Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information

Metal-free photoinduced hydrogen atom transfer assisted C(sp^3)–H thioarylation

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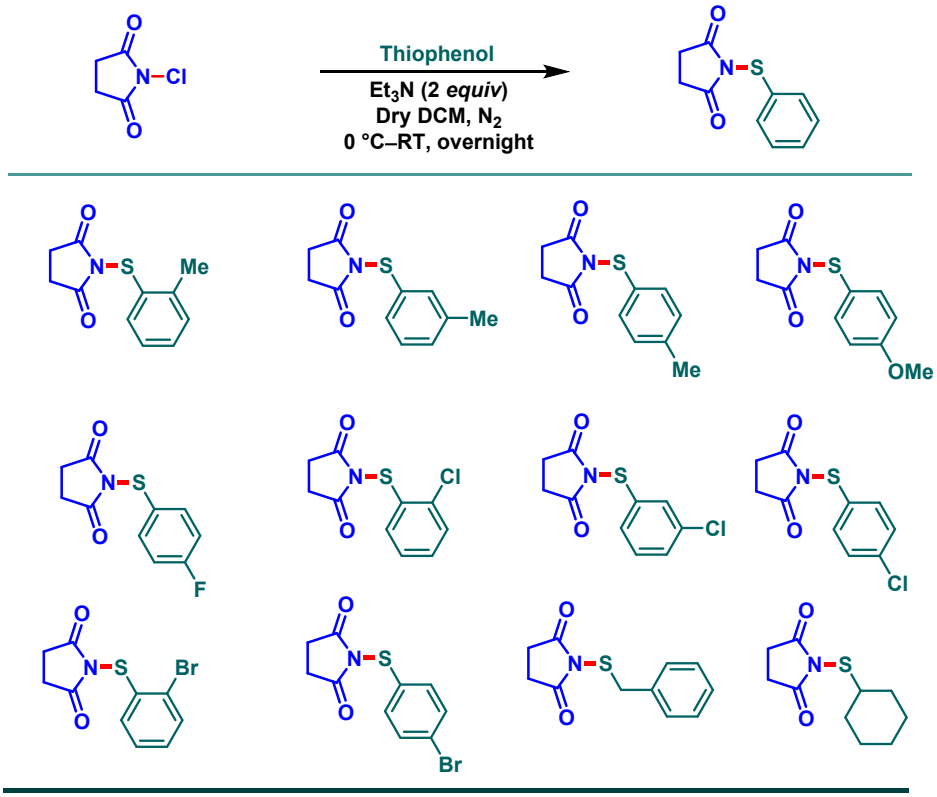
1. *General Information:*

All required fine chemicals were purchased from Sigma-Aldrich, Alfa-aesar, Spectrochem-India, TCI India, BLD Pharma, and used directly without purification unless stated otherwise. All organic reaction solvents were bought from commercial sources and purified according to the general purification technique. All air and moisture sensitive reactions were carried out under nitrogen atmosphere using standard Glove box technique. ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were acquired at various field strengths as indicated and were referenced to $CHCl_3$(7.26 and 77.20 ppm for ^{1}H and ^{13}C respectively). ^{1}H NMR coupling constants are reported in Hertz and refer to apparent multiplicities and not true coupling constants. Data for ^{1}H are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, h = hextet, hept = heptet, m = multiplet, br = broad), coupling constant (Hz), and integration. High resolution mass spectrometry (HRMS-ESI) experiments were performed on Agilent 6530 Accurate-Mass Q-TOF LC/MS in ESI mode. FT-IR were recorded on an Agilent Cary 630 instrument and are reported in wavenumbers (cm–1). Analytical TLC: aluminum backed plates pre-coated (0.25 mm) with Merck Silica Gel 60 F254. Compounds were visualized by exposure to UV-light or by dipping the plates in permanganate ($KMnO_4$) stain followed by heating or by dipping the plates in iodine chamber. Flash column chromatography was performed using Merck Silica Gel (100-200 mesh). All mixed solvent eluents are reported as v/v solutions. All the reactions were conducted in Borosilicate 16 mL glass tubes with threaded end; Kimble Black phenolic screw thread closures with open tops; Thermo Scientific National PTFE/Silicone septa for sample screw thread caps. Custom-made photo setup (with fan): Kessil PR160L Blue LEDs (390 nm) bulb arranged around the reaction tubes (~ 3 cm distance between the light source and the tube). Yield for optimization studies were determined form NMR of the crude reaction mixtures using 1,3,5-trimethoxybenzene (TMB) as internal standard.

2. Synthesis of starting materials

Preparation of thioarylating coupling partners:

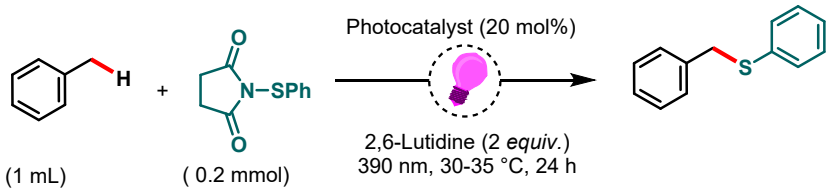
These were prepared according to the procedures known in the literature**.** All these starting materials are already known in the literatures.



3. Optimization of reaction conditions

Yield denoted are GC yields using *n*-decane as internal standard.

Table S1. Photocatalysts Optimization



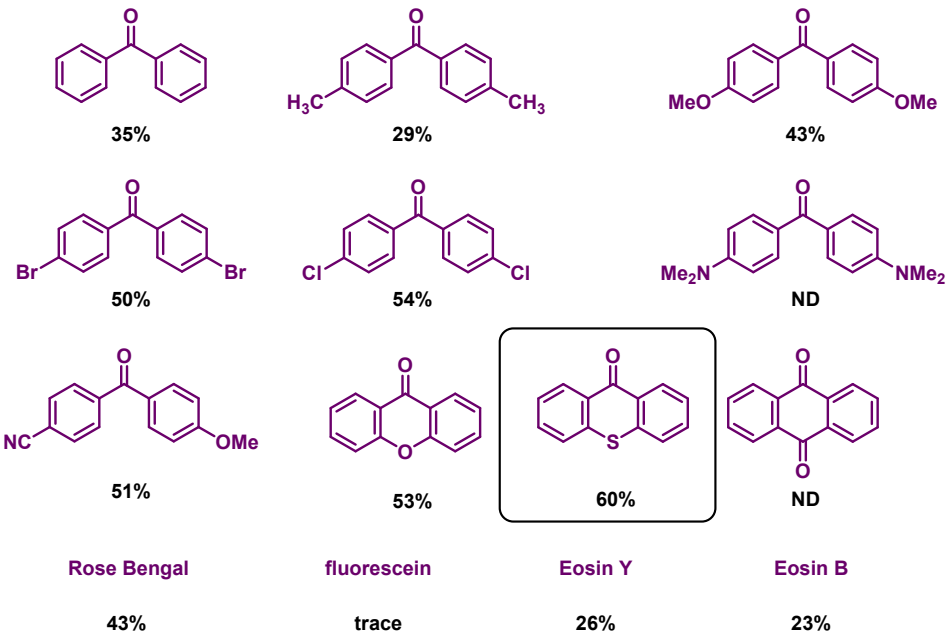
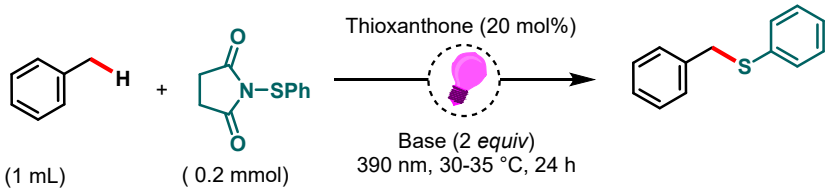
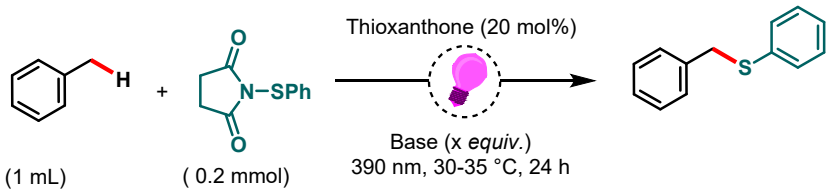


Table S2. Base Optimization



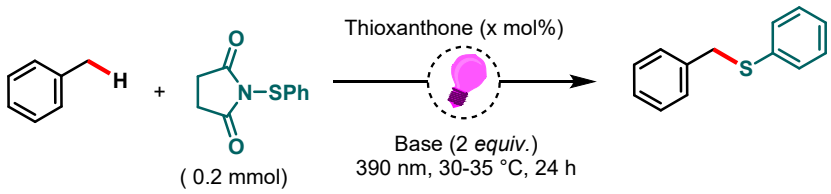
S. No.	Base	Yield
1.	Na_2CO_3	33%
2.	K_2CO_3	41%
3.	K_2HPO_4	58%
4.	**K_3PO_4**	**63%**
5.	DIPEA	51%
6.	2,6-lutidine	60%
7.	Tetramethylguanidine (TMG)	ND
8.	DBU	37%

Table S3. Base amount optimization



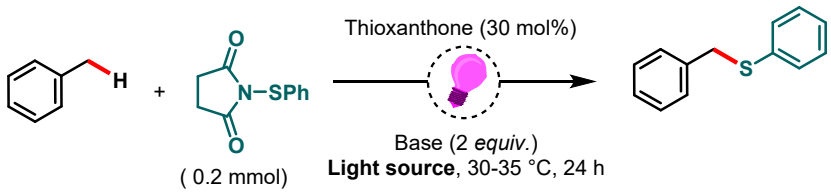
S. No.	K_3PO_4 (equiv)	Yield (%)
1.	1	41
2.	**2**	**63**
3.	3	58
4.	4	47

Table S4. Photocatalyst amount optimization



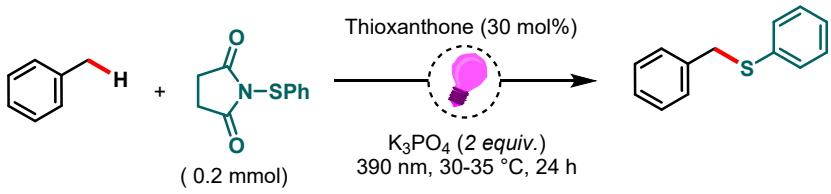
S. No.	Thioxanthone (mol%)	Yield (%)
1.	10	42
2.	20	63
3.	**30**	**68**
4.	40	65

Table S5. Light source optimization



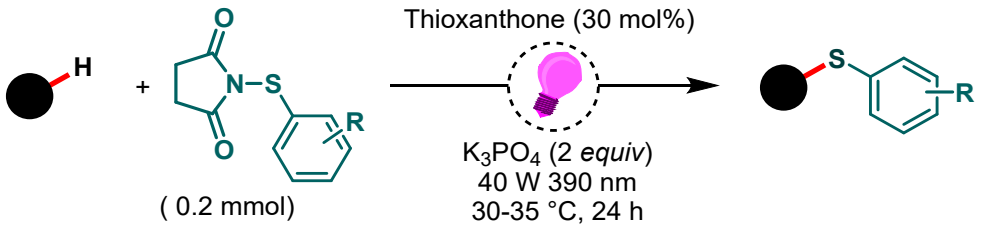
S. No.	Light	Yield (%)
1.	370 nm	62
2.	**390 nm**	**68**
3.	440 nm	7
4.	456 nm	9
5.	While CFL	44

Table S6: Control experiment



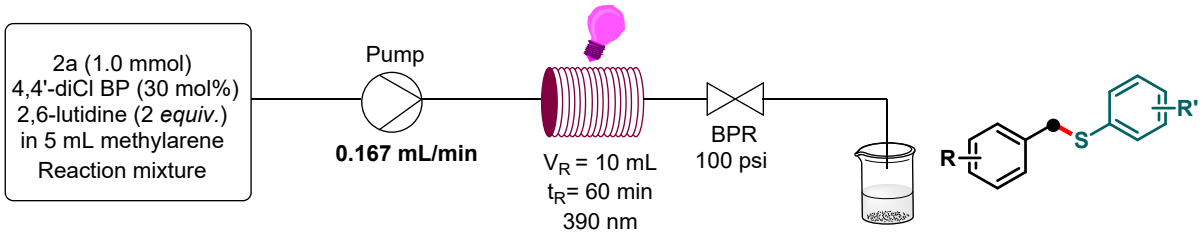
S. No.	Deviation	Yield (%)
1.	**None**	**68**
2.	No PC	ND
3.	No Base	21
4.	No light at RT	trace

4. General Procedure for the thioarylation reaction of methylarenes and alkanes under optimized conditions:



To an oven-dried screw cap reaction tube equipped with a magnetic stir bar was added thioxanthone (12.7 mg, 0.06 equiv.), K_3PO_4 (84.8 mg, 2.0 equiv.), 1-(phenylthio)pyrrolidine-2,5-dione derivative (0.2 mmol, 1.0 equiv.) inside the Glove box. The tube was sealed with a rubber fitted screw cap followed by sealing with parafilm, taken out from the Glove box. To this reaction vial was added 1 mL of respective methylarene/alkane under positive flow of nitrogen. The reaction vial was irradiated with 40 W, 390 nm Kessil PR-160L Blue LED from 3 cm away for 24 hours under the continuous flow of fan to maintain the temperature at 30-35 °C. The reaction was quenched by exposure to air, followed by water and subsequent extraction with EtOAc (3 x 5 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuum. The crude residue was purified by flash chromatography on silica gel (100-200 mesh size) to afford the desired thioarylated product.

5. Scale-up reaction set-up using continuous flow-photo reactor

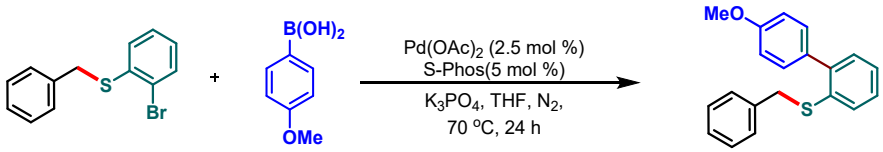


1-(phenylthio)pyrrolidine-2,5-dione (2.0 mmol) (2a), 4,4'-diChlorobenzophenone (0.3 *equiv.*), 2,6-lutidine (2 *equiv.*) were dissolved in 5 mL toluene. It was filled into a 10 mL syringe and then loaded onto a syringe pump. The reaction mixture was passed to a micro flow reactor which was illuminated with two 40 W, 390 nm Kessil PR-160L Blue LEDs. A fan was kept to prevent the reactor from heating. 60 min of the residence time was achieved by keeping the flow rate of 0.167 mL per min. The reaction was quenched by exposure to air, followed by water and subsequent extraction with EtOAc (3 x 5 mL). The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuum. The crude residue was purified by flash chromatography on silica gel (100-200 mesh size) to afford the desired thioarylated product.

6. Post synthetic modifications

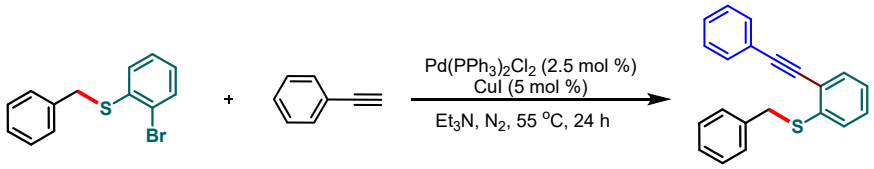
Post synthetic modifications were performed *via* literature known procedures.[1-4]

a. Suzuki coupling[1]: An oven-dried screw cap reaction tube was charged with a magnetic stir-bar. [$Pd(OAc)_2$] (2.5 mol%), S-Phos (5 mol%), bromo-thioarylated product (0.2 mmol), and 4-methoxy phenyl boronic acid (1.2 *equiv.*) and K_3PO_4 (3 *equiv.*) were added. Degassed THF (2 ml) was injected in it and the reaction was stirred at room temperature under nitrogen atmosphere. The reaction was stirred continuously for 24 h at 70 °C. Once the reaction was over the mixture was diluted with 5 ml EtOAc and filtered through the celite. The filtrate was evaporated under low pressure and the compound was isolated through silica column (100-200 mesh).



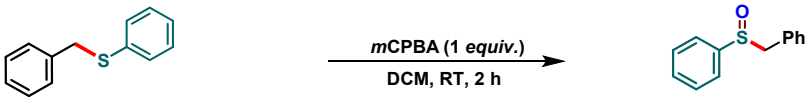
7, 92%

b. Sonogashira coupling[2]: An oven-dried screw cap reaction tube was charged with a magnetic stir-bar. [$Pd(PPh_3)_2Cl_2$] (2.5 mol%), CuI (5 mol%) and bromo-thioarylated product (0.2 mmol). Degassed Triethylamine (1 ml) was injected in it and the reaction was stirred at room temperature under nitrogen atmosphere. Under nitrogen atmosphere phenylacetylene (1.25 *equiv.*) was added to the reaction mixture slowly. Gradually the reaction turned dark. The reaction was stirred continuously for 24 h at 55 °C. Once the reaction was over the mixture was diluted with 5 ml EtOAc and filtered through the celite. The filtrate was evaporated under low pressure and the compound was isolated through silica column (100-200 mesh).



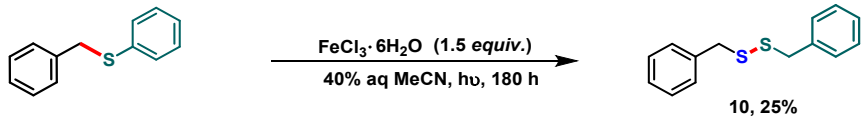
8, 70%

c. Sulfide to sulfoxide[3]: An oven-dried screw cap reaction tube was charged with a magnetic stir-bar. thioarylated product (0.2 mmol) and *meta*-chloroperoxybenzoic acid (1 *equiv.*) were added. DCM (1 ml) was injected in it and the reaction was stirred at room temperature for 2 hours. Once the reaction was over the mixture was diluted with 5 ml DCM and filtered through the celite. The filtrate was evaporated under low pressure and the compound was isolated through silica column (100-200 mesh).

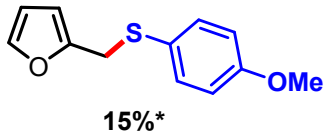


9, 85%

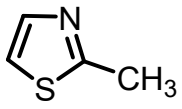
d. Sulfide to unsymmetrical-disulfide[4]: An oven-dried screw cap reaction tube was charged with a magnetic stir-bar. Thioarylated product (0.2 mmol) and $FeCl_3.6H_2O$ (1.5 *equiv.*) were added. 40% aqueous acetonitrile (5 ml) was added to it and the reaction was irradiated with a high-pressure mercury lamp through a Pyrex filter for about 180 h in air. The reaction mixture was evaporated under low pressure and isolated through silica column (100-200 mesh).



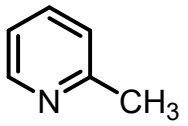
Failed or low yielding heteroarenes:



* determined by GC-MS



2-methylthiazole

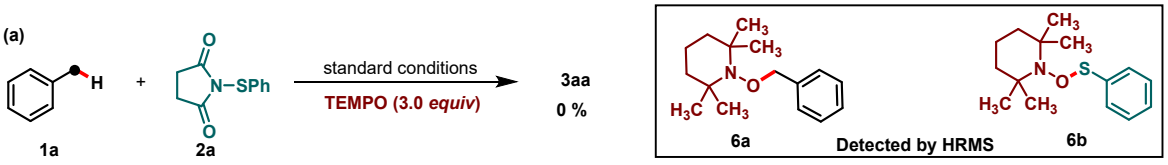


2-methylpyridine

7. Mechanistic investigations

a. Radical trapping experiment with TEMPO and BHT

When the radical scavenger TEMPO (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (3.0 *equiv.*) or BHT(Butylatedhydroxytoluene) (3.0 *equiv.*) was added to the standard reaction conditions, no conversion to the desired thioarylated product was observed, suggesting that the reaction proceeds via a radical pathway (Scheme S4a). The TEMPO and BHT adduct formation was detected through HRMS study. All these observations indicate the generation of benzyl and thioaryl radical.

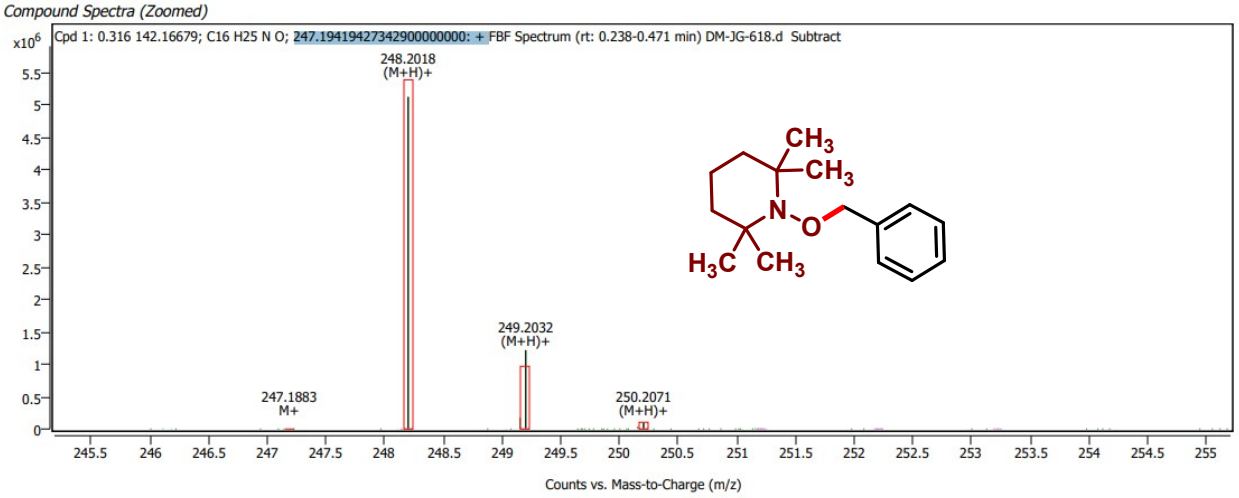


HRMS spectra of the benzylic radical adduct with TEMPO

Cpd. 1: C16 H25 N O

Formula	m/z	Observed M/Z	Difference_Da	Difference_PPM	Score
C16 H25 N O	248.2018	248.201845912194	0.579846228617953	2.34571685826747	87.44

Compound Spectra (Zoomed)



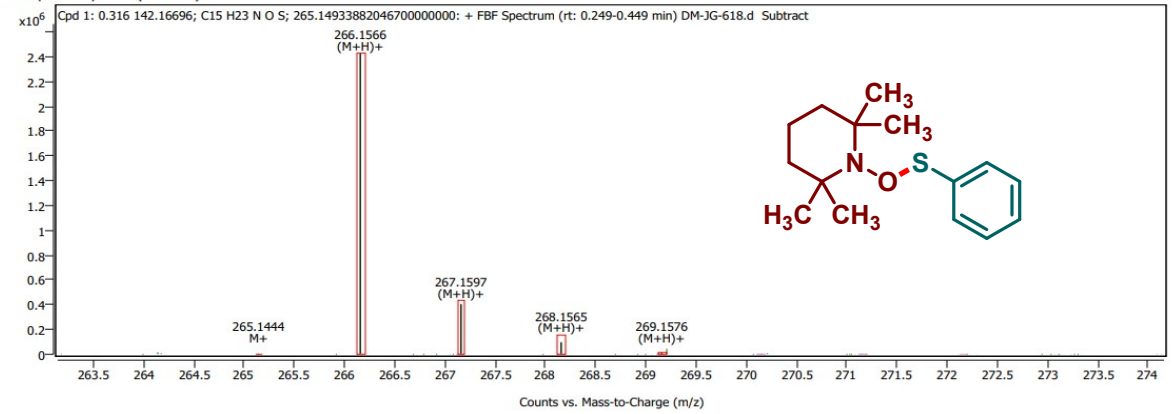
HRMS spectra of the thioaryl radical adduct with TEMPO

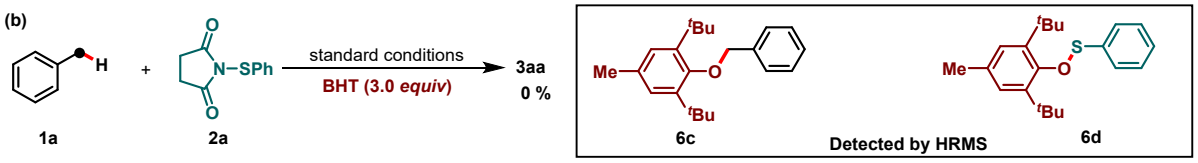
Compound Details

Cpd. 1: C15 H23 N O S

Formula	m/z	Observed M/Z	Difference_Da	Difference_PPM	Score
C15 H23 N O S	266.1566	266.156562817579	-0.69627293277108	-2.62595829008967	96.53

Compound Spectra (Zoomed)





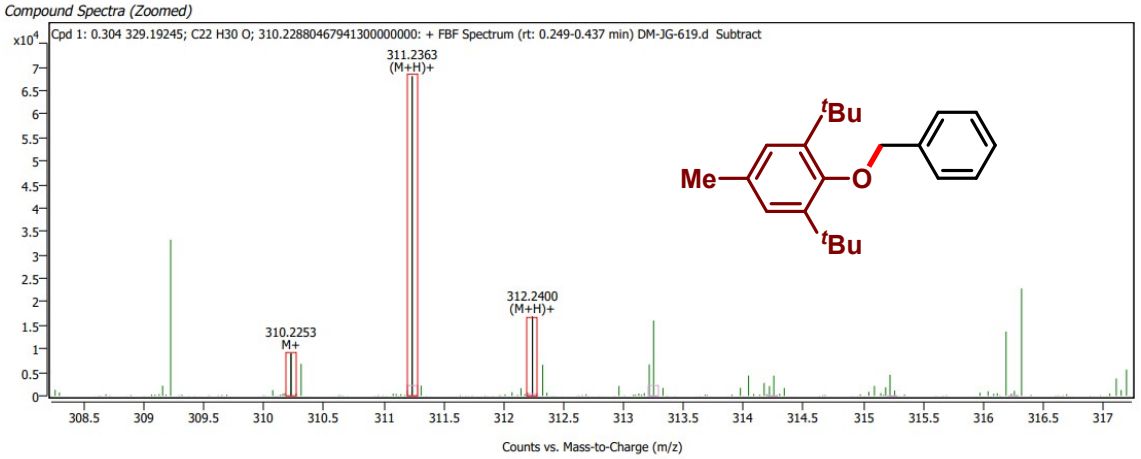
HRMS spectra of the benzylic radical adduct with BHT

Compound Details

Cpd. 1: C22 H30 O

Formula	m/z	Observed M/Z	Difference_Da	Difference_PPM	Score
C22 H30 O	311.2363	311.236334523362	-0.860899887243249	-2.77504050309202	83.92

Compound Spectra (Zoomed)



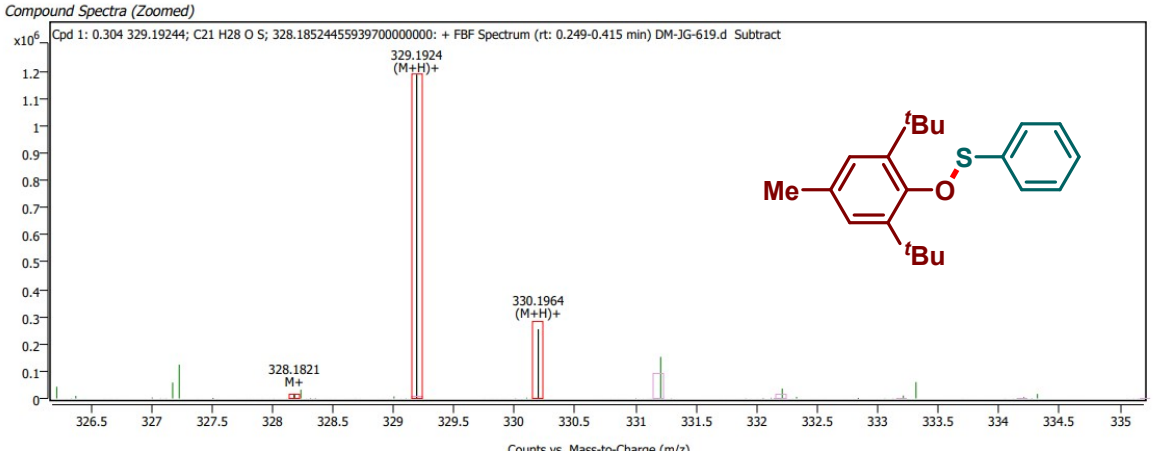
HRMS spectra of the thioaryl radical adduct with BHT

Compound Details

Cpd. 1: C21 H28 O S

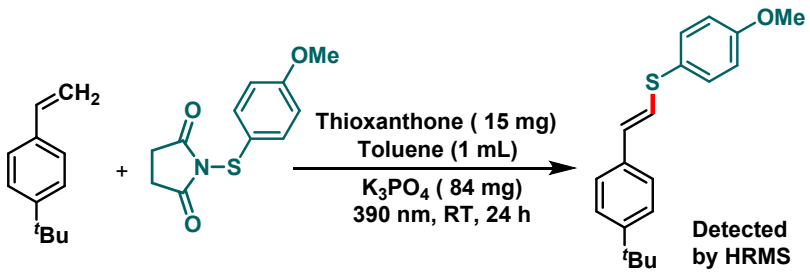
Formula	m/z	Observed M/Z	Difference_Da	Difference_PPM	Score
C21 H28 O S	329.1924	329.192437835529	-0.841686102887707	-2.56466114245344	73.05

Compound Spectra (Zoomed)



c. Radical trapping experiment with 4-tert butyl styrene

When *tert*-butyl styrene was added to the standard reaction conditions, no desired thioarylated product was observed rather the thioaryl group added to olefin was detected by HRMS.

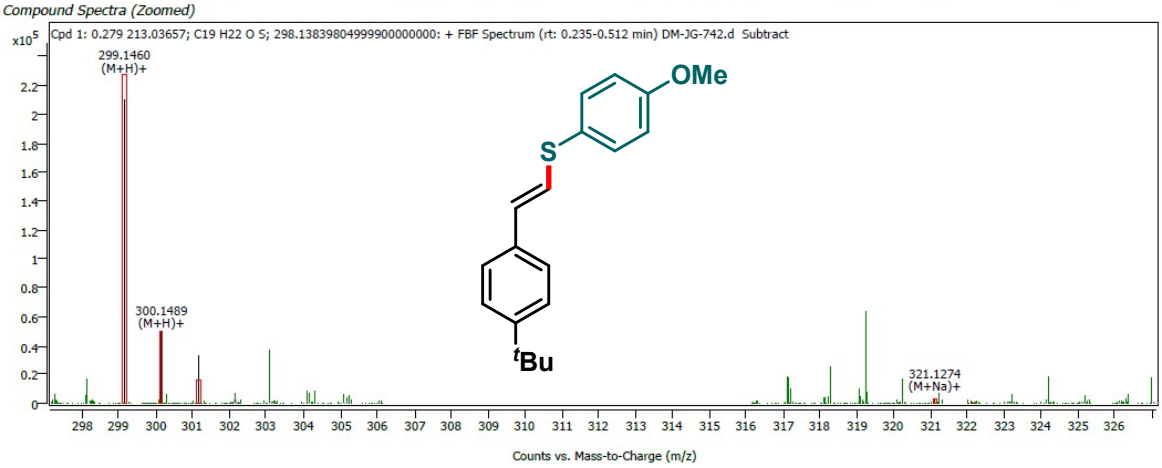


Compound Details

Cpd. 1: C19 H22 O S

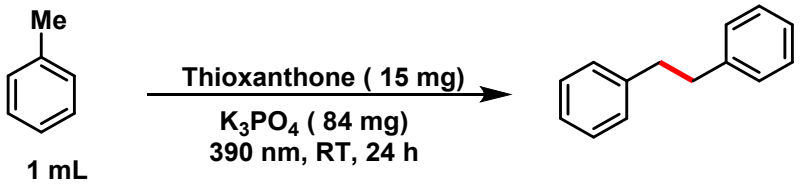
Formula	m/z	Observed M/Z	Difference_Da	Difference_PPM	Score
C19 H22 O S	299.1460	299.146035806869	-0.738004100696799	-2.47536807969713	80.65

Compound Spectra (Zoomed)



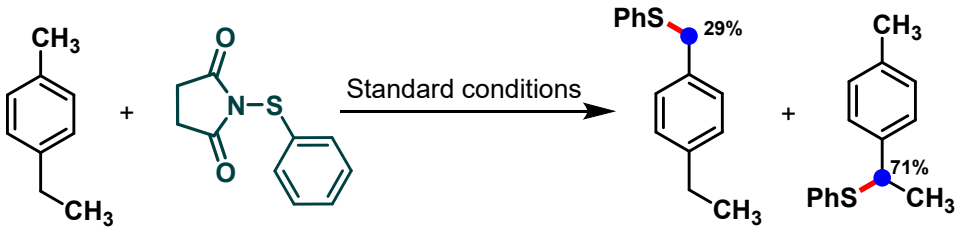
d. Radical probing experiment without coupling partner

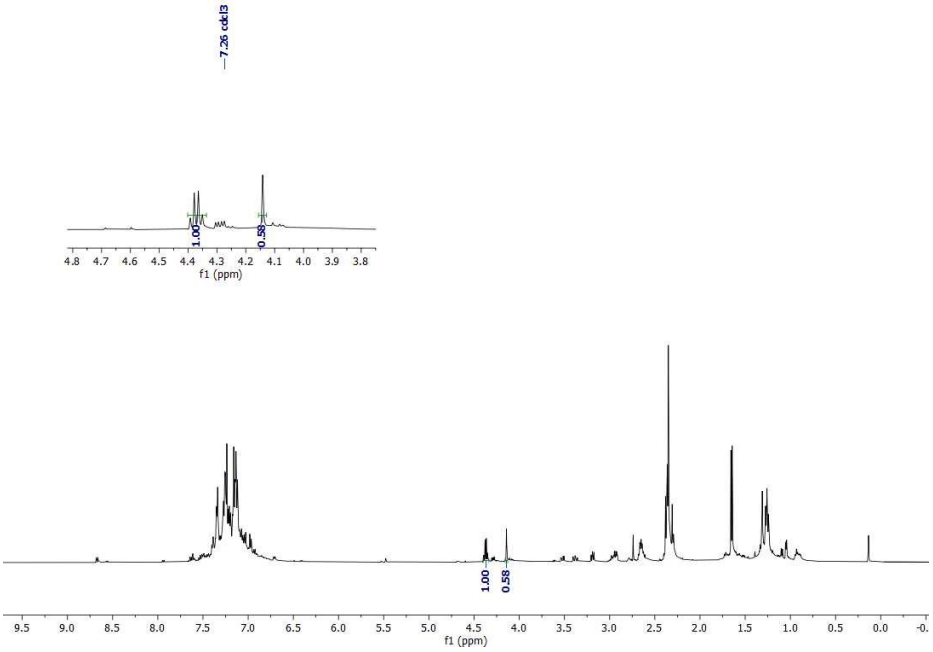
When no thioaryl coupling partner *w*as added to the standard reaction conditions, 1,2-diphenylethane was observed by GC–MS.



e. Intramolecular competitive experiment using 4-ethyl toluene as substrate

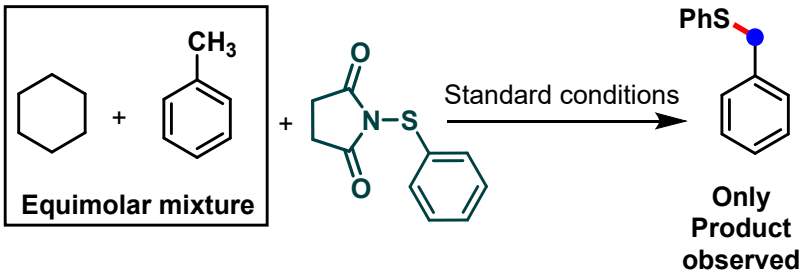
When 4–ethyl toluene was used as the substrate, we observed that product distribution was 29:71. 29% on the methyl (1°) and 71% on the methylene carbon (2°).

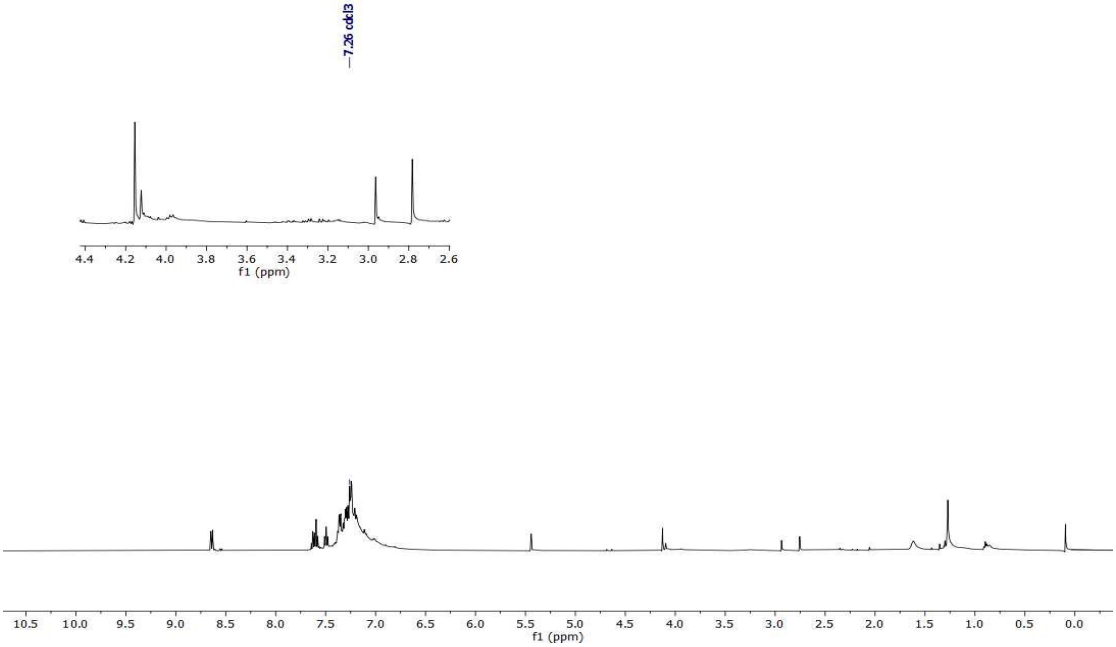




f. Intramolecular competitive experiment using 4-ethyl toluene as substrate

When toluene and cyclohexane equimolar mixture was used as the substrate, we observed that thioarylated product formed only with toluene.





g. Stern-volmer fluorescence quenching experiments:

Fluorescence quenching experiments were performed on a Horiba Duetta spectrometer and 1 mm High Precision Cell made of quartz from Hellma Analytics. In a typical experiment, a 10

mM solution of photocatalyst in benzene; thioxanthone in dry and distilled benzene was treated with the appropriate amount of quencher in a screw-top 1.0 cm quartz cuvette. All the reagents were dispensed in stock solutions prepared volumetrically inside a nitrogen filled glove box. All solutions were excited at λ = 390 nm and the luminescence was measured over a range of 450 – 700 nm (λ_{max} = 474 nm). It was clearly found that the both toluene (substrate) and thioaryl coupling partner (1-(phenylthio)pyrrolidine-2,5-dione) were able to quench the emission of thioxanthone (Fig. S2 and S3). Notably, dilution using benzene could not quench the emission of the photocatalyst.

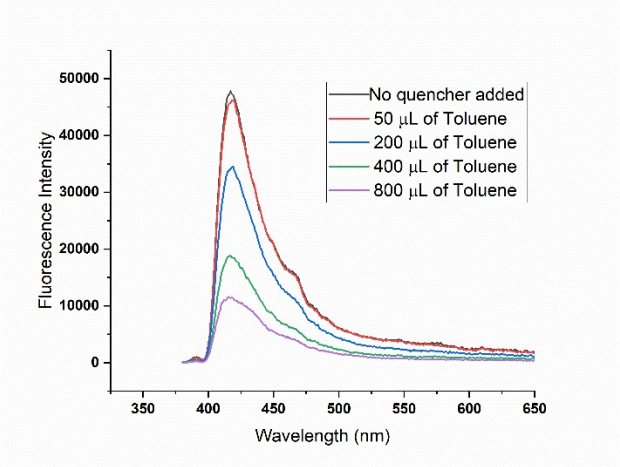


Figure S1. Emission Quenching of thioxanthone using toluene

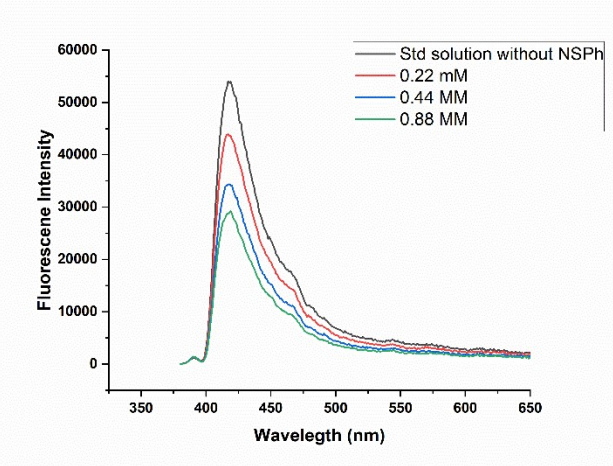
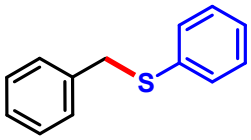


Figure S2. Emission Quenching of thioxanthone using NSPh

8. Characterization data



Benzyl(phenyl)sulfane (3aa).

The title compound 3aa was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

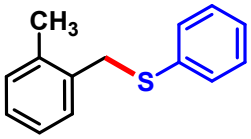
Isolated Yield: 68%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, J = 1.6 Hz, 1H), 7.31 – 7.27 (m, 5H), 7.27 – 7.22 (m, 3H), 7.21 – 7.15 (m, 1H), 4.12 (s, 2H).

^{13}C NMR (101 MHz, CDCl3) δ 137.64, 136.54, 130.03, 129.49, 129.00, 128.67, 127.35, 126.53, 39.25.

GCMS (m/z): calcd. 200.0660 for $C_{13}H_{12}S$ found 200.0.

IR (thin film, cm^{-1}): 735, 908, 1089, 1220, 740, 920, 1089, 1227, 2832.



(2-methylbenzyl)(phenyl)sulfane (3ab)

The title compound 3ab was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

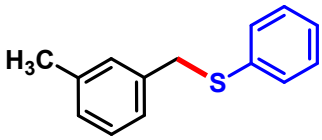
Isolated Yield: 65%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.31 (m, 2H), 7.26 (d, *J* = 4.5 Hz, 2H), 7.23 – 7.19 (m, 1H), 7.18 – 7.13 (m, 3H), 7.12 – 7.07 (m, 1H), 4.11 (s, 2H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.93, 136.80, 135.23, 130.65, 130.42, 129.96, 129.02, 127.70, 126.64, 126.19, 37.60, 19.37.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 740, 920, 1089, 1227, 1574, 1750, 2927, 3062.



(3-methylbenzyl)(phenyl)sulfane (3ac)

The title compound 3ac was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

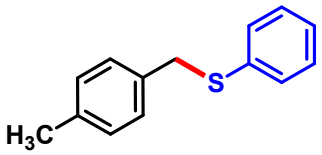
Isolated Yield: 68%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.27 (s, 1H), 7.23 (d, *J* = 9.9 Hz, 1H), 7.20 – 7.15 (m, 2H), 7.13 – 7.07 (m, 2H), 7.05 (d, *J* = 7.5 Hz, 1H), 4.09 (s, 2H), 2.32 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.35, 137.43, 136.79, 129.86, 129.76, 129.00, 128.56, 128.15, 126.43, 126.04, 39.18, 21.53.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 737, 945, 1075, 1240, 1765, 2920.



(4-methylbenzyl)(phenyl)sulfane (3ad)

The title compound 3ad was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

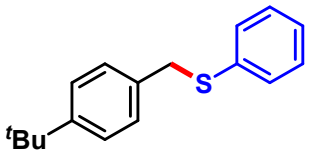
Isolated Yield: 70%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.27 (s, 1H), 7.24 (d, *J* = 8.0 Hz, 1H), 7.21 – 7.14 (m, 3H), 7.09 (d, *J* = 7.8 Hz, 2H), 4.09 (s, 2H), 2.32 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 137.03, 136.81, 134.49, 129.81, 129.37, 128.99, 128.88, 126.38, 38.87, 21.29.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 757, 965, 1085, 1247, 1774, 2932.



(4-(tert-butyl)benzyl)(phenyl)sulfane (3ae)

The title compound 3ae was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

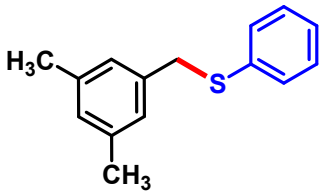
Isolated Yield: 68%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.33 (m, 4H), 7.33 – 7.26 (m, 5H), 4.16 (s, 2H), 1.35 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 150.34, 137.07, 134.40, 129.50, 129.00, 128.66, 126.27, 125.64, 38.63, 34.67, 31.51.

GCMS (m/z): calcd. 256.1286 for $C_{17}H_{20}S$, found 256.1

IR (thin film, cm^{-1}): 762, 971, 1081, 1257, 1781, 2941.



(3,5-dimethylbenzyl)(phenyl)sulfane (3af)

The title compound 3af was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

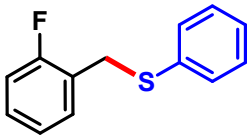
Isolated Yield: 70%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, J = 7.4 Hz, 2H), 7.28 (s, 1H), 7.25 (s, 1H), 7.21 – 7.14 (m, 1H), 6.92 (s, 2H), 6.88 (s, 1H), 4.06 (s, 2H), 2.28 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.24, 137.24, 137.03, 129.69, 129.07, 128.99, 126.82, 126.35, 39.12, 21.40.

HRMS: [ESI, (+) ve]: cald. 227.0894 for $C_{15}H_{15}S$ found: 227.0887.

IR (thin film, cm^{-1}): 728, 904, 1025, 1090, 1479, 1606, 2253, 2920.



(2-fluorobenzyl)(phenyl)sulfane (3ag)

The title compound 3ag was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

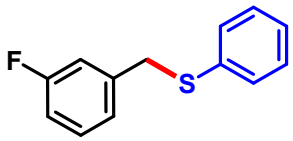
Isolated Yield: 47%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.35 (m, 2H), 7.29 (dd, *J* = 8.3, 6.5 Hz, 3H), 7.27 – 7.20 (m, 2H), 7.10 – 7.01 (m, 2H), 4.17 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 161.88, 159.91, 135.84, 130.98, 130.95, 130.62, 129.09, 129.03, 129.00, 126.83, 125.02, 124.90, 124.18, 124.15, 115.59, 115.42, 32.32, 32.30.

HRMS: [ESI, (+) ve]: cald. 217.0486 for $C_{13}H_{10}FS$ found 217.0482.

IR (thin film, cm^{-1}): 736, 906, 1088, 1185, 1232, 1489, 1795, 1948, 2249, 2933, 3062.



(3-fluorobenzyl)(phenyl)sulfane (3ah)

The title compound 3ah was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

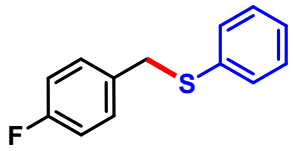
Isolated Yield: 44%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 8.2, 1.5 Hz, 2H), 7.34 – 7.29 (m, 2H), 7.30 – 7.27 (m, 1H), 7.27 – 7.23 (m, 1H), 7.15 – 7.04 (m, 2H), 6.98 (td, *J* = 8.5, 2.6 Hz, 1H), 4.14 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.86, 161.91, 140.30, 140.25, 135.82, 130.20, 130.03, 129.97, 129.03, 126.75, 124.56, 124.54, 115.88, 115.70, 114.29, 114.12, 38.76, 38.74.

HRMS: [ESI, (+) ve]: cald. 217.0486 for $C_{13}H_{10}FS$ found 217.0478.

IR (thin film, cm^{-1}): 732, 785, 909, 1045, 1241, 1373, 1446, 1734, 2256, 2985.



(3-fluorobenzyl)(phenyl)sulfane (3ai)

The title compound 3ai was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

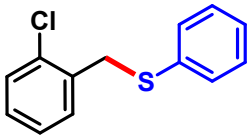
Isolated Yield: 51%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 3H), 7.22 – 7.16 (m, 3H), 6.99 – 6.90 (m, 2H), 4.07 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 162.95, 161.00, 135.86, 133.28, 133.26, 130.38, 130.32, 130.22, 128.89, 126.60, 115.41, 115.24, 38.46.

HRMS: [ESI, (+) ve]: cald. 217.0486 for $C_{13}H_{10}FS$ found 217.0471.

IR (thin film, cm^{-1}): 754, 96, 1063, 1232, 1451, 1734, 2276, 2960.



(2-chlorobenzyl)(phenyl)sulfane (3aj)

The title compound 3aj was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

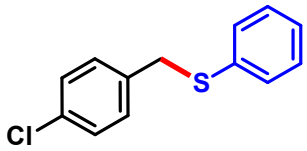
Isolated Yield: 56%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (ddd, J = 12.3, 7.8, 2.1 Hz, 3H), 7.31 – 7.18 (m, 4H), 7.18 – 7.09 (m, 2H), 4.22 (d, J = 2.1 Hz, 2H)

^{13}C NMR (101 MHz, $CDCl_3$) δ 135.87, 135.32, 134.17, 130.82, 130.76, 129.76, 129.01, 128.70, 126.88, 126.85, 37.08.

HRMS: [ESI, (+) ve]: cald. 233.0191 for $C_{13}H_{10}ClS$ found 233.0171.

IR (thin film, cm^{-1}): 691, 758, 928, 1038, 1215, 1476, 1581, 2401, 3020.



(4-chlorobenzyl)(phenyl)sulfane (3ak)

The title compound 3ak was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

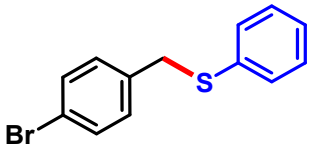
Isolated Yield: 59%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 3H), 7.25 (d, *J* = 1.7 Hz, 2H), 7.24 – 7.17 (m, 4H), 4.06 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.34, 135.85, 133.13, 130.49, 130.30, 129.10, 128.79, 126.88, 38.77.

HRMS: [ESI, (+) ve]: cald. 233.0191 for $C_{13}H_{10}ClS$ found 233.0170.

IR (thin film, cm^{-1}): 813, 907, 1011, 1095, 1215, 1389, 1475, 1573, 1883, 2925, 3030.



(4-bromobenzyl)(phenyl)sulfane (3al)

The title compound 3al was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid.

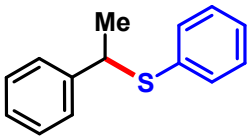
Isolated Yield: 55%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.4 Hz, 2H), 7.31 – 7.22 (m, 4H), 7.22 – 7.16 (m, 1H), 7.13 (d, *J* = 8.4 Hz, 2H), 4.04 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.84, 135.78, 131.73, 130.64, 130.45, 129.10, 126.87, 121.20, 38.78.

HRMS: [ESI, (+) ve]: cald. 276.9687 for $C_{13}H_{10}BrS$ observed 278.9698.

IR (thin film, cm^{-1}): 853, 1011, 1235, 1391, 1470, 1887, 2965, 3070.



Phenyl(1-phenylethyl)sulfane (3am)

The title compound 3am was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

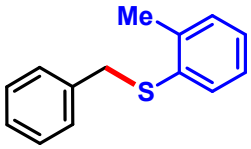
Isolated Yield: 69%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 – 7.34 (m, 6H), 7.33 – 7.25 (m, 4H), 4.44 (q, *J* = 7.0 Hz, 1H), 1.73 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 143.33, 135.27, 132.58, 128.80, 128.51, 127.40, 127.25, 127.22, 48.10, 22.47.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 695, 840, 968, 1088, 1221, 1450, 1583, 1802, 1875, 1947, 2970, 3061.



benzyl(o-tolyl)sulfane (3an)

The title compound 3an was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

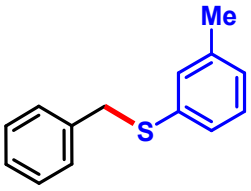
Isolated Yield: 62%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 6H), 7.24 – 7.14 (m, 3H), 4.14 (s, 2H), 2.40 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 138.01, 137.40, 135.91, 130.17, 129.06, 129.00, 128.61, 127.31, 126.54, 126.23, 38.43, 20.44.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 695, 854, 916, 1199, 1387, 1476, 1570, 1768, 1947, 2925.



benzyl(m-tolyl)sulfane (3ao)

The title compound 3ao was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

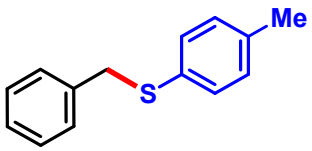
Isolated Yield: 64%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 5H), 7.24 (d, *J* = 8.2 Hz, 3H), 7.13 – 7.04 (m, 1H), 4.09 (s, 2H), 2.33 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.67, 137.64, 136.32, 130.46, 128.97, 128.80, 128.58, 127.27, 127.25, 126.75, 39.07, 21.43.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 685, 876, 976, 1180, 1371, 1416, 1538, 1771, 1949, 2968.



benzyl(m-tolyl)sulfane (3ap)

The title compound 3ap was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

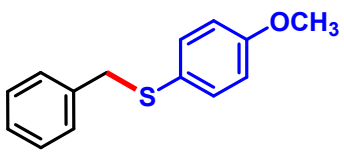
Isolated Yield: 65%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 4H), 7.24 (d, *J* = 8.2 Hz, 3H), 7.13 – 7.04 (m, 2H), 4.09 (s, 2H), 2.33 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.96, 136.71, 132.64, 130.86, 129.77, 128.99, 128.59, 127.23, 39.94, 21.21.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.0.

IR (thin film, cm^{-1}): 695, 854, 998, 1081, 1238, 1319, 1493, 1591, 1756, 1944, 2920, 3029.



benzyl(4-methoxyphenyl)sulfane (3aq)

The title compound 3aq was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

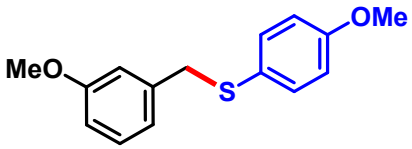
Isolated Yield: 72%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (ddd, *J* = 7.4, 5.3, 1.9 Hz, 4H), 7.28 (td, *J* = 7.3, 1.5 Hz, 3H), 6.86 (d, *J* = 8.8 Hz, 2H), 4.06 (s, 2H), 3.80 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.19, 138.15, 134.00, 128.91, 128.37, 126.99, 126.11, 114.45, 55.23, 41.15.

GCMS (m/z): calcd. 230.0765 for $C_{14}H_{14}OS$ found 230.0.

IR (thin film, cm^{-1}): 737, 824, 1030, 1177, 1247, 1498, 1580, 1712, 1888, 2050, 2530, 2949.



(3-methoxybenzyl)(4-methoxyphenyl)sulfane (3ar)

The title compound 3ar was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (96:4).

Physical State: Colourless liquid

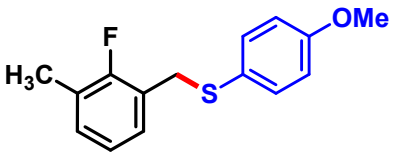
Isolated Yield: 71%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.7 Hz, 2H), 7.19 (t, *J* = 7.7 Hz, 1H), 6.91 – 6.58 (m, 5H), 3.99 (s, 2H), 3.78 (d, *J* = 1.1 Hz, 3H), 3.76 (d, *J* = 1.1 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.60, 159.25, 139.72, 134.05, 129.39, 126.16, 121.29, 114.49, 114.21, 112.86, 55.33, 55.18, 41.24.

HRMS: [ESI, (+) ve] Cald. 260.0871 for $C_{15}H_{16}O_2S$ found 260.0852.

IR (thin film, cm^{-1}): 706, 782, 910, 1102, 1243, 1455, 1491, 1590, 1732, 2043, 2530, 2835.



(2-fluoro-3-methylbenzyl)(4-methoxyphenyl)sulfane (3as)

The title compound 3as was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (96:4).

Physical State: Colourless liquid

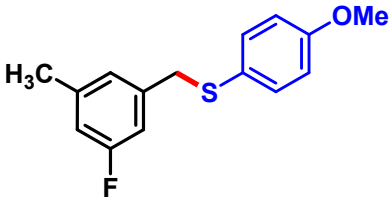
Isolated Yield: 64%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.7 Hz, 2H), 7.04 (td, *J* = 7.1, 2.7 Hz, 1H), 6.95 – 6.86 (m, 2H), 6.80 (d, *J* = 8.7 Hz, 2H), 4.00 (d, *J* = 1.1 Hz, 2H), 3.79 (s, 3H), 2.25 (d, *J* = 2.1 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 160.42, 159.57, 158.46, 134.73, 130.51, 130.47, 128.49, 128.46, 125.98, 125.08, 124.93, 123.50, 123.46, 114.58, 55.49, 34.70, 34.68, 14.75, 14.72.

HRMS: [ESI, (+) ve] Cald. 262.0828 $C_{15}H_{15}FOS$ found 262.0830.

IR (thin film, cm^{-1}): 735, 788, 919, 1051, 1247, 1375, 1449, 1732, 2256, 2987.



(3-fluoro-5-methylbenzyl)(4-methoxyphenyl)sulfane (3at)

The title compound 3at was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (96:4).

Physical State: Colourless liquid

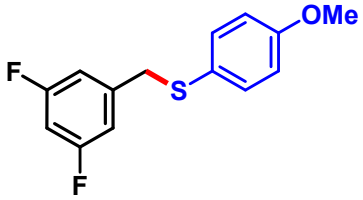
Isolated Yield: 68%

^{1}H NMR (500 MHz, CDCl3) δ 7.31 (d, J = 8.9 Hz, 2H), 6.85 (d, J = 8.8 Hz, 2H), 6.82 (s, 1H), 6.77 (dd, J = 9.5, 5.8 Hz, 2H), 3.96 (s, 2H), 3.81 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.76, 161.81, 159.45, 140.40, 140.35, 140.33, 140.28, 134.23, 125.85, 125.43, 125.41, 114.72, 114.60, 114.56, 112.90, 112.73, 55.37, 40.92, 40.91, 21.32, 21.30.

HRMS: [ESI, (+) ve] Cald. 262.0828 $C_{15}H_{15}FOS$ found 262.0830.

IR (thin film, cm^{-1}): 777, 791, 921, 1049, 1235, 1378, 1452, 1752, 2210, 2991.



(3,5-difluorobenzyl)(4-methoxyphenyl)sulfane (3au)

The title compound 3au was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (96:4).

Physical State: Colourless liquid

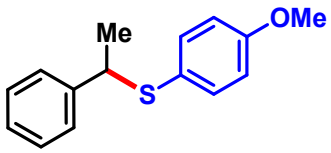
Isolated Yield: 51%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.74 – 6.59 (m, 3H), 3.91 (s, 2H), 3.78 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 164.22, 164.09, 161.75, 161.63, 159.73, 142.46, 134.63, 124.99, 114.74, 111.92, 111.85, 111.74, 111.67, 102.81, 102.55, 102.30, 55.41, 40.87.

HRMS: [ESI, (+) ve] Cald. 267.0650 $C_{14}H_{13}F_2OS$ found 267.0652.

IR (thin film, cm^{-1}): 788, 915, 1074, 1228, 1373, 1452, 1752, 2212, 2993.



