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Supporting Information

Electrophotocatalytic C–H Heterofunctionalization of Arenes

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Table of Contents

1. General Information

Commercially available reagents were purchased from Sigma Aldrich, Matrix Chemical, AKSci, Alfa Aesar, Oakwood Chemical, Combi-Blocks or TCI, and used as received unless otherwise noted. Silica gel 60 (230-400 mesh) from SiliCycle was used for chromatography, and Merck silica gel plates with a fluorescence F_{254} indicator were used for thin-layer chromatography (TLC) analysis. ^{1}H and ^{13}C NMR spectra were recorded on Mercury-300 (300 MHz), Inova-400 (400 MHz), and Inova-500 (500 MHz) spectrometers. Chemical shifts in ^{1}H NMR spectra are reported in parts per million (ppm) relative to residual chloroform (7.26 ppm) or dimethyl sulfoxide (2.50 ppm) as internal standards. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet), coupling constant in Hertz (Hz) and number of hydrogen atoms based on integration intensities. ^{13}C NMR chemical shifts are reported in ppm relative to the central peak of $CDCl_3$ (77.16 ppm) CD_3OD (49.00 ppm) or $(CD_3)_2SO$ (39.52 ppm) as internal standards. ^{19}F NMR chemical shifts are reported in ppm relative to the central peak of $C_6H_5CF_3$ (-63.72 ppm) as an internal standard. The mass spectral (MS) data were obtained on a Thermo Fisher Scientific Exactive series DART Mass Spectrometer.

2. General Procedures

Electrode preparation can be found in our previous report[1].

Materials used for set-up:

Platinum wire (13039-BU from Alfa Aesar, 25 cm). Blue LED strips (Solid Apollo). Holmes Lil' Blizzard 8-inch oscillating table fan (Amazon). June gold 2.0 mm 2B pencil lead refills (Amazon). DC Power supply (Amazon, 30V/5A). Carbon felt (cut around 7 mm x 7 mm x 7 mm) from C200 Soft Carbon Battery Felt (fuelcellstore, Product Code: 1595010).

The set-up is shown in Fig. S1.

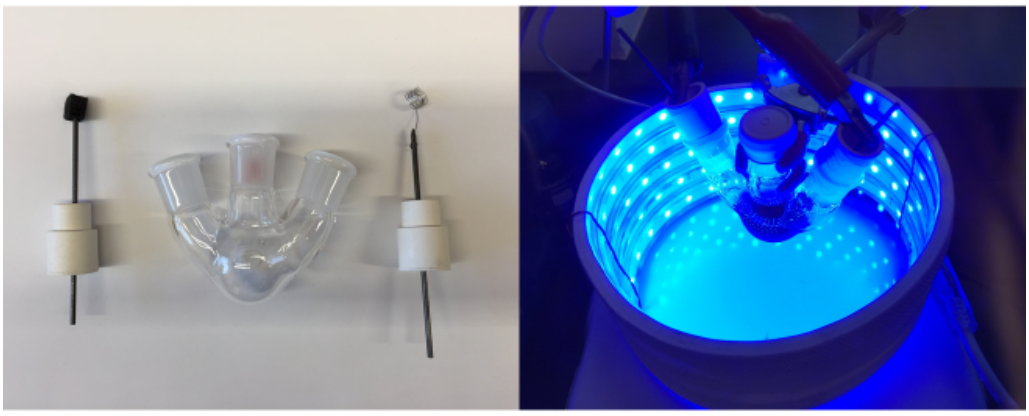


Fig. S1. Experiment set up

Procedure A for compounds 11-24

To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (9.1 mg, 0.04 mmol), arene (0.4 mmol, if solid), and $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL), water (361 μL, 20 mmol), arene (0.4 mmol, if liquid), and acetic acid (229 μL, 4.0 mmol). The reaction mixture was then purged with nitrogen gas for an additional 5 min. The solution was then stirred at room temperature with irradiation from a blue LED strip and under a controlled potential of 1.5 V for 48 h. The reaction mixture was then poured into water (ca. 10 mL). The carbon felt anode was washed with EtOAc (3×5 mL) in an ultrasonic bath. The aqueous layer was separated and extracted with EtOAc (3×10 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration *in vacuo*, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Procedure B for compounds 25, 27-32

To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (9.1 mg, 0.04 mmol), arene (0.4 mmol, if solid), and $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL), arene (0.4 mmol, if liquid), alcohol (8 – 20 mmol), and acetic acid (229 μL, 4.0 mmol). The reaction mixture was then purged with nitrogen gas for an additional 5 min. The solution was then stirred at room temperature with irradiation from a blue LED strip and under a controlled potential of 1.5 V for 48 h. The reaction mixture was subsequently poured into a saturated sodium bicarbonate solution (ca. 20 mL). The carbon felt anode was washed with EtOAc (3×5 mL) in an ultrasonic bath. The aqueous layer was separated and extracted with EtOAc (3×10 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration *in vacuo*, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Procedure C for compounds 33-34

To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (9.1 mg, 0.04 mmol), benzene (0.4 mmol), acid (4 - 6 mmol), and $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL). The reaction mixture was then purged with nitrogen gas for an additional 5 min. The solution was then stirred at room temperature with irradiation from a blue LED strip and under a controlled potential of 1.5 V for 48 h. The reaction mixture was subsequently poured into a saturated sodium bicarbonate solution (ca. 20 mL). The carbon felt anode was washed with EtOAc (3×5 mL) in an ultrasonic bath. The aqueous layer was separated and extracted with EtOAc (3×10 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration *in vacuo*, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Procedure D for compounds 35-46

To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (9.1 mg, 0.04 mmol), arene (0.4 mmol, if solid), amide (1.2 - 6 mmol) and $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with

nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL), arene (0.4 mmol, if liquid), and acetic acid (229 μL, 4.0 mmol). The reaction mixture was then purged with nitrogen gas for an additional 5 min. The solution was then stirred at room temperature with irradiation from a blue LED strip and under a controlled potential of 1.5 V for 48 h. The reaction mixture was subsequently poured into a saturated sodium bicarbonate solution (ca. 20 mL). The carbon felt anode was washed with EtOAc (3×5 mL) in an ultrasonic bath. The aqueous layer was separated and extracted with EtOAc (3×10 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration *in vacuo*, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product.

Procedure E for direct electrolysis in Table 1

To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL), water (361 μL, 20 mmol), benzene (35.6 μL, 0.4 mmol), and acetic acid (229 μL, 4.0 mmol). The reaction mixture was then purged with nitrogen gas for an additional 5 min. The solution was then stirred at room temperature under a controlled potential of 3.0 V for 48 h. Yields and conversions were determined by ^1H NMR with dibromomethane as an internal standard.

Notes for procedures:

1. Because the carbon felt can absorb a significant amount of reaction solution, it should be rinsed in an ultrasonic bath for 5 min or more to obtain optimal product yields.

2. In the undivided cell, it is best to keep the anode and cathode relatively close (~0.5 cm) to one another; however, they should not touch.

3. For the cathode, the platinum wire should be immersed in the solution, but not the pencil lead to which it is attached.

4. The carbon felt should be replaced for each reaction.

5. The system was kept cool using a fan.

6. High stirring speed is required.

7. After the reaction, care should be taken when removing the septum in case of pressure build up from hydrogen gas generation.

3. Gram Scale Procedures

Electrode preparation can be found in our previous report[2].

Materials used for set-up:

Platinum foil (42456-FF, 0.05 mm thick from Alfa Aesar, 25 * 25 mm). DC Power supply (Amazon, 30V/5A). Carbon felt (cut around 20 mm x 10 mm x 7 mm) from C200 Soft Carbon Battery Felt (fuelcellstore, Product Code: 1595010).

The set-up is shown in Fig. S2.

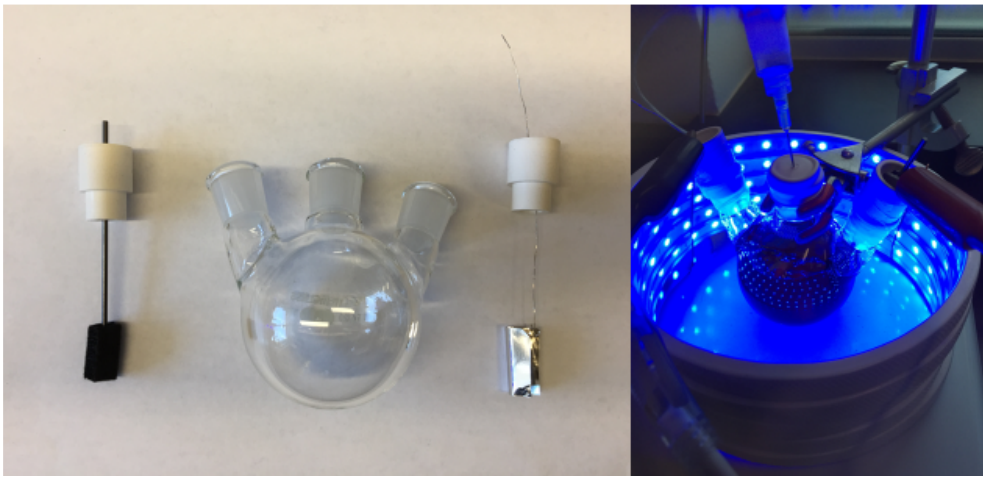


Fig. S2. Large scale experiment set-up

Procedure for gram scale reaction

To an oven-dried 100-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (90.8 mg, 0.4 mmol) and $LiClO_4$ (2.55 g, 24 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 15 min, followed by the sequential addition via syringe of CH_3CN (60 mL), water (3.6 mL, 200 mmol), benzene (0.36 mL, 4 mmol), and acetic acid (2.29 mL, 40 mmol). The reaction mixture was then purged with nitrogen gas for an additional 15 min. A nitrogen gas balloon was connected to the flask by a needle. The solution was then stirred at room temperature with irradiation from a blue LED strip and under a controlled potential of 1.5 V for 96 h. The reaction mixture was subsequently poured into water (ca. 30 mL). The carbon felt anode was washed with EtOAc (3×30 mL) in an ultrasonic bath. The aqueous layer was separated and extracted with EtOAc (3×60 mL), and the combined organic layers were washed with brine and dried over anhydrous Na_2SO_4. Following concentration *in vacuo*, the crude residue was subjected to flash column chromatography on silica gel to yield the desired product in 52% yield (195.7 mg).

4. Electrophotocatalysis in Flow

Materials used for set-up:

Peristaltic pump (INZOK 12V DC large flow dosing pump, Amazon), PFA tubing (1514L, IDEX-HS), silicone tubing (96410-13, Cole Parmer).

Photo reactor channel set up:

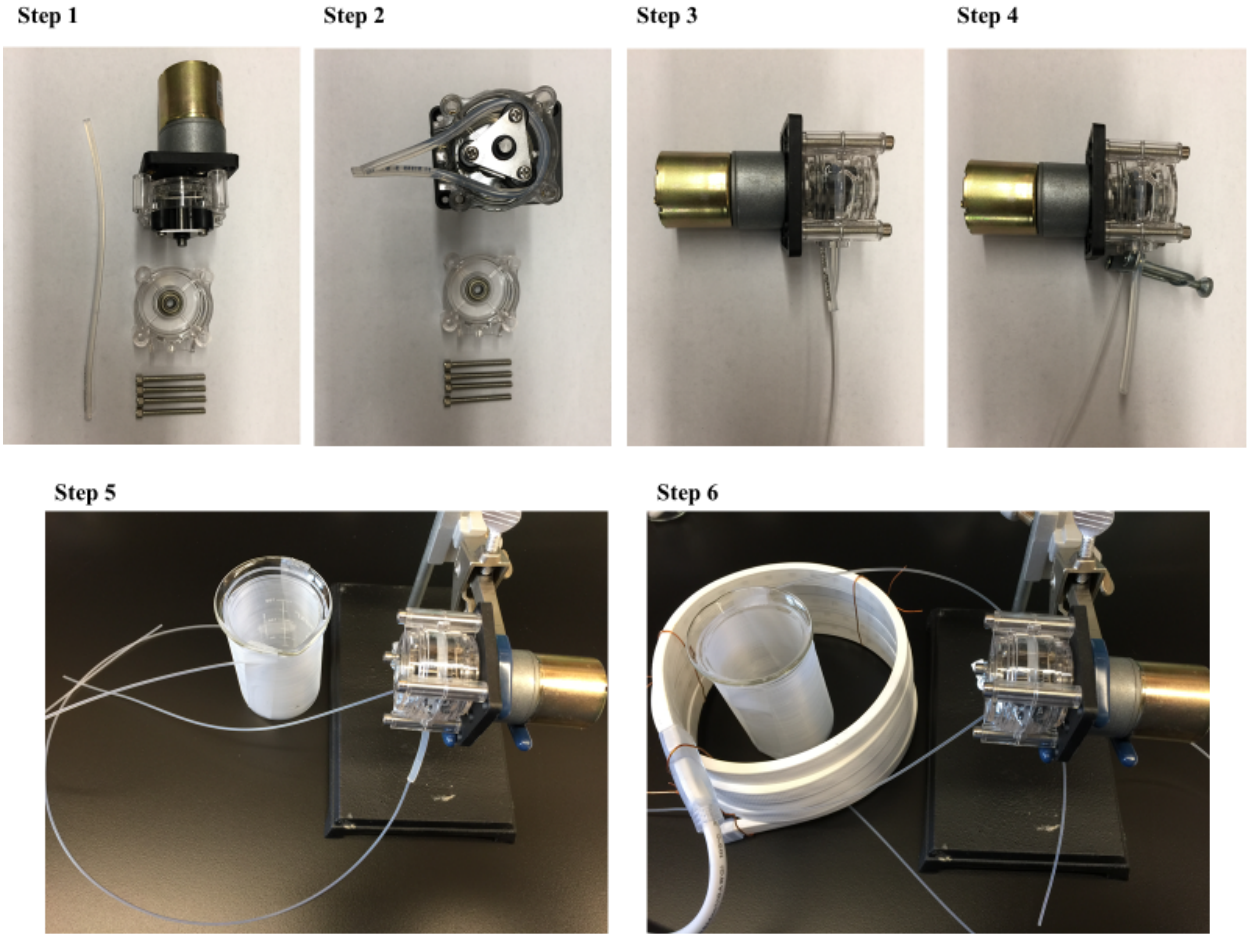


Fig. S3. Photo reactor channel set-up

The original pump tubing was replaced with ~18 of cm silicone tubing (Step 1→Step 2). A piece of PFA tubing was inserted into one side of the silicone tubing (Step 3), with a clamp placed on the connecting part to hold the tubing in place (Stepp 4). PFA tubing was wound around the outside of a small beaker and connected to the other side of the silicone tubing (Step 5). A blue LED strip was coiled around the beaker. (Step 6).

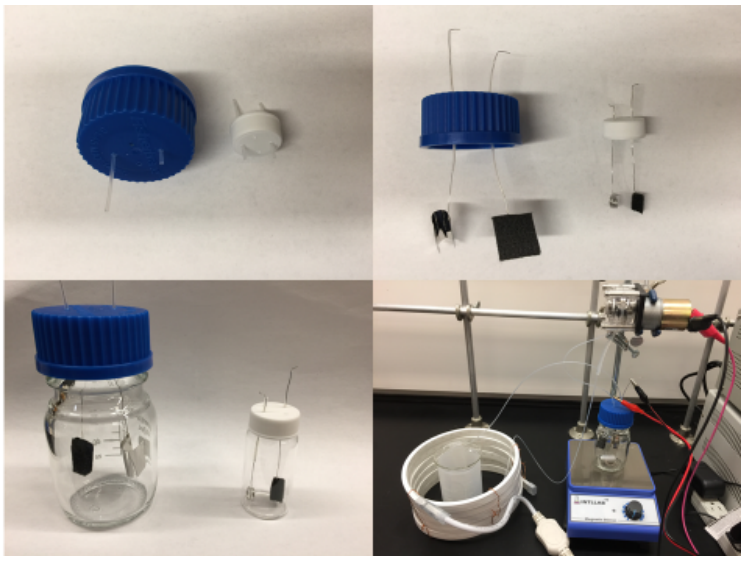


Fig. S4. Undivided cell set-up

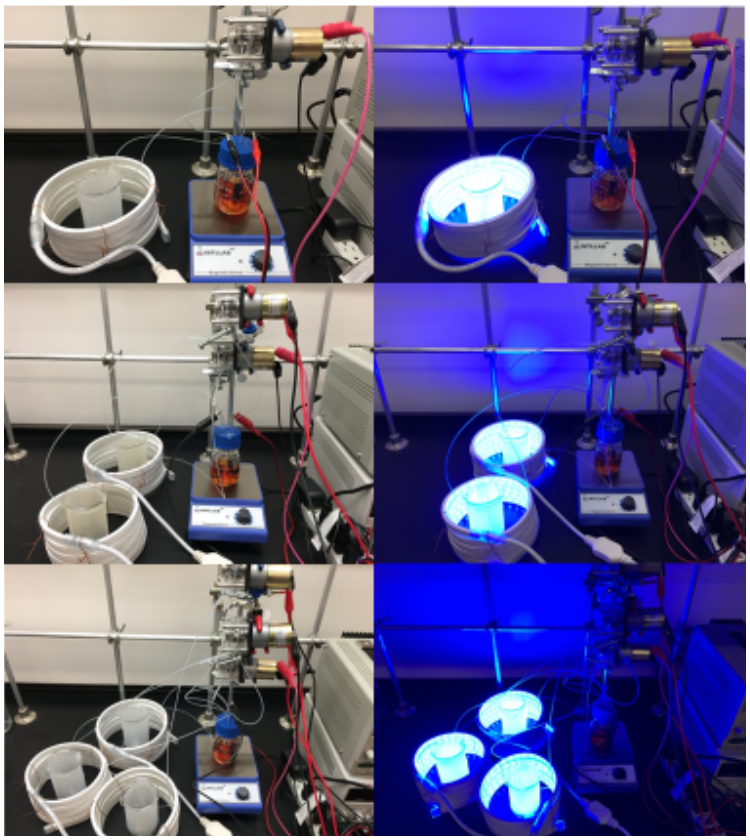


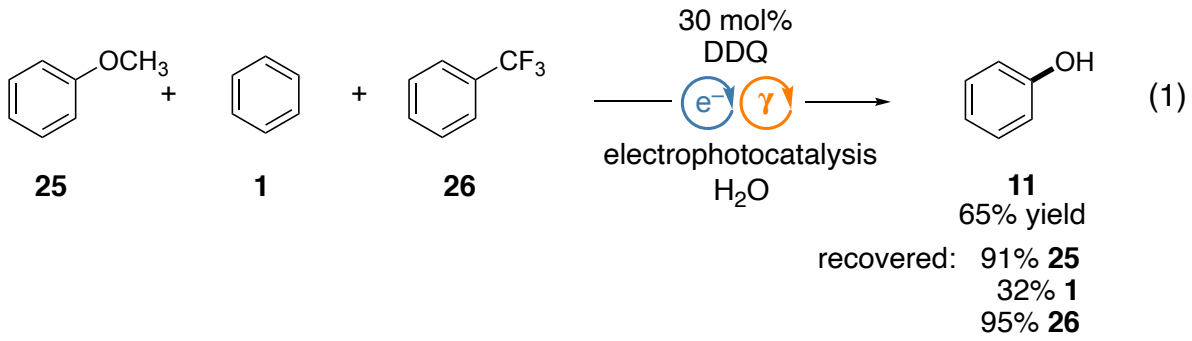
Fig. S5. Electrophotocatalysis in flow using one, two, or three photo reactor channels

Procedures for the reactions in flow

For 4 mmol scale: to an oven-dried 100-mL bottle equipped with a stir bar were added DDQ (90.8 mg, 0.4 mmol) and $LiClO_4$ (2.55 g, 24 mmol). The cell was sealed by screwing on the lid, attached to which were a carbon felt anode (~ 20 mm x 20 mm x 7 mm) and a platinum wire cathode (Fig. S4). The bottle was then flushed with nitrogen gas for 15 min, followed by the sequential addition via syringe of CH_3CN (60 mL), water (3.6 mL, 200 mmol), benzene (0.36 mL, 4 mmol), and acetic acid (2.29 mL, 40 mmol). The reaction mixture was then purged with nitrogen gas for an additional 15 min with stirring. The reaction mixture was then pumped through one, two, or three photo reactor channels with a 3 min residence time. The solution in the bottle was stirred at room temperature under a controlled potential of 1.5 V, with the photoreactor channel(s) under irradiation from a blue LED strip. After completion, the yield was determined by UHPLC with dimethyl maleate as an internal standard (calibration curve).

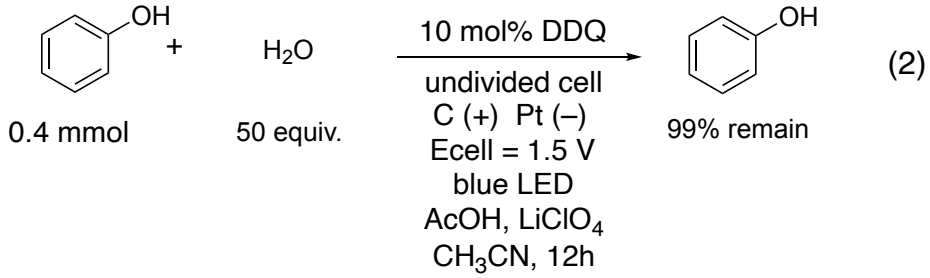
For 15 mmol scale: to an oven-dried 250-mL bottle equipped with a stir bar were added DDQ (340.5 mg, 1.5 mmol) and $LiClO_4$ (9.56 g, 90 mmol). The cell was sealed by screwing on the lid, attached to which were a carbon felt anode (~ 35 mm x 20 mm x 7 mm) and a platinum wire cathode (Fig. S4). The bottle was then flushed with nitrogen gas for 15 min, followed by the sequential addition via syringe of CH_3CN (225 mL), water (13.5 mL, 750 mmol), benzene (1.35 mL, 15 mmol), and acetic acid (8.59 mL, 150 mmol). The reaction mixture was then purged with nitrogen gas for an additional 15 min with stirring. The reaction mixture was then pumped through three photo reactor channels with a 3 min residence time. The solution was stirred at room temperature under a controlled potential of 1.5 V, with the photoreactor channels under irradiation from a blue LED strip. After completion, the yield was determined by UHPLC with dimethyl maleate as an internal standard (calibration curve).

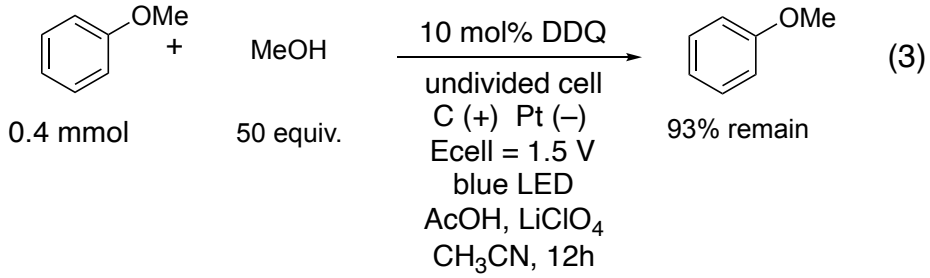
5. Demonstration of Chemoselectivity



For eq. 1: To an oven-dried 10-mL three-neck flask equipped with a stir bar, a carbon felt anode, and a platinum wire cathode were added DDQ (27.3 mg, 0.12 mmol) and $LiClO_4$ (255.3 mg, 2.4 mmol). The cell was sealed using a rubber septum and parafilm and then flushed with nitrogen gas for 5 min, followed by the sequential addition via syringe of CH_3CN (6 mL), water (361 µL, 20 mmol), anisole (8.7 µL, 0.08 mmol), benzene (36 µL, 0.4 mmol), trifluorotoluene (49 µL, 0.4 mmol), and acetic acid (229 µL, 4.0 mmol). The reaction mixture was then purged with nitrogen gas carefully for an additional 2 min. The solution was then stirred at room temperature under irradiation from a blue LED strip and at a controlled potential of 1.5 V for 36 h. Yields and conversions were determined by ^1H NMR with dibromomethane as an internal standard.

6. Control Experiments





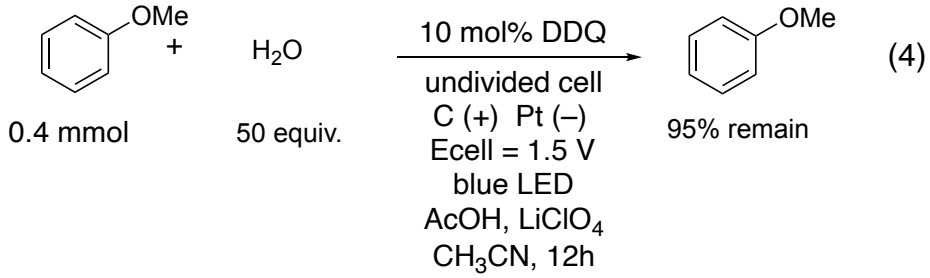
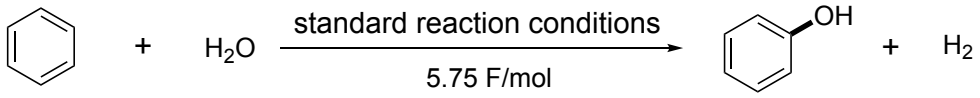


Fig. S6. Control experiments

7. Current Profile



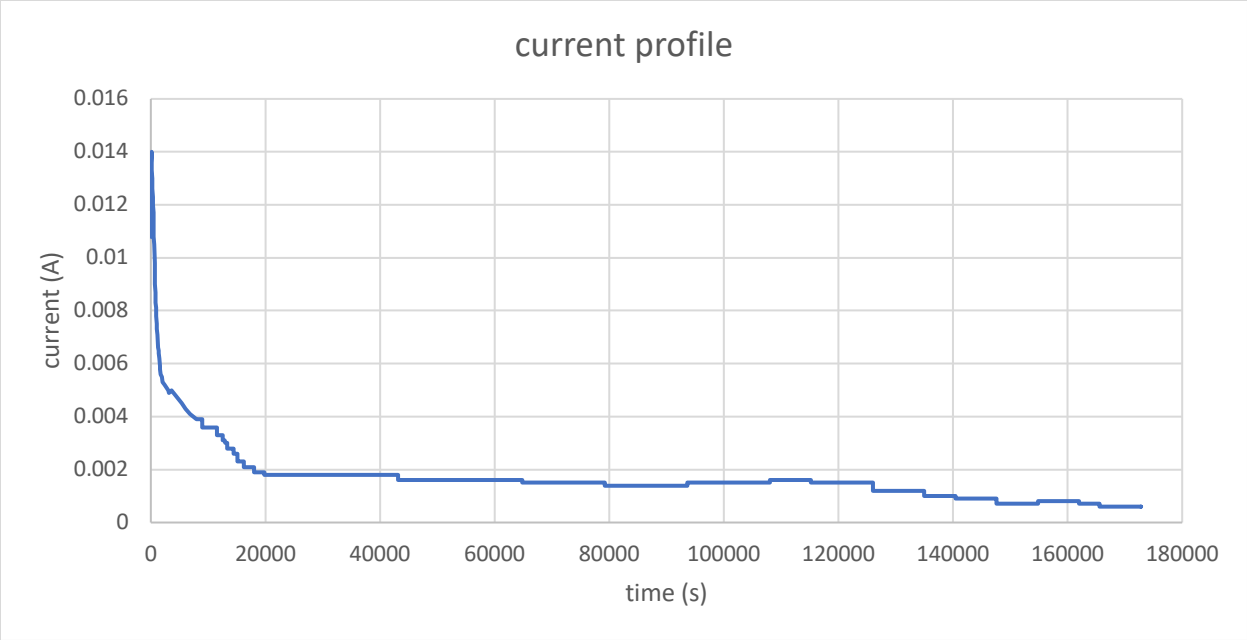
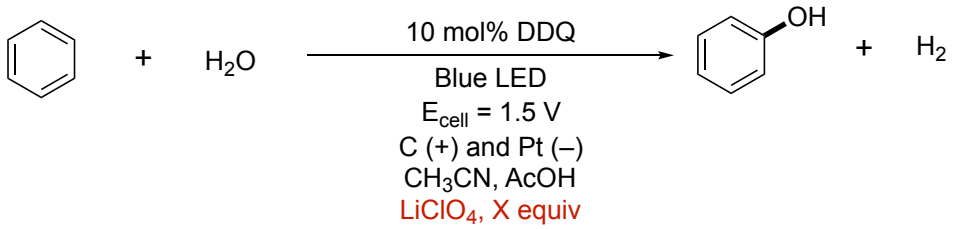


Fig S7. Current profile

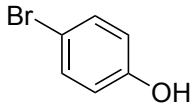
8. Optimization of Reaction Conditions



$LiClO_4$	**yield**
$LiClO_4$, 1 equiv, 48 h	**33%**
$LiClO_4$, 3 equiv, 48 h	**67%**
$LiClO_4$, 3 equiv, 60 h	**75%**
$LiClO_4$, 6 equiv, 48 h	**80%**

Fig. S8. Reaction condition optimization

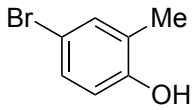
9. Characterization



4-bromophenol **(12)**: The title compounds were prepared from bromobenzene (0.4 mmol, 41.9 μL) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 20% EtOAc/hexanes to furnish a colorless oil in 55% yield (38.1 mg).

12: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 (d, *J* = 8.8 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 5.12 (br, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 154.1, 129.5, 125.7, 116.7.

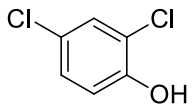
MS (DART) exact mass: calculated for $(M-H)^-$: 170.9451; found: 170.9450.



4-bromo-2-methylphenol **(13)**: The title compounds were prepared from 1-bromo-3-methylbenzene (0.4 mmol, 68.4 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 50% yield (37.4 mg).

13: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.32 – 7.23 (m, 1H), 7.19 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.67 (d, *J* = 8.5 Hz, 1H), 5.32 (br, 1H), 2.25 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 152.9, 133.5, 129.8, 126.2, 116.5, 112.6, 15.6.

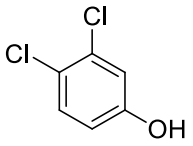
MS (DART) exact mass: calculated for $(M-H)^-$: 184.9608; found:184.9601.



2,4-dichlorophenol **(14)**: The title compound was prepared from 1,3-dichlorobenzene (0.4 mmol, 45.6 μL) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 36 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 69% yield (45.0 mg).

14: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 2.5 Hz, 1H), 7.18 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.98 (d, *J* = 8.8 Hz, 1H), 5.52 (br, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 150.2, 128.6, 128.6, 125.6, 120.4, 117.1.

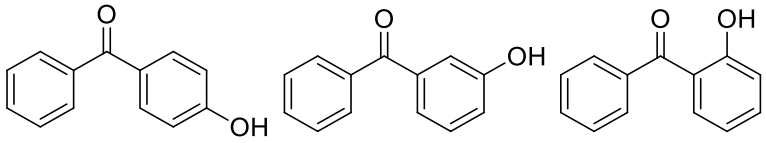
MS (DART) exact mass: calculated for $(M-H)^-$: 160.9566; found:160.9558.



***3,4-dichlorophenol* (15)**: The title compound was prepared from 1,2-dichlorobenzene (0.4 mmol, 45.2 µL) and water (20 mmol, 361 µL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 20% EtOAc/hexanes to furnish a colorless oil in 41% yield (26.7 mg).

15: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 (d, *J* = 8.8 Hz, 1H), 6.99 (d, *J* = 2.9 Hz, 1H), 6.72 (dd, *J* = 8.7, 2.9 Hz, 1H), 5.14 (s, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 154.6, 133.0, 130.9, 124.2, 117.5, 115.3.

MS (DART) exact mass: calculated for $(M-H)^-$: 160.9566; found: 160.9559.



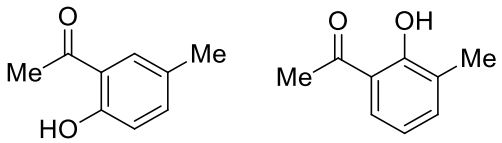
***4-hydroxybenzophenone, 3-hydroxybenzophenone, and 2-hydroxybenzophenone* (16a, 16b** and **16c)**: The title compound was prepared from benzophenone (0.4 mmol, 72.9 mg) and water (20 mmol, 361 µL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 46% yield (36.5 mg).

16a: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.83 – 7.76 (m, 4H), 7.62 – 7.49 (m, 4H), 6.94 (d, *J* = 8.6 Hz, 2H), 6.01 (br, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 196.0, 159.9, 138.1, 133.0, 132.1, 130.2, 129.8, 128.3, 115.2.

MS (DART) exact mass: calculated for $(M-H)^-$: 197.0608; found:197.0603.

16b: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.88 – 7.77 (m, 2H), 7.67 – 7.58 (m, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 7.42 – 7.32 (m, 3H), 7.15 – 7.05 (m, 1H), 5.40 (br, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 196.6, 155.7, 139.0, 137.4, 132.6, 130.1, 129.6, 128.3, 122.9, 119.7, 116.5.

MS (DART) exact mass: calculated for $(M-H)^-$: 197.0608; found: 197.0603.

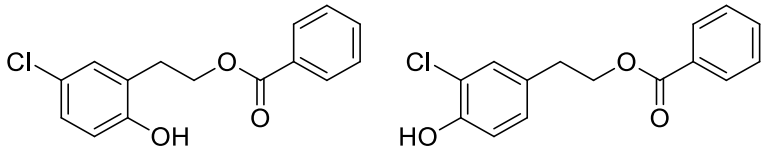


***2-hydroxy-5-methylacetophenone and 2-hydroxy-3-methylacetophenone* (17a and 17b)**: The title compounds were prepared from 3-methylacetophenone (0.4 mmol, 53.7 mg) and water (20 mmol, 361 µL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was

purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 52% yield (31.2 mg).

20: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **20a and 20b**) δ 12.34 (br, 1H), 7.66 – 7.47 (m, 1H), 7.43 – 7.29 (m, 1H), 6.98 – 6.75 (m, 1H), 2.65 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **20a and 20b**) δ 204.8, 204.4, 160.9, 160.3, 137.5, 137.2, 130.4, 128.3, 128.0, 127.5, 119.4, 119.0, 118.2, 118.2, 26.7, 26.6, 20.5, 15.4.

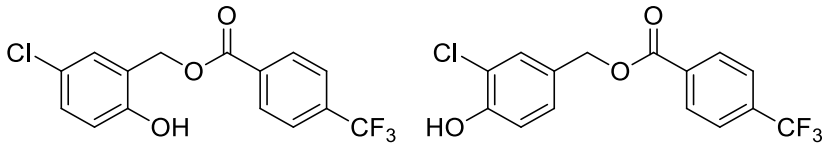
MS (DART) exact mass: calculated for $(M-H)^-$: 149.0608; found:149.0602.



5-chloro-2-hydroxyphenethyl benzoate and 3-chloro-4-hydroxyphenethyl benzoate **(18a and 18b)**: The title compounds were prepared from 3-chlorophenethyl benzoate (0.4 mmol, 104.3 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 43% yield (47.6 mg).

18: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **18a** and **18b**) δ 8.13 – 8.01 (m, 2H), 7.60 (q, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 2H), 7.28 – 7.08 (m, 2H), 6.99 (d, *J* = 8.3 Hz, 0.27H), 6.82 (d, *J* = 8.5 Hz, 0.76H), 4.59 – 4.45 (m, 2H), 3.13 – 2.96 (m, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **18a** and **18b**) δ 167.4, 153.2, 133.3, 133.1, 130.6, 130.2, 129.8, 129.7, 129.6, 129.3, 129.0, 128.5, 128.4, 128.1, 125.3, 125.1, 117.1, 116.3, 65.3, 64.4, 34.2, 30.1.

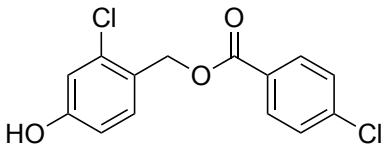
MS (DART) exact mass: calculated for $(M-H)^-$: 275.0480; found:275.0476.



5-chloro-2-hydroxybenzyl 4-(trifluoromethyl)benzoate and 3-chloro-4-hydroxybenzyl 4-(trifluoromethyl)benzoate **(19a and 19b)**: The title compounds were prepared from 3-chlorobenzyl 4-(trifluoromethyl)benzoate (0.4 mmol, 125.9 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 62% yield (82.0 mg).

19: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **19a** and **19b**) δ 8.19 (d, *J* = 7.8 Hz, 2H), 7.78 – 7.68 (m, 2H), 7.47 – 7.36 (m, 1H), 7.30 – 7.23 (m, 1H), 7.06 (d, *J* = 8.3 Hz, 0.24H), 6.92 (d, *J* = 8.7 Hz, 0.81H), 5.35 (s, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **19a** and **19b**) δ 167.4, 154.1, 135.2 (q, *J* = 33.3 Hz), 131.3, 130.4, 129.5, 129.0, 125.6, 125.6, 125.6, 125.5 (q, *J* = 3.8 Hz), 125.4 (q, *J* = 3.8 Hz), 125.3, 123.7 (q, *J* = 271.3 Hz), 122.9, 119.2, 116.5, 66.4, 63.4. ^{19}F NMR (470 MHz, Chloroform-*d*, mixture of **19a** and **19b**) δ -63.15, -63.24.

