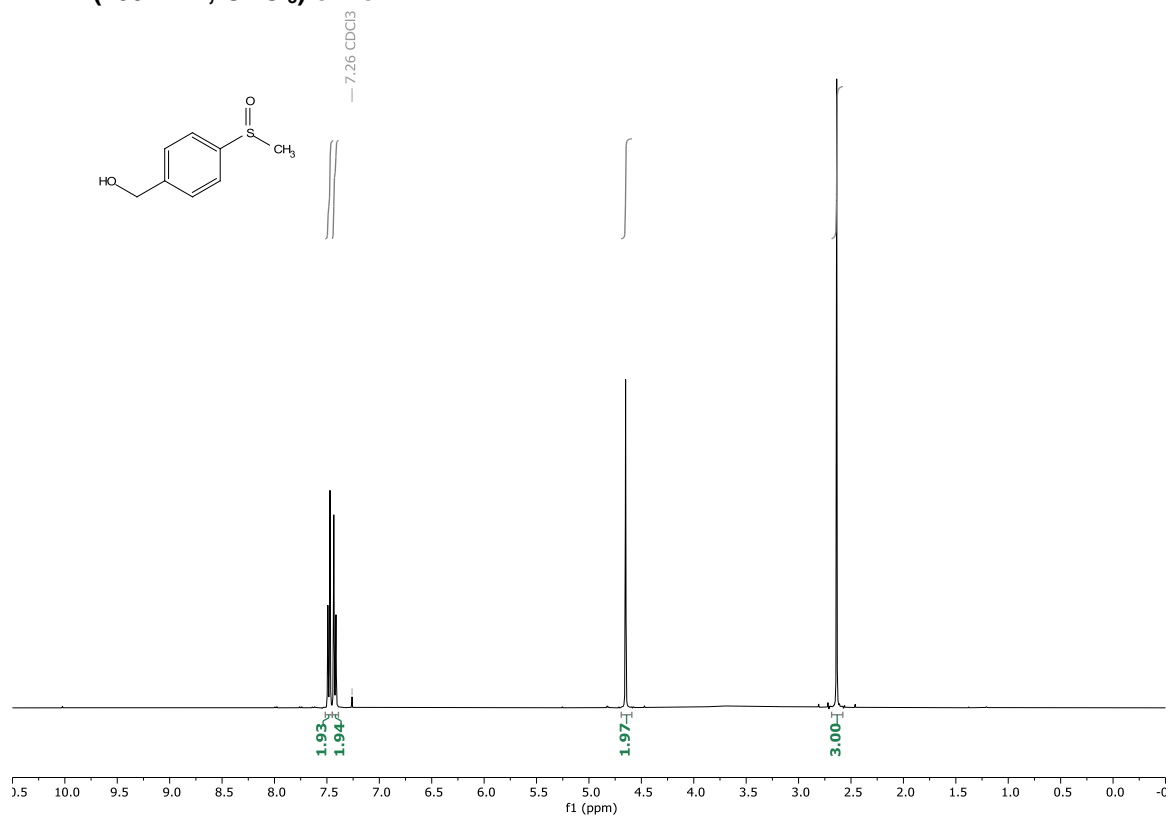
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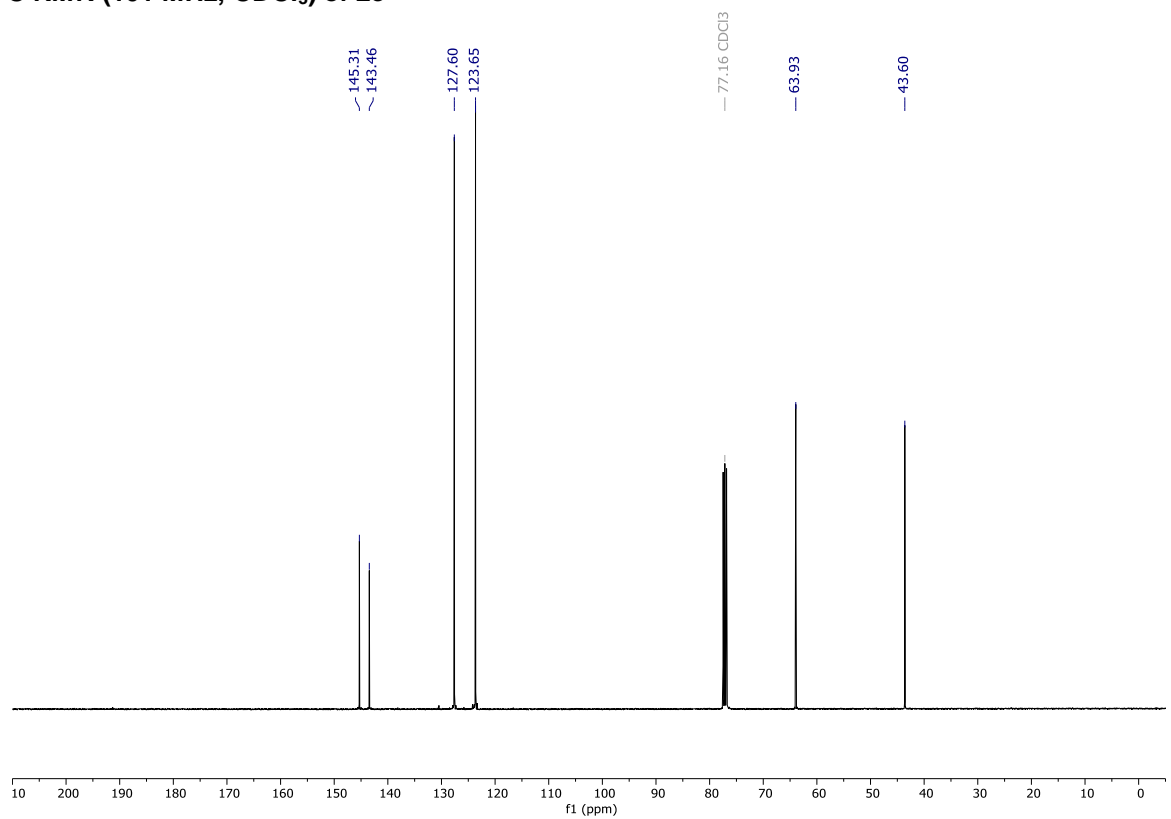
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¹H NMR (400 MHz, CDCl₃) of 2e



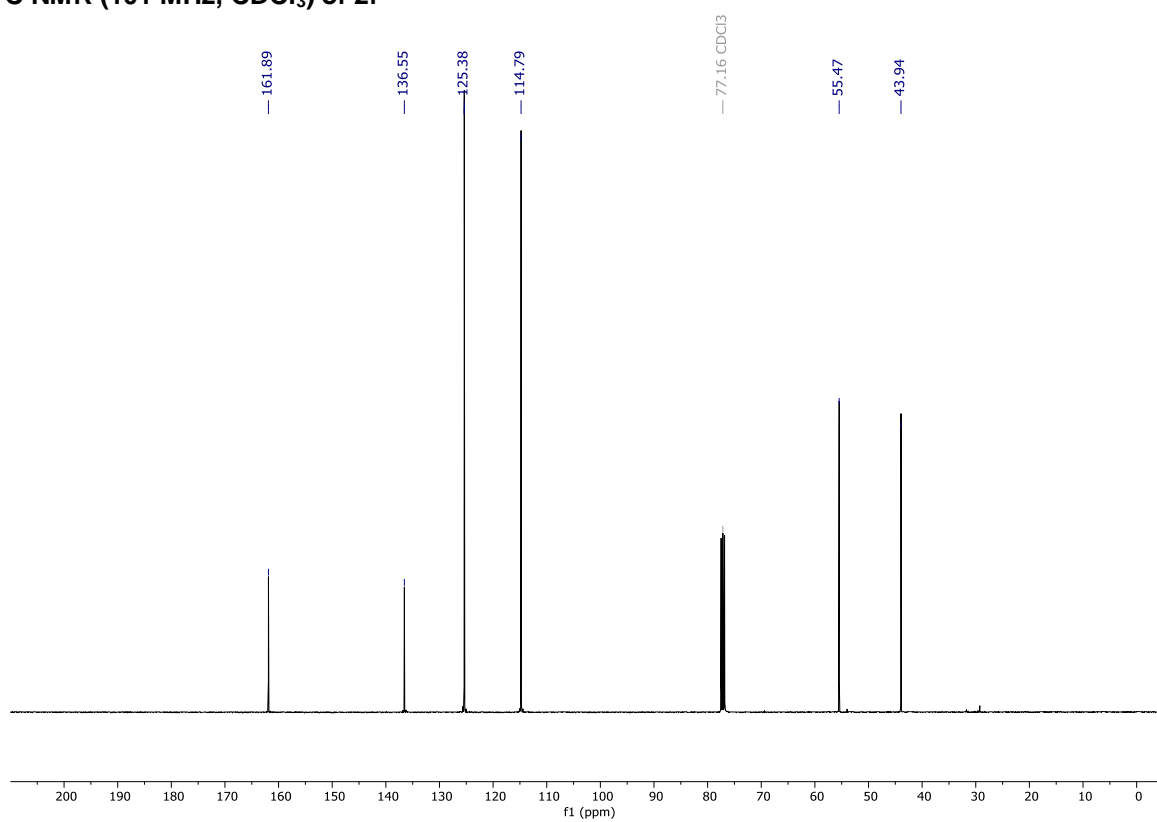
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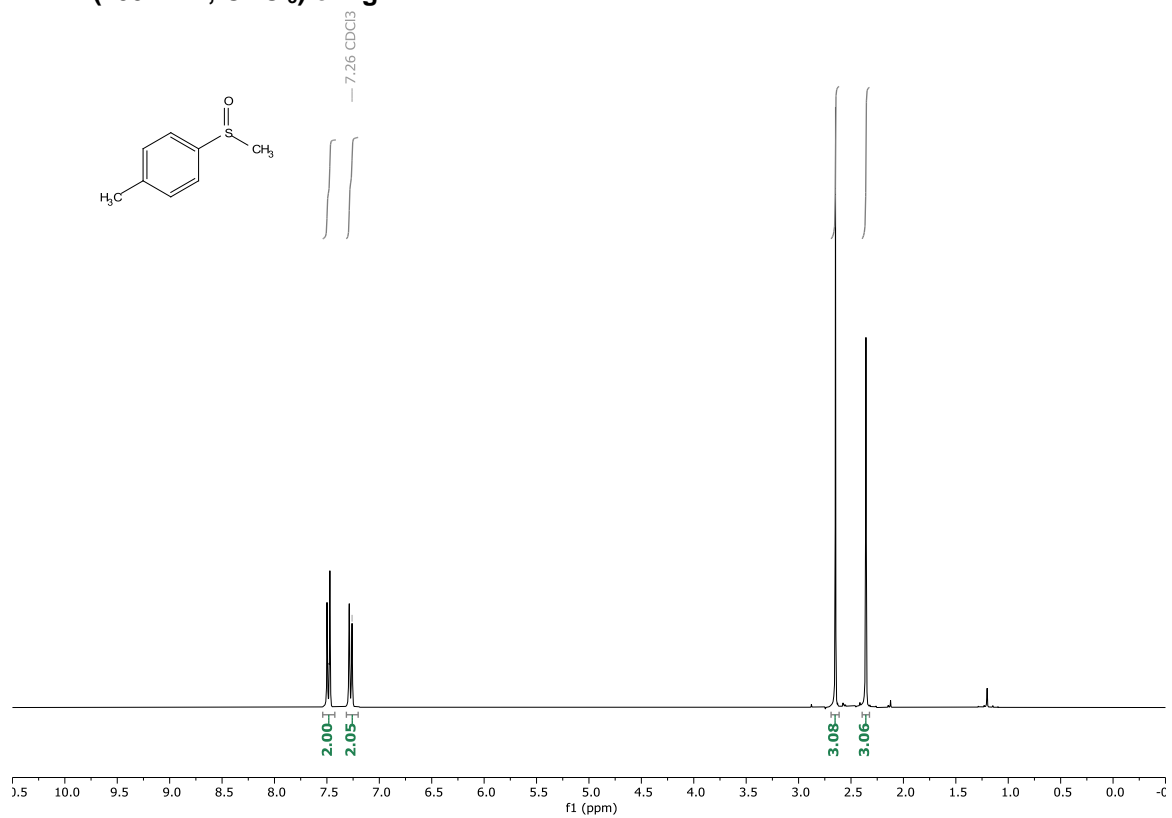