(4-methoxyphenyl)(1-phenylethyl)sulfane (3av)

The title compound 3av was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

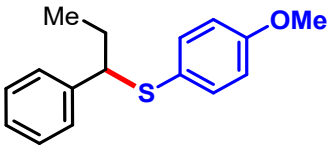
Isolated Yield: 71%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 – 7.21 (m, 2H), 7.25 – 7.18 (m, 5H), 6.76 (d, *J* = 8.7 Hz, 2H), 4.17 (q, *J* = 7.0 Hz, 1H), 3.77 (s, 3H), 1.60 (d, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.77, 143.55, 136.18, 128.44, 127.51, 127.17, 125.29, 114.36, 55.44, 49.30, 21.97.

HRMS: [ESI, (+) ve]: Cald. 244.0922 for $C_{15}H_{16}OS$ found 244.0915.

IR (thin film, cm^{-1}): 698, 967, 1102, 1245, 1373, 1454, 1591, 1678, 1733, 1883, 2044, 2855, 3027.



(4-methoxyphenyl)(1-phenylpropyl)sulfane (3aw)

The title compound 3aw was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

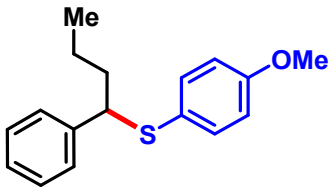
Isolated Yield: 72%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 2H), 7.22 – 7.14 (m, 5H), 6.74 (d, *J* = 8.9 Hz, 2H), 3.88 (dd, *J* = 8.8, 6.2 Hz, 1H), 3.76 (s, 3H), 2.05 – 1.84 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.48, 142.14, 135.91, 130.93, 129.46, 128.20, 127.93, 126.90, 114.15, 56.63, 55.26, 28.80, 12.35.

HRMS: [ESI, (+) ve]: Cald. 258.1078 for $C_{16}H_{18}OS$ found 259.1132.

IR (thin film, cm^{-1}): 756, 830, 916, 1176, 1243, 1460, 1609, 1885, 1949, 2048, 2315, 2543, 2835, 2956.



(4-methoxyphenyl)(1-phenylbutyl)sulfane (3ax)

The title compound 3ax was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

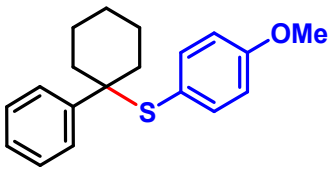
Isolated Yield: 69%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (dd, *J* = 7.0, 1.3 Hz, 2H), 7.22 – 7.06 (m, 5H), 6.73 (d, *J* = 8.8 Hz, 2H), 3.96 (dd, *J* = 8.6, 6.6 Hz, 1H), 3.76 (s, 3H), 2.00 – 1.80 (m, 2H), 1.44 – 1.21 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.63, 142.62, 136.05, 129.49, 128.34, 128.04, 127.02, 114.29, 55.41, 54.79, 37.95, 20.99, 13.95.

HRMS: [ESI, (+) ve]: Cald. 271.1157 for $C_{17}H_{19}OS$ found 271.1160.

IR (thin film, cm^{-1}): 777, 830, 1182, 1477, 1621, 1951, 2021, 2547, 2835, 2956.



(4-methoxyphenyl)(1-phenylcyclohexyl)sulfane (3ay)

The title compound 3ay was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

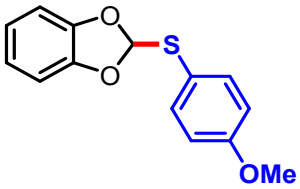
Isolated Yield: 74%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 4H), 7.19 – 7.14 (m, 1H), 6.89 (d, *J* = 8.7 Hz, 2H), 6.66 (d, *J* = 8.7 Hz, 2H), 3.75(s, 3H), 2.28 – 2.16 (m, 2H), 2.08 – 1.92 (m, 2H), 1.88 – 1.71 (m, 2H), 1.52 – 1.35 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.28, 144.61, 138.73, 128.00, 127.20, 126.30, 122.76, 113.75, 55.37, 55.16, 36.33, 26.11, 23.05.

HRMS: [ESI, (+) ve]: cald. 297.1312 for $C_{19}H_{21}OS$ found 297.1307.

IR (thin film, cm^{-1}): 667, 751, 828, 895, 1032, 1113, 1173, 1245, 1285, 1446, 1492, 1591, 1889, 2044, 2856, 2932, 3021.



2-((4-methoxyphenyl)thio)benzo[d][1,3]dioxole (3az)

The title compound 3az was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (98:2).

Physical State: Colourless liquid

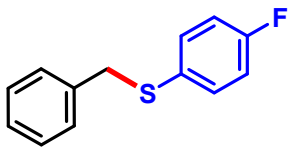
Isolated Yield: 72%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 8.7 Hz, 2H), 7.38 (s, 1H), 6.87 – 6.84 (m, 4H), 6.84 (d, J = 3.1 Hz, 2H), 3.79 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 160.76, 146.28, 137.51, 122.30, 119.15, 117.20, 114.75, 109.21, 55.50.

HRMS: [ESI, (+) ve]: cald 260.0507 for $C_{14}H_{12}O_3S$ found 260.0511.

IR (thin film, cm^{-1}): 790, 917, 1112, 1257, 1475, 1570, 2143, 2532, 2735.



benzyl(4-fluorophenyl)sulfane (3ba)

The title compound 3ba was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

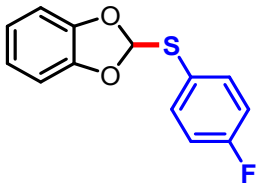
Isolated Yield: 59%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.20 (m, 7H), 6.96 (t, J = 8.7 Hz, 2H), 4.05 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 163.45, 161.00, 137.66, 133.57, 133.50, 130.91, 130.88, 128.99, 128.60, 127.33, 116.15, 115.93, 40.56.

HRMS: [ESI, (+) ve]: Cald. 217.04867 for $C_{13}H_{10}FS$ found 217.0485.

IR (thin film, cm^{-1}): 826, 906, 1090, 1226, 1489, 1589, 1888, 2250, 2924, 3031.



2-((4-fluorophenyl)thio)benzo[d][1,3]dioxole (3bb)

The title compound 3bb was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: White solid.

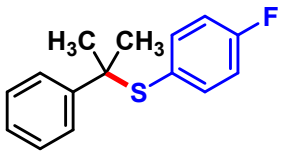
Isolated Yield: 76%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.42 (s, 1H), 7.00 (t, *J* = 8.6 Hz, 2H), 6.84 (s, 4H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 164.68, 162.69, 146.17, 137.82, 137.76, 122.46, 116.75, 116.73, 116.41, 116.24, 109.25.

HRMS: [ESI, (+) ve]: Cald. 248.0307 for $C_{13}H_9FO_2S$ found 248.0305.

IR (thin film, cm^{-1}): 790, 927, 1109, 1261, 1458, 1570, 2043, 2532, 2652.



(4-fluorophenyl)(2-phenylpropan-2-yl)sulfane (3bc)

The title compound 3bc was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

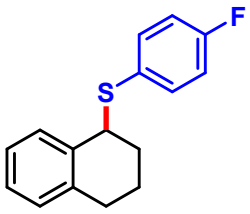
Isolated Yield: 61%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.31 – 7.25 (m, 2H), 7.24 – 7.16 (m, 1H), 7.13 – 7.01 (m, 2H), 6.86 (t, *J* = 8.7 Hz, 2H), 1.68 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 146.21, 138.76, 138.69, 128.09, 126.80, 126.73, 115.58, 115.41, 29.90, 29.63.

GCMS (m/z): cald. 246.0878 for $C_{15}H_{15}FS$ found 246.0.

IR (thin film, cm^{-1}): 833, 916, 1078, 1233, 1491, 1578, 1878, 2247, 2932, 3034.



(4-fluorophenyl)(1,2,3,4-tetrahydronaphthalen-1-yl)sulfane (3bd)

The title compound 3bd was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

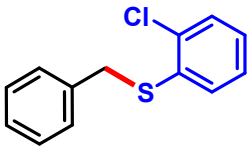
Isolated Yield: 64%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 – 7.42 (m, 2H), 7.36 (dd, *J* = 6.7, 2.3 Hz, 1H), 7.19 – 7.12 (m, 2H), 7.12 – 7.07 (m, 1H), 7.07 – 6.96 (m, 2H), 4.45 (t, *J* = 4.1 Hz, 1H), 2.94 – 2.60 (m, 2H), 2.36 – 2.10 (m, 1H), 2.02 – 1.87 (m, 2H), 1.86 – 1.67 (m, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.61, 161.64, 137.74, 135.56, 135.40, 135.34, 130.94, 130.61, 129.47, 127.32, 125.86, 116.27, 116.10, 48.91, 29.25, 28.55, 18.72.

GCMS (m/z): cald. 258.0878 for $C_{16}H_{15}FS$ found 258.0.

IR (thin film, cm^{-1}): 835, 9017, 1074, 1231, 1479, 1589, 1887, 2247, 2946, 3052.



benzyl(2-chlorophenyl)sulfane (3be)

The title compound 3be was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

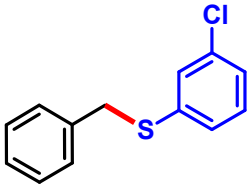
Isolated Yield: 61%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.27 (m, 7H), 7.18 (dtd, *J* = 21.9, 7.4, 1.6 Hz, 2H), 4.20 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.32, 135.89, 133.55, 129.64, 129.13, 129.11, 128.96, 128.62, 127.44, 127.13, 126.83, 37.46.

HRMS: [ESI, (+) ve]: Cald. 233.0192 for $C_{13}H_{10}ClS$ found 233.0198.

IR (thin film, cm^{-1}): 743, 907, 1034, 1114, 1237, 1256, 1451, 1494, 1575, 1899, 2925.



benzyl(3-chlorophenyl)sulfane (3bf)

The title compound 3bf was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

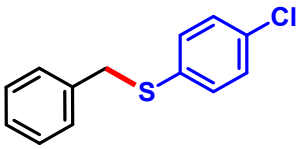
Isolated Yield: 64%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 5.1 Hz, 5H), 7.37 – 7.32 (m, 1H), 7.23 (d, *J* = 6.2 Hz, 3H), 4.20 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 138.76, 136.81, 134.62, 129.90, 128.92, 128.68, 127.49, 127.33, 126.33, 38.64

HRMS: [ESI, (+) ve]: Cald. 233.0192 for $C_{13}H_{10}ClS$ found 233.0196.

IR (thin film, cm^{-1}): 697, 763, 864, 909, 995, 1084, 1164, 1238, 1402, 1460, 1576, 1752, 1945, 2925.



benzyl(4-chlorophenyl)sulfane (3bg)

The title compound 3bg was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: White solid.

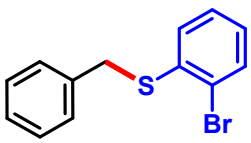
Isolated Yield: 60%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 5H), 7.24 (s, 4H), 4.11 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.20, 134.82, 132.53, 131.45, 129.05, 128.91, 128.66, 127.43, 39.35.

HRMS: [ESI, (+) ve]: Cald. 233.0192 for $C_{13}H_{10}ClS$ observed 233.0196.

IR (thin film, cm^{-1}): 745, 985, 1164, 1252, 1460, 1581, 1777, 1947, 2951.



benzyl(2-bromophenyl)sulfane (3bh)

The title compound 3bh was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

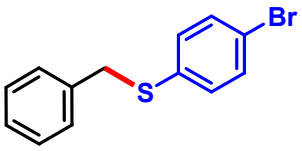
Isolated Yield: 59%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.54 (m, 1H), 7.37 (d, *J* = 7.0 Hz, 2H), 7.34 – 7.24 (m, 3H), 7.21 (dd, *J* = 6.4, 1.6 Hz, 2H), 7.03 (ddd, *J* = 8.5, 6.5, 2.5 Hz, 1H), 4.16 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.02, 136.26, 133.06, 129.10, 128.95, 128.75, 127.86, 127.58, 127.04, 123.78, 38.04.

HRMS: [ESI, (+) ve]: Cald. 277.9765 for $C_{13}H_{11}BrS$ observed 277.9768.

IR (thin film, cm^{-1}): 767, 921, 1110, 1217, 1452, 1517, 1890, 2923, 3029.



benzyl(4-bromophenyl)sulfane (3bi)

The title compound 3bi was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: White solid.

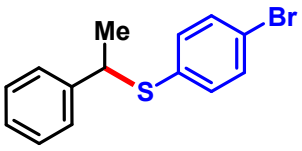
Isolated Yield: 58%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.5 Hz, 2H), 7.38 – 7.33 (m, 4H), 7.33 – 7.28 (m, 1H), 7.20 (d, *J* = 8.5 Hz, 2H), 4.13 (s, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 137.04, 135.57, 131.89, 131.35, 128.84, 128.61, 127.38, 120.29, 39.00.

HRMS: [ESI, (+) ve]: Cald. 277.9765 for $C_{13}H_{11}BrS$ observed 277.9762.

IR (thin film, cm^{-1}): 758, 908, 1215, 1334, 1472, 1563, 1886, 2923, 3029.



(4-bromophenyl)(1-phenylethyl)sulfane (3bj)

The title compound 3bj was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

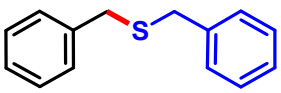
Isolated Yield: 64%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (td, *J* = 5.4, 2.4 Hz, 2H), 7.37 – 7.33 (m, 4H), 7.32 – 7.26 (m, 1H), 7.23 – 7.15 (m, 2H), 4.44 – 4.33 (m, 1H), 1.75 – 1.61 (m, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 142.94, 142.92, 134.39, 134.06, 131.86, 131.84, 128.59, 128.57, 127.42, 127.40, 127.37, 127.35, 121.42, 48.16, 22.36.

HRMS: [ESI, (+) ve]: Cald. 291.9921 for $C_{14}H_{13}BrS$ observed 291.9923.

IR (thin film, cm^{-1}): 698, 815, 1090, 1384, 1563, 1802, 1946, 2866, 3028.



dibenzylsulfane (3bk)

The title compound 3bk was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

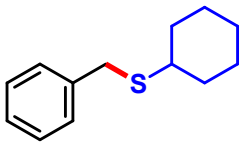
Isolated Yield: 61%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 10H), 3.65 (s, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 138.28, 129.14, 128.60, 127.10, 35.72.

GCMS (m/z): calcd. 214.0816 for $C_{14}H_{14}S$ found 214.3.

IR (thin film, cm^{-1}): 906, 1028, 1070, 1196, 1236, 1320, 1452, 1493, 1601, 1805, 1878, 1947, 2248, 2916, 3029.



benzyl(cyclohexyl)sulfane (3bl)

The title compound 3bl was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

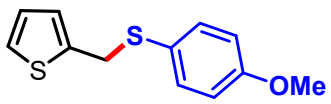
Isolated Yield: 32%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.27 (m, 4H), 7.25 – 7.17 (m, 1H), 3.75 (s, 2H), 2.56 (ddt, *J* = 10.5, 7.5, 3.7 Hz, 1H), 2.05 – 1.81 (m, 2H), 1.75 (td, *J* = 6.4, 3.5 Hz, 2H), 1.43 – 1.06 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.13, 128.92, 128.61, 126.93, 43.08, 34.76, 33.55, 26.17, 26.04.

GCMS (m/z): cald. 206.1129 for $C_{13}H_{18}S$ observed 206.1.

IR (thin film, cm^{-1}): 691, 886, 1090, 1263, 1339, 1583, 1877, 1945, 2852, 3059.



2-(((4-methoxyphenyl)thio)methyl)thiophene (3bm)

The title compound 3bm was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (98:2).

Physical State: Colourless liquid

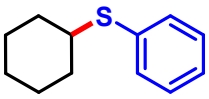
Isolated Yield: 67%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.28 (m, 3H), 6.82 (d, J = 8.6 Hz, 2H), 6.78 (d, J = 3.7 Hz, 1H), 6.48 (d, J = 3.7 Hz, 1H), 4.08 (s, 1H), 3.79 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.64, 143.40, 134.77, 134.58, 129.35, 126.39, 114.60, 111.15, 55.33, 36.03.

GCMS (m/z): cald. 236.0330 for $C_{12}H_{12}OS_2$ observed 236.0.

IR (thin film, cm^{-1}): 677, 878, 1071, 1250, 1341, 1574, 1874, 2895, 3066.



cyclohexyl(phenyl)sulfane (5aa)

The title compound 5aa was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

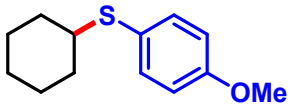
Isolated Yield: 47%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.38 (m, 2H), 7.29 (dd, J = 8.2, 6.6 Hz, 2H), 7.25 – 7.19 (m, 1H), 3.24 – 3.02 (m, 1H), 2.07 – 1.93 (m, 2H), 1.84 – 1.72 (m, 2H), 1.70 – 1.55 (m, 1H), 1.50 – 1.15 (m, 5H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 135.30, 131.99, 128.88, 126.71, 46.69, 33.48, 26.20, 25.91.

GCMS (m/z): calcd. 192.0973 for $C_{12}H_{16}S$ found 192.0.

IR (thin film, cm^{-1}): 691, 886, 1090, 1263, 1339, 1583, 1877, 1945, 2852, 3059.



cyclohexyl(4-methoxyphenyl)sulfane (5ab)

The title compound 5ab was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

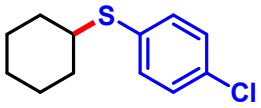
Isolated Yield: 51%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.7 Hz, 2H), 3.80 (s, 3H), 2.99 – 2.81 (m, 1H), 1.92 (d, *J* = 11.3 Hz, 2H), 1.82 – 1.69 (m, 2H), 1.35 – 1.19 (m, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.48, 135.77, 125.15, 114.46, 55.49, 48.10, 33.55, 26.30, 25.95.

HRMS: [ESI, (+) ve]: Cald 222.1078 for $C_{13}H_{18}OS$ observed 221.1003.

IR (thin film, cm^{-1}): 677, 749, 1051, 1107, 1217, 1584, 2024, 2815, 3100.



(4-chlorophenyl)(cyclohexyl)sulfane (5ac)

The title compound 5ac was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

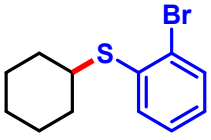
Isolated Yield: 45%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.32 (m, 2H), 7.28 (d, *J* = 1.1 Hz, 2H), 3.29 – 2.87 (m, 1H), 1.98 (ddt, *J* = 9.1, 3.6, 2.0 Hz, 2H), 1.89 – 1.73 (m, 2H), 1.52 – 1.12 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 133.44, 129.53, 129.50, 129.07, 47.08, 33.42, 26.20, 25.88.

GCMS (m/z): calcd. 226.0583 for $C_{12}H_{15}ClS$ found 226.0.

IR (thin film, cm^{-1}): 668, 762, 928, 1012, 1095, 1214, 1475, 2401, 2855, 2932, 3020.



(2-bromophenyl)(cyclohexyl)sulfane (5ad)

The title compound 5ad was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

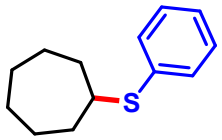
Isolated Yield: 43%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 8.0, 1.4 Hz, 1H), 7.36 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.26 – 7.21 (m, 1H), 7.04 (ddd, *J* = 7.9, 7.3, 1.6 Hz, 1H), 3.38 – 3.15 (m, 1H), 2.14 – 1.93 (m, 2H), 1.90 – 1.74 (m, 2H), 1.52 – 1.16 (m, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 133.35, 131.05, 128.91, 127.86, 127.73, 127.31, 45.79, 33.13, 26.19, 25.94.

HRMS: [ESI, (+) ve]: Cald. 269.0000 for $C_{12}H_{14}BrS$ observed 270.9957.

IR (thin film, cm^{-1}): 670, 777, 974, 1254, 1457, 2499, 2847, 3027.



cycloheptyl(phenyl)sulfane (5ae)

The title compound 5ae was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

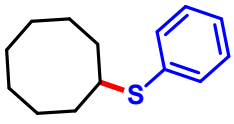
Isolated Yield: 49%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 7.5, 1.7 Hz, 2H), 7.32 (dd, *J* = 8.4, 6.8 Hz, 2H), 7.27 – 7.20 (m, 1H), 3.38 (tt, *J* = 9.0, 4.2 Hz, 1H), 2.19 – 1.98 (m, 2H), 1.85 – 1.71 (m, 2H), 1.72 – 1.58 (m, 6H), 1.56 – 1.45 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.37, 131.29, 128.90, 126.43, 48.06, 34.77, 28.38, 26.08.

GCMS (m/z): calcd. 206.1129 for $C_{13}H_{18}S$ found 206.1.

IR (thin film, cm^{-1}): 751, 886, 1090, 1263, 1341, 1882, 1950, 2872, 3061.



cyclooctyl(phenyl)sulfane (5af)

The title compound 5af was prepared following the general procedure 4.

Eluent: petroleum ether (100%).

Physical State: Colourless liquid

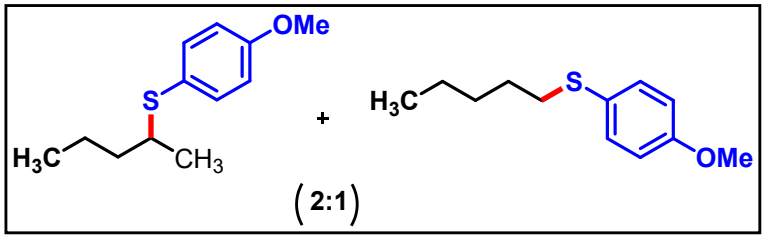
Isolated Yield: 51%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.36 (m, 2H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.16 (m, 1H), 3.41 (tt, *J* = 8.9, 3.8 Hz, 1H), 1.98 (ddt, *J* = 15.0, 8.4, 3.4 Hz, 2H), 1.82 – 1.73 (m, 2H), 1.73 – 1.65 (m, 2H), 1.64 – 1.46 (m, 8H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 136.28, 131.51, 128.93, 126.52, 47.79, 32.10, 27.27, 25.99, 25.31.

GCMS (m/z): calcd. 220.1286 for $C_{14}H_{20}S$ found 220.1.

IR (thin film, cm^{-1}): 753, 887, 1091, 1271, 1352, 1891, 1970, 2901, 3066.



(4-methoxyphenyl)(pentan-2-yl)sulfane + (4-methoxyphenyl)(pentyl)sulfane (2:1) (5ag)

The title compound 5ag was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

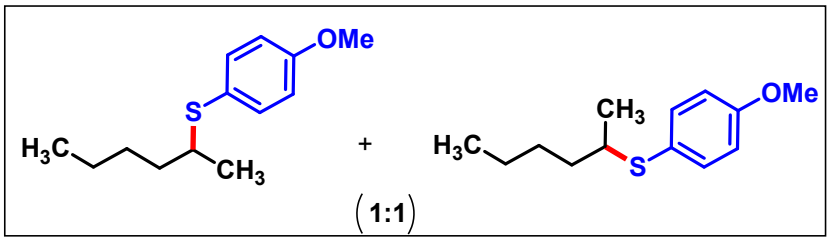
Isolated Yield: 45%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, J = 8.8 Hz, 2H), 6.84 (d, J = 8.8 Hz, 2H), 3.80 (s, 3H), 3.01 (m, 0.74H), 2.78(t, 0.33H), 1.54 – 1.38 (m, 5H), 1.21 (d, J = 6.7 Hz, 2H), 1.00 (t, J = 7.4 Hz, 1H), 0.90 (t, J = 7.1 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 159.49, 159.37, 135.81, 135.67, 125.41, 115.19, 114.48, 55.49, 44.54, 38.92, 26.73, 21.27, 20.43, 14.10, 11.40.

HRMS: [ESI, (+) ve]: Cald. 210.1078 for $C_{12}H_{18}OS$ observed 210.1062.

IR (thin film, cm^{-1}): 668, 751, 1035, 1102, 1214, 1478, 1592, 2043, 2872, 2961, 3136.



hexan-2-yl(4-methoxyphenyl)sulfane:hexan-2-yl(4-methoxyphenyl)sulfane (1:1) (5ah)

The title compound 5ah was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (99:1).

Physical State: Colourless liquid

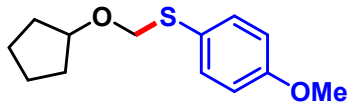
Isolated Yield: 43%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 3.4 Hz, 1H), 7.36 (d, *J* = 3.4 Hz, 1H), 6.84 (d, *J* = 3.4 Hz, 1H), 6.82 (d, *J* = 3.3 Hz, 1H), 3.80 (d, *J* = 1.3 Hz, 3H), 3.09 – 2.93 (m, 0.5H), 2.88 – 2.71 (m, 0.5H), 1.48 (tt, *J* = 4.5, 1.3 Hz, 1H), 1.43 – 1.36 (m, 2H), 1.33 – 1.23 (m, 3H), 1.21 (d, *J* = 6.7 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 1H), 0.89 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.50, 159.39, 135.80, 135.69, 125.69, 125.47, 114.49, 55.49, 52.00, 44.82, 36.45, 36.25, 29.44, 27.28, 22.76, 21.30, 20.23, 14.21, 14.18, 11.29.

HRMS: [ESI, (+) ve]: cald. 223.1157 for $C_{13}H_{20}OS$ observed 223.1141.

IR (thin film, cm^{-1}): 679, 1041, 1275, 1471, 1587, 2015, 2961, 3130.



((cyclopentyloxy)methyl)(4-methoxyphenyl)sulfane (5ai)

The title compound 5ai was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (98:2).

Physical State: Colourless liquid

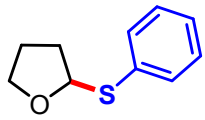
Isolated Yield: 65%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.9 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 4.84 (s, 1H), 4.42 – 4.29 (m, 1H), 3.79 (s, 3H), 1.82 – 1.60 (m, 6H), 1.57 – 1.50 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.36, 133.76, 126.44, 114.65, 78.87, 75.42, 55.48, 32.19, 23.70.

HRMS: [ESI, (+) ve]: cald. $[M + H]^+$: 239.1100 for $C_{13}H_{19}O_2S$ Found 239.1098

IR (thin film, cm^{-1}): 698, 749, 1051, 1107, 1217, 1584, 2024, 2815, 3100.



2-(phenylthio)tetrahydrofuran (5aj)

The title compound 5aj was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

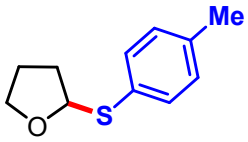
Isolated Yield: 65%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 – 7.49 (m, 2H), 7.36 – 7.28 (m, 2H), 7.27 – 7.20 (m, 1H), 5.66 (dd, *J* = 7.2, 3.8 Hz, 1H), 4.09 – 3.88 (m, 2H), 2.37 (dtd, *J* = 12.2, 7.3, 4.7 Hz, 1H), 2.09 – 1.82 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 135.79, 131.08, 128.87, 126.83, 87.18, 67.32, 32.71, 24.92.

HR-ESI-MS (m/z): calcd. for $C_{10}H_{12}OSNa$ $[M + Na]^+$: 203.0501, found 203.0502.

IR (thin film, cm^{-1}): 698, 750, 1032, 1177, 1235, 1575, 2024, 2817, 3117.



2-(*p*-tolylthio)tetrahydrofuran (5ak)

The title compound 5ak was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

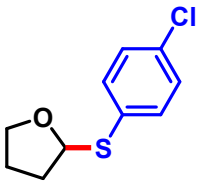
Physical State: Colourless liquid

Isolated Yield: 68%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.31 (m, 2H), 7.11 (dd, *J* = 8.2, 3.1 Hz, 2H), 5.57 (dd, *J* = 7.2, 3.8 Hz, 1H), 4.06 – 3.90 (m, 2H), 2.39 – 2.35 (m, 1H), 2.32 (s, 3H), 2.07 – 1.79 (m, 3H).

HR-ESI-MS (m/z): calcd. for $C_{11}H_{14}OSNa$ $[M + Na]^+$: 217.0658, found 217.0663

IR (thin film, cm^{-1}): 691, 732, 1025, 1171, 1555, 2014, 2857, 3129.



2-((4-chlororophenyl)thio)tetrahydrofuran (5al)

The title compound 5al was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

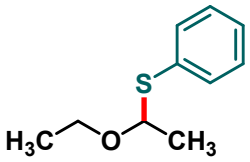
Isolated Yield: 61%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.6 Hz, 2H), 7.25 (d, *J* = 8.6 Hz, 2H), 5.60 (dd, *J* = 7.3, 3.8 Hz, 1H), 4.17 – 3.69 (m, 2H), 2.35 (ddt, *J* = 12.1, 6.9, 3.1 Hz, 1H), 2.13 – 1.78 (m, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 134.38, 133.00, 132.48, 129.00, 87.32, 67.38, 32.69, 24.91.

HR-ESI-MS (m/z): calcd. for $C_{10}H_{11}ClOSNa$ $[M + Na]^+$: 237.0111, found 237.0109.

IR (thin film, cm^{-1}): 671, 735, 1023, 1175, 1246, 1575, 2021, 2845, 3121.



(1-ethoxyethyl)(phenyl)sulfane (5am)

The title compound 5am was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

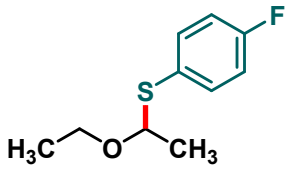
Isolated Yield: 73%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 7.9, 1.8 Hz, 2H), 7.34 – 7.25 (m, 3H), 4.90 (q, *J* = 6.3 Hz, 1H), 4.01 – 3.91 (m, 1H), 3.56 – 3.45 (m, 1H), 1.51 (d, *J* = 6.2 Hz, 3H), 1.23 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 133.86, 133.01, 128.71, 127.54, 84.54, 63.39, 22.71, 14.94.

HR-ESI-MS (m/z): calcd 182.0765 for $C_9H_{12}OS$ $[M]^+$: found 182.0764.

IR (thin film, cm^{-1}): 694, 798, 1054, 1178, 1541, 2017, 2861, 3133.



(1-ethoxyethyl)(4-fluorophenyl)sulfane (5an)

The title compound 5an was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

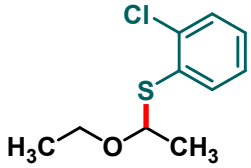
Isolated Yield: 71%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (dd, *J* = 8.7, 5.5 Hz, 2H), 7.00 (t, *J* = 8.7 Hz, 2H), 4.80 (q, *J* = 6.2 Hz, 1H), 3.95 (dq, *J* = 9.3, 7.1 Hz, 1H), 3.48 (dq, *J* = 9.3, 7.0 Hz, 1H), 1.45 (d, *J* = 6.4 Hz, 3H), 1.22 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 163.85, 161.88, 136.59, 136.52, 127.66, 127.63, 115.97, 115.80, 84.59, 63.56, 22.66, 15.00.

HRMS: [ESI, (+) ve]: calcd for 200.0671 for $[M]^+$: $C_{10}H_{13}FOS$ found 200.0669.

IR (thin film, cm^{-1}): 678, 741, 1059, 1157, 1541, 2022, 2897, 3152



(1-ethoxyethyl)(2-chlorophenyl)sulfane (5ao)

The title compound 5ao was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

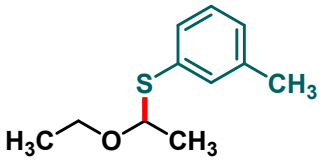
Isolated Yield: 68%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 – 7.57 (m, 1H), 7.40 (dd, *J* = 7.1, 2.3 Hz, 1H), 7.24 – 7.10 (m, 2H), 5.08 – 5.03 (m, 1H), 3.98 – 3.81 (m, 1H), 3.64 – 3.42 (m, 1H), 1.57 (dd, *J* = 6.4, 0.8 Hz, 3H), 1.20 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 136.81, 134.52, 133.32, 129.91, 128.46, 127.07, 84.43, 63.12, 22.41, 15.08.

HRMS: [ESI, (+) ve]: calcd. 239.0268 for $C_{10}H_{13}ClOSNa$ $[M + Na]^+$ found 239.0256.

IR (thin film, cm^{-1}): 681, 733, 1063, 1175, 1526, 2027, 2888, 3175



(1-ethoxyethyl)(*m*-tolyl)sulfane (5ap)

The title compound 5ap was prepared following the general procedure 4.

Eluent: petroleum ether:ethyl acetate (100:0).

Physical State: Colourless liquid

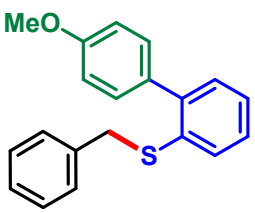
Isolated Yield: 74%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (d, *J* = 8.1 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.10 (d, *J* = 7.6 Hz, 1H), 4.91 (q, *J* = 6.3 Hz, 1H), 3.98 (dq, *J* = 9.2, 7.1 Hz, 1H), 3.52 (dq, *J* = 9.3, 7.0 Hz, 1H), 2.35 (s, 3H), 1.54 (d, *J* = 6.3 Hz, 3H), 1.26 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 138.39, 134.44, 132.83, 130.83, 128.56, 128.39, 84.55, 63.35, 22.79, 21.35, 14.99.

HRMS: [ESI, (+) ve]: calcd. 197.0989 for $C_{11}H17OS$ for $[M+H]^+$ found 197.0987

IR (thin film, cm^{-1}): 687, 729, 1052, 1167, 1523, 2019, 2879, 3154



benzyl(4'-methoxy-[1,1'-biphenyl]-2-yl)sulfane (7)

The title compound 7 was prepared according to the literature procedure.[1]

Eluent: petroleum ether:ethyl acetate (98:2).

Physical State: White solid.

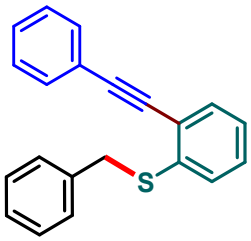
Isolated Yield: 92%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.38 (m, 1H), 7.36 (d, *J* = 8.6 Hz, 2H), 7.30 – 7.19 (m, 8H), 6.97 (d, *J* = 8.7 Hz, 2H), 3.97 (s, 2H), 3.86 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 159.10, 142.17, 137.14, 135.52, 133.19, 130.69, 130.54, 129.04, 128.90, 128.55, 127.87, 127.67, 127.24, 126.07, 114.31, 113.57, 55.38, 38.53.

HRMS: [ESI, (+) ve]: Calcd. 306.1078 for $C_{20}H_{18}OS$ found 306.1082.

IR (thin film, cm^{-1}): 732, 827, 905, 1031, 1102., 1174, 1244, 1285, 1460, 1492, 1592, 1677, 1885, 2044, 2251, 2963, 3028.



benzyl(2-(phenylethynyl)phenyl)sulfane (8)

The title compound 8 was prepared according to the literature procedure.[2]

Eluent: petroleum ether (100%).

Physical State: Colourless liquid.

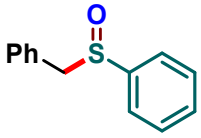
Isolated Yield: 70%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.54 (m, 2H), 7.41 – 7.33 (m, 5H), 7.33 – 7.27 (m, 4H), 7.25 – 7.15 (m, 3H), 4.23 (s, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.68, 137.00, 132.77, 131.82, 129.11, 128.79, 128.69, 128.60, 128.50, 128.37, 128.04, 127.41, 125.70, 123.35, 95.67, 87.42, 37.71.

HRMS: [ESI, (+) ve]: calcd. 300.0973 for $C_{21}H_{16}S$ observed 300.0957.

IR (thin film, cm^{-1}): 2943, 2210, 1617, 1463, 1323, 1125, 1064, 842.



(benzylsulfinyl)benzene (9)

The title compound 9 was prepared according to the literature procedure.[3]

Eluent: petroleum ether:ethyl acetate (60:40).

Physical State: White solid

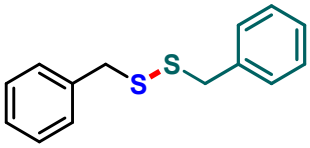
Isolated Yield: 85%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.30 (m, 5H), 7.28 – 7.11 (m, 3H), 6.95 (dd, *J* = 8.0, 1.7 Hz, 2H), 4.22 – 3.74 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.75, 131.16, 130.36, 129.14, 128.85, 128.43, 128.23, 124.41, 63.51.

GCMS (m/z): Calcd. 216.0609 for $C_{13}H_{12}OS$ found 216.2.

IR (thin film, cm^{-1}): 775, 930, 1072, 1247, 1372, 2876.



1,2-dibenzyldisulfane (10)

The title compound 10 was prepared according to the literature procedure.[4]

Eluent: petroleum ether (100%).

Physical State: White solid

Isolated Yield: 25%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 6H), 7.27 – 7.23 (m, 4H), 3.62 (s, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.53, 129.59, 128.65, 127.60, 43.43.

GCMS (m/z): Calcd. 246.0537 for $C_{14}H_{14}S_2$ found 246.3.

IR (thin film, cm^{-1}): 755, 918, 1082, 1227, 2832,2875.

7. References

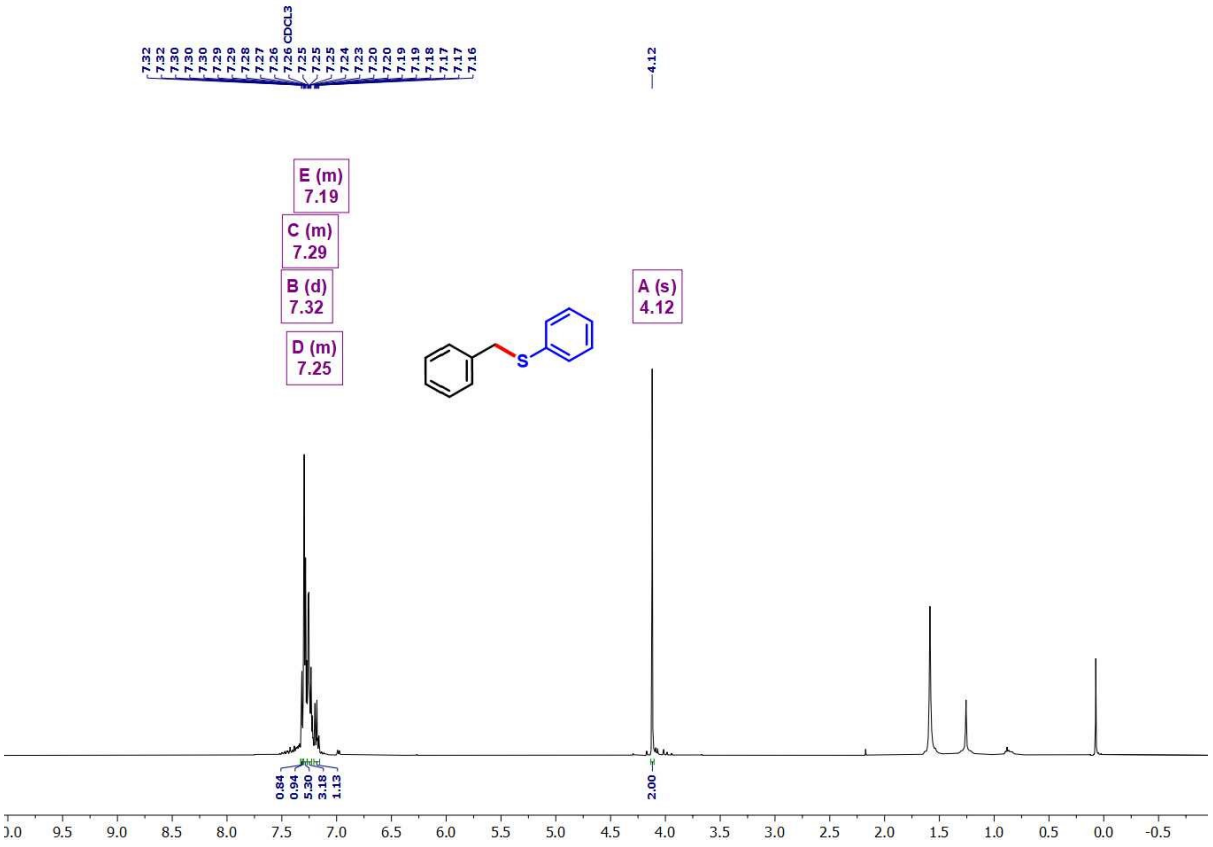
1. Bag, S.; Patra, T.; Modak, A.; Deb, A.; Maity, S.; Dutta, U.; Dey, A.; Kancherla, R.; Maji, A.; Hazra, A.; Bera, M.; Maiti, D. Remote Para-C–H Functionalization of Arenes by a D-Shaped Biphenyl Template-Based Assembly. *J. Am. Chem. Soc.* 2015, *137* (37), 11888–11891.

2. Liu, M.; Sun, J.; Erbay, T. G.; Ni, H.-Q.; Martín-Montero, R.; Liu, P.; Engle, K. M. PdII-Catalyzed C(Alkenyl)−H Activation Facilitated by a Transient Directing Group**. *Angewandte Chemie International Edition* 2022, *61* (25), e202203624.

3. Pospíšil, J.; Markó, I. E. Total Synthesis of Jerangolid D. *J. Am. Chem. Soc.* 2007, *129* (12), 3516–3517.

4. Seguchi, K.; Hirota, S. Photo-oxidation of aryl ethers in the presence of ferric chloride. *Chem. Lett.* 1982, *11* (3), 385–386.