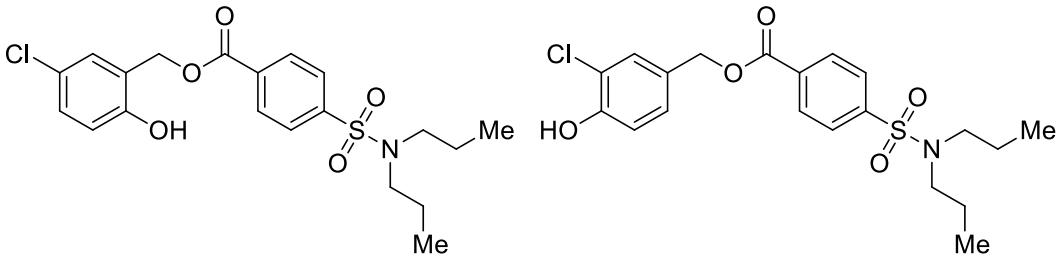
MS (DART) exact mass: calculated for $(M-H)^-$: 329.0198; found: 329.0192.



2-chloro-4-hydroxybenzyl 4-chlorobenzoate **(20a)**: The title compounds were prepared from 2-chlorobenzyl 4-chlorobenzoate (0.4 mmol, 112.4 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 36 hours. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a white solid in 57% yield (67.7 mg).

20a: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.03 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.6 Hz, 2H), 7.29 (d, *J* = 3.2 Hz, 1H), 7.00 (d, *J* = 3.0 Hz, 1H), 6.80 (dd, *J* = 8.6, 3.0 Hz, 1H), 5.42 (s, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.6, 154.5, 139.8, 134.6, 131.2, 130.6, 128.9, 128.2, 124.9, 116.6, 116.6, 64.2.

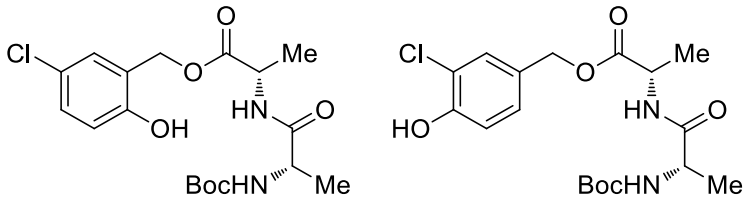
MS (DART) exact mass: calculated for $(M-H)^-$: 294.9934; found: 294.9930.



5-chloro-2-hydroxybenzyl 4-(N,N-dipropylsulfamoyl)benzoate and 5-chloro-4-hydroxybenzyl 4-(N,N-dipropylsulfamoyl)benzoate **(21a and 21b)**: The title compounds were prepared from 3-chlorobenzyl 4-(N,N-dipropylsulfamoyl)benzoate (0.4 mmol, 164.0 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 60% EtOAc/hexanes to furnish a white solid in 41% yield (69.8 mg).

21: ^{1}H NMR (500 MHz Chloroform-*d*, mixture of **21a** and **21b**) δ 8.20 (dd, *J* = 10.2, 8.3 Hz, 2H), 7.97 – 7.89 (m, 2H), 7.35 (d, *J* = 5.2 Hz, 2H), 7.27 – 7.21 (m, 0.5H), 6.91 (d, *J* = 8.7 Hz, 0.5H), 5.37 (s, 2H), 3.18 – 2.89 (m, 4H), 1.63 – 1.41 (m, 4H), 0.95 – 0.80 (m, 6H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **21a** and **21b**) δ 164.9, 154.1, 145.0, 144.4, 137.4, 133.5, 131.5, 131.0, 130.6, 130.5, 130.4, 130.0, 128.7, 128.3, 127.1, 127.1, 127.0, 126.4, 125.3, 122.9, 119.1, 66.5, 63.4, 49.9, 45.1, 23.0, 21.9, 11.1, 11.0.

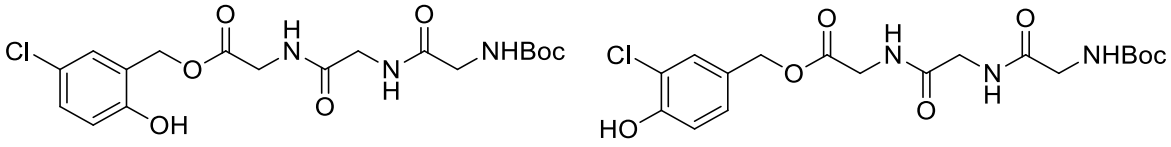
MS (DART) exact mass: calculated for $(M-H)^-$: 424.0991; found: 424.0988.



(S)-5-chloro-2-hydroxybenzyl 2-((S)-2-((tert-butoxycarbonyl)amino)propanamido)propanoate and (S)-3-chloro-4-hydroxybenzyl 2-((S)-2-((tert-butoxycarbonyl)amino)propanamido)propanoate **(22a and 22b)**: The title compounds were prepared from (S)-3-chlorobenzyl 2-((S)-2-((tert-butoxycarbonyl)amino)propanamido)propanoate (0.4 mmol, 154.0 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 70% EtOAc/hexanes to furnish a colorless oil in 37% yield (59.3 mg).

22: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **22a** and **22b**) δ 8.21 (d, *J* = 9.1 Hz, 1H), 7.25–6.98 (m, 3H), 6.83 (dd, *J* = 8.6, 1.5 Hz, 1H), 5.35 (d, *J* = 7.7 Hz, 1H), 5.21–5.02 (m, 2H), 4.57 (t, *J* = 7.3 Hz, 1H), 4.32 – 4.05 (m, 1H), 1.55–1.24 (m, 15H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **22a** and **22b**) δ 173.3, 173.1, 154.0, 154.0, 130.3, 130.3, 130.1, 124.6, 123.2, 117.8, 117.8, 62.9, 48.3, 48.3, 28.3, 18.3, 18.1, 17.9, 17.8.

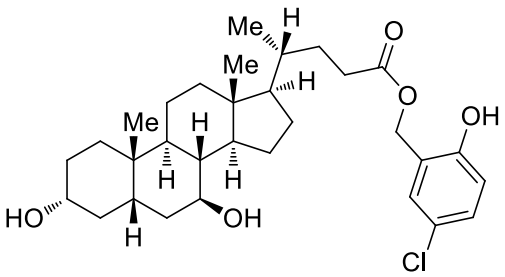
MS (DART) exact mass: calculated for $(M+H)^+$: 401.1474; found: 401.1476.

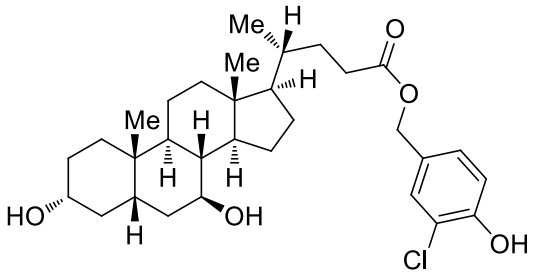


5-chloro-2-hydroxybenzyl 2,2-dimethyl-4,7,10-trioxo-3-oxa-5,8,11-triazatridecan-13-oate and 5-chloro-4-hydroxybenzyl 2,2-dimethyl-4,7,10-trioxo-3-oxa-5,8,11-triazatridecan-13-oate **(23a and 23b)**: The title compounds were prepared from 3-chlorobenzyl 2,2-dimethyl-4,7,10-trioxo-3-oxa-5,8,11-triazatridecan-13-oate (0.4 mmol, 165.6 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 90% EtOAc/hexanes to furnish a colorless oil in 32% yield (55.0 mg).

23: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **23a** and **23b**) δ 8.64 (br, 1H), 7.78 – 7.47 (m, 2H), 7.20 – 7.00 (m, 2H), 6.80 (d, *J* = 8.6 Hz, 1H), 5.85 (d, *J* = 5.9 Hz, 1H), 5.09 (s, 2H), 4.13 – 3.60 (m, 6H), 1.40 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **23a** and **23b**) δ 171.3, 170.4, 170.3, 156.9, 154.0, 130.3, 130.0, 124.4, 123.1, 117.5, 80.7, 62.8, 44.1, 42.7, 41.4, 28.3.

MS (DART) exact mass: calculated for $(M+H)^+$: 430.1376; found: 430.1378.

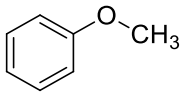




(R)-5-chloro-2-hydroxybenzyl 4-((3R,5S,7S,8R,9S,10S,13R,14S,17R)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate and (R)-5-chloro-4-hydroxybenzyl 4-((3R,5S,7S,8R,9S,10S,13R,14S,17R)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate **(24a and 24b)**: The title compounds were prepared from (R)-3-chlorobenzyl 4-((3R,5S,7S,8R,9S,10S,13R,14S,17R)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate (0.4 mmol, 206.8 mg) and water (20 mmol, 361 μL) according to general procedure A with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 100% EtOAc/hexanes to furnish a white solid in 45% yield (96.0 mg).

24: ^{1}H NMR (500 MHz Chloroform-*d*, mixture of **24a** and **24b**) δ 8.38 (br, 1H), 7.26 – 7.11 (m, 2H), 6.82 (d, *J* = 8.6 Hz, 1H), 5.07 (q, *J* = 12.6 Hz, 2H), 3.73 – 3.38 (m, 2H), 2.56 – 2.13 (m, 3H), 2.12 – 0.95 (m, 25H), 0.96 – 0.80 (m, 6H), 0.61 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **24a** and **24b**) δ 175.9, 154.0, 130.6, 130.1, 124.7, 123.8, 118.2, 71.4, 62.0, 55.8, 54.8, 43.7, 43.5, 42.5, 40.1, 39.4, 37.2, 36.9, 35.2, 34.9, 34.0, 31.2, 30.9, 30.1, 28.6, 26.9, 23.4, 21.2, 18.4, 12.1.

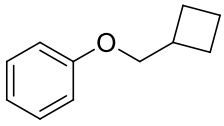
MS (DART) exact mass: calculated for $(M+H)^+$: 533.3028; found: 533.3032.



Anisole **(25)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and methanol (20 mmol, 809 μL) according to general procedure B with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 5% EtOAc/hexanes to yield a colorless oil. Yield was determined by UHPLC with dimethyl maleate as an internal standard (calibration curve).

25: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.40 – 7.30 (m, 2H), 7.04 – 6.90 (m, 3H), 3.85 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.6, 129.5, 120.7, 113.9, 55.2.

MS (DART) exact mass: calculated for $(M+H)^+$: 109.0648; found: 109.0657.

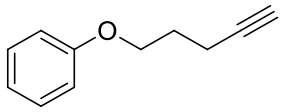


(cyclobutylmethoxy)benzene **(27)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and cyclobutylmethanol (12 mmol, 1.13 mL) according to general procedure B with an

irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 10% EtOAc/hexanes to furnish a colorless oil in 43% yield (27.9 mg).

27: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 – 7.28 (m, 2H), 7.06 – 6.77 (m, 3H), 3.96 (dd, *J* = 6.7, 1.9 Hz, 2H), 2.92 – 2.71 (m, 1H), 2.29 – 2.10 (m, 2H), 2.07 – 1.85 (m, 4H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 159.3, 129.4, 120.5, 114.6, 72.0, 34.7, 24.9, 18.6

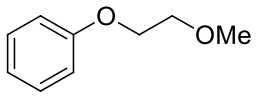
MS (DART) exact mass: calculated for $(M+H)^+$: 163.1117; found: 163.1124.



***(pent-4-yn-1-yloxy)benzene* (28)**: The title compound was prepared from benzene (0.4 mmol, 35.6 µL) and 4-pentyn-1-ol (8 mmol, 744 µL) according to general procedure B with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 20% EtOAc/hexanes to furnish a colorless oil in 31% yield (19.9 mg).

28: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.32 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.05 – 6.88 (m, 3H), 4.10 (t, *J* = 6.1 Hz, 2H), 2.45 (td, *J* = 7.0, 2.7 Hz, 2H), 2.09 – 1.91 (m, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.9, 129.5, 120.7, 114.5, 83.5, 68.9, 66.0, 28.2, 15.2.

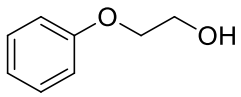
MS (DART) exact mass: calculated for $(M+H)^+$: 161.0961; found: 161.0970.



***(2-methoxyethoxy)benzene* (29)**: The title compound was prepared from benzene (0.4 mmol, 35.6 µL) and 2-methoxyethanol (20 mmol, 1.58 mL) according to general procedure B with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a colorless oil in 53% yield (32.3 mg).

29: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 (dd, *J* = 8.6, 7.3 Hz, 2H), 7.01 – 6.93 (m, 3H), 4.18 – 4.12 (m, 2H), 3.81 – 3.76 (m, 2H), 3.48 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.8, 129.4, 120.9, 114.6, 71.1, 67.1, 59.2.

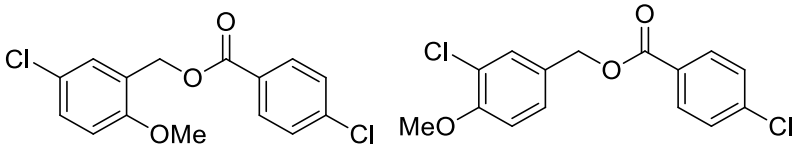
MS (DART) exact mass: calculated for $(M+H)^+$: 153.0910; found: 153.0919.



***2-phenoxyethanol* (30)**: The title compound was prepared from benzene (0.4 mmol, 35.6 µL) and ethylene glycol (20 mmol, 1.12 mL) according to general procedure B with an irradiation/electrolysis time of 36 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a colorless oil in 51% yield (28.2 mg).

30: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.32 (t, *J* = 7.9 Hz, 2H), 7.07 – 6.87 (m, 3H), 4.11 (t, *J* = 4.5 Hz, 2H), 3.98 (t, *J* = 4.5 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 158.6, 129.6, 121.2, 114.6, 69.1, 61.5.

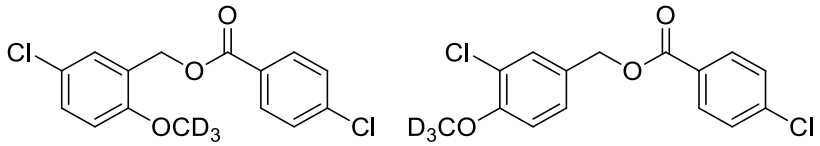
MS (DART) exact mass: calculated for $(M+H)^+$: 139.0754; found: 139.0758.



3-chloro-4-methoxybenzyl 4-chlorobenzoate and 5-chloro-2-methoxybenzyl 4-chlorobenzoate **(31a and 31b)**: The title compounds were prepared from 3-chlorobenzyl 4-chlorobenzoate (0.4 mmol, 112.4 mg) and methanol (20 mmol, 809 μL) according to general procedure B with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnnish a white solid in 82% yield (102.1 mg).

31: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **31a** and **31b**) δ 8.02 (m, 2H), 7.51 – 7.30 (m, 4H), 6.95 (d, *J* = 8.4 Hz, 0.6H), 6.86 (d, *J* = 8.4 Hz, 0.4H), 5.33 (s, 2H), 3.90 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **31a** and **31b**) δ 165.5, 155.1, 139.6, 131.1, 131.1, 130.6, 129.1, 129.0, 128.9, 128.8, 128.8, 128.5, 128.3, 122.6, 112.0, 111.7, 66.0, 61.7, 56.2, 55.8.

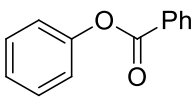
MS (DART) exact mass: calculated for $(M+H)^+$: 311.0236; found: 311.0239.



3-chloro-4-d3-methoxybenzyl 4-chlorobenzoate and 5-chloro-2-d3-methoxybenzyl 4-chlorobenzoate **(32a and 32b)**: The title compounds were prepared from 3-chlorobenzyl 4-chlorobenzoate (0.4 mmol, 112.4 mg) and methanol-*d4* (20 mmol, 813 μL) according to general procedure B with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 30% EtOAc/hexanes to furnish a colorless oil in 76% yield (95.5 mg).

32: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **32a** and **32b**) δ 8.09 – 7.92 (m, 2H), 7.51 – 7.29 (m, 4H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.85 (d, *J* = 8.7 Hz, 1H), 5.33 (s, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **32a** and **32b**) δ 165.5, 155.1, 139.6, 131.1, 131.1, 130.6, 129.1, 129.0, 128.9, 128.8, 128.8, 128.6, 128.5, 128.3, 126.0, 122.6, 111.9, 111.7, 66.0, 61.7, 55.6 (septet, *J* = 21.4 Hz), 54.8 (septet, *J* = 21.4 Hz).

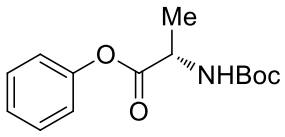
MS (DART) exact mass: calculated for $(M+H)^+$: 314.0425; found: 314.0430.



***phenyl benzoate* (33)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and benzoic acid (6 mmol, 733 mg) according to general procedure C with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 52% yield (41.2 mg).

33: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.25 (dd, *J* = 8.3, 1.4 Hz, 2H), 7.71 – 7.63 (m, 1H), 7.59 – 7.52 (m, 2H), 7.47 (dd, *J* = 8.4, 7.4 Hz, 2H), 7.34 – 7.29 (m, 1H), 7.27 – 7.21 (m, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 165.2, 151.0, 133.6, 130.2, 129.6, 129.5, 128.6, 125.9, 121.8.

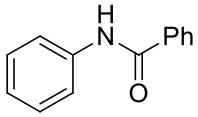
MS (DART) exact mass: calculated for $(M+H)^+$: 199.0754; found: 199.0762.



***(S)-phenyl 2-((tert-butoxycarbonyl)amino)propanoate* (34)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and Boc-Ala-OH (4 mmol, 757 mg) according to general procedure C with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 70% EtOAc/hexanes to furnish a colorless oil in 30% yield (31.8 mg).

34: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.45 – 7.36 (m, 2H), 7.28 – 7.23 (m, 1H), 7.12 (dd, *J* = 7.5, 1.6 Hz, 2H), 5.14 (br, 1H), 4.58 (t, *J* = 7.6 Hz, 1H), 1.58 (d, *J* = 7.3 Hz, 3H), 1.49 (s, 9H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 172.1, 155.2, 150.5, 129.5, 126.1, 121.3, 80.1, 49.5, 28.3, 18.5.

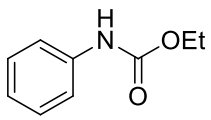
MS (DART) exact mass: calculated for $(M+H)^+$: 266.1387; found: 266.1395.



***N*-phenylbenzamide (35)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and benzamide (4 mmol, 484 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 70% EtOAc/hexanes to furnish a white solid in 55% yield (43.4 mg).

35: ^{1}H NMR (500 MHz, DMSO-d_6) δ 10.26 (br, 1H), 8.02 – 7.93 (m, 2H), 7.85 – 7.74 (m, 2H), 7.65 – 7.57 (m, 1H), 7.57 – 7.48 (m, 2H), 7.36 (dd, *J* = 8.5, 7.3 Hz, 2H), 7.16 – 7.05 (m, 1H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 166.0, 139.7, 135.5, 132.0, 129.1, 128.8, 128.1, 124.1, 120.8.

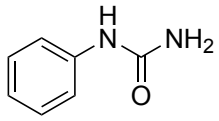
MS (DART) exact mass: calculated for $(M+H)^+$: 198.0913; found: 198.0918.



***ethyl phenylcarbamate* (36)**: The title compound was prepared from benzene (0.4 mmol, 35.6 μL) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 70% yield (46.3 mg).

36: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 7.9 Hz, 2H), 7.32 (t, *J* = 7.9 Hz, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.84 (br, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*) δ 153.8, 138.1, 129.0, 123.3, 61.2, 14.6.

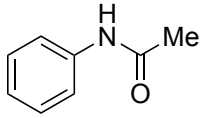
MS (DART) exact mass: calculated for $(M+H)^+$: 166.0863; found: 166.0871.



***1-phenylurea* (37)**: The title compounds were prepared from benzene (0.4 mmol, 35.6 μL) and urea (2 mmol, 120.2 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 90% EtOAc/hexanes to furnish a white solid in 50% yield (27.2 mg).

37: ^{1}H NMR (500 MHz, DMSO) δ 8.53 (br, 1H), 7.45 – 7.36 (m, 2H), 7.22 (dd, *J* = 8.5, 7.3 Hz, 2H), 6.94 – 6.85 (m, 1H), 5.87 (br, 2H). ^{13}C NMR (126 MHz, DMSO) δ 156.6, 141.0, 129.1, 121.6, 118.3.

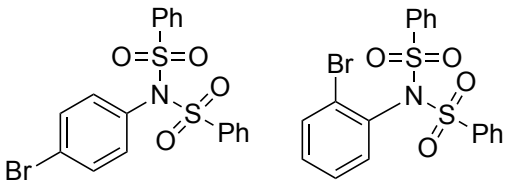
MS (DART) exact mass: calculated for $(M+H)^+$: 137.0709; found: 137.0718.



***N-phenylacetamide* (38)**: The title compounds were prepared from benzene (0.4 mmol, 35.6 μL) and acetamide (2 mmol, 118.2 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 26% yield (14.1 mg).

38: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (br, 1H), 7.60 – 7.49 (m, 2H), 7.38 – 7.25 (m, 2H), 7.16 – 7.05 (m, 1H), 2.16 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.1, 138.1, 128.9, 124.3, 120.2, 24.4.

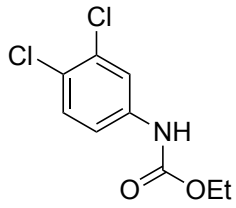
MS (DART) exact mass: calculated for $(M+H)^+$: 136.0757; found: 136.0766.



***N-(4-bromophenyl)-N-(phenylsulfonyl)benzenesulfonamide with N-(2-bromophenyl)-N-(phenylsulfonyl)benzenesulfonamide* (40a and 40b)**: The title compounds were prepared from bromobenzene (0.4 mmol, 41.9 μL) and dibenzenesulfonimide (1.2 mmol, 356.9 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 60% EtOAc/hexanes to furnish a light yellow in 55% yield (99.5 mg).

40: ^{1}H NMR (500 MHz, $CDCl_3$, mixture of **40a** and **40b**) δ 8.00 – 7.92 (m, 4H), 7.77 – 7.67 (m, 2H), 7.63 – 7.56 (m, 4H), 7.55 – 7.48 (m, 1.9H), 7.37 – 7.35 (m, 0.3H), 6.98 – 6.97 (m, 0.2H), 6.93 – 6.87 (m, 1.7H). ^{13}C NMR (126 MHz, $CDCl_3$, mixture of **40a** and **40b**) δ 139.2, 134.2, 133.2, 133.1, 132.8, 132.6, 129.6, 129.1, 128.6, 128.6 125.0.

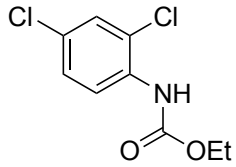
MS (DART) exact mass: calculated for $(M+H)^+$: 451.9620; found: 451.9627.



***ethyl (3,4-dichlorophenyl)carbamate* (41)**: The title compounds were prepared from 1,2-dichlorobenzene (0.4 mmol, 45.2 μL) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 24 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 80% yield (74.9 mg).

41: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 – 7.56 (m, 1H), 7.35 (d, J = 8.7 Hz, 1H), 7.22 (dd, J = 8.8, 2.5 Hz, 1H), 6.82 (s, 1H), 4.25 (q, J = 7.1 Hz, 2H), 1.32 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.3, 137.6, 132.8, 130.5, 126.5, 120.3, 117.9, 61.7, 14.5.

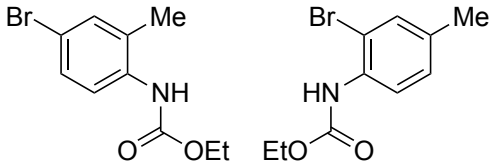
MS (DART) exact mass: calculated for $(M+H)^+$: 234.0083; found: 234.0090.



***ethyl (2,4-dichlorophenyl)carbamate* (42)**: The title compounds were prepared from 1,3-dichlorobenzene (0.4 mmol, 45.6 μL) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 30 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 85% yield (79.6 mg).

42: ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 (d, J = 9.0 Hz, 1H), 7.36 (d, J = 2.3 Hz, 1H), 7.25 (dd, J = 8.9, 2.4 Hz, 1H), 7.09 (s, 1H), 4.26 (q, J = 7.1 Hz, 2H), 1.35 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 153.1, 133.6, 128.7, 128.0, 127.9, 122.4, 120.6, 61.8, 14.5.

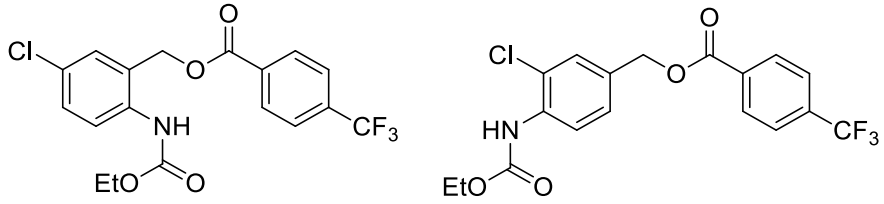
MS (DART) exact mass: calculated for $(M+H)^+$: 234.0083; found: 234.0091.



ethyl (4-bromo-2-methylphenyl)carbamate with ethyl (2-bromo-4-methylphenyl)carbamate **(43a and 43b)**: The title compounds were prepared from 1-bromo-3-methylbenzene (0.4 mmol, 68.4 mg) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 38 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a light yellow oil in 49% yield (50.6 mg).

43: ^{1}H NMR (500 MHz, $CDCl_3$, mixture of **43a** and **43b**) δ 7.74 (br, 0.8H), 7.47 – 7.45 (m, 0.1H), 7.39 – 7.30 (m, 1.7H), 7.21 – 7.20 (m, 0.2H), 7.08 – 7.05 (m, 0.1H), 6.34 (s, 0.9H), 4.25 (q, J = 7.1 Hz, 2H), 2.25 (s, 3H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, mixture of **43a** and **43b**) δ 153.7, 135.1, 133.0, 130.3, 129.8, 129.1, 122.3, 116.6, 61.5, 19.1, 17.5, 14.5.

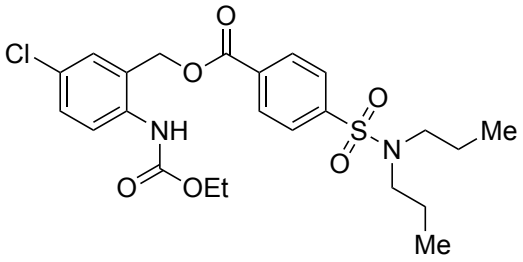
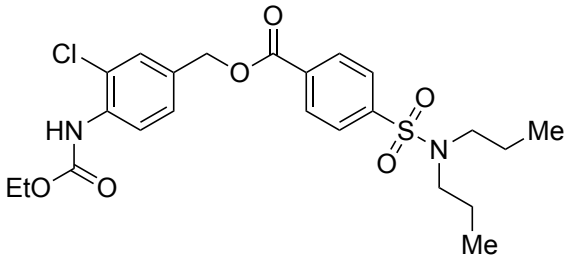
MS (DART) exact mass: calculated for $(M+H)^+$: 258.0124; found: 258.0133.



3-chloro-4-((ethoxycarbonyl)amino)benzyl 4-(trifluoromethyl)benzoate compound with 5-chloro-2-((ethoxycarbonyl)amino)benzyl 4-(trifluoromethyl)benzoate **(44a and 44b)**: The title compounds were prepared from 3-chlorobenzyl 4-(trifluoromethyl)benzoate (0.4 mmol, 125.9 mg) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 60% EtOAc/hexanes to furnish a colorless oil in 43% yield (69.1 mg).

44: ^{1}H NMR (500 MHz, Chloroform-*d*, mixture of **44a** and **44b**) δ 8.30 – 8.11 (m, 2.5H), 7.89 – 7.75 (m, 1H), 7.72 (dd, J = 8.2, 5.3 Hz, 2H), 7.49 – 7.32 (m, 2H), 7.18 (br, 0.5H), 5.36 (s, 1.1H), 5.32 (s, 0.9H), 4.29 – 4.25 (m, 2H), 1.37 – 1.33 (m, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*, mixture of **44a** and **44b**) δ 165.7, 165.1, 154.1, 153.1, 135.6, 135.1, 132.5, 131.0, 130.9, 130.2, 130.1, 130.0, 129.2, 128.1, 125.6, 125.6 (q, J = 3.8 Hz), 125.5 (q, J = 3.8 Hz), 123.2 (q, J = 270.9 Hz), 122.0, 119.8, 66.2, 63.8, 61.7, 61.6, 14.6, 14.4. ^{19}F NMR (470 MHz, Chloroform-*d*, mixture of **44a** and **44b**) δ -63.14, -63.21.

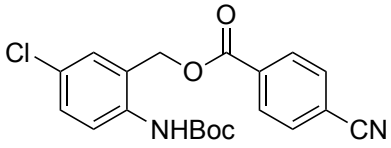
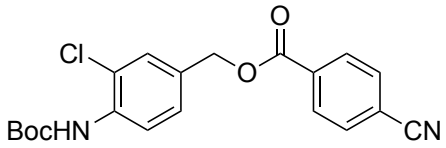
MS (DART) exact mass: calculated for $(M+H)^+$: 402.0714; found: 402.0720.

5-chloro-2-((ethoxycarbonyl)amino)benzyl 4-(N,N-dipropylsulfamoyl)benzoate with 3-chloro-4-((ethoxycarbonyl)amino)benzyl 4-(N,N-dipropylsulfamoyl)benzoate **(45a and 45b)**: The title compounds were prepared from 3-chlorobenzyl 4-(N,N-dipropylsulfamoyl)benzoate (0.4 mmol, 164.0 mg) and ethyl carbamate (6 mmol, 534.6 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 70% EtOAc/hexanes to furnish a colorless oil in 71% yield (141.1 mg).

45: ^{1}H NMR (500 MHz, $CDCl_3$, mixture of **45a** and **45b**) δ 8.23 – 8.17 (m, 2H), 7.99 – 7.78 (m, 3H), 7.49 – 7.44 (m, 1H), 7.38 – 7.34 (m, 2H), 5.37 (m, 2H), 4.81 – 4.16 (m, 2H), 3.17 – 2.89 (m, 4H), 1.62 – 1.46 (m, 4H), 1.35 (m, 3H), 0.88 (m, 6H). ^{13}C NMR (126 MHz, $CDCl_3$, mixture of **45a** and **45b**) δ 164.9, 144.4, 137.4, 135.6, 134.6, 133.4, 130.9, 130.5, 130.5, 130.4, 130.1, 130.0, 129.3, 128.7, 128.3, 128.2, 127.1, 127.1, 127.0, 126.4, 119.9, 66.4, 66.3, 63.8, 61.7, 61.6, 49.9, 49.9, 45.0, 23.0, 21.9, 21.9, 14.6, 11.2, 11.2, 11.1.

MS (DART) exact mass: calculated for $(M+H)^+$: 497.1508; found: 497.1517.

2-((tert-butoxycarbonyl)amino)-5-chlorobenzyl 4-cyanobenzoate with 4-((tert-butoxycarbonyl)amino)-3-chlorobenzyl 4-cyanobenzoate **(46a and 46b)**: The title compounds were prepared from 3-chlorobenzyl 4-cyanobenzoate (0.4 mmol, 108.7 mg) and tert-butyl carbamate (2.0 mmol, 234.4 mg) according to general procedure D with an irradiation/electrolysis time of 48 h. The crude residue was purified by column chromatography on silica gel with an eluent of hexanes to 50% EtOAc/hexanes to furnish a colorless oil in 62% yield (95.9 mg).

46: ^{1}H NMR (500 MHz, $CDCl_3$, mixture of **46a** and **46b**) δ 8.23 – 8.13 (m, 2H), 7.85 – 7.73 (m, 2.9H), 7.48 – 7.32 (m, 2.9H), 7.11 – 7.10 (m, 0.1H), 6.58 (br, 0.1H), 5.36 (s, 1.78H), 5.32 (s, 0.22H), 1.55 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$, mixture of **46a** and **46b**) δ 165.1, 153.1, 139.9, 137.7, 135.8, 133.2, 132.4, 132.3, 130.7, 130.3, 130.3, 130.0, 129.1, 117.8, 117.0, 81.1, 66.6, 64.1, 28.3.

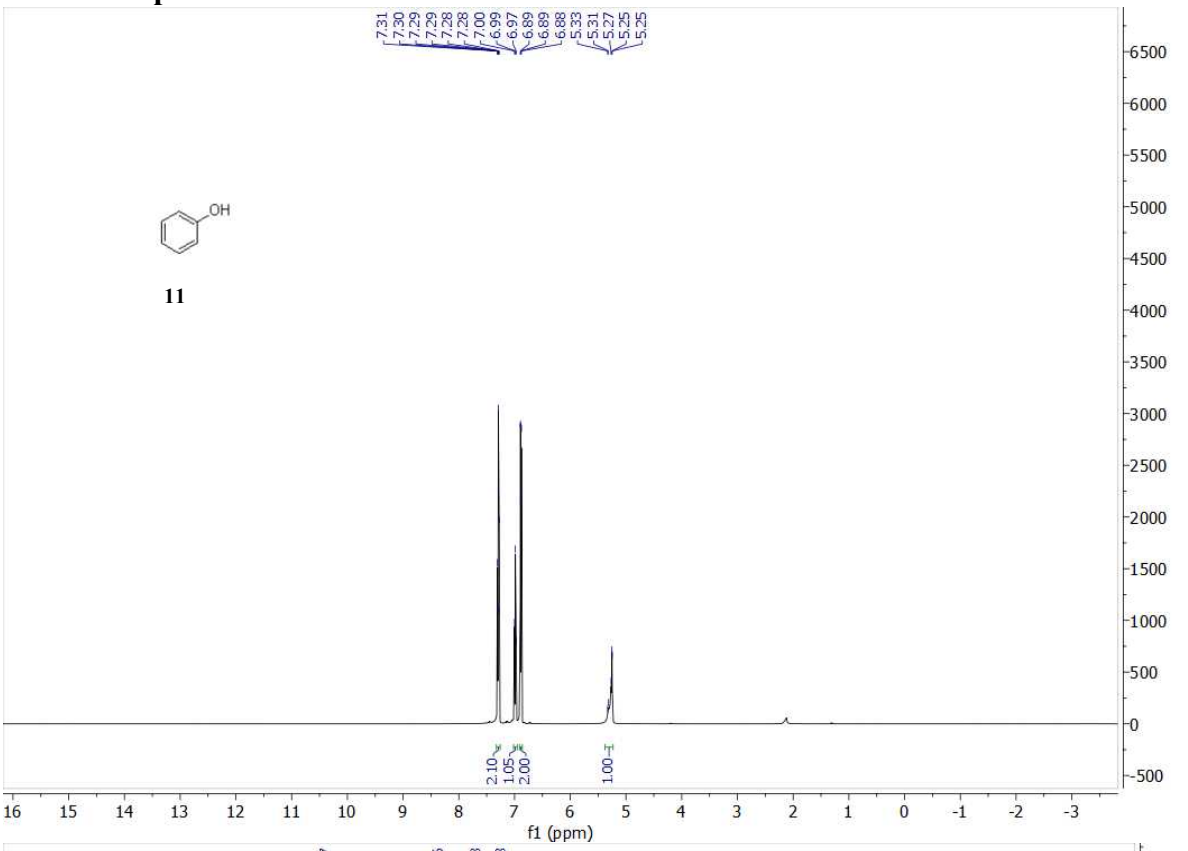
MS (DART) exact mass: calculated for $(M+H)^+$: 387.1106; found: 387.1116.