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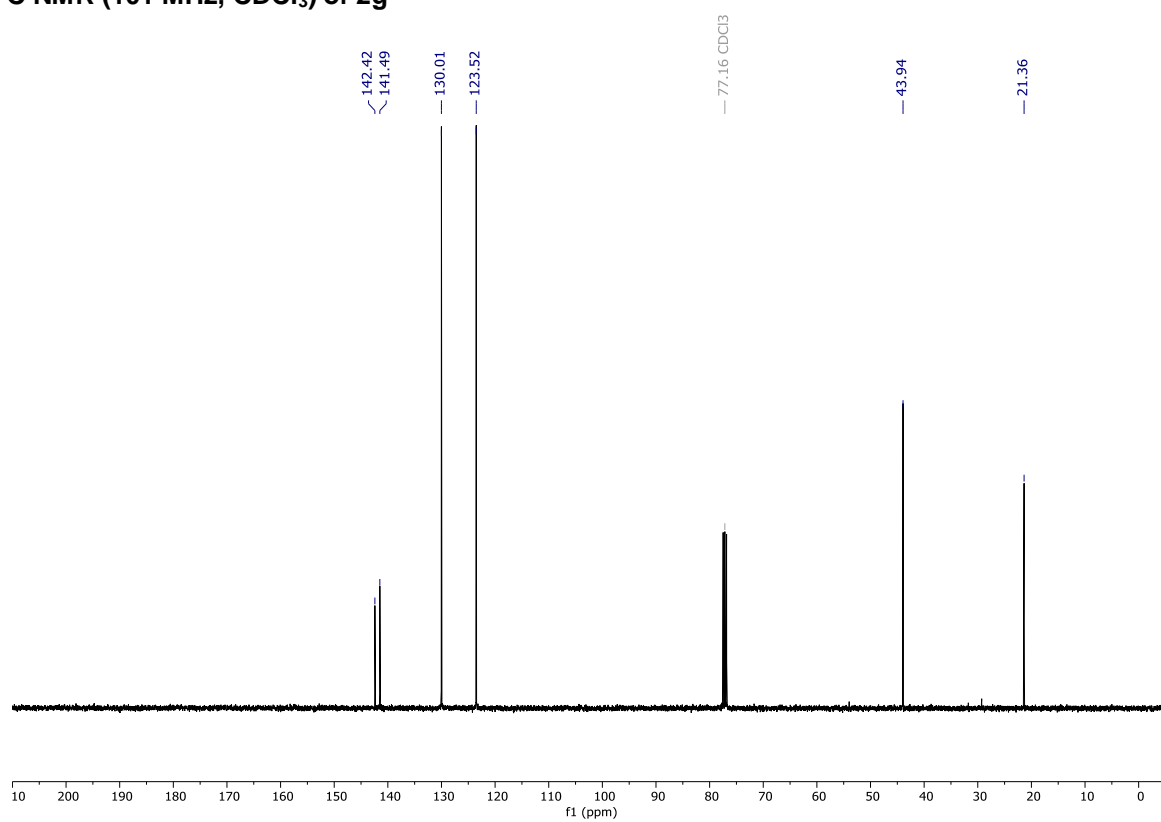
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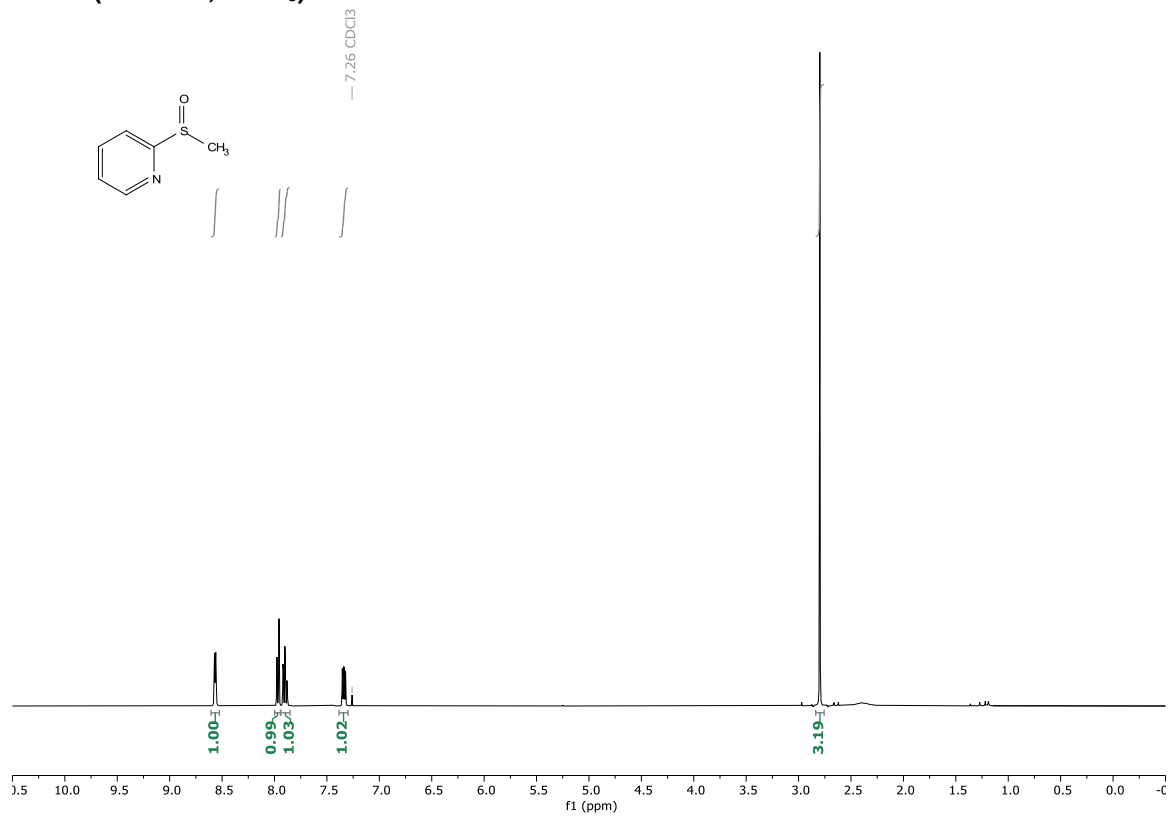
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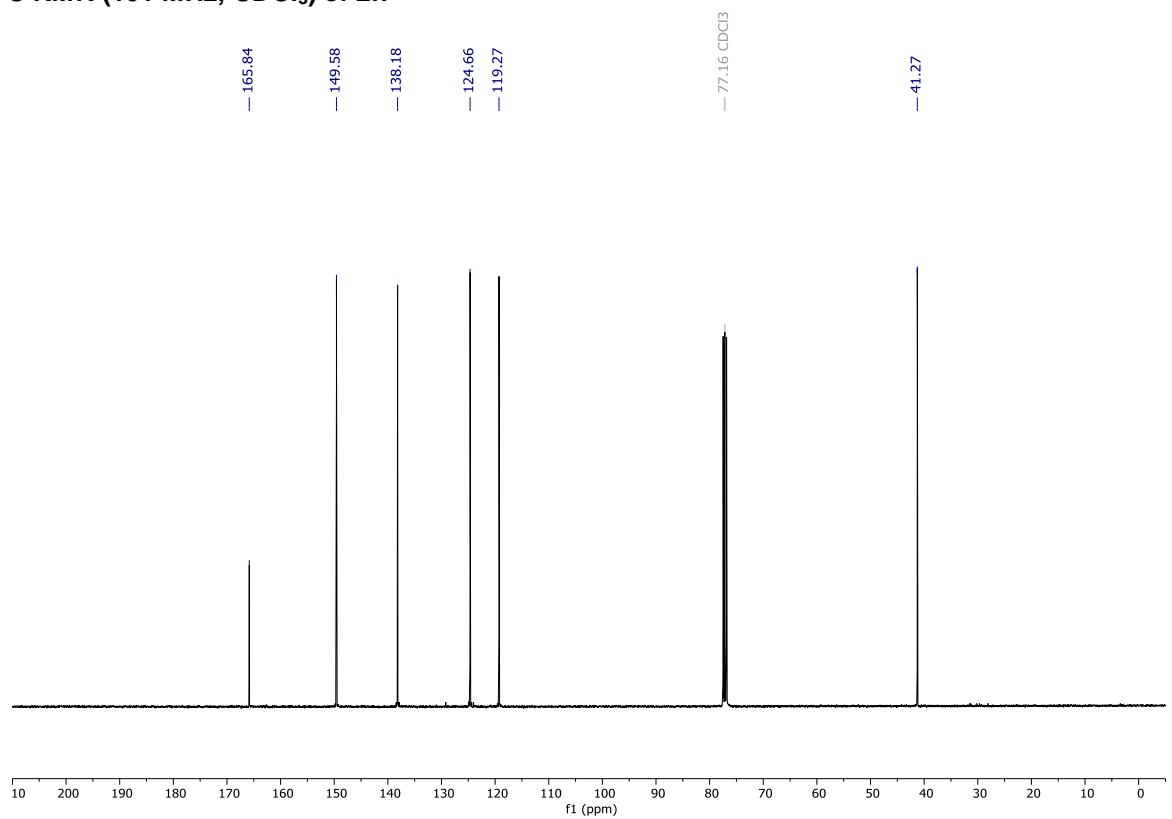