NMR Spectra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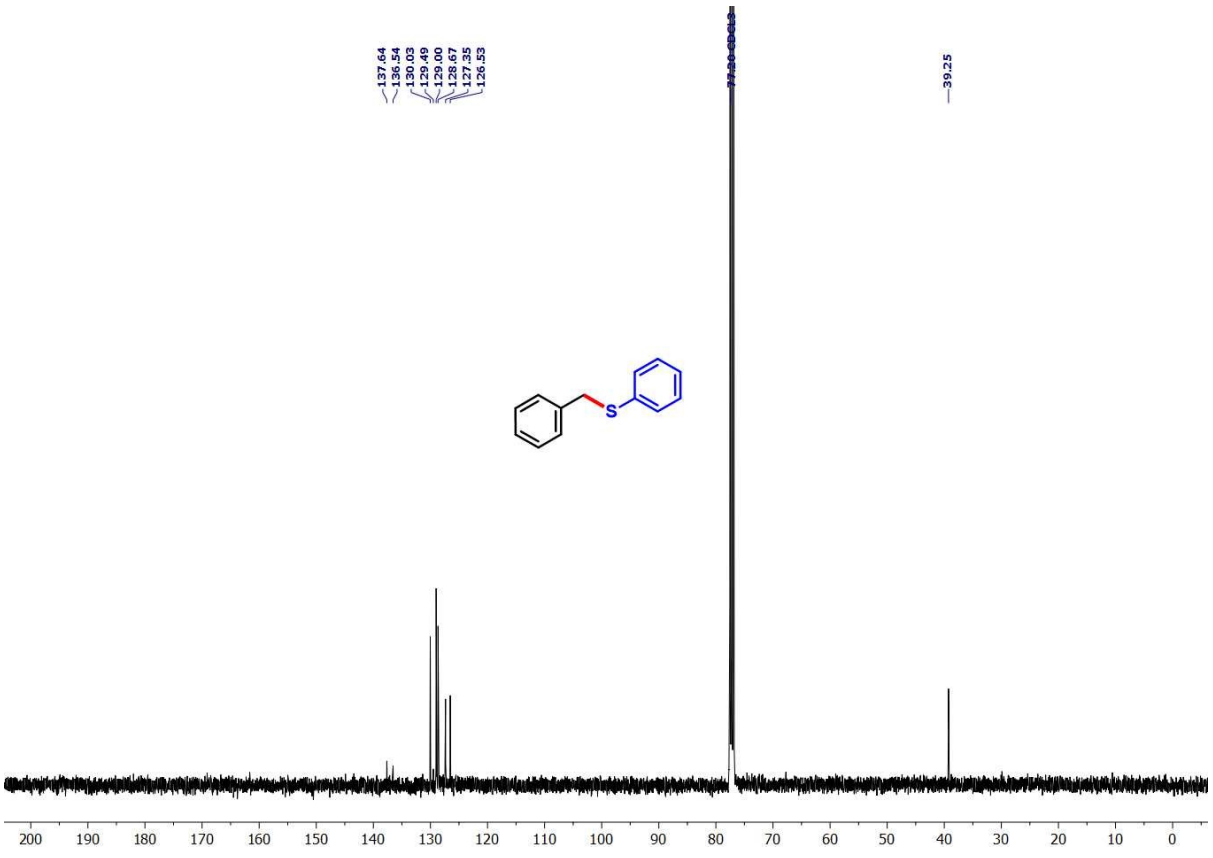
7.30
7.30
7.29
7.29
7.28
7.27
7.26
7.26
7.25
7.25
7.25
7.24
7.23
7.20
7.20
7.19
7.19
7.18
7.17
7.17
7.17
7.16
4.12
E (m)
7.19
C (m)
7.29
B (d)
7.32
D (m)
7.25
A (s)
4.12
S
0.84
0.94
5.30
3.18
1.13
2.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5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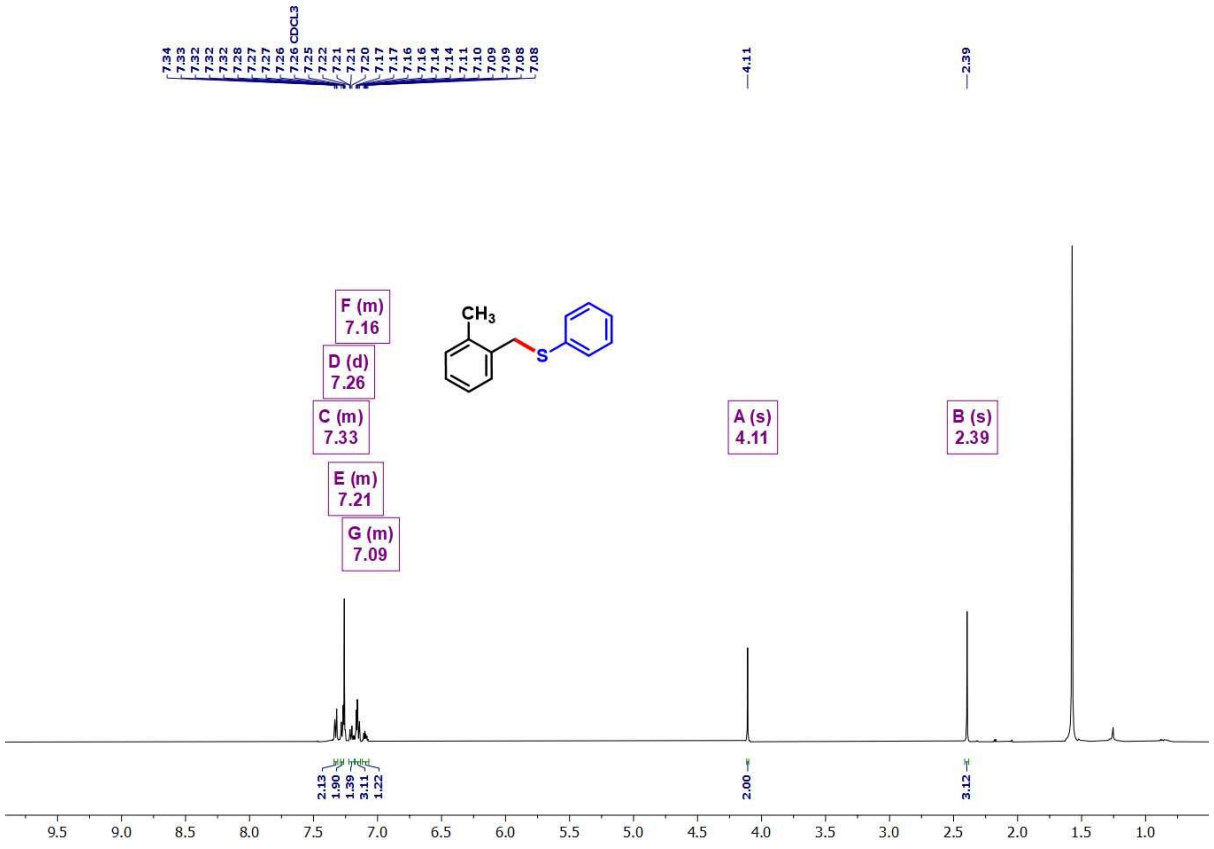
136.54
130.03
129.49
129.00
128.67
127.35
126.53
77.20 CDCL3
39.25
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.33
7.32
7.32
7.32
7.28
7.27
7.27
7.26
CDCL3
7.26
7.25
7.22
7.21
7.21
7.20
7.17
7.17
7.16
7.16
7.14
7.14
7.11
7.10
7.09
7.09
7.08
7.08
4.11
2.39
F (m)
7.16
D (d)
7.26
C (m)
7.33
E (m)
7.21
G (m)
7.09
CH3
S
A (s)
4.11
B (s)
2.39
2.13
1.90
1.39
3.11
1.22
2.00
3.12
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
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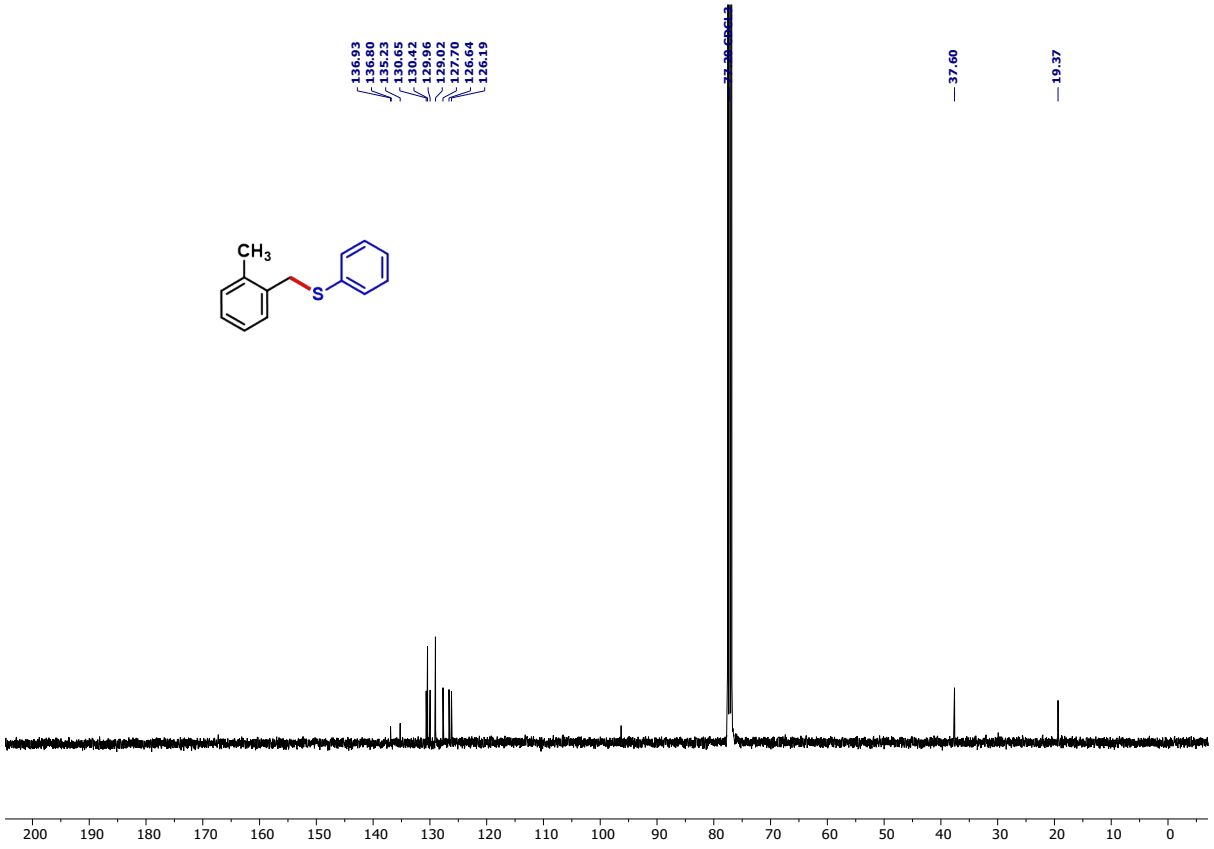
1.0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130.65
130.42
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129.02
127.70
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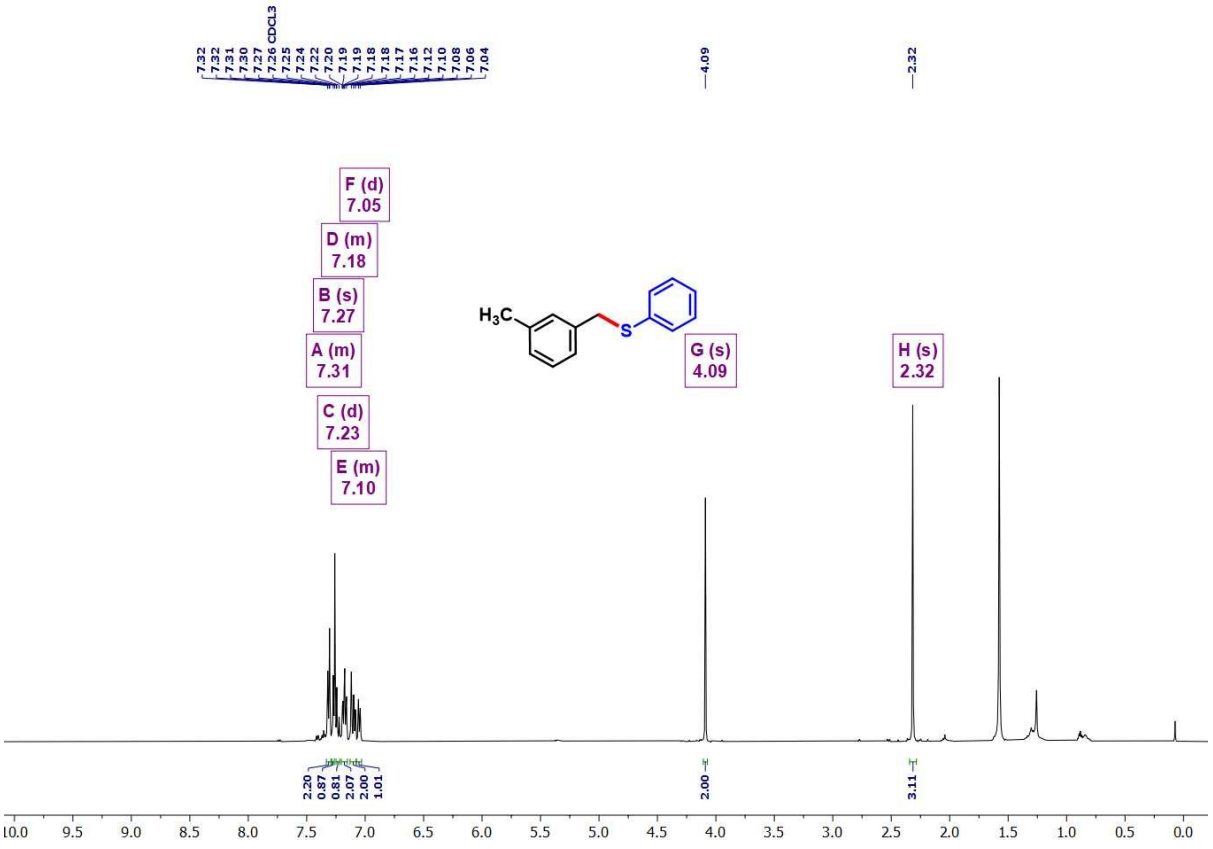
126.19
77.36 CDCL3
37.60
19.37
CH3
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.32
7.31
7.30
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7.25
7.24
7.22
7.20
7.19
7.19
7.18
7.18
7.17
7.16
7.12
7.10
7.08
7.06
7.04
4.09
2.32
F (d)
7.05
D (m)
7.18
B (s)
7.27
A (m)
7.31
C (d)
7.23
E (m)
7.10
G (s)
4.09
H (s)
2.32
H3C
S
2.20
0.87
0.81
2.07
2.00
1.01
2.00
3.11
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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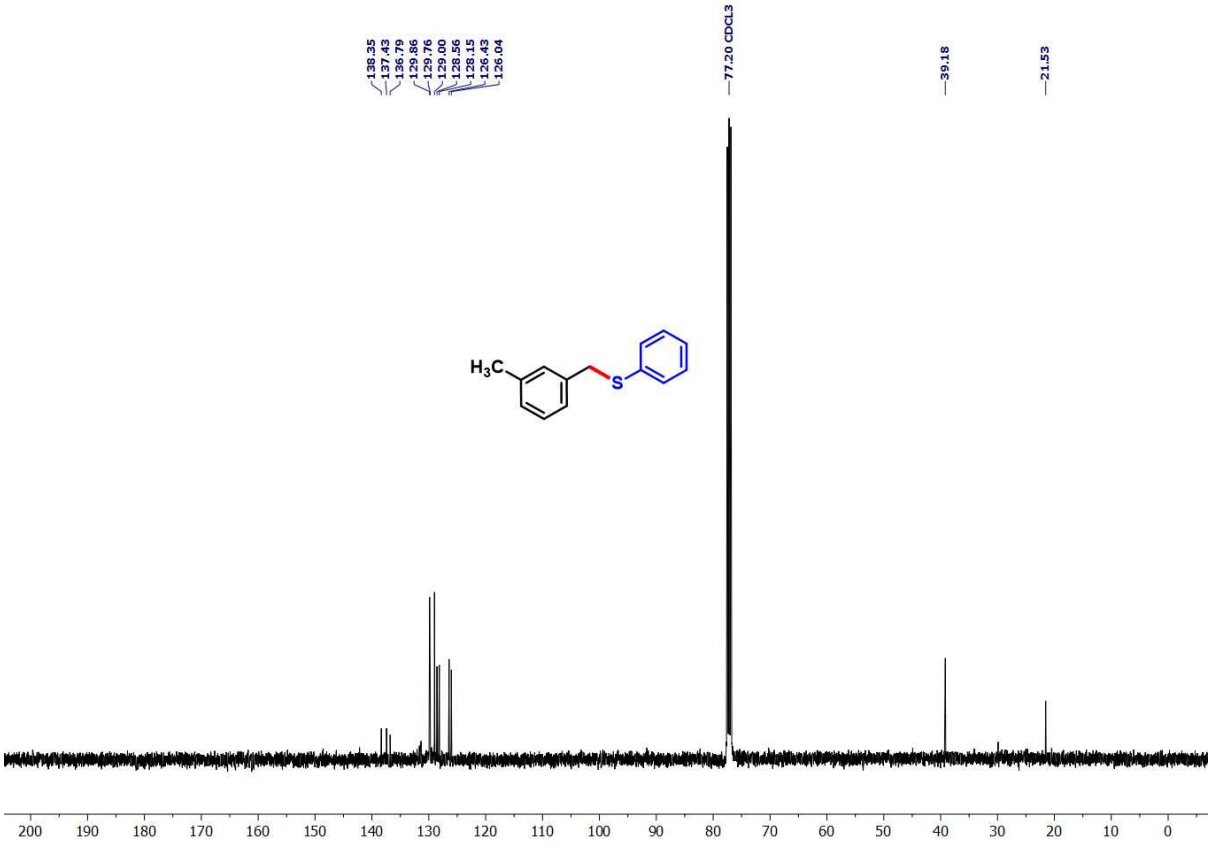
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128.15
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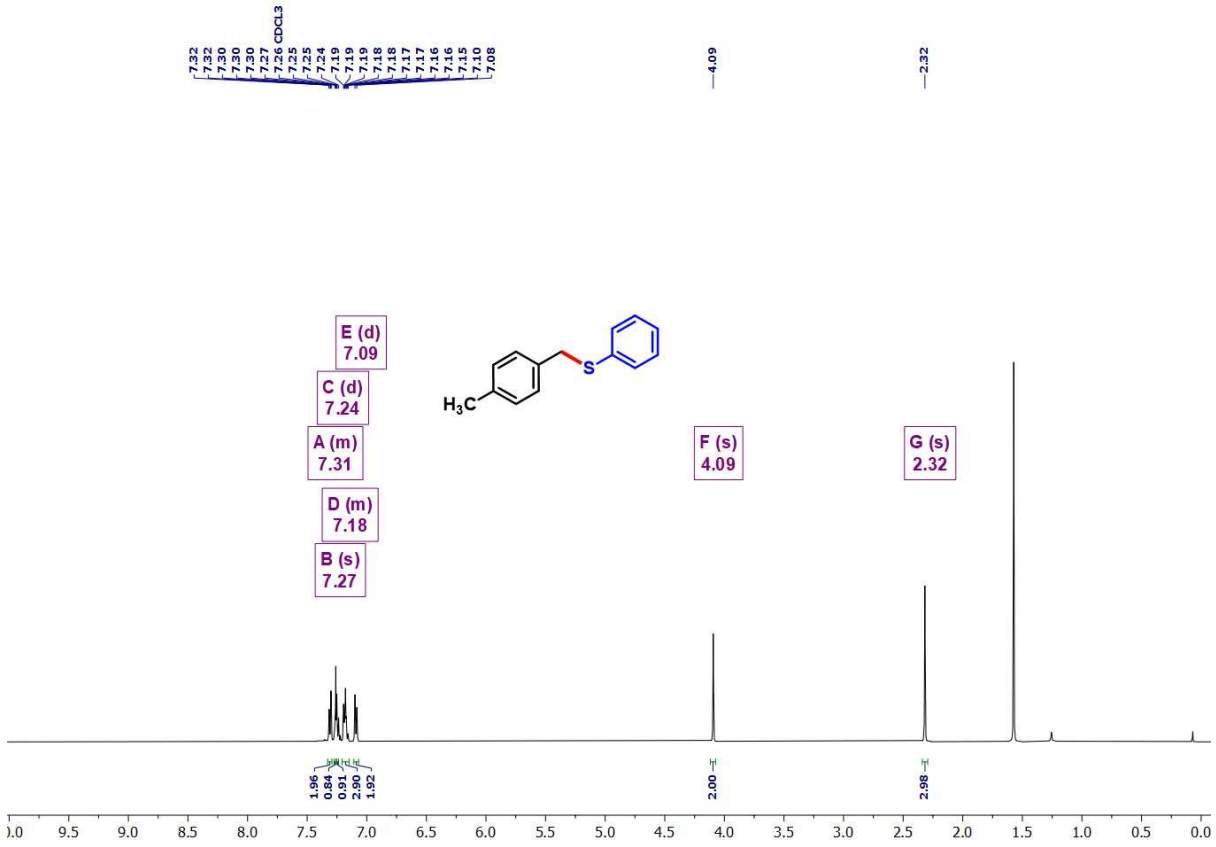
126.04
77.20 CDCl3
39.18
21.53
H3C
S
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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7.32
7.30
7.30
7.30
7.27
7.26 CDCL3
7.25
7.25
7.24
7.19
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7.19
7.18
7.18
7.17
7.17
7.16
7.16
7.15
7.10
7.08
4.09
2.32
E (d)
7.09
C (d)
7.24
A (m)
7.31
D (m)
7.18
B (s)
7.27
F (s)
4.09
G (s)
2.32
H3C
S
1.96
0.84
0.91
2.90
1.92
2.00
2.98
9.5
9.0
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8.0
7.5
7.0
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6.0
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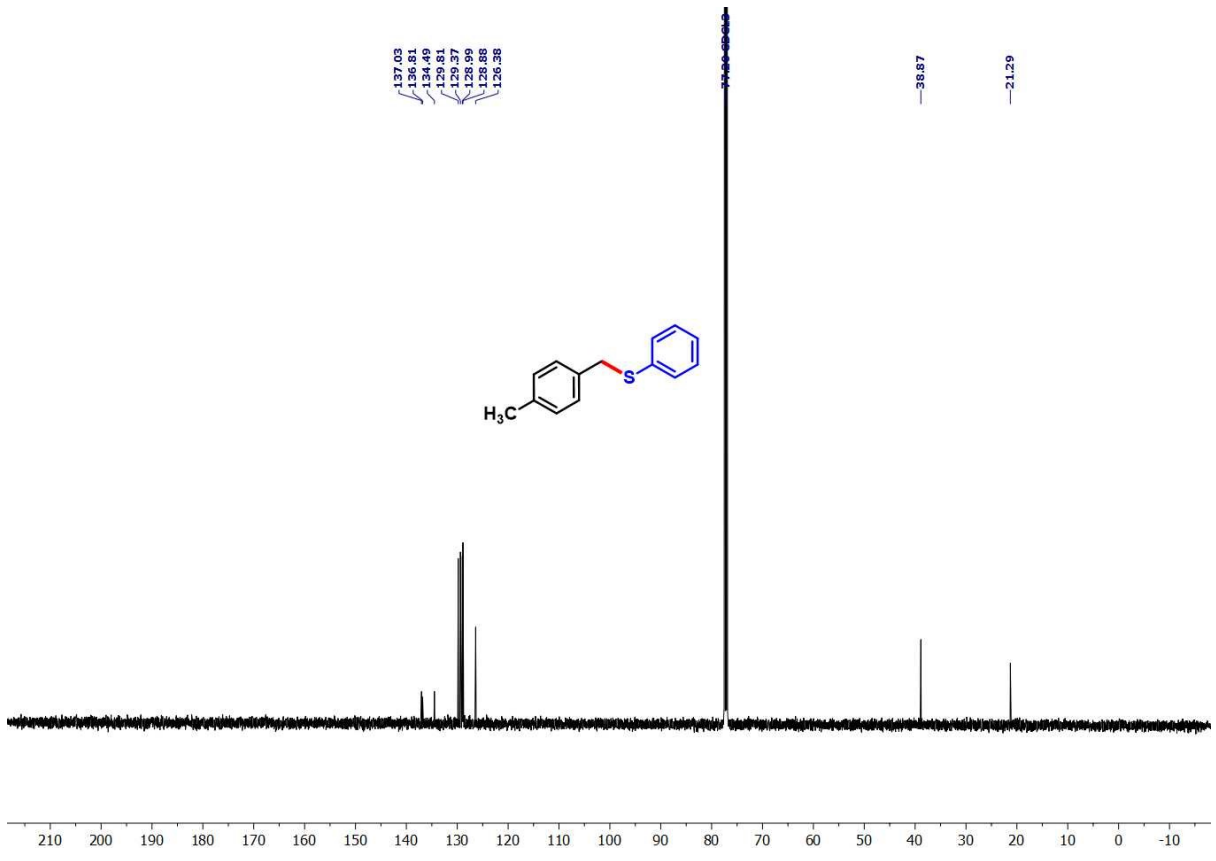
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H3C
S
210
200
190
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50
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30
20
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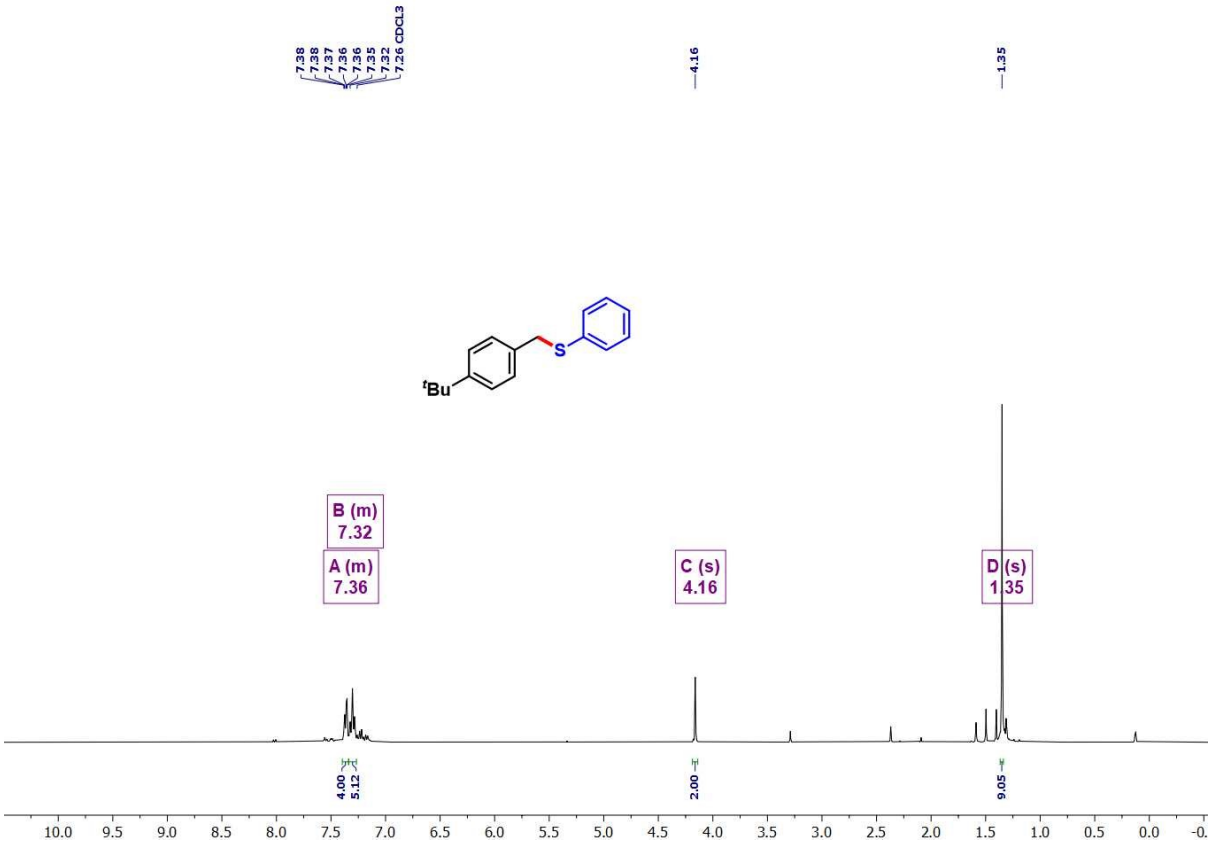
-10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7.38
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7.35
7.32
7.26 CDCL3
4.16
1.35
tBu
S
B (m)
7.32
A (m)
7.36
C (s)
4.16
D (s)
1.35
4.00
5.12
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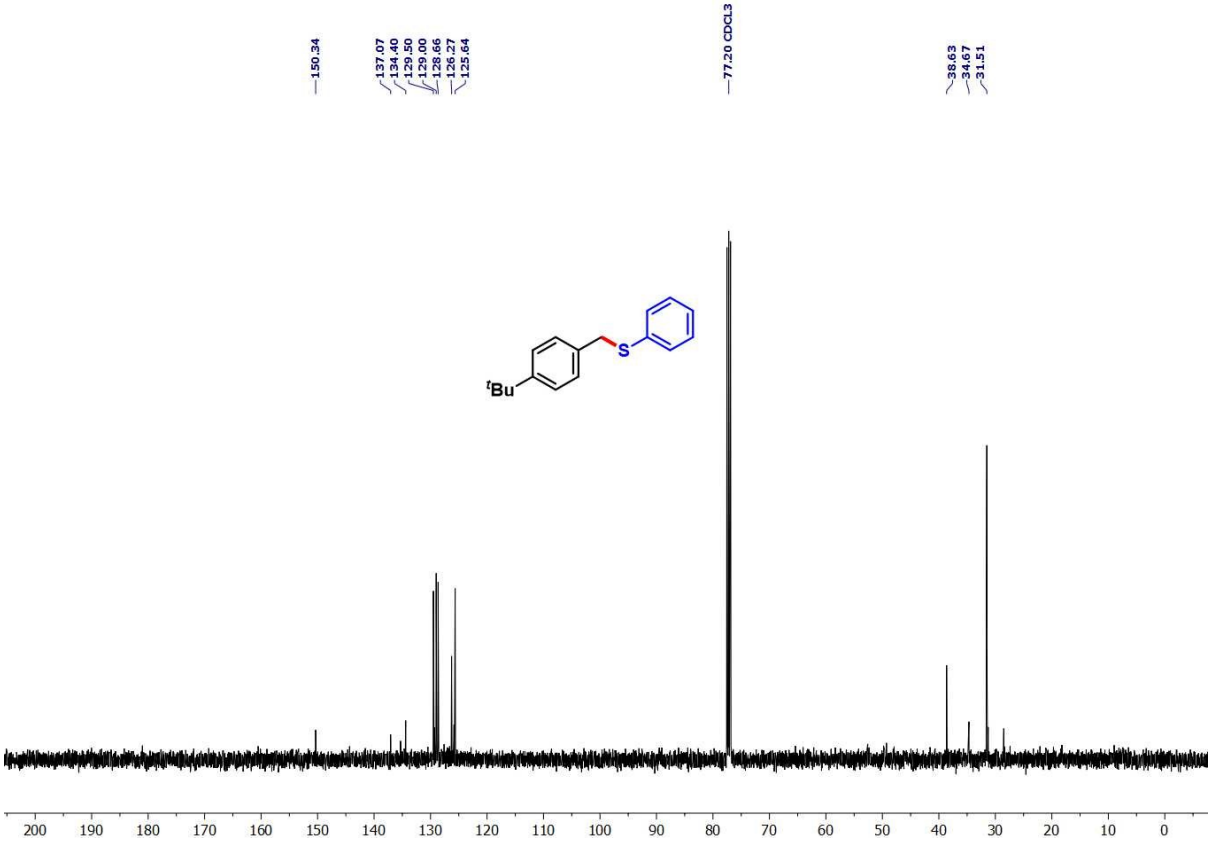
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tBu
S
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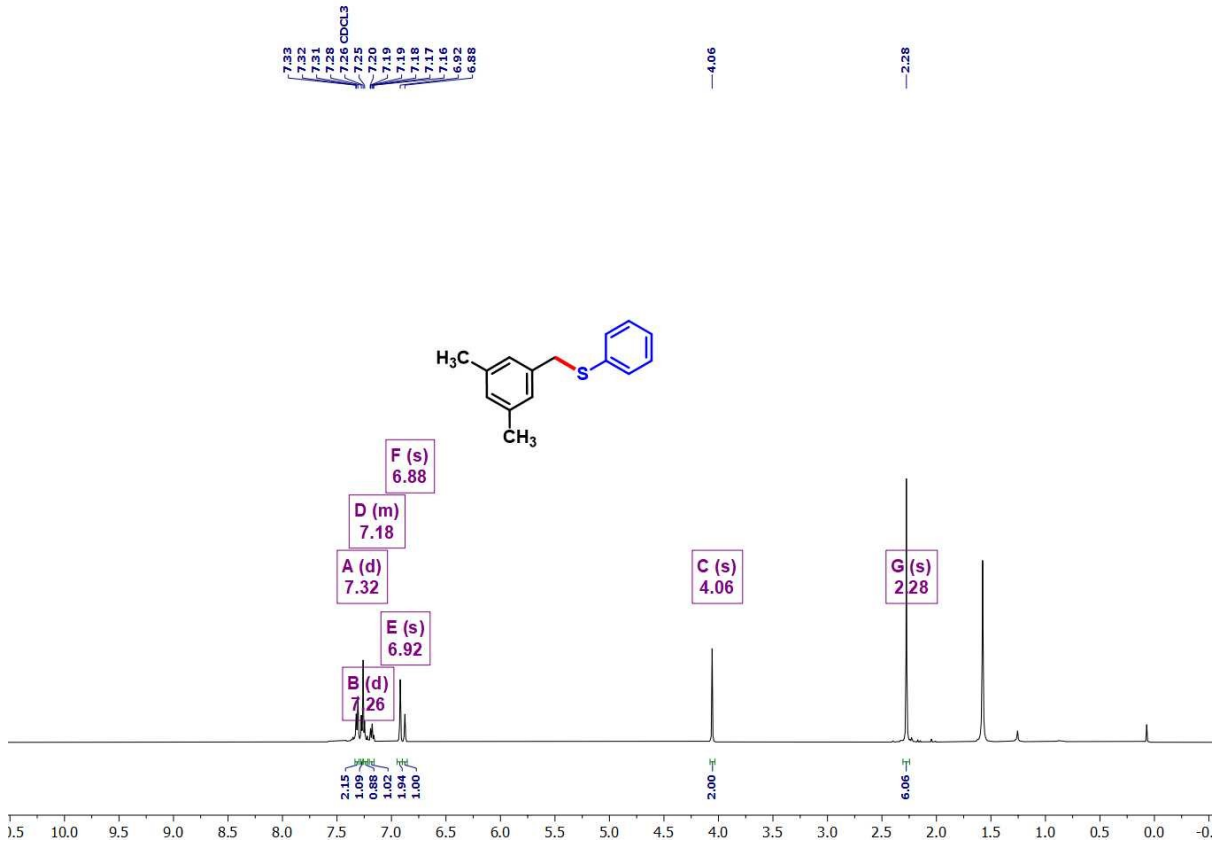
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7.17
7.16
6.92
6.88
4.06
2.28
H3C
S
CH3
F (s)
6.88
D (m)
7.18
A (d)
7.32
C (s)
4.06
G (s)
2.28
E (s)
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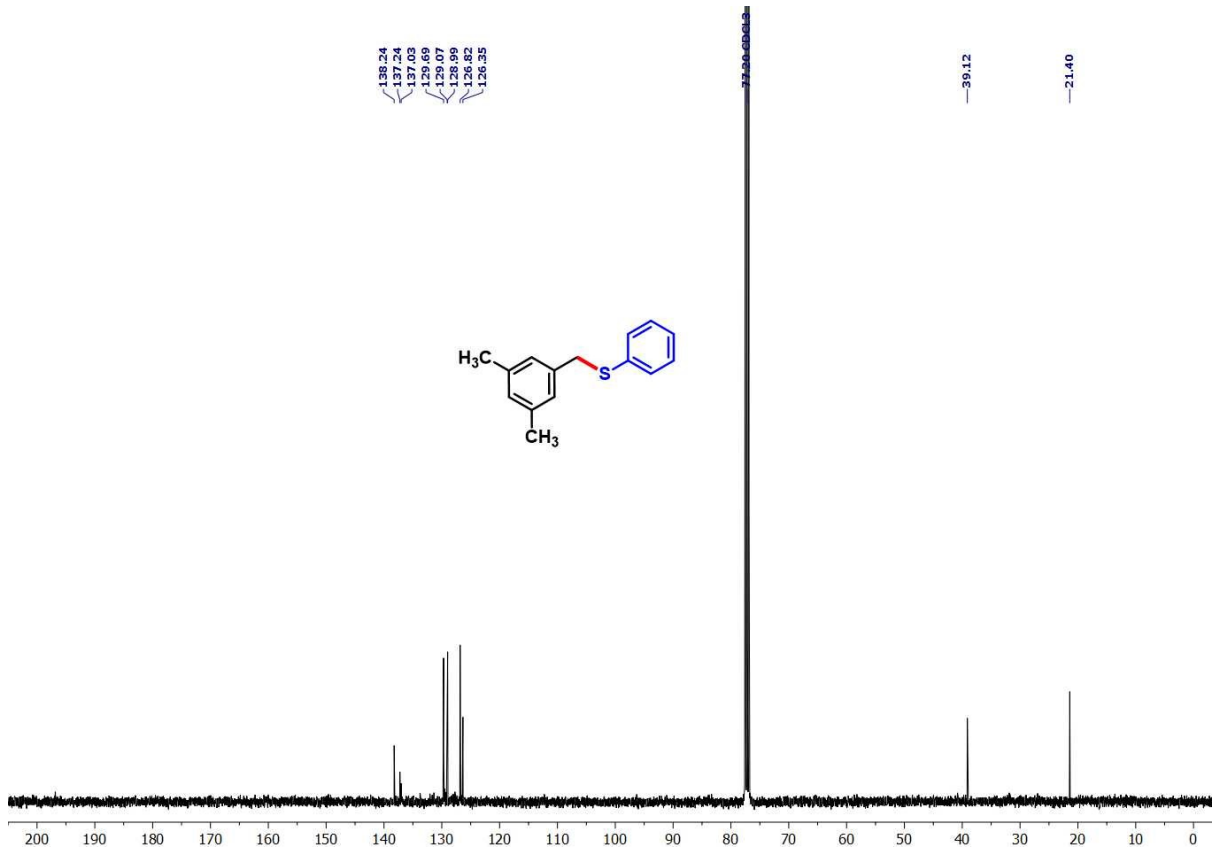
1.02
1.94
1.00
2.00
6.06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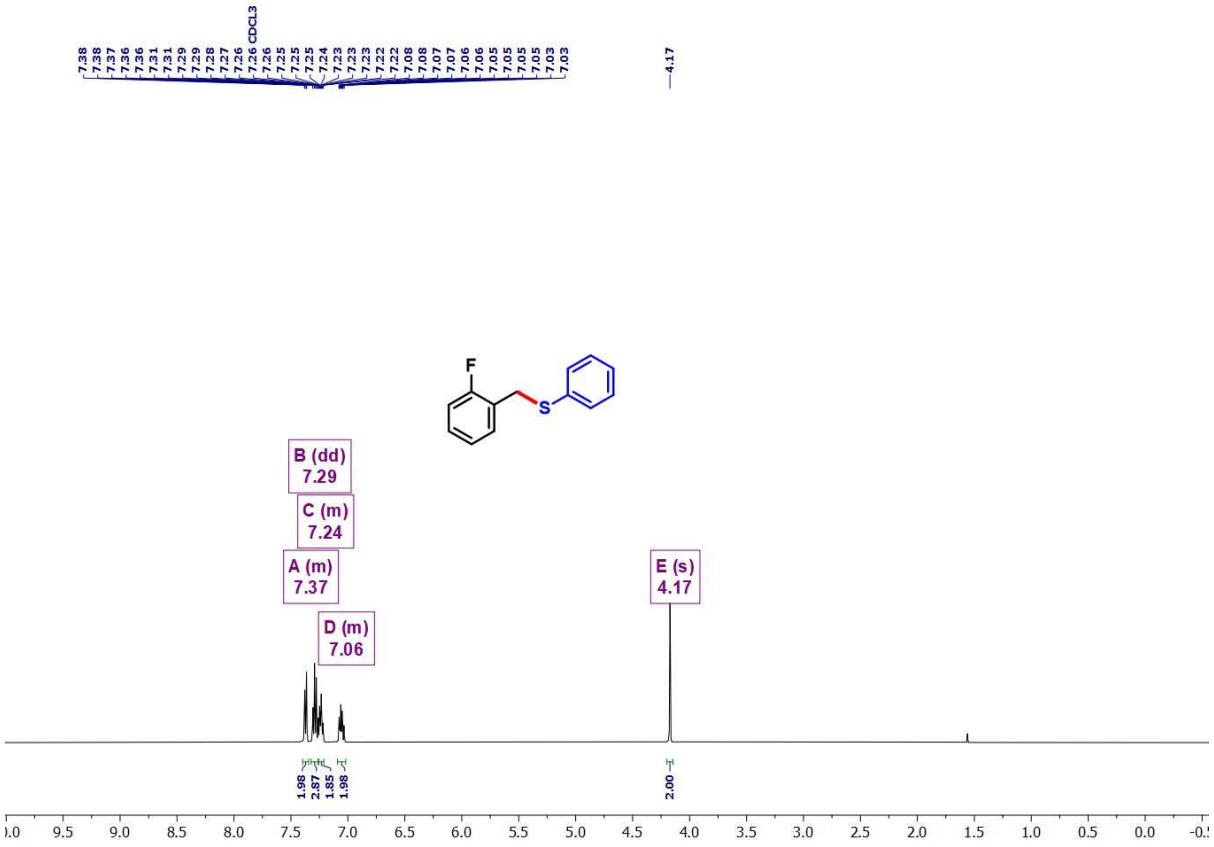
S
CH3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7.08
7.08
7.07
7.07
7.06
7.06
7.05
7.05
7.05
7.03
7.03
4.17
F
S
B (dd)
7.29
C (m)
7.24
A (m)
7.37
D (m)
7.06
E (s)
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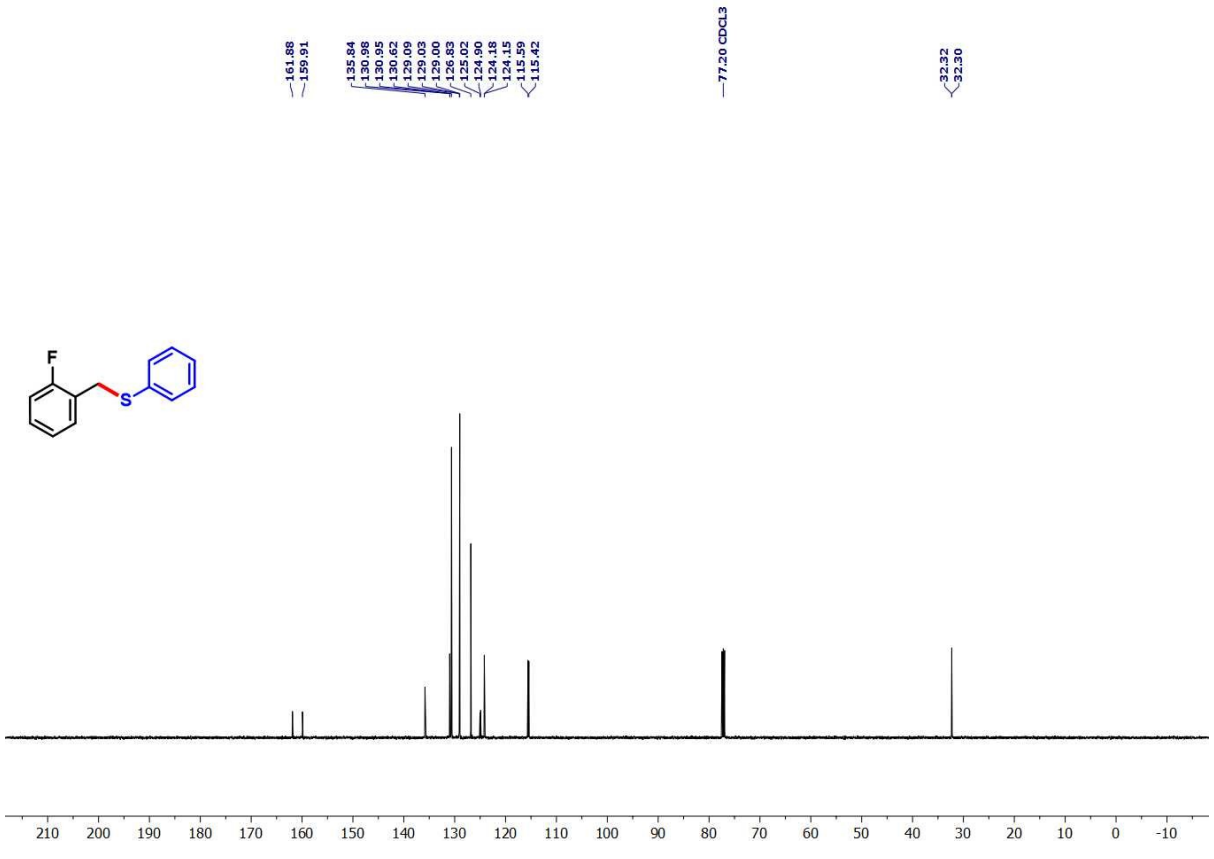
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F
S
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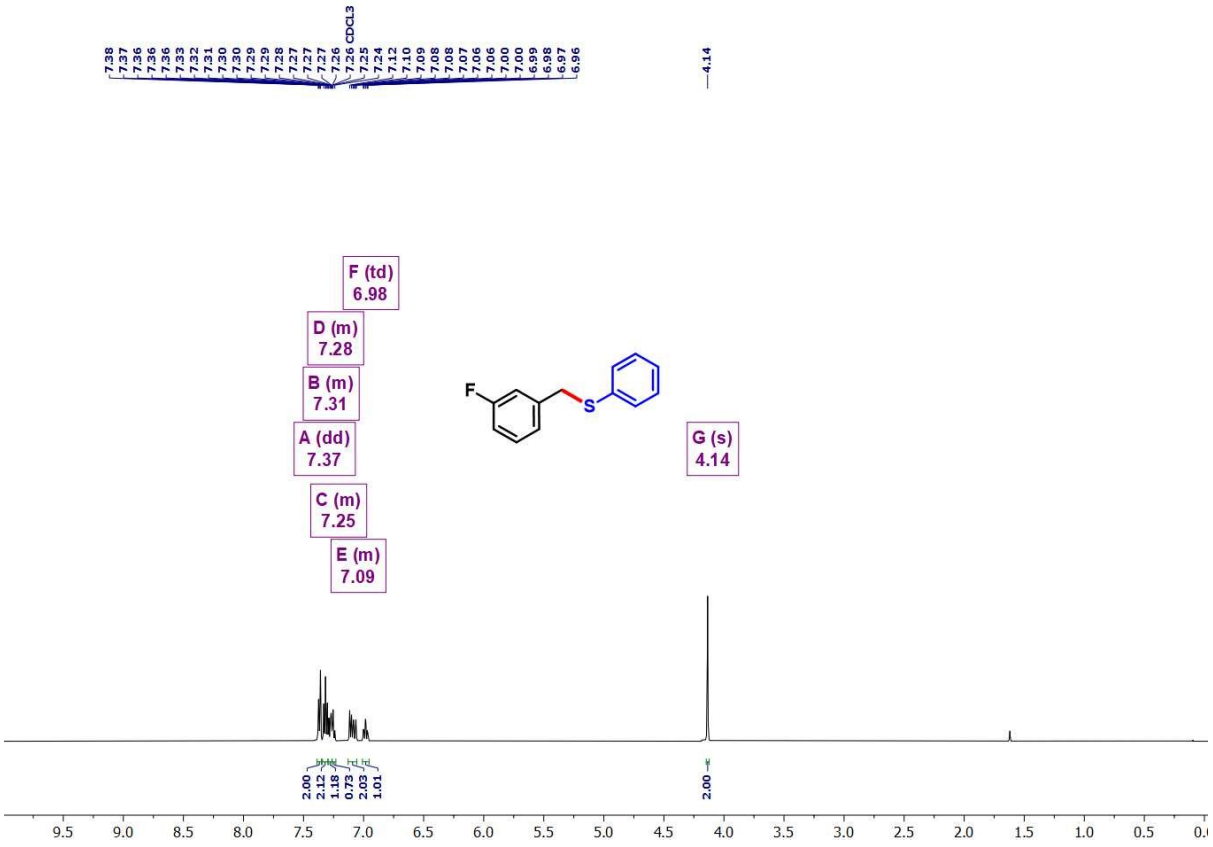
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7.08
7.07
7.06
7.06
7.00
7.00
6.99
6.98
6.97
6.96
4.14
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6.98
D (m)
7.28
B (m)
7.31
A (dd)
7.37
C (m)
7.25
E (m)
7.09
G (s)
4.14
F
S
2.00
2.12
1.18
0.73
2.03
1.01
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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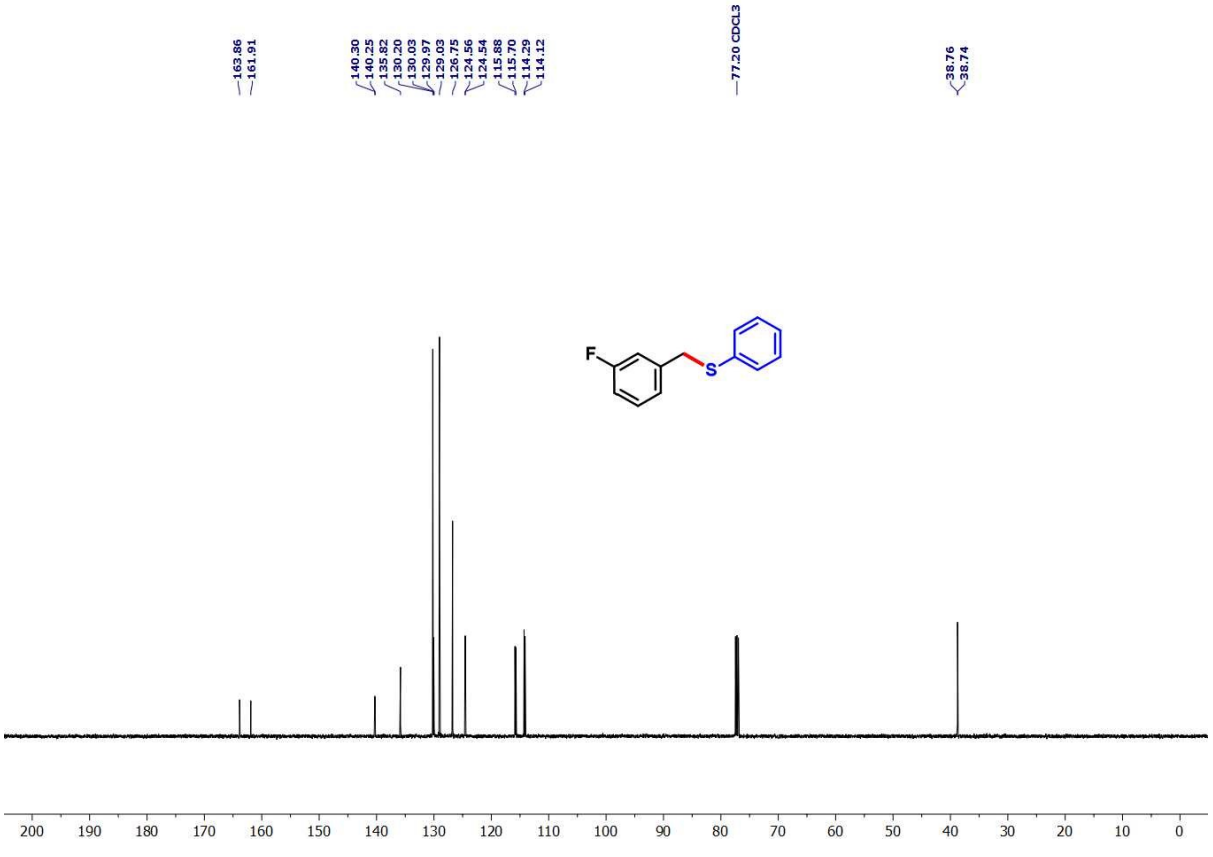
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129.03
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124.56
124.54
115.88
115.70
114.29
114.12
77.20 CDCL3
38.76
38.74
F
S
200
190
180
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150
140
130
120
110
100
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80
70
60
50
40
30
20
10
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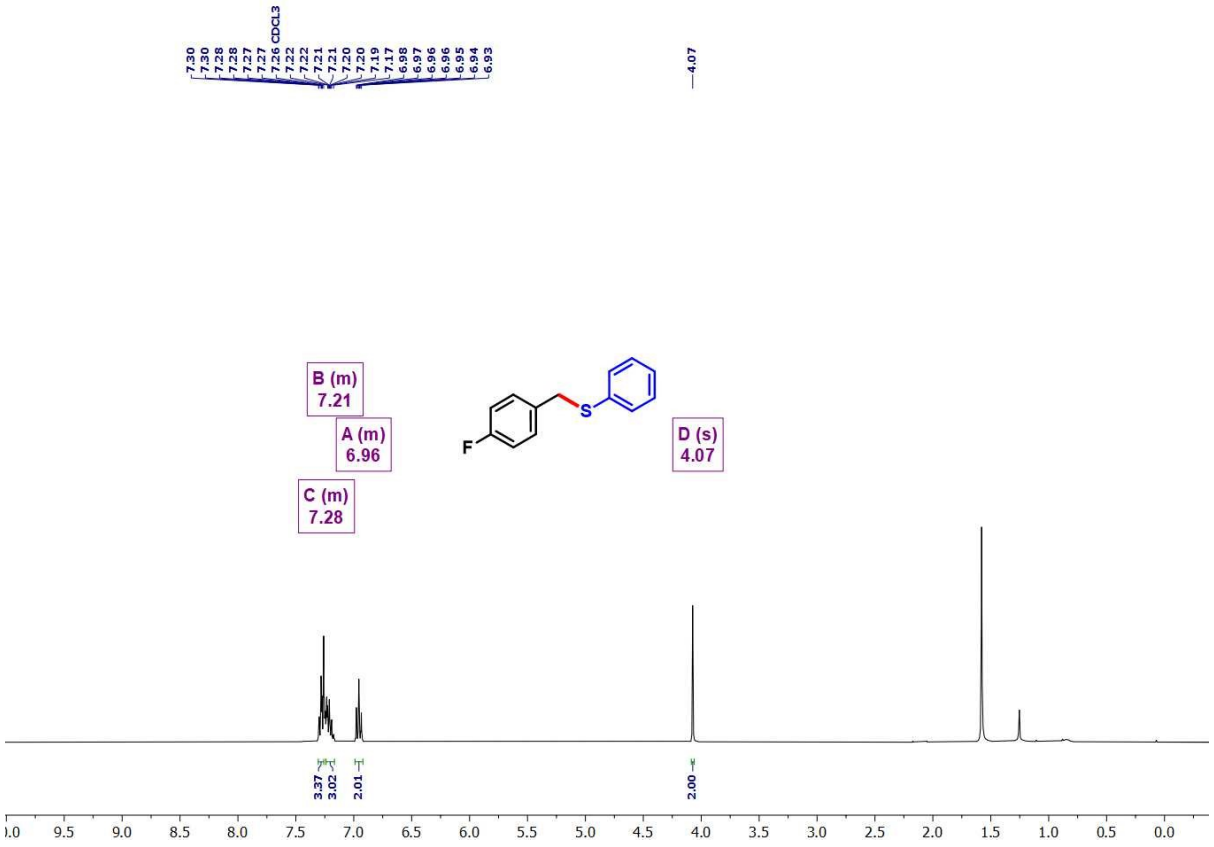
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7.20
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7.17
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6.97
6.96
6.96
6.95
6.94
6.93
4.07
B (m)
7.21
A (m)
6.96
C (m)
7.28
D (s)
4.07
F
S
3.37
3.02
2.01
2.00
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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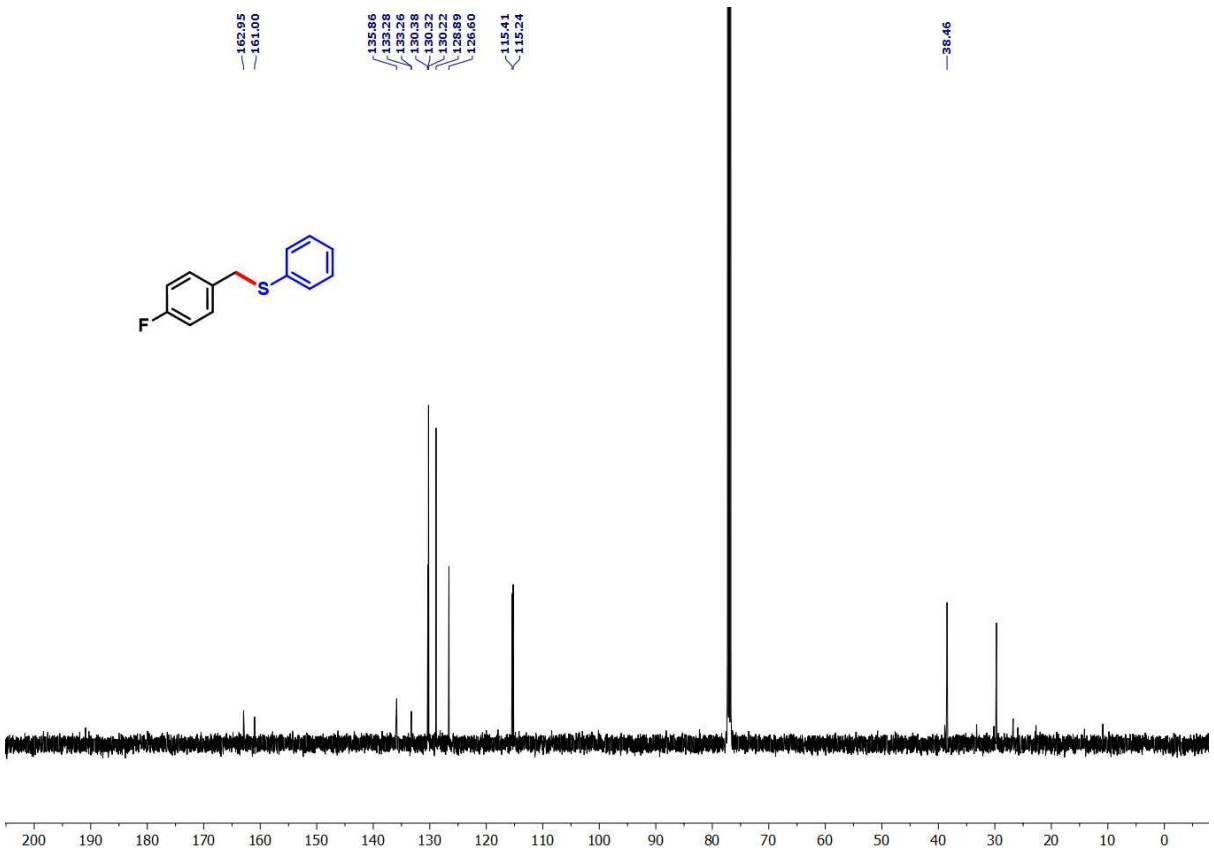
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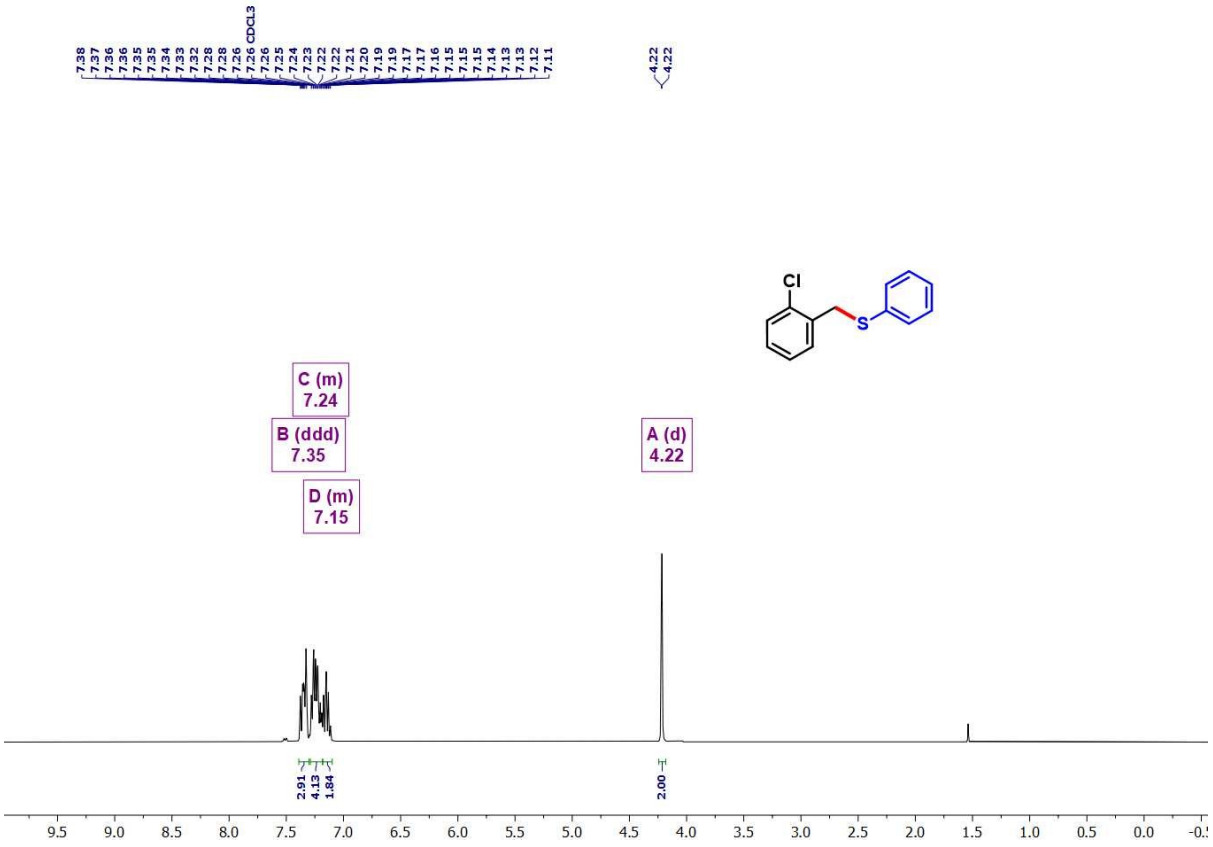
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7.17
7.16
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7.14
7.13
7.12
7.11
4.22
4.22
Cl
S
C (m)
7.24
B (ddd)
7.35
D (m)
7.15
A (d)
4.22
2.91
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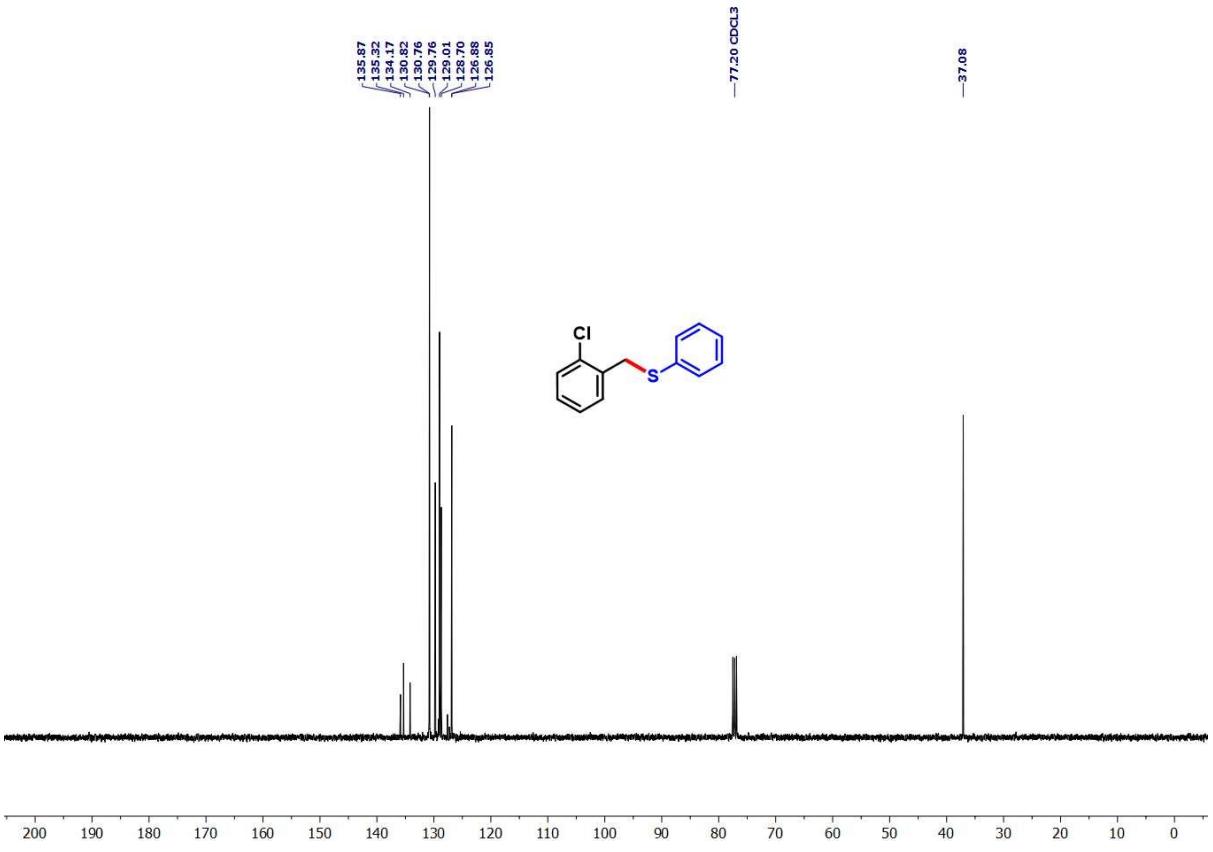
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Cl
S
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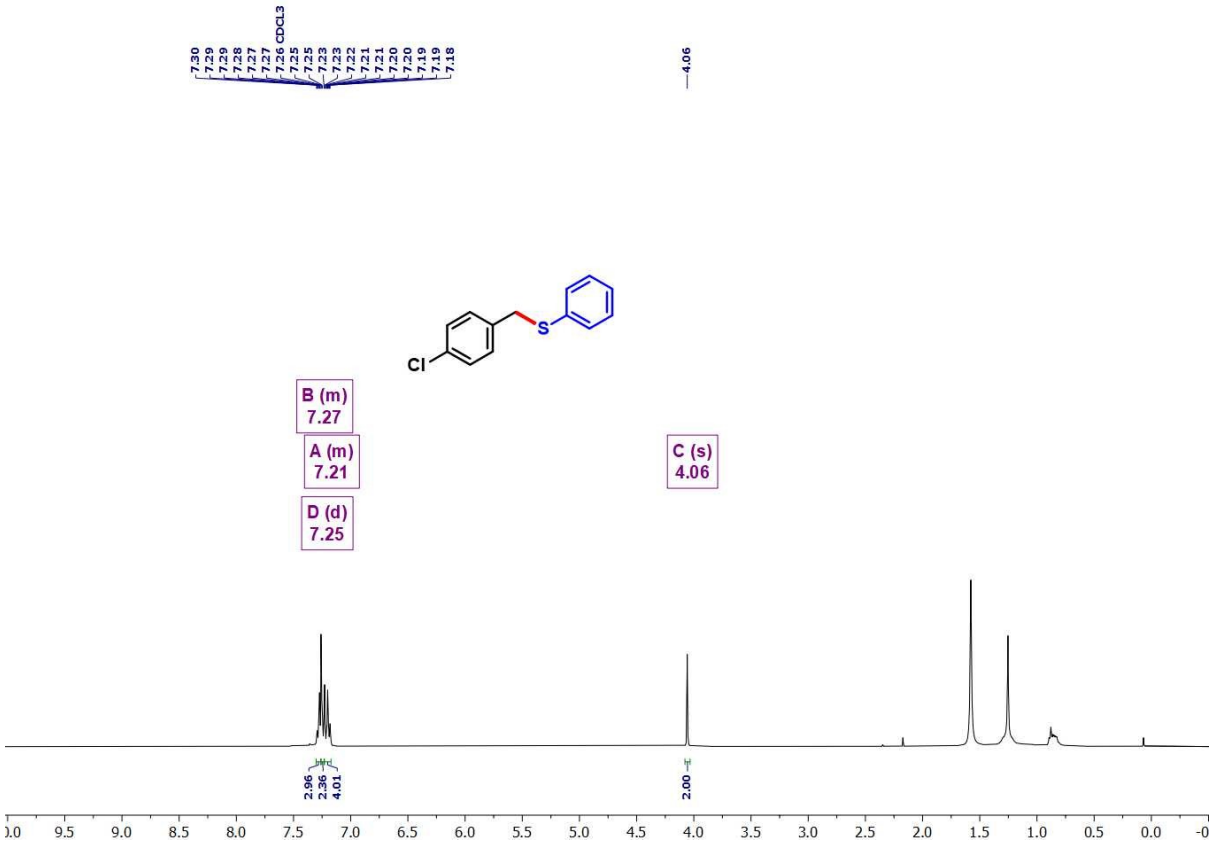
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7.21
7.21
7.20
7.20
7.19
7.19
7.18
4.06
Cl
S
B (m)
7.27
A (m)
7.21
D (d)
7.25
C (s)
4.06
2.96
2.36
4.01
2.00
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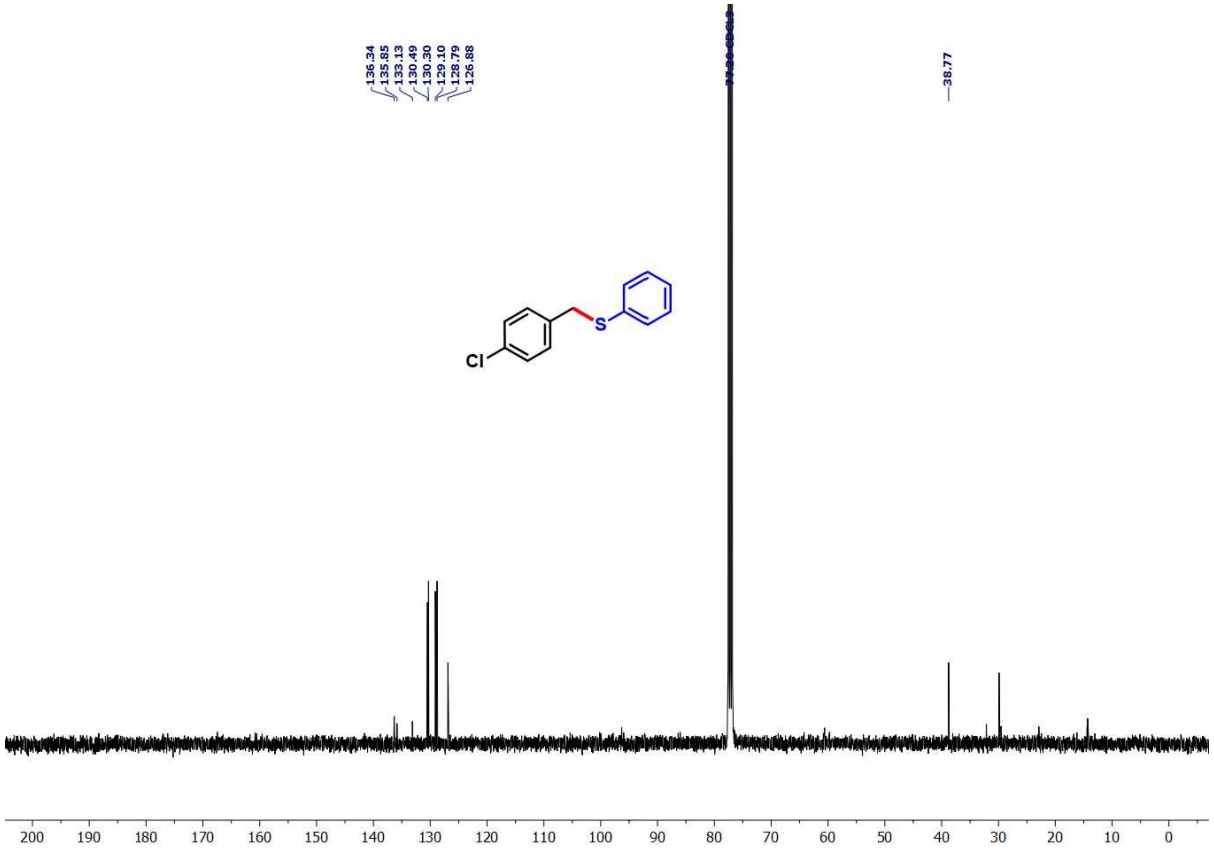
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76.68
38.77
Cl
S
200
190
180
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160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