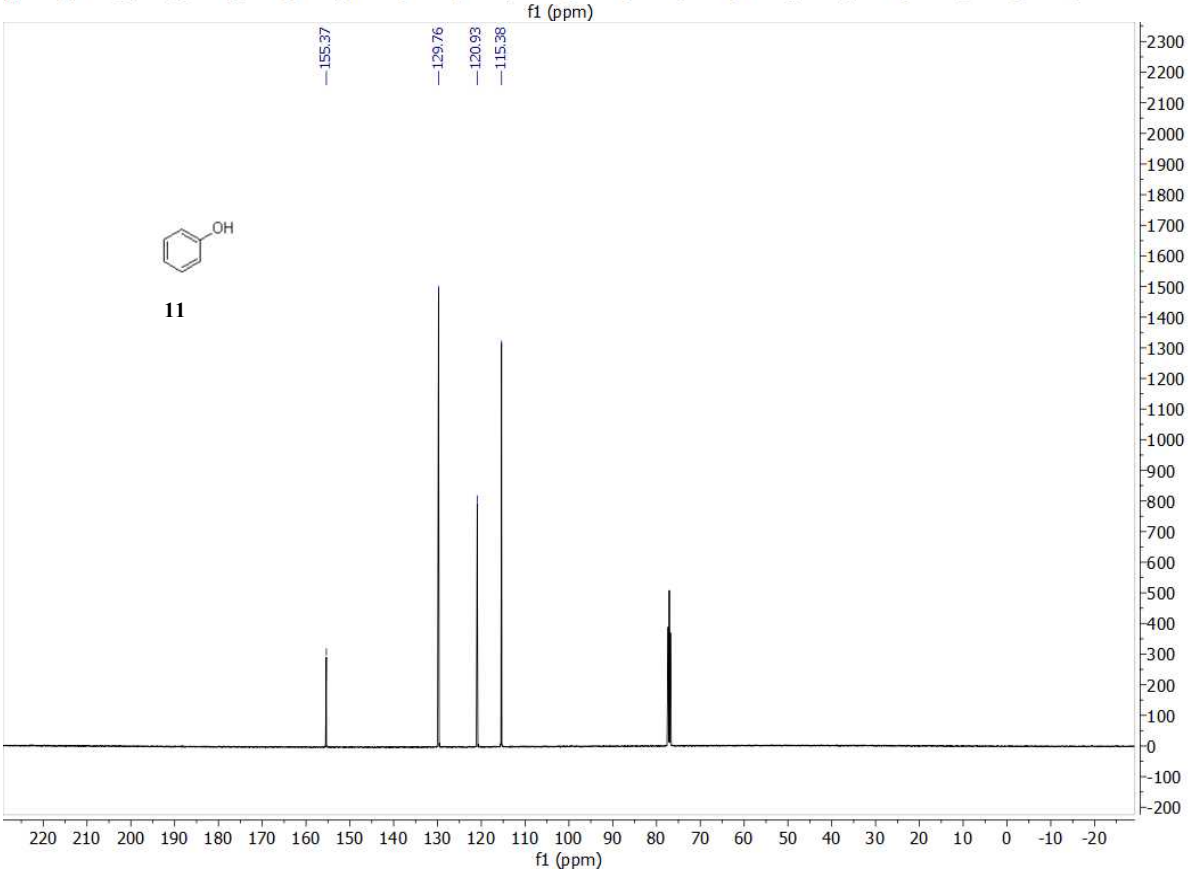
10. References

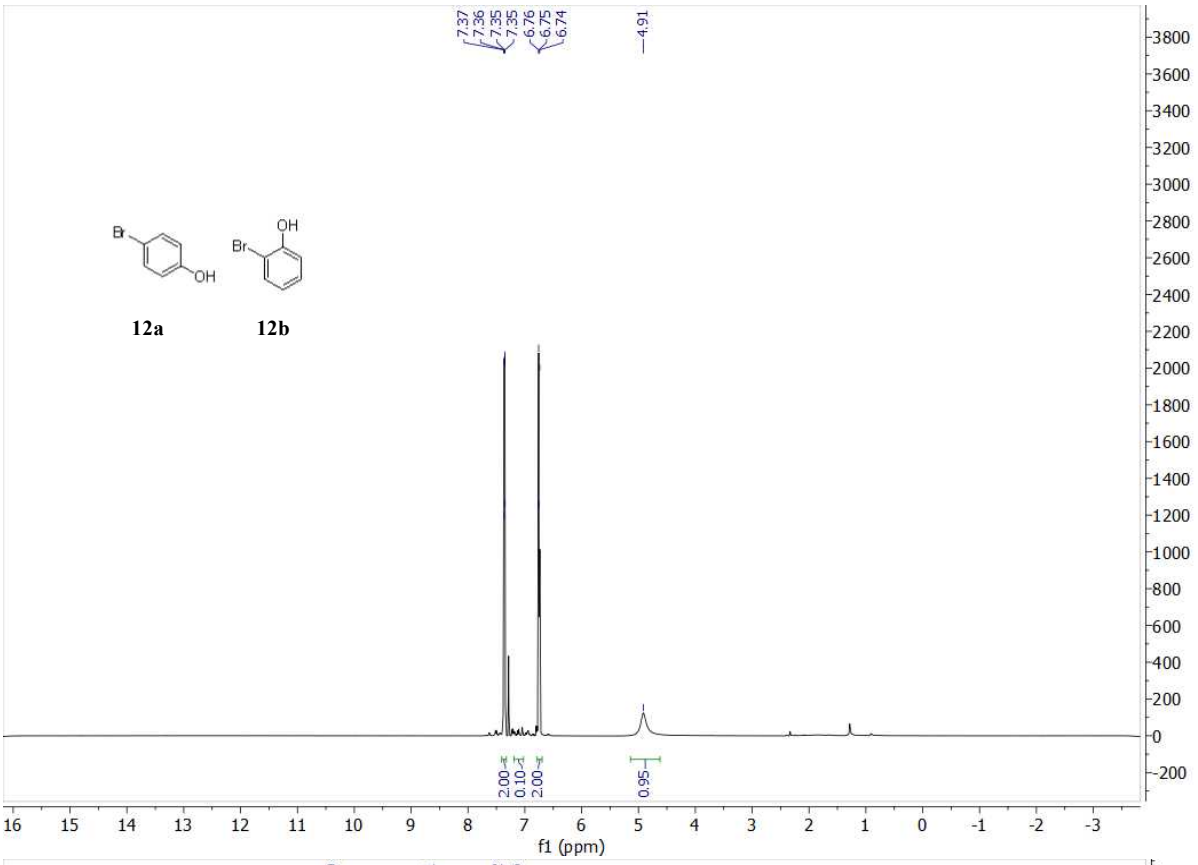
[1] Huang, H.; Strater, Z. M.; Rauch, M.; Shee, J.; Sisto, T. J.; Nuckolls, C.; Lambert, T. H., Electrophotocatalysis with a trisaminocyclopropenium radical dication. *Angew. Chem. Int. Ed.* **2019**, *58*, 13318-13322.

[2] Huang, H.; Lambert, T. H., Electrophotocatalytic SNAr reactions of unactivated aryl fluorides at ambient temperature and without base. *Angew. Chem. Int. Ed.* **2020,** *59*, 658-662.

11. NMR Spectral Data



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7.36
7.35
7.35
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6.75
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OH
Br
OH
12a
12b
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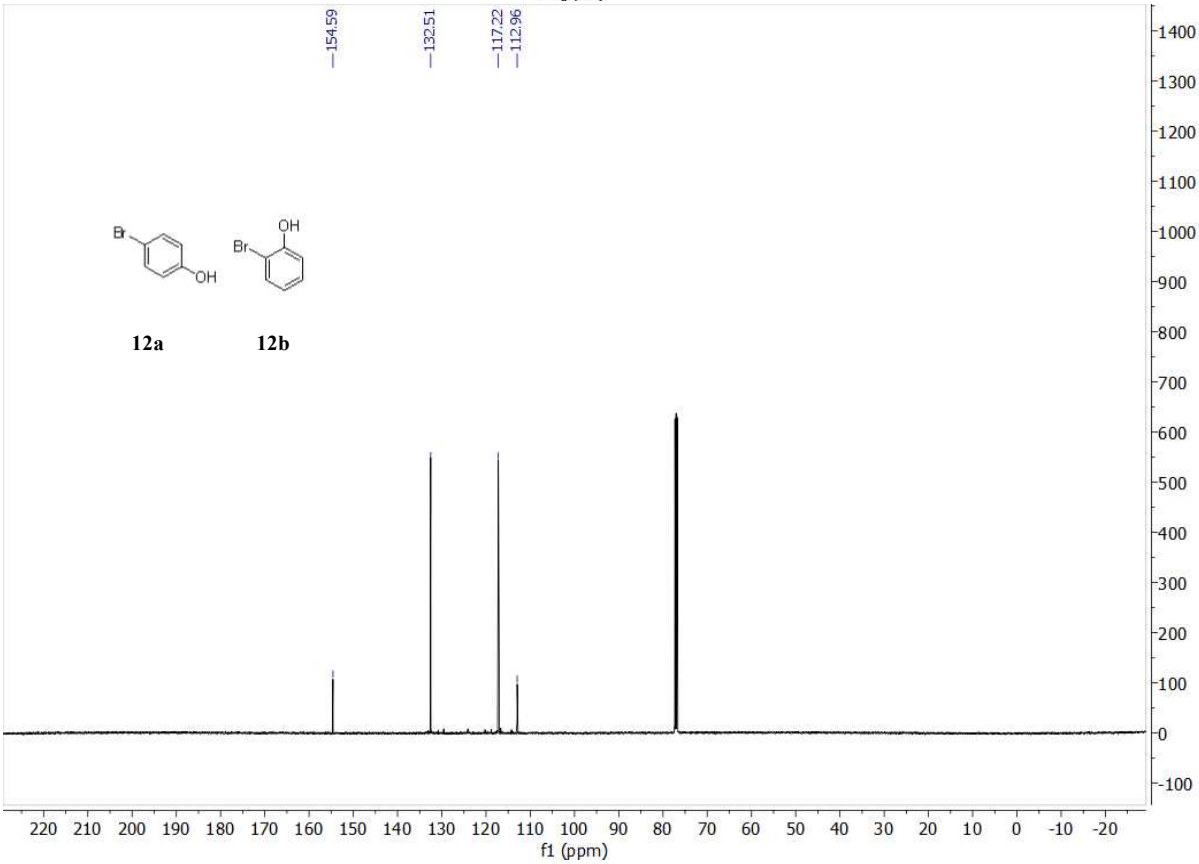
0.10
2.00
0.95
f1 (ppm)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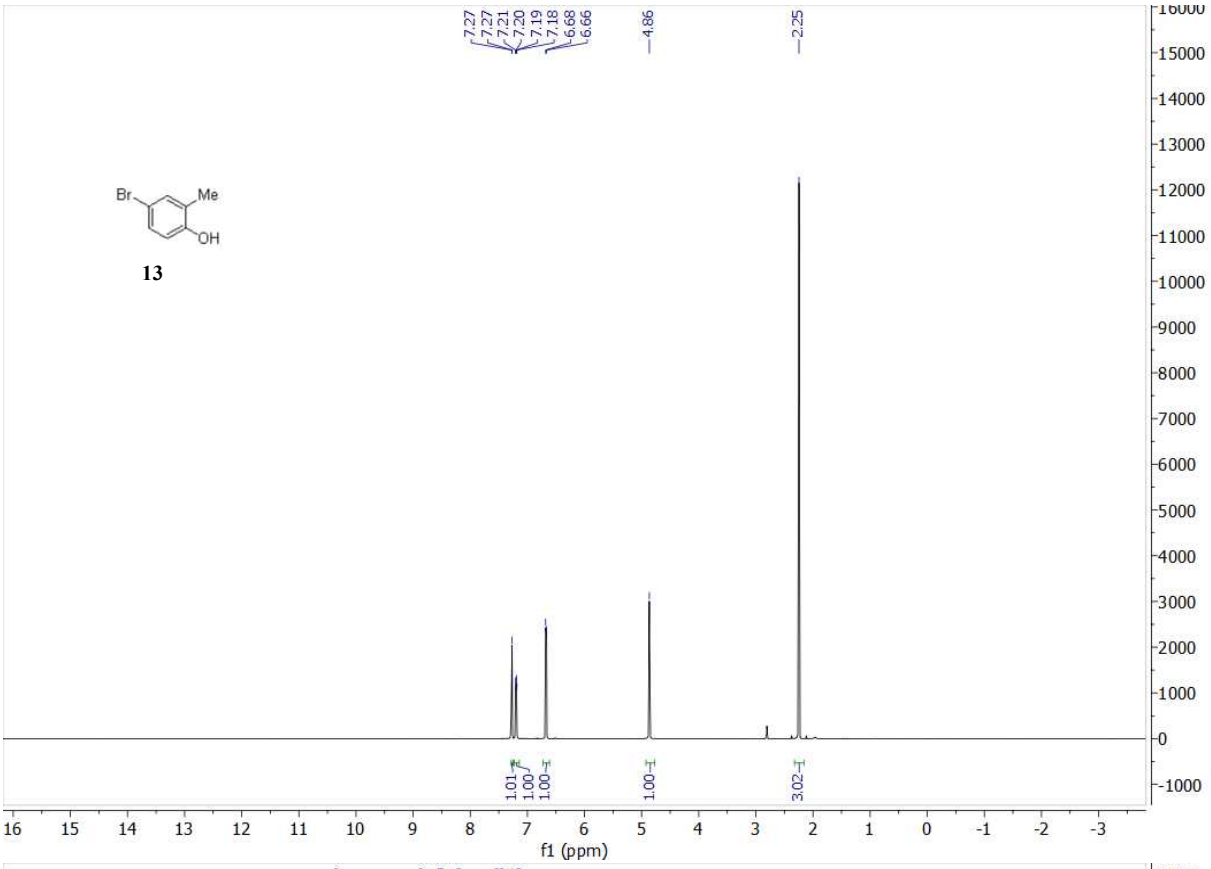
Br
OH
Br
OH
12a
12b
f1 (ppm)


7.27
7.27
7.21
7.20
7.19
7.18
6.68
6.66
4.86
2.25
Br
Me
OH
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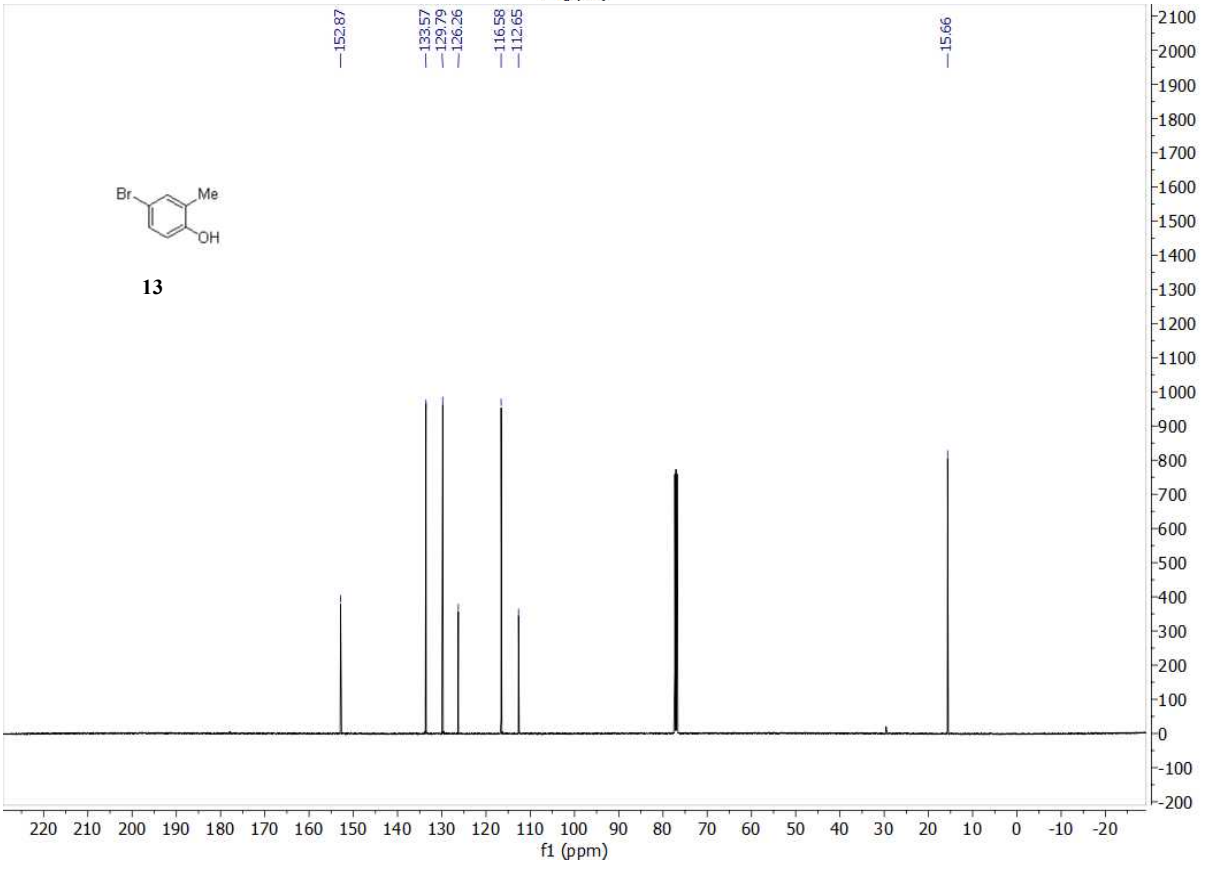
1.00
1.00
1.00
3.02
f1 (ppm)

13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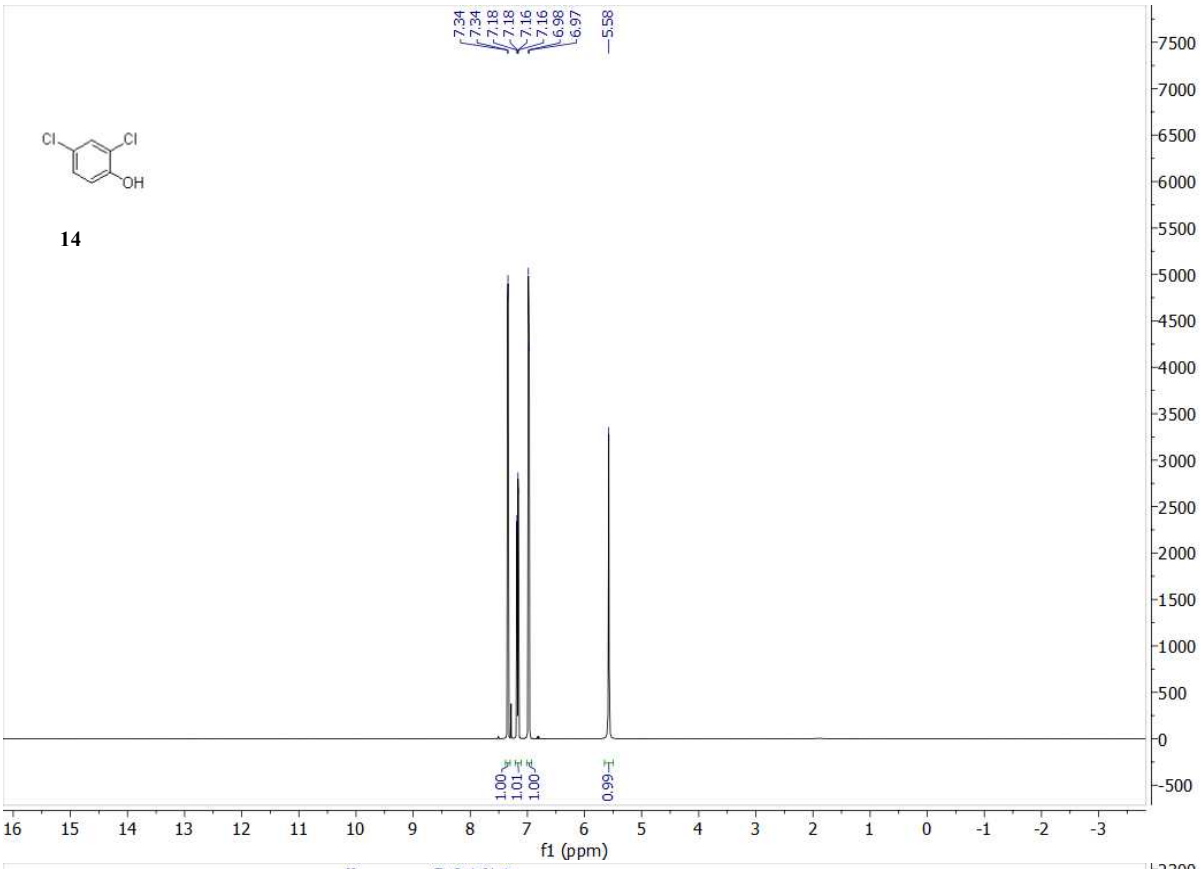
Br
Me
OH
f1 (ppm)

13


7.34
7.34
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7.18
7.16
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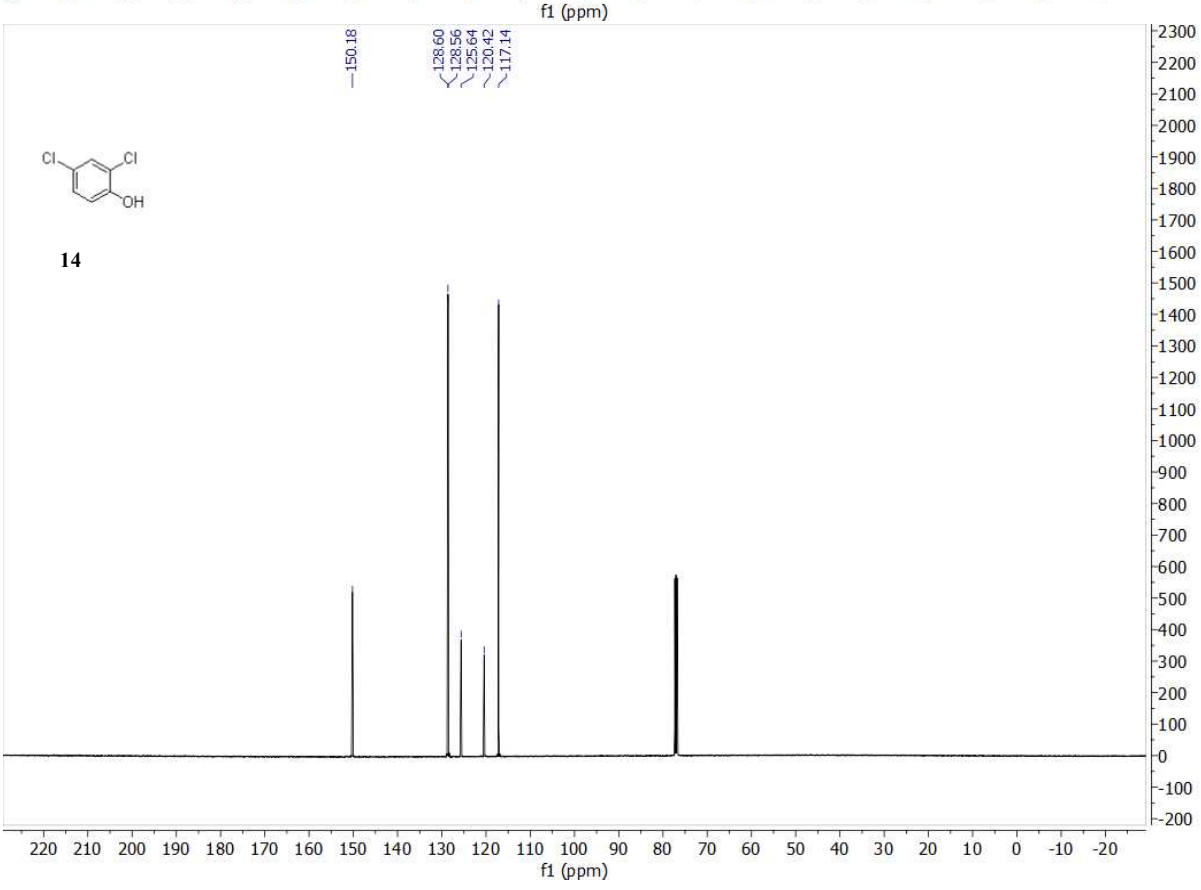
6.97
5.58
Cl
Cl
OH
1.00
1.01
1.00
0.99
f1 (ppm)

14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Cl
OH
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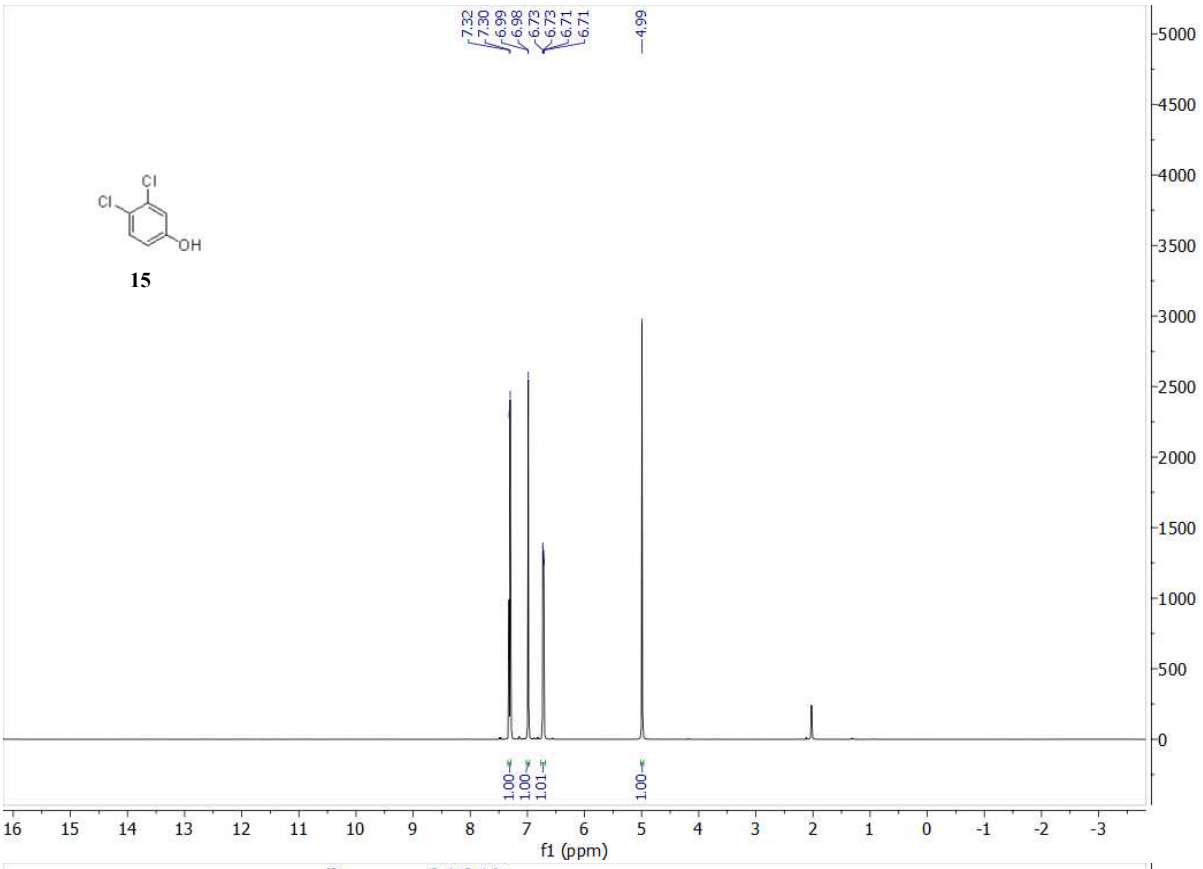
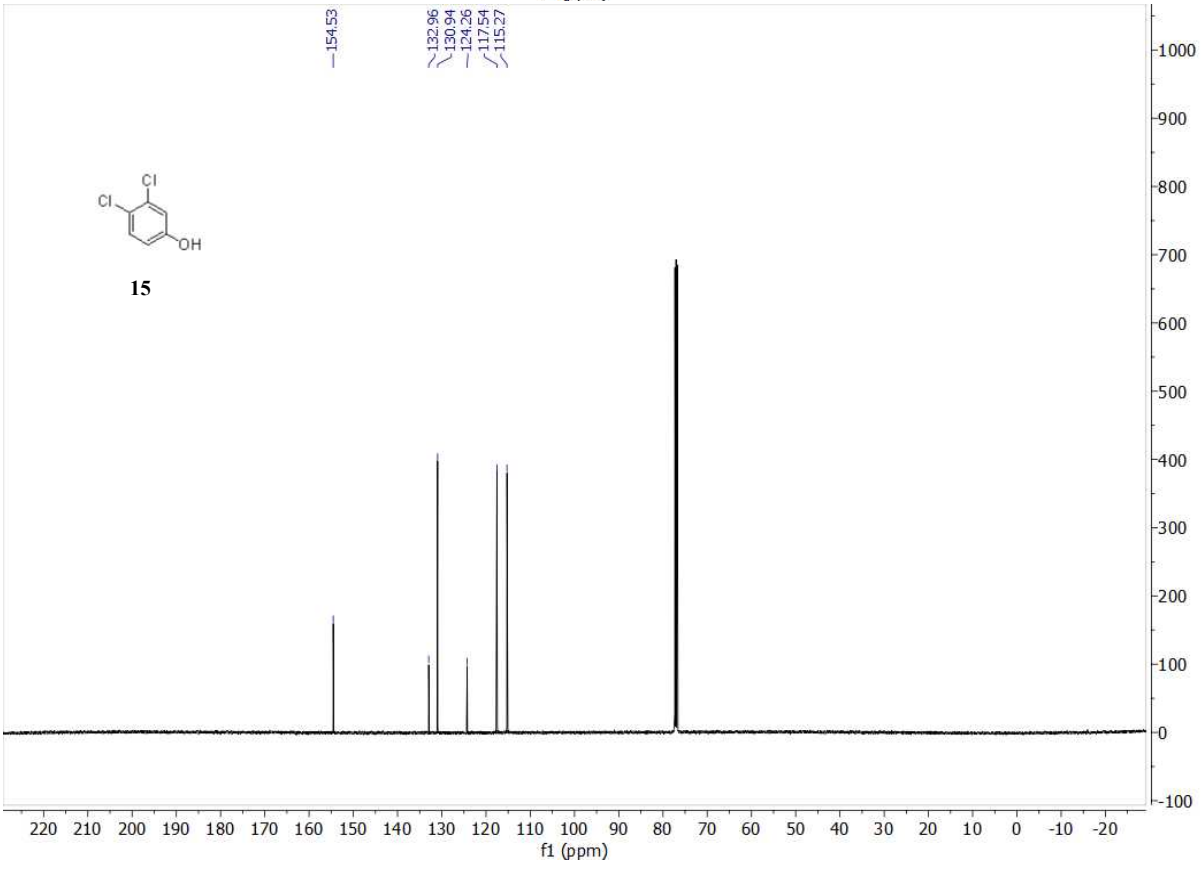
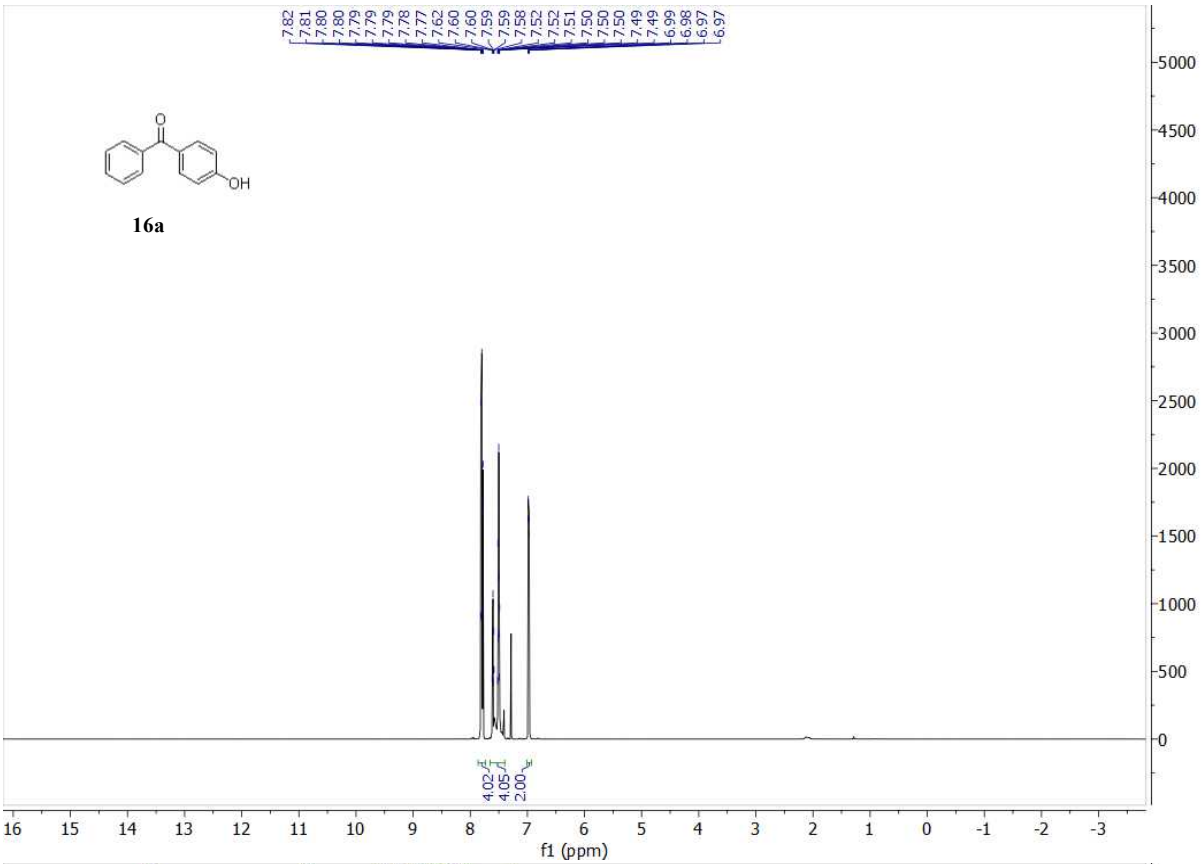
14


7.32
7.30
6.99
6.98
6.73
6.73
6.71
6.71
4.99
Cl
Cl
OH
1.00
1.00
1.01
1.00
f1 (ppm)

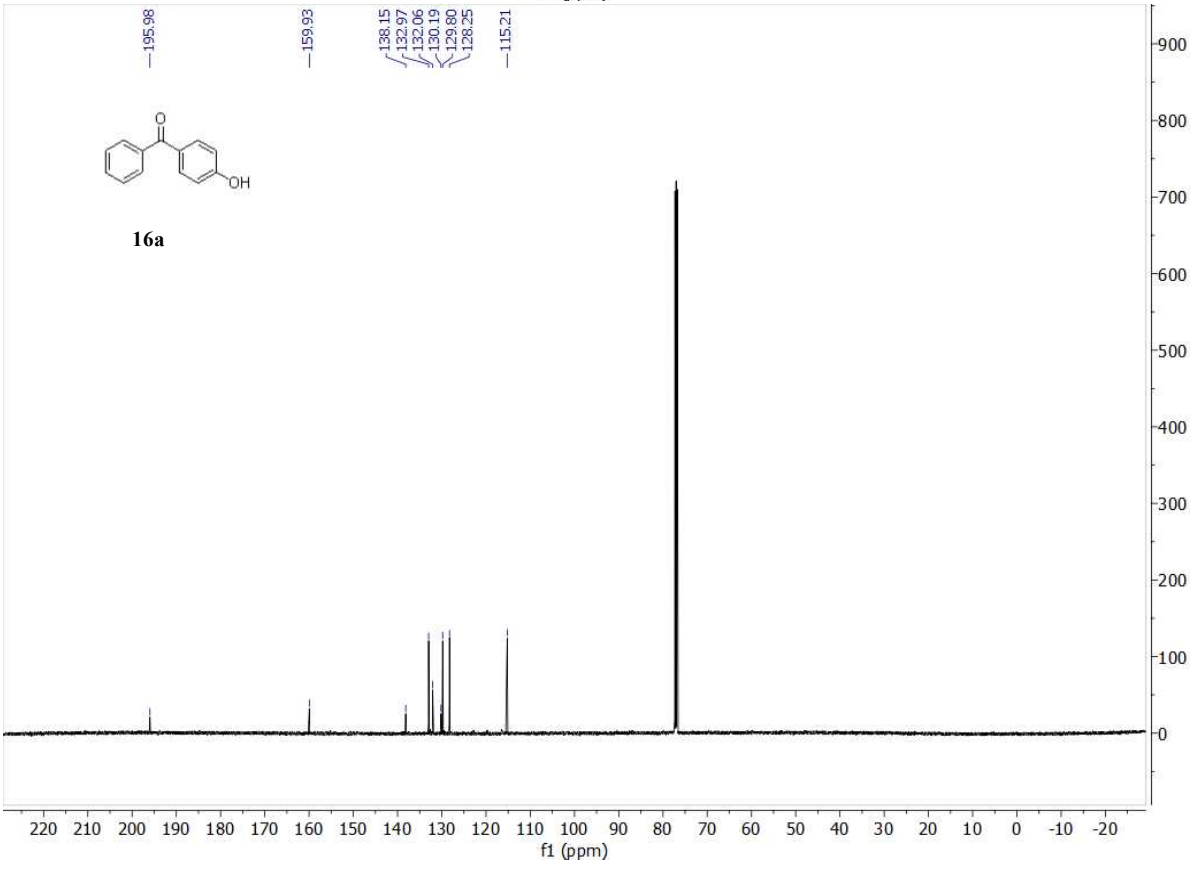
15


154.53
132.96
130.94
124.26
117.54
115.27
Cl
Cl
OH
f1 (ppm)