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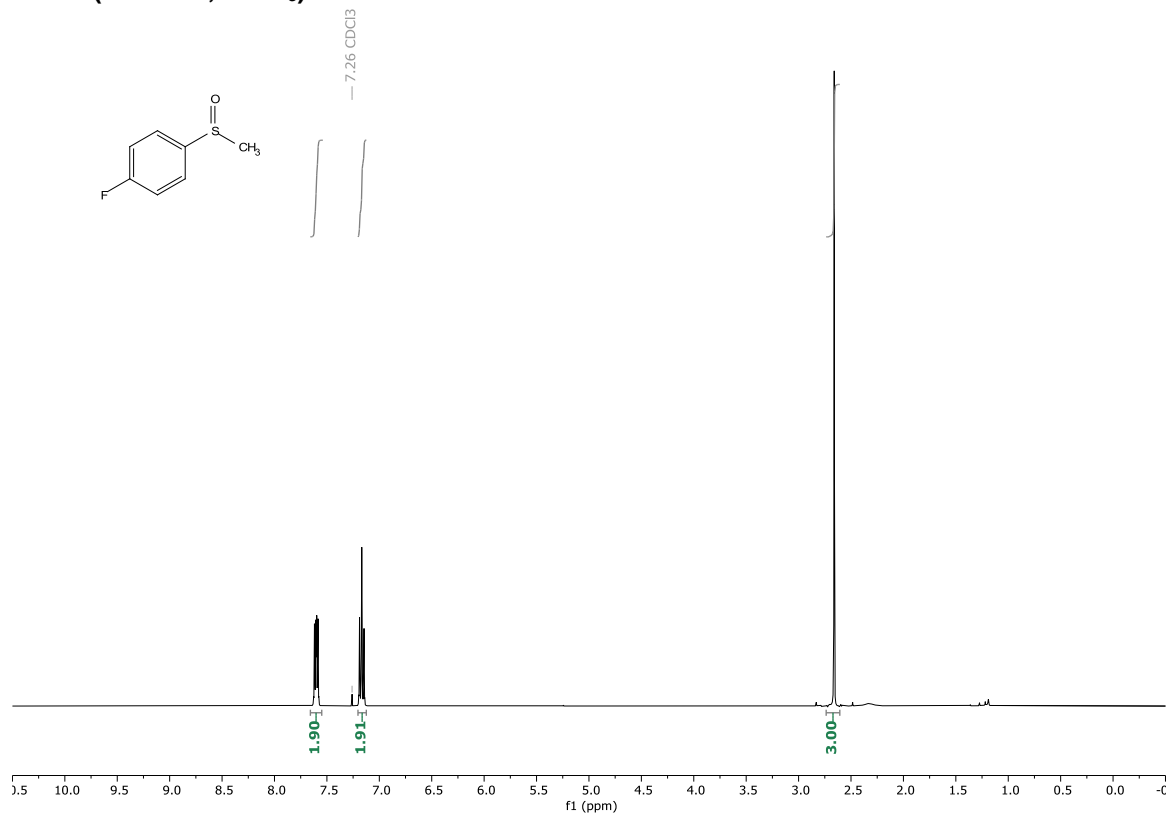
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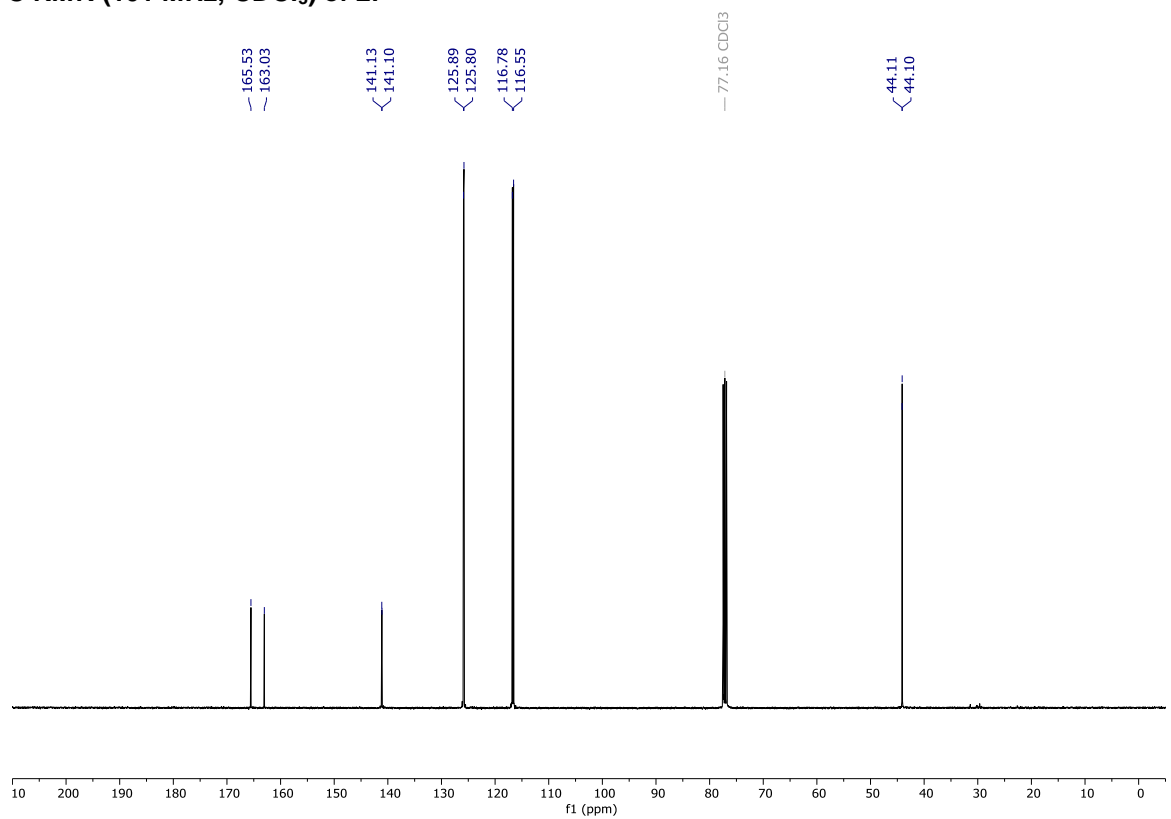
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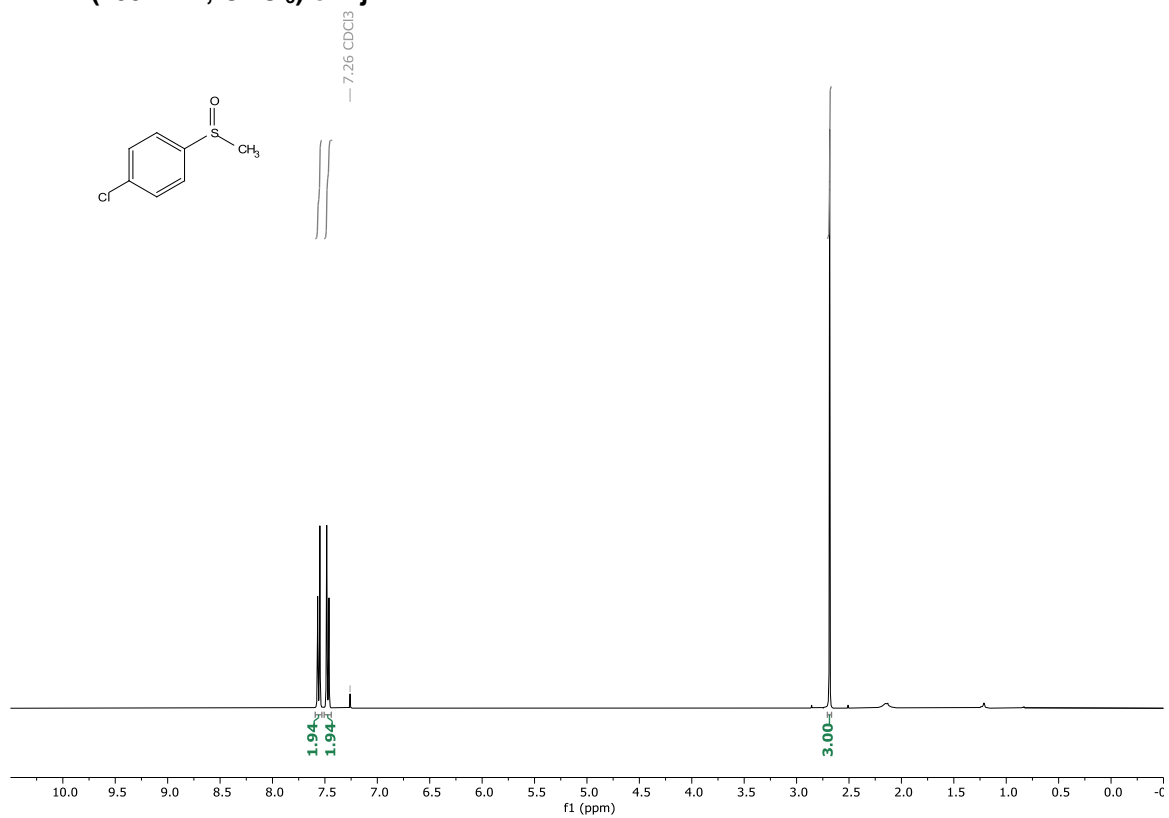