7.40
7.37
7.29
7.29
7.28
7.28
7.27
7.27
7.26
CDCL3
7.26
7.25
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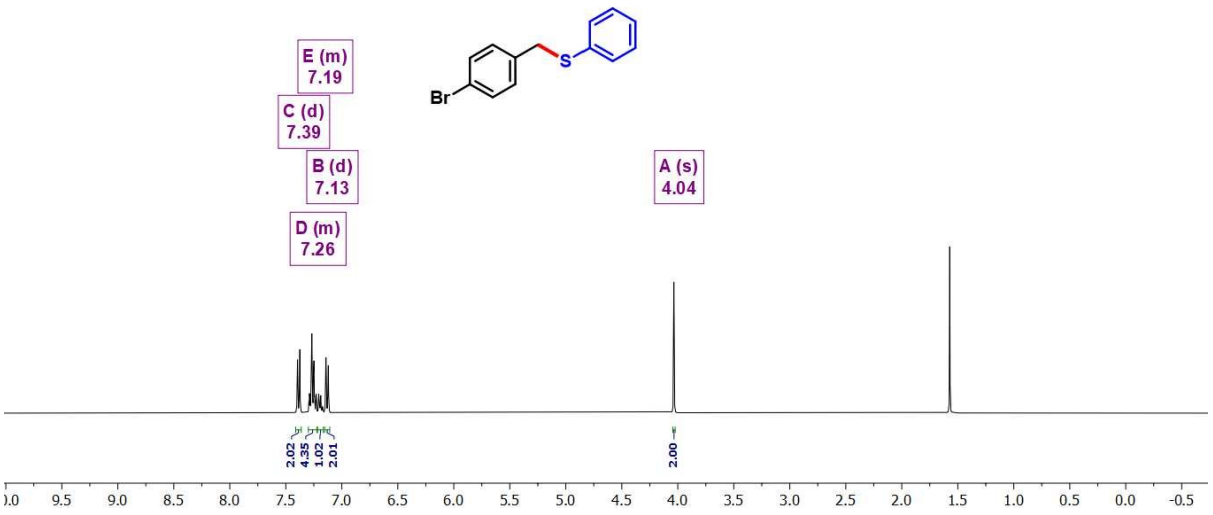
E (m)
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C (d)
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B (d)
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D (m)
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A (s)
4.04
Br
S
2.02
4.35
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7.0
6.5
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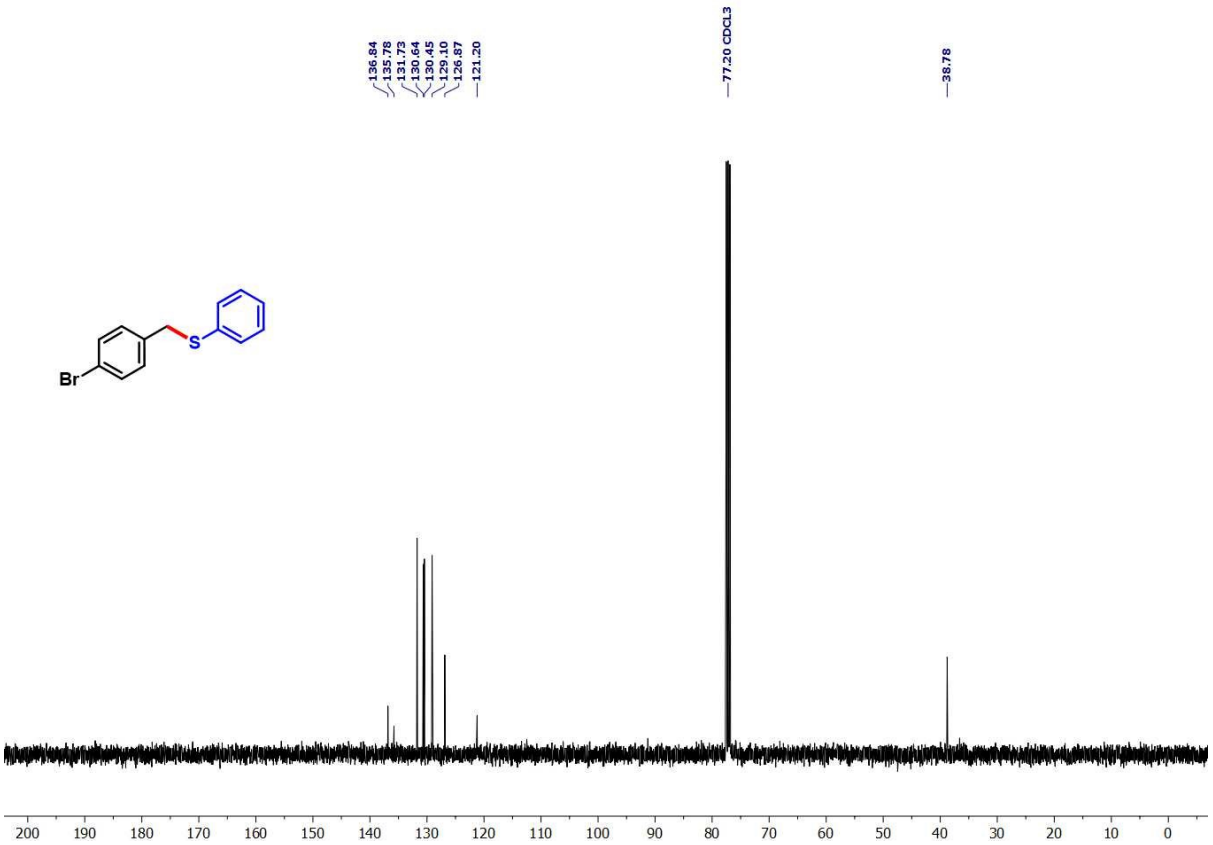
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Br
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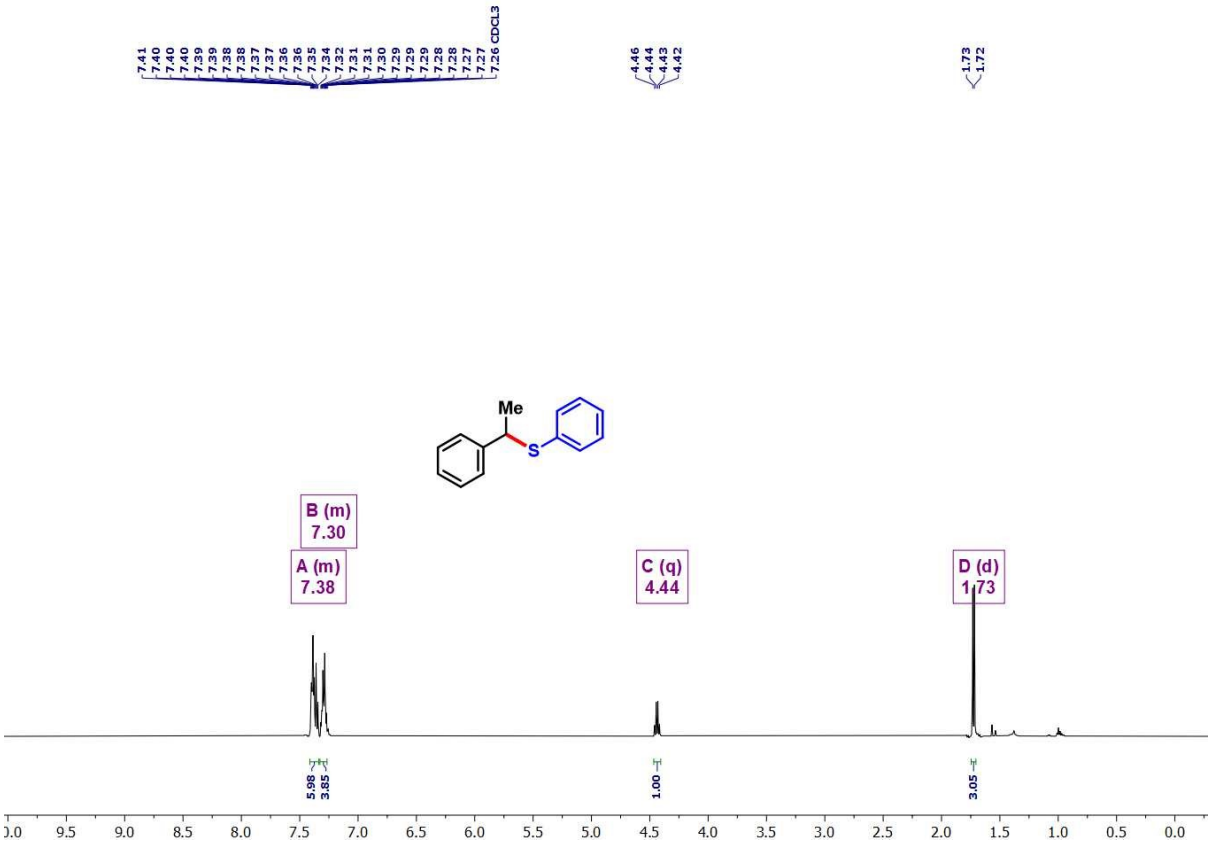
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7.29
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7.27
7.26 CDCL3
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4.44
4.43
4.42
1.73
1.72
Me
S
B (m)
7.30
A (m)
7.38
C (q)
4.44
D (d)
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3.85
1.00
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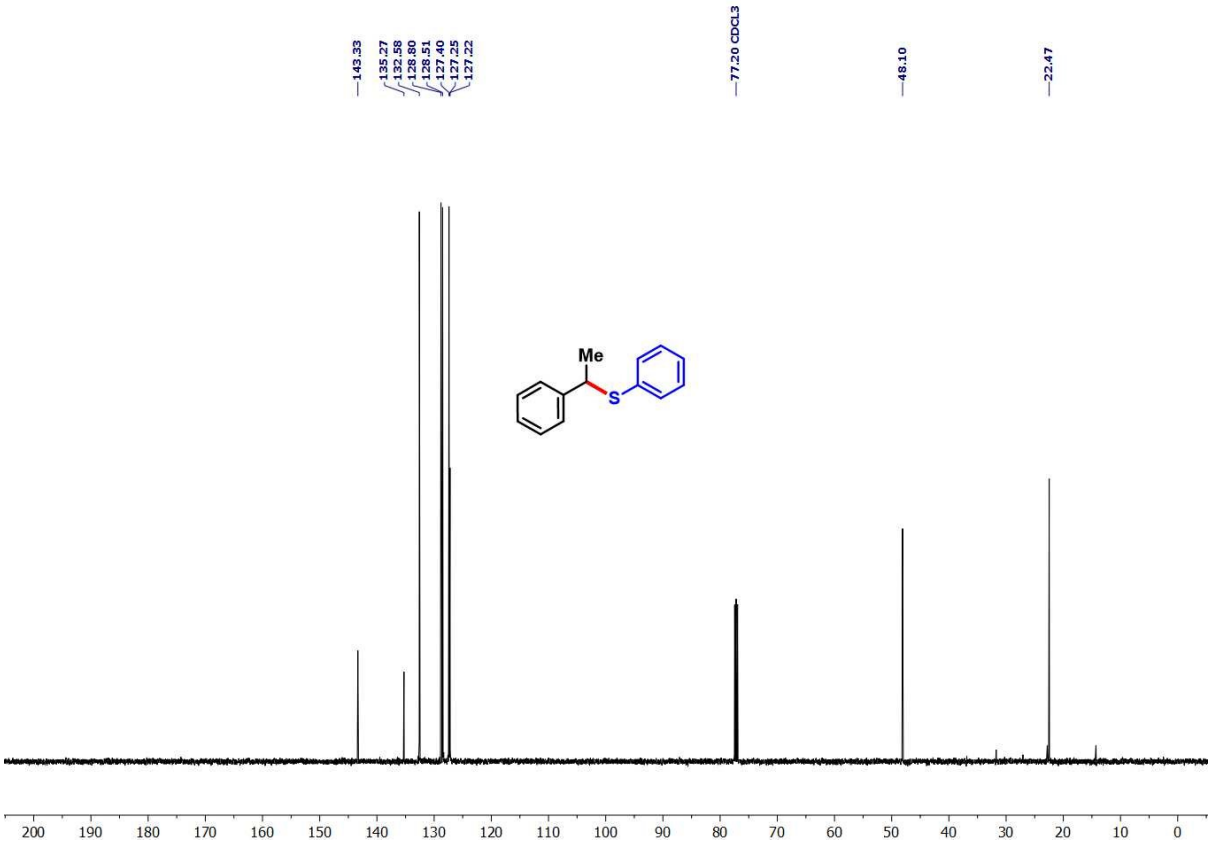
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S
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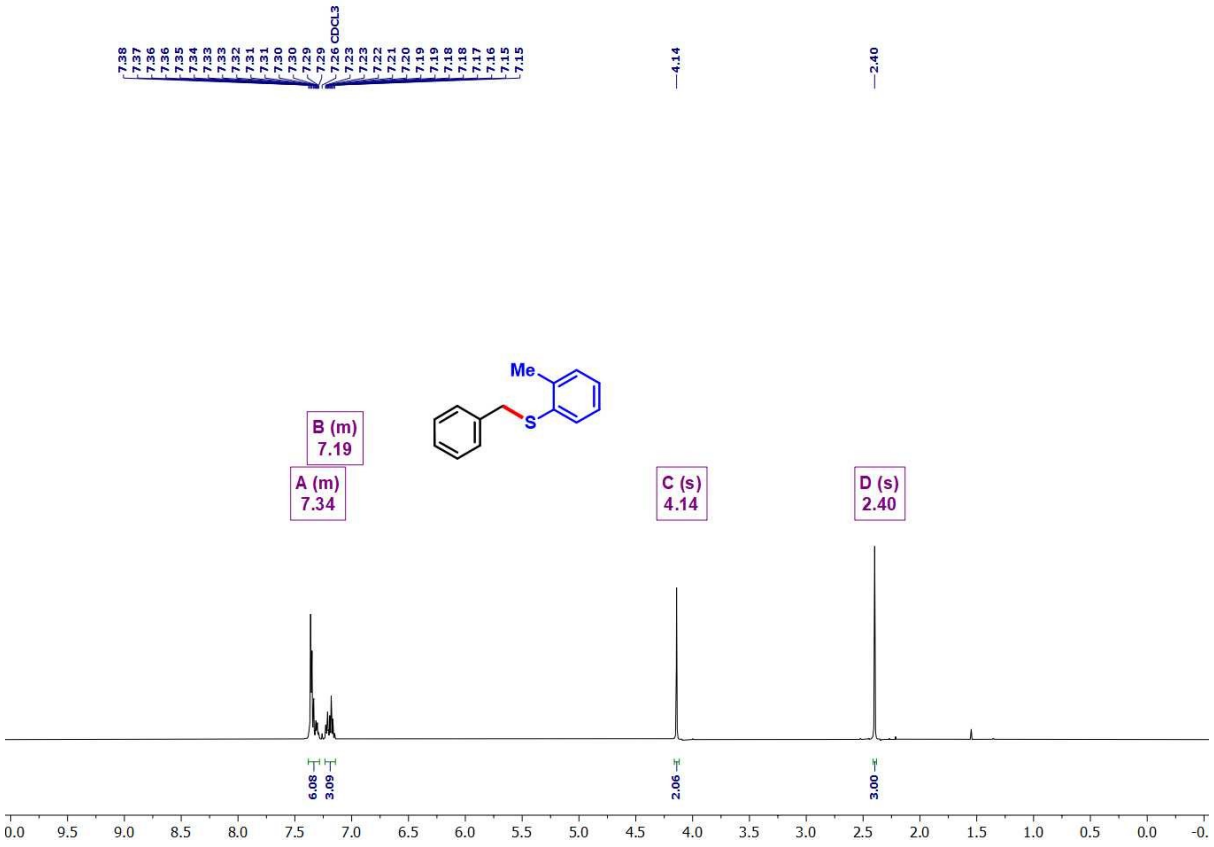
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CDCl3
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7.19
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7.18
7.17
7.16
7.15
7.15
4.14
2.40
Me
S
B (m)
7.19
A (m)
7.34
C (s)
4.14
D (s)
2.40
6.08
3.09
2.06
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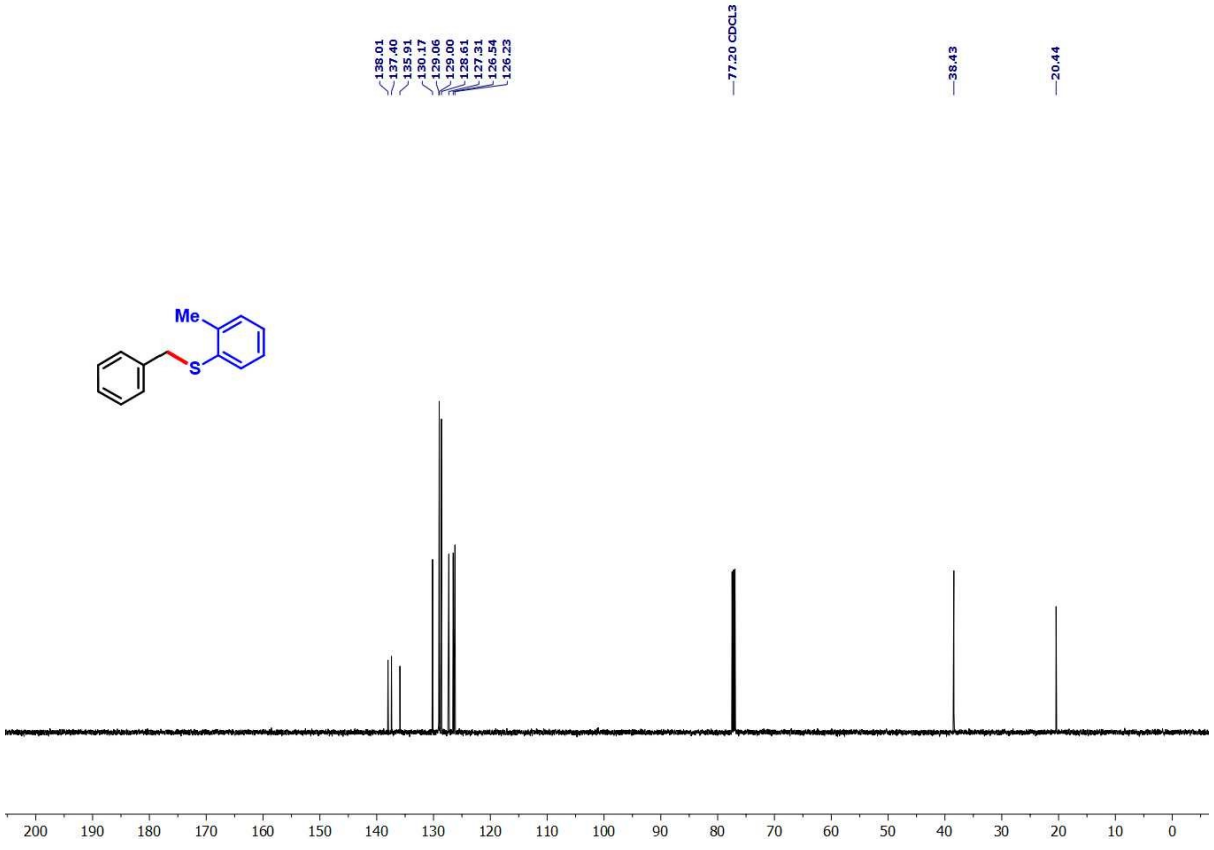
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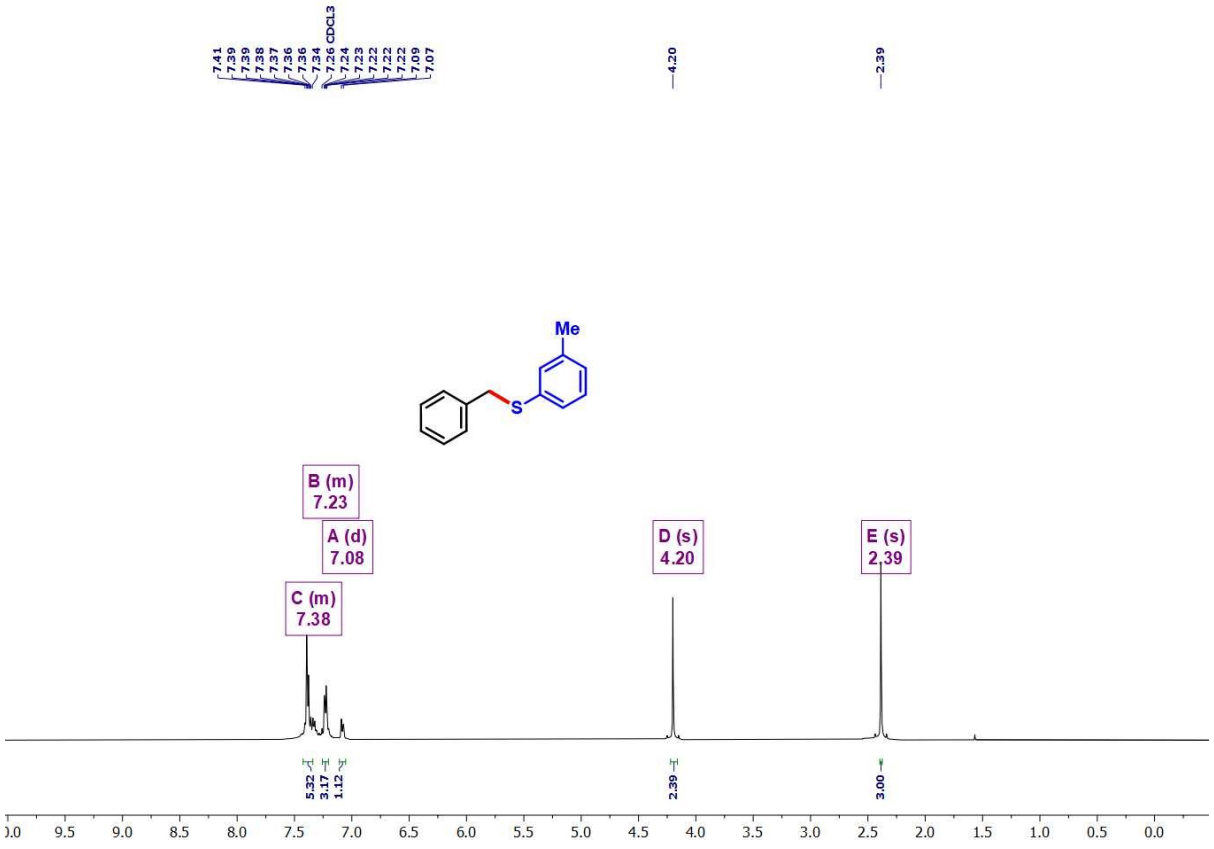
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7.22
7.22
7.09
7.07
4.20
2.39
Me
S
B (m)
7.23
A (d)
7.08
C (m)
7.38
D (s)
4.20
E (s)
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3.17
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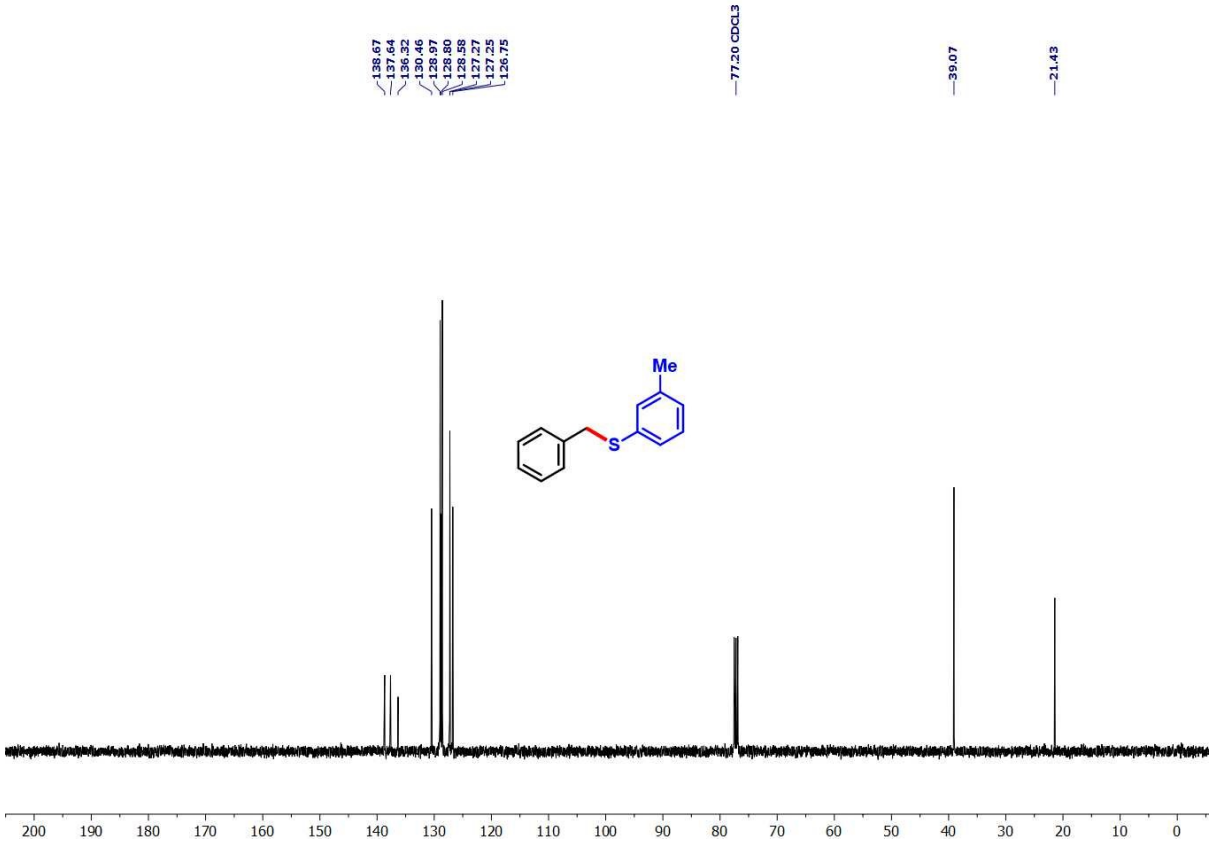
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Me
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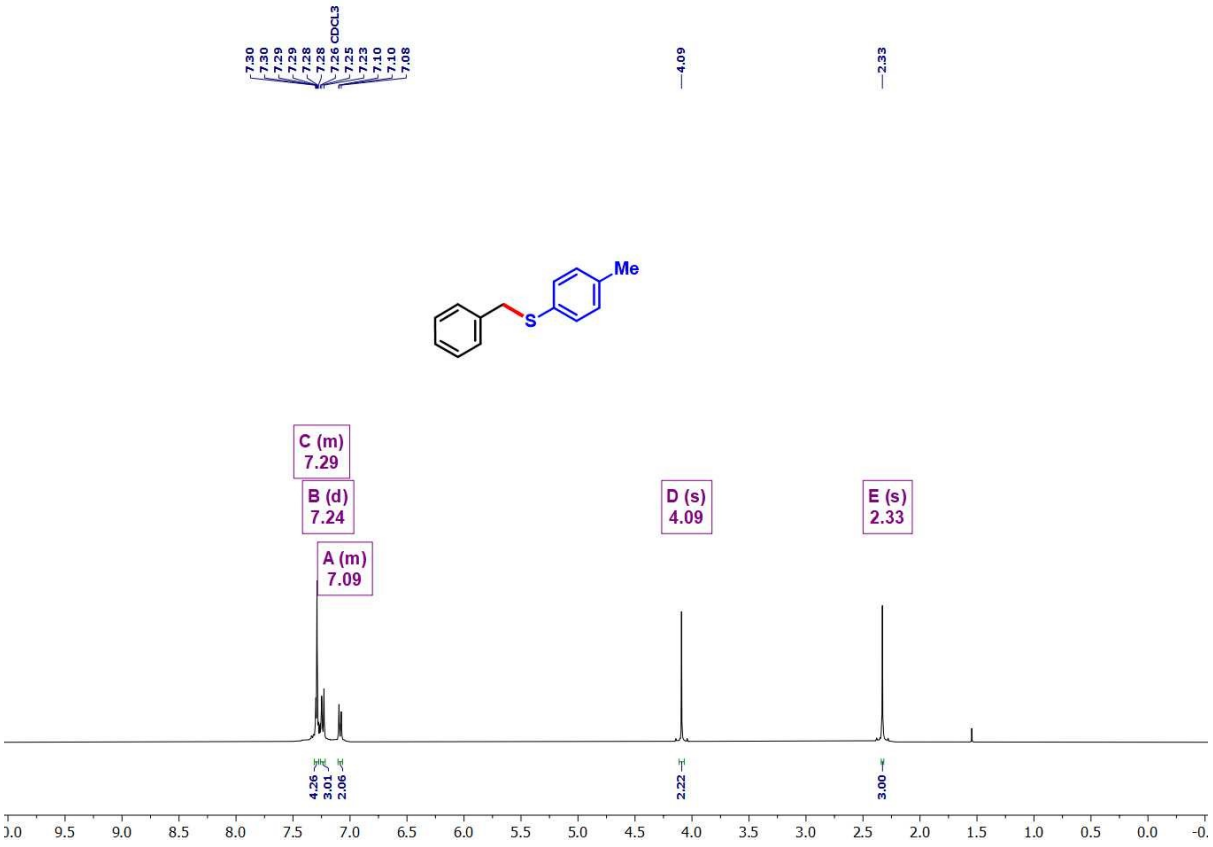
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7.10
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7.08
4.09
2.33
Me
S
C (m)
7.29
B (d)
7.24
A (m)
7.09
D (s)
4.09
E (s)
2.33
4.26
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2.06
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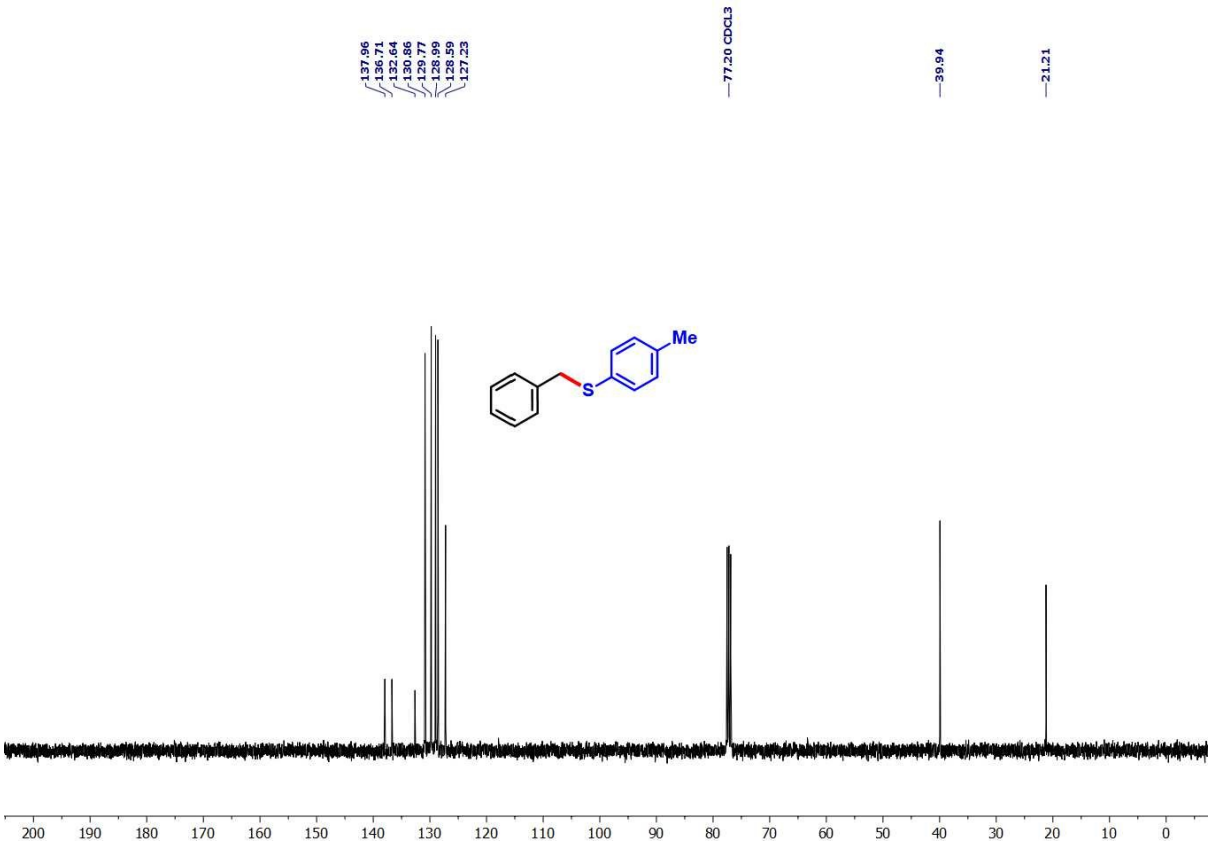
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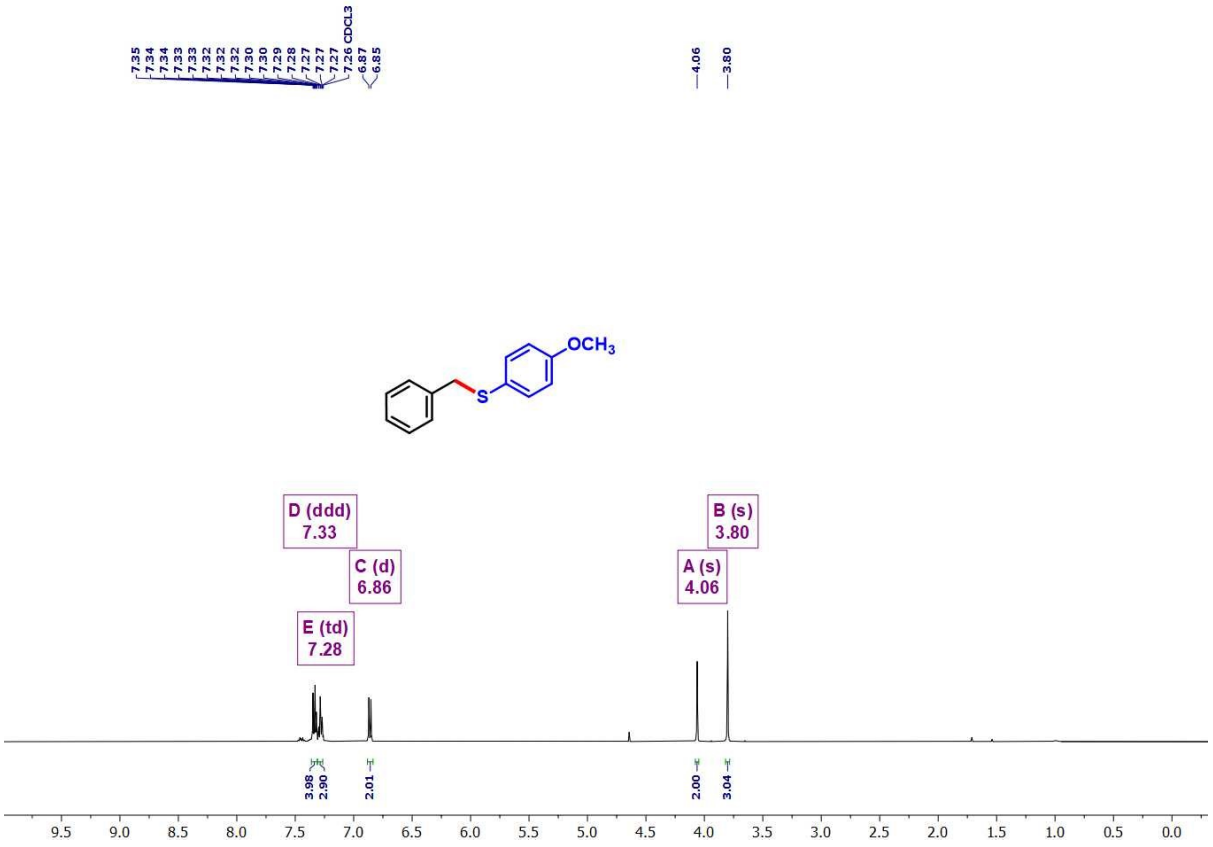
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7.27
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6.85
4.06
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OCH3
S
D (ddd)
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C (d)
6.86
E (td)
7.28
B (s)
3.80
A (s)
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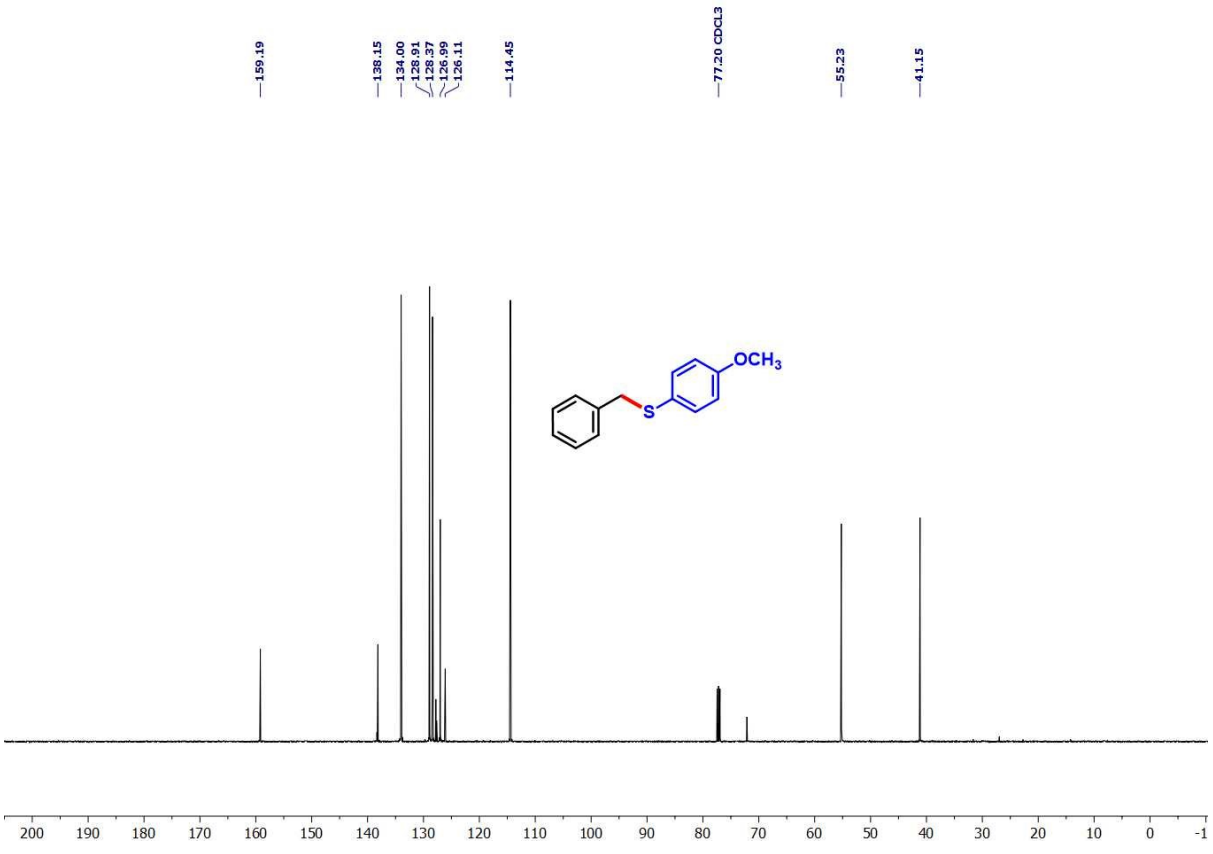
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OCH3
S
200
190
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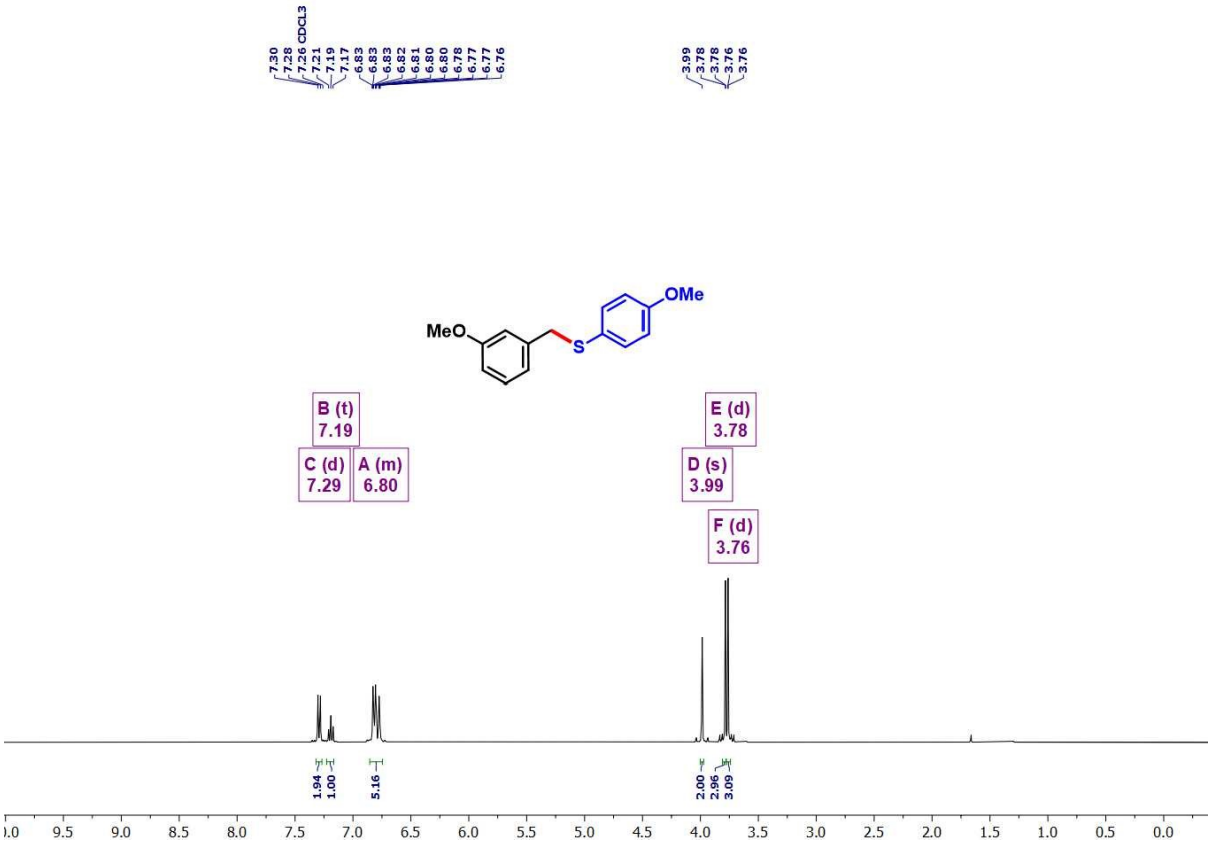
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6.80
6.78
6.77
6.77
6.76
3.99
3.78
3.78
3.76
3.76
MeO
S
OMe
B (t)
7.19
C (d)
7.29
A (m)
6.80
E (d)
3.78
D (s)
3.99
F (d)
3.76
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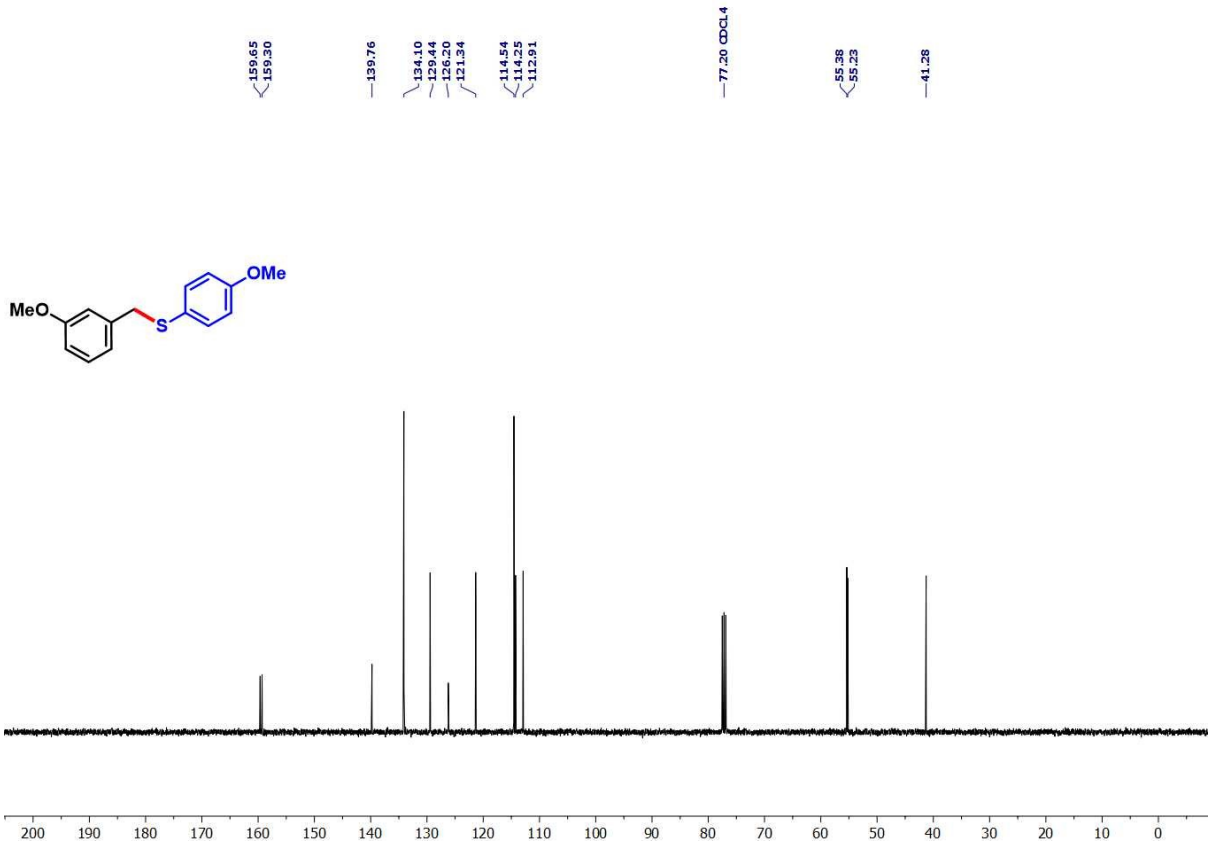
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S
OMe
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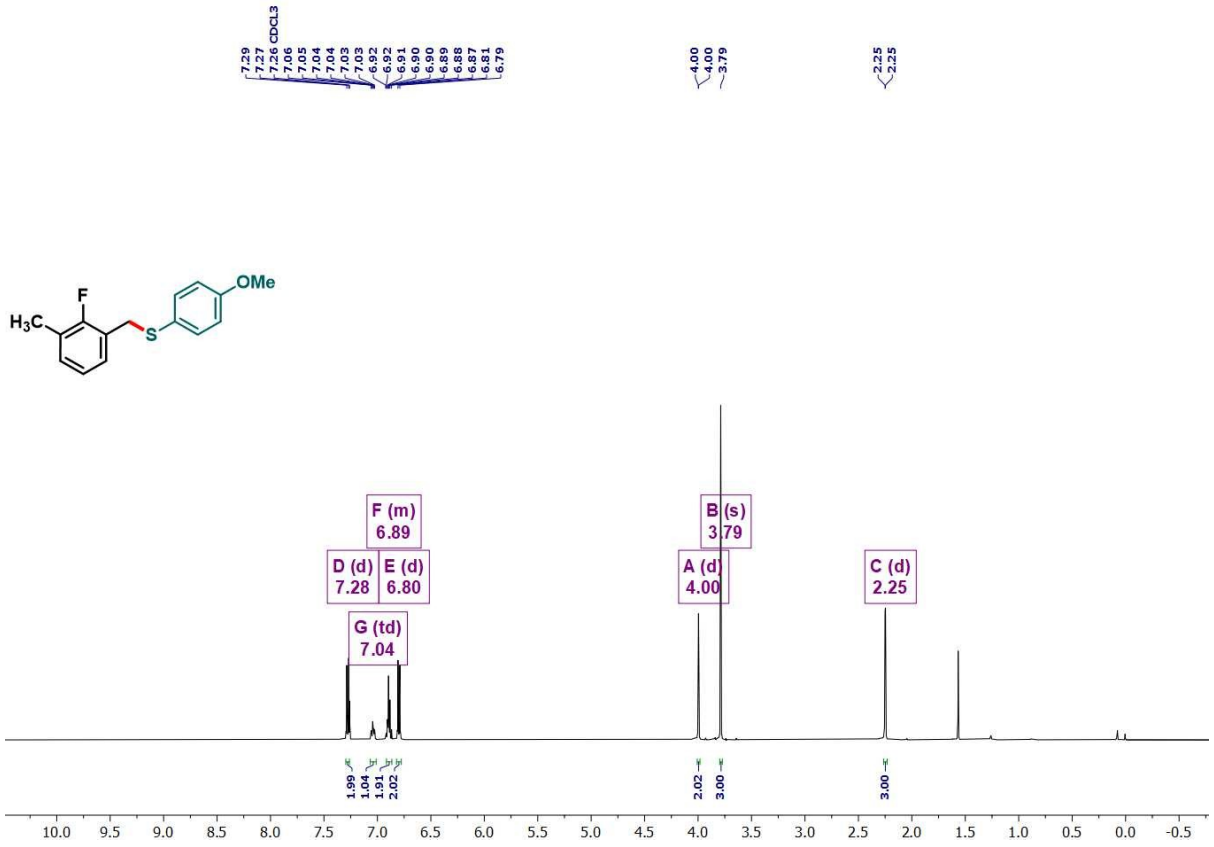
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6.90
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6.87
6.81
6.79
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4.00
3.79
2.25
2.25
F
H3C
S
OMe
F (m)
6.89
B (s)
3.79
D (d)
7.28
E (d)
6.80
A (d)
4.00
C (d)
2.25
G (td)
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1.04
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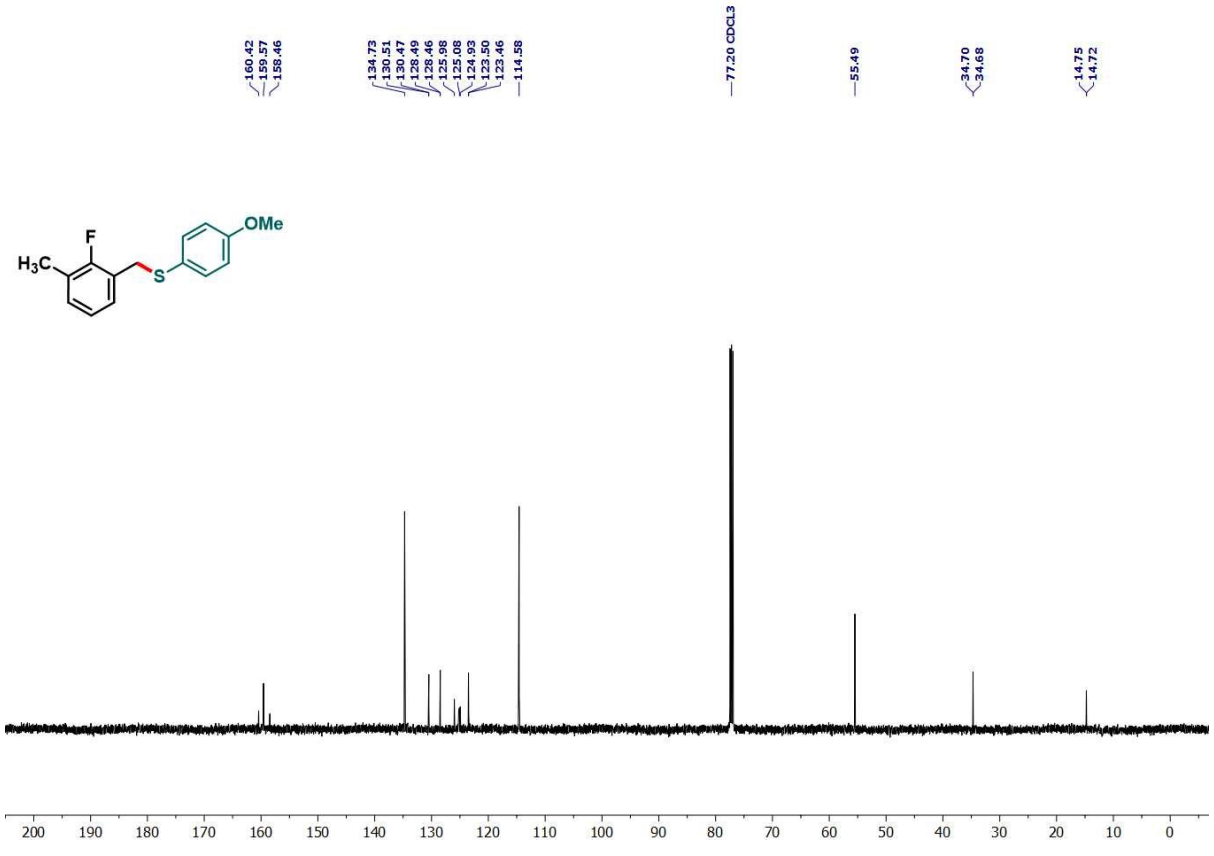
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H3C
S
OMe
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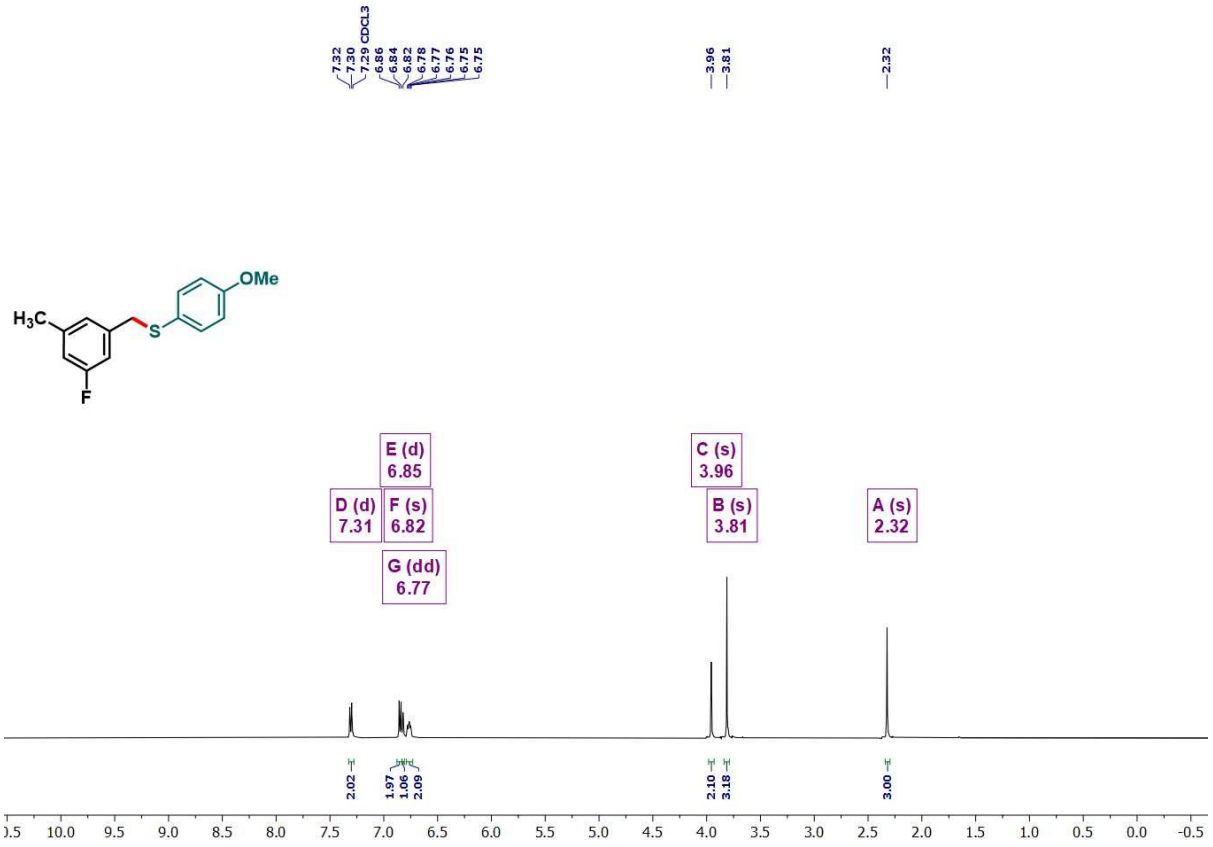
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6.75
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3.81
2.32
OMe
H3C
S
F
E (d)
6.85
D (d)
7.31
F (s)
6.82
G (dd)
6.77
C (s)
3.96
B (s)
3.81
A (s)
2.32
2.02
1.97
1.06
2.09
2.10
3.18
3.00
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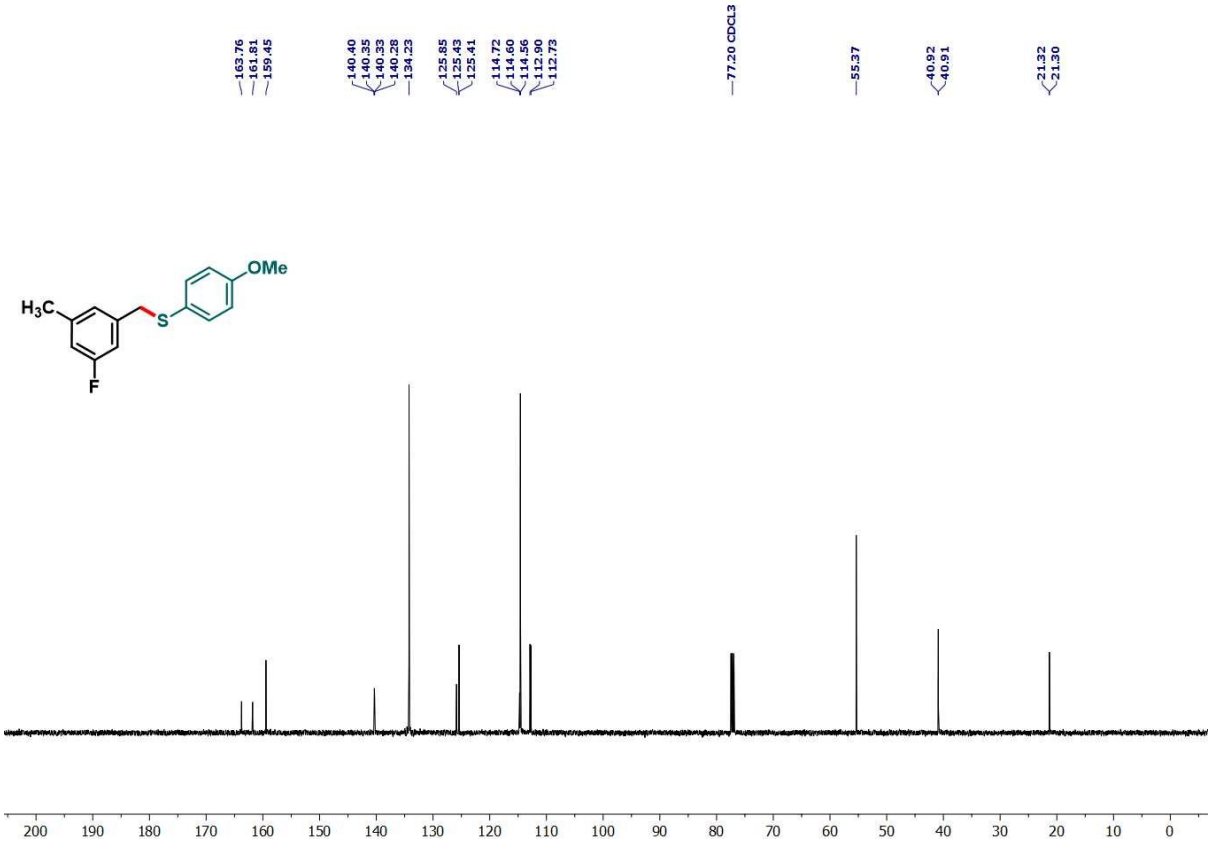
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OMe
H3C
S
F
200
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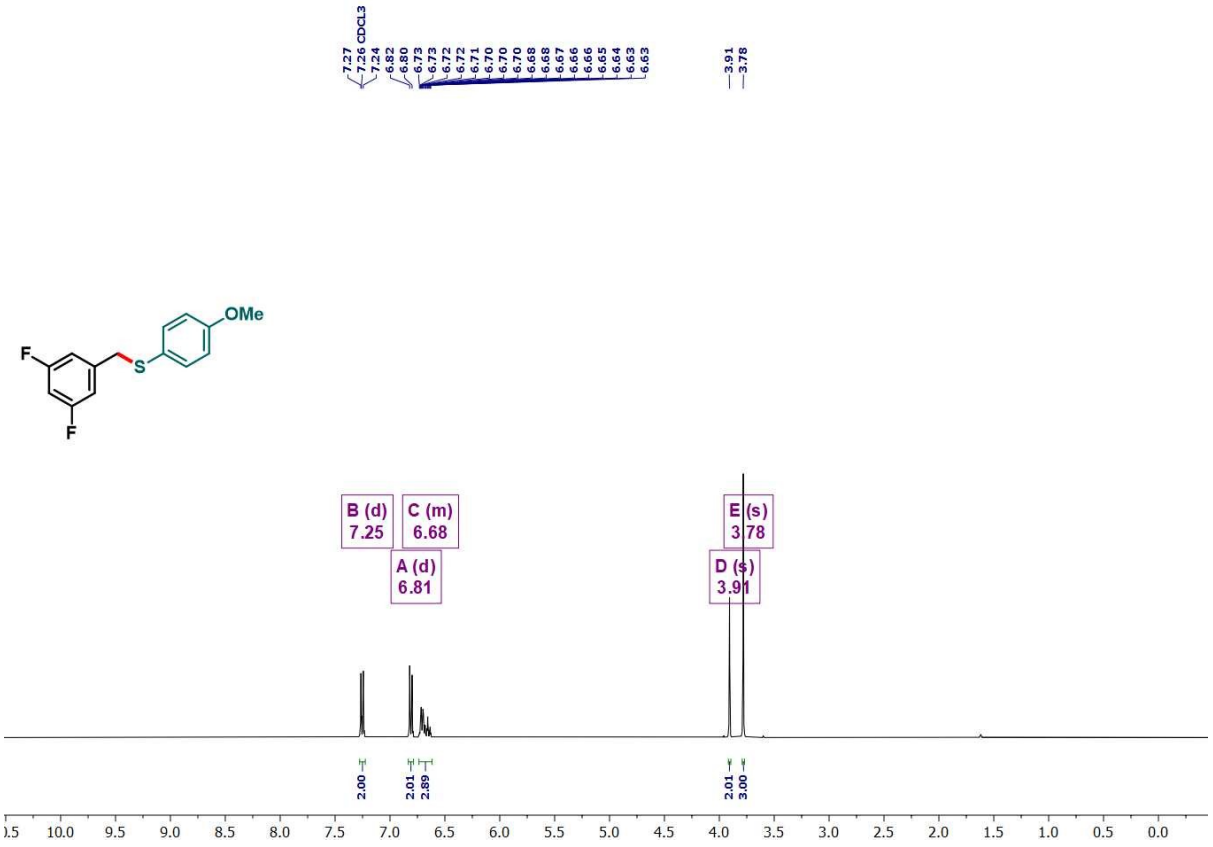
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6.80
6.73
6.73
6.72
6.72
6.71
6.70
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6.70
6.68
6.68
6.67
6.66
6.66
6.65
6.64
6.63
6.63
3.91
3.78
OMe
F
S
F
B (d)
7.25
C (m)
6.68
A (d)
6.81
E (s)
3.78
D (s)
3.91
2.00
2.01
2.89
2.01
3.00
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8.0
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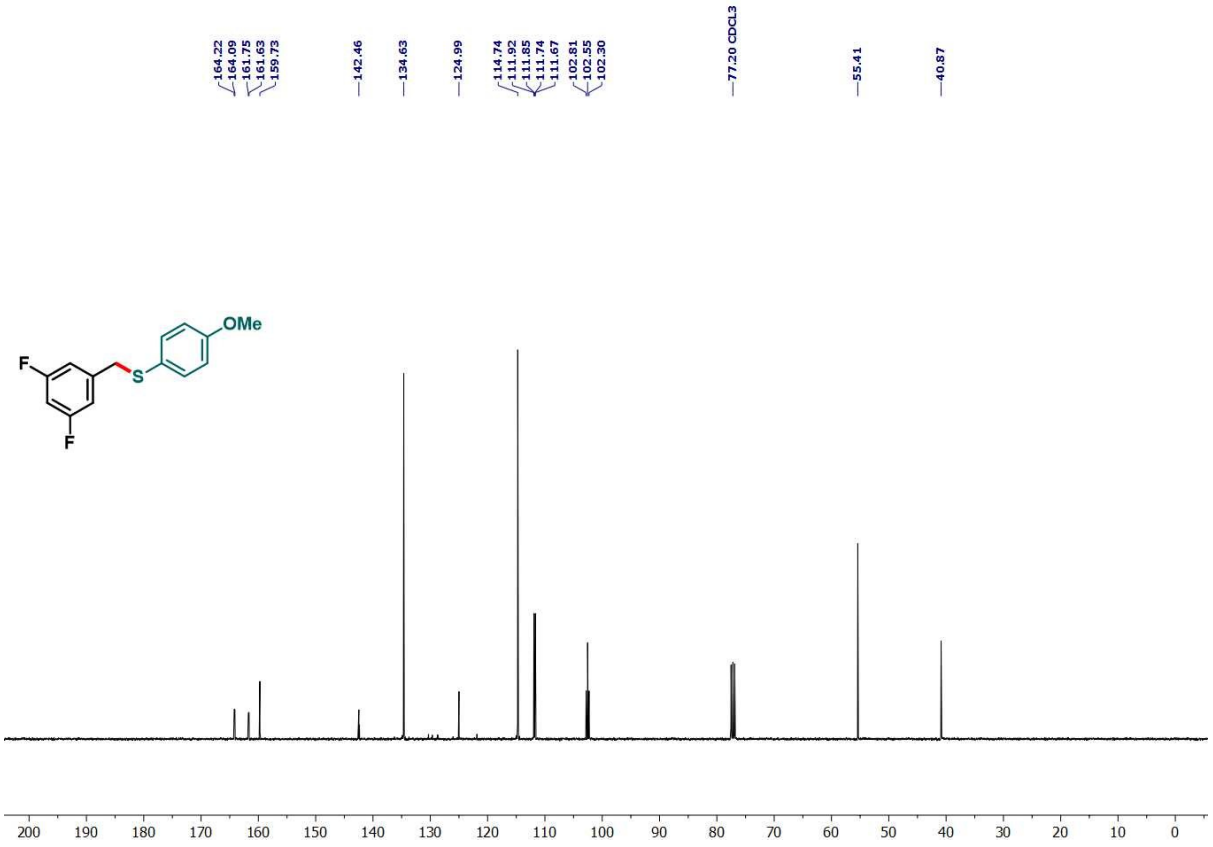
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102.81
102.55
102.30
77.20 CDCL3
55.41
40.87
OMe
F
S
F
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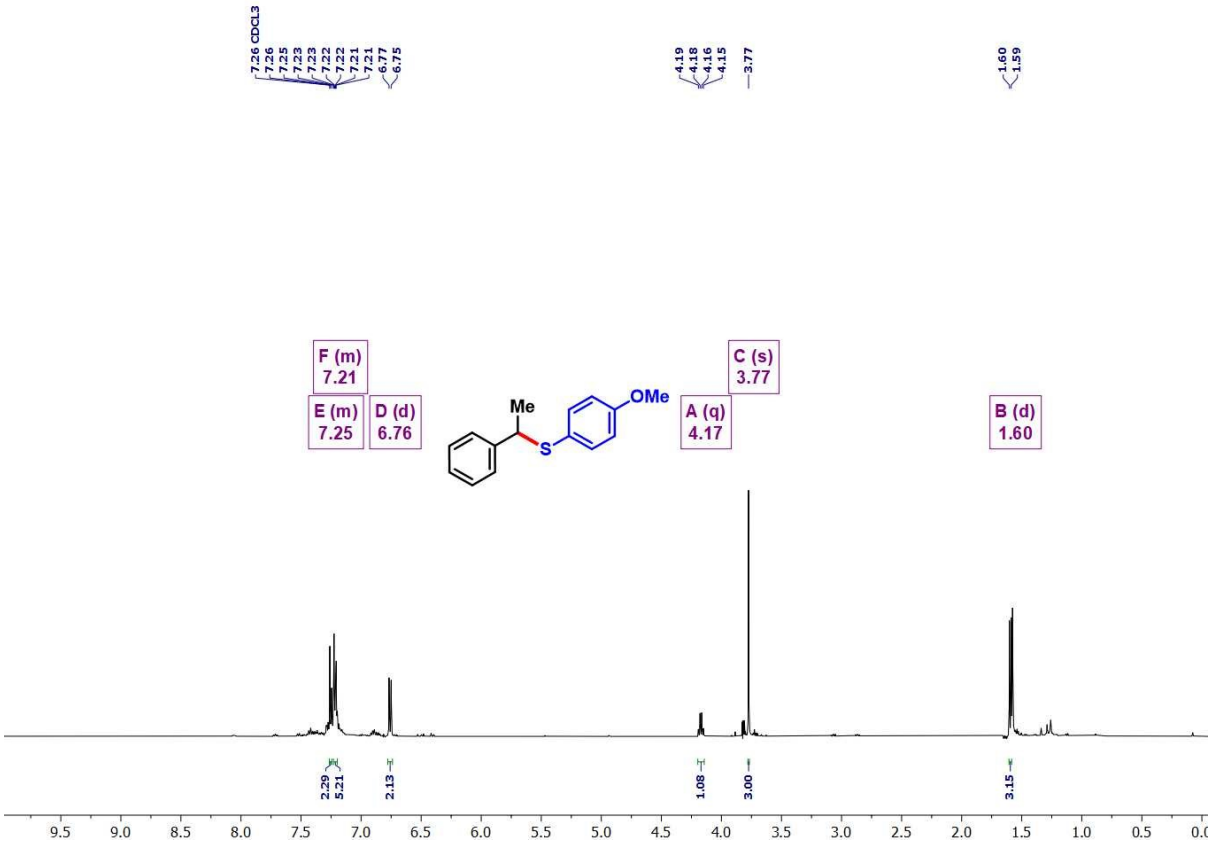
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7.25
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7.23
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7.21
7.21
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6.75
4.19
4.18
4.16
4.15
3.77
1.60
1.59
F (m)
7.21
E (m)
7.25
D (d)
6.76
C (s)
3.77
A (q)
4.17
B (d)
1.60
Me
OMe
S
2.29
5.21
2.13
1.08
3.00
3.15
9.5
9.0
8.5
8.0
7.5
7.0
6.5
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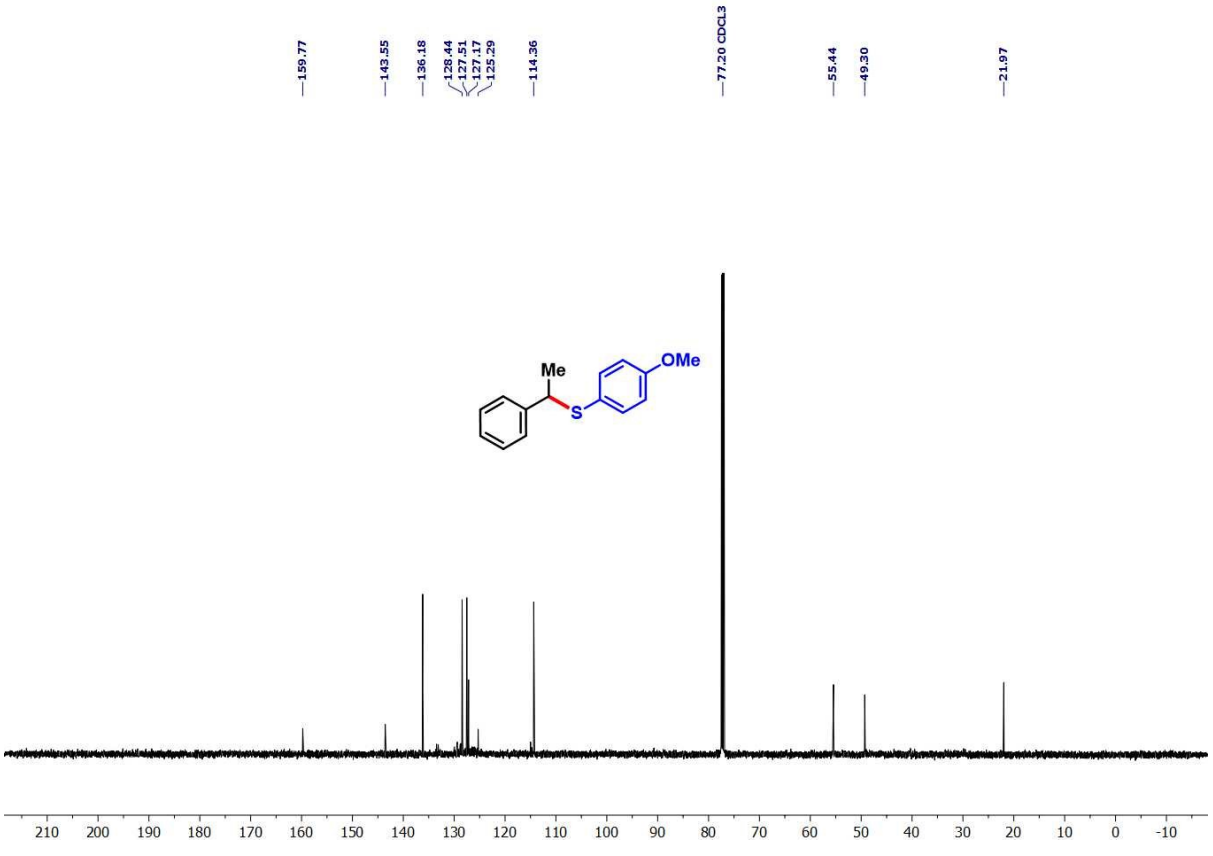
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55.44
49.30
21.97
Me
OMe
S
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