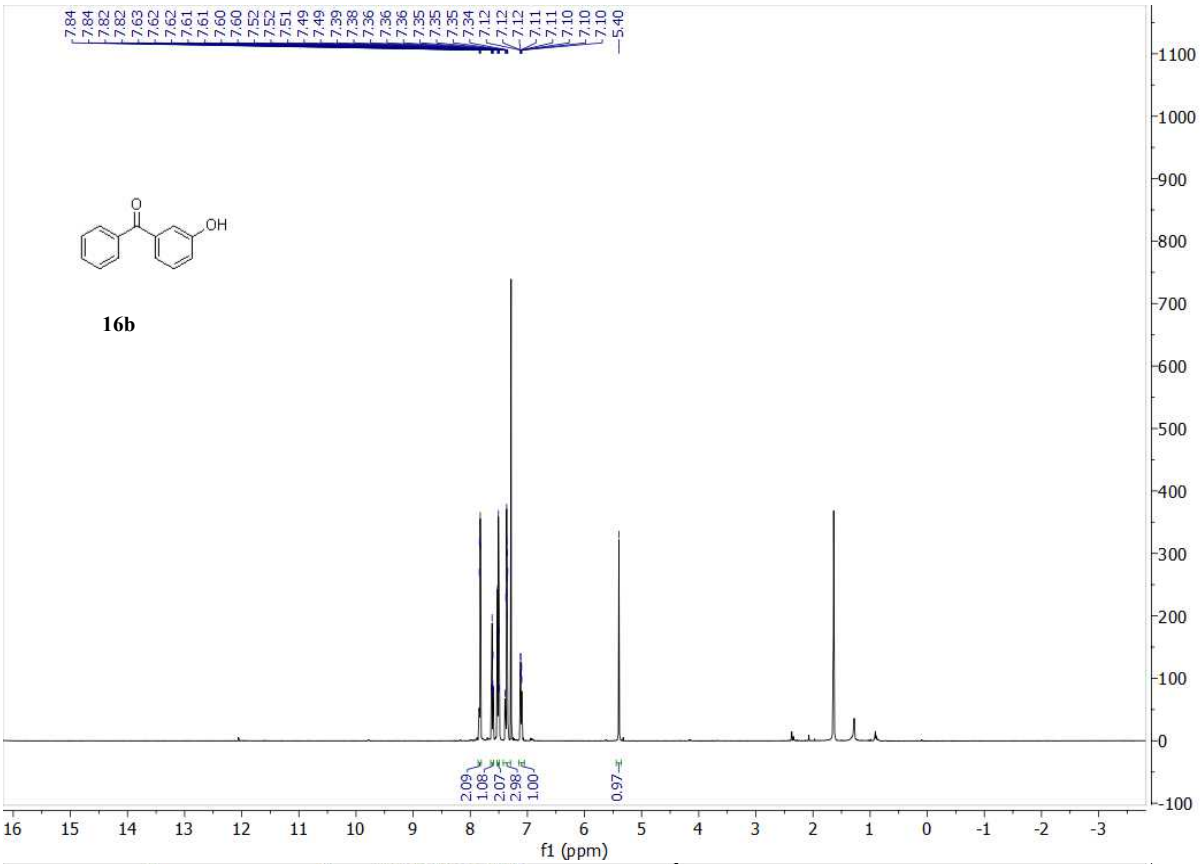
15

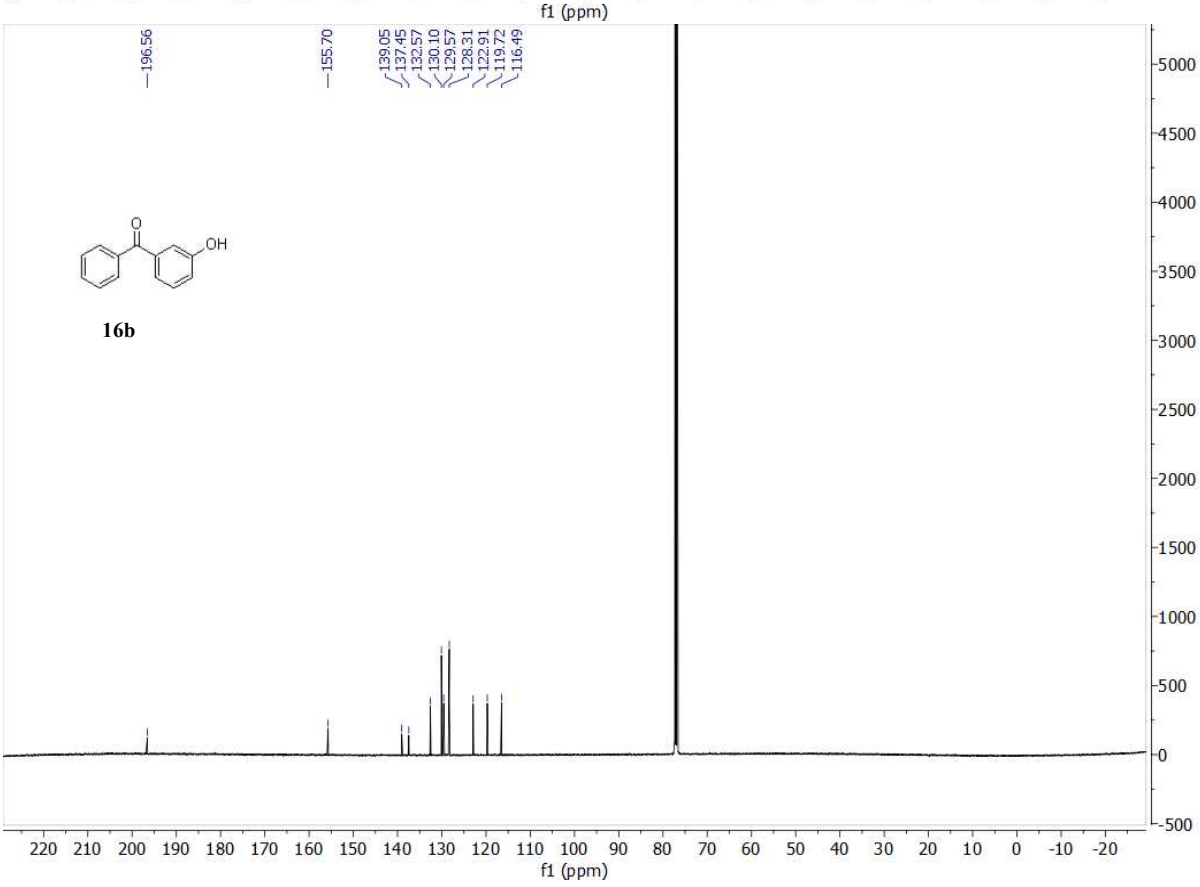

7.82
7.81
7.80
7.80
7.79
7.79
7.79
7.78
7.77
7.62
7.60
7.60
7.59
7.59
7.58
7.52
7.52
7.51
7.50
7.50
7.50
7.49
7.49
6.99
6.98
6.97
6.97
O
OH
4.02
4.05
2.00
f1 (ppm)

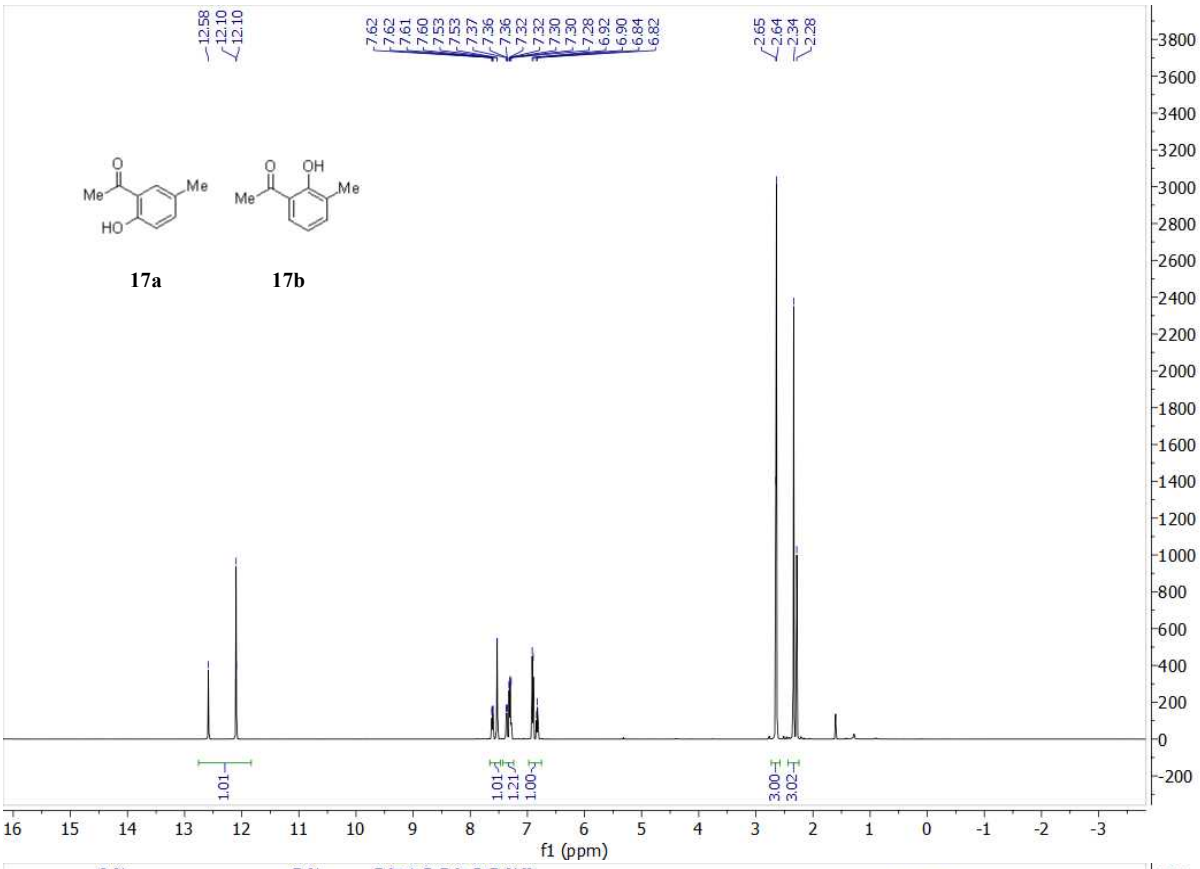
16a

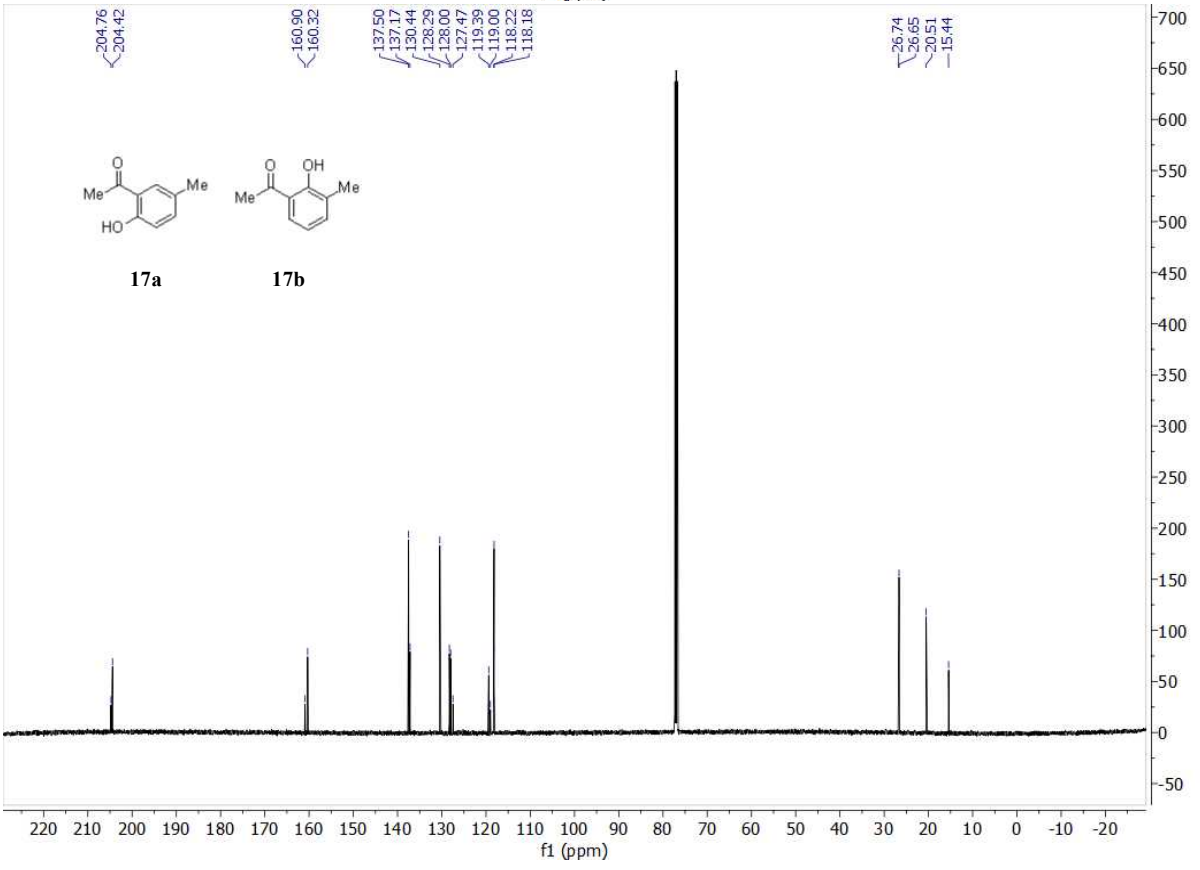

195.98
159.93
138.15
132.97
132.06
130.19
129.80
128.25
115.21
O
OH
f1 (ppm)

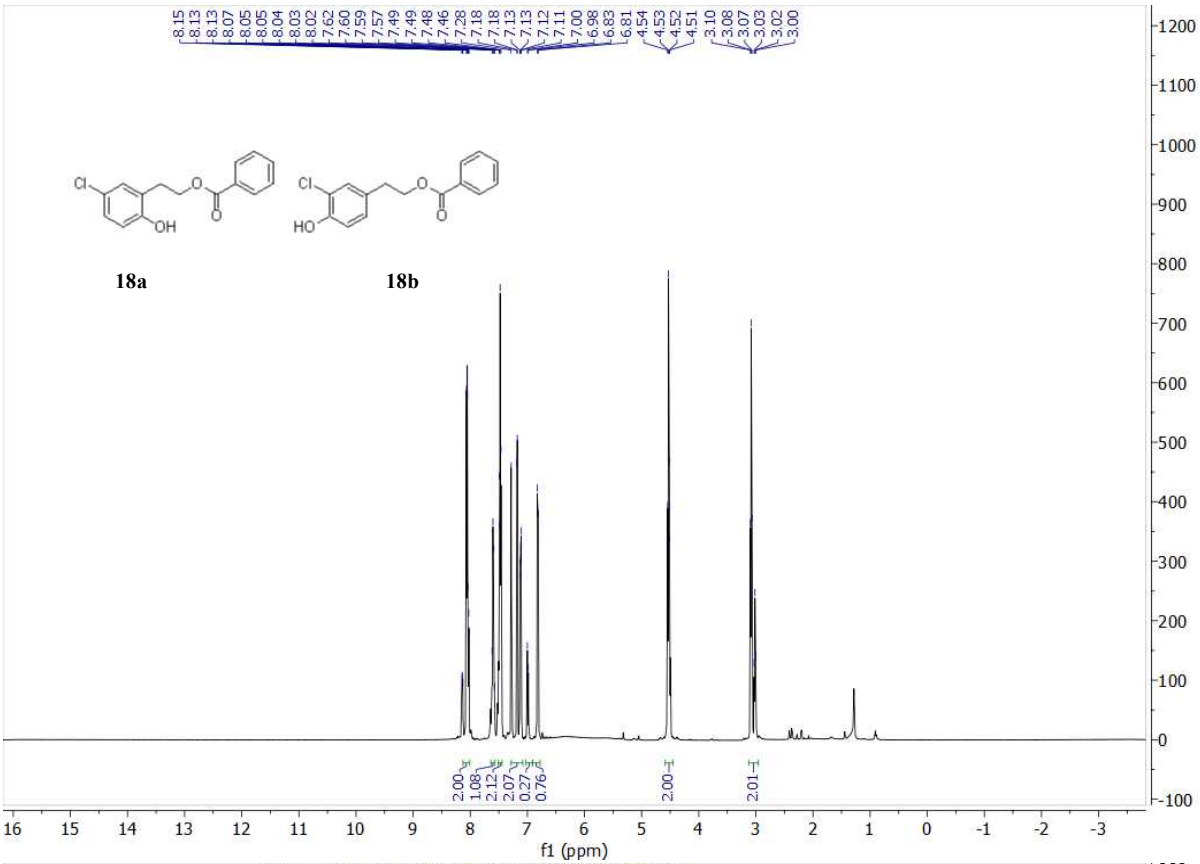
16a

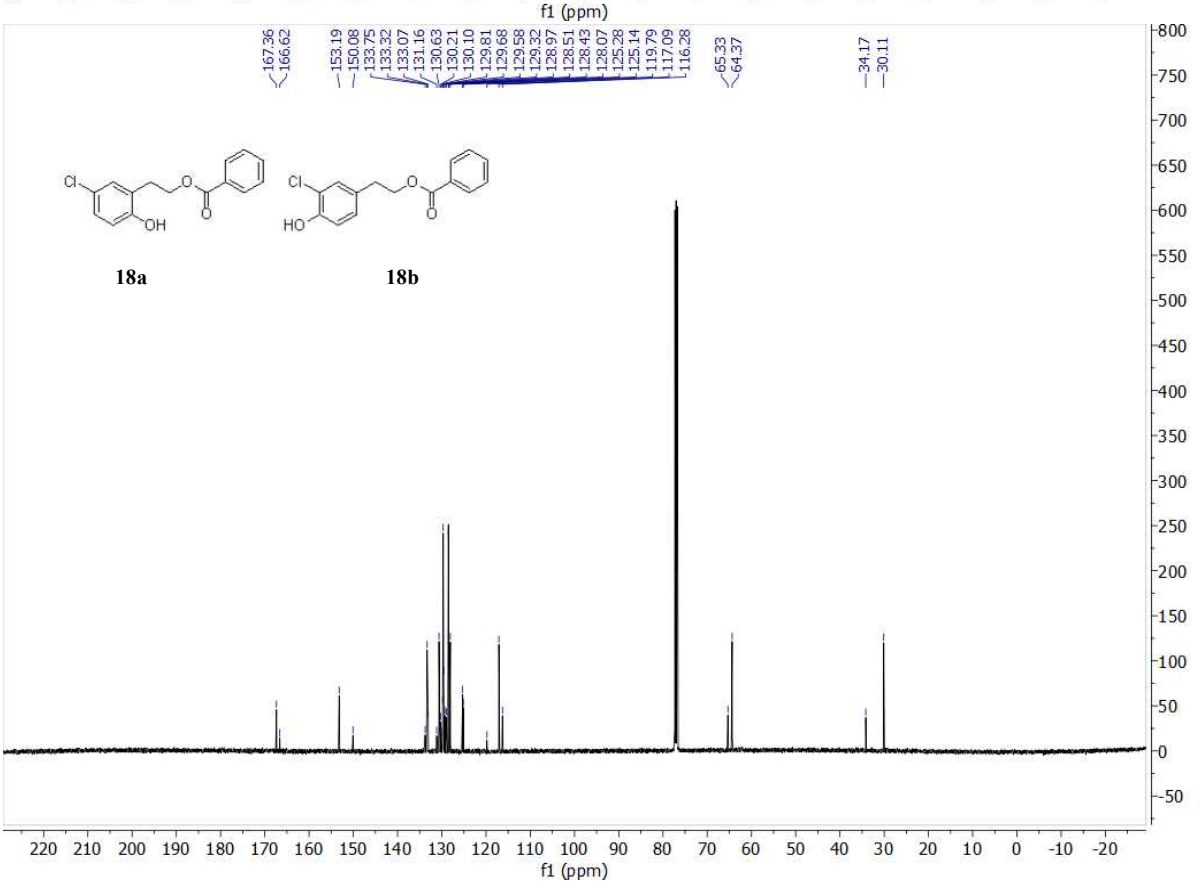

7.84
7.84
7.82
7.82
7.63
7.62
7.62
7.61
7.61
7.60
7.60
7.52
7.52
7.51
7.49
7.49
7.39
7.38
7.36
7.36
7.36
7.35
7.35
7.35
7.34
7.12
7.12
7.11
7.11
7.10
7.10
7.10
5.40
O
OH
16b
2.09
1.08
2.07
2.98
1.00
0.97
f1 (ppm)

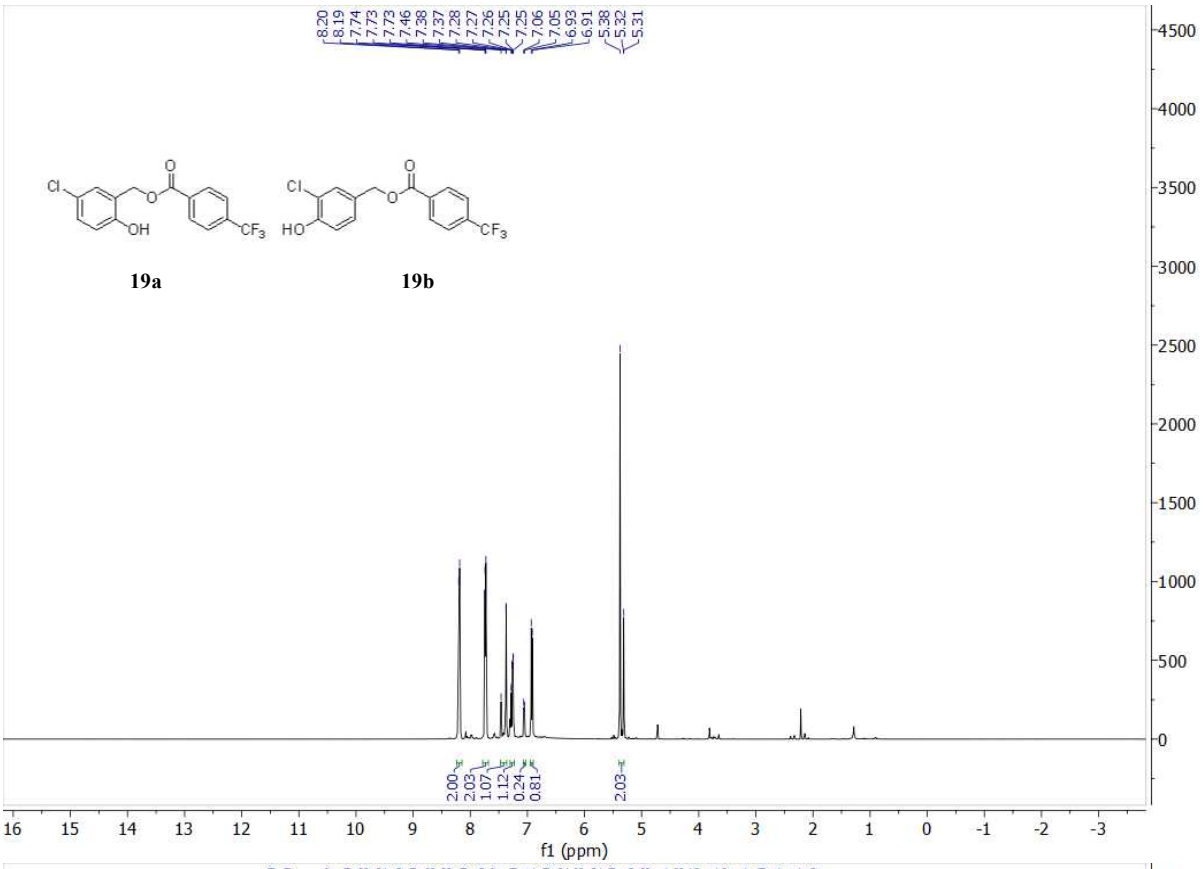

196.56
155.70
139.05
137.45
132.57
130.10
129.57
128.31
122.91
119.72
116.49
O
OH
16b
f1 (ppm)


12.58
12.10
12.10
7.62
7.62
7.61
7.60
7.53
7.53
7.37
7.36
7.36
7.32
7.32
7.30
7.30
7.28
6.92
6.90
6.84
6.82
2.65
2.64
2.34
2.28
Me
O
HO
Me
17a
Me
O
OH
Me
17b
1.01
1.01
1.21
1.00
3.00
3.02
f1 (ppm)

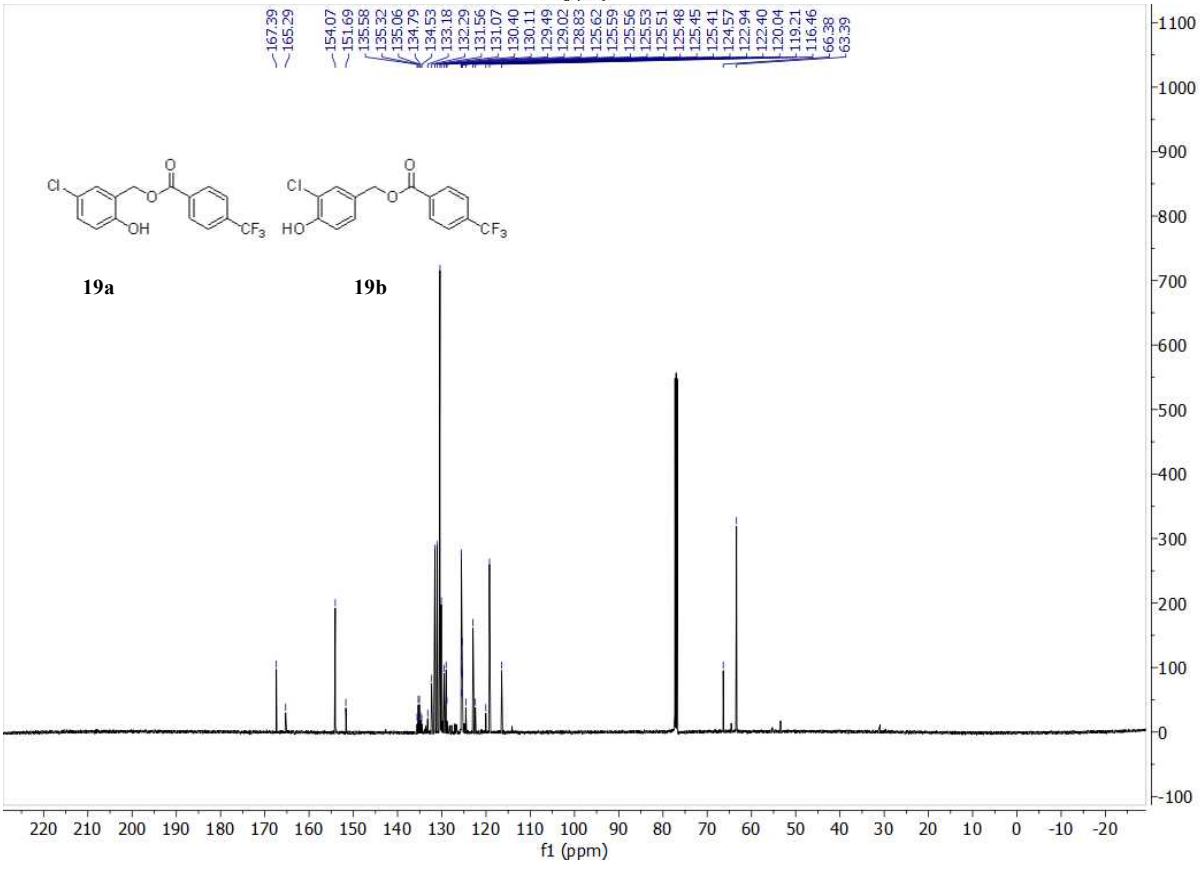

204.76
204.42
160.90
160.32
137.50
137.17
130.44
128.29
128.00
127.47
119.39
119.00
118.22
118.18
26.74
26.65
20.51
15.44
Me
O
HO
Me
17a
Me
O
OH
Me
17b
f1 (ppm)


8.15
8.13
8.13
8.07
8.05
8.05
8.04
8.03
8.02
7.62
7.60
7.59
7.57
7.49
7.49
7.48
7.46
7.28
7.18
7.18
7.13
7.13
7.12
7.11
7.00
6.98
6.83
6.81
4.54
4.53
4.52
4.51
3.10
3.08
3.07
3.03
3.02
3.00
Cl
O
OH
O
18a
Cl
HO
O
O
18b
2.00
1.08
2.12
2.07
0.27
0.76
2.00
2.01
f1 (ppm)


167.36
166.62
153.19
150.08
133.75
133.32
133.07
131.16
130.63
130.21
130.10
129.81
129.68
129.58
129.32
128.97
128.51
128.43
128.07
125.28
125.14
119.79
117.09
116.28
65.33
64.37
34.17
30.11
Cl
O
OH
O
18a
Cl
HO
O
O
18b
f1 (ppm)

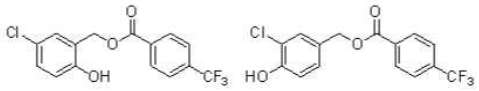


19a **19b**



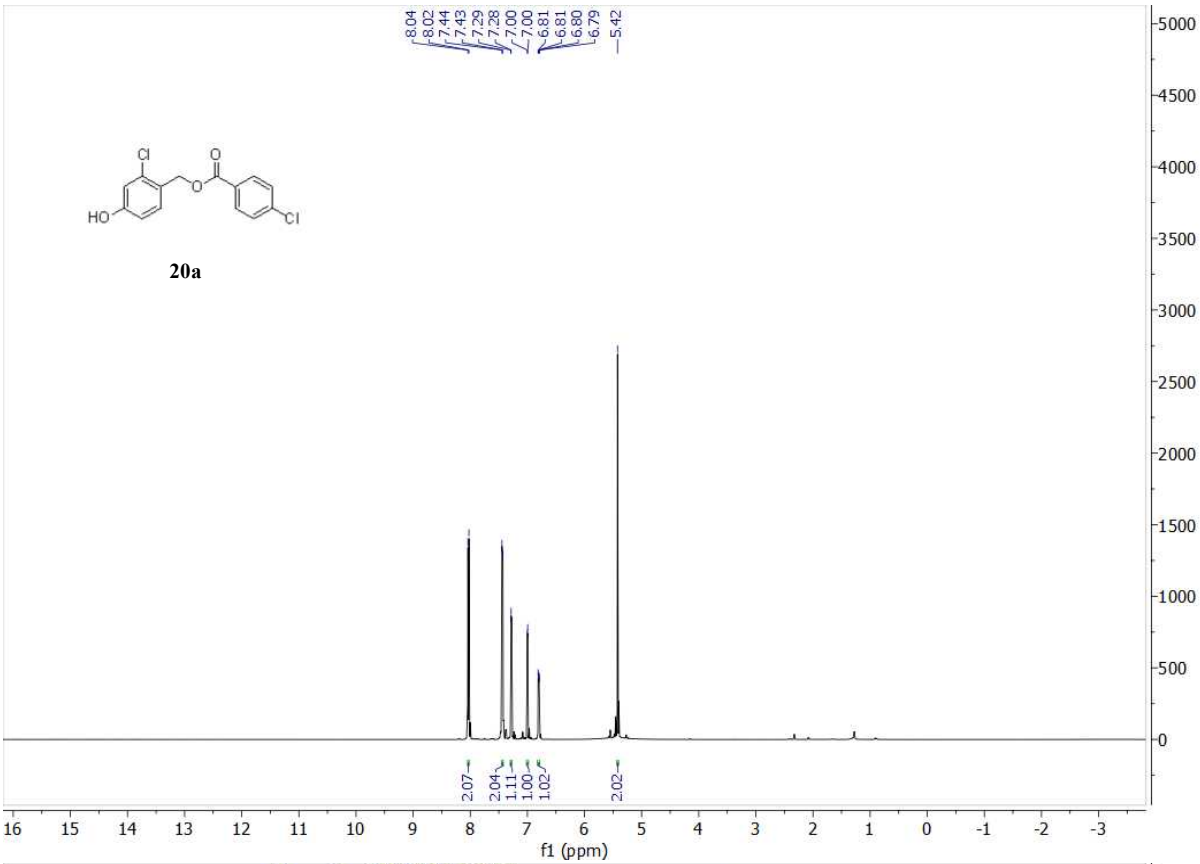
19a **19b**

^{19}F NMR (470 MHz)