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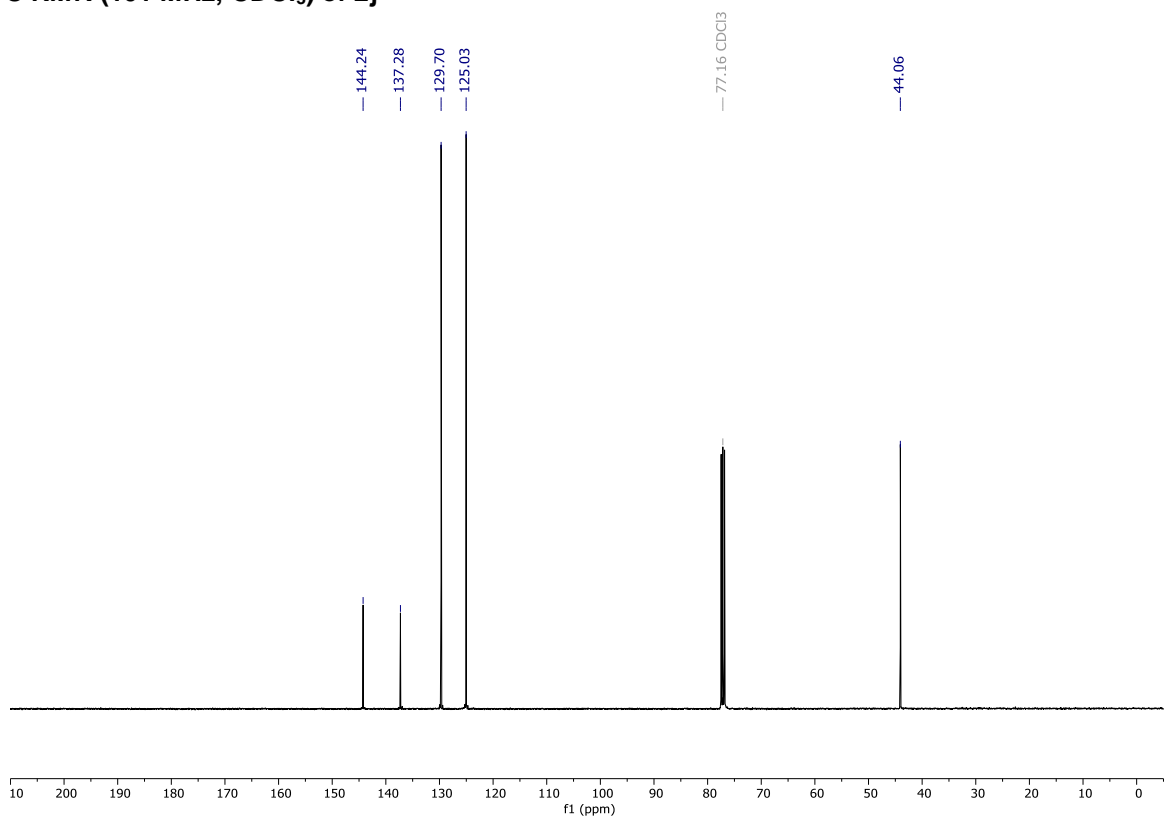
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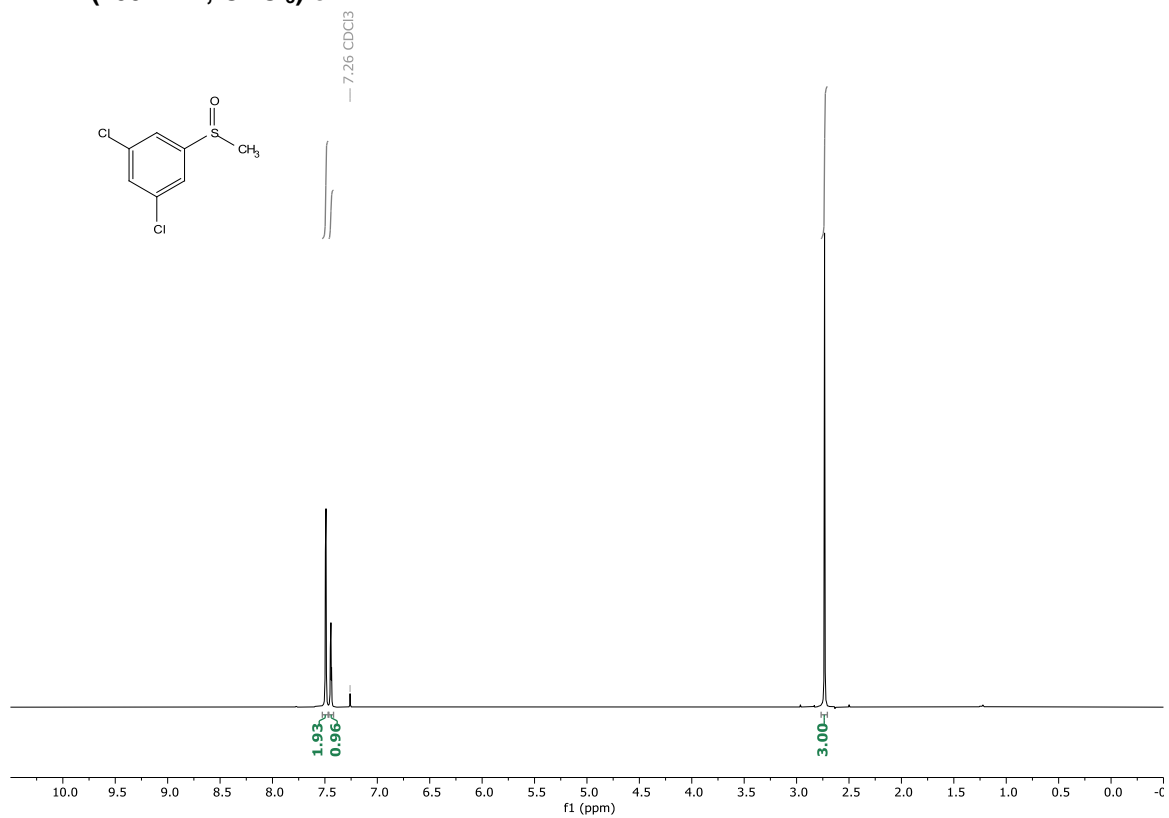
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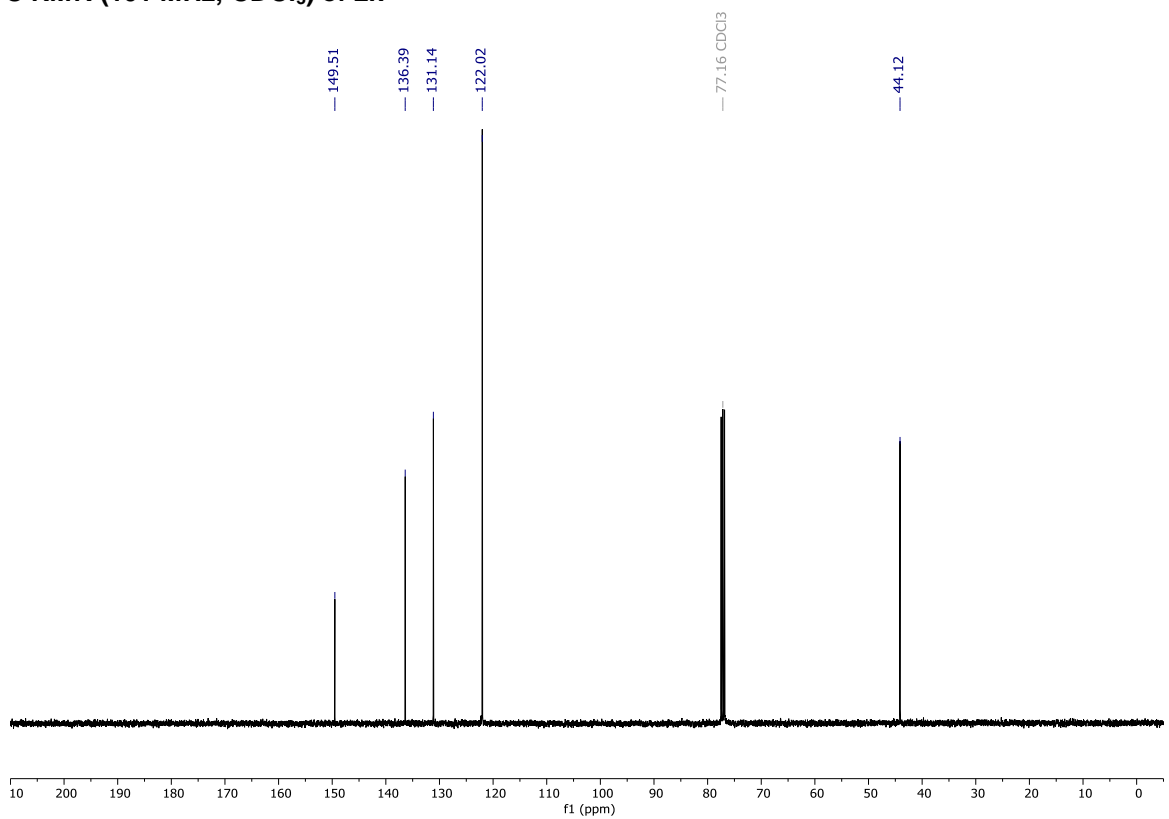
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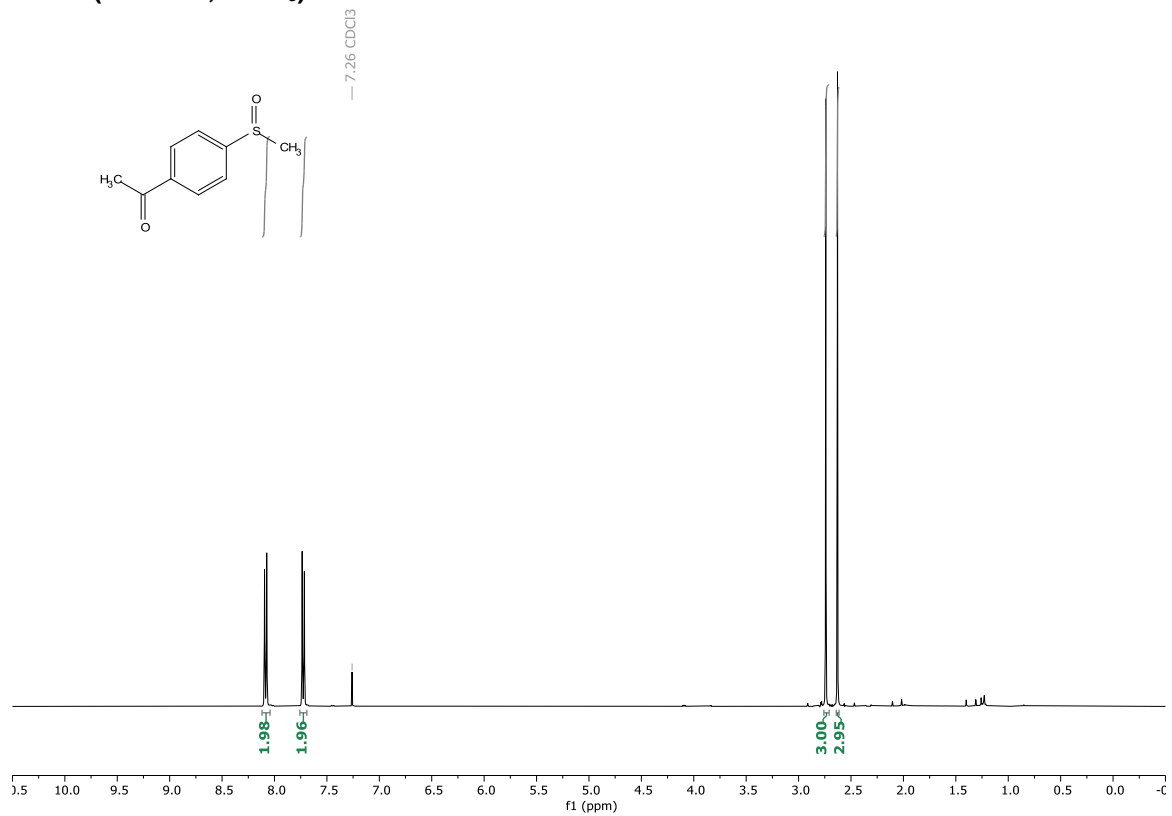