7.26 CDCL3
7.25
7.25
7.23
7.23
7.23
7.22
7.21
7.21
7.21
7.20
7.20
7.20
7.19
7.19
7.19
7.19
7.18
7.17
7.17
7.17
7.16
7.16
7.15
7.15
6.75
6.73
3.90
3.88
3.87
3.86
3.76
2.03
2.01
2.01
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1.99
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1.94
1.94
1.93
1.92
1.91
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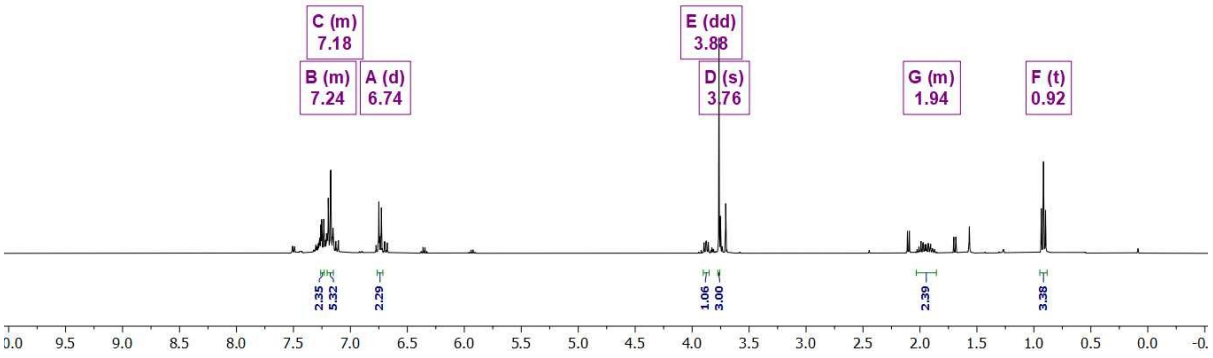
1.89
1.87
0.93
0.92
0.90


Me
OMe
S


C (m)
7.18
B (m)
7.24
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6.74
E (dd)
3.88
D (s)
3.76
G (m)
1.94
F (t)
0.92
2.35
5.32
2.29
1.06
3.00
2.39
3.38
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


159.63
142.30
136.06
129.61
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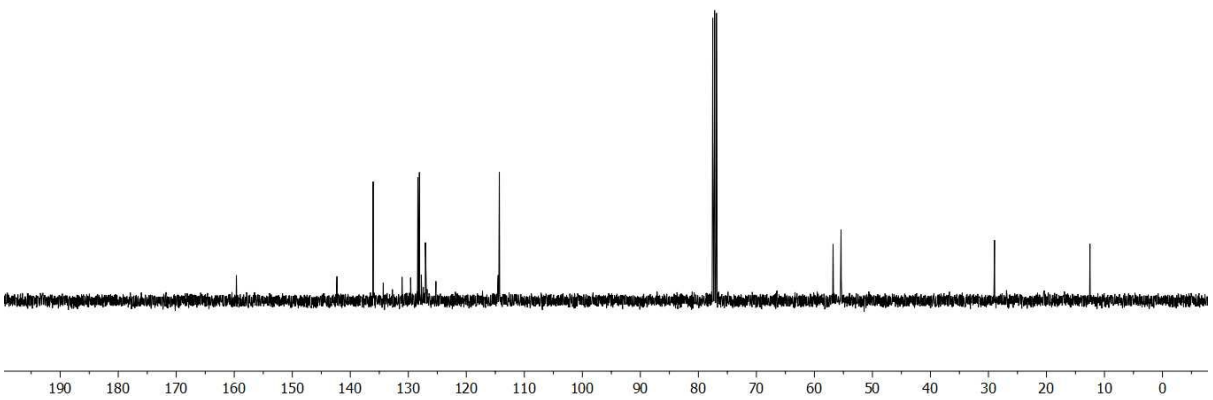
127.06
114.31
77.20 CDCL3
56.78
55.41
28.95
12.50


Me
OMe
S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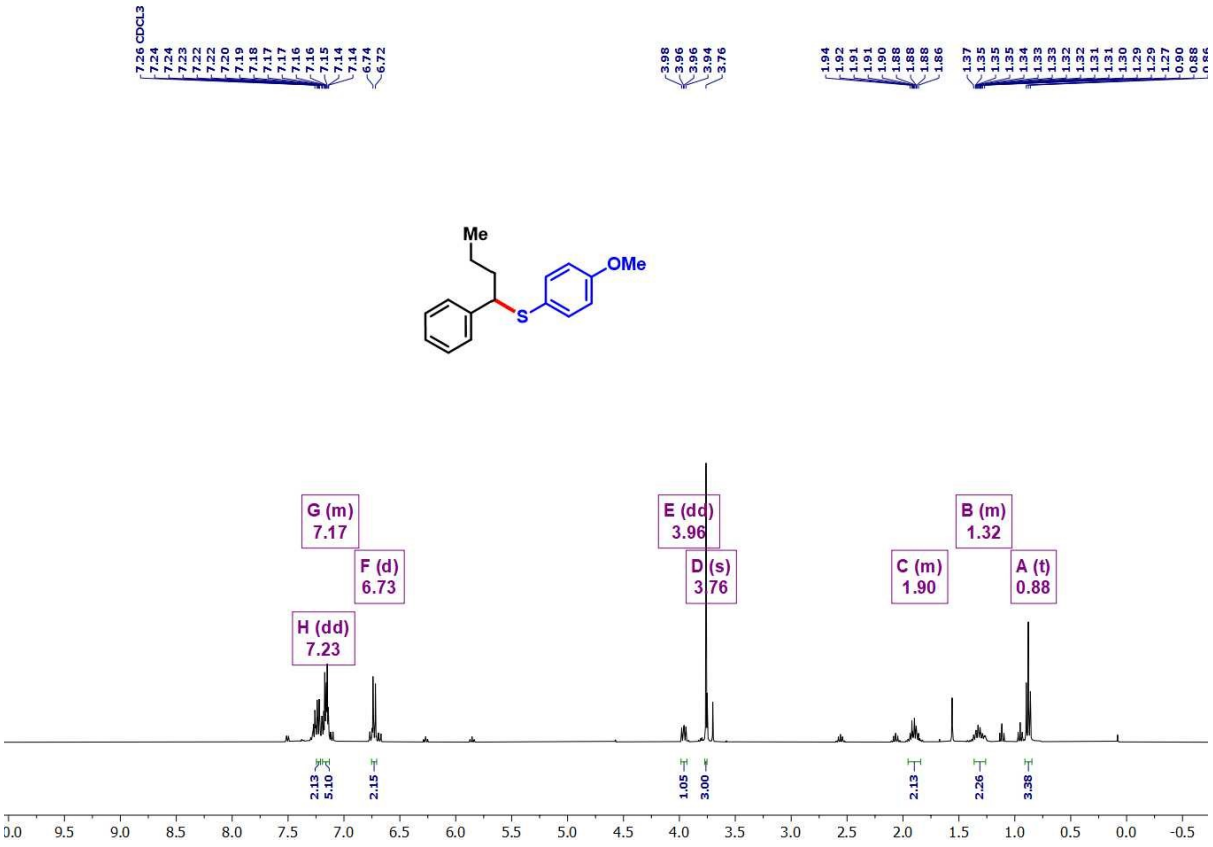
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7.22
7.20
7.19
7.18
7.17
7.17
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7.16
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7.14
6.74
6.72
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3.96
3.96
3.94
3.76
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1.92
1.91
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1.90
1.88
1.88
1.88
1.86
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1.35
1.34
1.33
1.33
1.32
1.32
1.31
1.31
1.30
1.29
1.29
1.27
0.90
0.88
Me
OMe
S
G (m)
7.17
F (d)
6.73
H (dd)
7.23
E (dd)
3.96
D (s)
3.76
C (m)
1.90
B (m)
1.32
A (t)
0.88
2.13
5.10
2.15
1.05
3.00
2.13
2.26
3.38
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
1.5
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0.0
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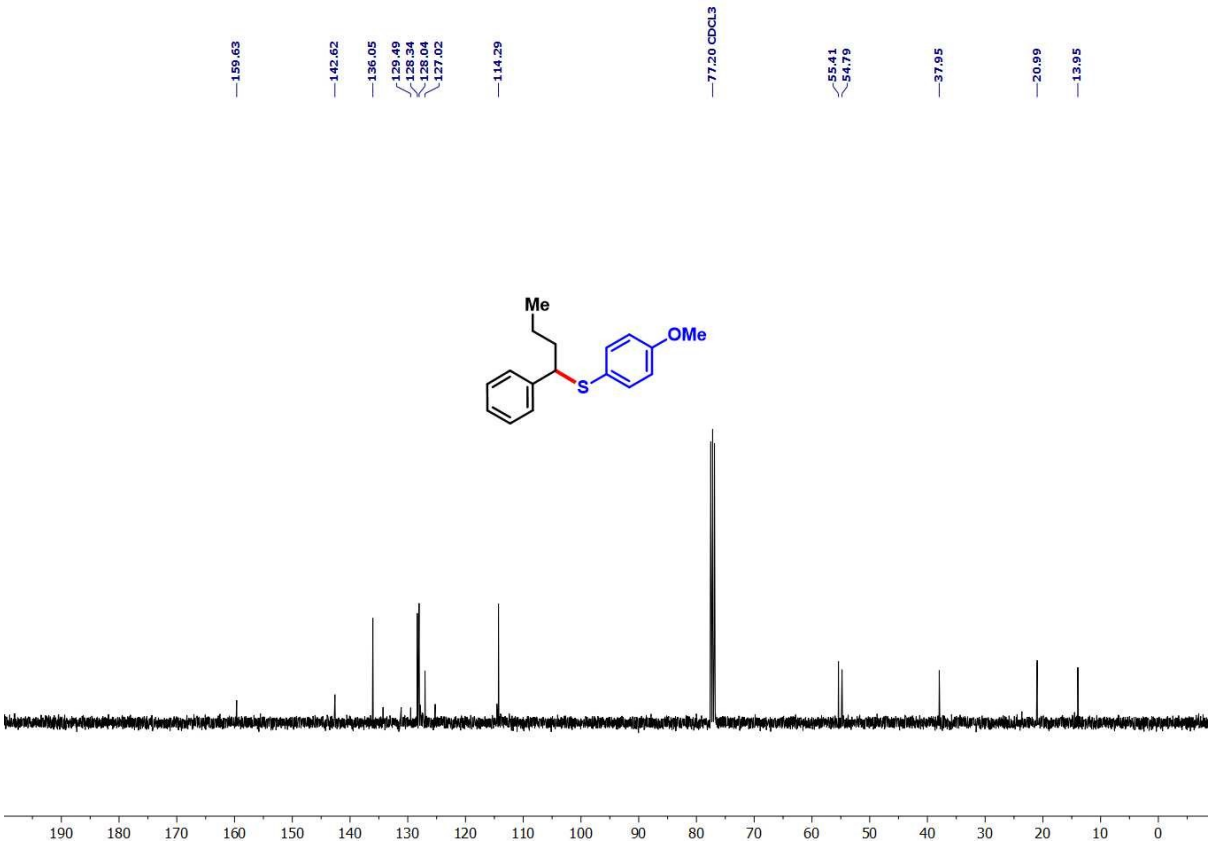
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128.04
127.02
114.29
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55.41
54.79
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20.99
13.95
Me
OMe
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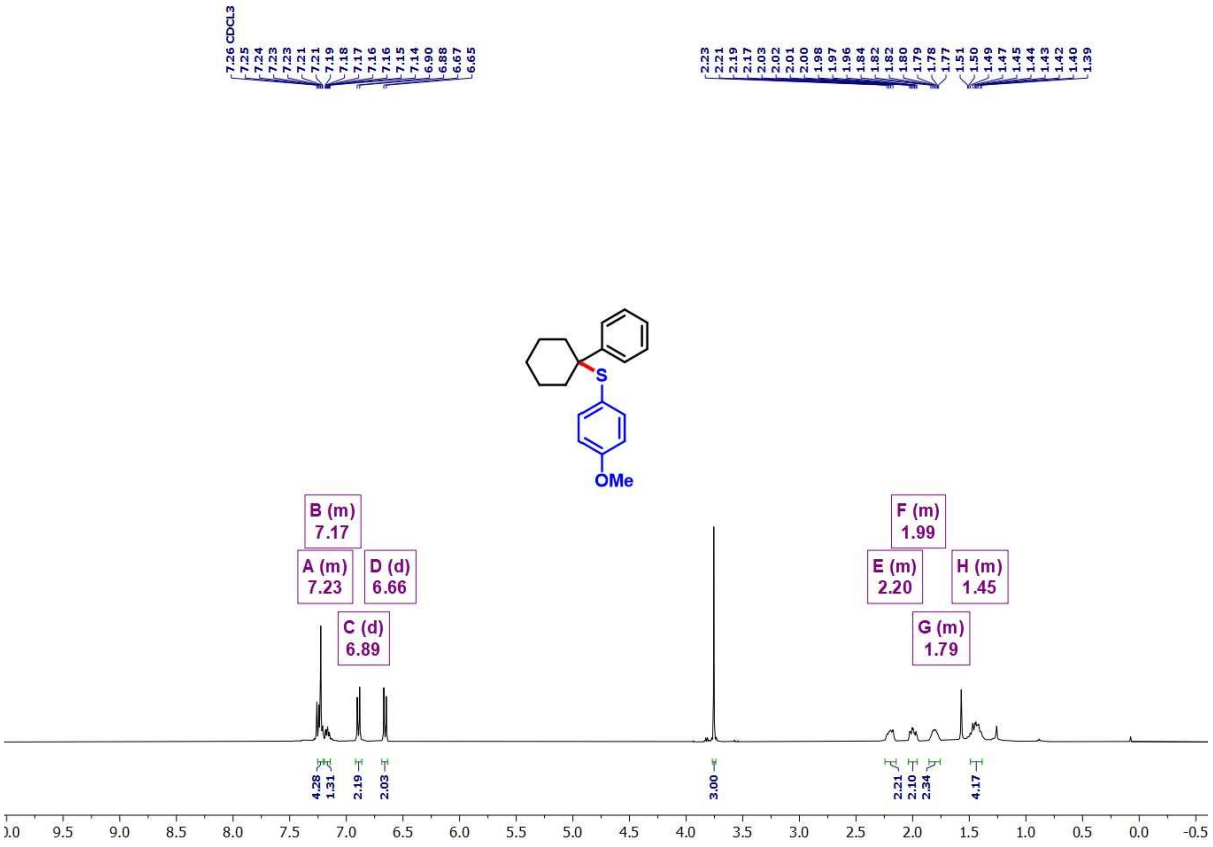
10
0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7.23
7.23
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7.19
7.18
7.17
7.16
7.16
7.15
7.14
6.90
6.88
6.67
6.65
2.23
2.21
2.19
2.17
2.03
2.02
2.01
2.00
1.98
1.97
1.96
1.84
1.82
1.82
1.80
1.79
1.78
1.77
1.51
1.50
1.49
1.47
1.45
1.44
1.43
1.42
1.40
1.39
S
OMe
B (m)
7.17
A (m)
7.23
D (d)
6.66
C (d)
6.89
F (m)
1.99
E (m)
2.20
H (m)
1.45
G (m)
1.79
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1.31
2.19
2.03
3.00
2.21
2.10
2.34
4.17
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9.0
8.5
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7.5
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6.0
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0.0
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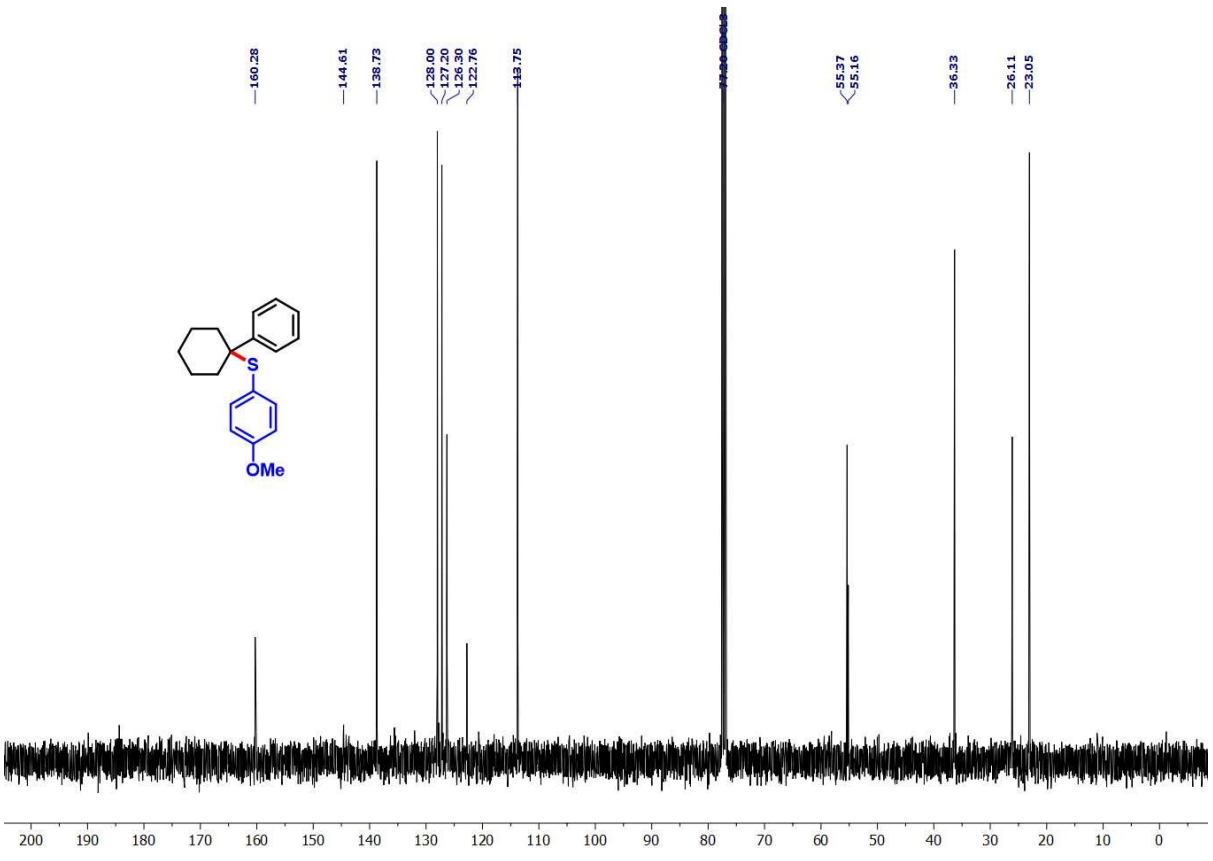
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122.76
113.75
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55.16
36.33
26.11
23.05
S
OMe
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180
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150
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110
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30
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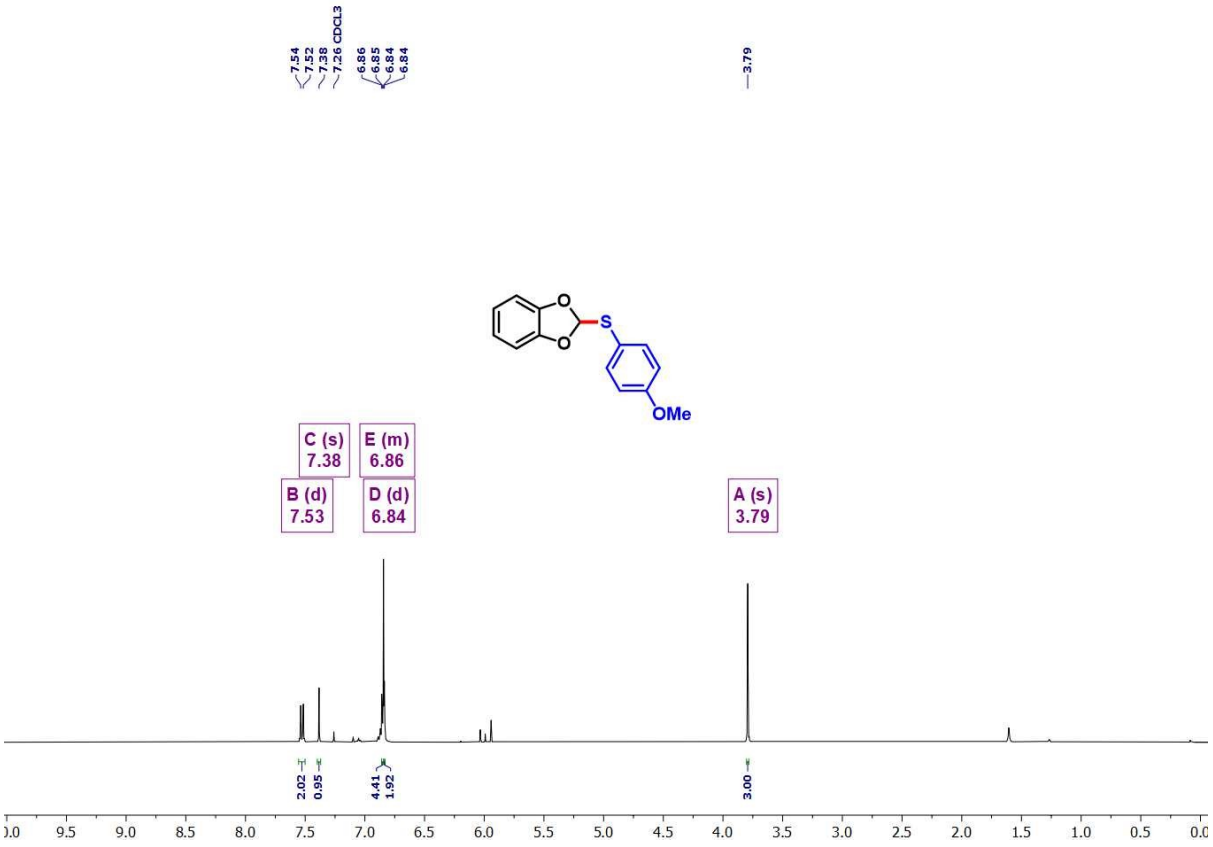
10
0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7.52
7.38
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6.86
6.85
6.84
6.84
3.79
S
O
O
OMe
C (s)
7.38
E (m)
6.86
B (d)
7.53
D (d)
6.84
A (s)
3.79
2.02
0.95
4.41
1.92
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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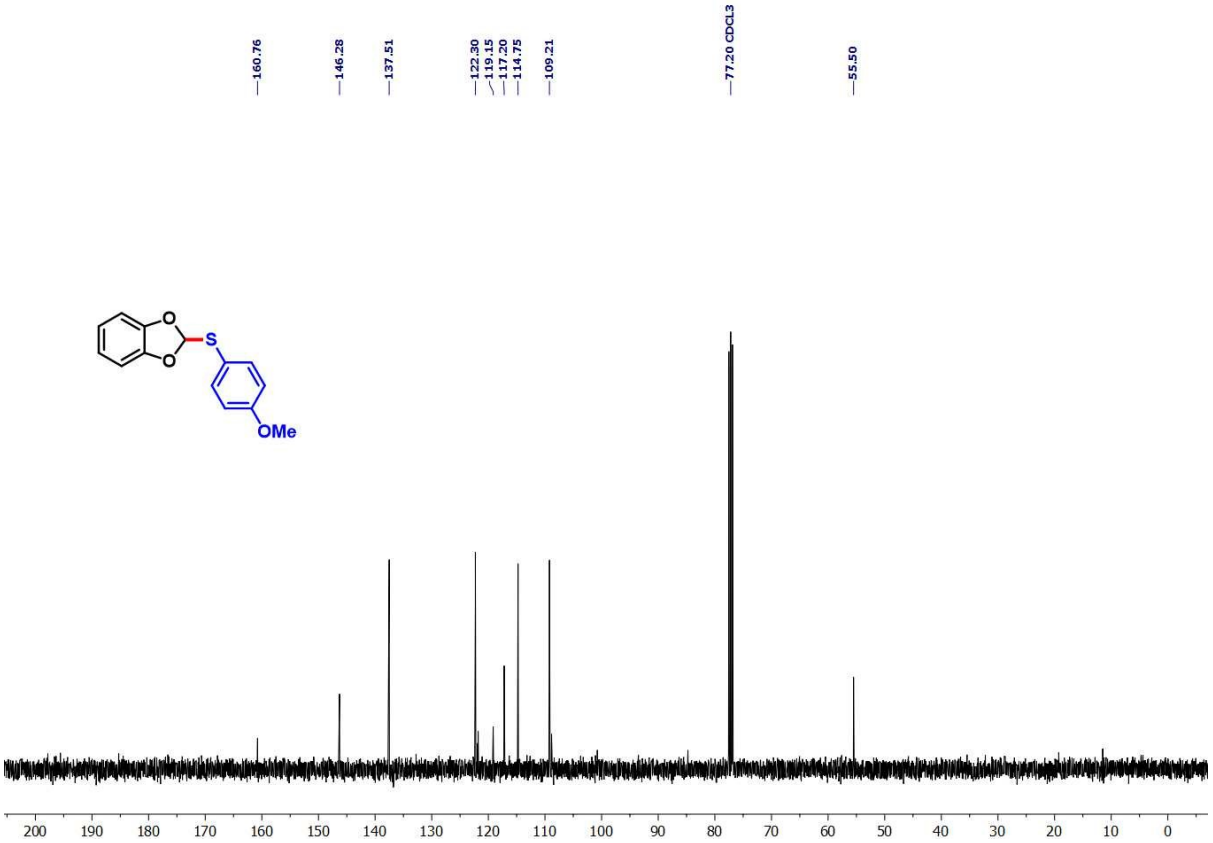
2.0
1.5
1.0
0.5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109.21
77.20 CDCL3
55.50
S
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O
OMe
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10
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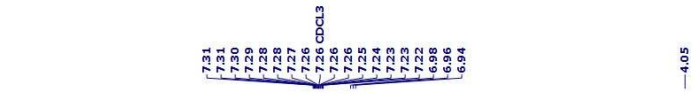
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7.28
7.27
7.26
CDCL3
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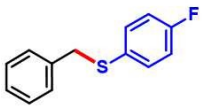
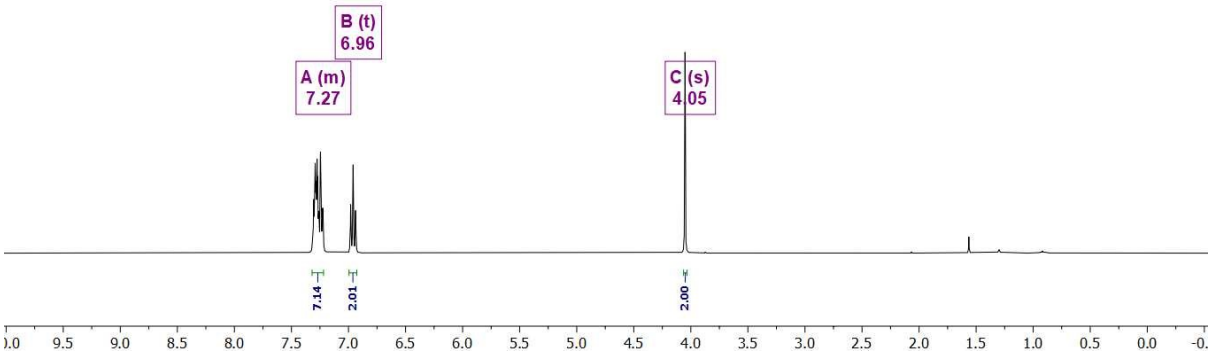
7.26
7.25
7.24
7.23
7.23
7.22
6.98
6.96
6.94
4.05


F
S


B (t)
6.96
A (m)
7.27
C (s)
4.05
7.14
2.01
2.00
9.5
9.0
8.5
8.0
7.5
7.0
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6.0
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5.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


163.45
161.00
137.66
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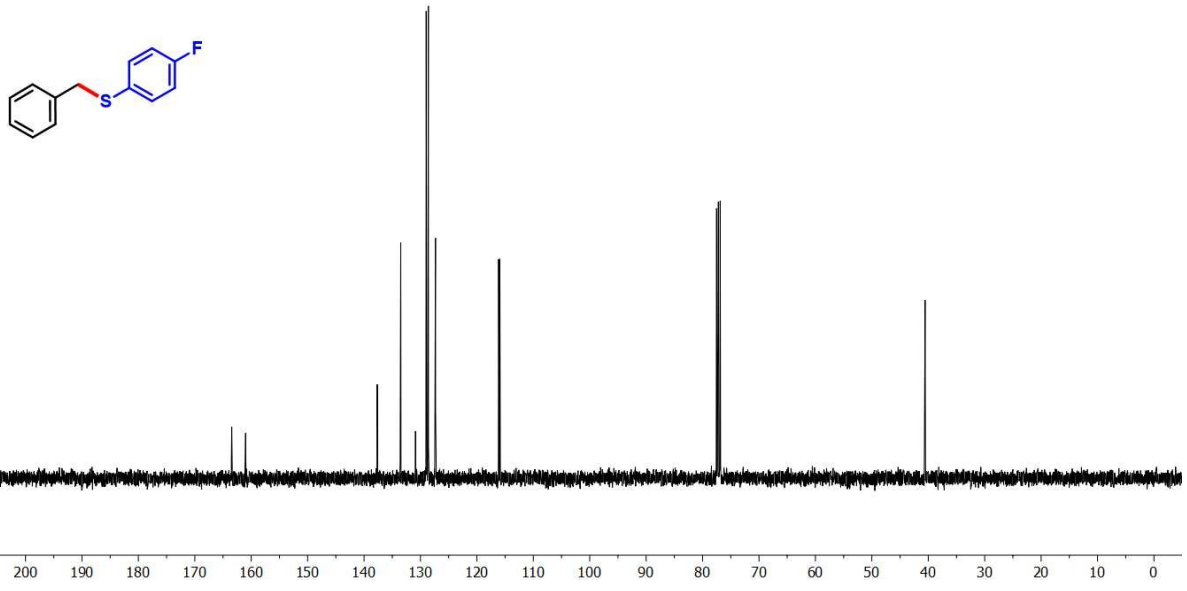
130.88
128.99
128.60
127.33
116.15
115.93
77.20 CDCL3
40.56


F
S
200
190
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60
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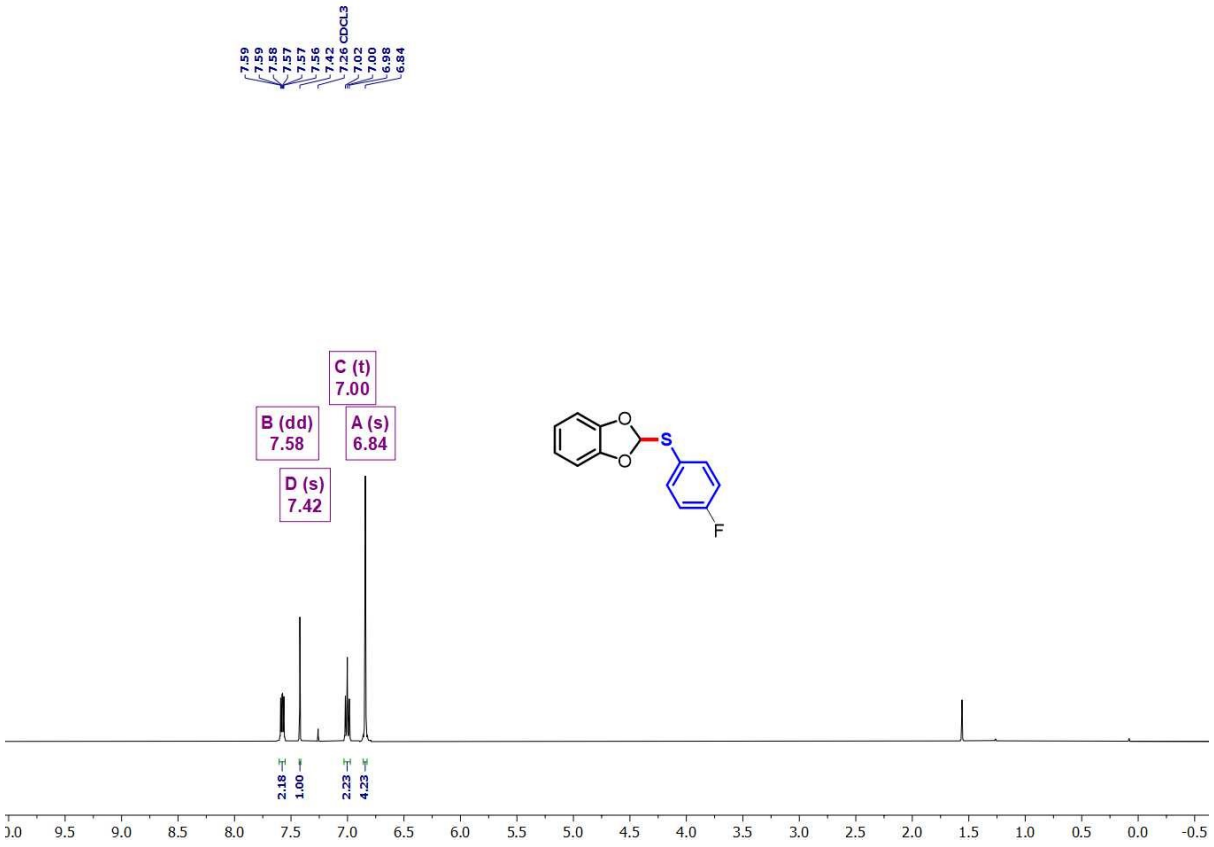
20
10
0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7.02
7.00
6.98
6.84
C (t)
7.00
B (dd)
7.58
A (s)
6.84
D (s)
7.42
S
F
2.18
1.00
2.23
4.23
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9.0
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6.0
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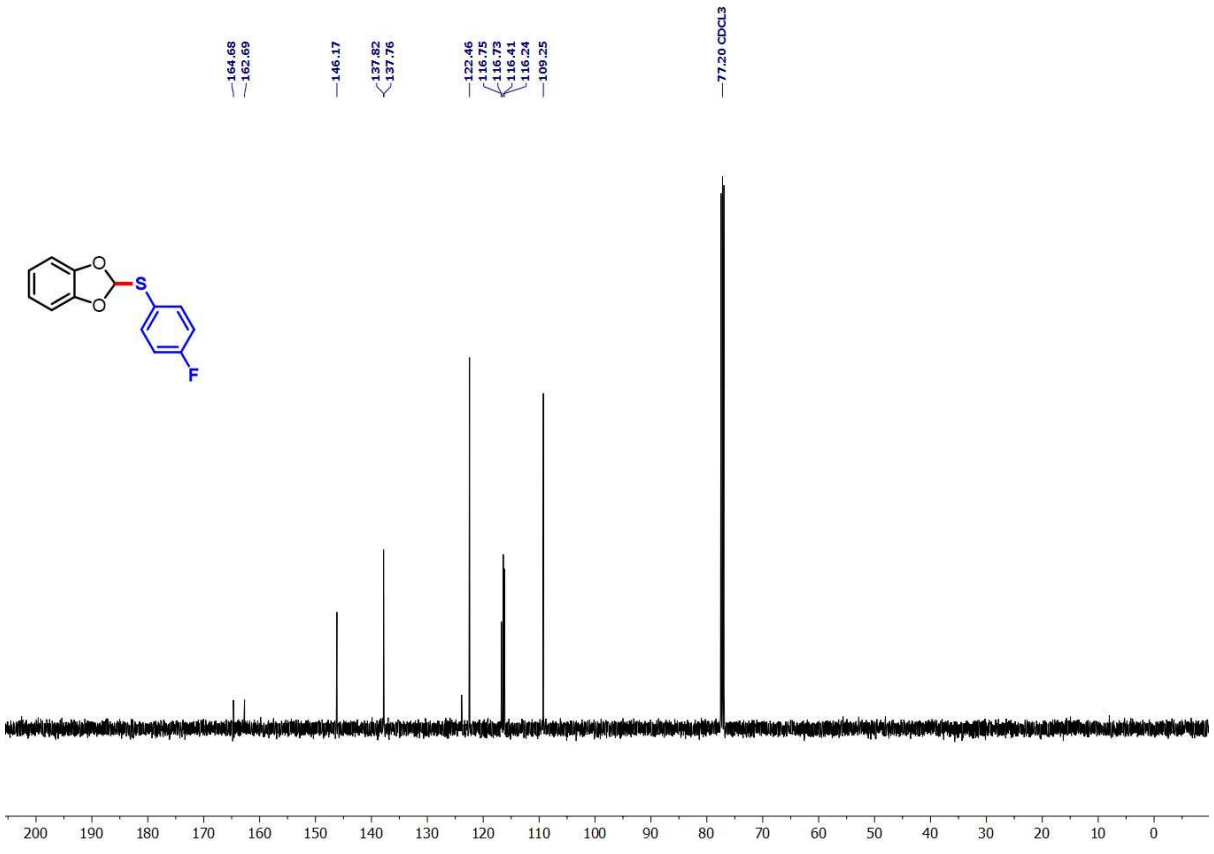
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116.24
109.25
77.20 CDCL3
S
F
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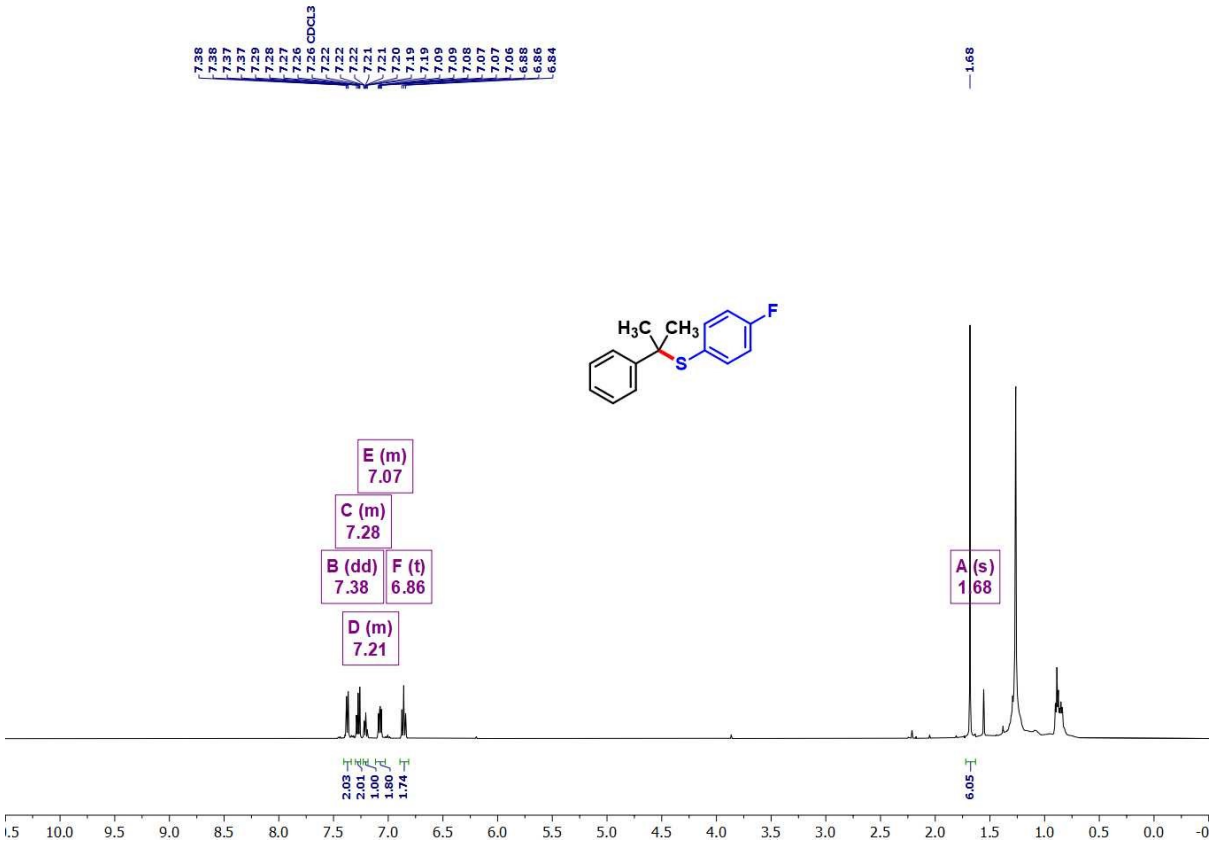
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7.26
7.26 CDCL3
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7.21
7.20
7.19
7.19
7.09
7.09
7.08
7.07
7.07
7.06
6.88
6.86
6.84
1.68
H3C CH3
S
F
E (m)
7.07
C (m)
7.28
B (dd)
7.38
F (t)
6.86
D (m)
7.21
A (s)
1.68
2.03
2.01
1.00
1.80
1.74
6.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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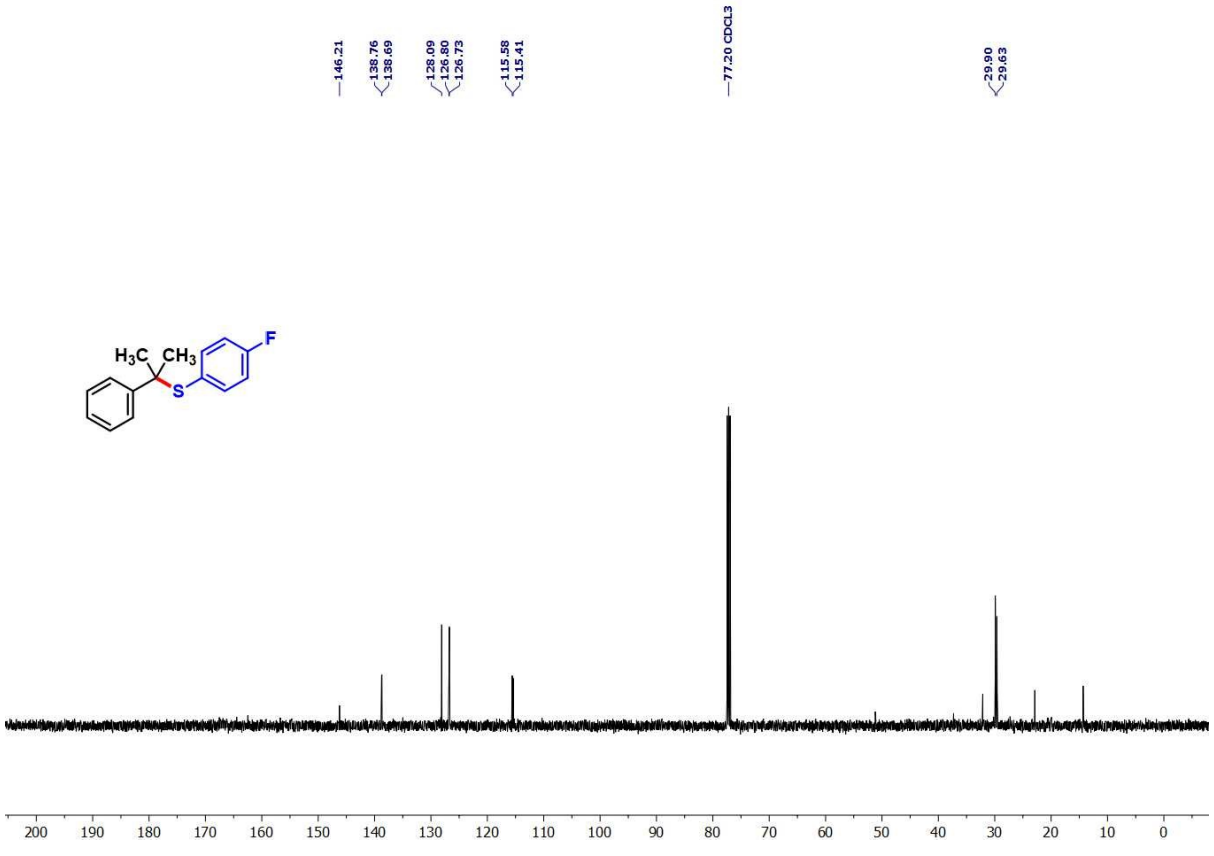
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115.41
77.20 CDCL3
29.90
29.63
H3C CH3
S
F
200
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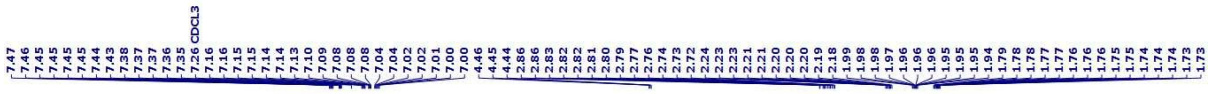
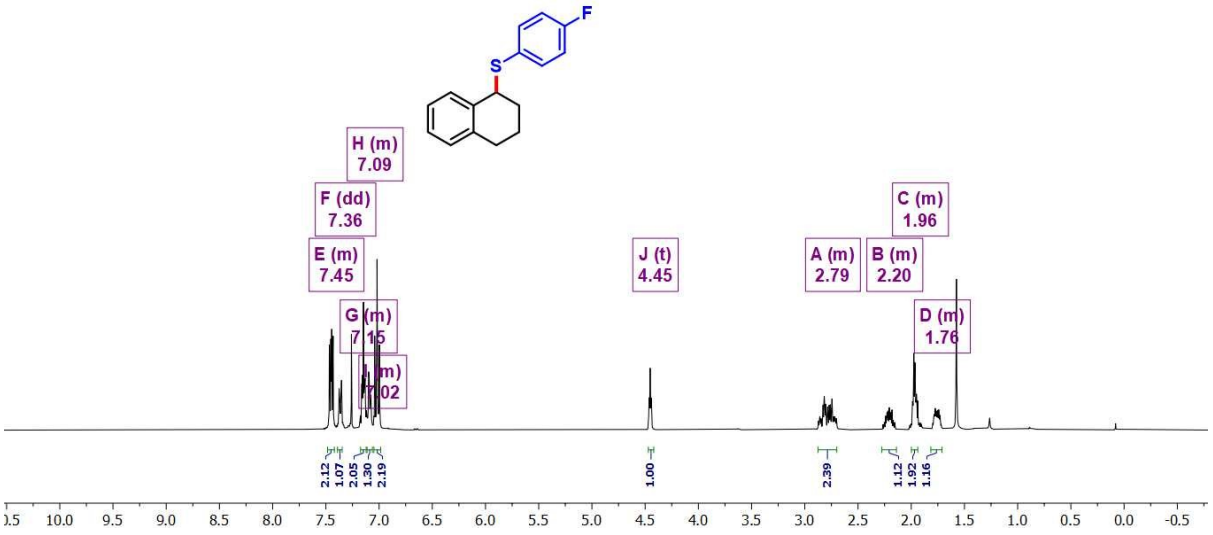
10
0