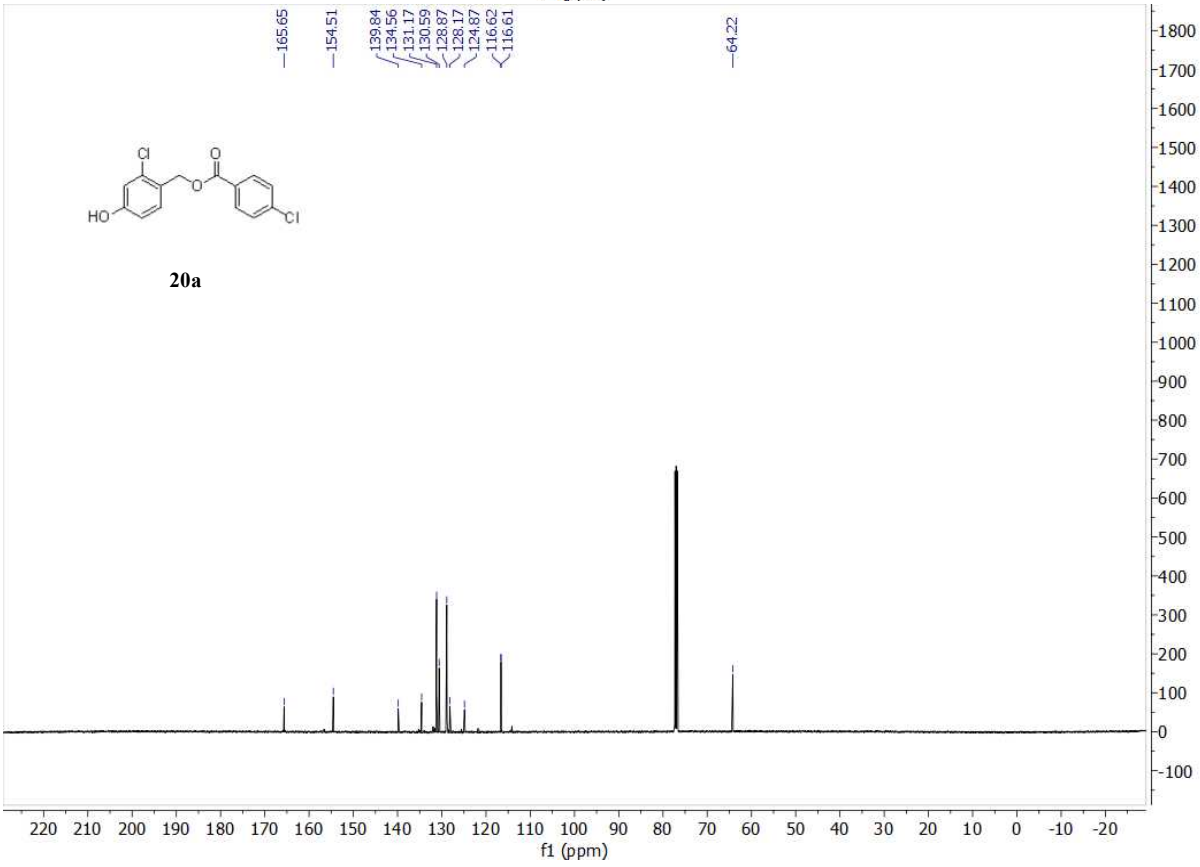


19a

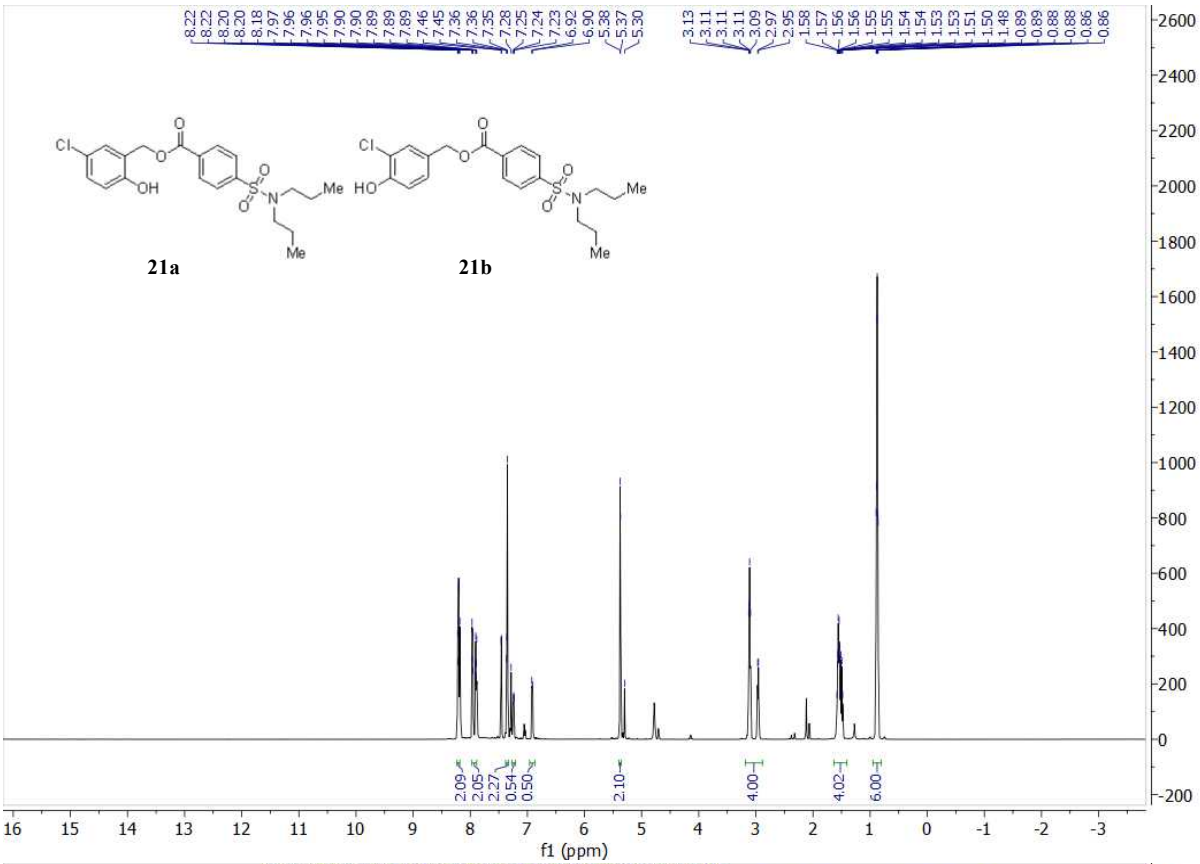
19b

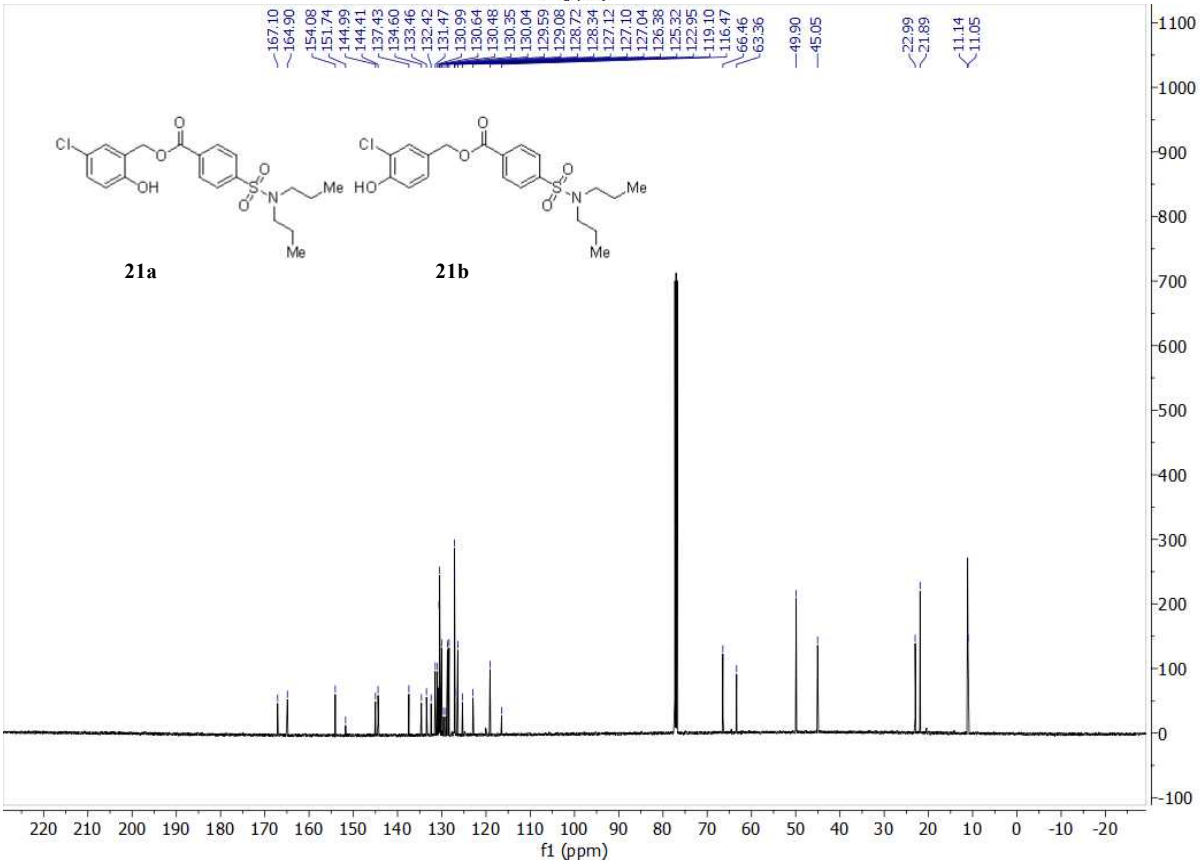

8.04
8.02
7.44
7.43
7.29
7.28
7.00
7.00
6.81
6.81
6.80
6.79
5.42
Cl
O
O
HO
Cl
2.07
2.04
1.11
1.00
1.02
2.02
f1 (ppm)

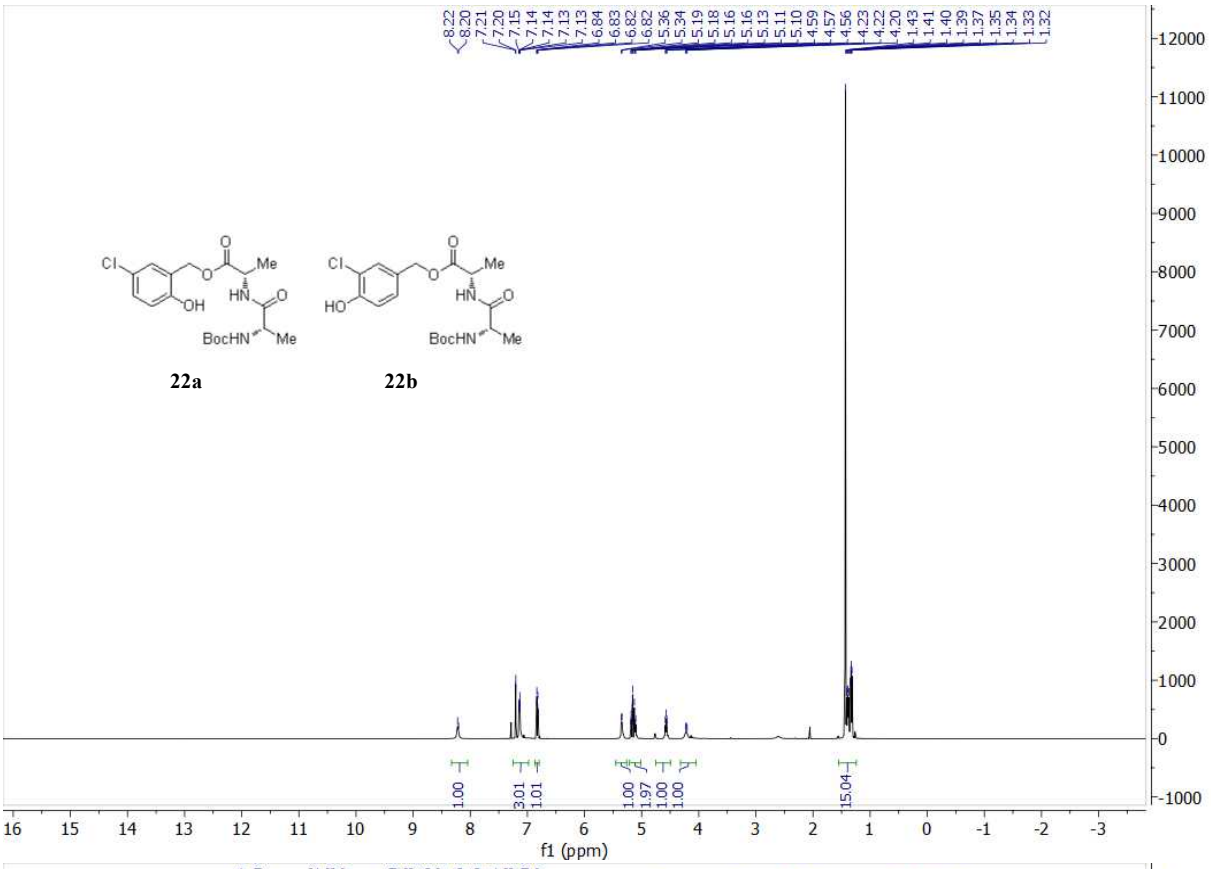
20a


165.65
154.51
139.84
134.56
131.17
130.59
128.87
128.17
124.87
116.62
116.61
64.22
Cl
O
O
HO
Cl
f1 (ppm)

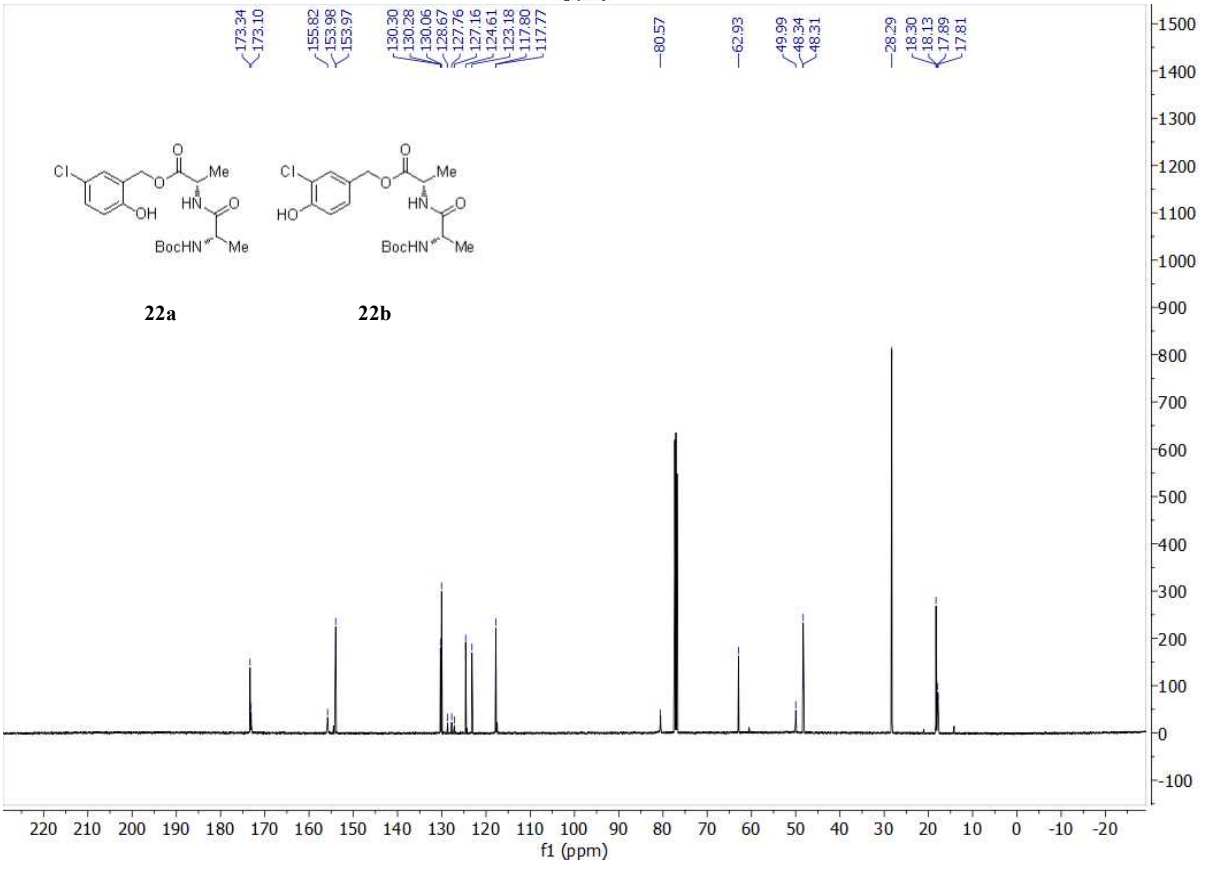
20a


21a
21b
f1 (ppm)

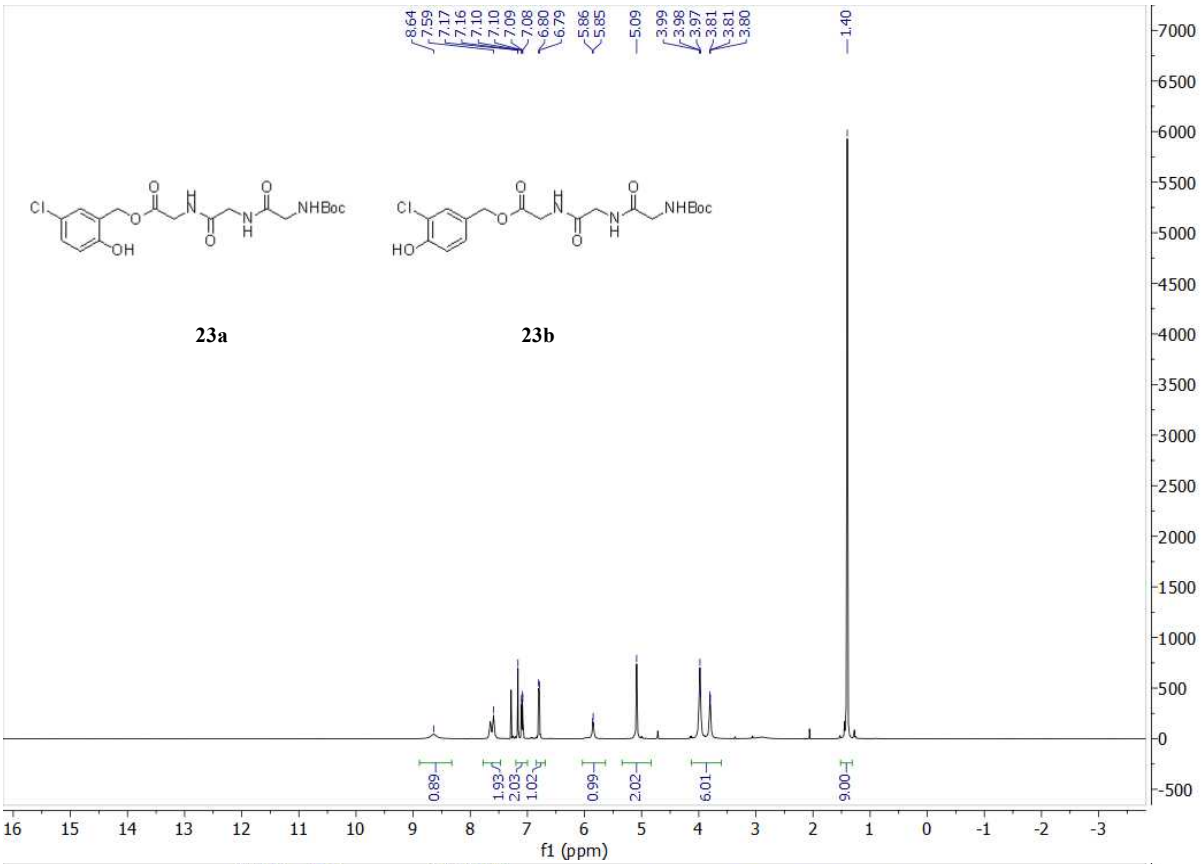

21a
21b
f1 (ppm)



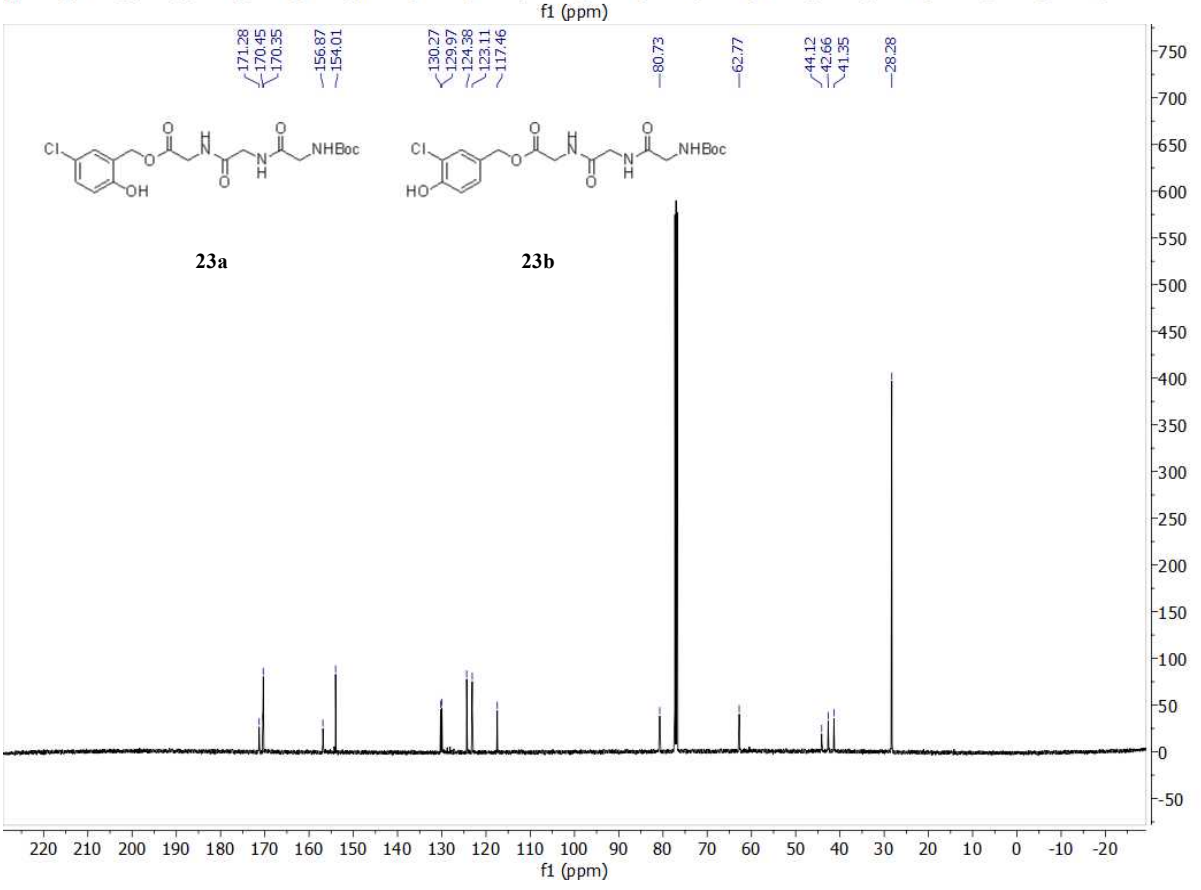
22a **22b**



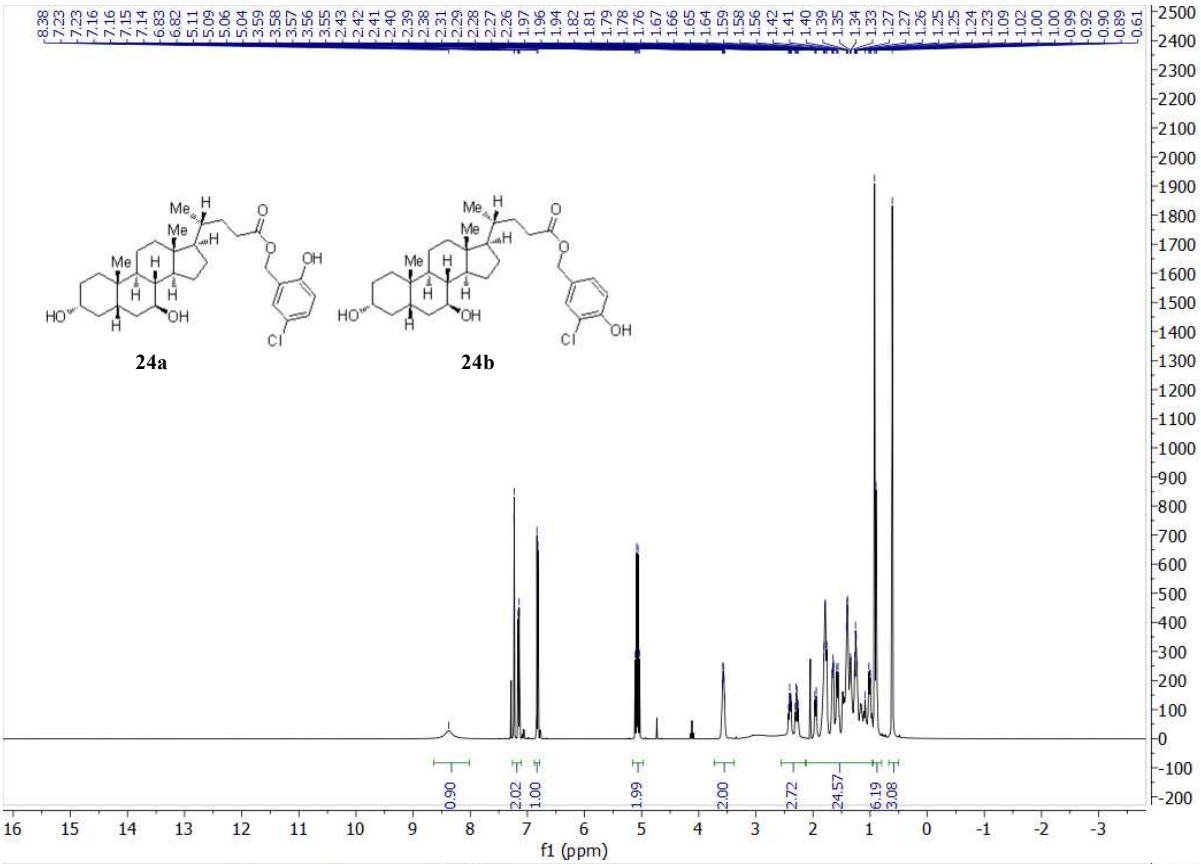
22a **22b**

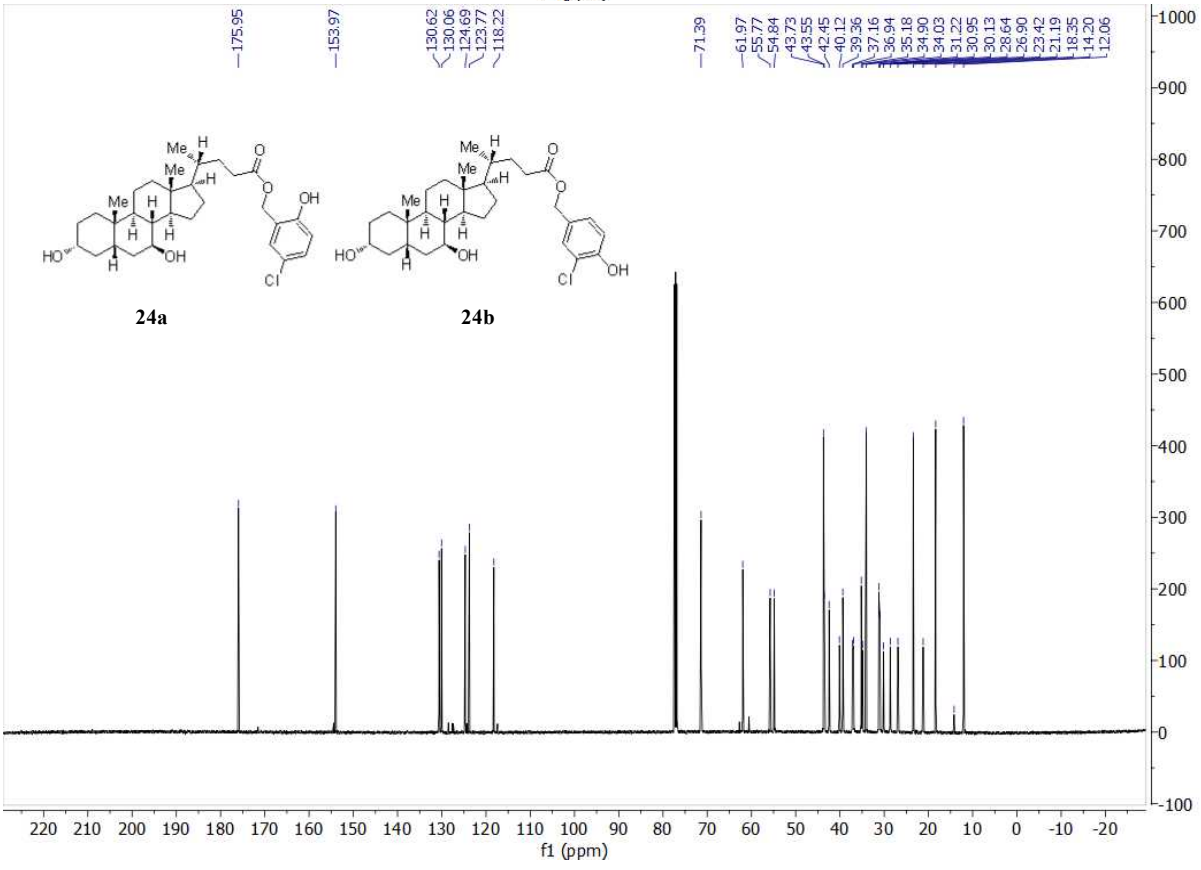


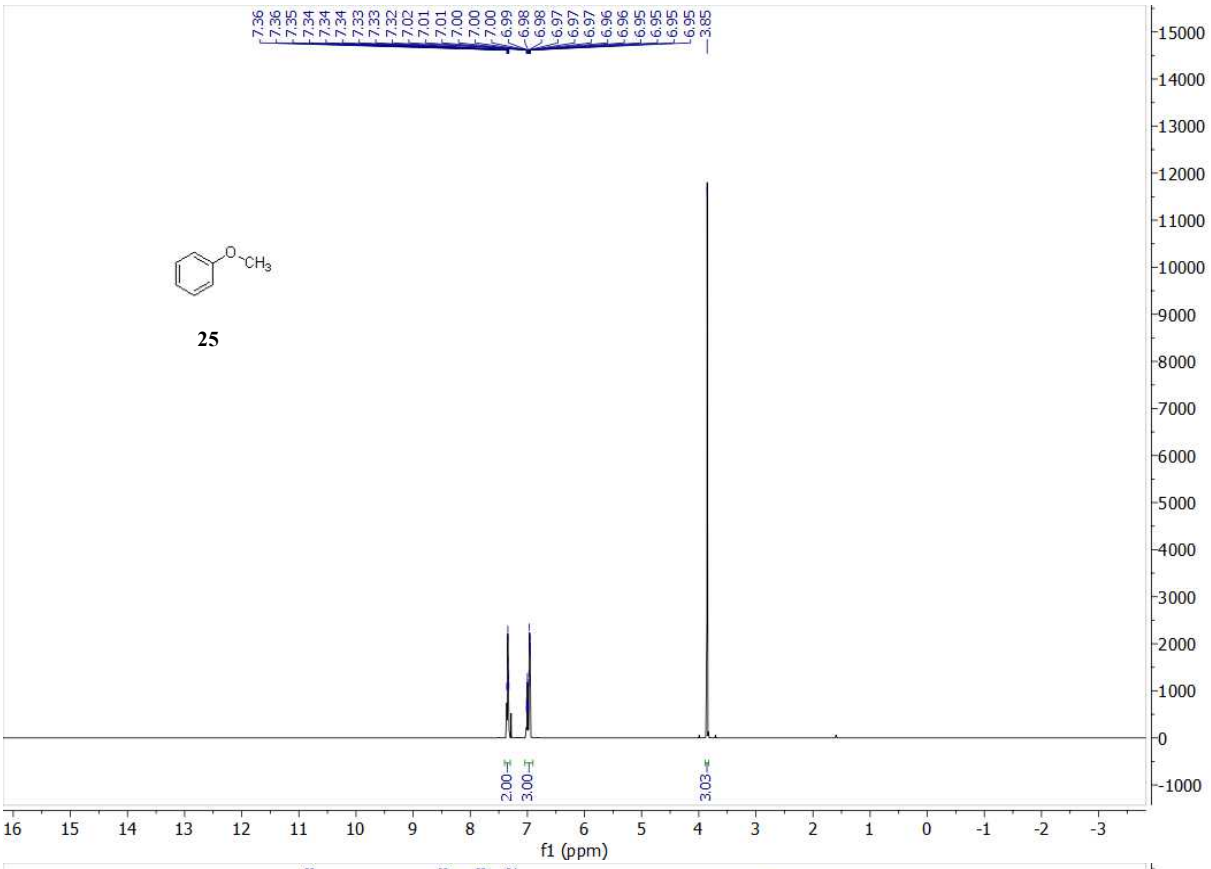
23a **23b**



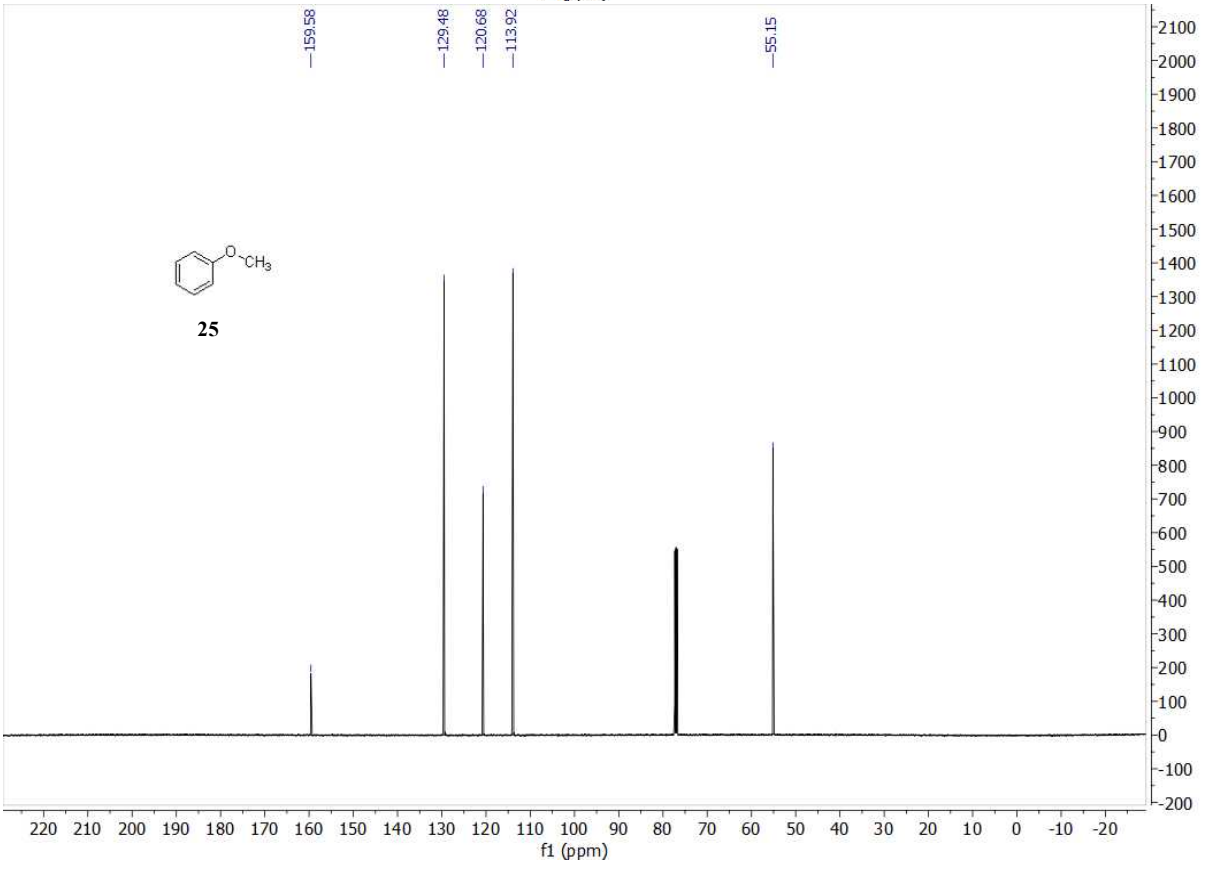
23a **23b**


24a
24b
f1 (ppm)

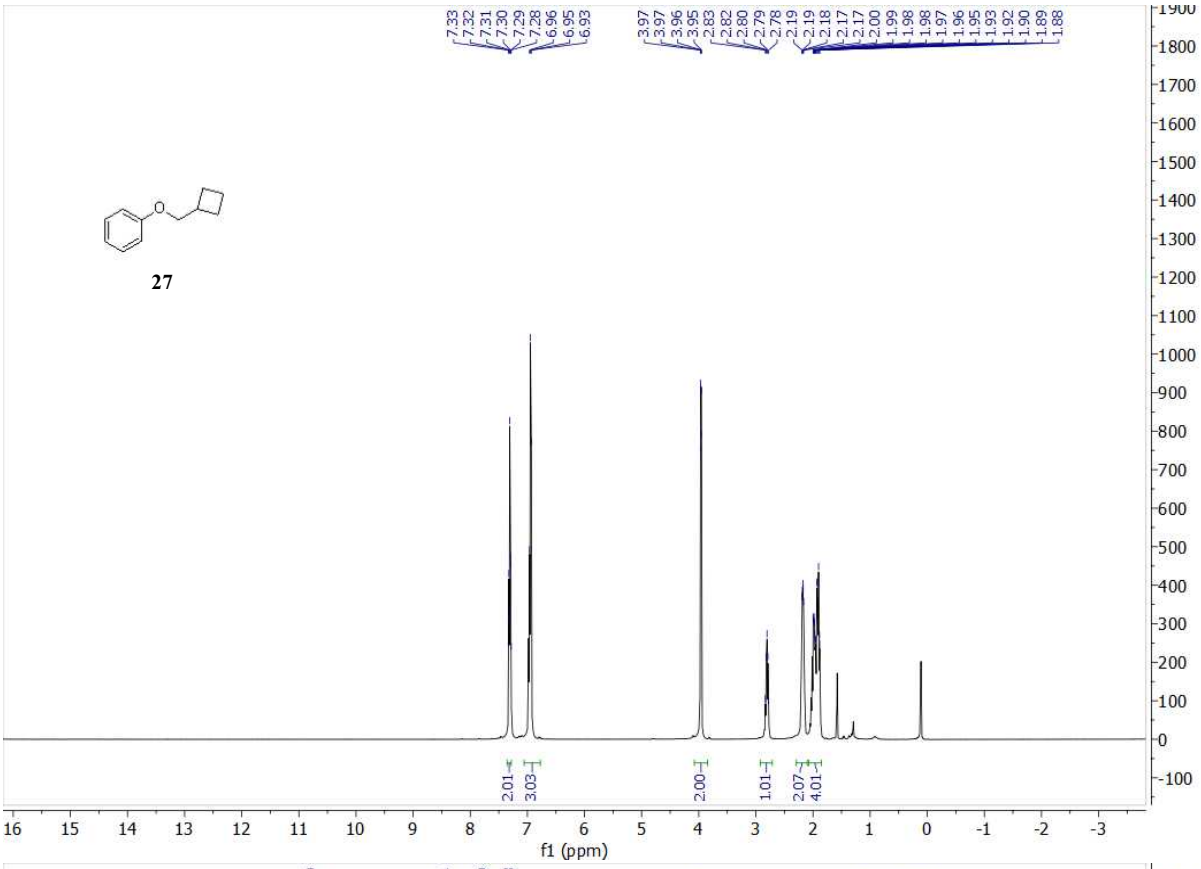

24a
24b
f1 (ppm)


7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.33
7.32
7.02
7.01
7.01
7.00
7.00
7.00
6.99
6.98
6.98
6.97
6.97
6.97
6.96
6.96
6.95
6.95
6.95
6.95
3.85
O
CH3
2.00
3.00
3.03
f1 (ppm)