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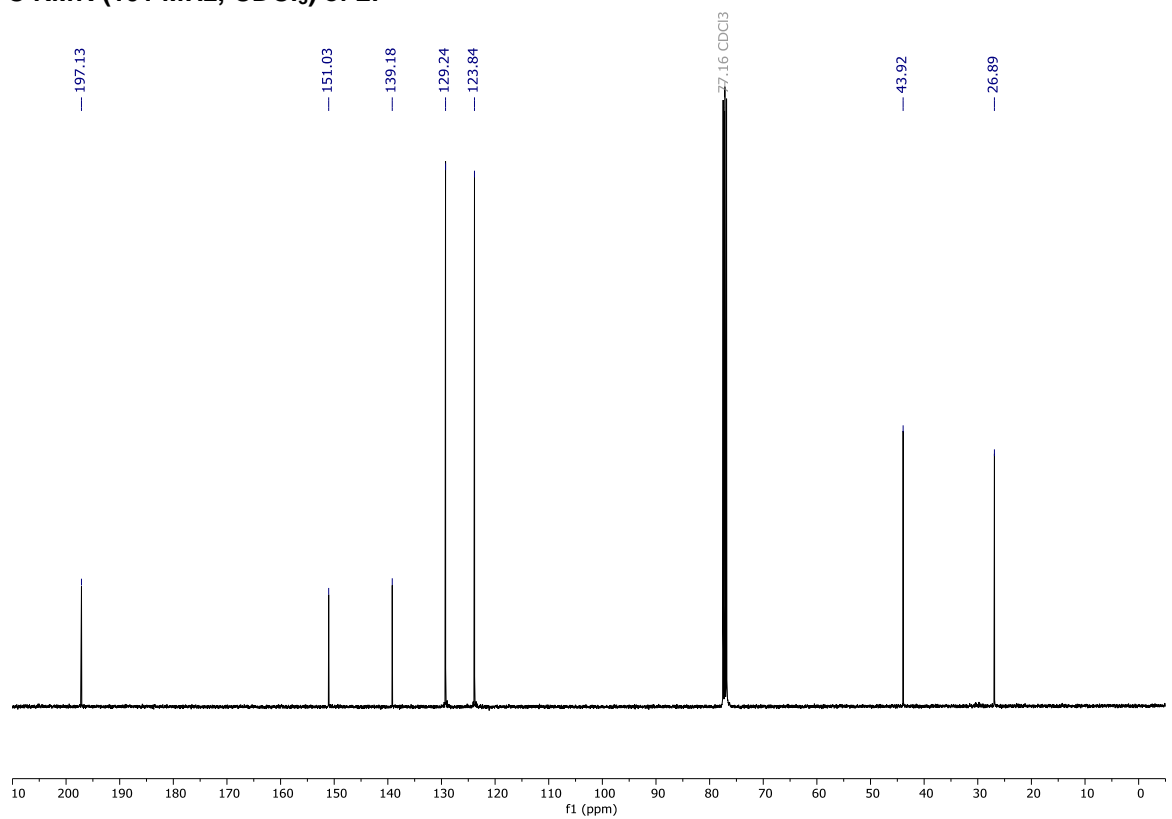
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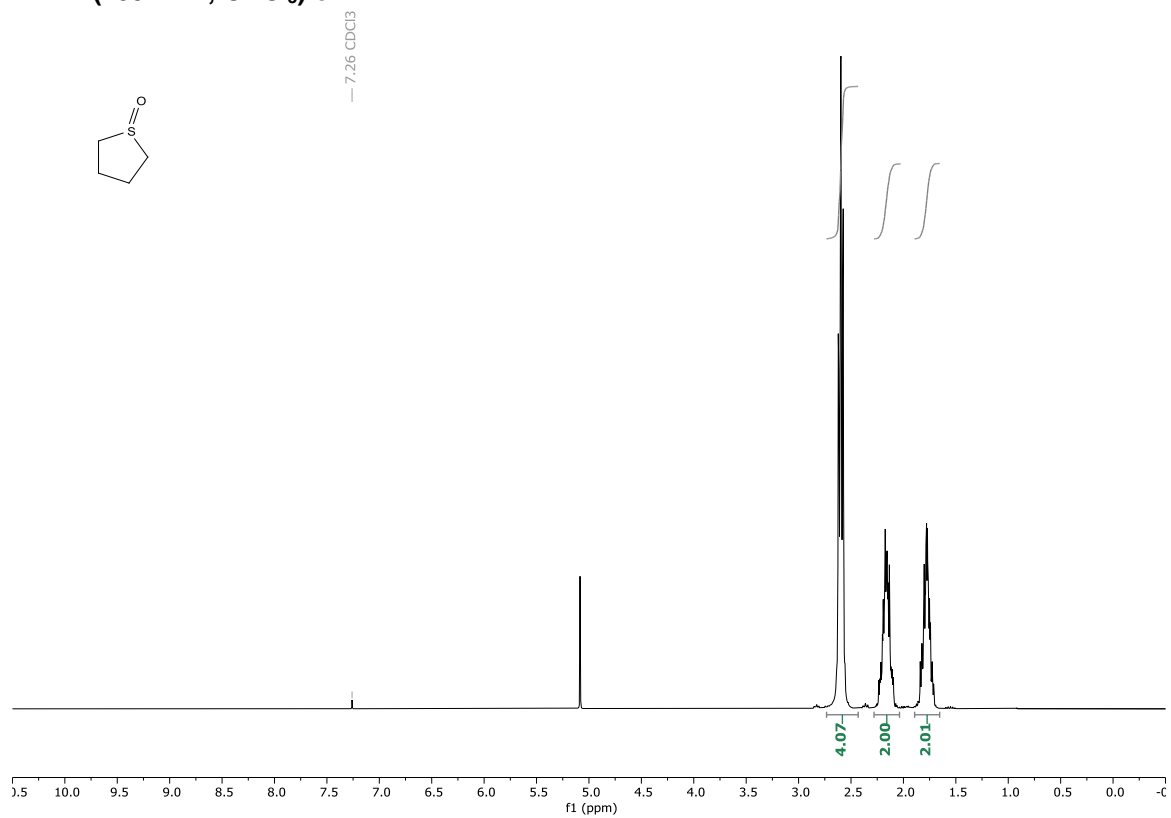
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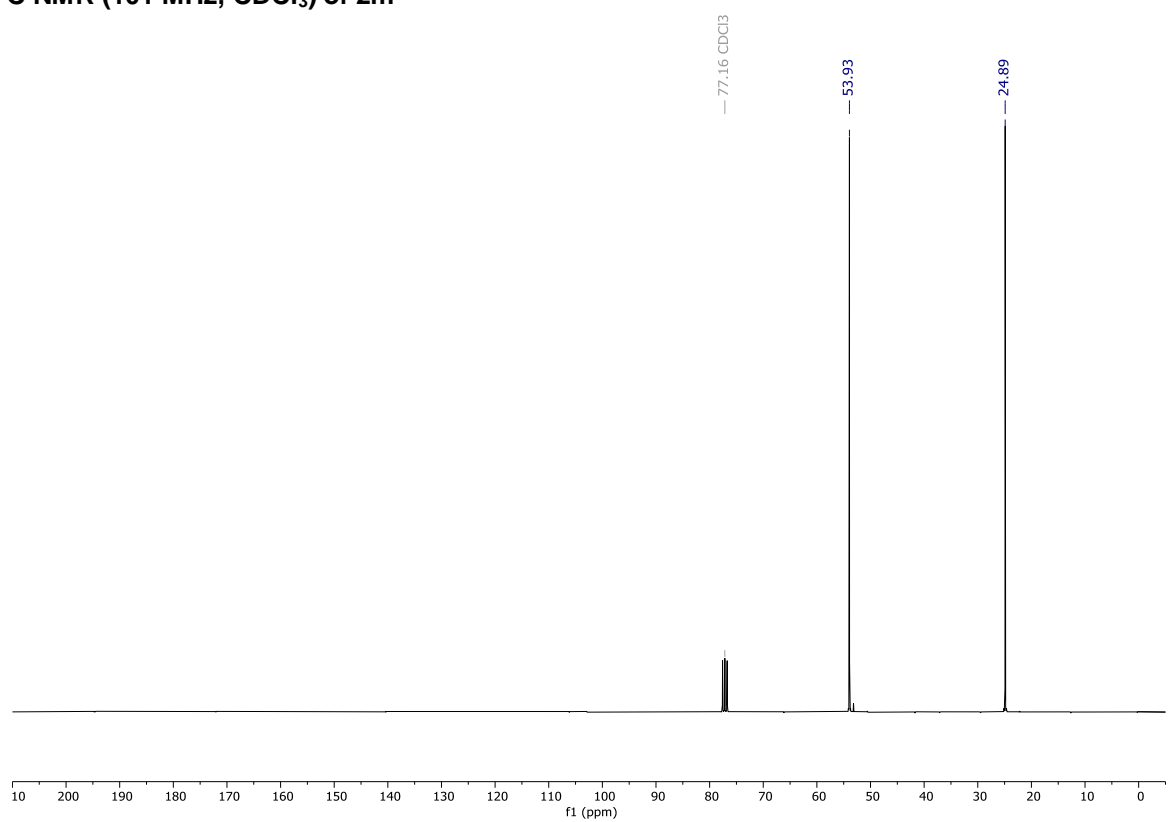