F
S
H (m)
7.09
F (dd)
7.36
E (m)
7.45
G (m)
7.15
I (m)
7.02
J (t)
4.45
A (m)
2.79
B (m)
2.20
C (m)
1.96
D (m)
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2.12
1.07
2.05
1.30
2.19
1.00
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1.12
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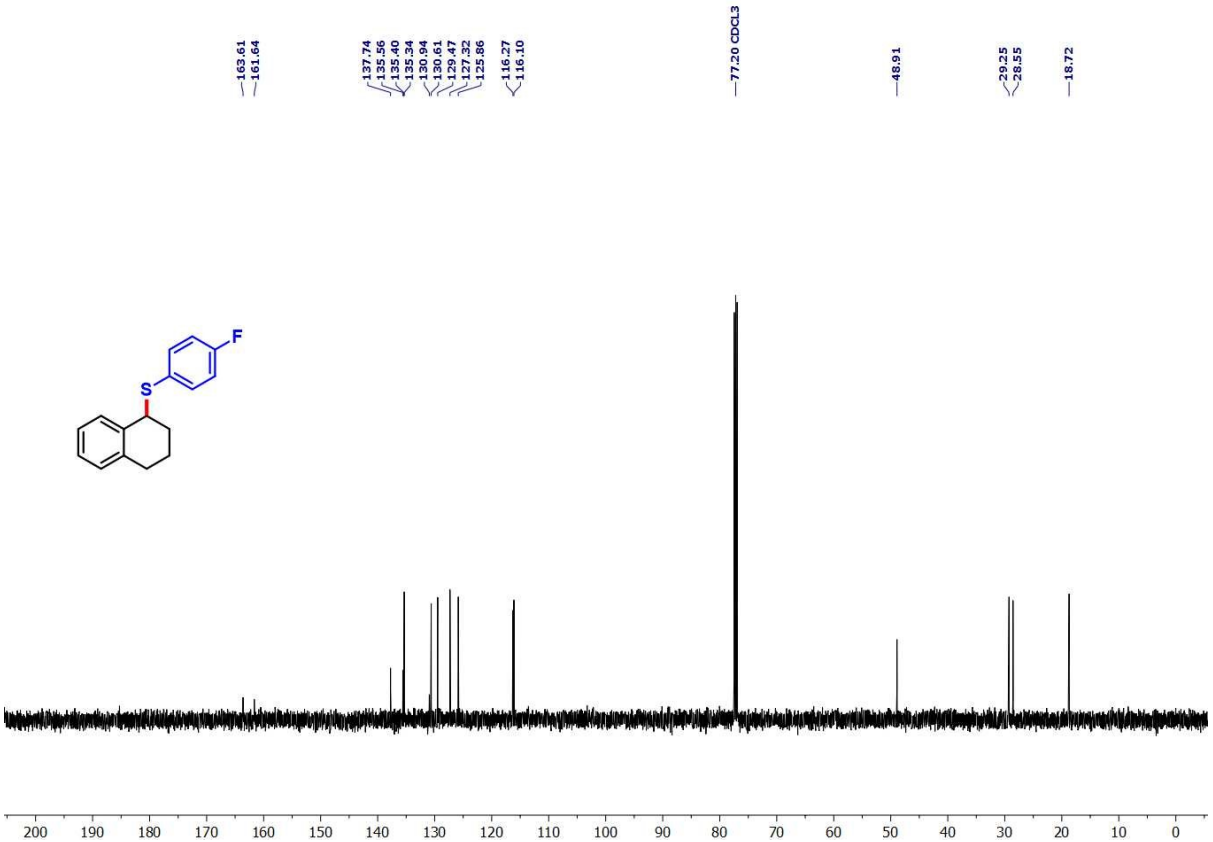
1.16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116.27
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77.20 CDCl3
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29.25
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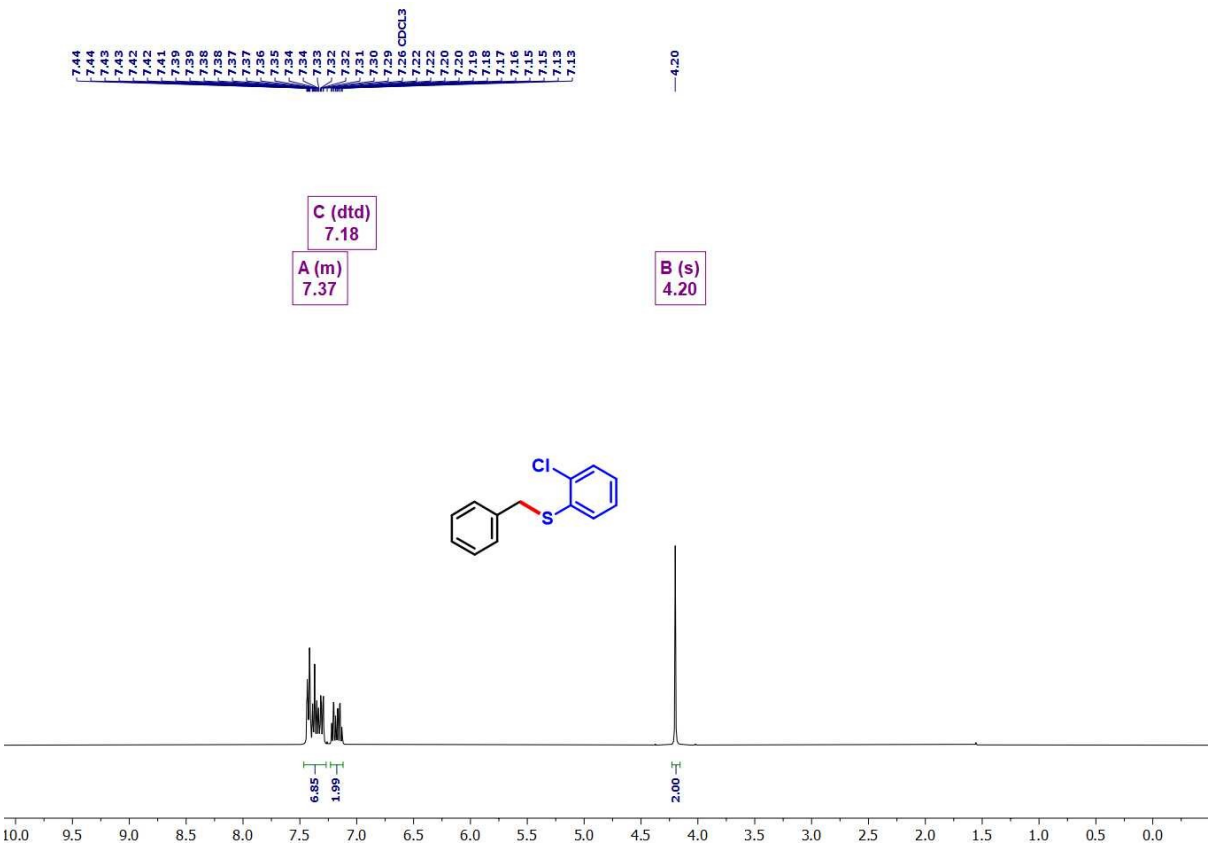
18.72
F
S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CDCL3
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7.15
7.13
7.13
4.20
C (dtd)
7.18
A (m)
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B (s)
4.20
Cl
S
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10.0
9.5
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8.0
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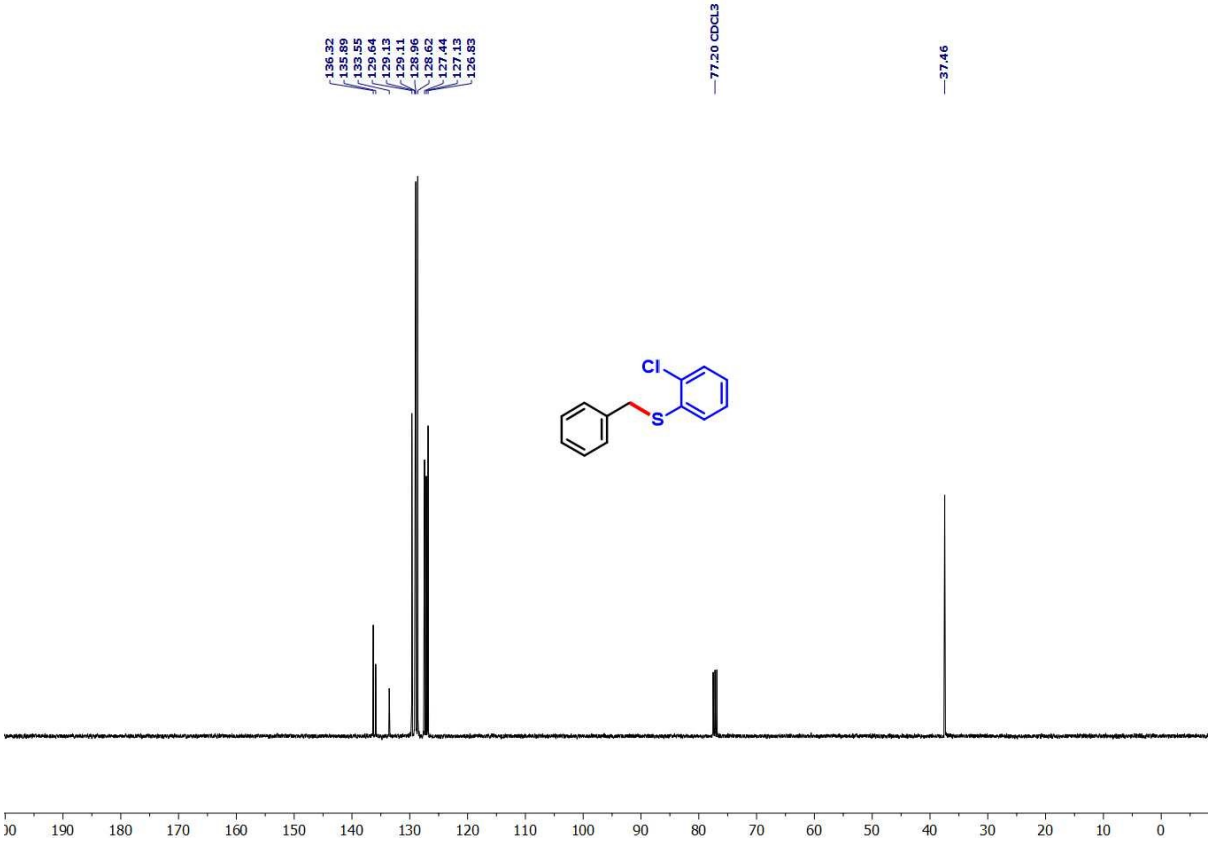
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128.62
127.44
127.13
126.83
77.20 CDCL3
37.46
Cl
S
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150
140
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120
110
100
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70
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30
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10
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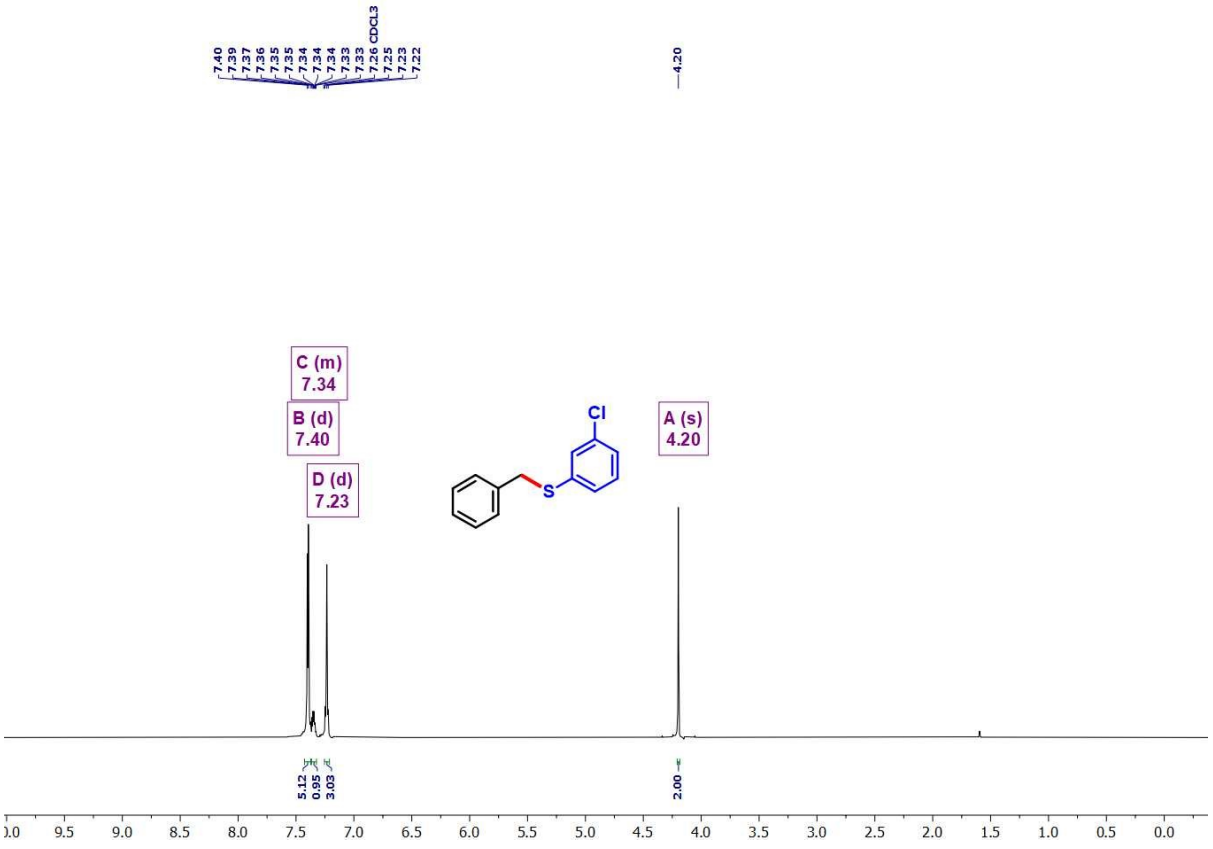
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7.33
7.26 CDCL3
7.25
7.23
7.22
4.20
C (m)
7.34
B (d)
7.40
D (d)
7.23
A (s)
4.20
Cl
S
5.12
0.95
3.03
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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6.0
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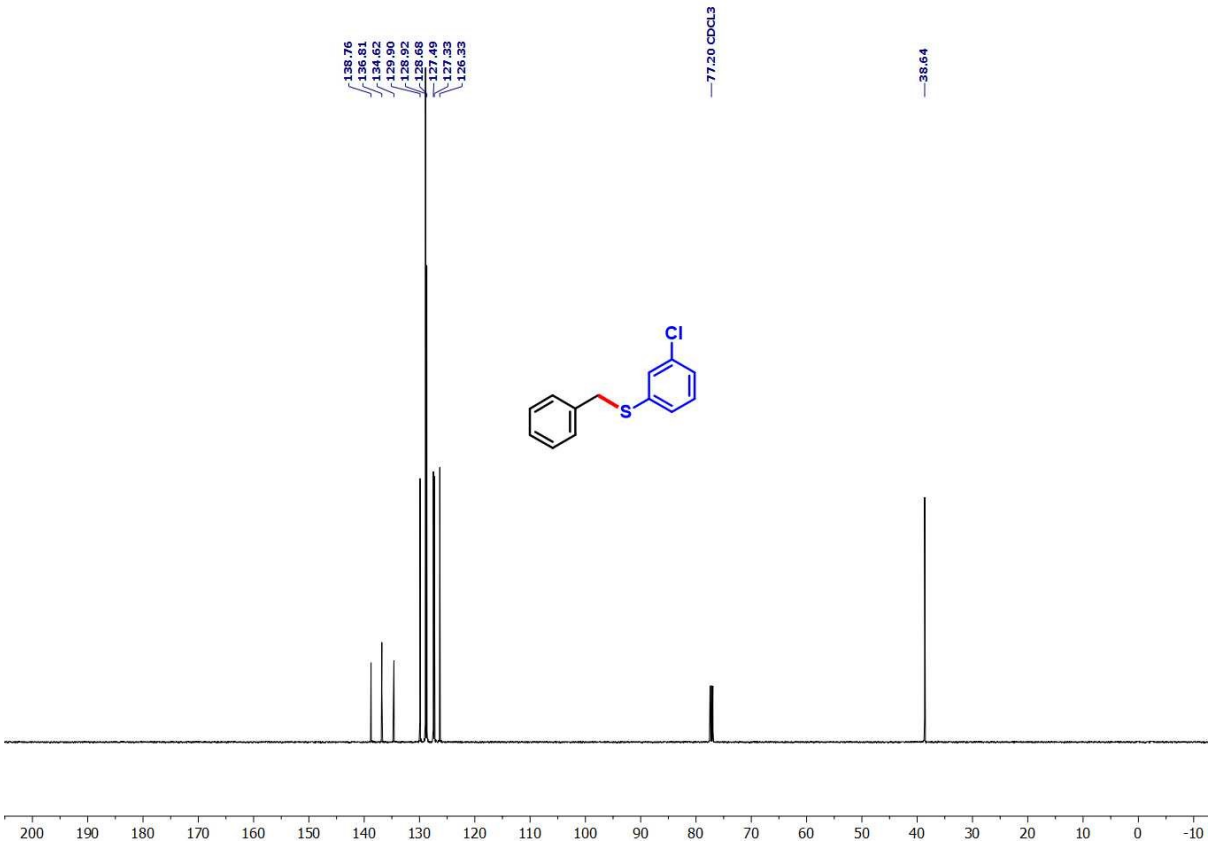
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77.20 CDCL3
38.64
Cl
S
200
190
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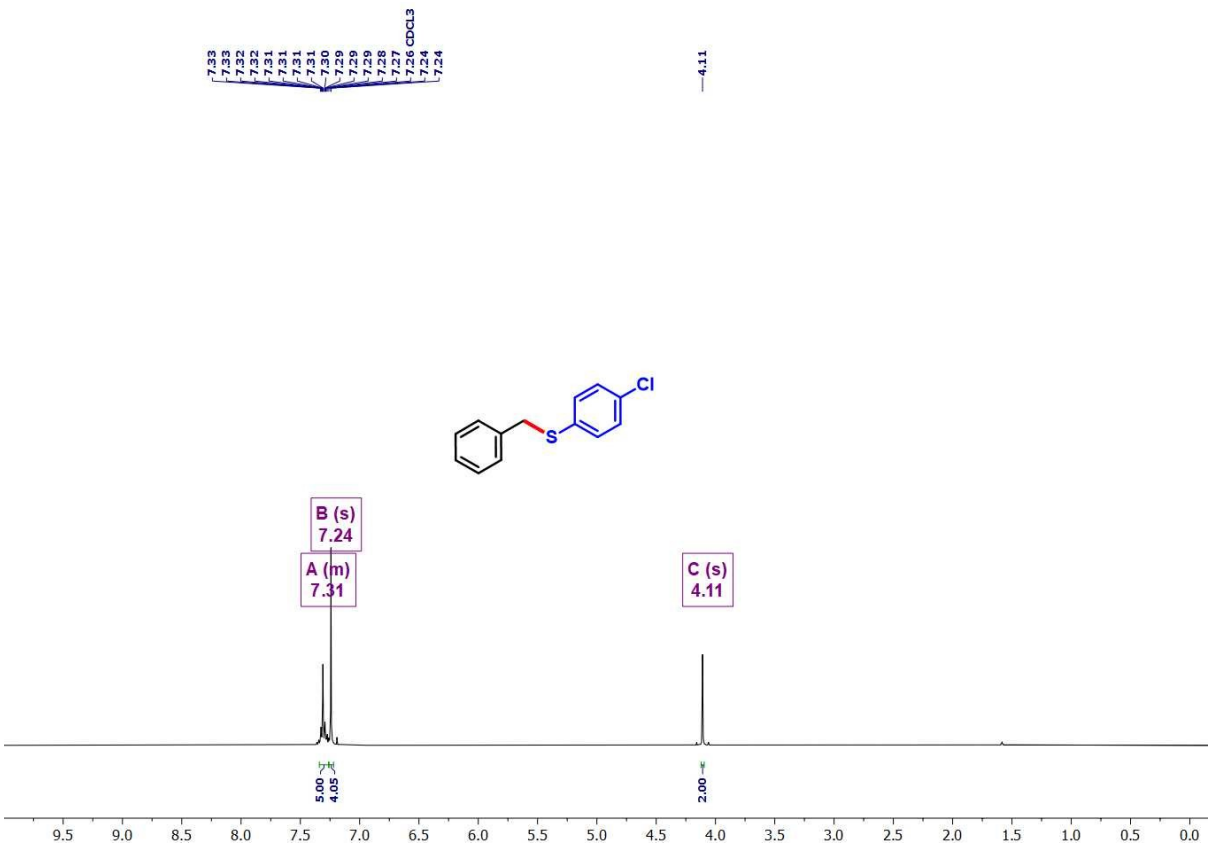
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7.29
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7.28
7.27
7.26 CDCL3
7.24
7.24
4.11
Cl
S
B (s)
7.24
A (m)
7.31
C (s)
4.11
5.00
4.05
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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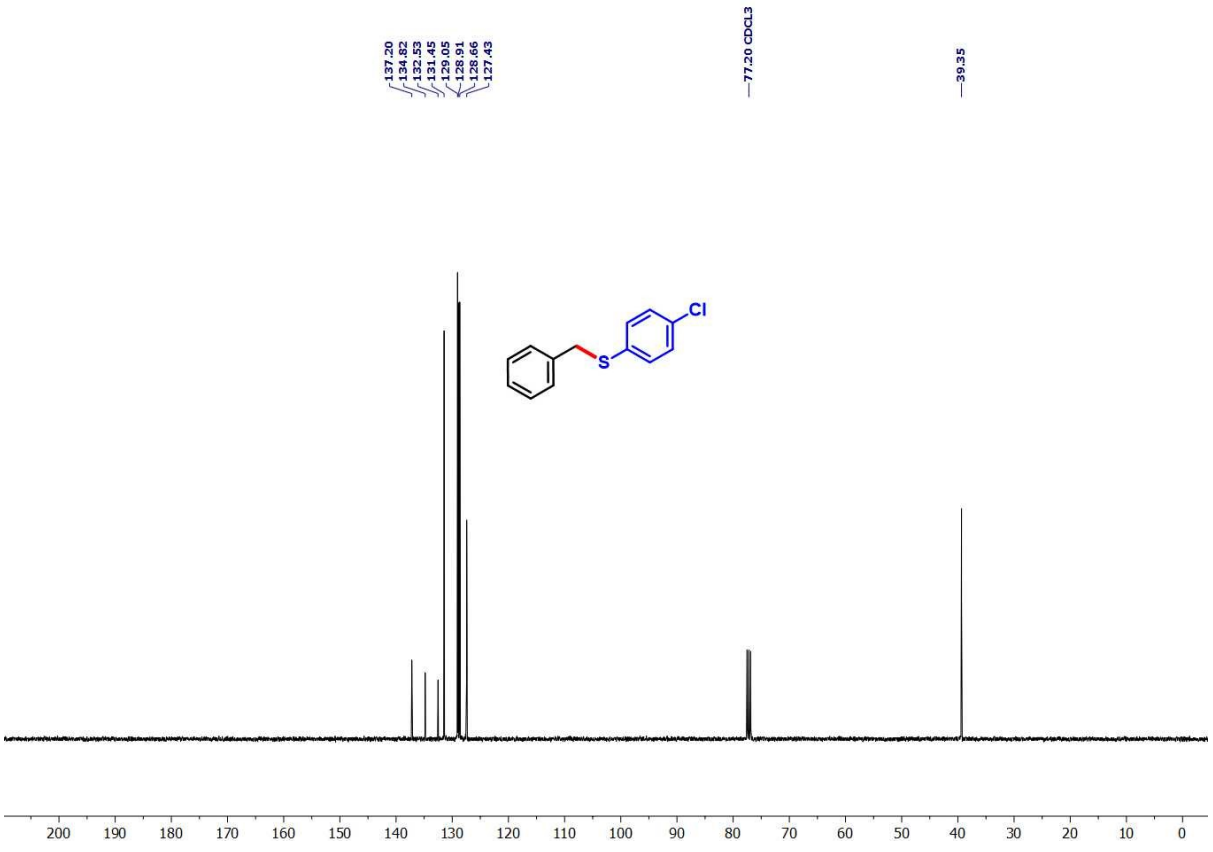
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128.91
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127.43
77.20 CDCL3
39.35
Cl
S
200
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150
140
130
120
110
100
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80
70
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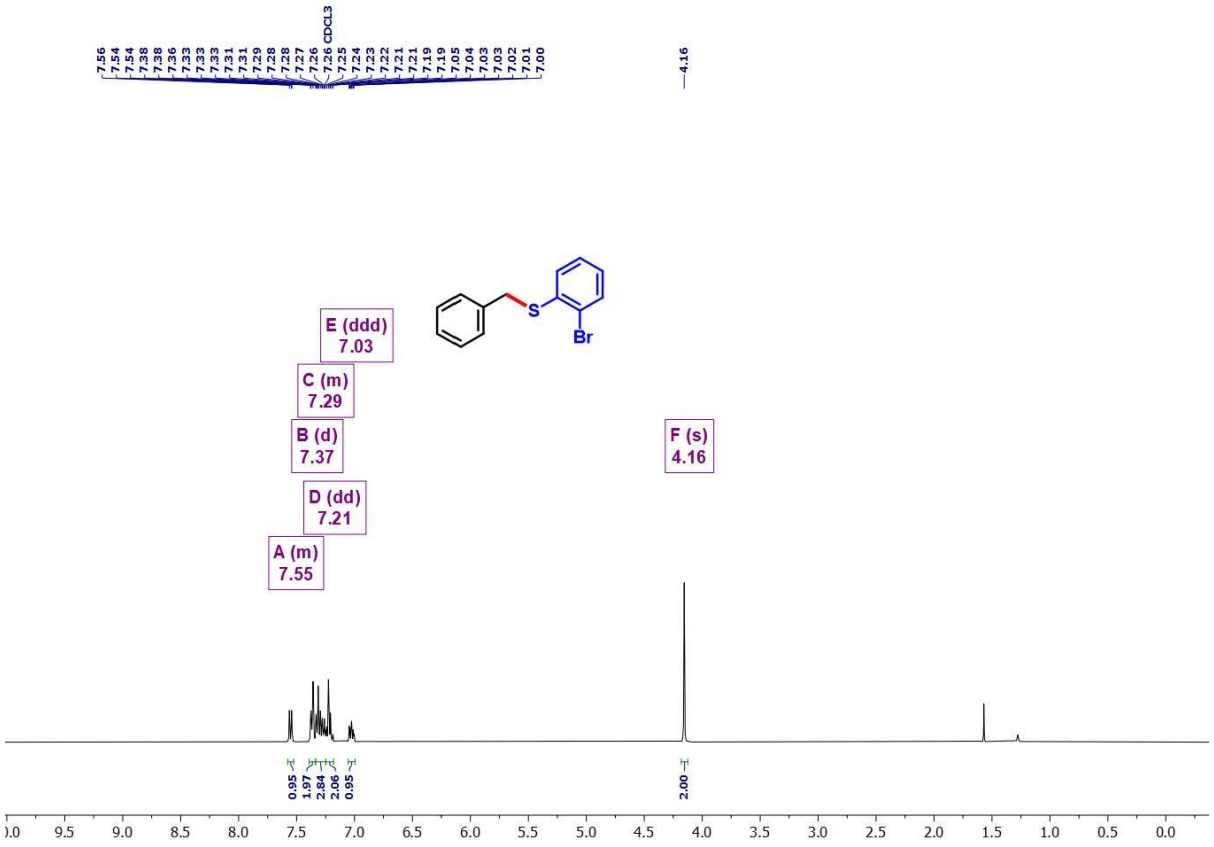
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7.29
7.28
7.28
7.27
7.26
CDCL3
7.26
7.25
7.24
7.23
7.22
7.21
7.21
7.19
7.19
7.05
7.04
7.03
7.03
7.02
7.01
7.00
4.16
S
Br
E (ddd)
7.03
C (m)
7.29
B (d)
7.37
D (dd)
7.21
A (m)
7.55
F (s)
4.16
0.95
1.97
2.84
2.06
0.95
2.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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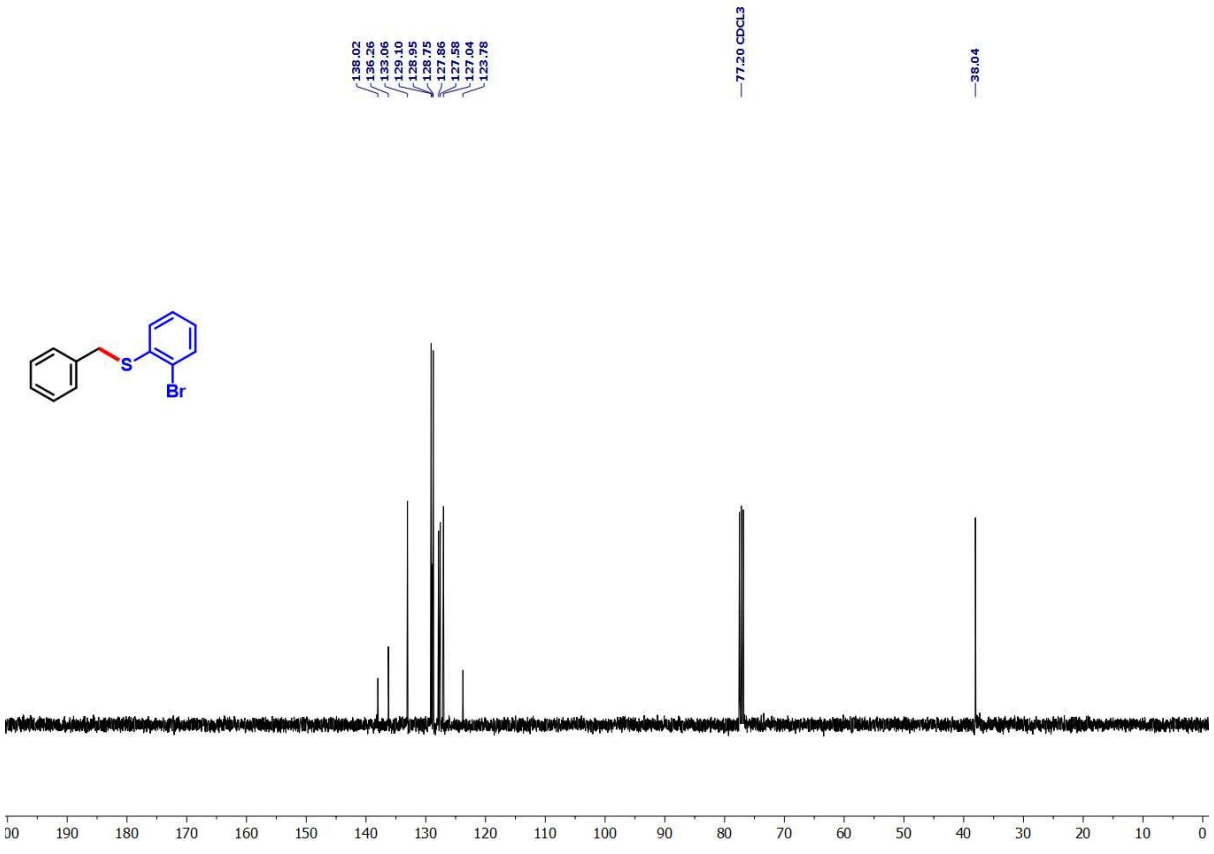
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127.58
127.04
123.78
77.20 CDCL3
38.04
S
Br
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150
140
130
120
110
100
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70
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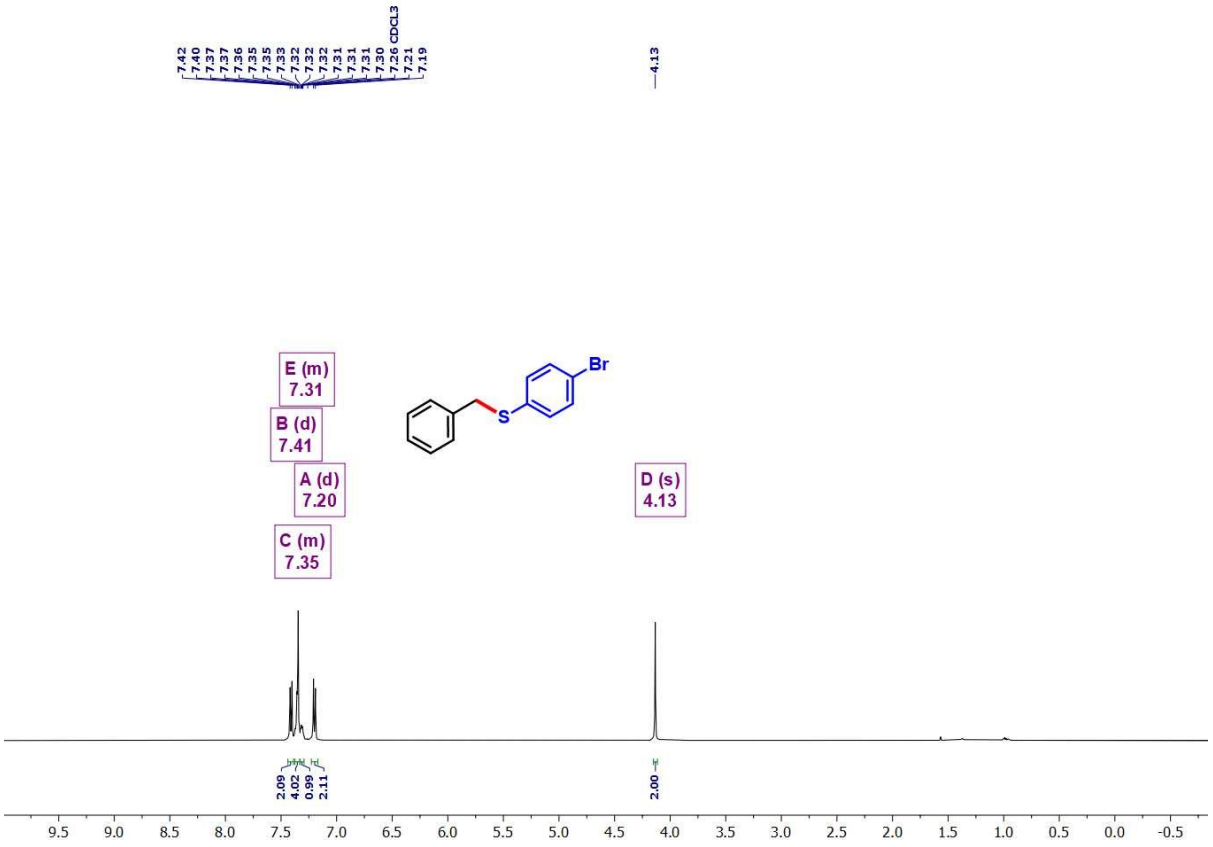
10
0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7.32
7.32
7.31
7.31
7.31
7.30
7.26 CDCL3
7.21
7.19
4.13
E (m)
7.31
B (d)
7.41
A (d)
7.20
C (m)
7.35
D (s)
4.13
Br
S
2.09
4.02
0.99
2.11
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.5
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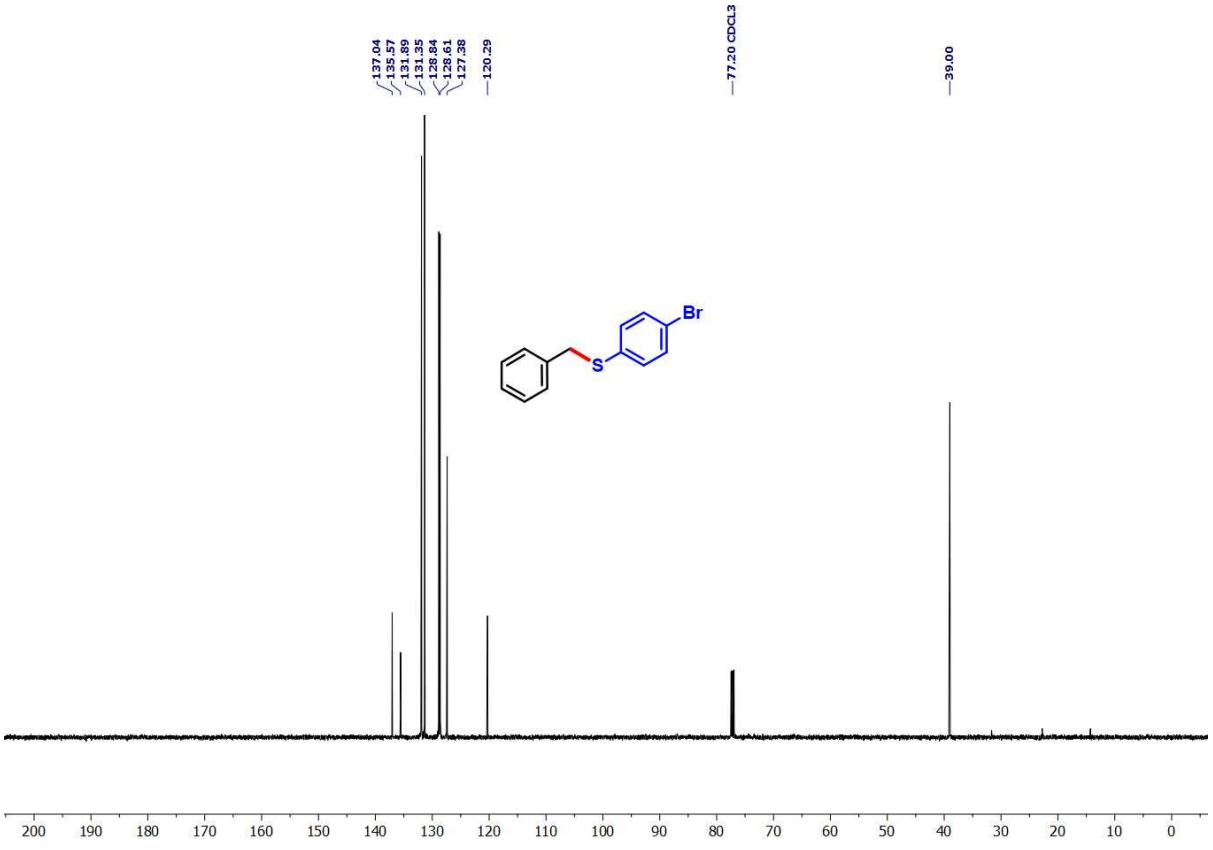
0.5
0.0
-0.5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128.61
127.38
120.29
77.20 CDCL3
39.00
Br
S
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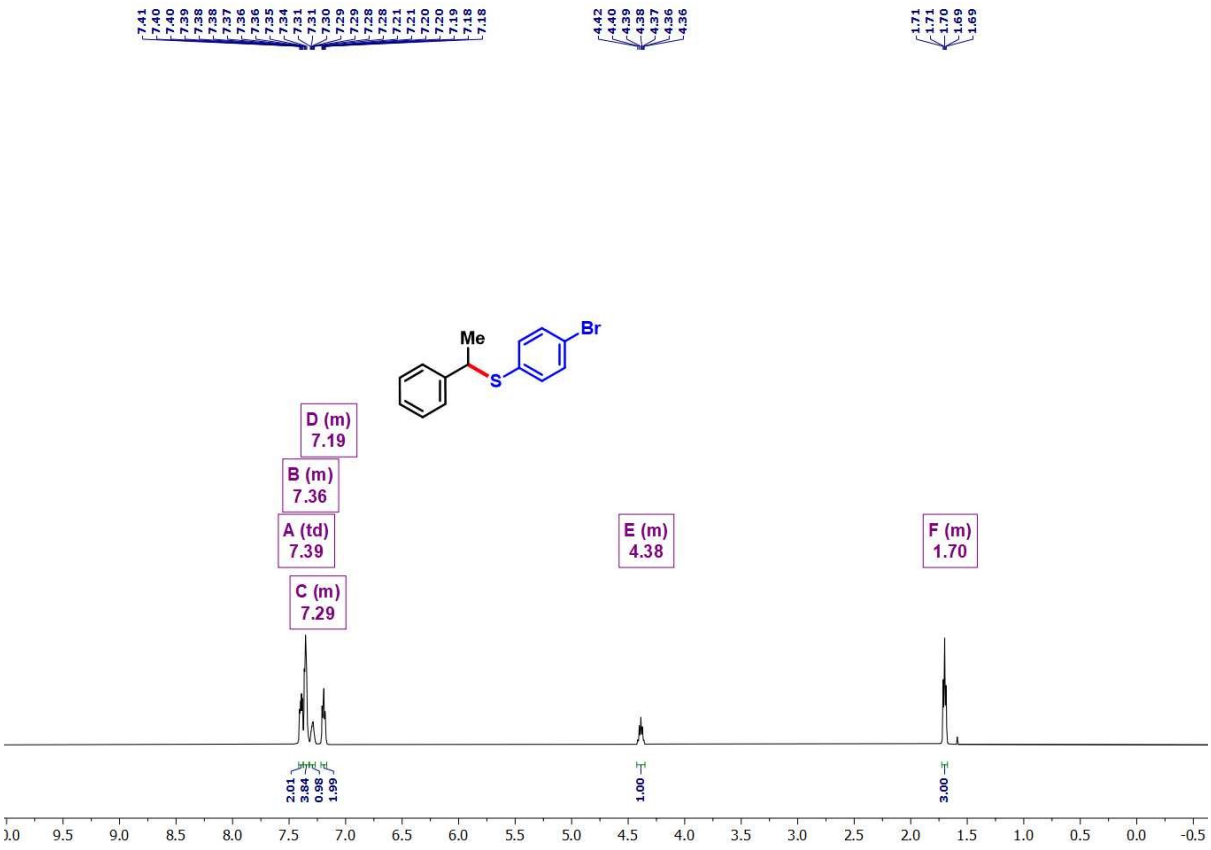
10
0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7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.21
7.21
7.20
7.20
7.19
7.18
7.18
4.42
4.40
4.39
4.38
4.37
4.36
4.36
1.71
1.71
1.70
1.69
1.69
Me
S
Br
D (m)
7.19
B (m)
7.36
A (td)
7.39
C (m)
7.29
E (m)
4.38
F (m)
1.70
2.01
3.84
0.98
1.99
1.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
1.5
1.0
0.5
0.0
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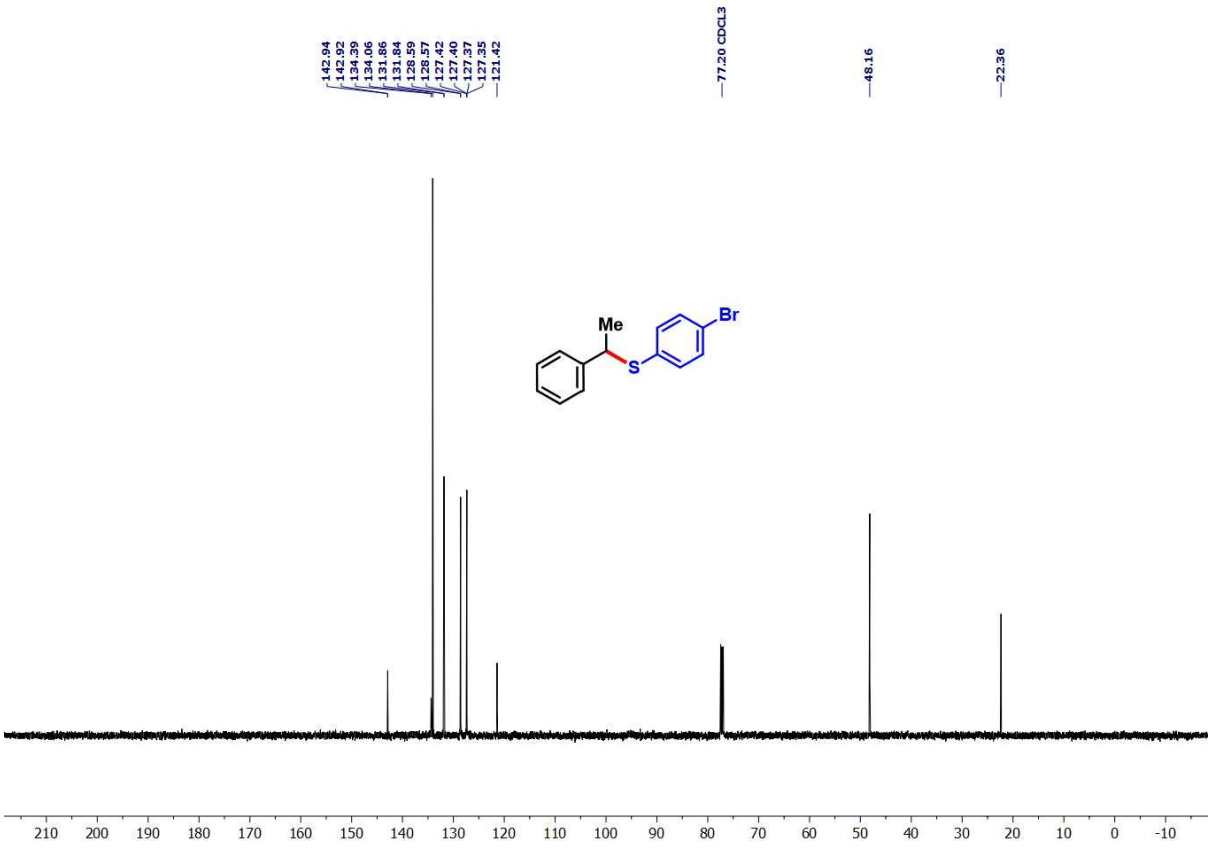
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142.92
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131.84
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128.57
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127.40
127.37
127.35
121.42
77.20 CDCl3
48.16
22.36
Me
S
Br
210
200
190
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140
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100
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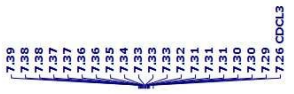
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7.38
7.38
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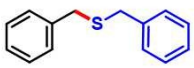
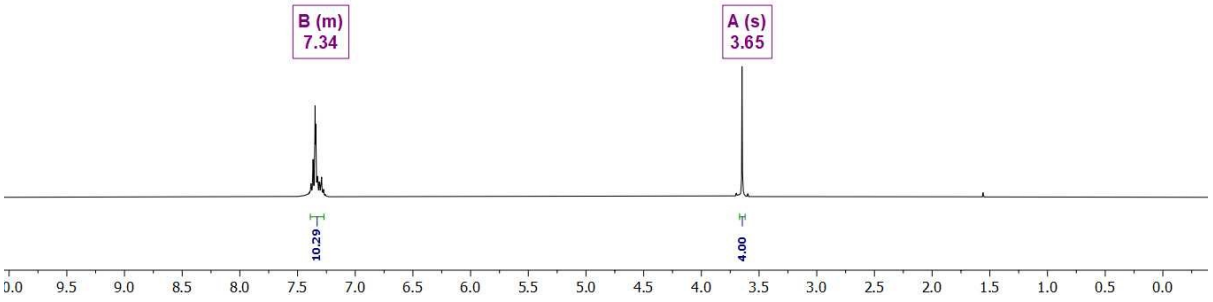
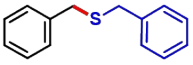
7.31
7.31
7.31
7.30
7.30
7.29
7.26 CDCL3
3.65

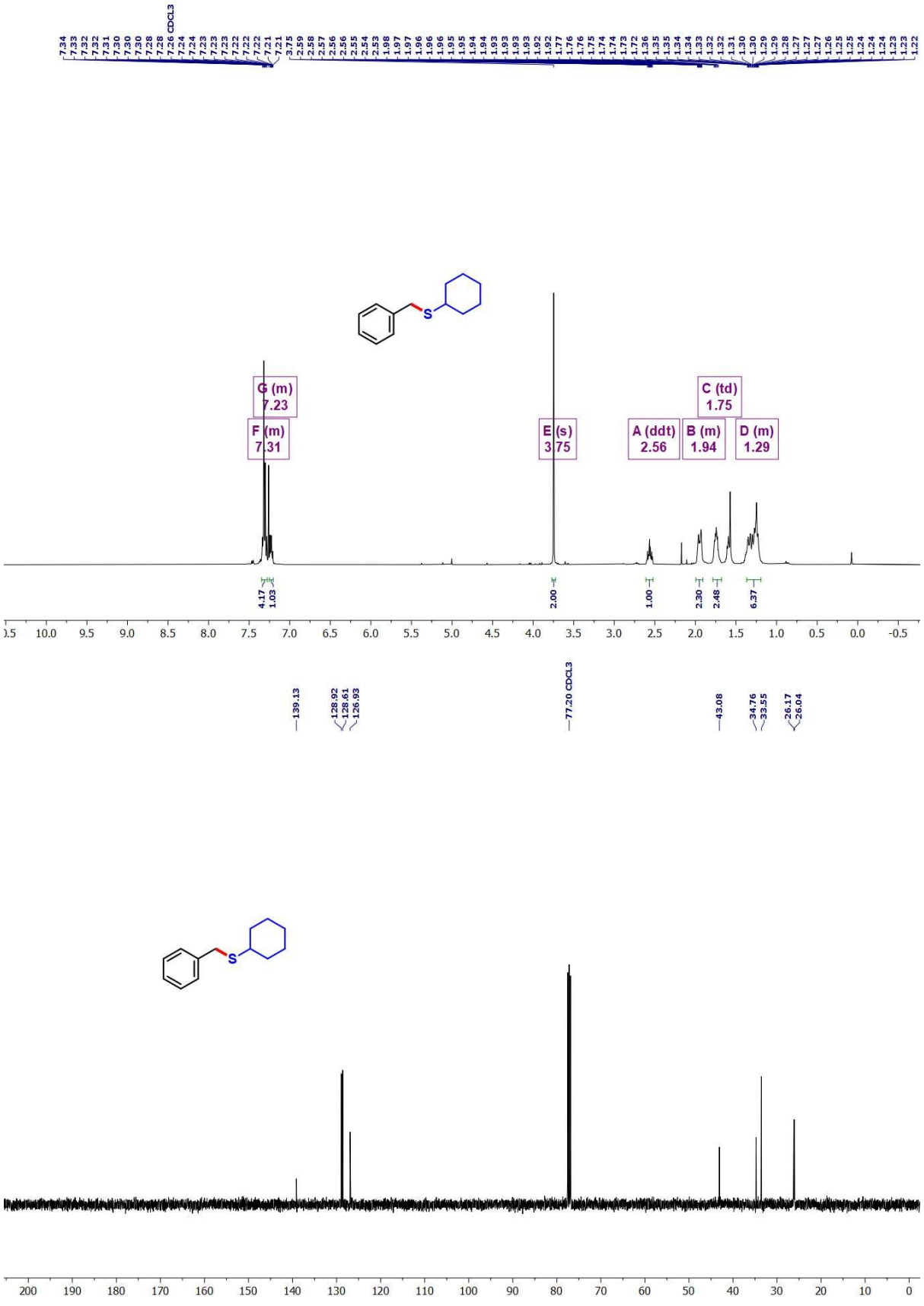

S

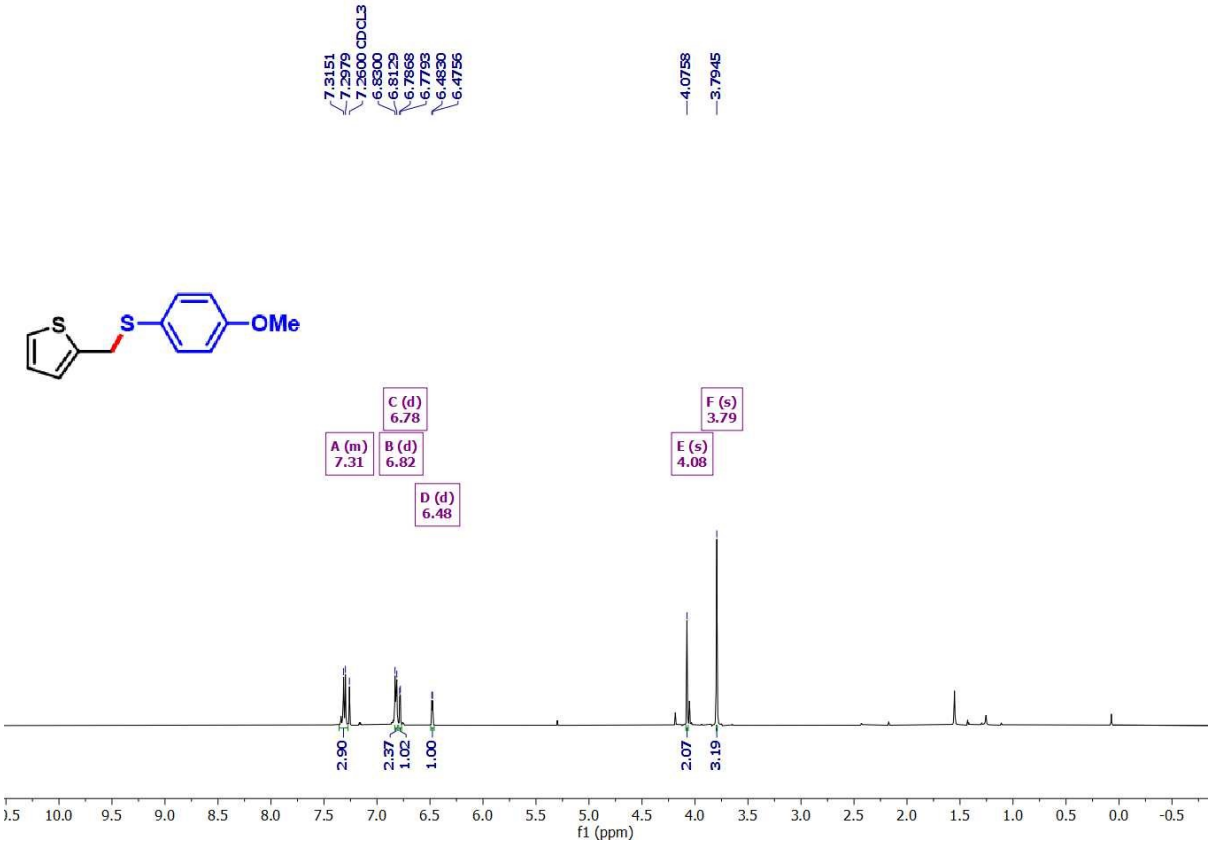

B (m)
7.34
A (s)
3.65
10.29
4.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

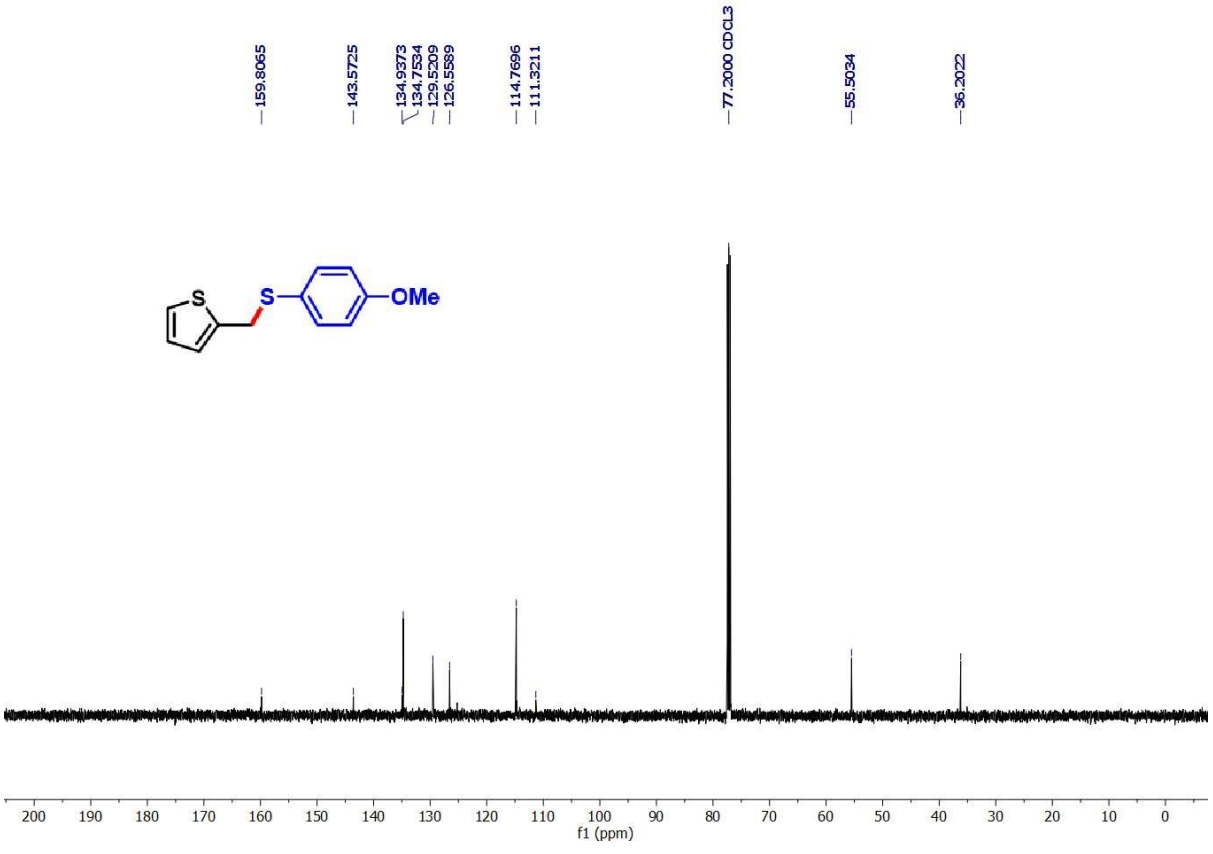

138.28
129.14
128.60
127.10
77.20 CDCL3
35.72


S

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


CDCL3
G (m)
7.23
F (m)
7.31
E (s)
3.75
A (ddt)
2.56
B (m)
1.94
C (td)
1.75
D (m)
1.29
4.17
1.03
2.00
1.00
2.30
2.48
6.37
139.13
128.92
128.61
126.93
77.20 CDCL3
43.08
34.76
33.55
26.17
26.04


7.3151
7.2979
7.2600 CDCL3
6.8300
6.8129
6.7868
6.7793
6.4830
6.4756
4.0758
3.7945
S
S
OMe
C (d)
6.78
A (m)
7.31
B (d)
6.82
D (d)
6.48
F (s)
3.79
E (s)
4.08
2.90
2.37
1.02
1.00
2.07
3.19
f1 (ppm)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134.9373
134.7534
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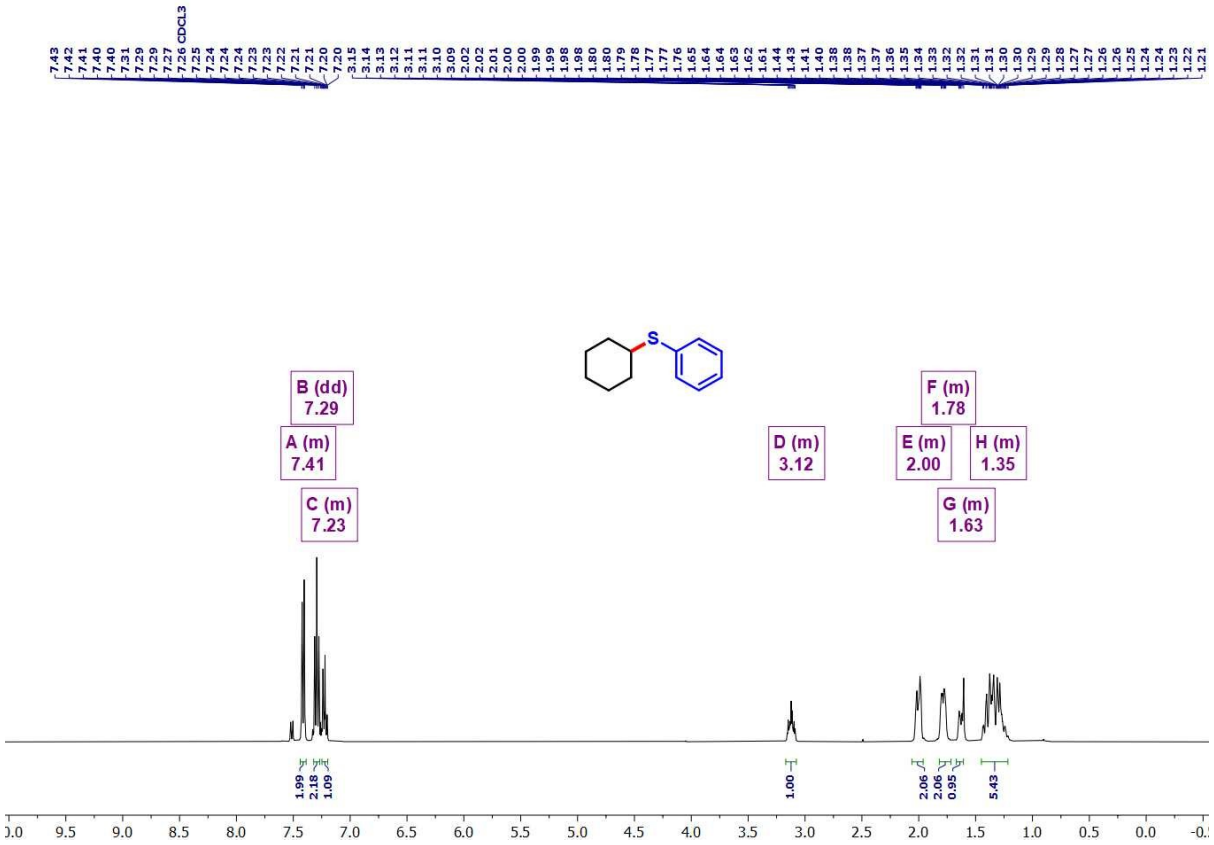
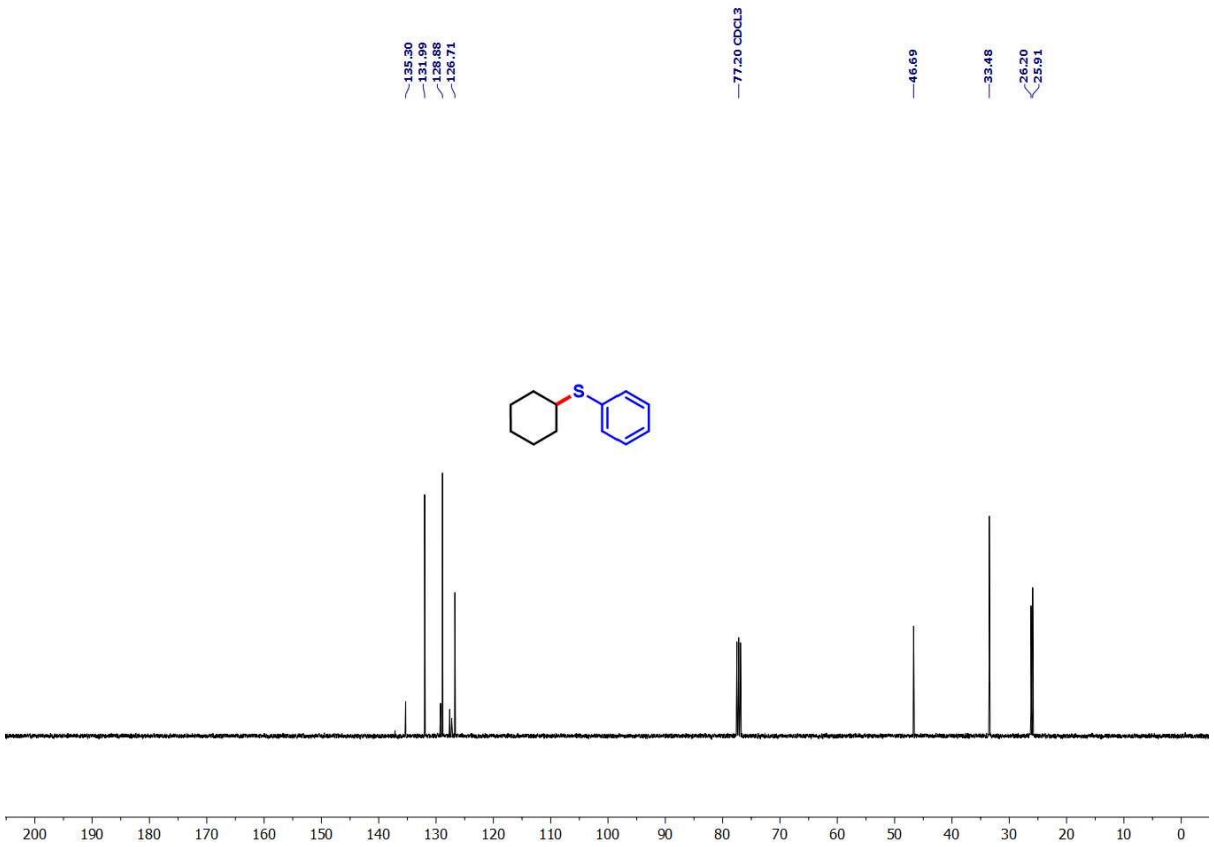
114.7696
111.3211
77.2000 CDCL3
55.5034
36.2022
S
S
OMe
f1 (ppm)