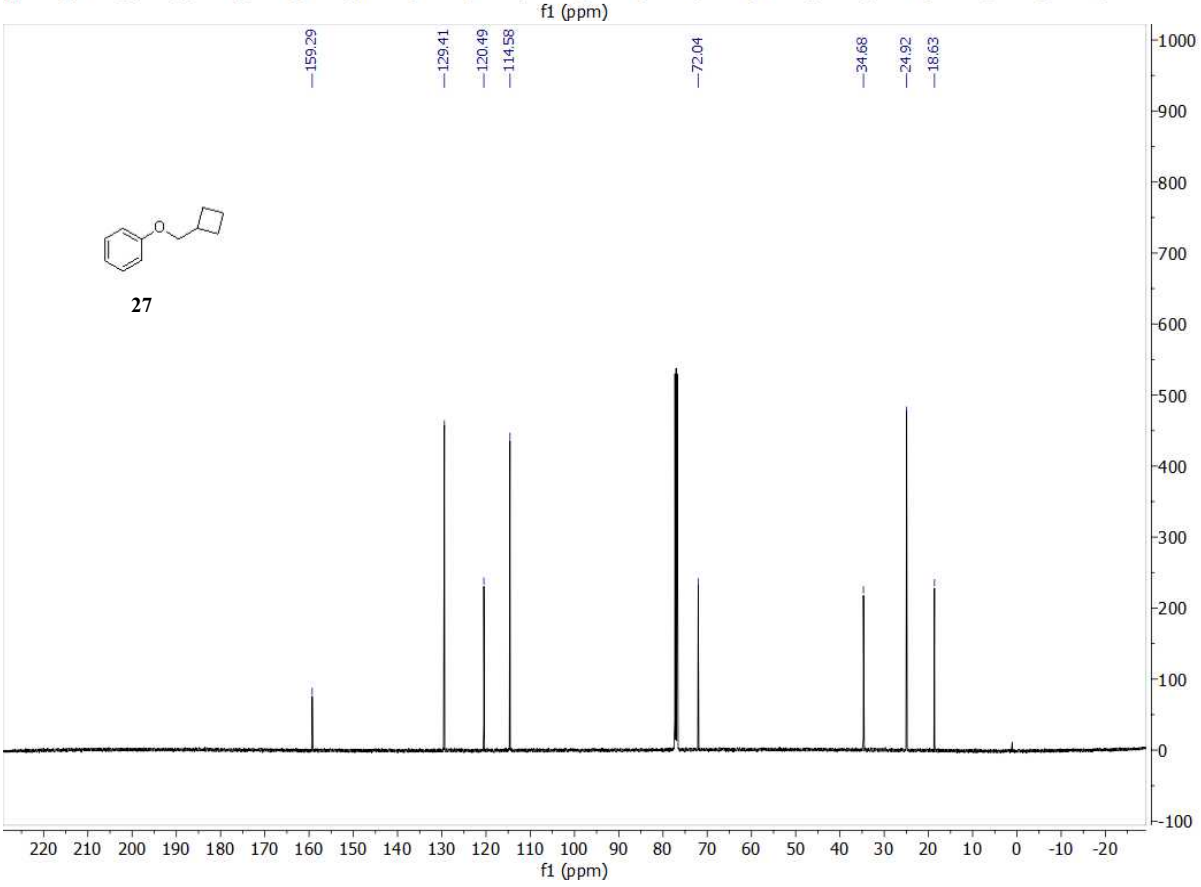
25


159.58
129.48
120.68
113.92
55.15
O
CH3
f1 (ppm)

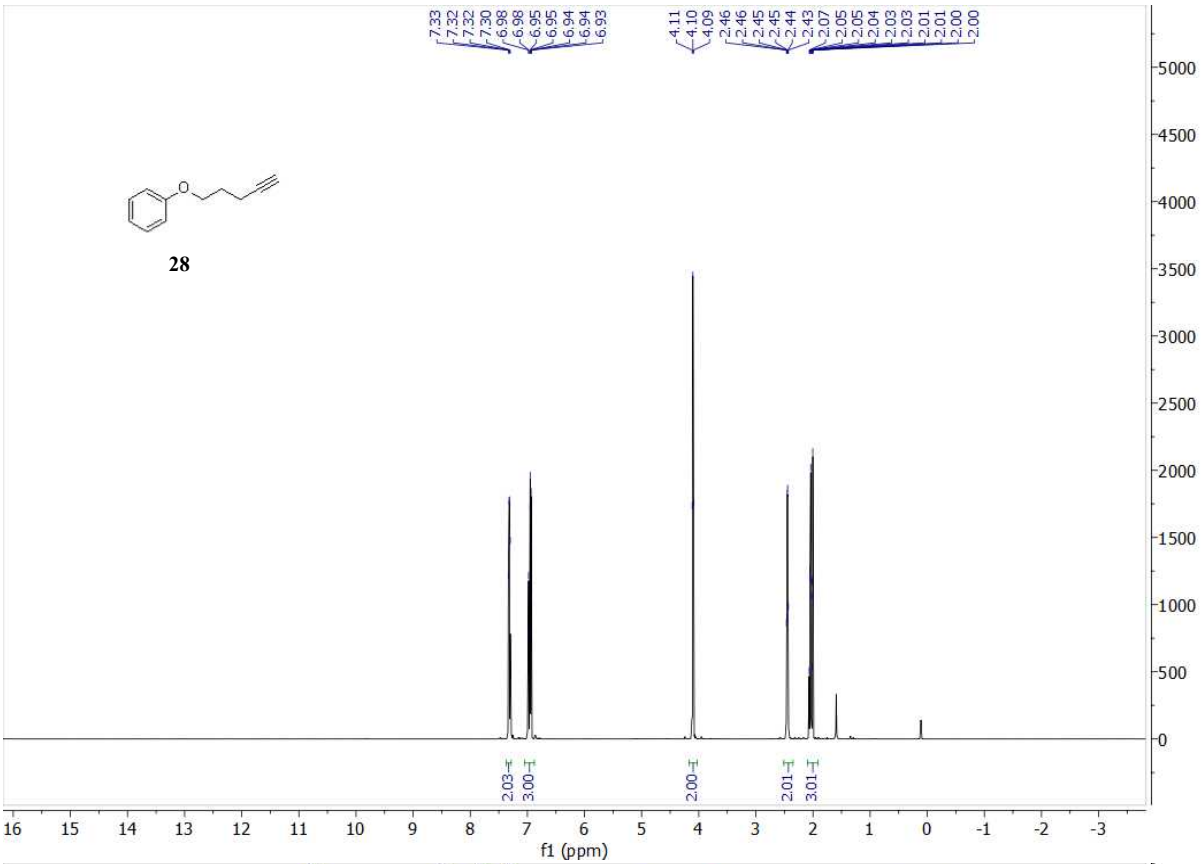
25


7.33
7.32
7.31
7.30
7.29
7.28
6.96
6.95
6.93
3.97
3.97
3.96
3.95
2.83
2.82
2.80
2.79
2.78
2.19
2.19
2.18
2.17
2.17
2.00
1.99
1.98
1.98
1.97
1.96
1.95
1.93
1.92
1.90
1.89
1.88
2.01
3.03
2.00
1.01
2.07
4.01
f1 (ppm)

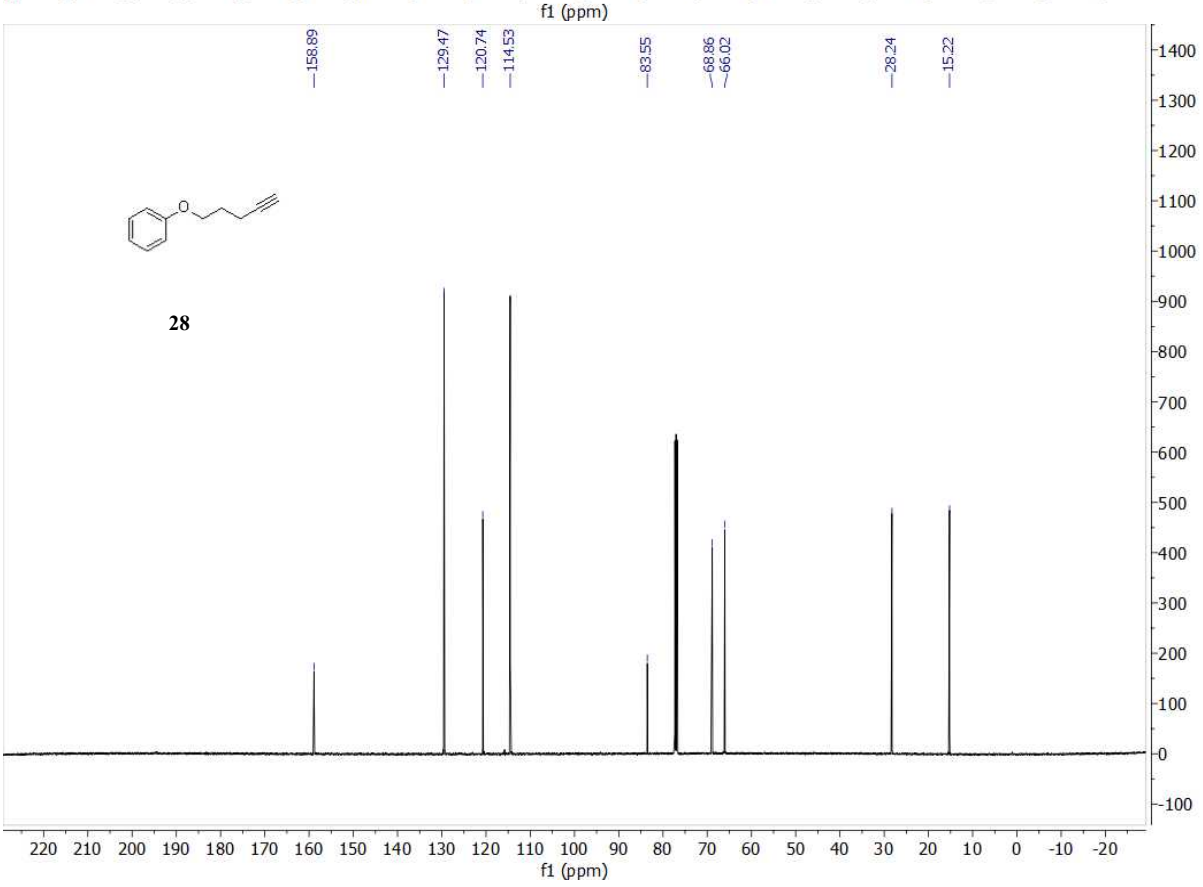
27


159.29
129.41
120.49
114.58
72.04
34.68
24.92
18.63
f1 (ppm)

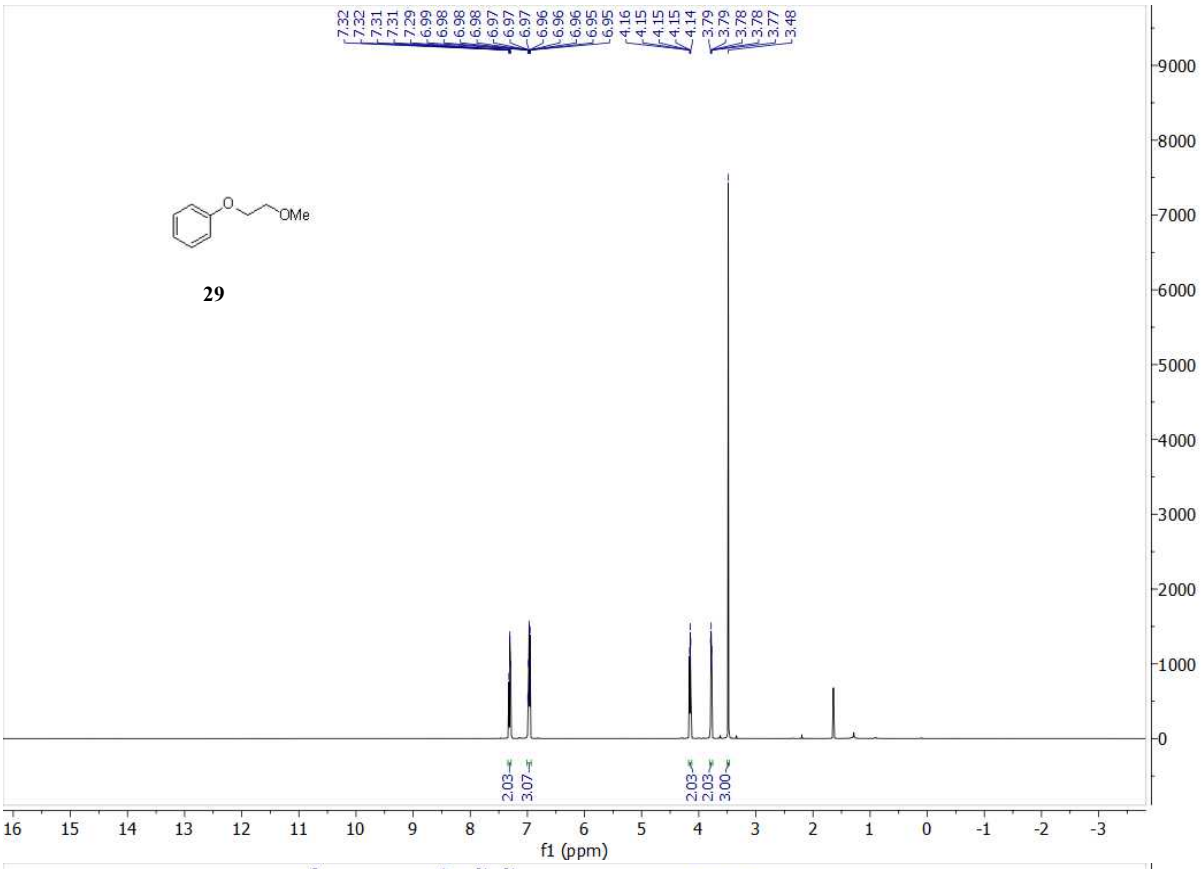
27


7.33
7.32
7.32
7.30
6.98
6.98
6.95
6.95
6.94
6.94
6.93
4.11
4.10
4.09
2.46
2.46
2.45
2.45
2.44
2.43
2.07
2.05
2.05
2.04
2.03
2.03
2.01
2.01
2.00
2.00
2.03
3.00
2.00
2.01
3.01
f1 (ppm)

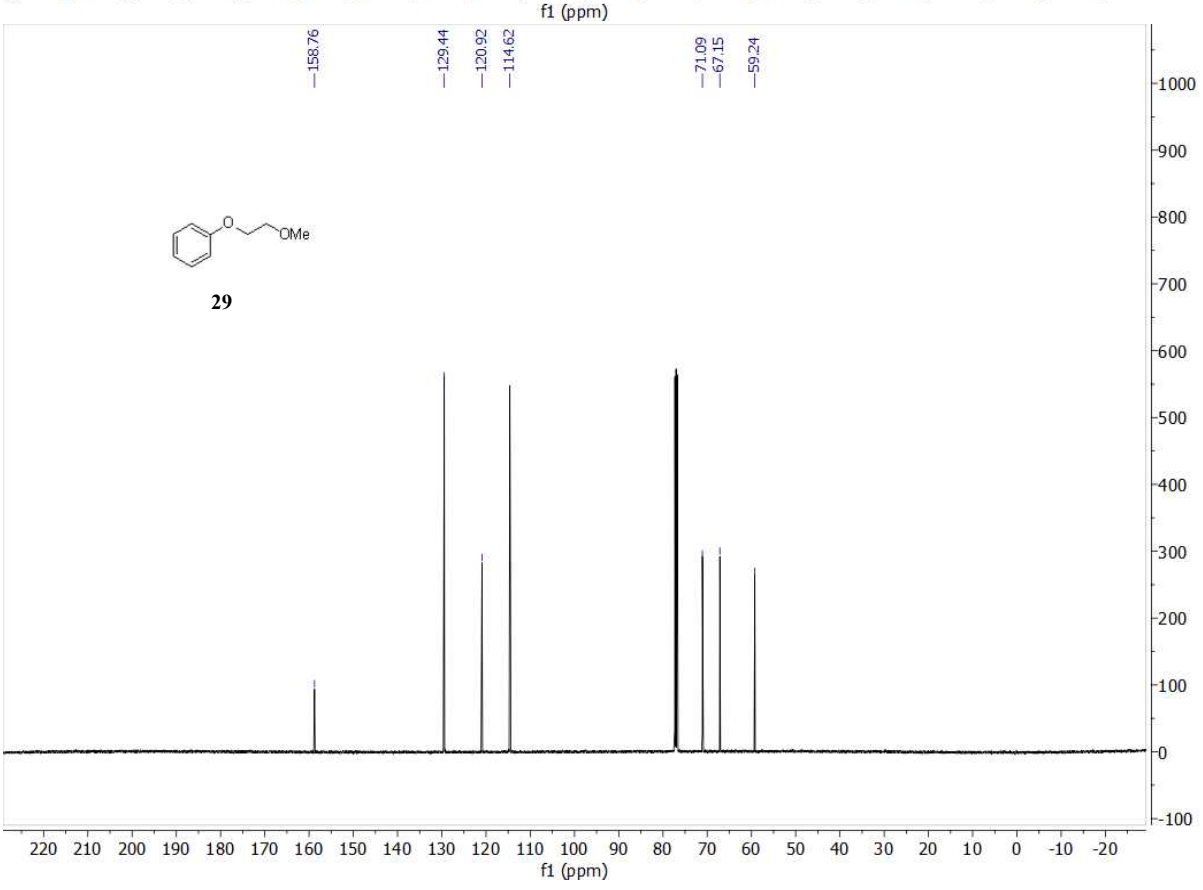
28


158.89
129.47
120.74
114.53
83.55
68.86
66.02
28.24
15.22
f1 (ppm)

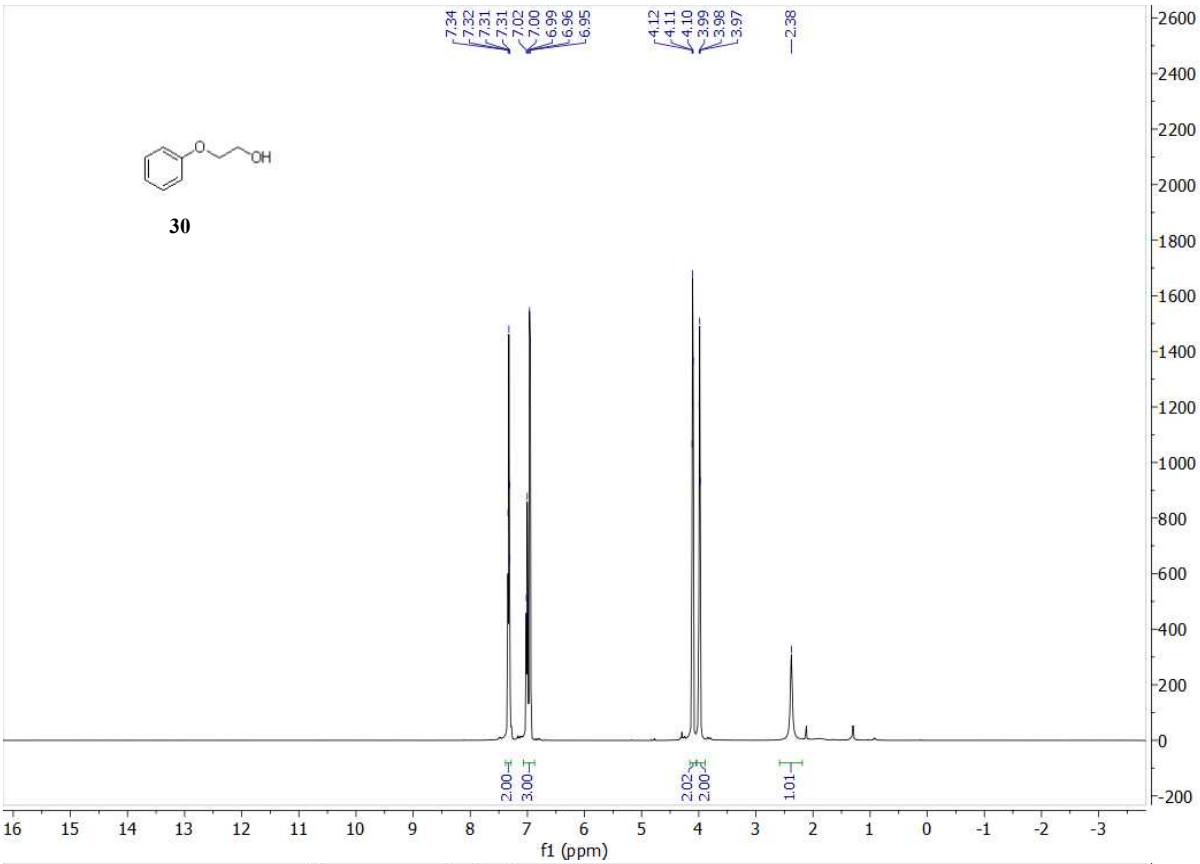
28

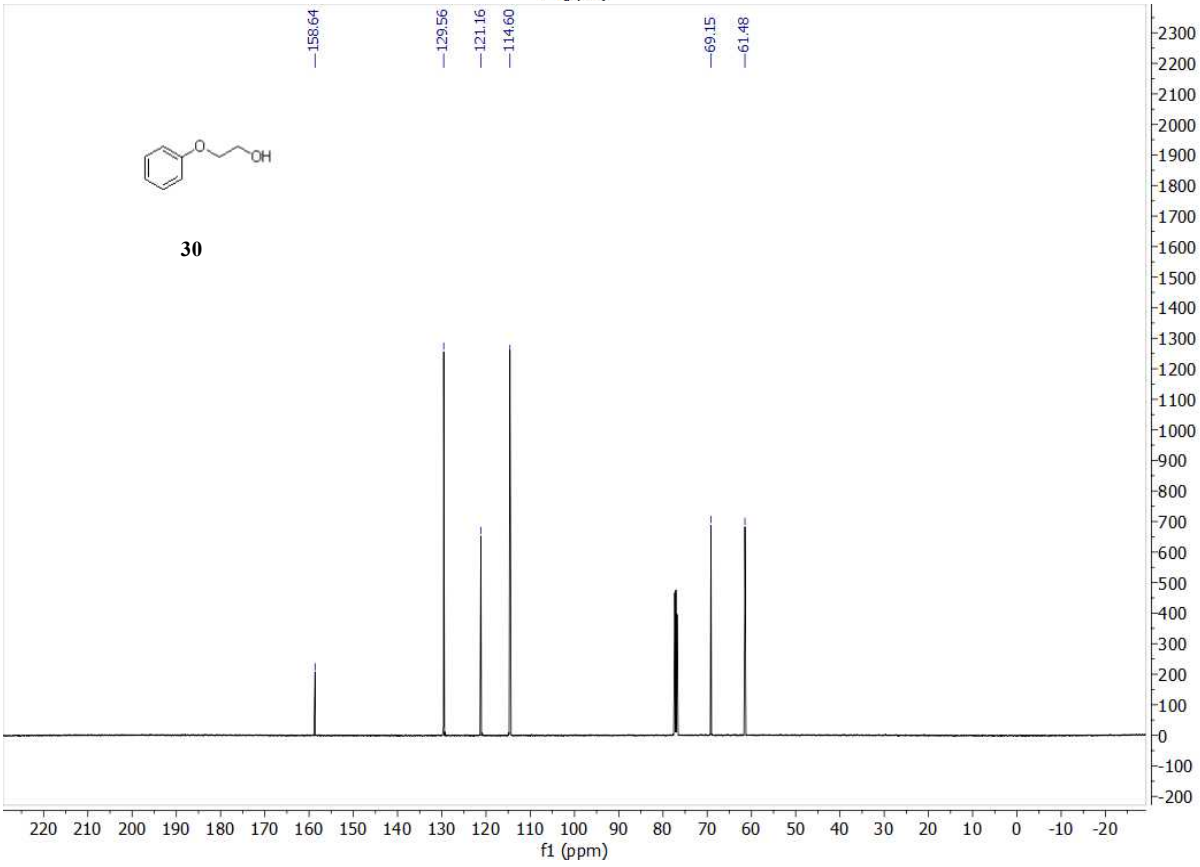

7.32
7.32
7.31
7.31
7.29
6.99
6.98
6.98
6.98
6.97
6.97
6.97
6.96
6.96
6.96
6.96
6.95
6.95
4.16
4.15
4.15
4.15
4.14
3.79
3.79
3.78
3.78
3.77
3.48
O
OMe
2.03
3.07
2.03
2.03
3.00
f1 (ppm)

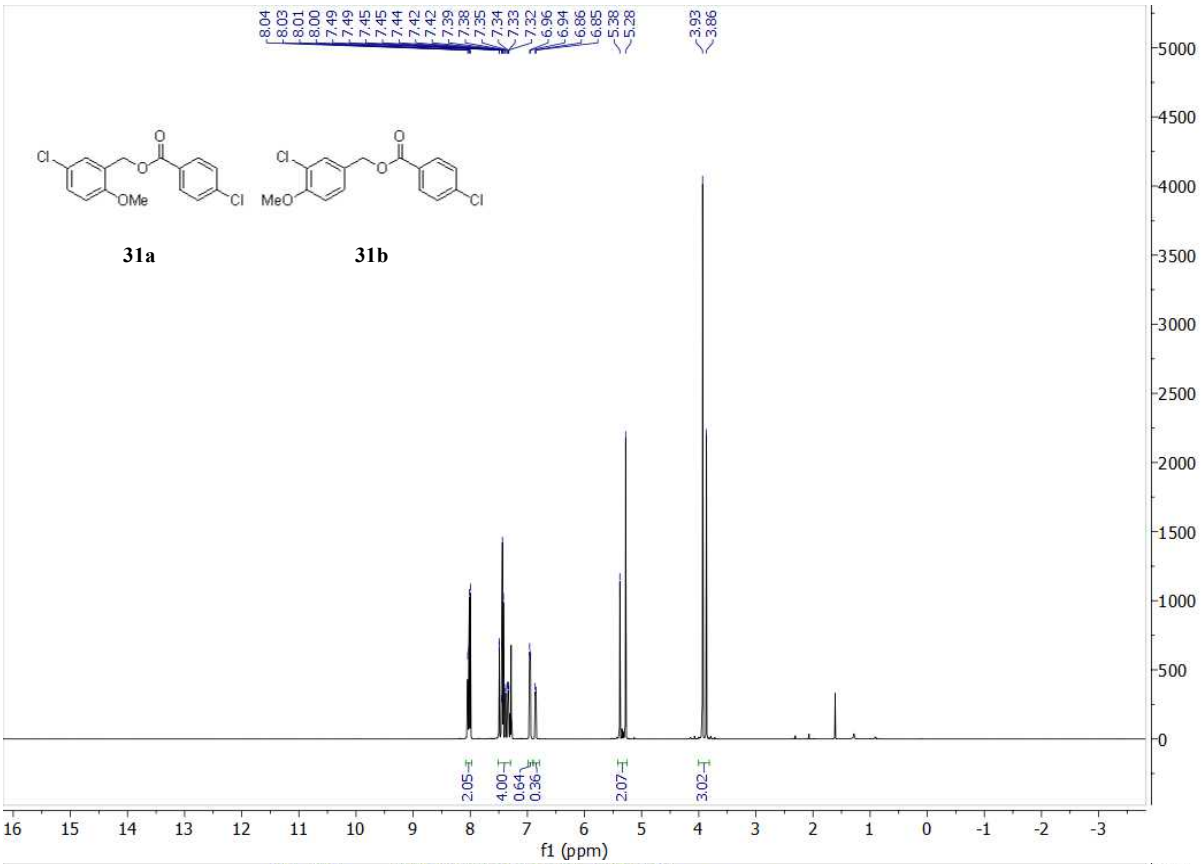
29


158.76
129.44
120.92
114.62
71.09
67.15
59.24
O
OMe
f1 (ppm)

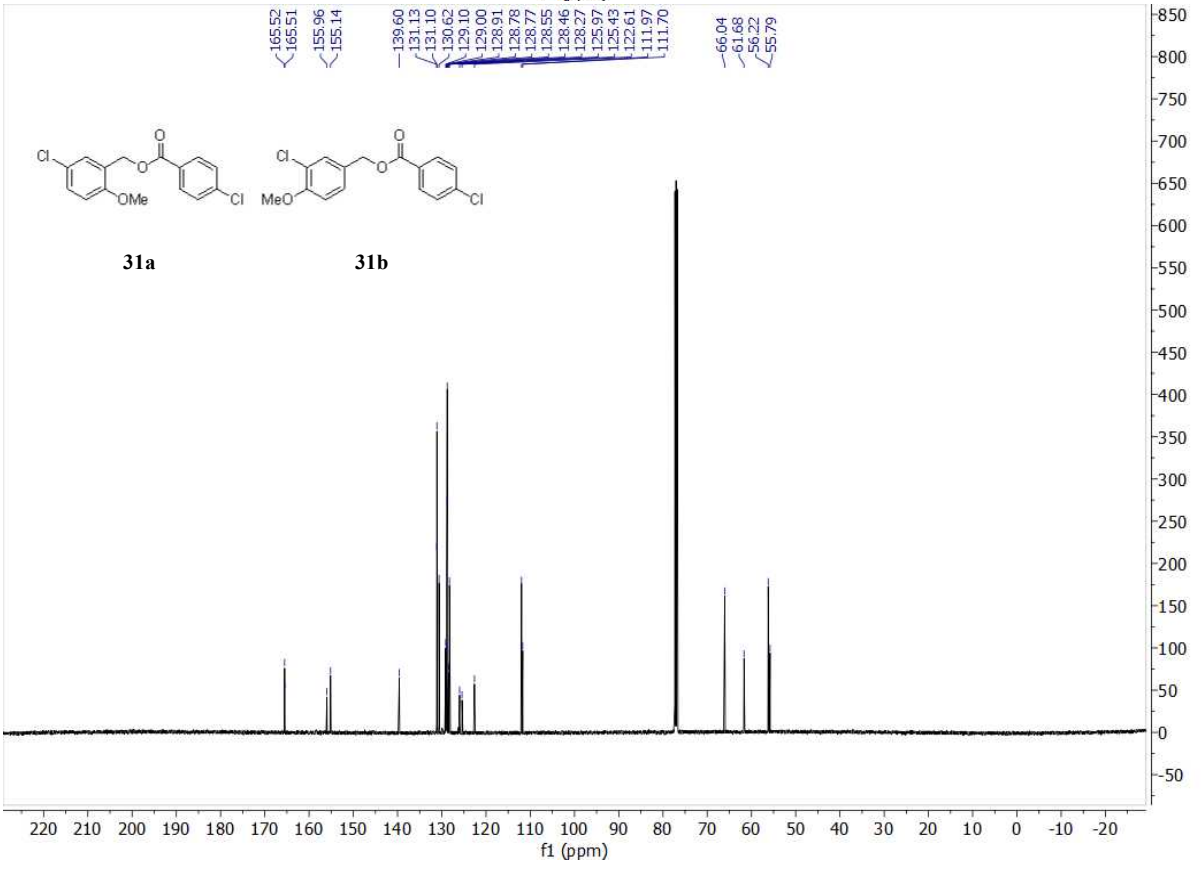
29


7.34
7.32
7.31
7.31
7.02
7.00
6.99
6.96
6.95
4.12
4.11
4.10
3.99
3.98
3.97
2.38
OH
30
2.00
3.00
2.02
2.00
1.01
f1 (ppm)

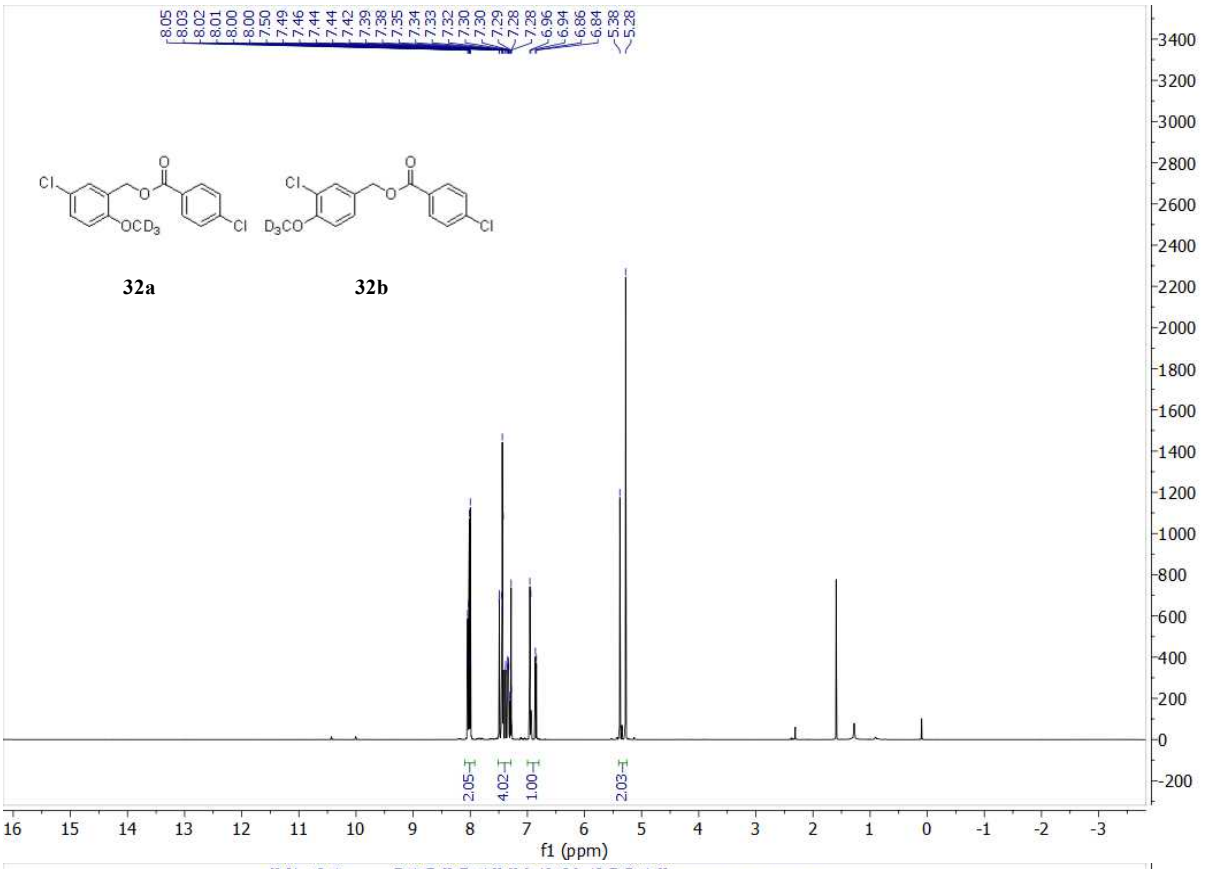

158.64
129.56
121.16
114.60
69.15
61.48
OH
30
f1 (ppm)