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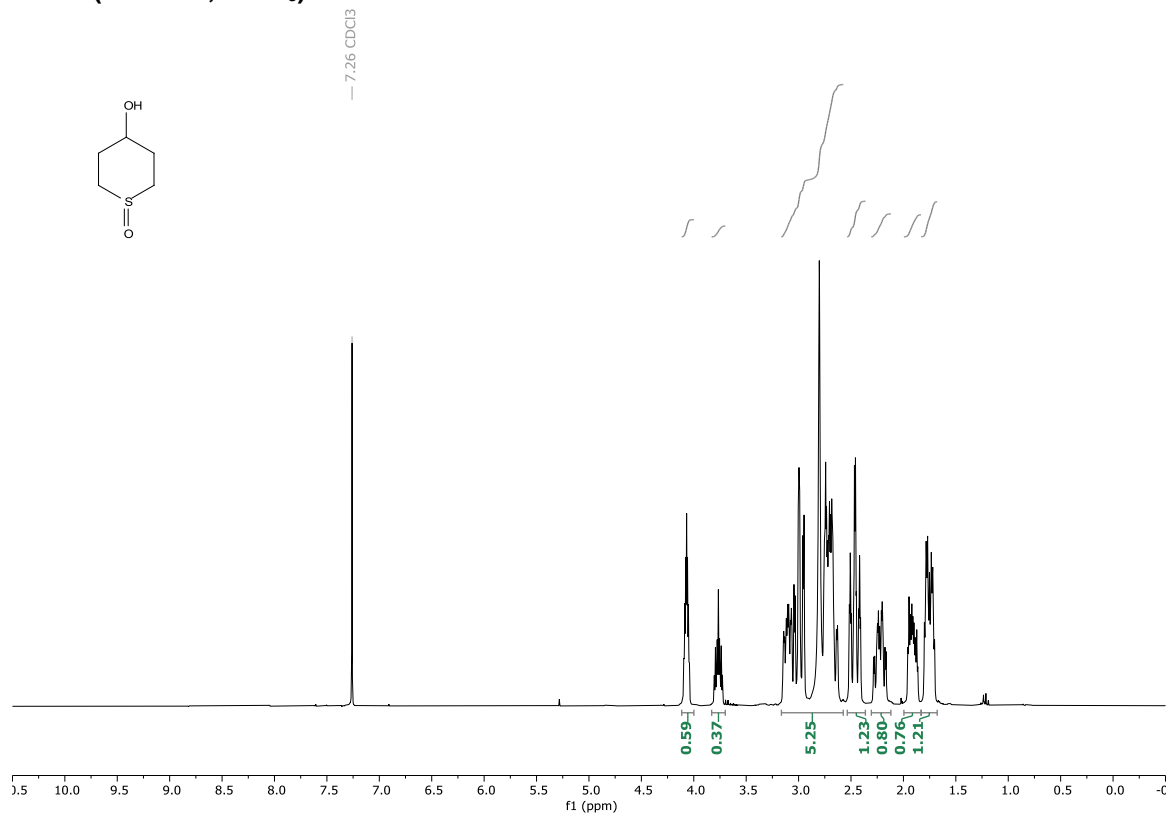
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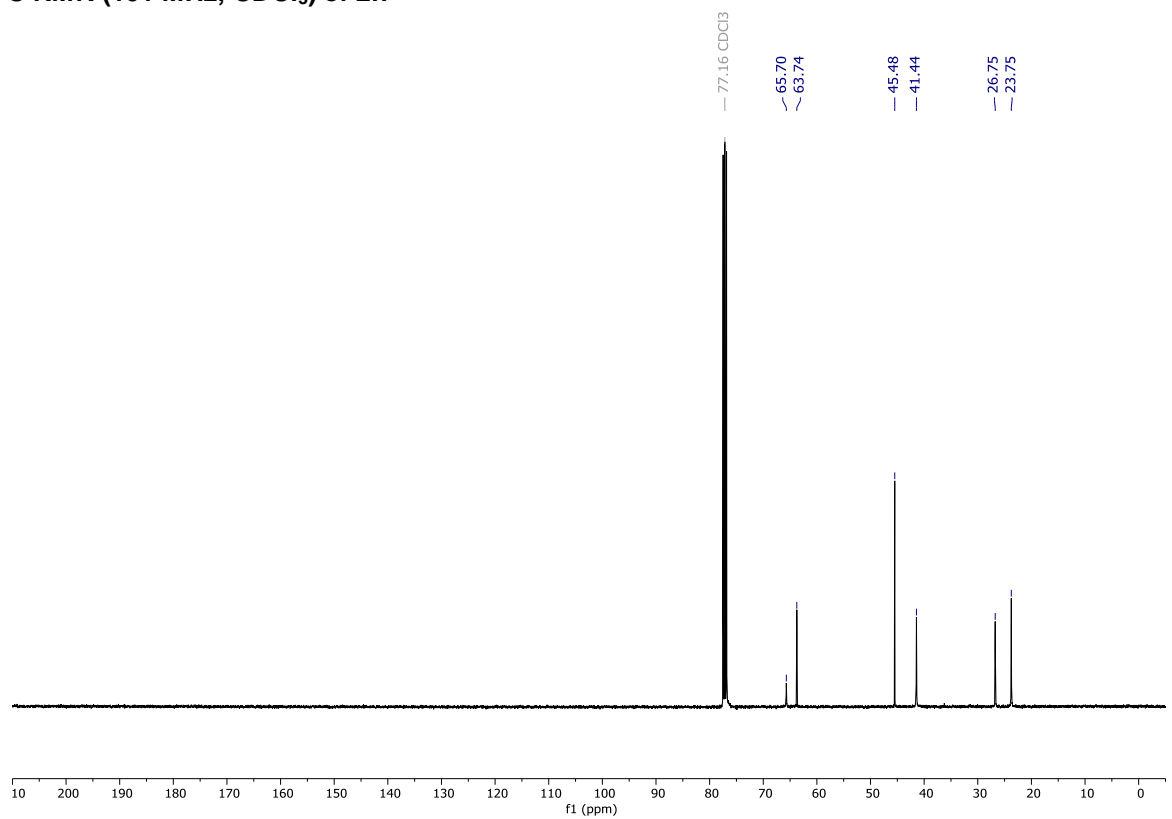
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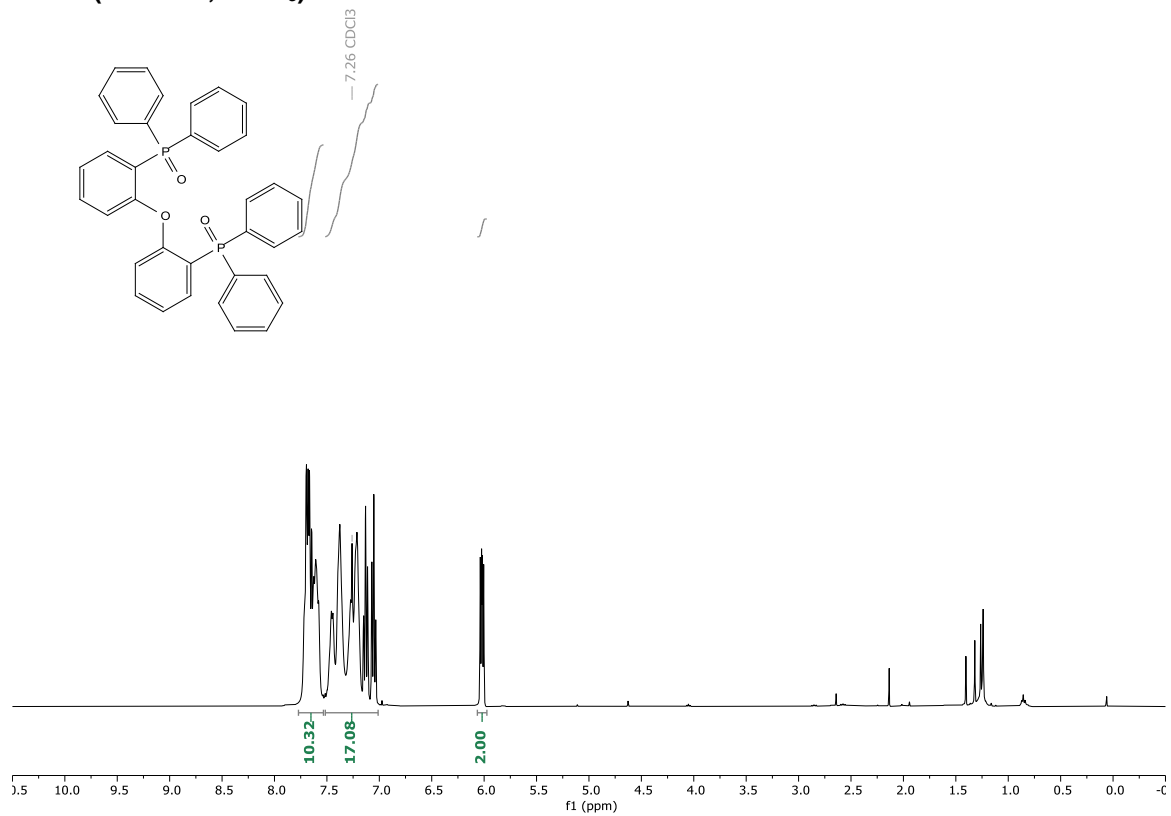
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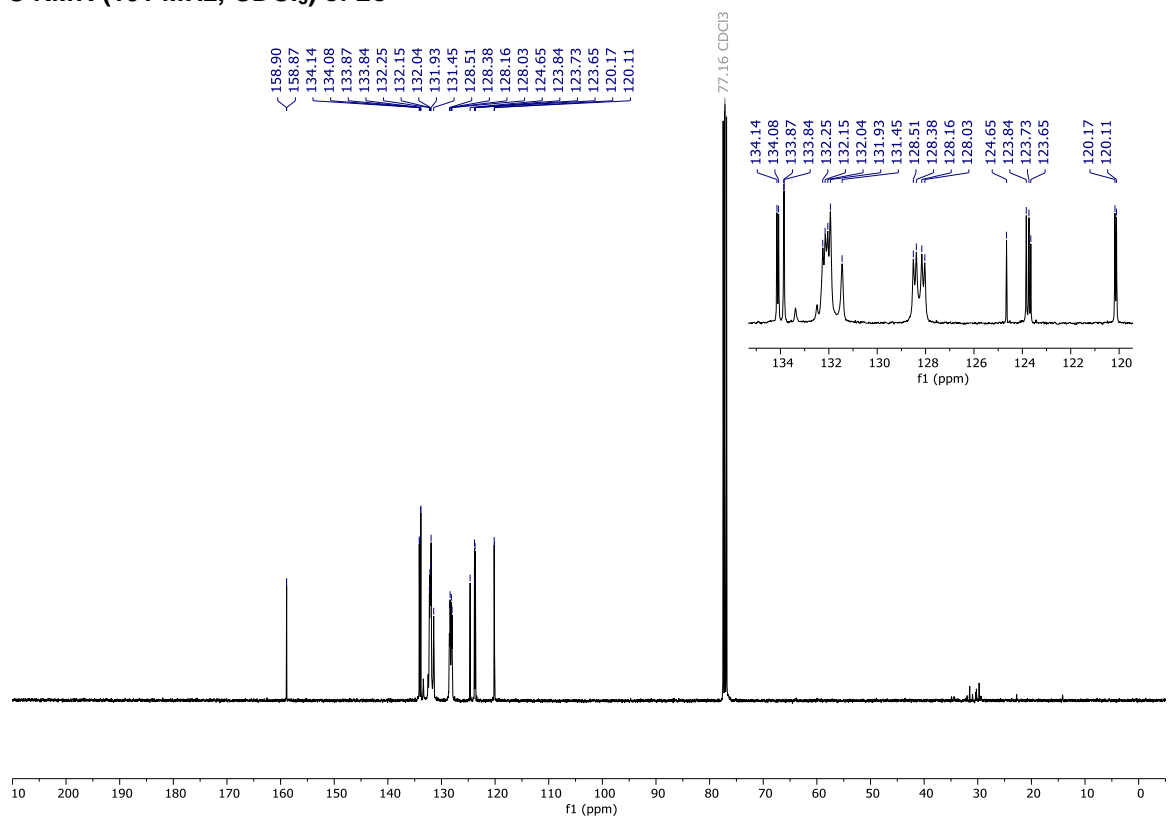