CDCL3
S
B (dd)
7.29
A (m)
7.41
C (m)
7.23
D (m)
3.12
F (m)
1.78
E (m)
2.00
H (m)
1.35
G (m)
1.63
1.99
2.18
1.09
1.00
2.06
2.06
0.95
5.43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126.71
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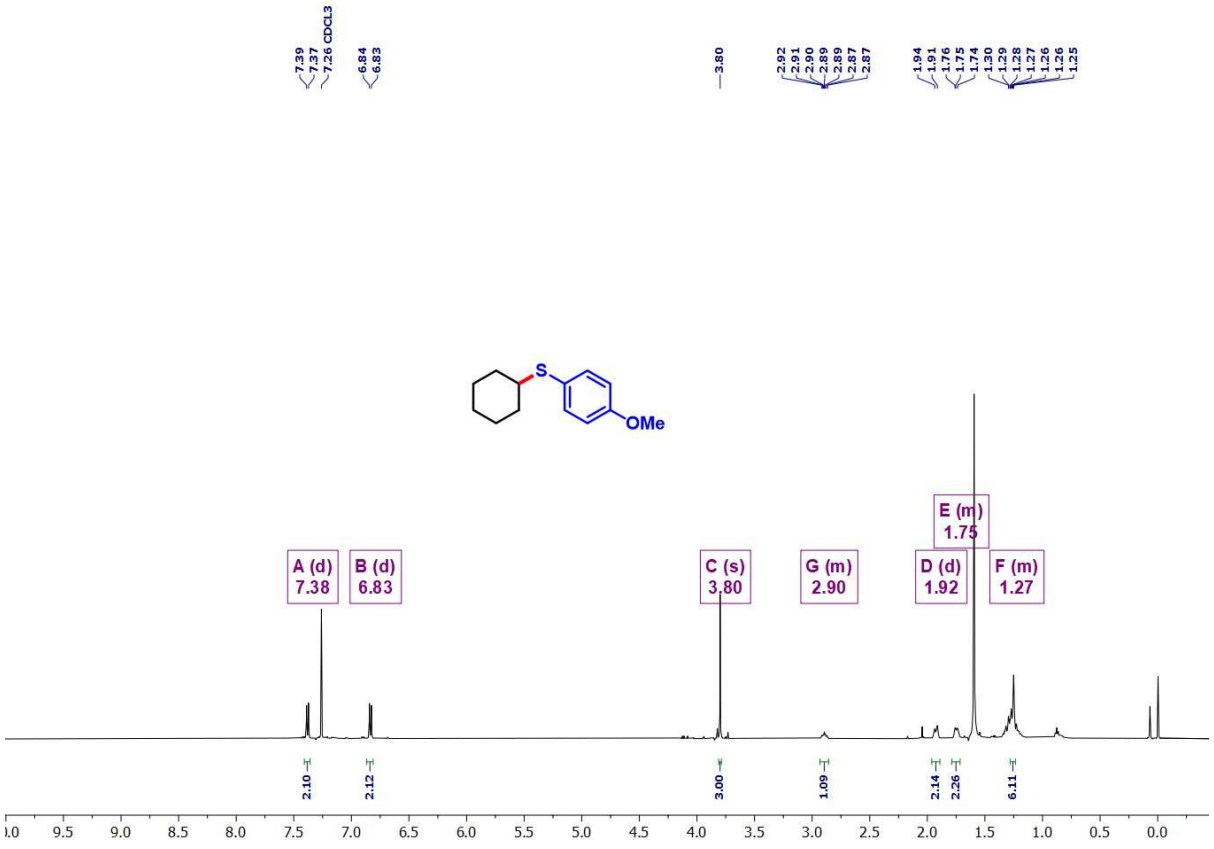
46.69
33.48
26.20
25.91
S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7.37
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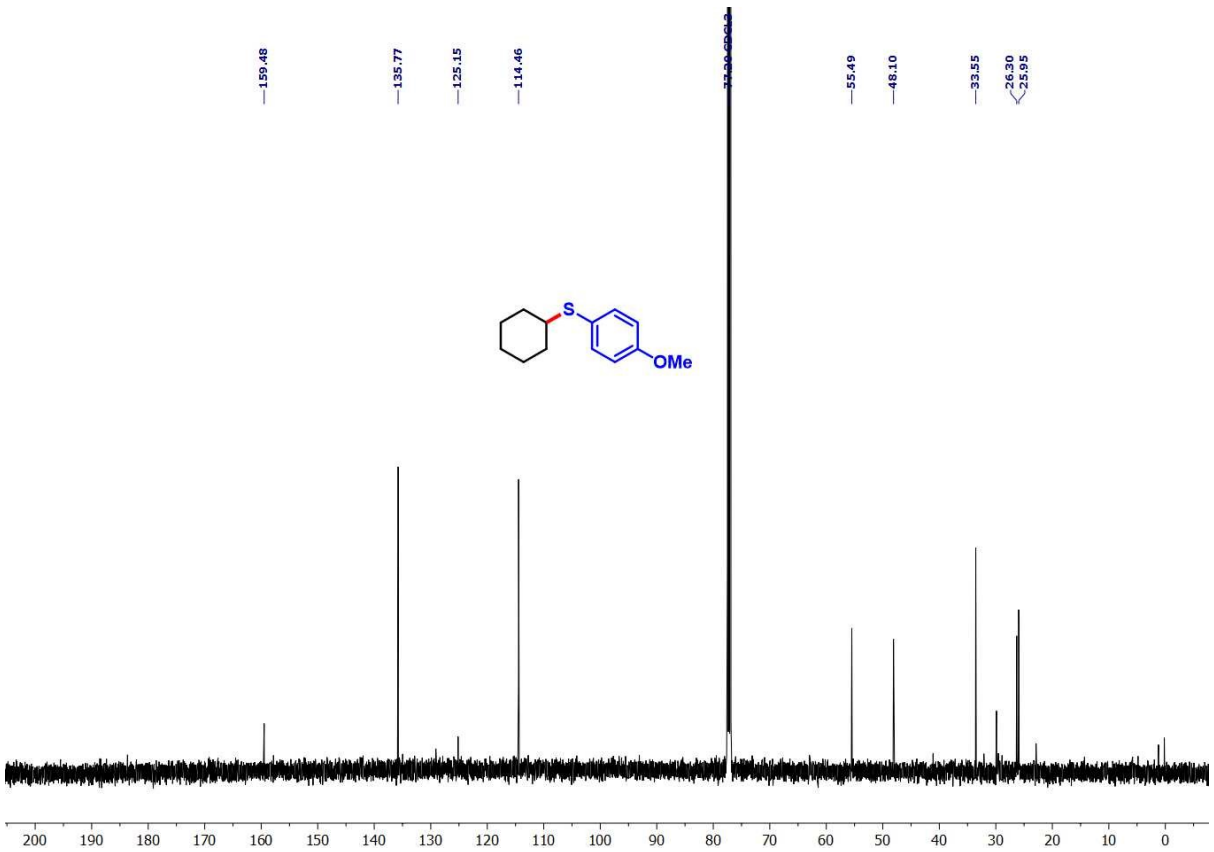
6.84
6.83
3.80
2.92
2.91
2.90
2.89
2.89
2.87
2.87
1.94
1.91
1.76
1.75
1.74
1.30
1.29
1.28
1.27
1.26
1.26
1.25
S
OMe
A (d)
7.38
B (d)
6.83
C (s)
3.80
G (m)
2.90
E (m)
1.75
D (d)
1.92
F (m)
1.27
2.10
2.12
3.00
1.09
2.14
2.26
6.11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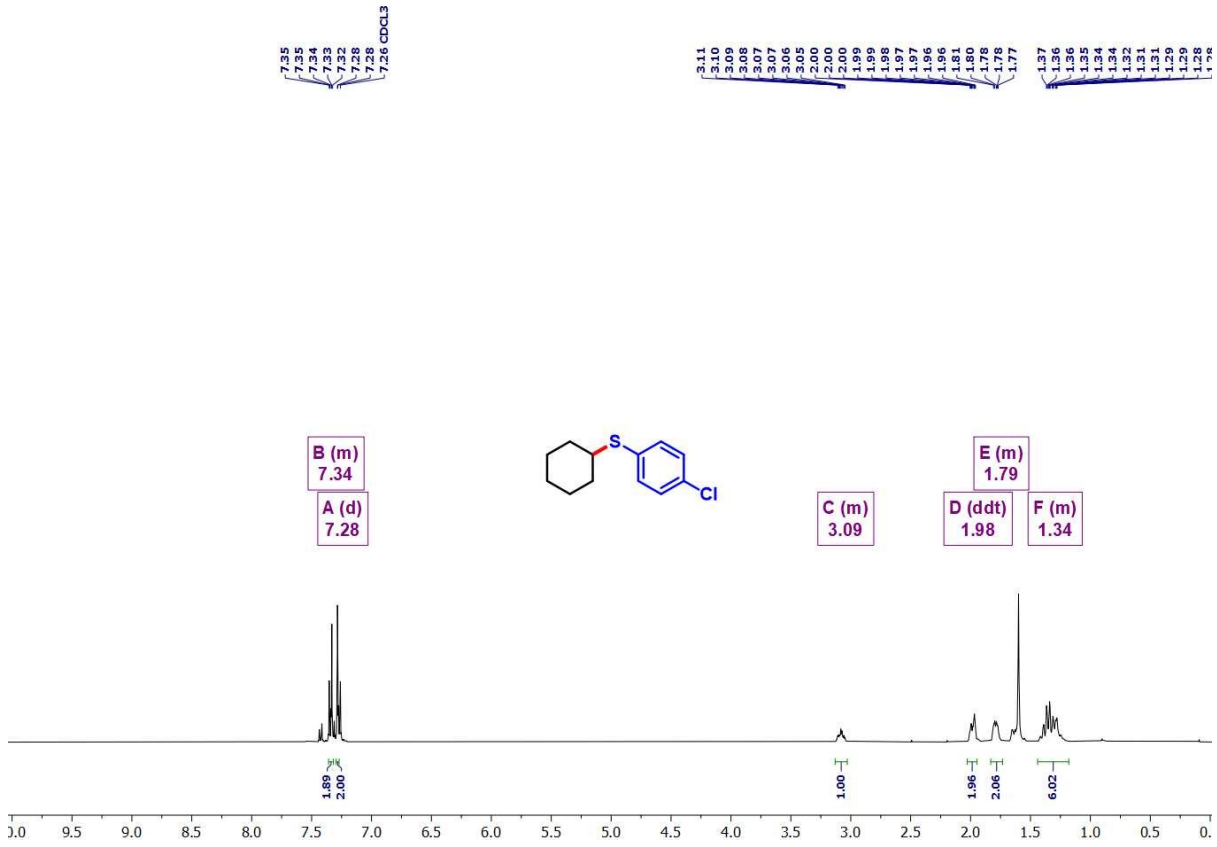
48.10
33.55
26.30
25.95
S
OMe


7.35
7.35
7.34
7.33
7.32
7.28
7.28
7.26 CDCL3
3.11
3.10
3.09
3.08
3.07
3.07
3.06
3.05
2.00
2.00
2.00
1.99
1.99
1.98
1.97
1.97
1.96
1.96
1.81
1.80
1.78
1.78
1.77
1.37
1.36
1.36
1.35
1.34
1.34
1.32
1.31
1.31
1.29
1.29
1.28
B (m)
7.34
A (d)
7.28
S
Cl
C (m)
3.09
E (m)
1.79
D (ddt)
1.98
F (m)
1.34
1.89
2.00
1.00
1.96
2.06
6.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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3.5
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2.0
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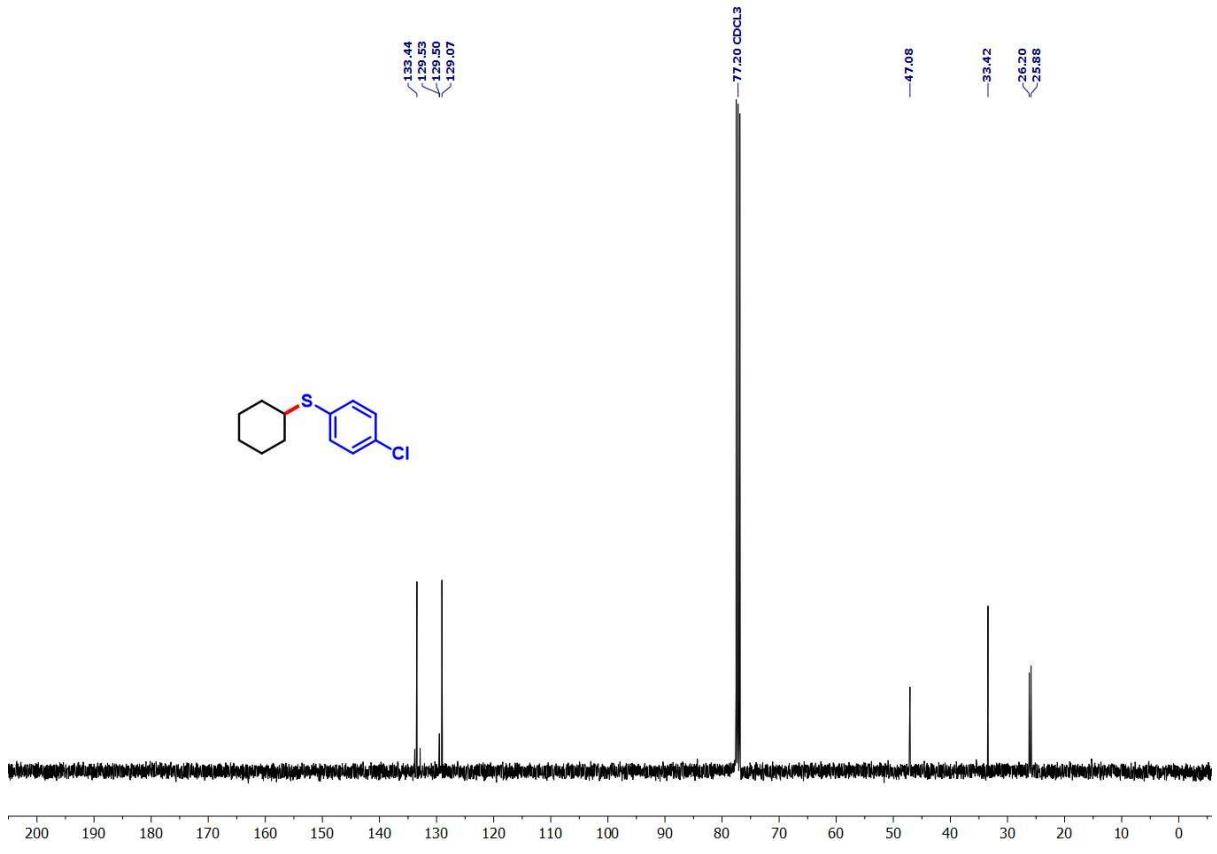
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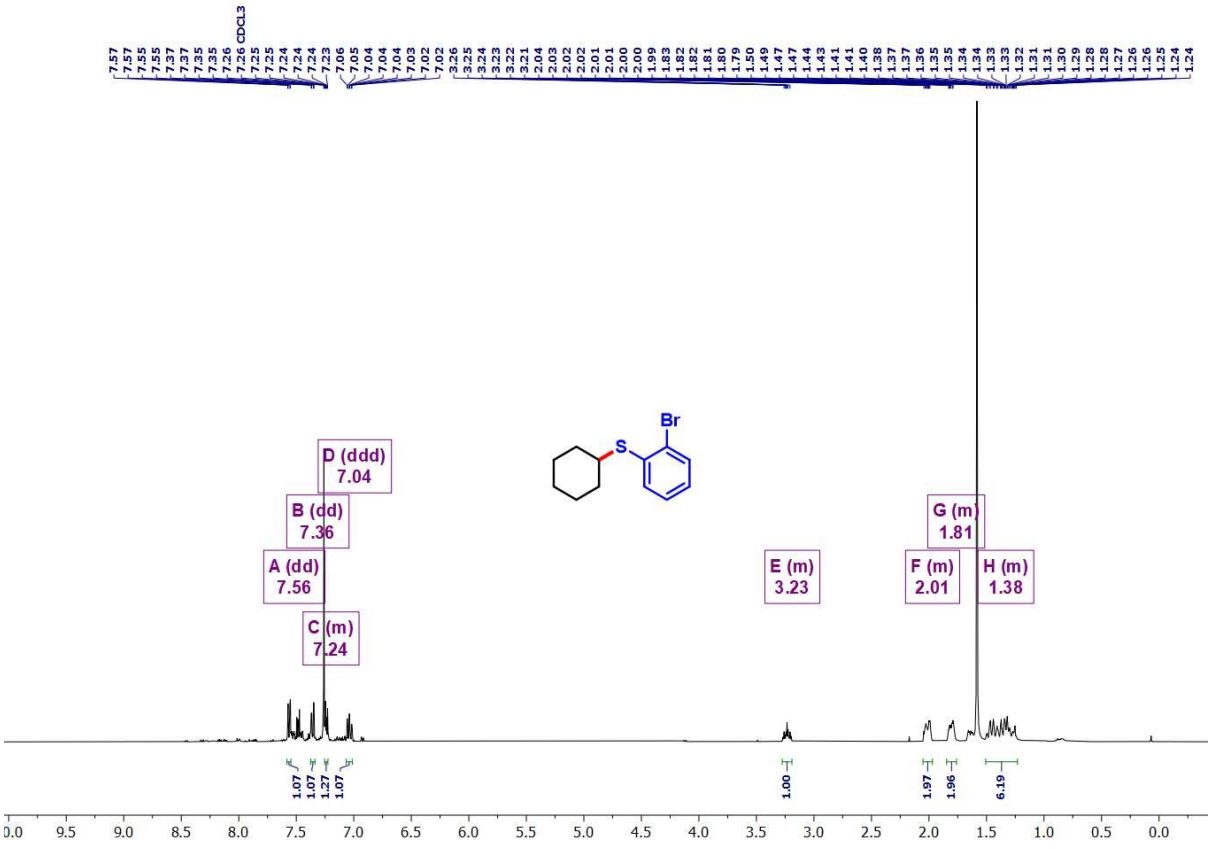
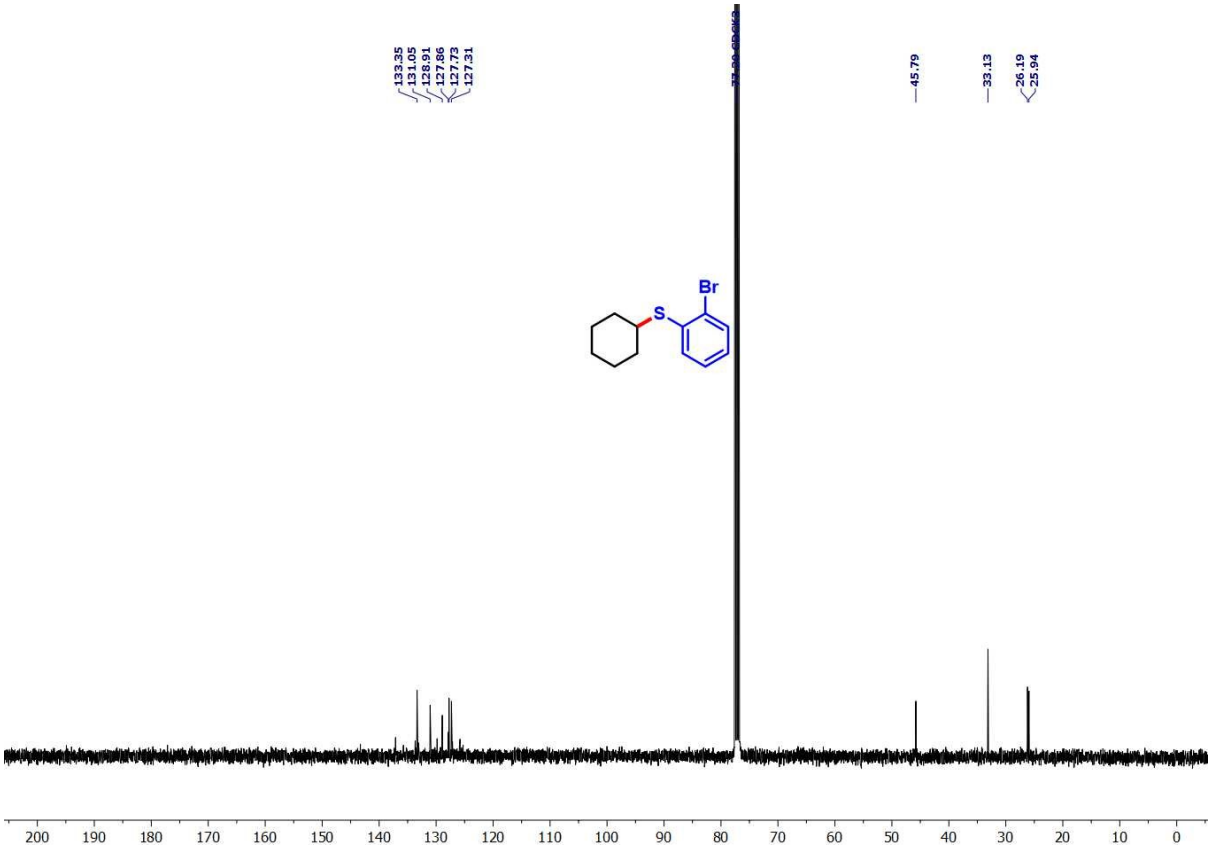
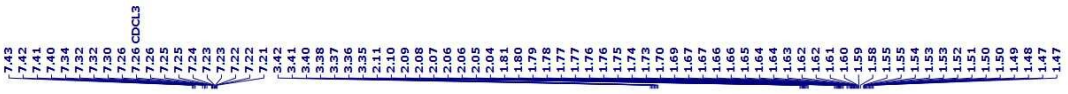
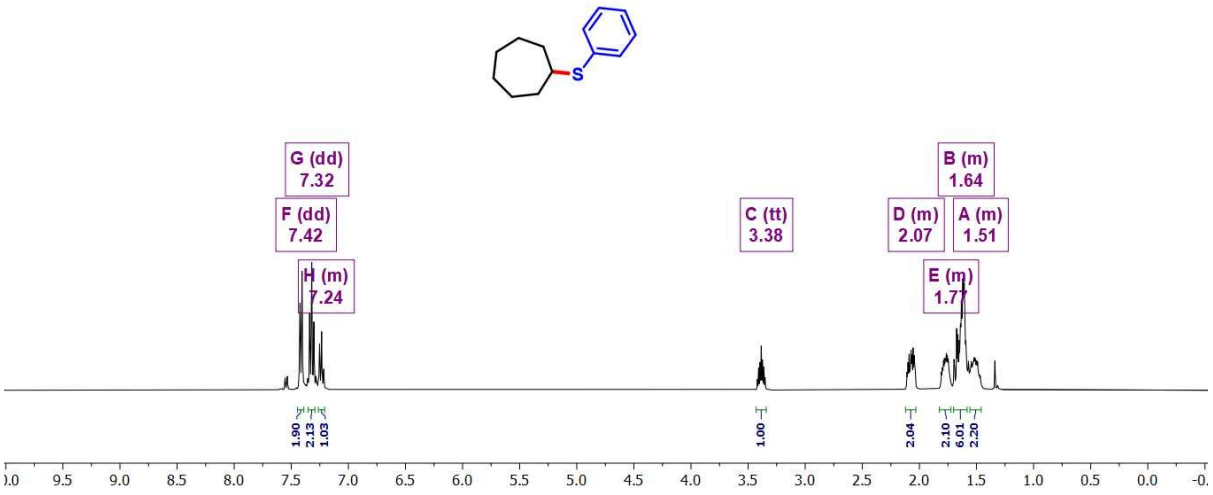
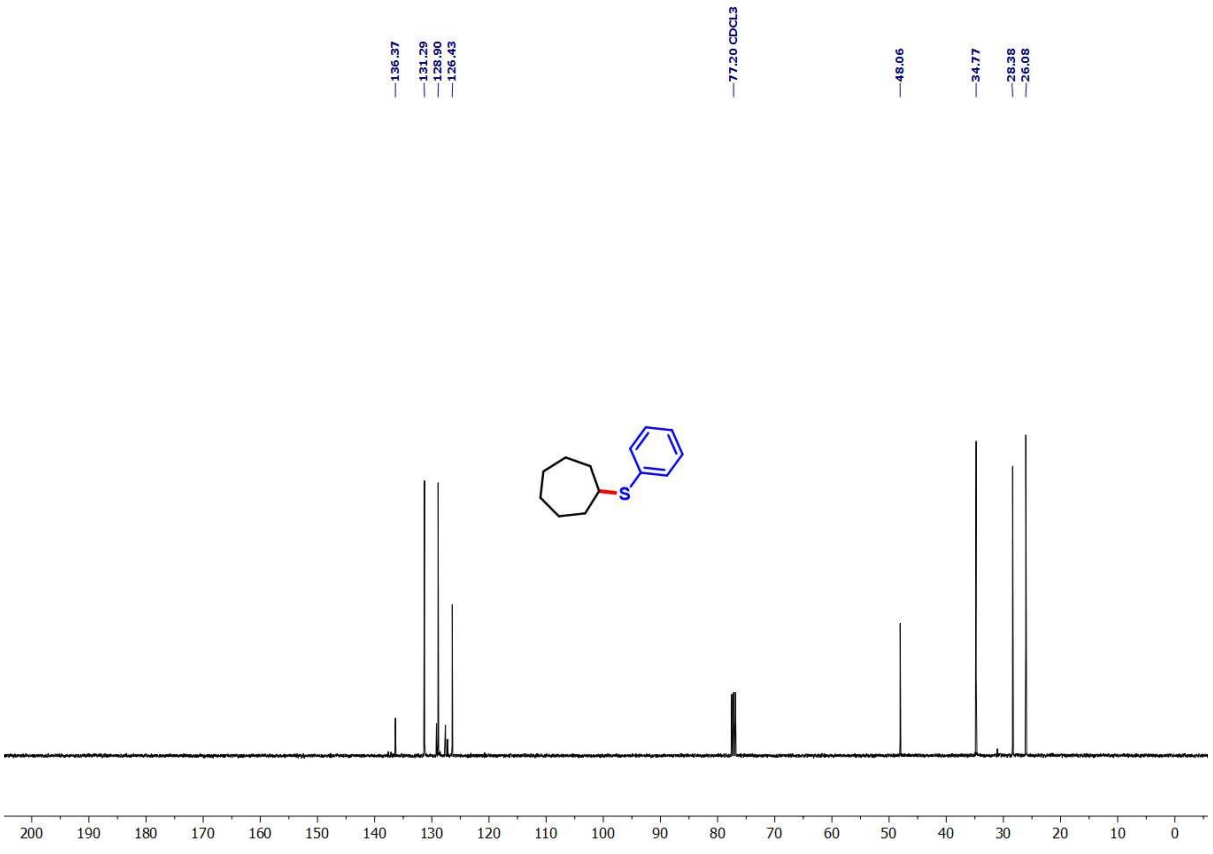
129.53
129.50
129.07
77.20 CDCL3
47.08
33.42
26.20
25.88
S
Cl
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


CDCL3
D (ddd)
7.04
B (dd)
7.36
A (dd)
7.56
C (m)
7.24
E (m)
3.23
G (m)
1.81
F (m)
2.01
H (m)
1.38
Br
S
1.07
1.07
1.27
1.07
1.00
1.97
1.96
6.19


133.35
131.05
128.91
127.86
127.73
127.31
45.79
33.13
26.19
25.94
Br
S




G (dd)
7.32
F (dd)
7.42
H (m)
7.24
C (tt)
3.38
B (m)
1.64
D (m)
2.07
A (m)
1.51
E (m)
1.77
1.90
2.13
1.03
1.00
2.04
2.10
6.01
2.20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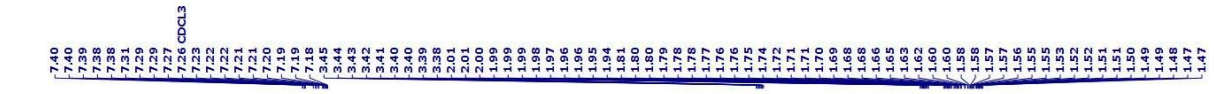
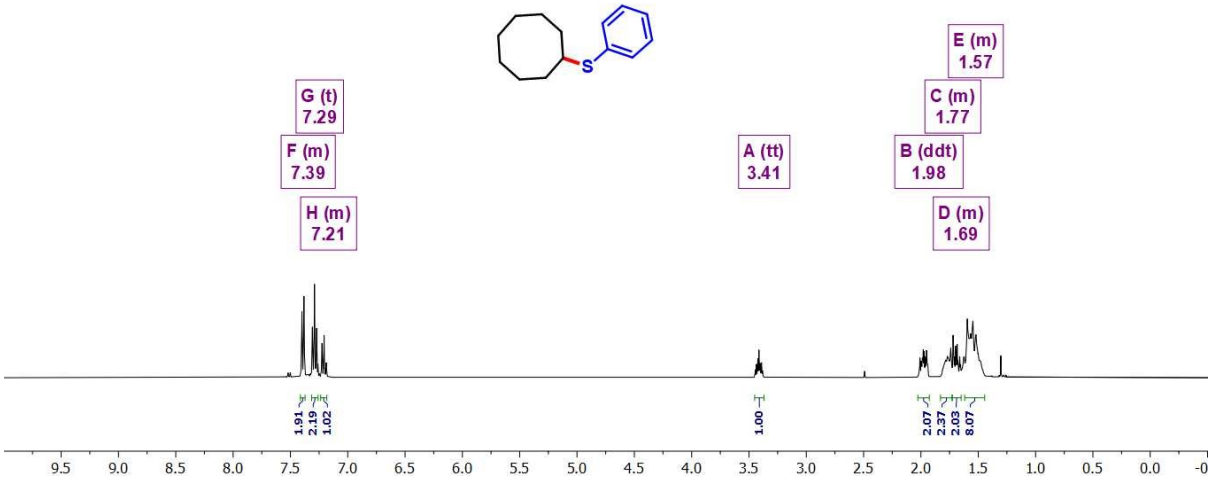
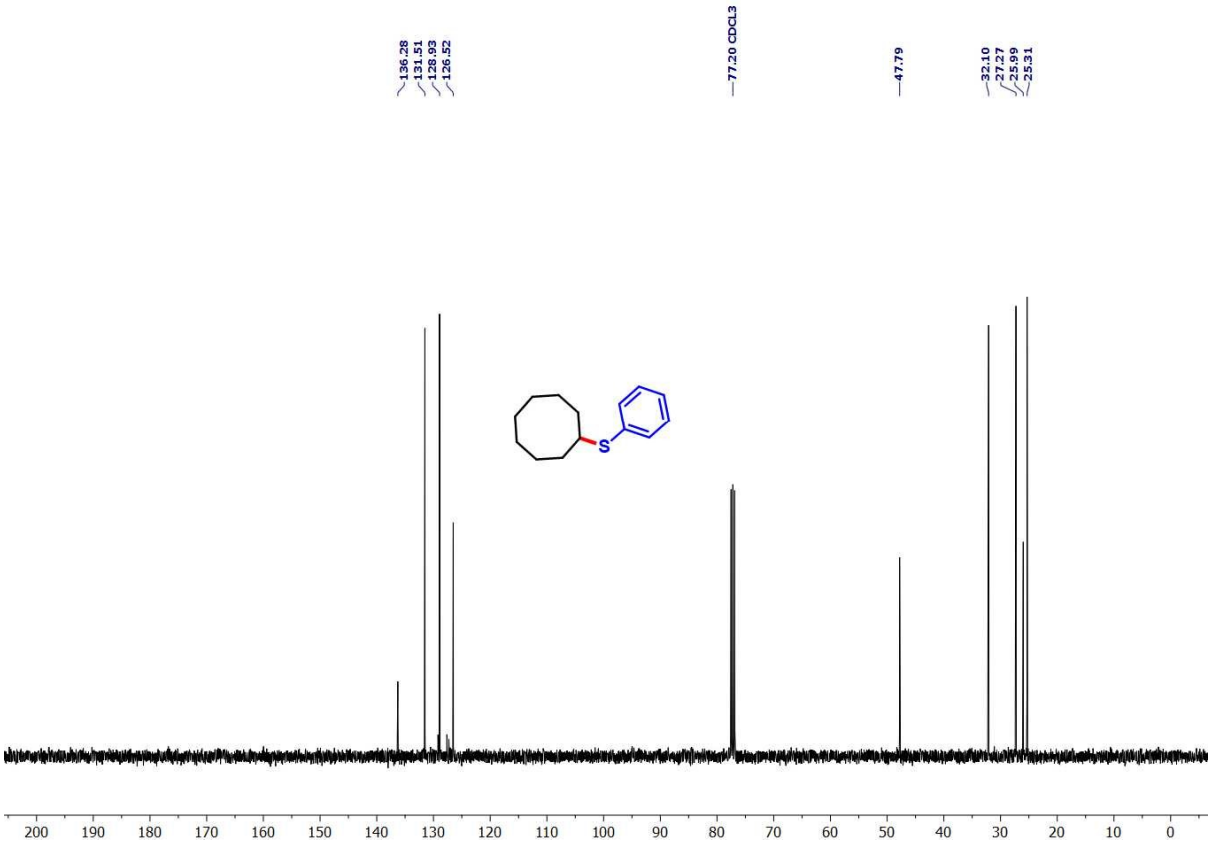
126.43
77.20 CDCl3
48.06
34.77
28.38
26.08


7.40 7.40 7.39 7.38 7.38 7.31 7.29 7.29 7.27 7.26 CDCL3 7.23 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 3.45 3.44 3.43 3.42 3.41 3.40 3.40 3.39 3.38 2.01 2.01 2.00 1.99 1.99 1.99 1.98 1.97 1.96 1.96 1.95 1.94 1.81 1.80 1.80 1.79 1.78 1.78 1.77 1.76 1.76 1.75 1.74 1.72 1.71 1.71 1.70 1.69 1.68 1.68 1.66 1.65 1.63 1.62 1.60 1.60 1.58 1.58 1.57 1.57 1.56 1.55 1.53 1.52 1.52 1.51 1.51 1.50 1.49 1.49 1.48 1.47 1.47


S
G (t) 7.29
F (m) 7.39
H (m) 7.21
A (tt) 3.41
E (m) 1.57
C (m) 1.77
B (ddt) 1.98
D (m) 1.69
1.91
2.19
1.02
1.00
2.07
2.37
2.03
8.07
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0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32.10
27.27
25.99
25.31
S
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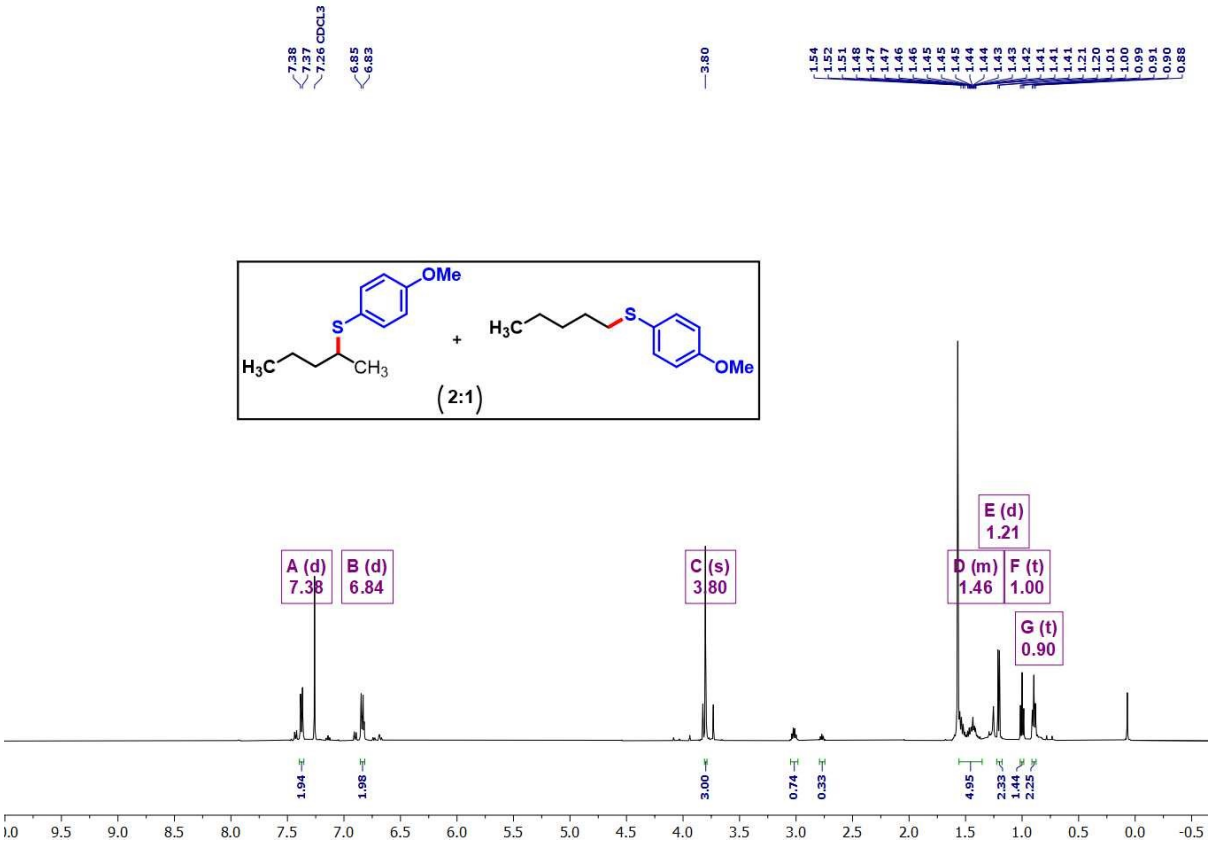
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7.37
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6.83
3.80
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1.52
1.51
1.48
1.47
1.47
1.46
1.46
1.45
1.45
1.45
1.44
1.44
1.43
1.43
1.42
1.41
1.41
1.41
1.21
1.20
1.01
1.00
0.99
0.91
0.90
0.88
OMe
S
H3C
CH3
+
H3C
S
OMe
(2:1)
A (d)
7.38
B (d)
6.84
C (s)
3.80
E (d)
1.21
D (m)
1.46
F (t)
1.00
G (t)
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1.94
1.98
3.00
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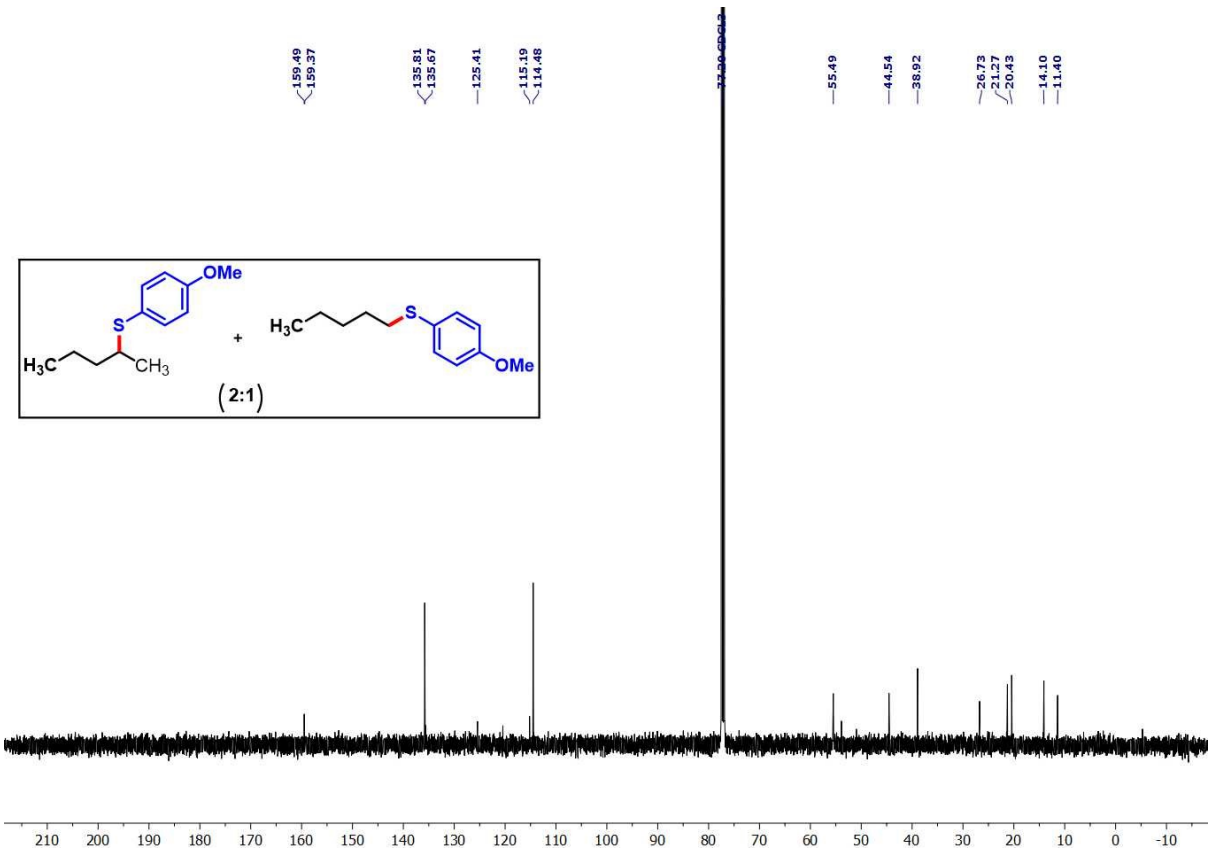
0.33
4.95
2.33
1.44
2.25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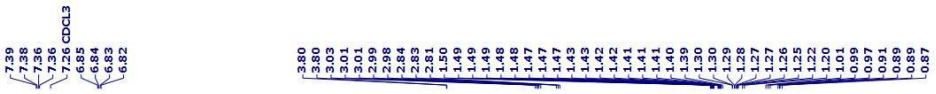
115.19
114.48
77.20 CDCL3
55.49
44.54
38.92
26.73
21.27
20.43
14.10
11.40
OMe
S
H3C
CH3
+
H3C
S
OMe
(2:1)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7.36
CDCL3
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6.85
6.84
6.83
6.82
3.80
3.80
3.03
3.01
3.01
2.99
2.98
2.84
2.83
2.81
1.50
1.49
1.49
1.49
1.48
1.48
1.47
1.47
1.47
1.43
1.42
1.42
1.41
1.41
1.41
1.40
1.39
1.30
1.30
1.29
1.28
1.27
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1.22
1.20
1.01
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0.97
0.91
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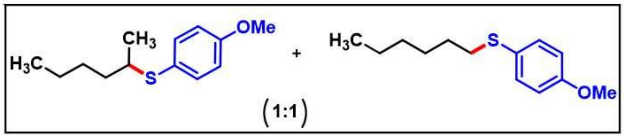
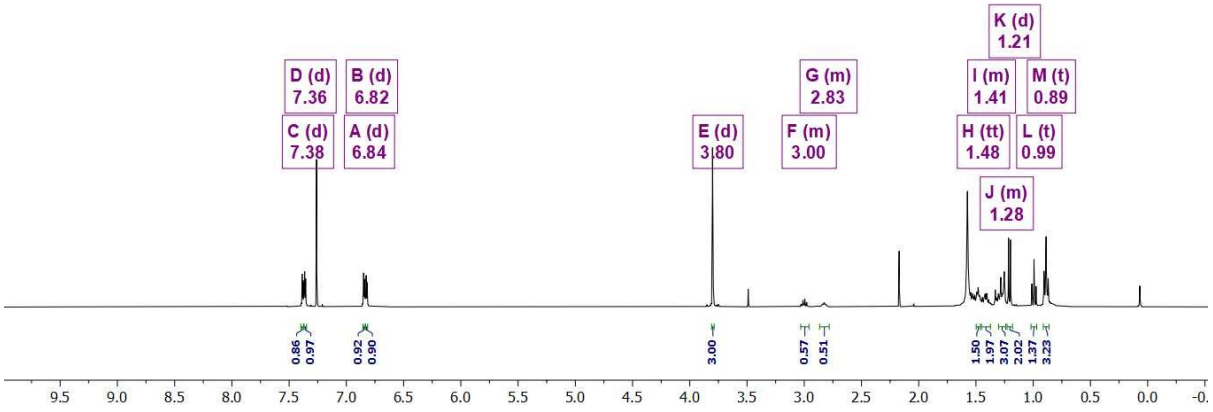
0.89
0.87


CH3
OMe
H3C
S
+
H3C
S
OMe
(1:1)


D (d)
7.36
B (d)
6.82
C (d)
7.38
A (d)
6.84
E (d)
3.80
G (m)
2.83
F (m)
3.00
K (d)
1.21
I (m)
1.41
M (t)
0.89
H (tt)
1.48
L (t)
0.99
J (m)
1.28
0.86
0.97
0.92
0.90
3.00
0.57
0.51
1.50
1.97
3.07
2.02
1.37
3.23
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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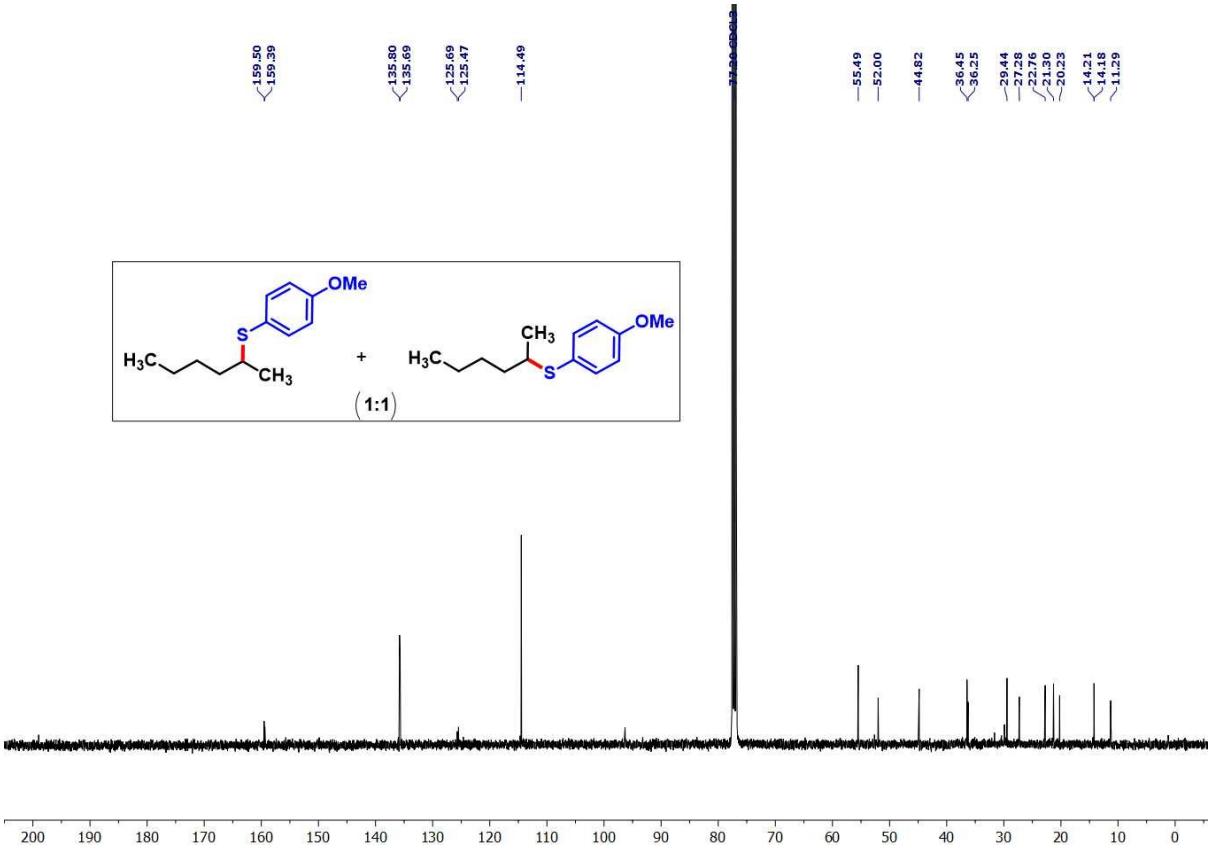
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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135.80
135.69
125.69
125.47
114.49
55.49
52.00
44.82
36.45
36.25
29.44
27.28
22.76
21.30
20.23
14.21
14.18
11.29
OMe
S
H3C
CH3
+
H3C
CH3
OMe
S
(1:1)
200
190
180
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160
150
140
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120
110
100
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70
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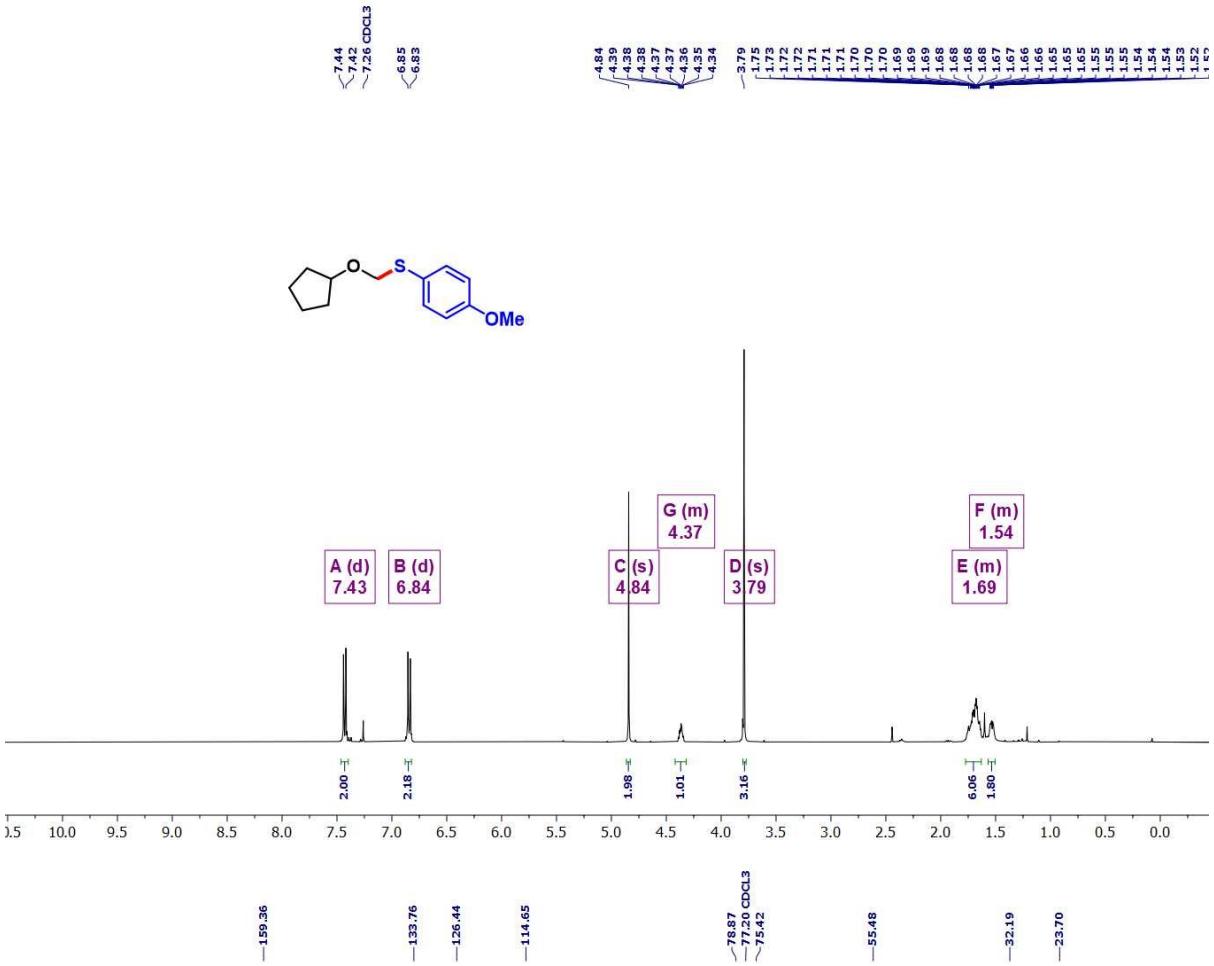
20
10
0


7.44
7.42
7.26 CDCL3
6.85
6.83
4.84
4.39
4.38
4.38
4.37
4.37
4.36
4.35
4.34
3.79
1.75
1.73
1.72
1.72
1.71
1.71
1.71
1.70
1.70
1.70
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1.67
1.66
1.66
1.65
1.65
1.55
1.55
1.55
1.54
1.54
1.54
1.53
1.52
O
S
OMe
A (d)
7.43
B (d)
6.84
C (s)
4.84
G (m)
4.37
D (s)
3.79
F (m)
1.54
E (m)
1.69
2.00
2.18
1.98
1.01
3.16
6.06
1.80
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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3.0
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2.0
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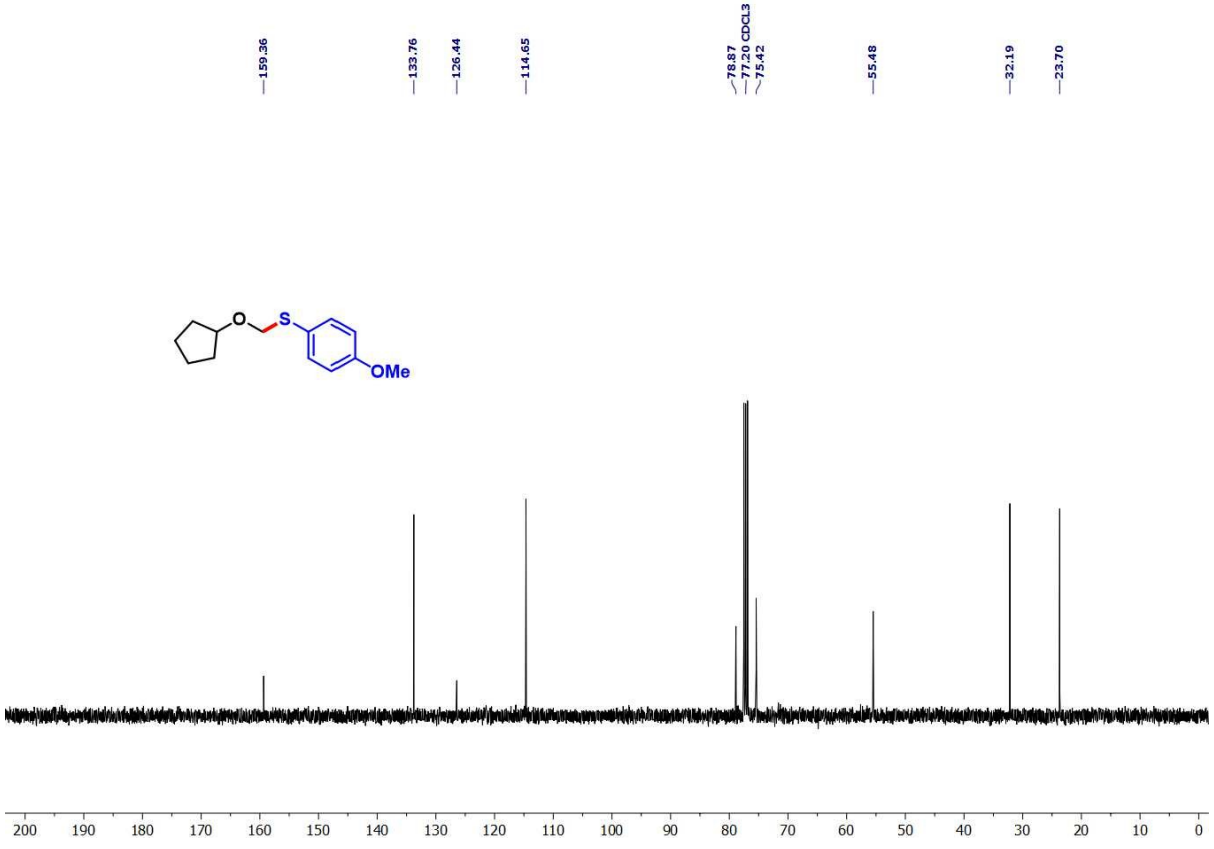
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126.44
114.65
78.87
77.20 CDCL3
75.42
55.48
32.19
23.70
O
S
OMe
200
190
180
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150
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30
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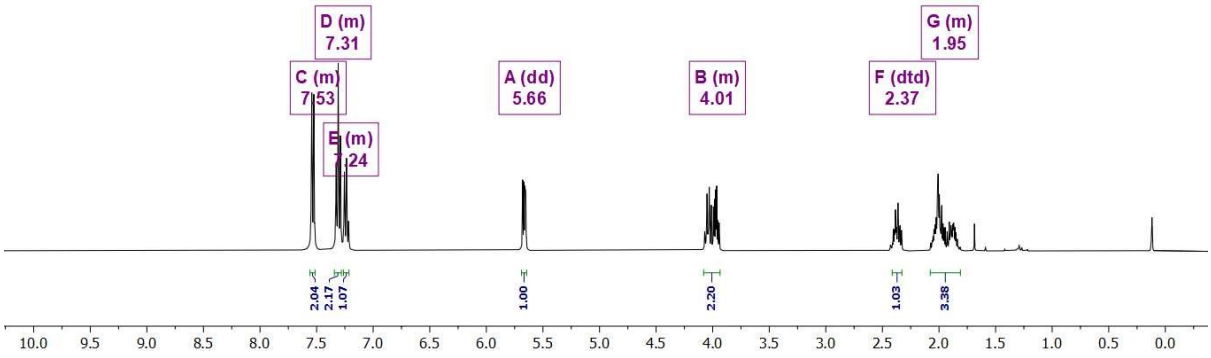
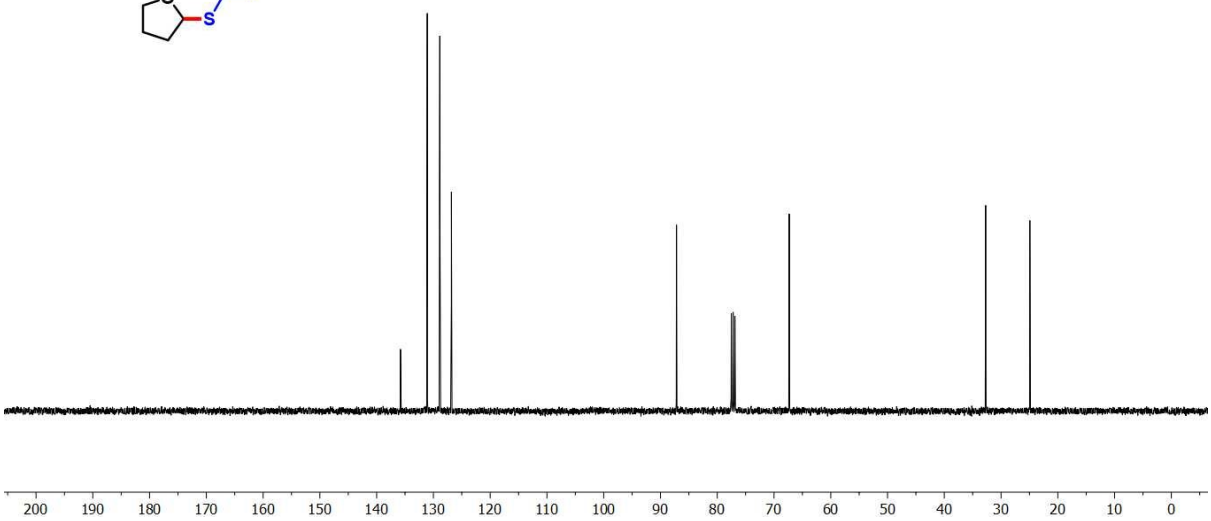
10
0