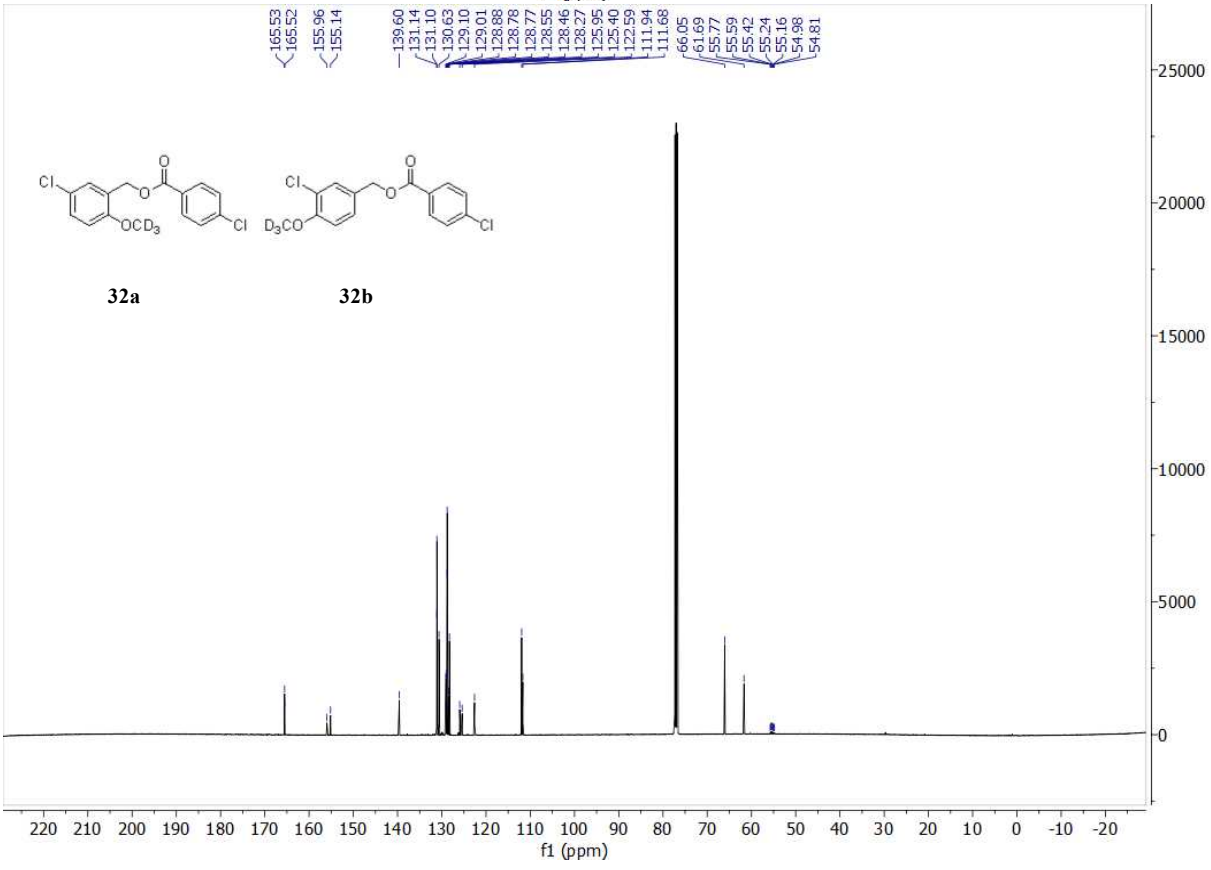
31a **31b**



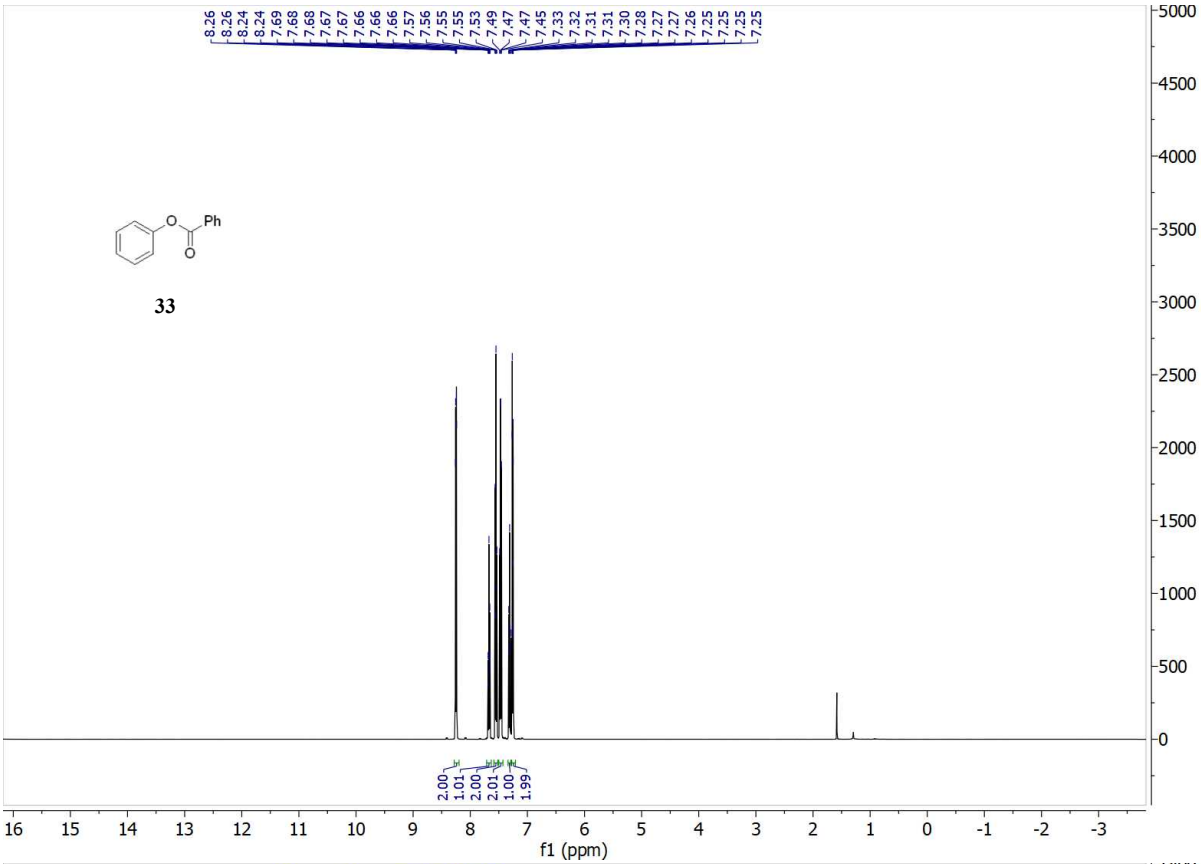
31a **31b**

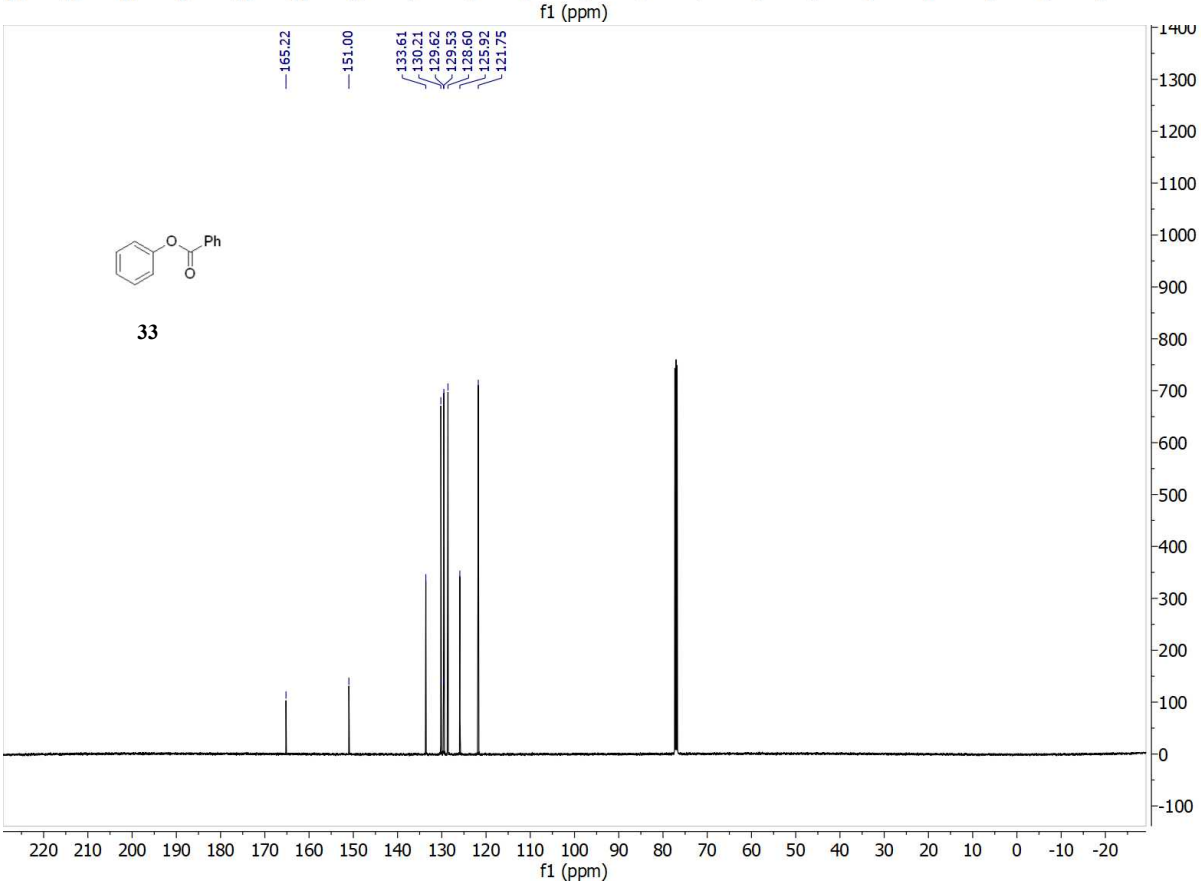


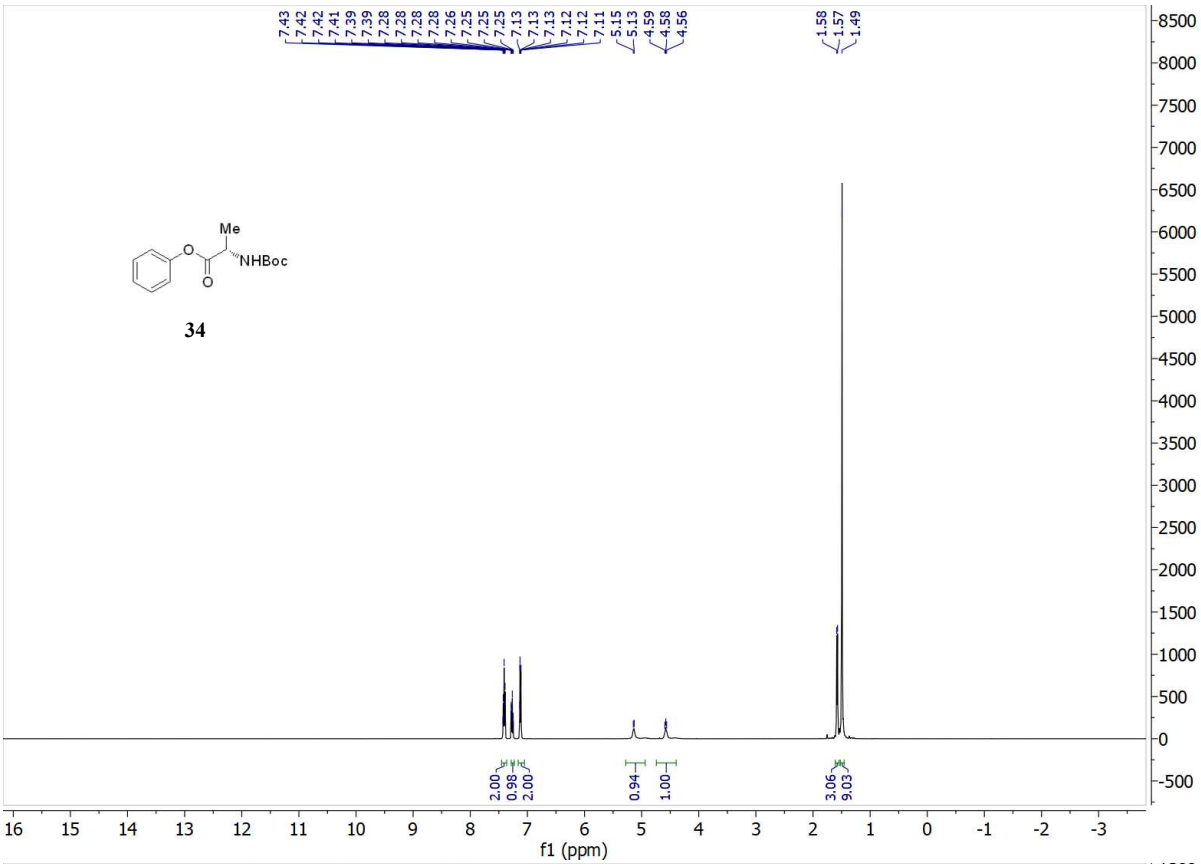
32a **32b**



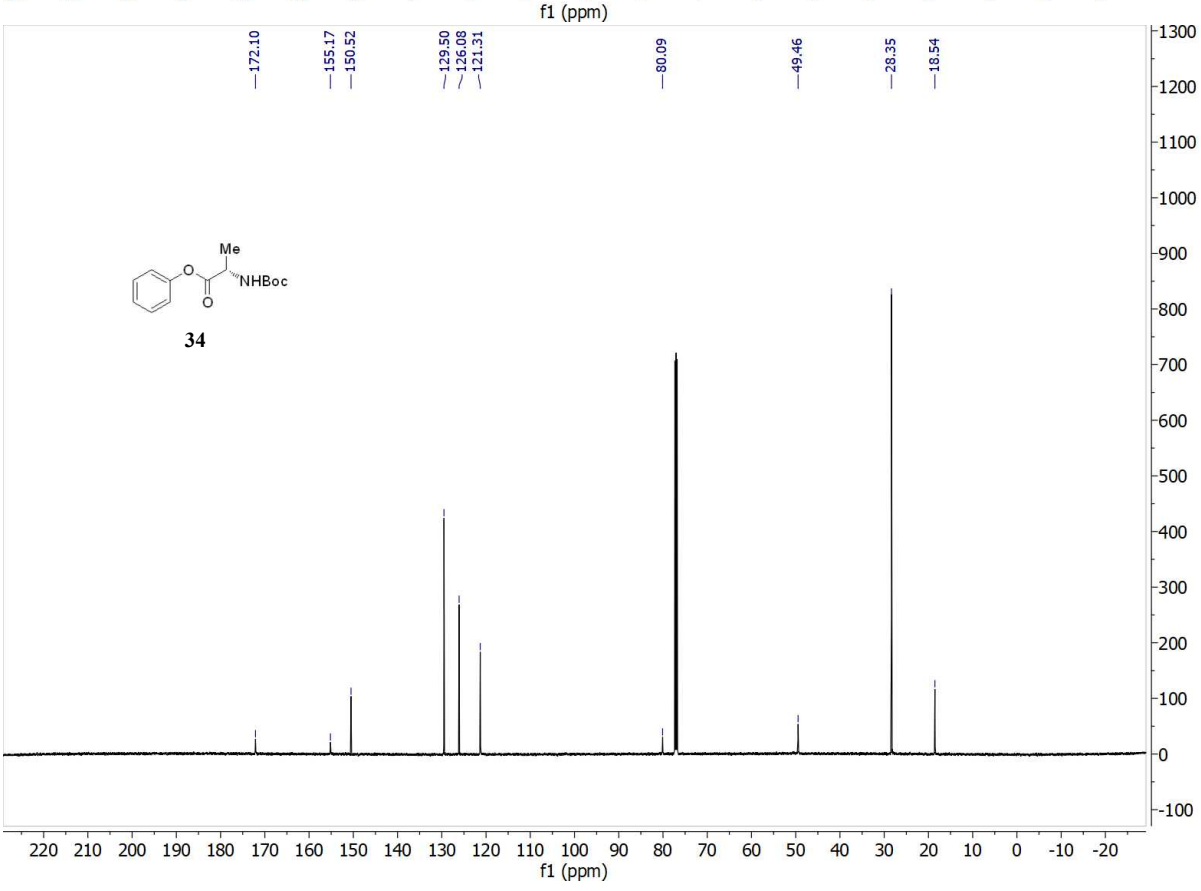
32a **32b**


8.26
8.26
8.24
8.24
7.69
7.68
7.68
7.67
7.67
7.66
7.66
7.66
7.66
7.57
7.56
7.55
7.55
7.53
7.49
7.47
7.47
7.45
7.33
7.32
7.31
7.31
7.30
7.28
7.27
7.27
7.26
7.25
7.25
7.25
7.25
O
Ph
O
33
2.00
1.01
2.00
2.01
1.00
1.99
f1 (ppm)

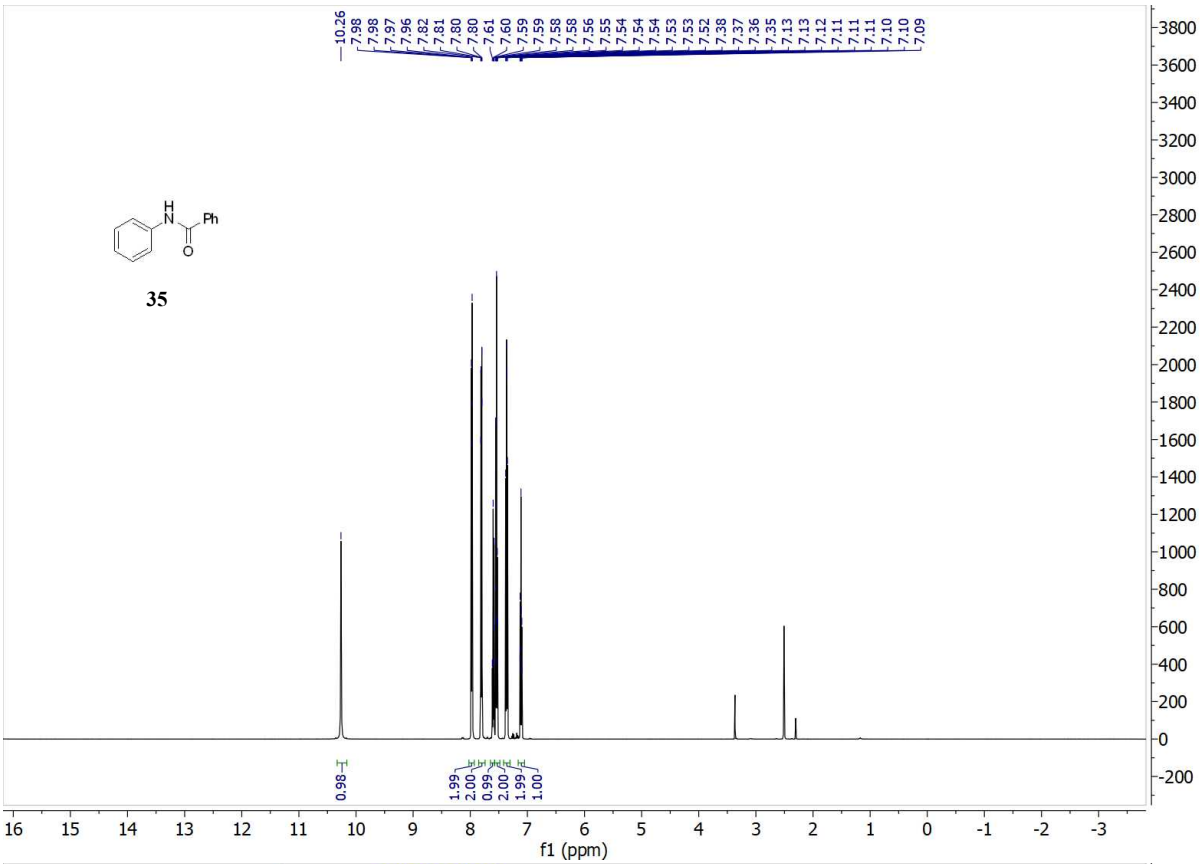

165.22
151.00
133.61
130.21
129.62
129.53
128.60
125.92
121.75
O
Ph
O
33
f1 (ppm)

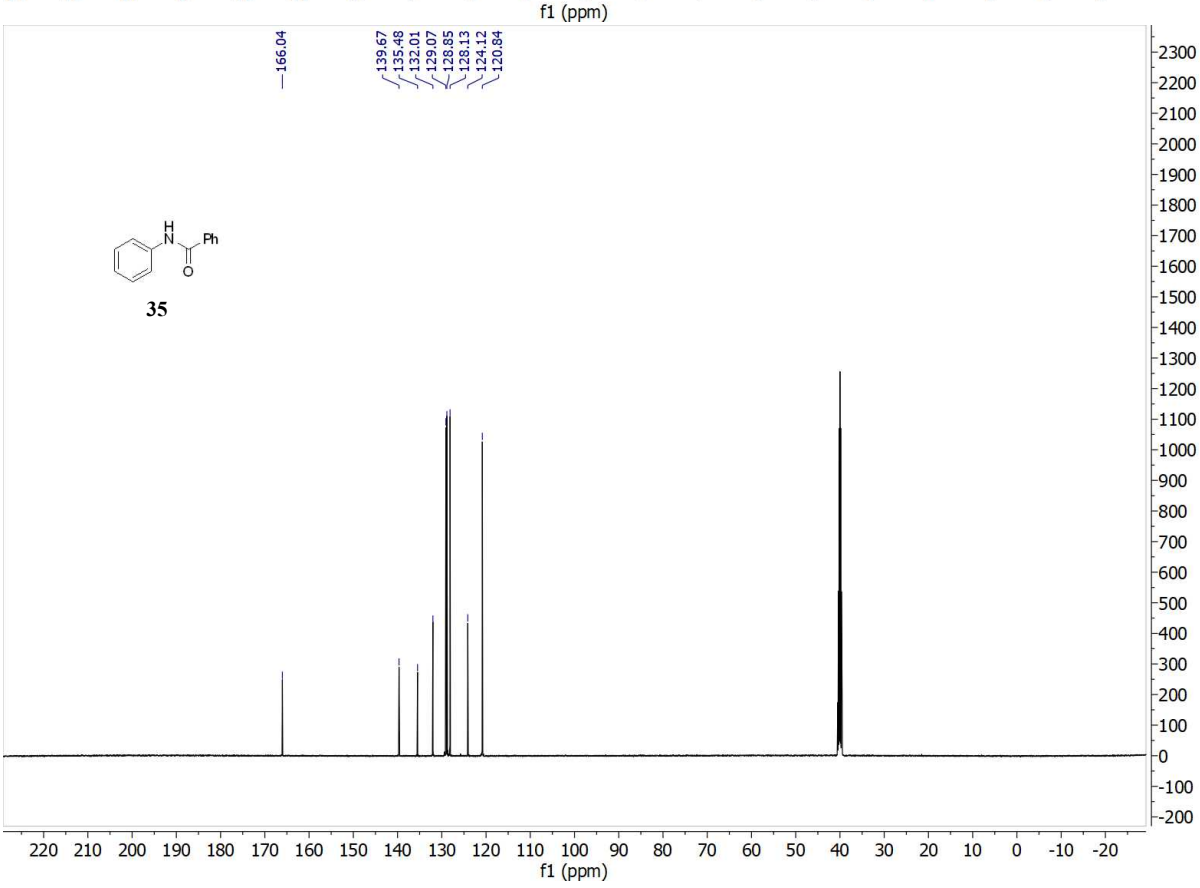

7.43
7.42
7.42
7.41
7.39
7.39
7.28
7.28
7.28
7.28
7.26
7.25
7.25
7.25
7.13
7.13
7.13
7.12
7.12
7.11
5.15
5.13
4.59
4.58
4.56
1.58
1.57
1.49
Me
NHBoc
O
O
2.00
0.98
2.00
0.94
1.00
3.06
9.03
f1 (ppm)

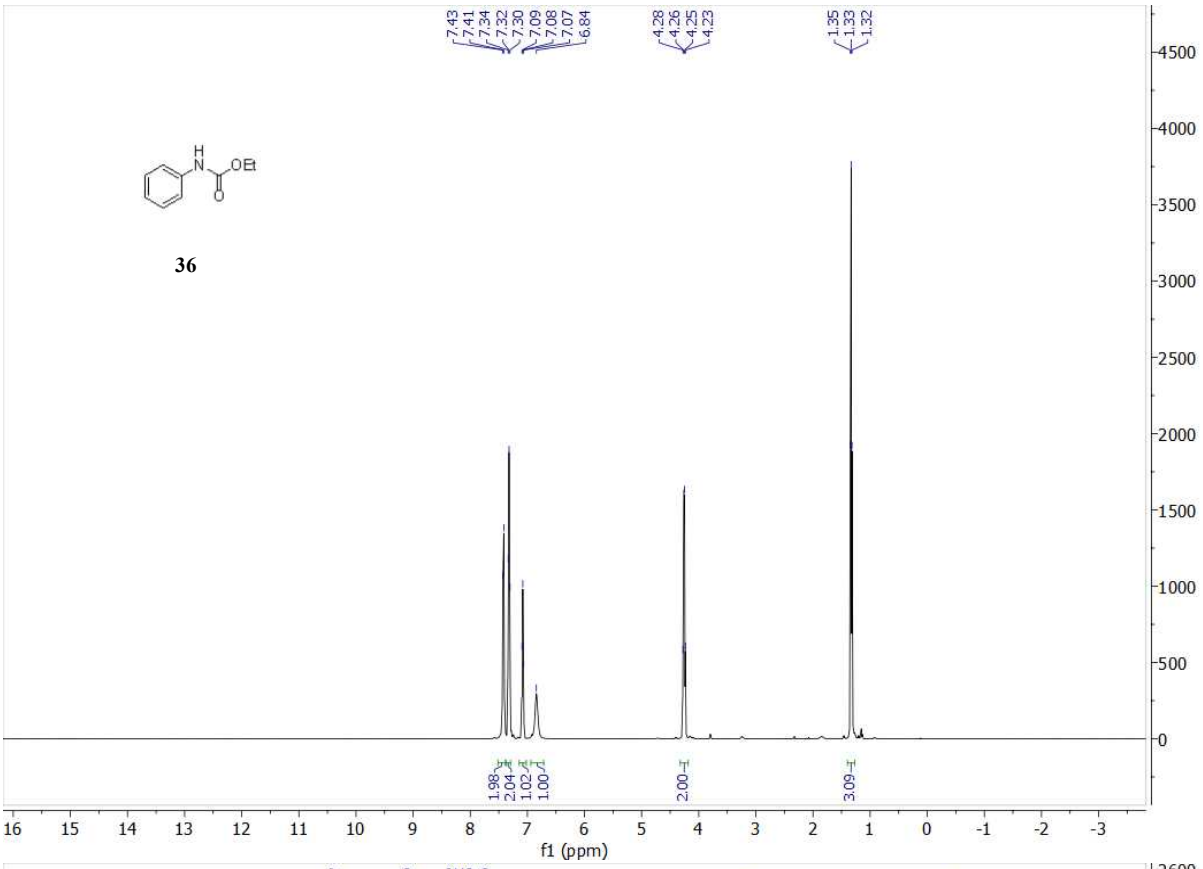
34


172.10
155.17
150.52
129.50
126.08
121.31
80.09
49.46
28.35
18.54
Me
NHBoc
O
O
f1 (ppm)

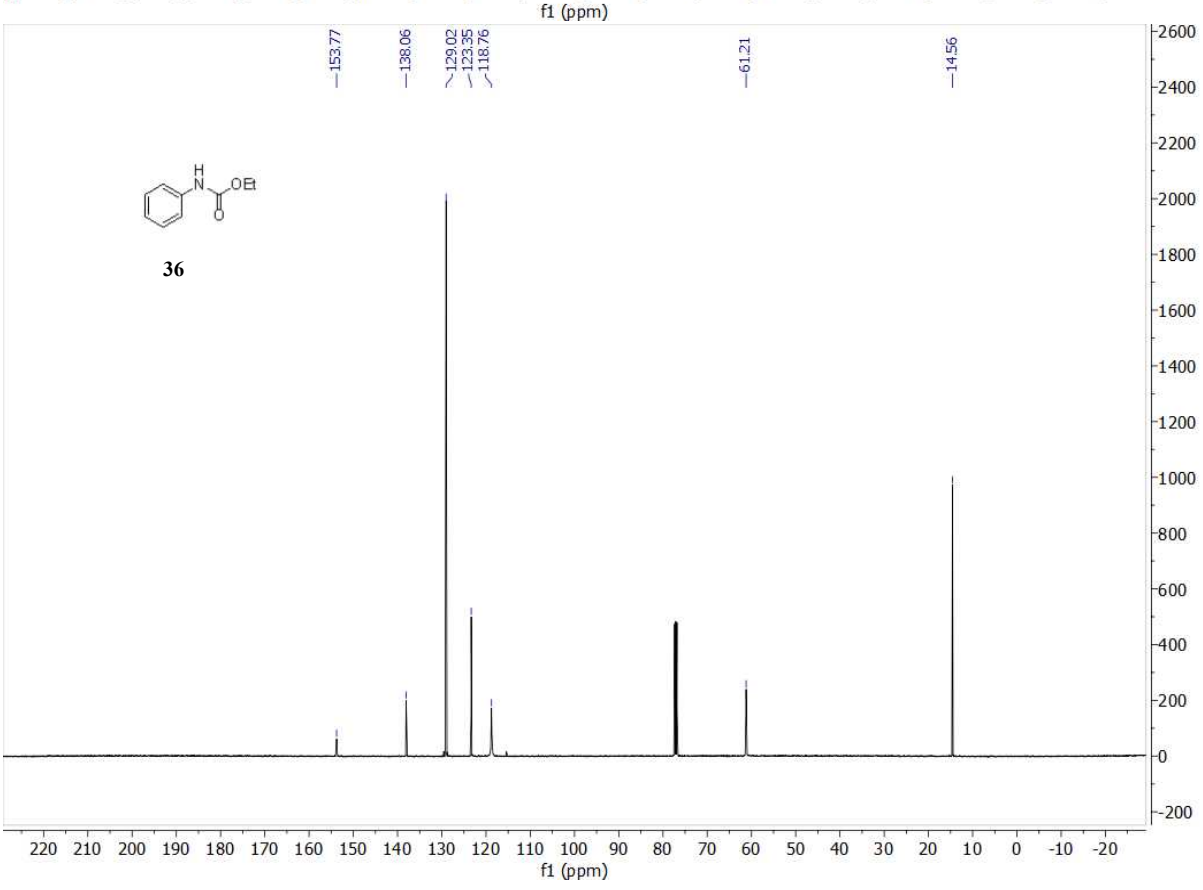
34


10.26
7.98
7.98
7.97
7.96
7.82
7.81
7.80
7.80
7.61
7.60
7.59
7.59
7.58
7.58
7.56
7.55
7.54
7.54
7.54
7.53
7.53
7.52
7.38
7.37
7.36
7.35
7.13
7.13
7.12
7.11
7.11
7.11
7.10
7.10
7.09
H
N
O
Ph
35
0.98
1.99
2.00
0.99
2.00
1.99
1.00
f1 (ppm)

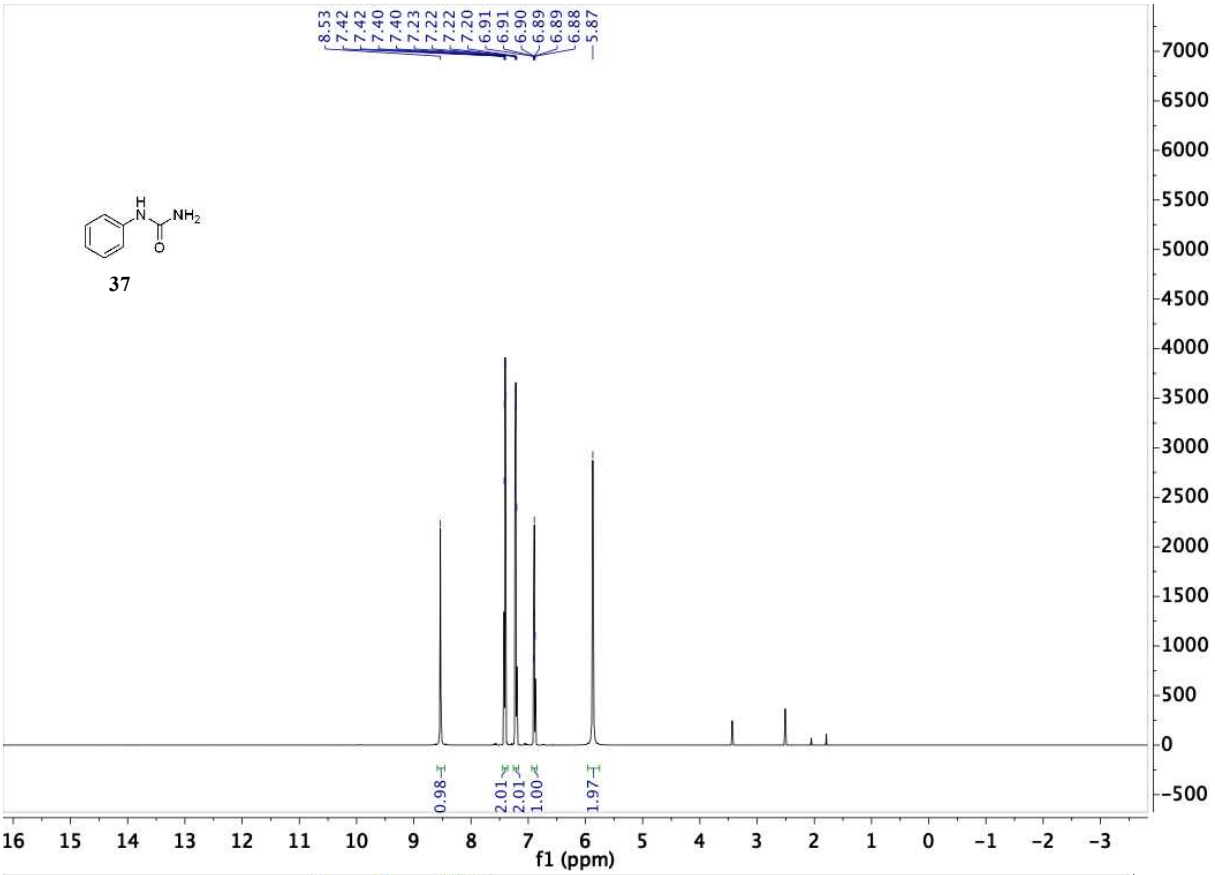

166.04
139.67
135.48
132.01
129.07
128.85
128.13
124.12
120.84
H
N
O
Ph
35
f1 (ppm)

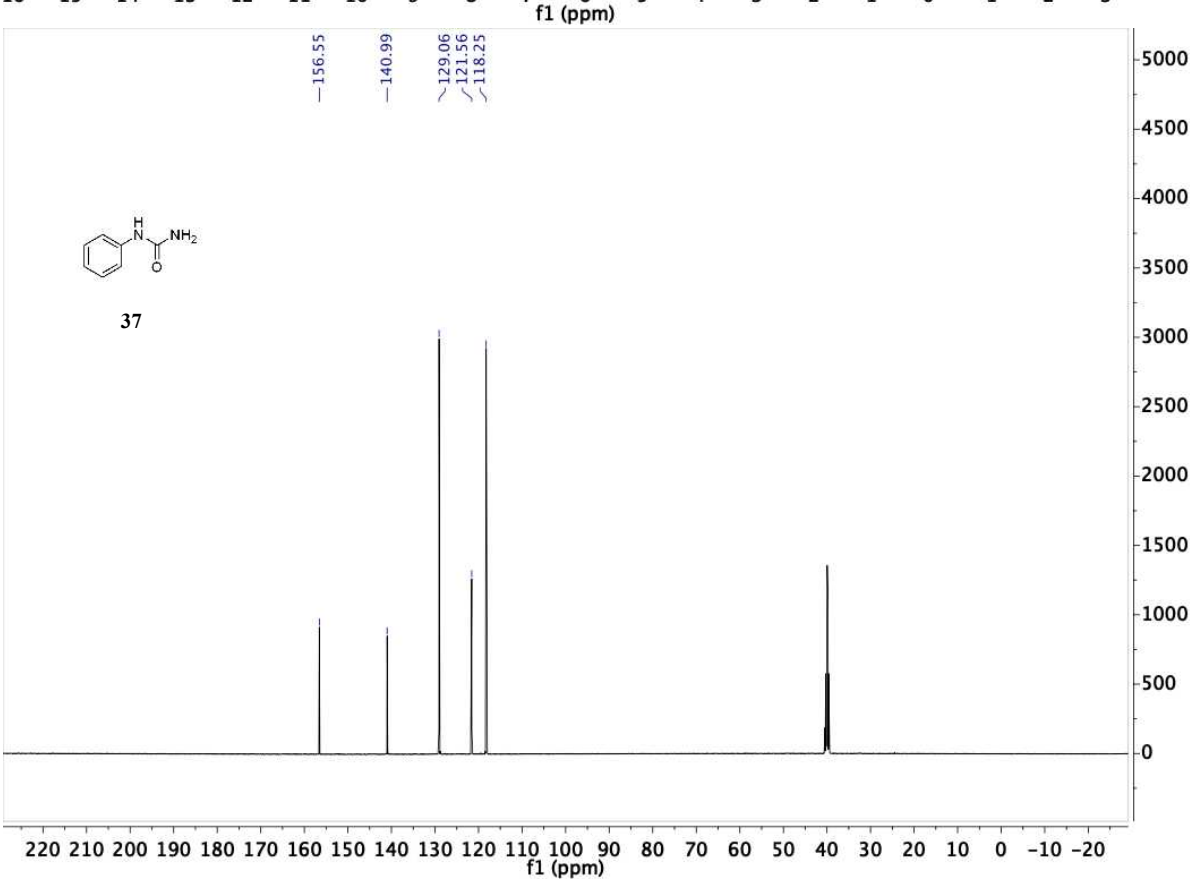

7.43
7.41
7.34
7.32
7.30
7.09
7.08
7.07
6.84
4.28
4.26
4.25
4.23
1.35
1.33
1.32
OEt
1.98
2.04
1.02
1.00
2.00
3.09
f1 (ppm)