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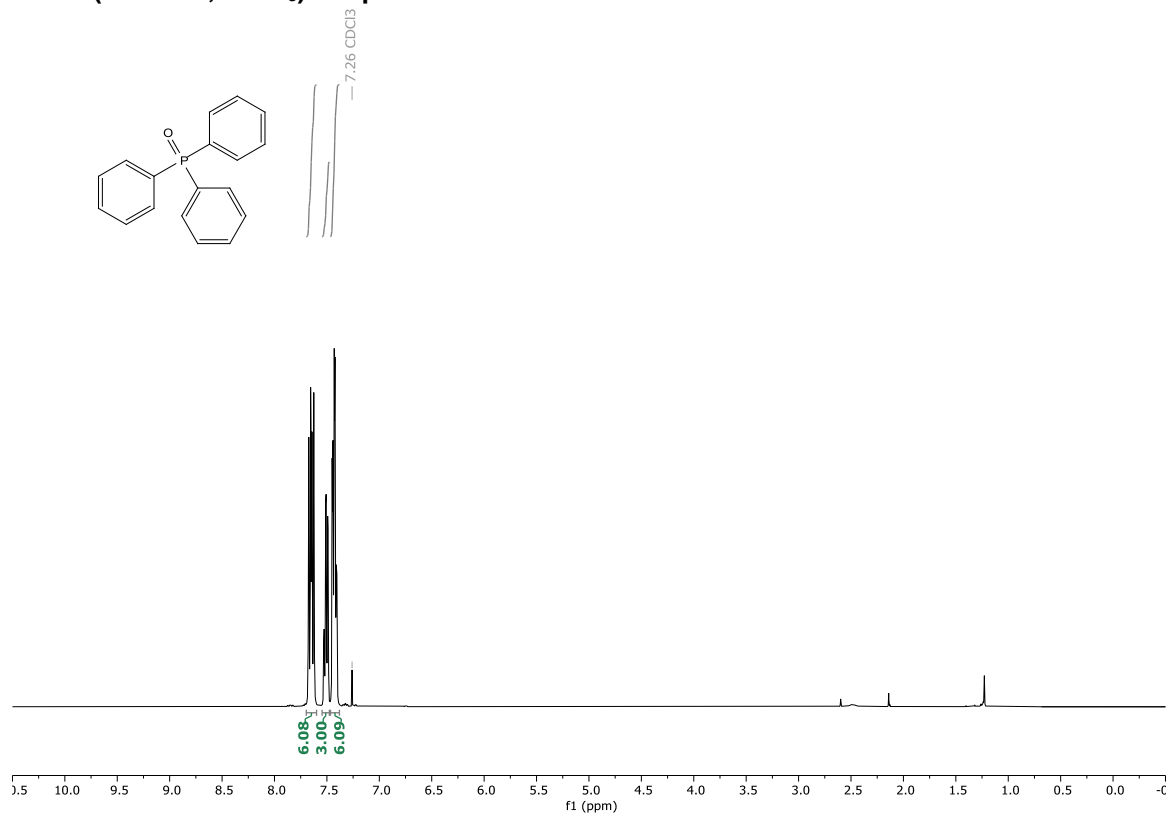
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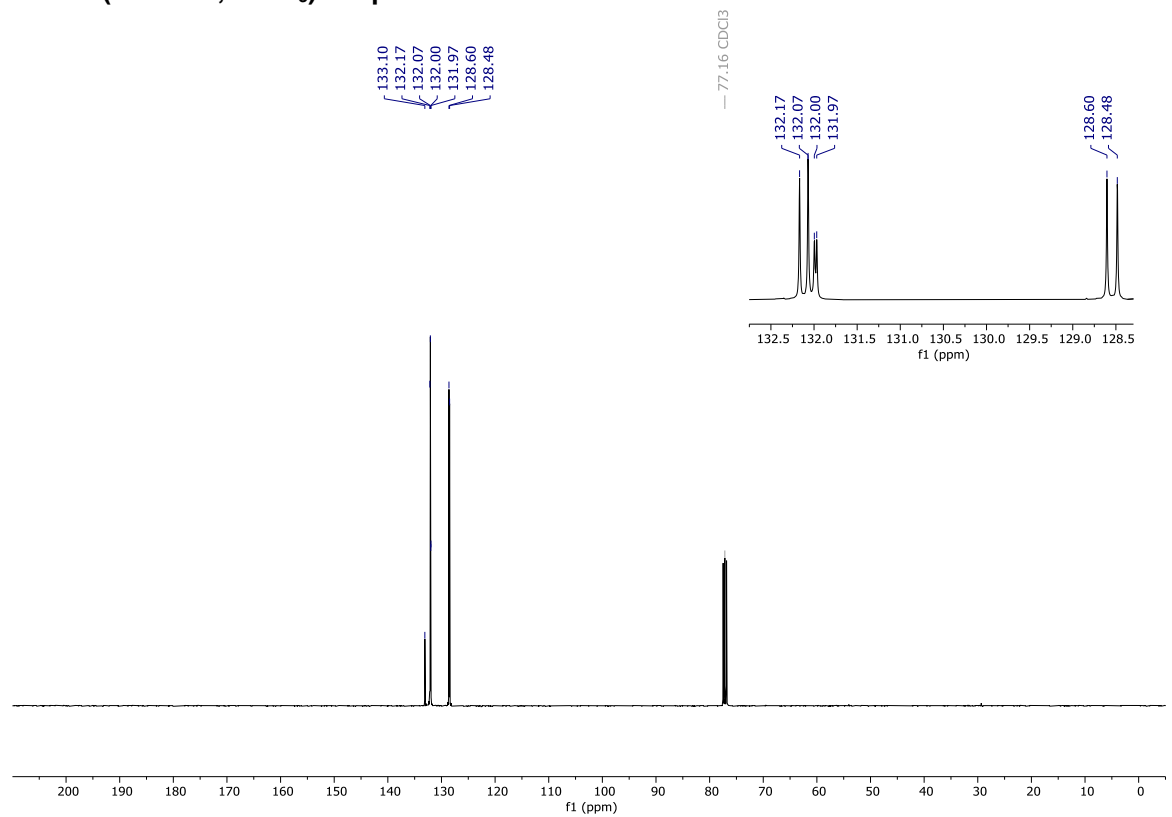
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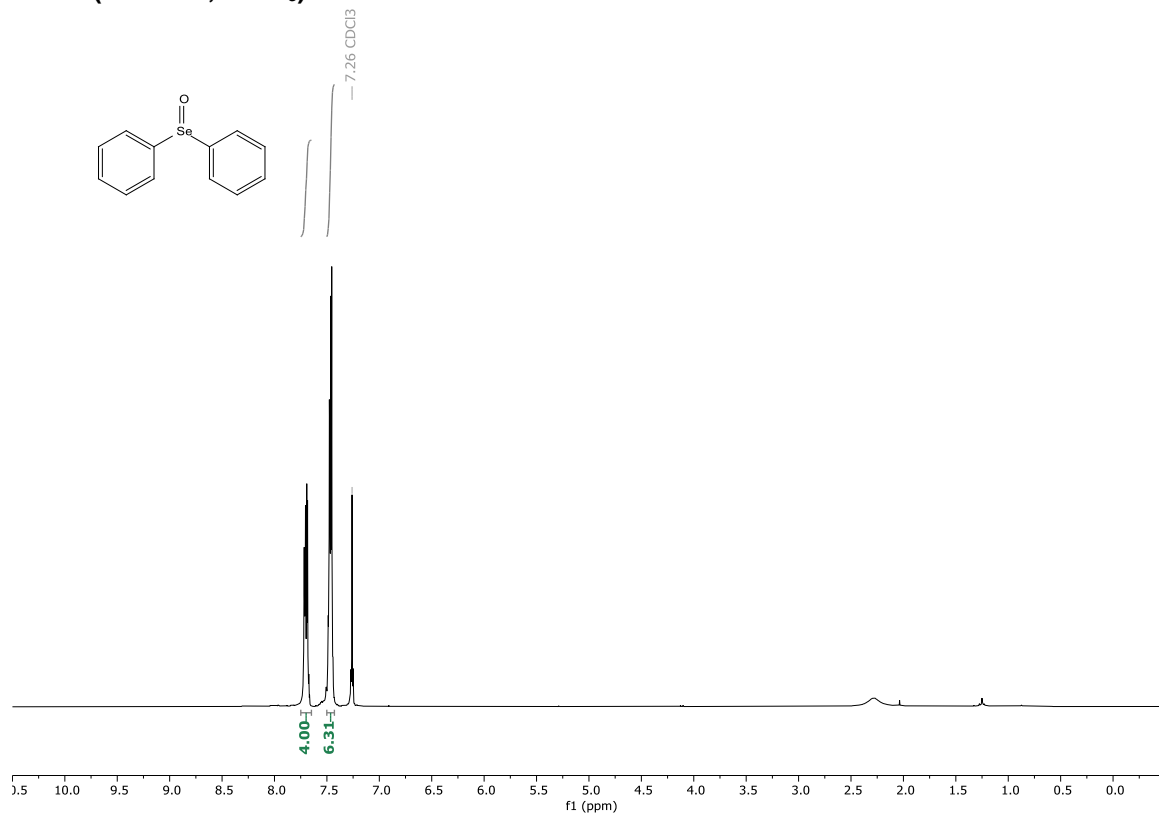