D (m)
7.31
C (m)
7.53
B (m)
7.24
A (dd)
5.66
B (m)
4.01
F (dtd)
2.37
G (m)
1.95
2.04
2.17
1.07
1.00
2.20
1.03
3.38
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


135.79
131.08
128.87
126.83
87.18
77.20 CDCL3
67.32
32.71
24.92


200
190
180
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160
150
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100
90
80
70
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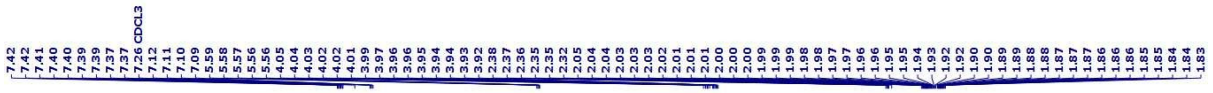
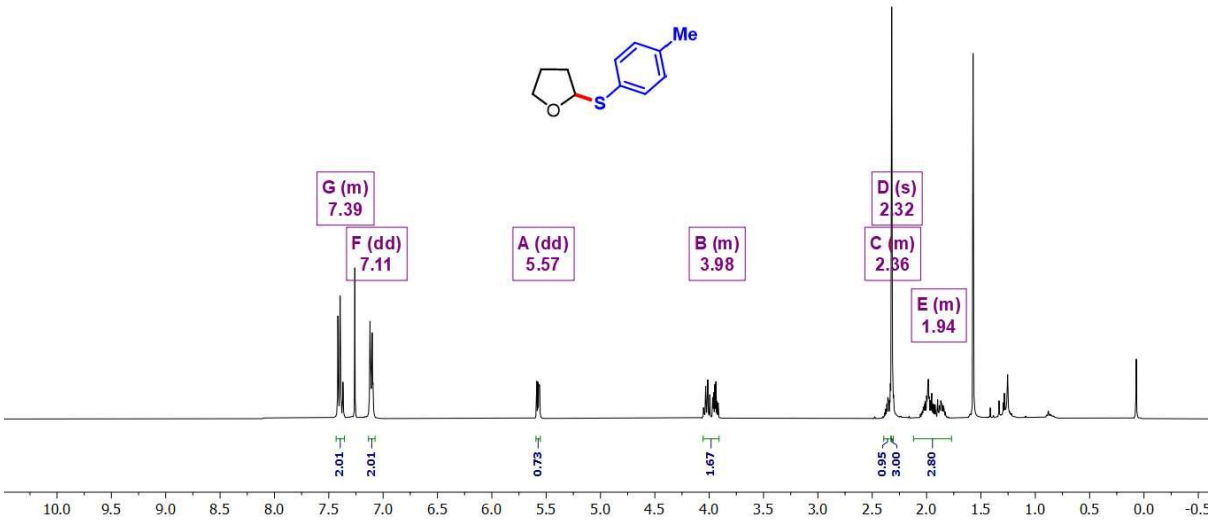
40
30
20
10
0




Me
S
G (m)
7.39
F (dd)
7.11
A (dd)
5.57
B (m)
3.98
D (s)
2.32
C (m)
2.36
E (m)
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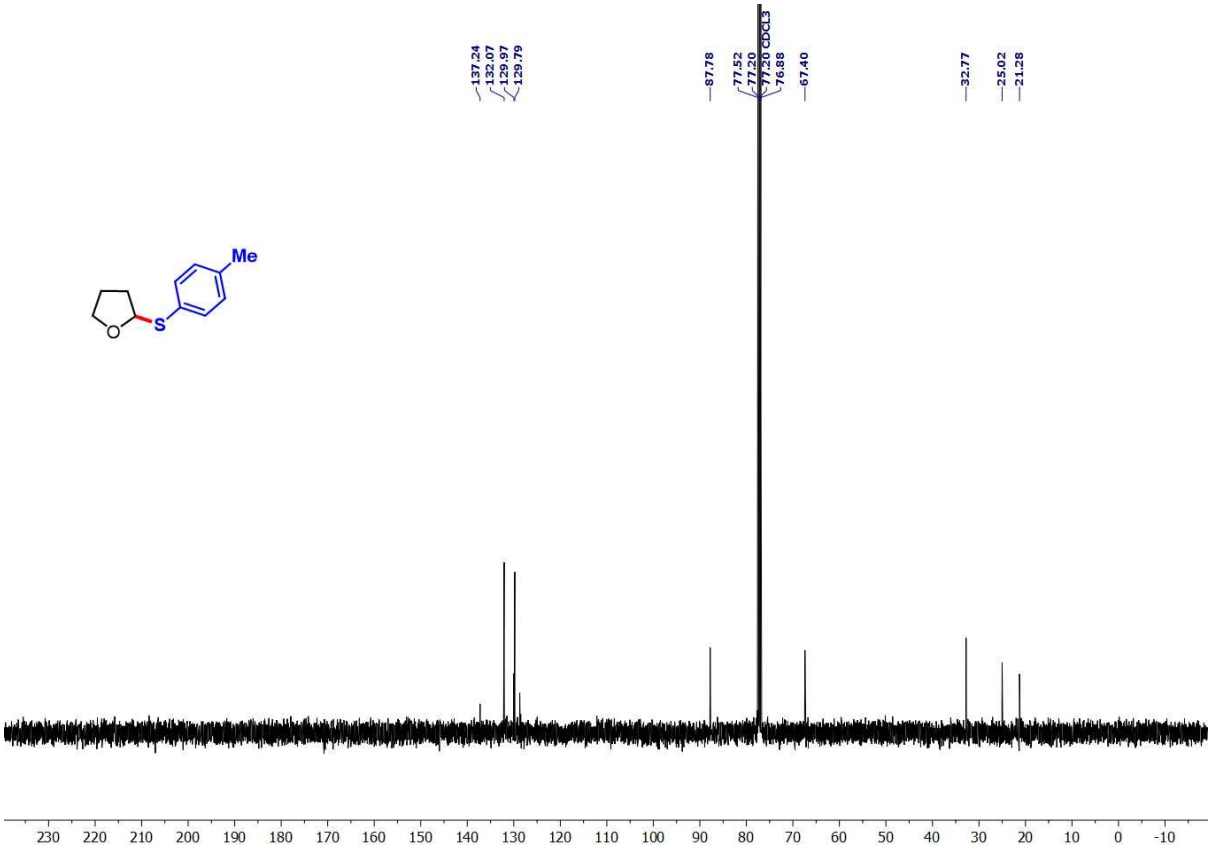
2.01
2.01
0.73
1.67
0.95
3.00
2.80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87.78
77.52
77.20
77.20 CDCl3
76.88
67.40
32.77
25.02
21.28
Me
S


7.44
7.42
7.27
CDCL3
7.26
7.24
5.61
5.60
5.59
5.58
4.04
4.03
4.03
4.02
4.00
4.00
4.00
3.98
3.97
3.97
3.96
3.95
3.94
3.93
2.39
2.38
2.38
2.37
2.36
2.36
2.35
2.35
2.34
2.33
2.33
2.32
2.05
2.04
2.04
2.03
2.03
2.02
2.02
2.01
2.01
2.01
2.00
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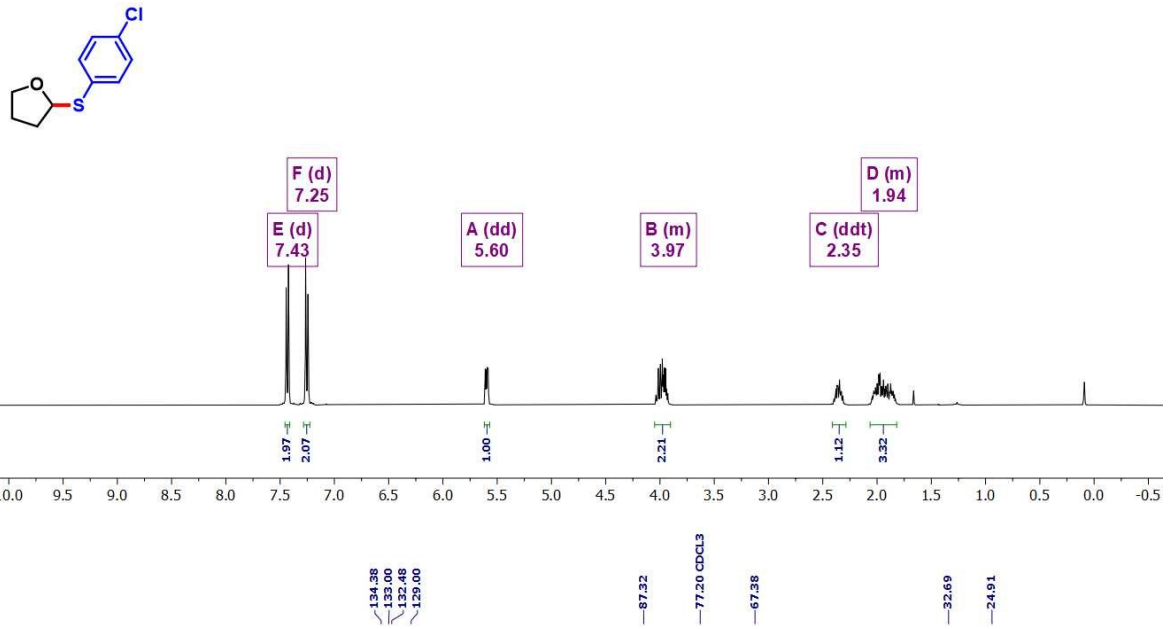
1.85
1.85
1.84
1.84
1.83


Cl
S
O
F (d)
7.25
E (d)
7.43
A (dd)
5.60
B (m)
3.97
C (ddt)
2.35
D (m)
1.94
1.97
2.07
1.00
2.21
1.12
3.32
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

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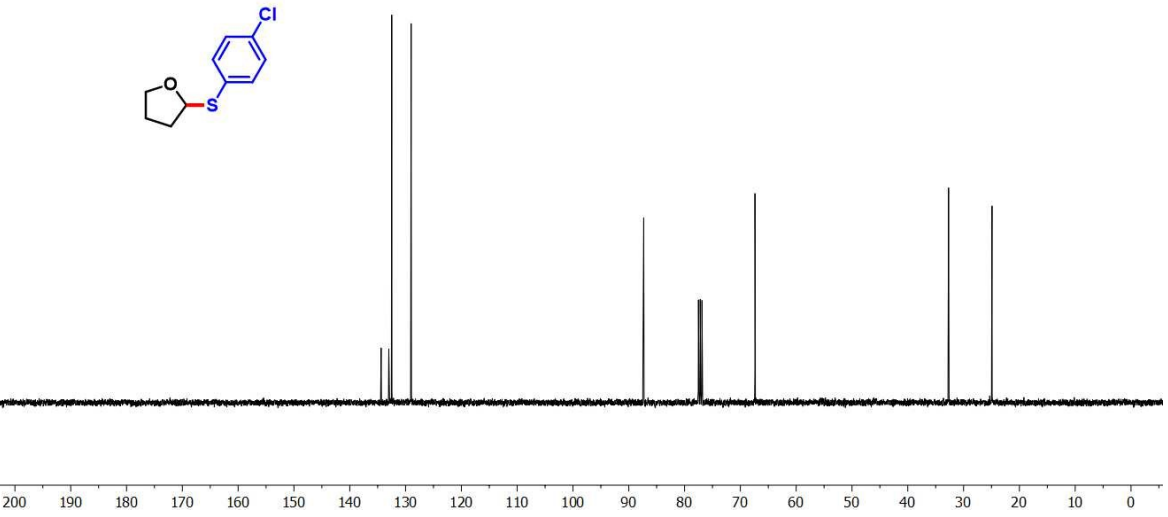
132.48
129.00
87.32
77.20 CDCL3
67.38
32.69
24.91


Cl
S
O
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200
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180
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30
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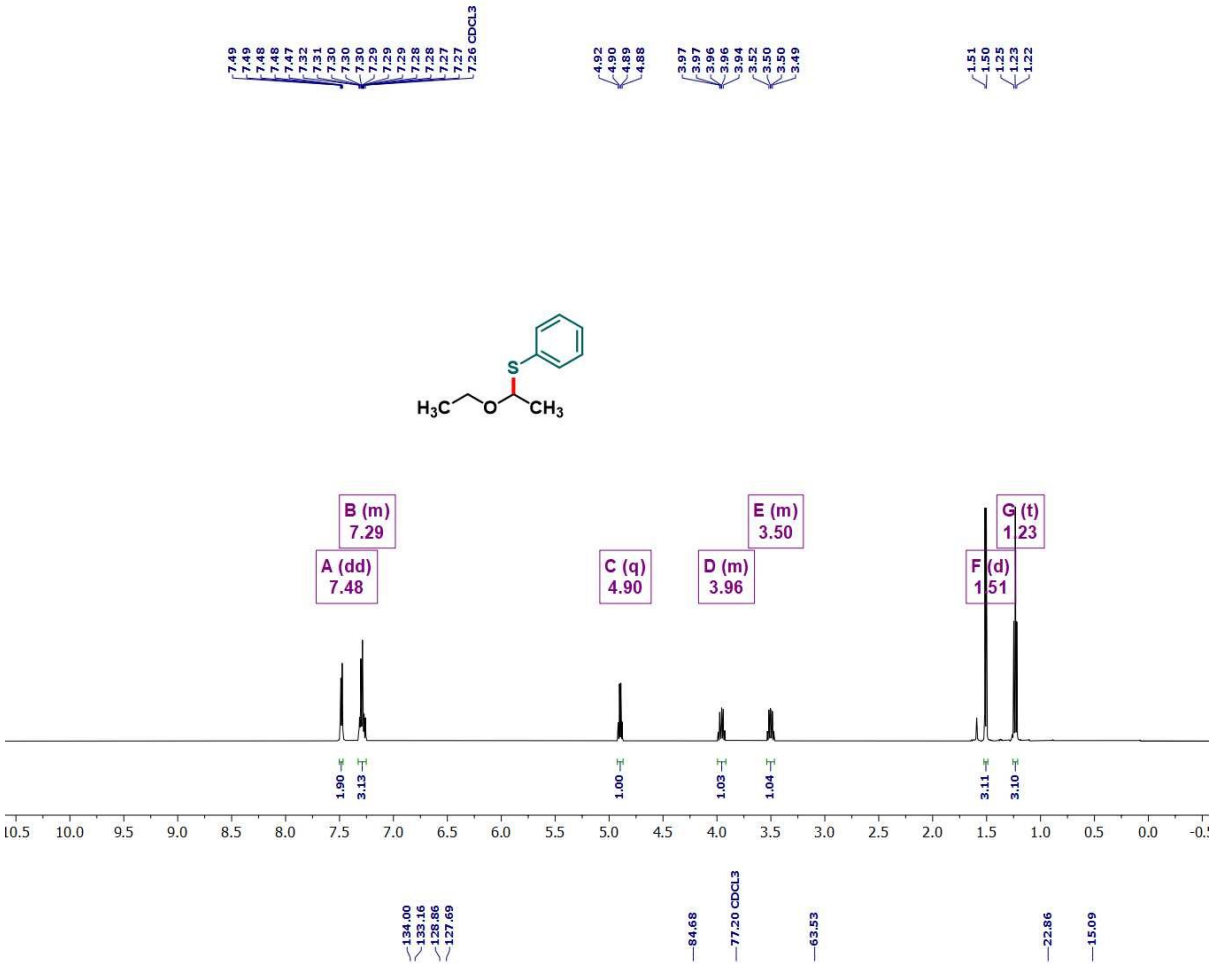
10
0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7.30
7.30
7.30
7.29
7.29
7.29
7.28
7.28
7.27
7.27
7.26 CDCL3
4.92
4.90
4.89
4.88
3.97
3.97
3.96
3.96
3.94
3.52
3.50
3.50
3.49
1.51
1.50
1.25
1.23
1.22
S
H3C
O
CH3
B (m)
7.29
A (dd)
7.48
C (q)
4.90
D (m)
3.96
E (m)
3.50
G (t)
1.23
F (d)
1.51
1.90
3.13
1.00
1.03
1.04
3.11
3.10
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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2.0
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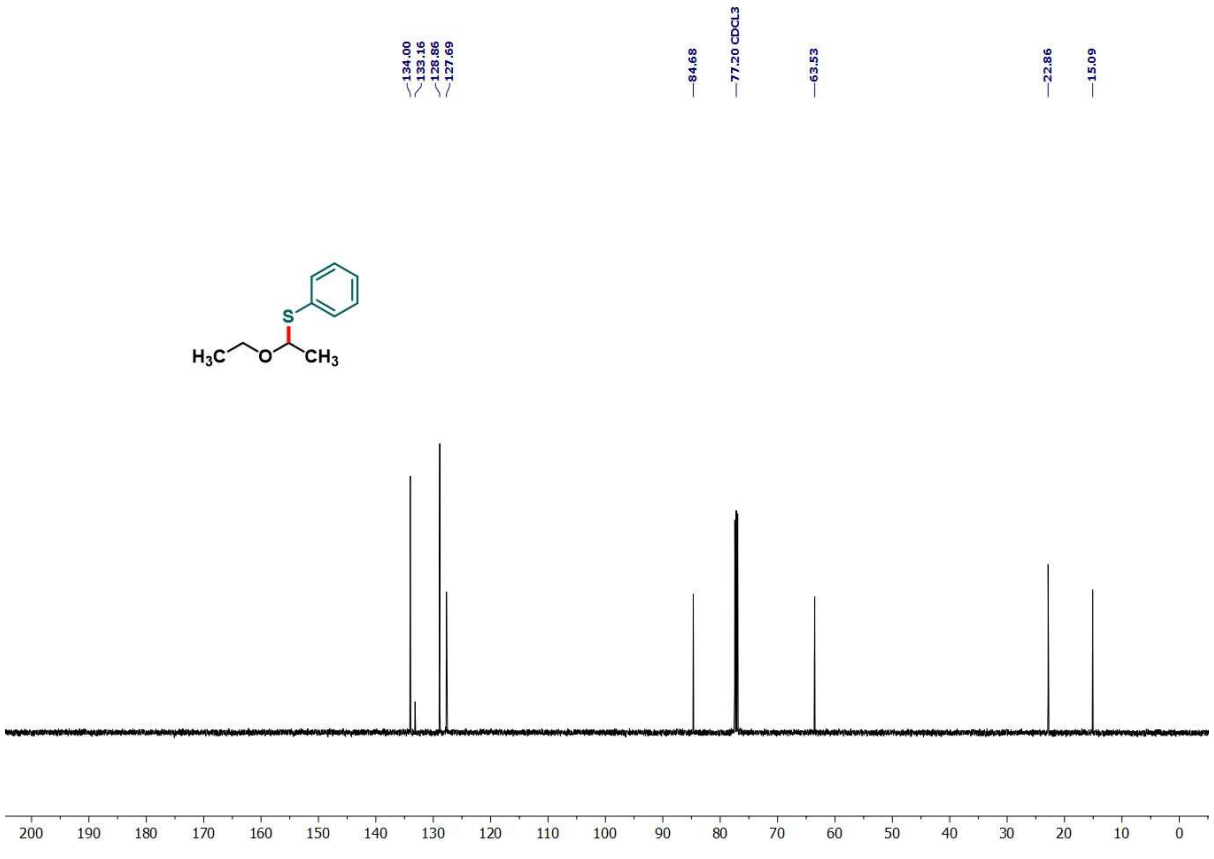
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127.69
84.68
77.20 CDCL3
63.53
22.86
15.09
S
H3C
O
CH3
200
190
180
170
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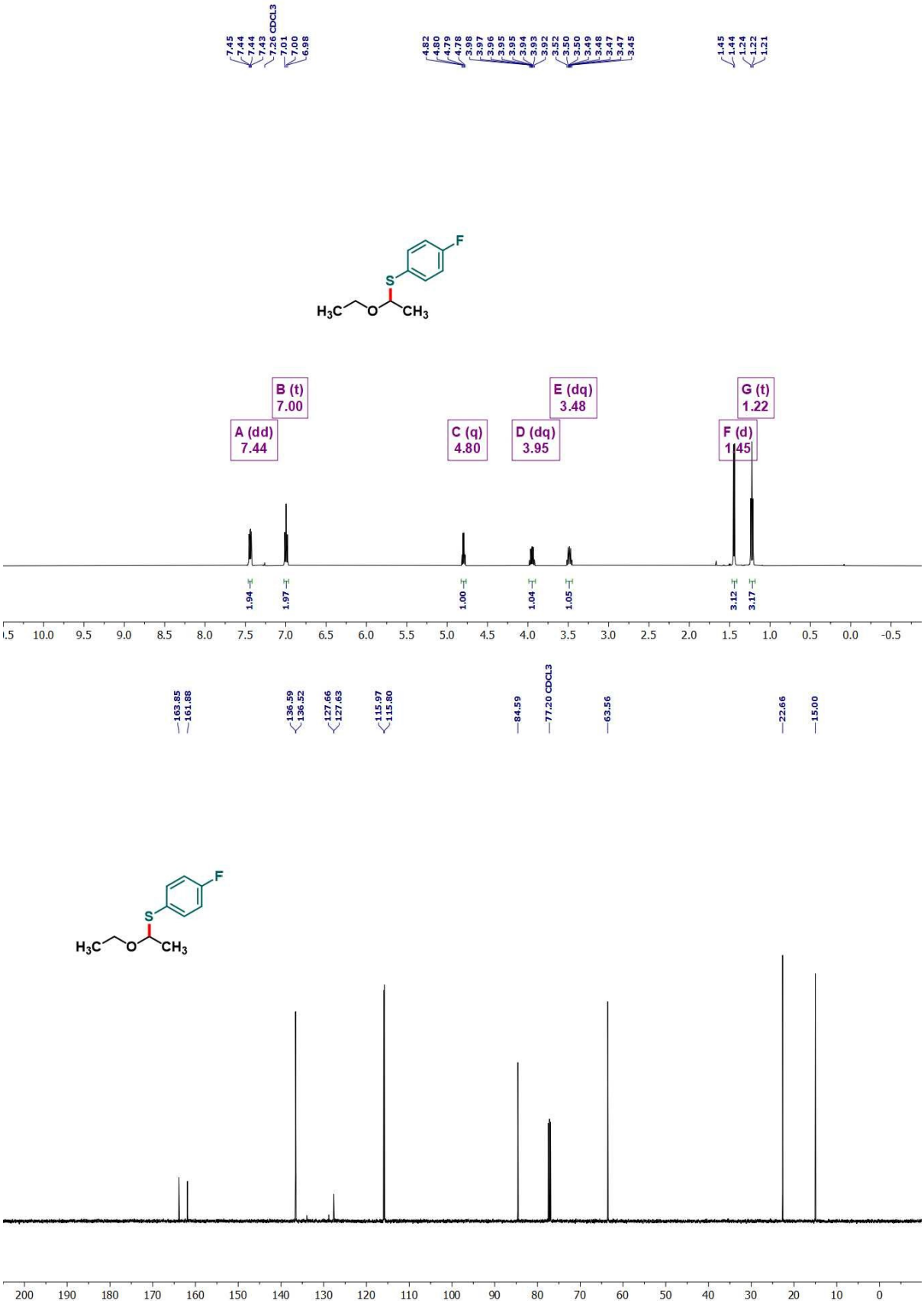
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


7.45
7.44
7.43
7.26 CDCL3
7.01
7.00
6.98
4.82
4.80
4.79
4.78
3.98
3.97
3.96
3.95
3.95
3.94
3.93
3.92
3.52
3.50
3.50
3.49
3.48
3.47
3.47
3.45
1.45
1.44
1.24
1.22
1.21
F
S
H3C
O
CH3
A (dd)
7.44
B (t)
7.00
C (q)
4.80
D (dq)
3.95
E (dq)
3.48
F (d)
1.45
G (t)
1.22
1.94
1.97
1.00
1.04
1.05
3.12
3.17
163.85
161.88
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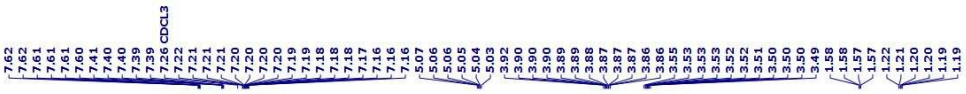
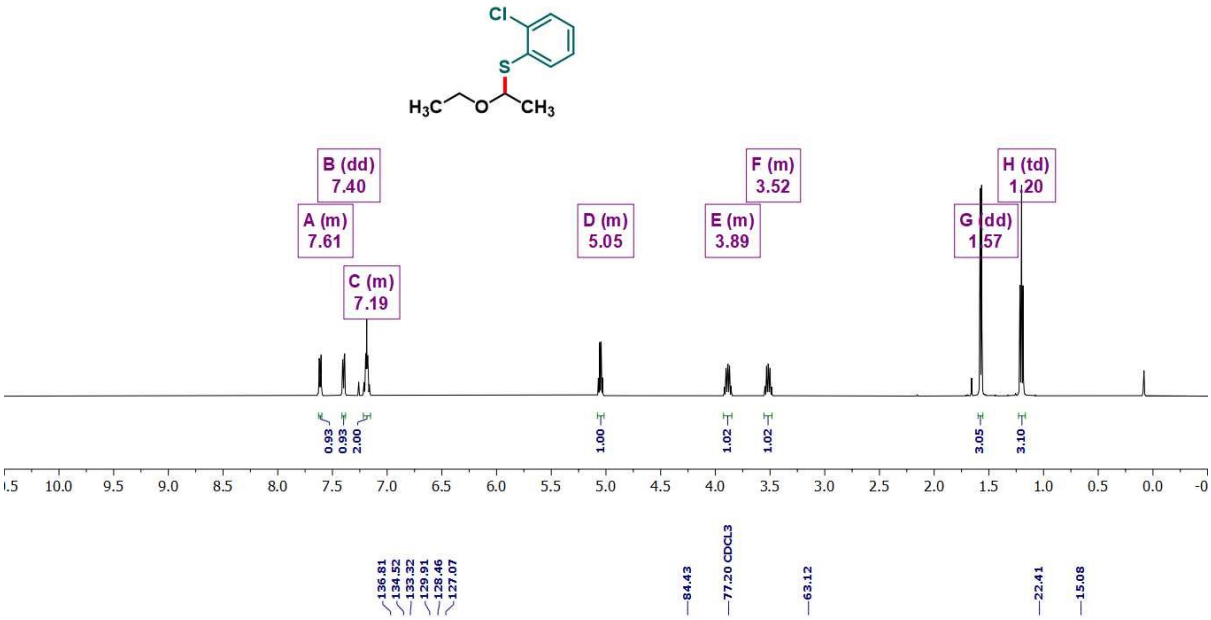
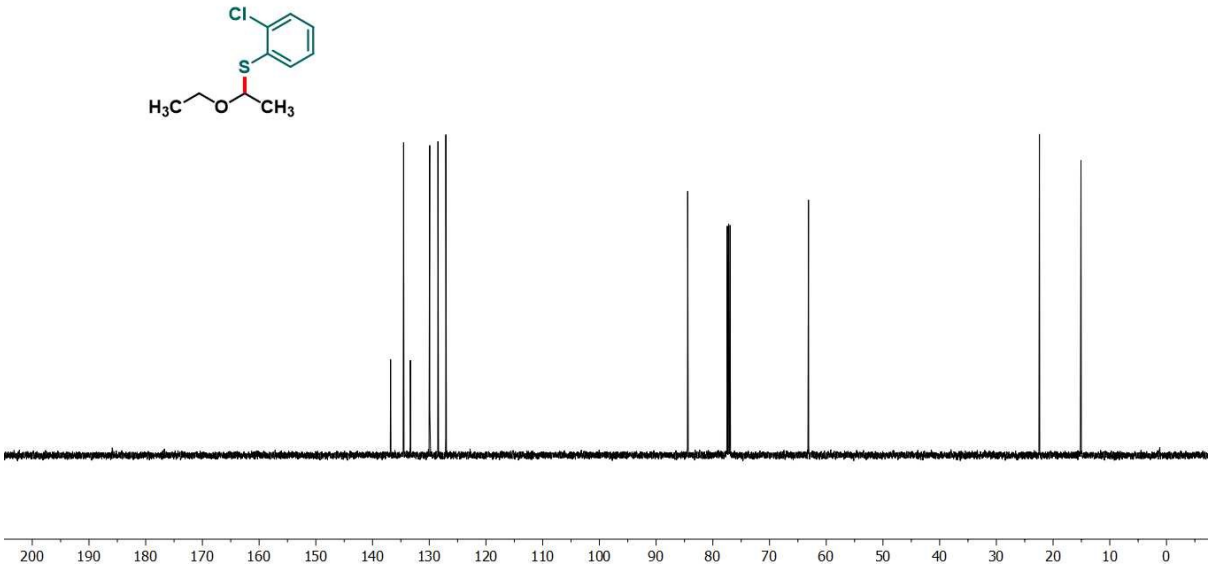
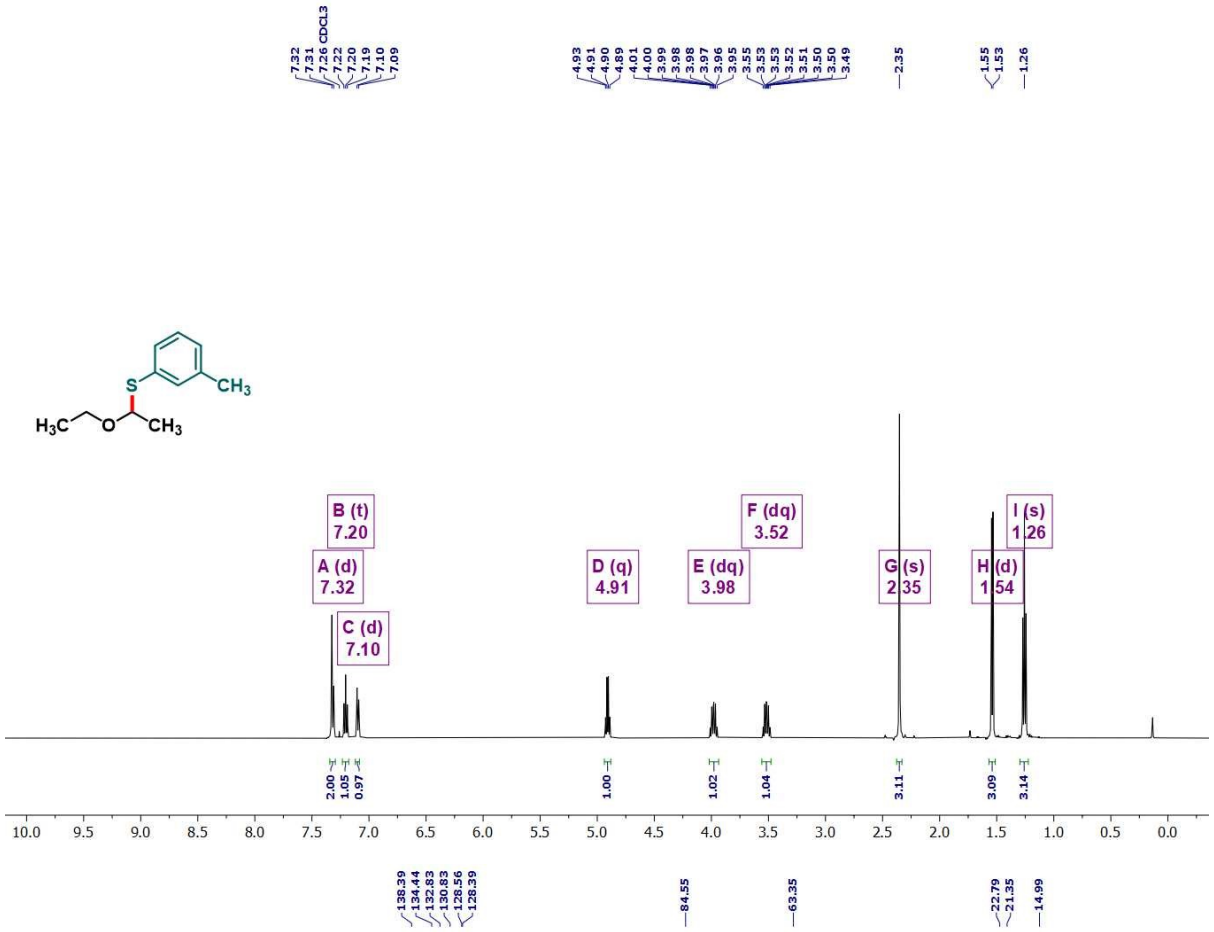
84.59
77.20 CDCL3
63.56
22.66
15.00




Cl
S
H3C
O
CH3
B (dd)
7.40
A (m)
7.61
C (m)
7.19
D (m)
5.05
E (m)
3.89
F (m)
3.52
G (dd)
1.57
H (td)
1.20
0.93
0.93
2.00
1.00
1.02
1.02
3.05
3.10

136.81
134.52
133.32
129.91
128.46
127.07
84.43
77.20 CDCl3
63.12
22.41
15.08


Cl
S
H3C
O
CH3


7.32
7.31
7.26 CDCL3
7.22
7.20
7.19
7.10
7.09
4.93
4.91
4.90
4.89
4.01
4.00
3.99
3.98
3.98
3.97
3.96
3.95
3.55
3.53
3.53
3.52
3.51
3.50
3.50
3.49
2.35
1.55
1.53
1.26
S
CH3
H3C
O
CH3
B (t)
7.20
A (d)
7.32
C (d)
7.10
D (q)
4.91
E (dq)
3.98
F (dq)
3.52
G (s)
2.35
H (d)
1.54
I (s)
1.26
2.00
1.05
0.97
1.00
1.02
1.04
3.11
3.09
3.14
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
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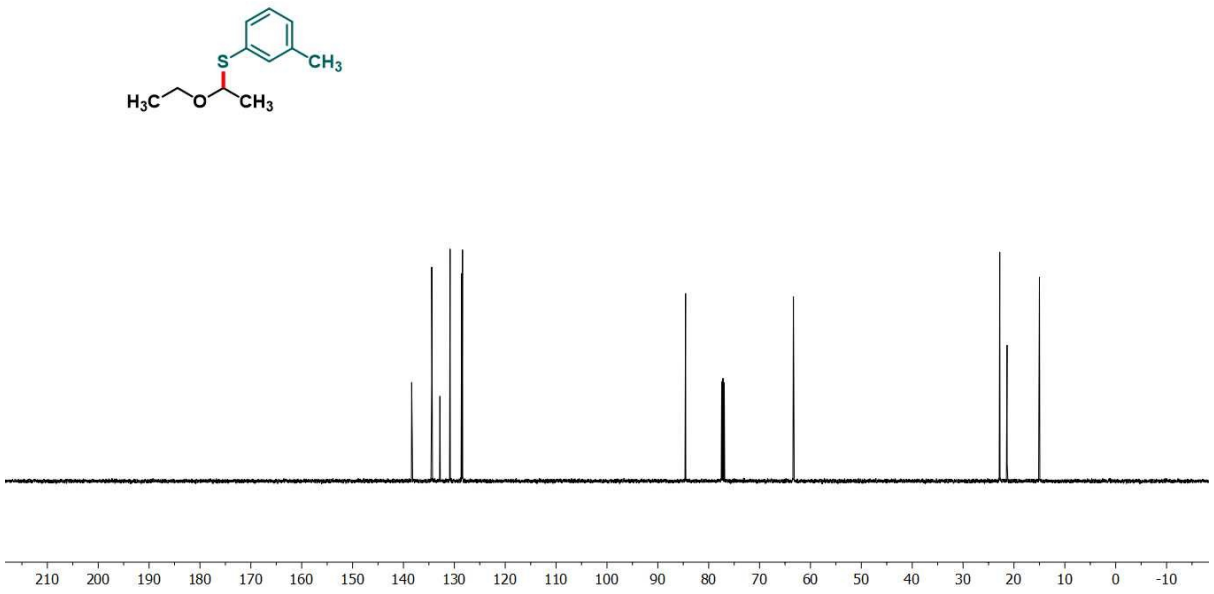
134.44
132.83
130.83
128.56
128.39
84.55
63.35
22.79
21.35
14.99


S
CH3
H3C
O
CH3
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200
190
180
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150
140
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120
110
100
90
80
70
60
50
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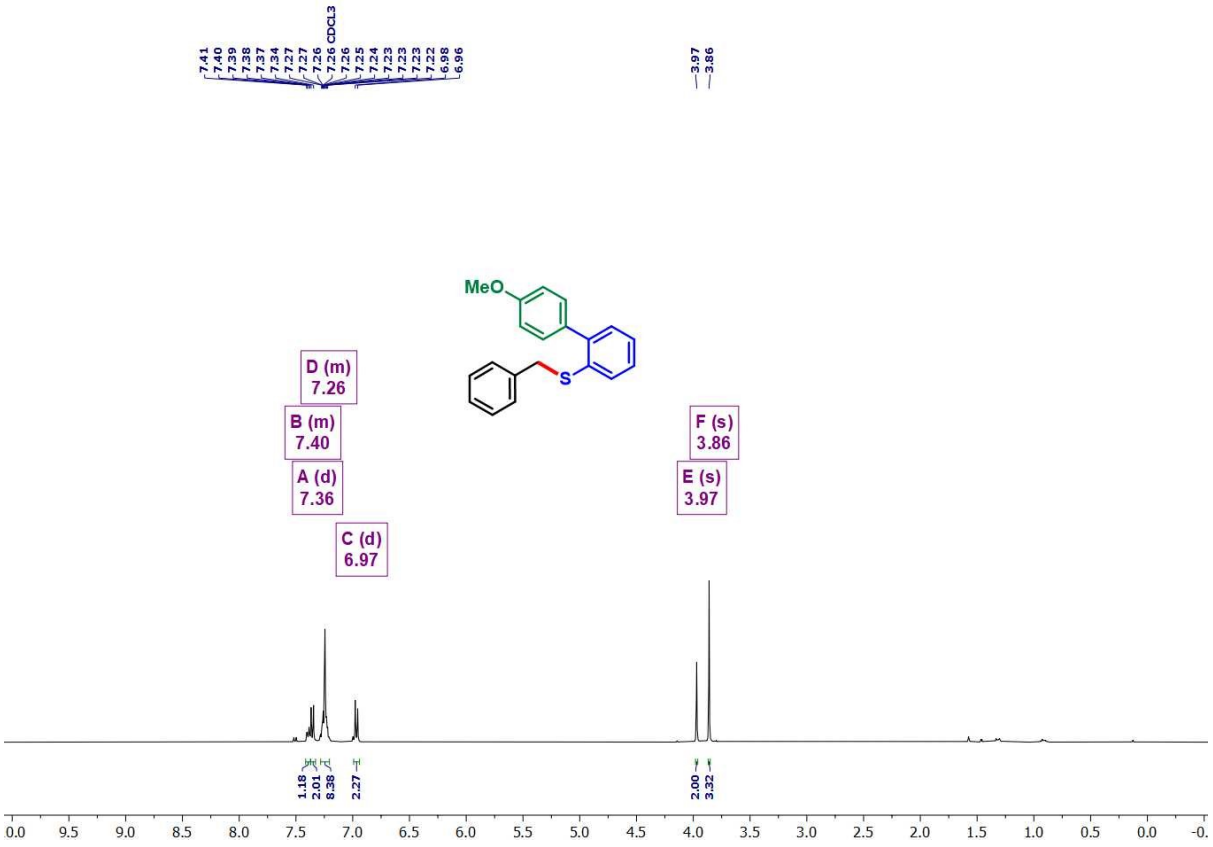
30
20
10
0
-10


7.41
7.40
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7.27
7.27
7.26
CDCL3
7.26
7.26
7.25
7.24
7.23
7.23
7.23
7.22
6.98
6.96
3.97
3.86
MeO
S
D (m)
7.26
B (m)
7.40
A (d)
7.36
C (d)
6.97
F (s)
3.86
E (s)
3.97
1.18
2.01
8.38
2.27
2.00
3.32
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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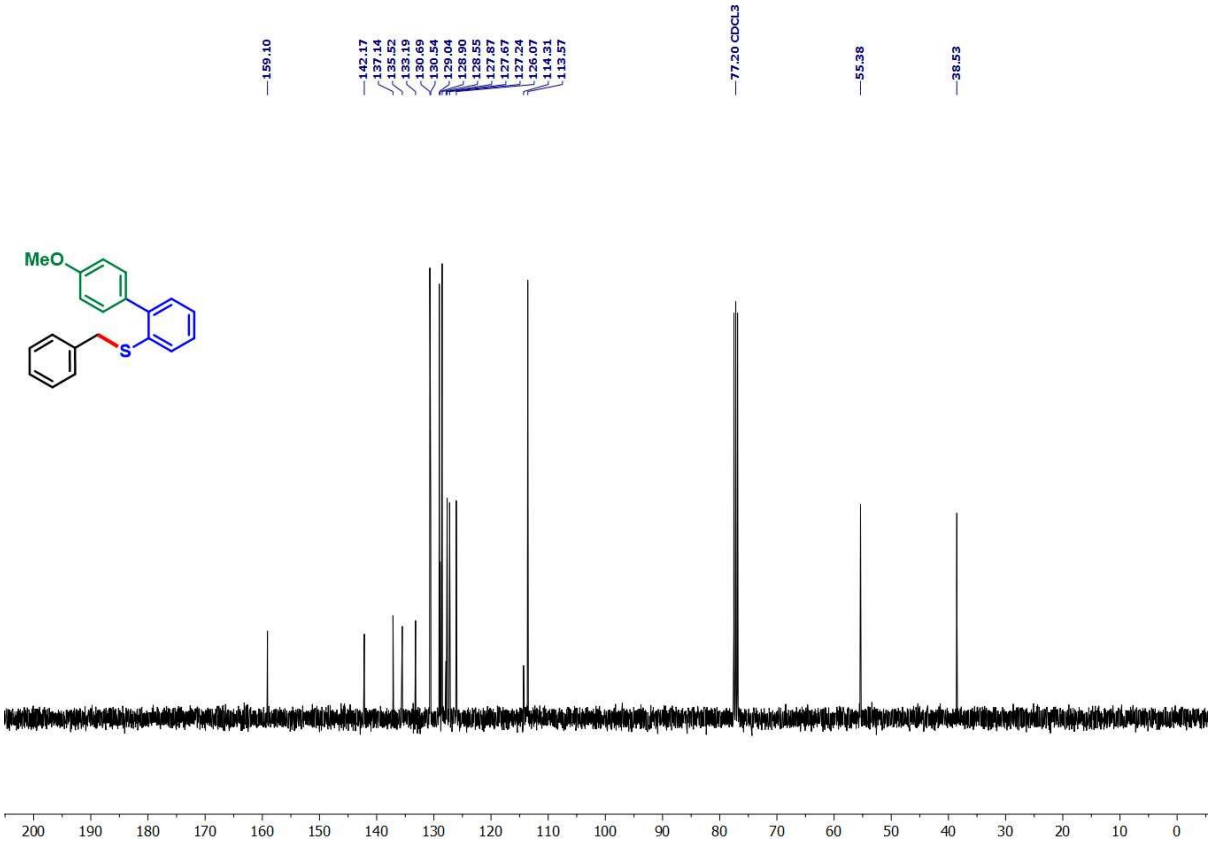
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.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135.52
133.19
130.69
130.54
129.04
128.90
128.55
127.87
127.67
127.24
126.07
114.31
113.57
77.20 CDCL3
55.38
38.53
MeO
S
200
190
180
170
160
150
140
130
120
110
100
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80
70
60
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10
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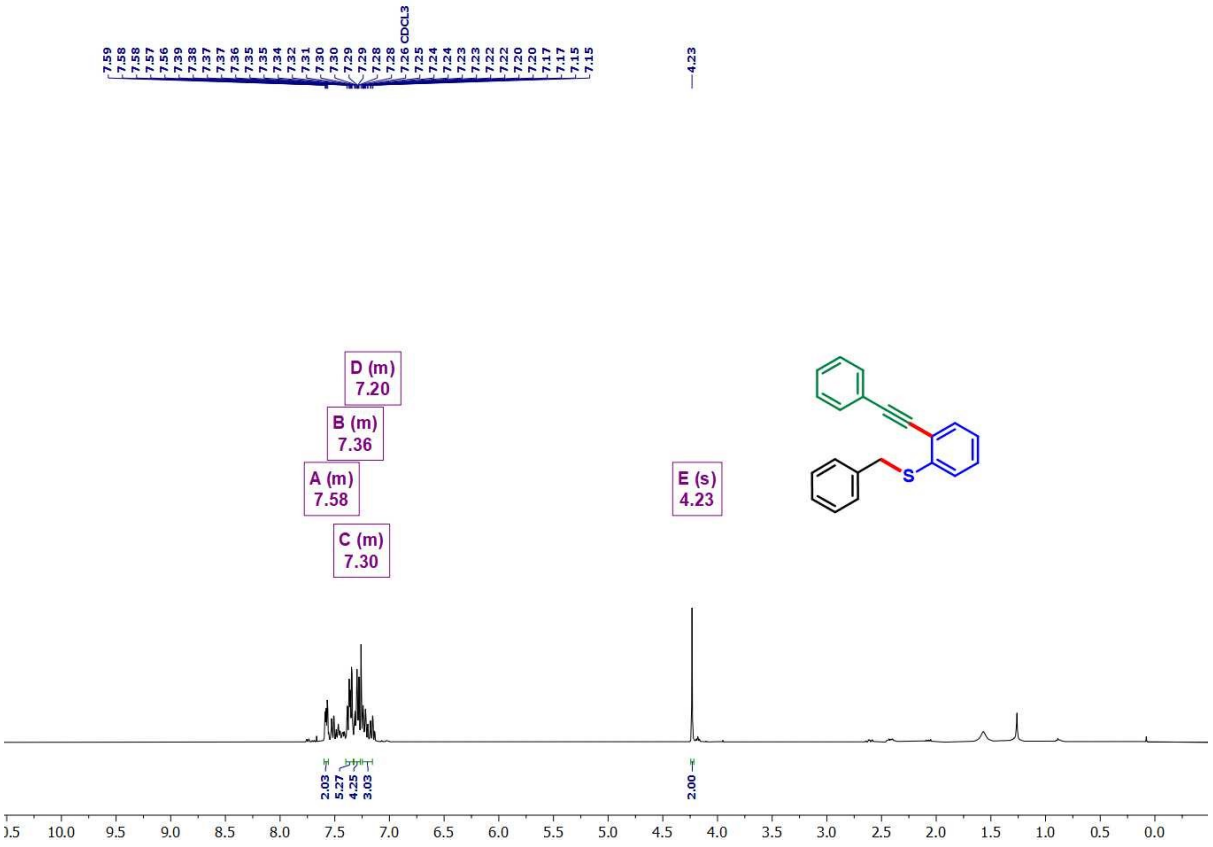
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7.36
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7.32
7.31
7.30
7.30
7.29
7.29
7.28
7.28
7.26 CDCl3
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.20
7.17
7.17
7.15
7.15
4.23
D (m)
7.20
B (m)
7.36
A (m)
7.58
C (m)
7.30
E (s)
4.23
S
2.03
5.27
4.25
3.03
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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2.0
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1.0
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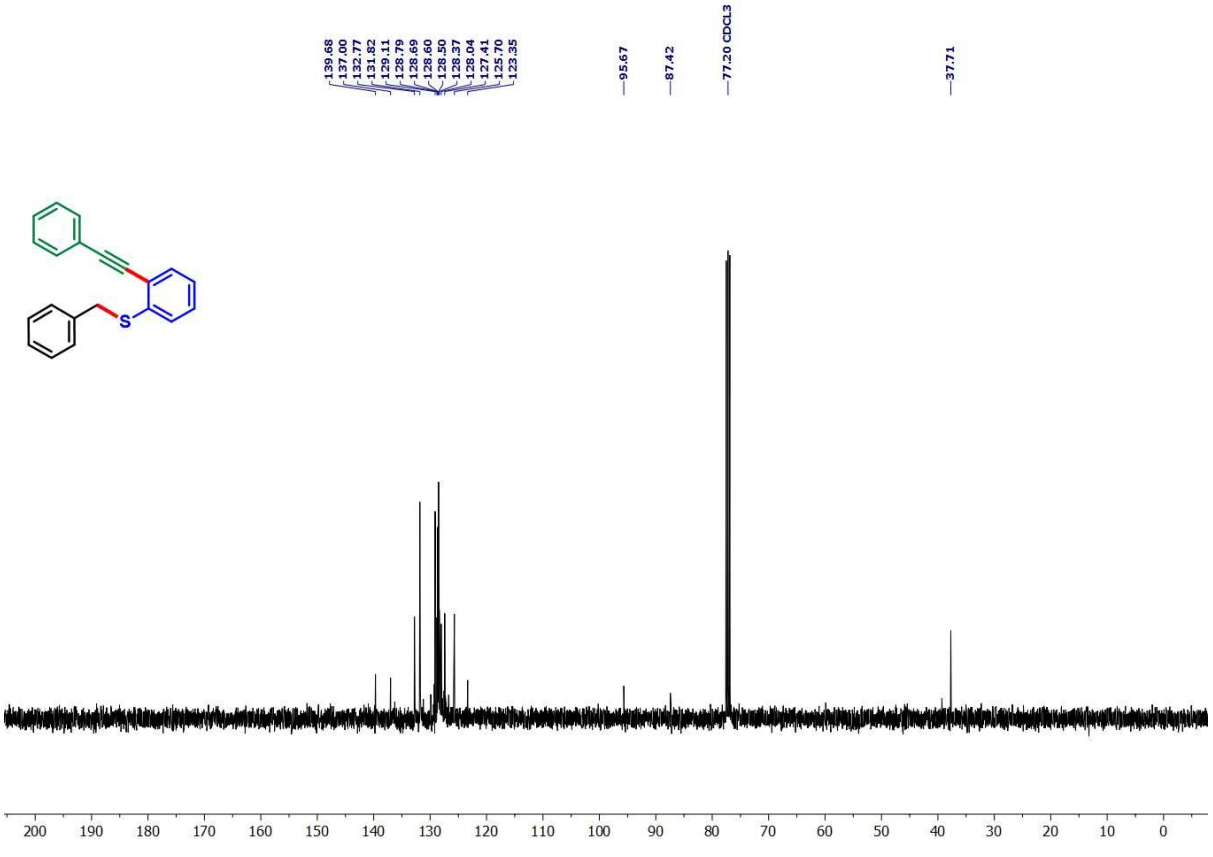
0.0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128.66
128.50
128.37
128.04
127.41
125.70
123.35
95.67
87.42
77.20 CDCl3
37.71
S
200
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180
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150
140
130
120
110
100
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80
70
60
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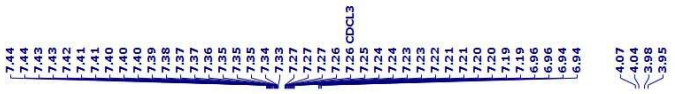
30
20
10
0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7.37
7.37
7.36
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7.35
7.34
7.33
7.27
7.27
7.27
CDCL3
7.26
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.21
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7.19
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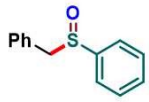
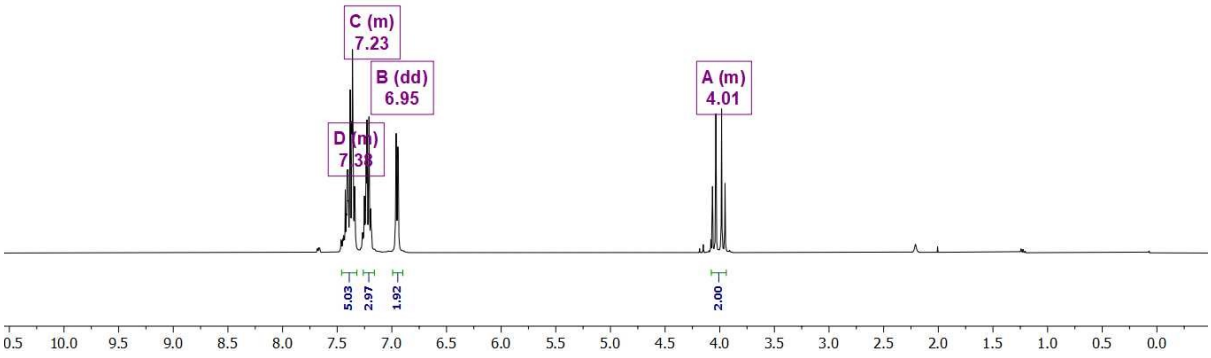
6.96
6.94
6.94
4.07
4.04
3.98
3.95


O
S
Ph


C (m)
7.23
B (dd)
6.95
D (m)
7.38
A (m)
4.01
5.03
2.97
1.92
2.00
0.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0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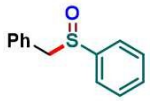
129.14
128.85
128.43
128.23
124.41
77.20 CDCL3
63.51


O
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Ph

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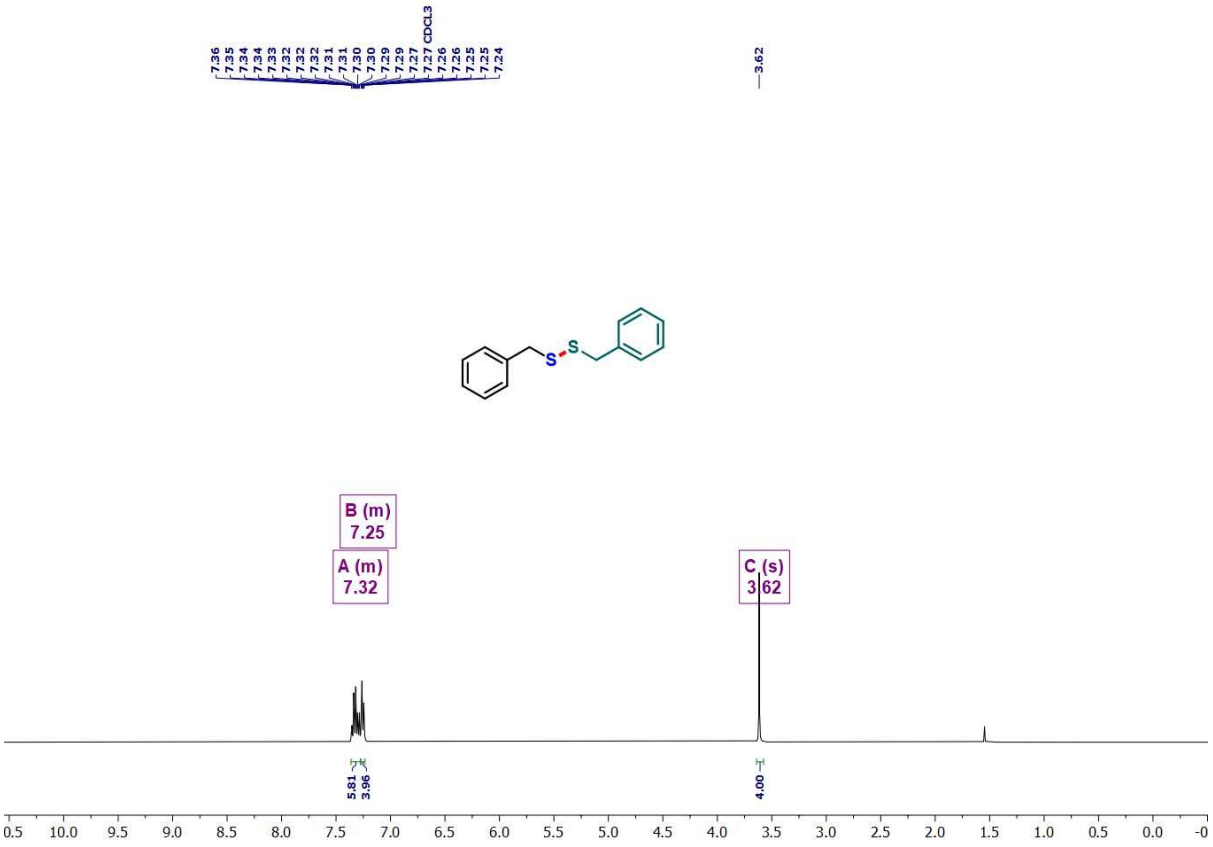
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7.30
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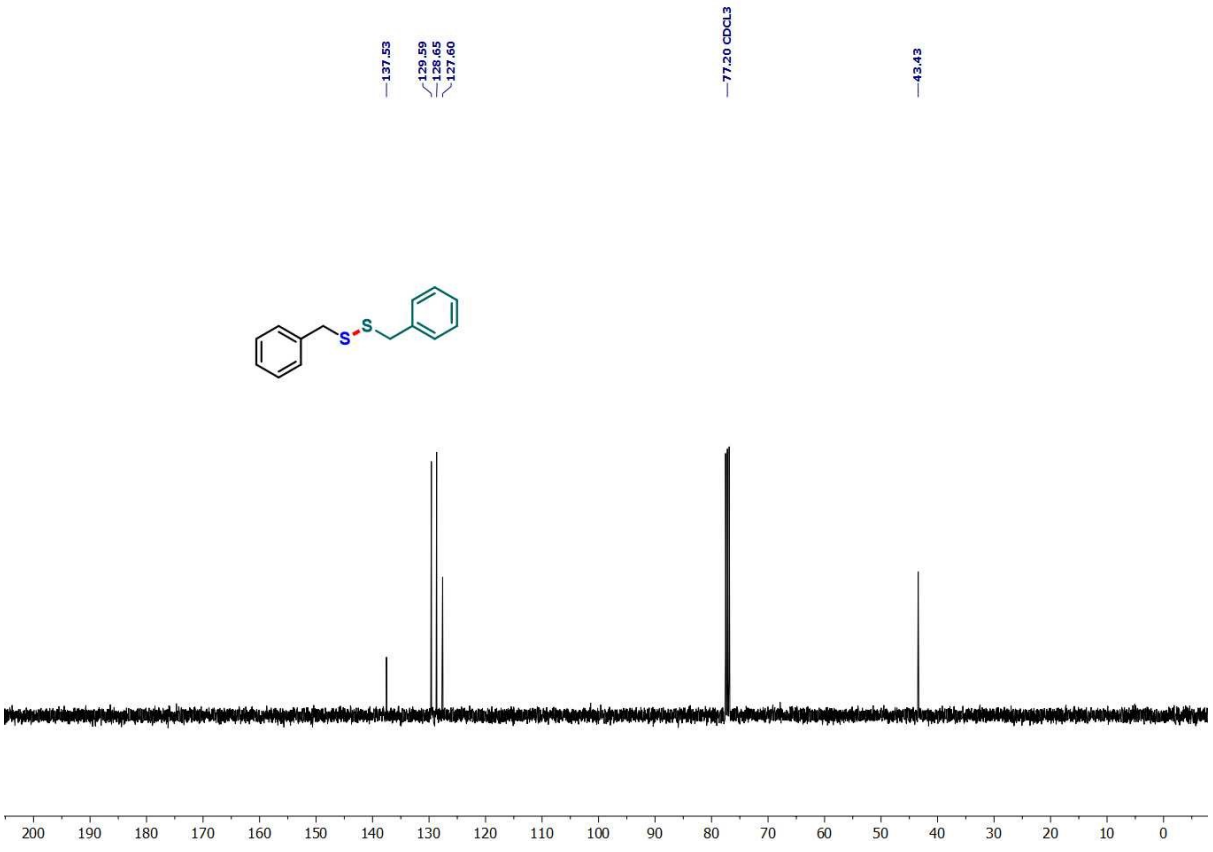
7.29
7.29
7.27
CDCL3
7.26
7.26
7.25
7.25
7.24
3.62
B (m)
7.25
A (m)
7.32
C (s)
3.62
5.81
3.96
4.00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128.65
127.60
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43.43