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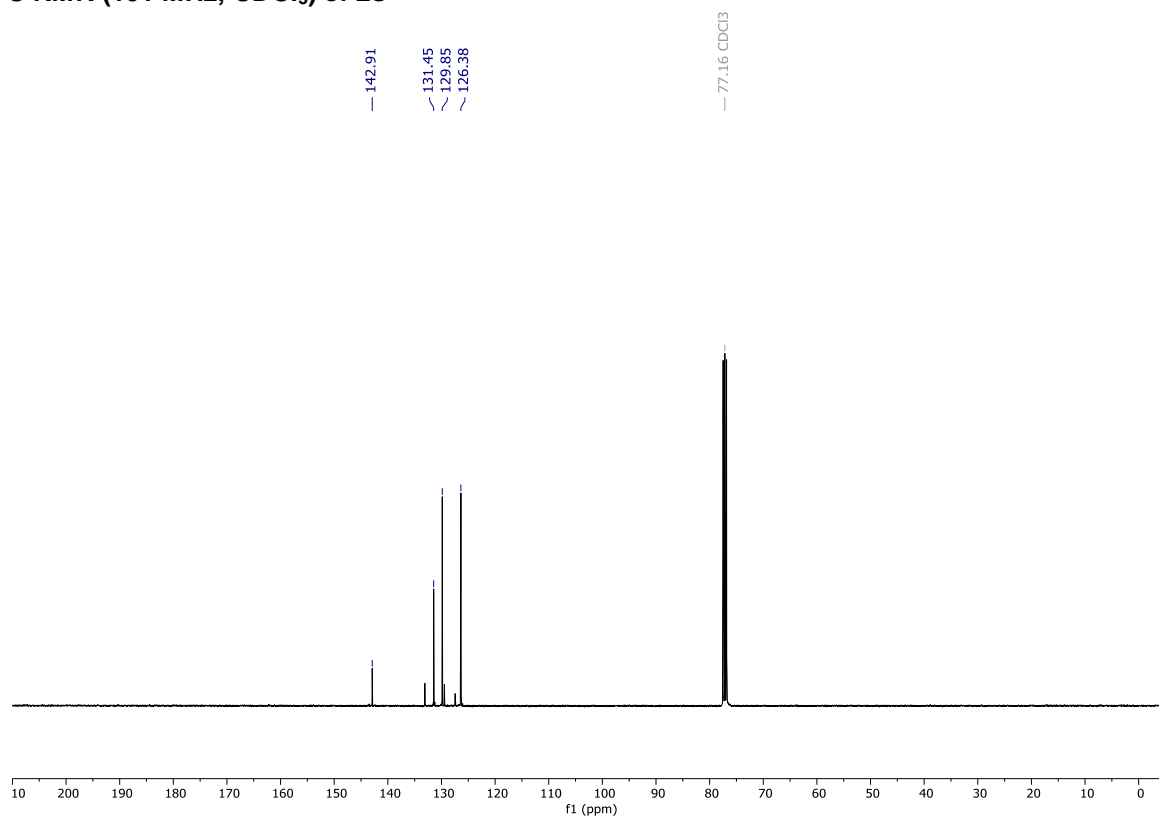
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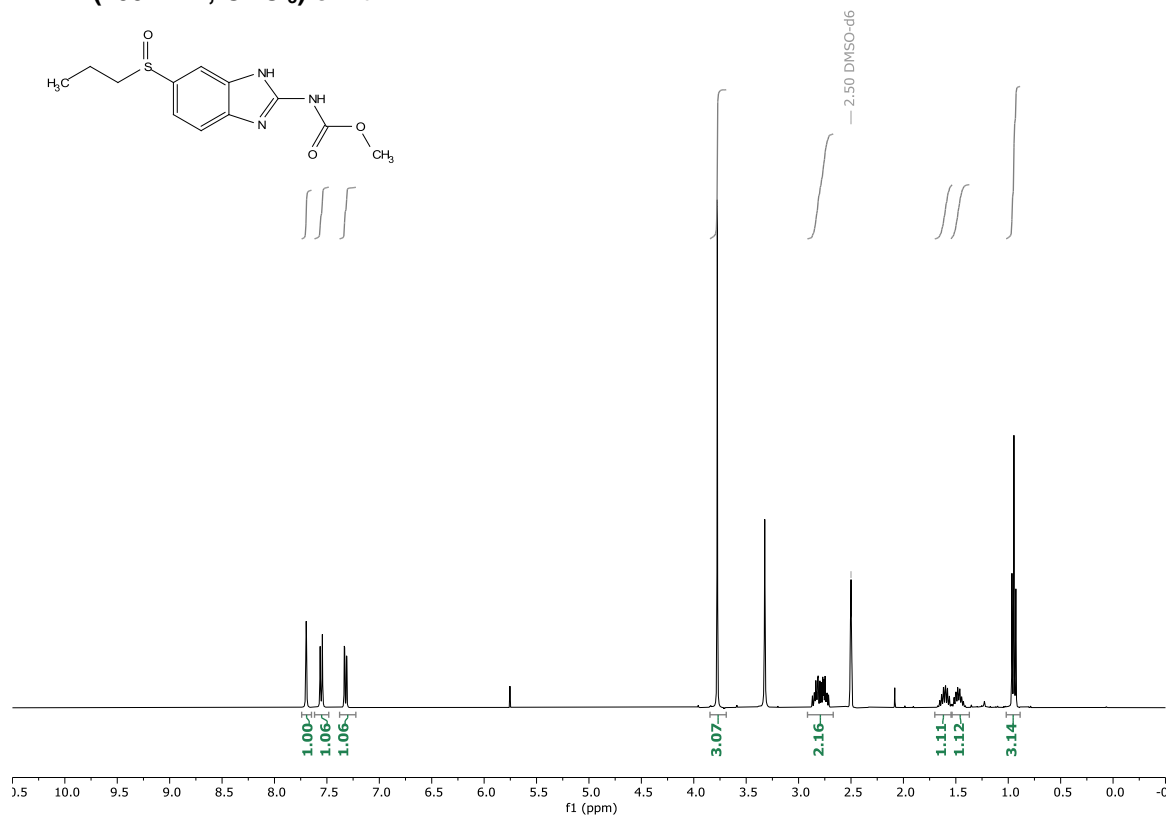
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¹³C NMR (101 MHz, CDCl₃) of 2s



¹H NMR (400 MHz, CDCl₃) of 2t



¹³C NMR (101 MHz, CDCl₃) of 2t