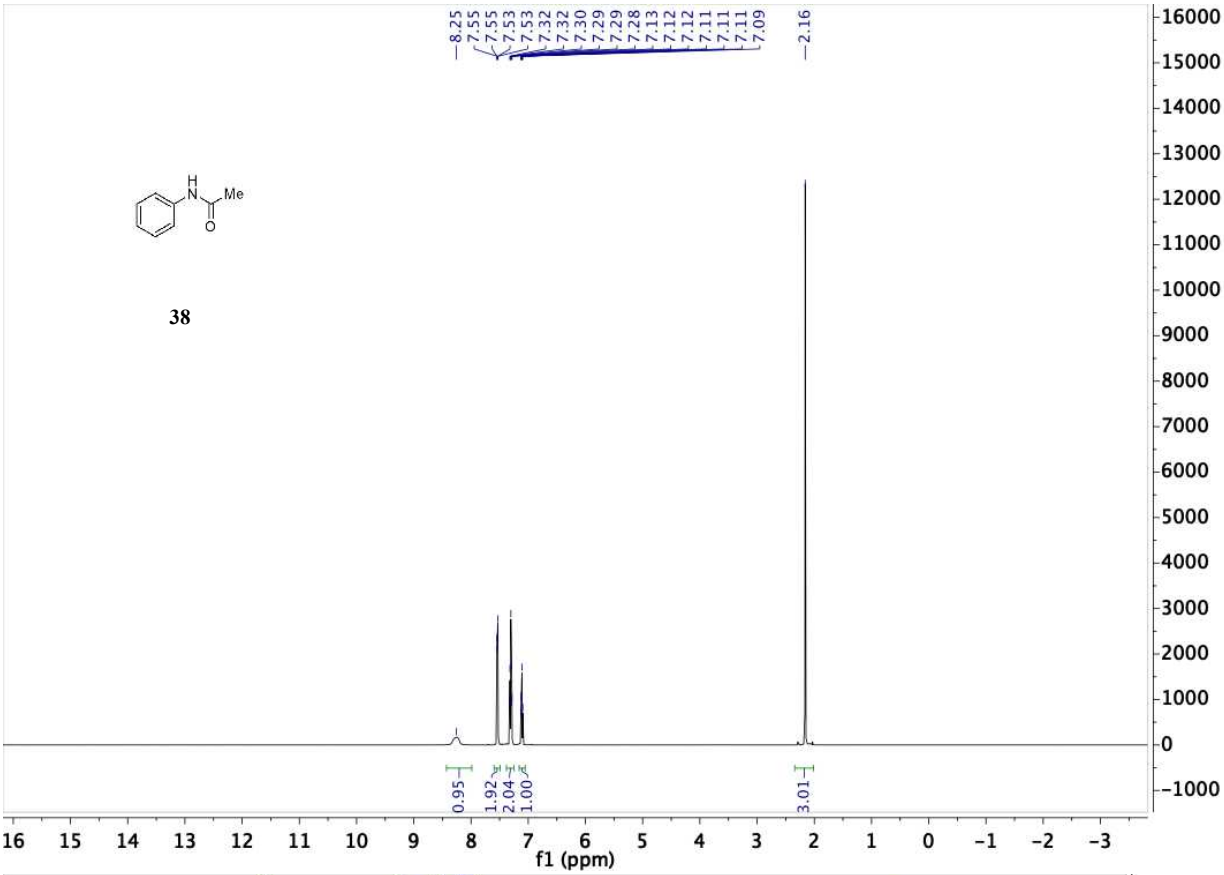
36


153.77
138.06
129.02
123.35
118.76
61.21
14.56
OEt
f1 (ppm)

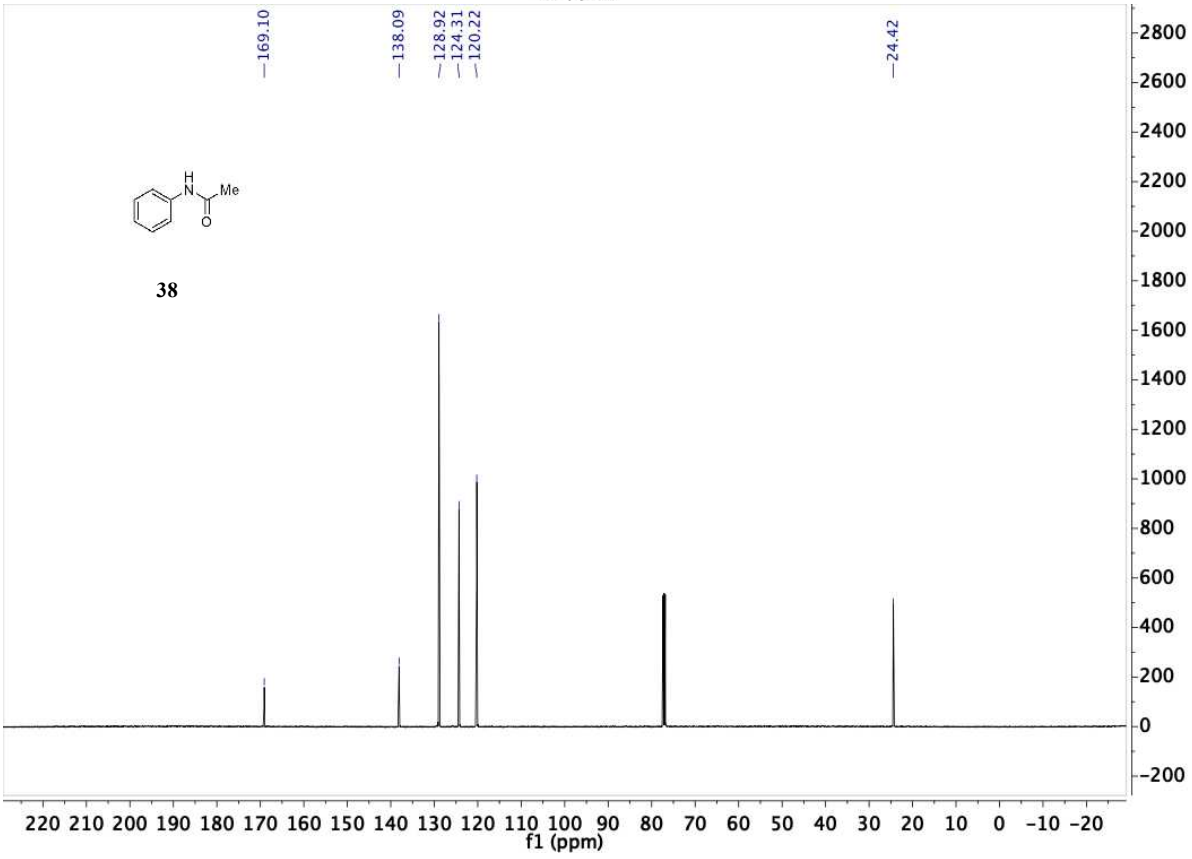
36


8.53
7.42
7.42
7.40
7.40
7.23
7.22
7.22
7.20
6.91
6.90
6.89
6.89
6.88
5.87
37
0.98
2.01
2.01
1.00
1.97
f1 (ppm)

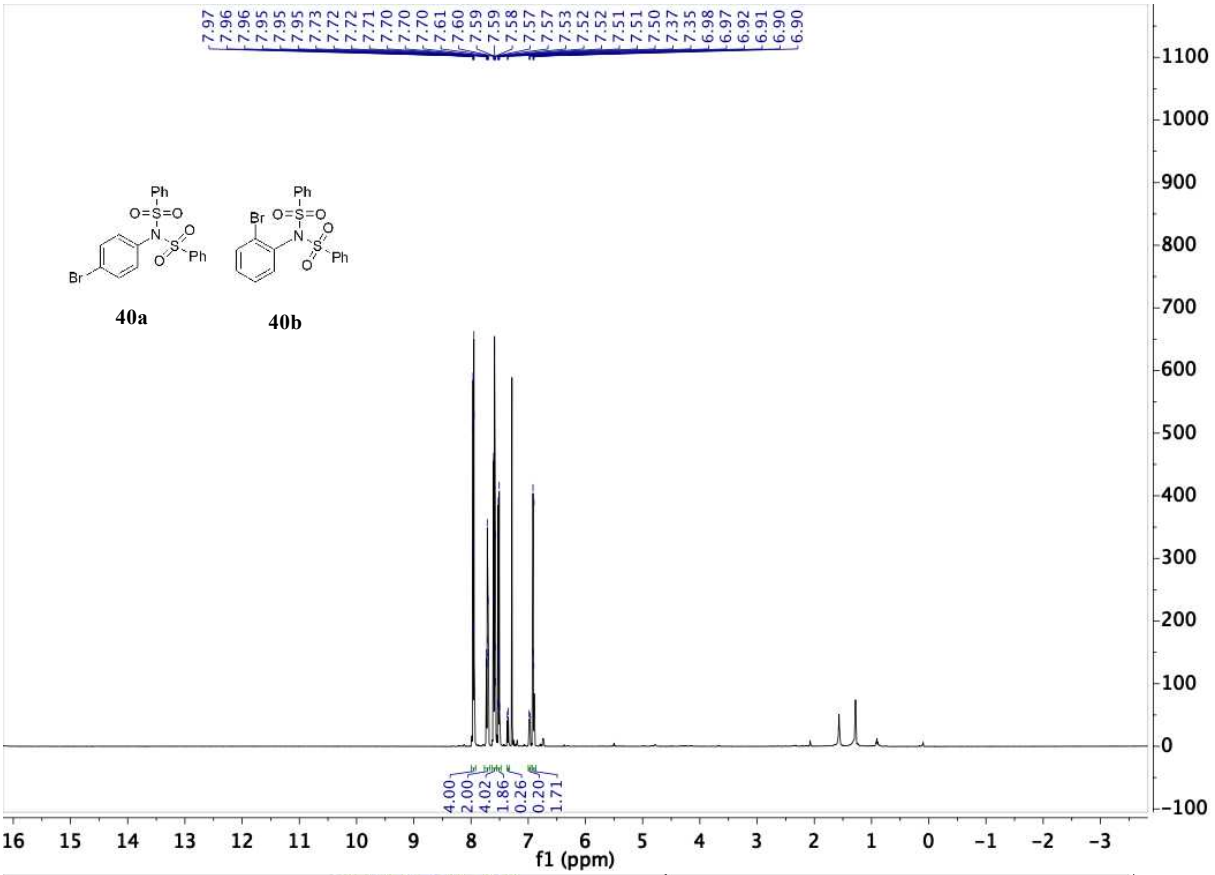

156.55
140.99
129.06
121.56
118.25
37
f1 (ppm)

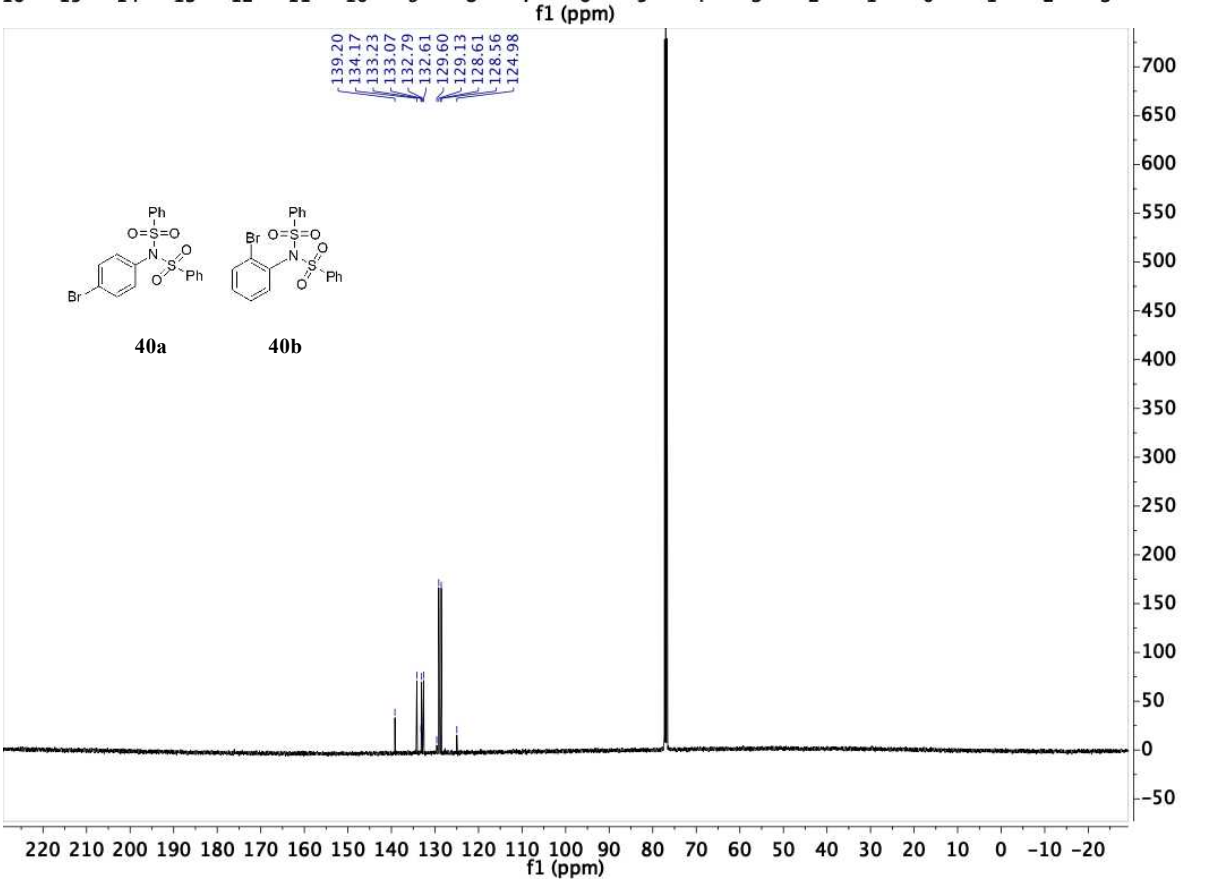

8.25
7.55
7.55
7.53
7.53
7.32
7.32
7.30
7.29
7.29
7.28
7.13
7.12
7.12
7.11
7.11
7.11
7.09
2.16
0.95
1.92
2.04
1.00
3.01
f1 (ppm)

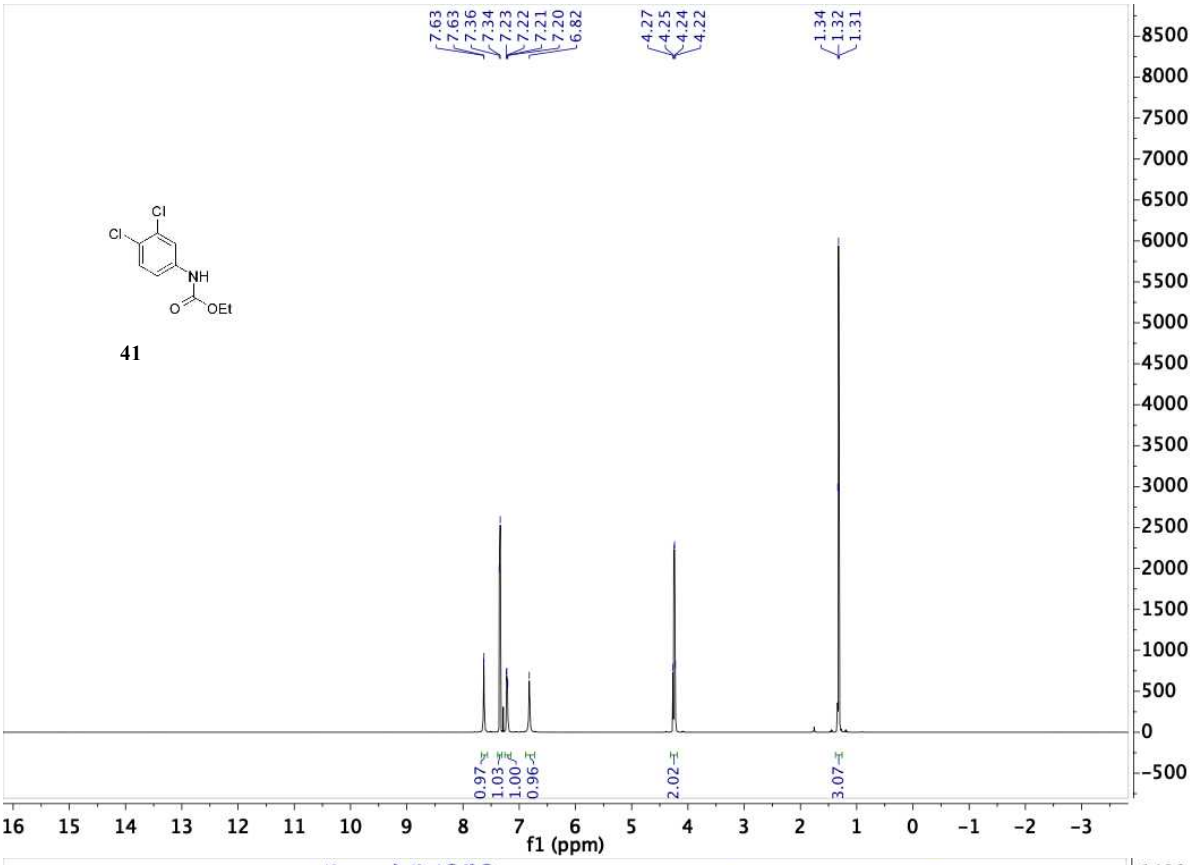
38


169.10
138.09
128.92
124.31
120.22
24.42
f1 (ppm)

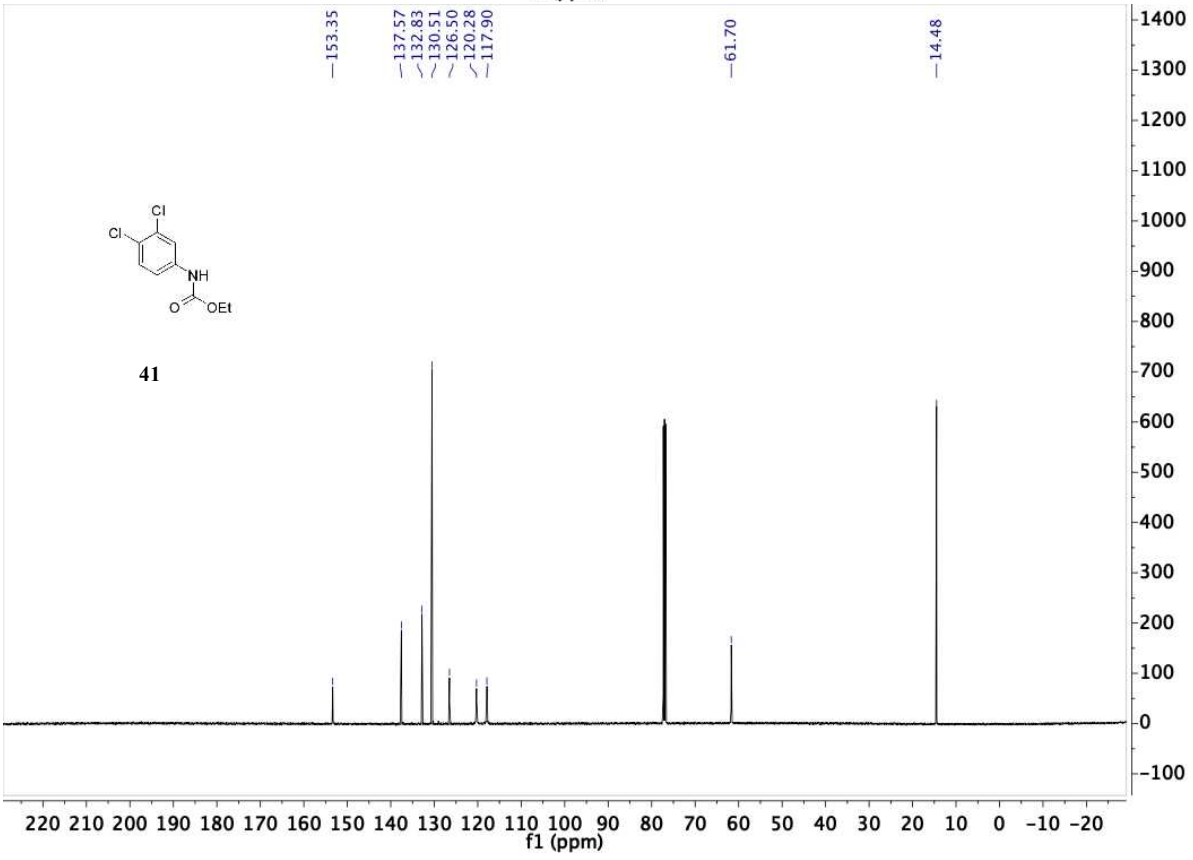
38


7.97
7.96
7.96
7.95
7.95
7.95
7.73
7.72
7.72
7.71
7.70
7.70
7.70
7.61
7.60
7.59
7.59
7.58
7.57
7.57
7.53
7.52
7.52
7.51
7.51
7.50
7.37
7.35
6.98
6.97
6.92
6.91
6.90
6.90
Ph
O=S=O
N
O=S=O
Ph
Br
40a
40b
4.00
2.00
4.02
1.86
0.26
0.20
1.71
f1 (ppm)

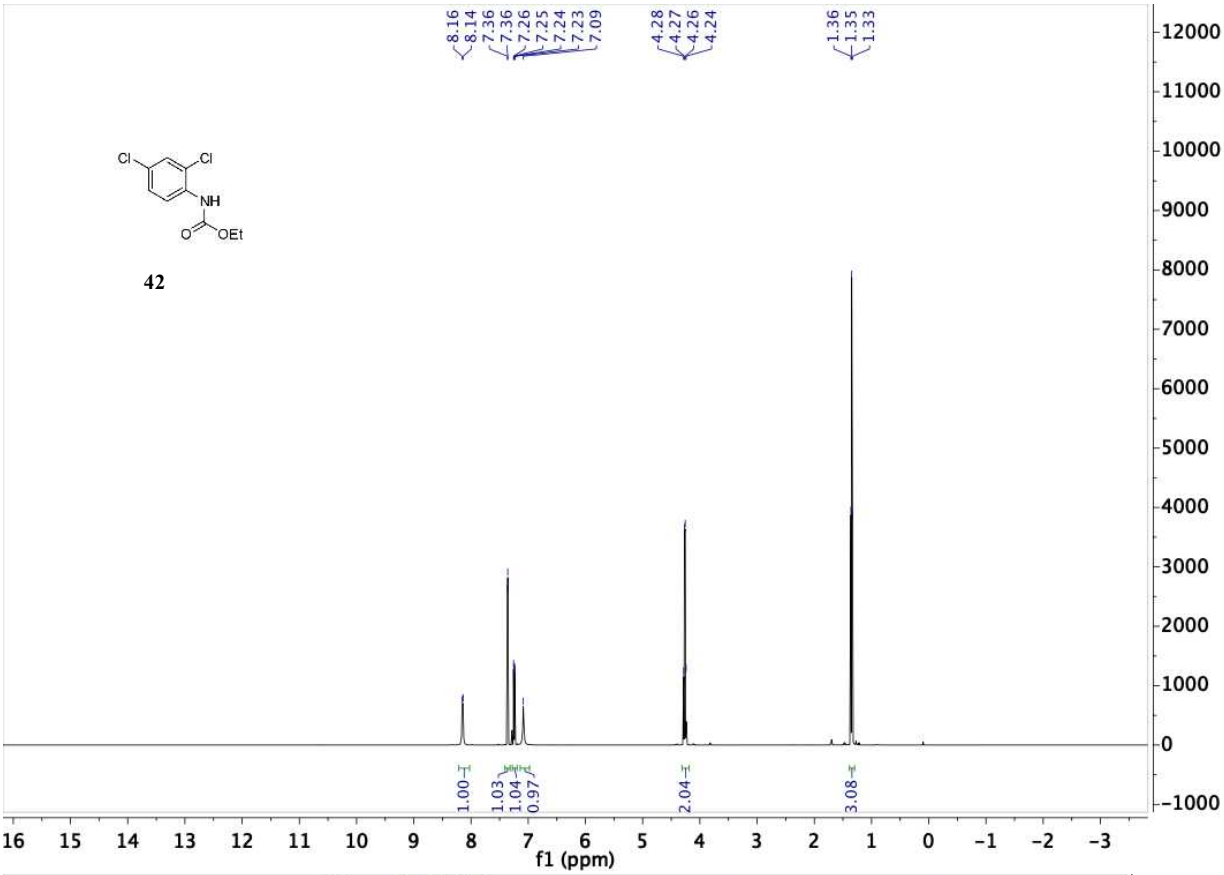

139.20
134.17
133.23
133.07
132.79
132.61
129.60
129.13
128.61
128.56
124.98
Ph
O=S=O
N
O=S=O
Ph
Br
40a
40b
f1 (ppm)

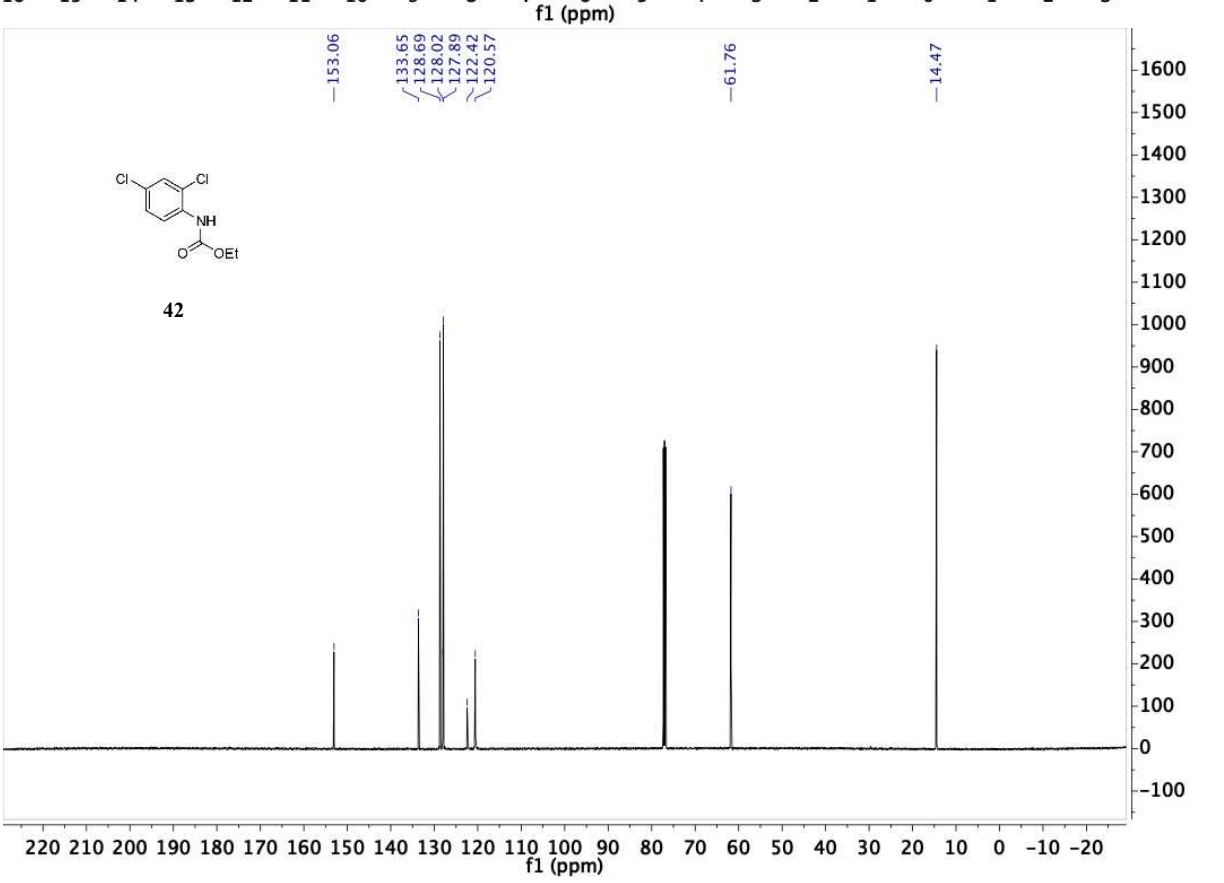

7.63
7.63
7.36
7.34
7.23
7.22
7.21
7.20
6.82
4.27
4.25
4.24
4.22
1.34
1.32
1.31
Cl
Cl
NH
O
OEt
0.97
1.03
1.00
0.96
2.02
3.07
f1 (ppm)

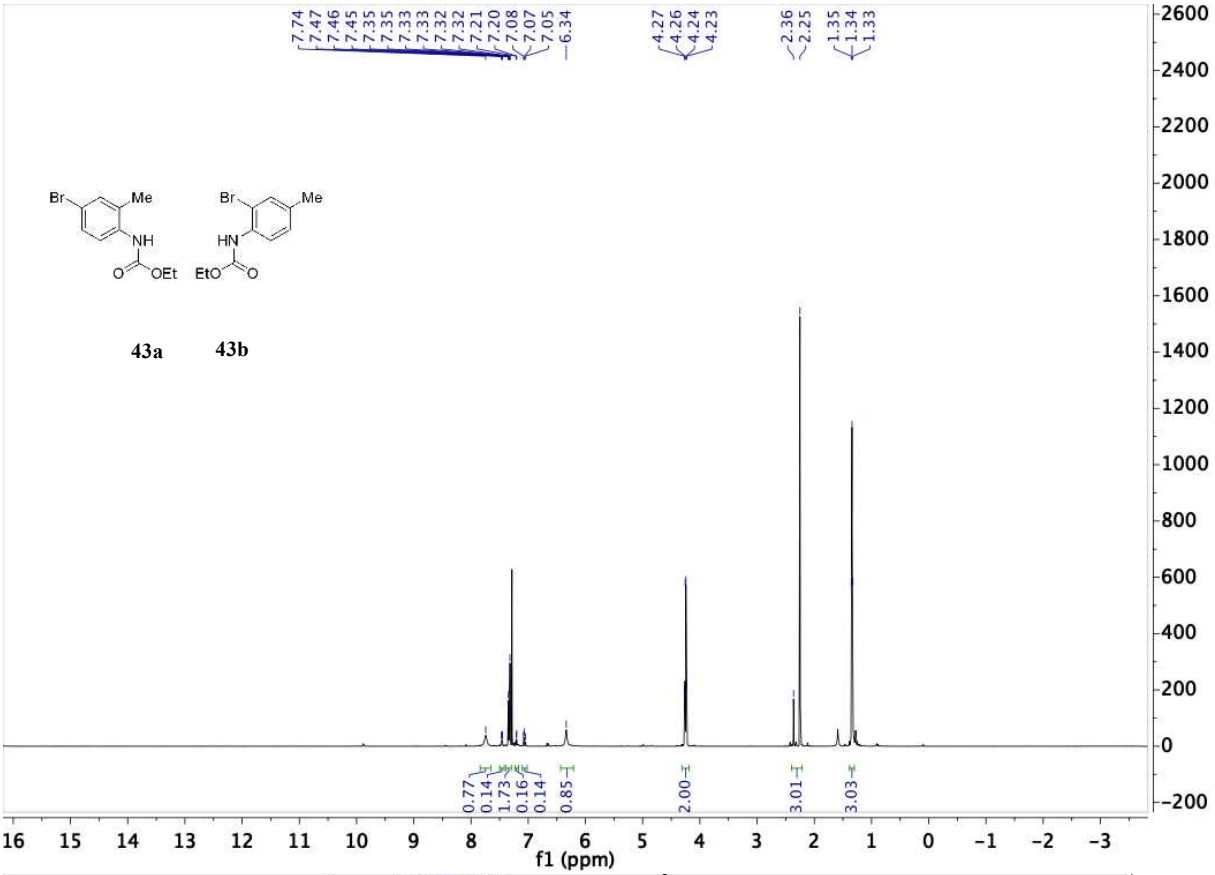
41

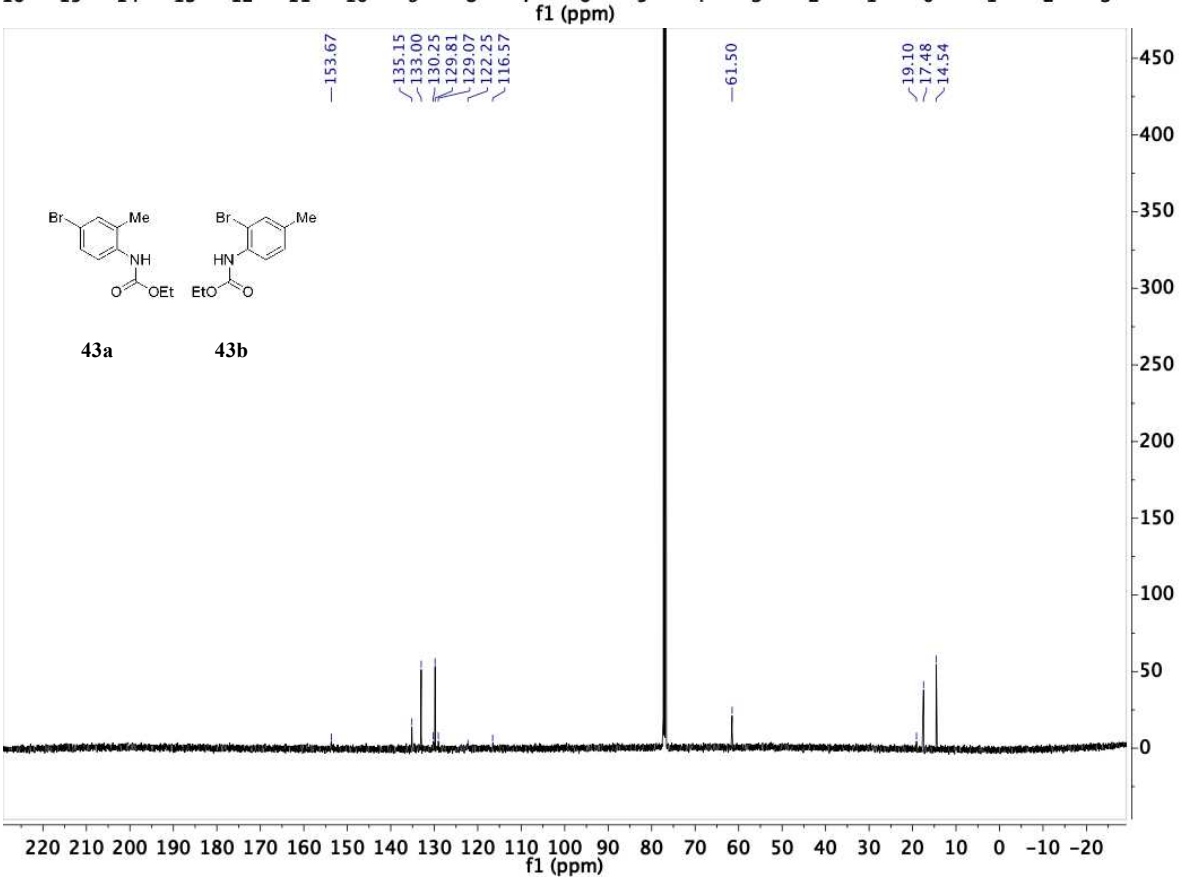

153.35
137.57
132.83
130.51
126.50
120.28
117.90
61.70
14.48
Cl
Cl
NH
O
OEt
f1 (ppm)

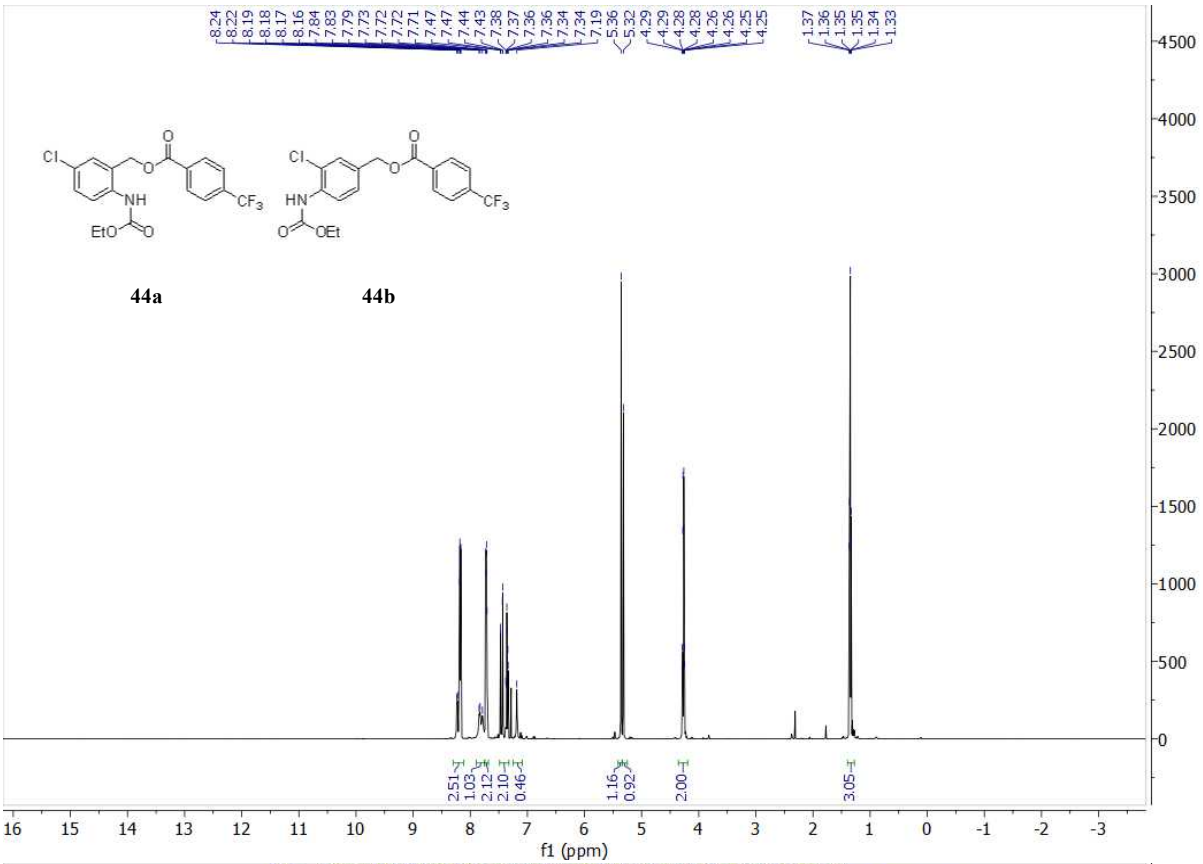
41

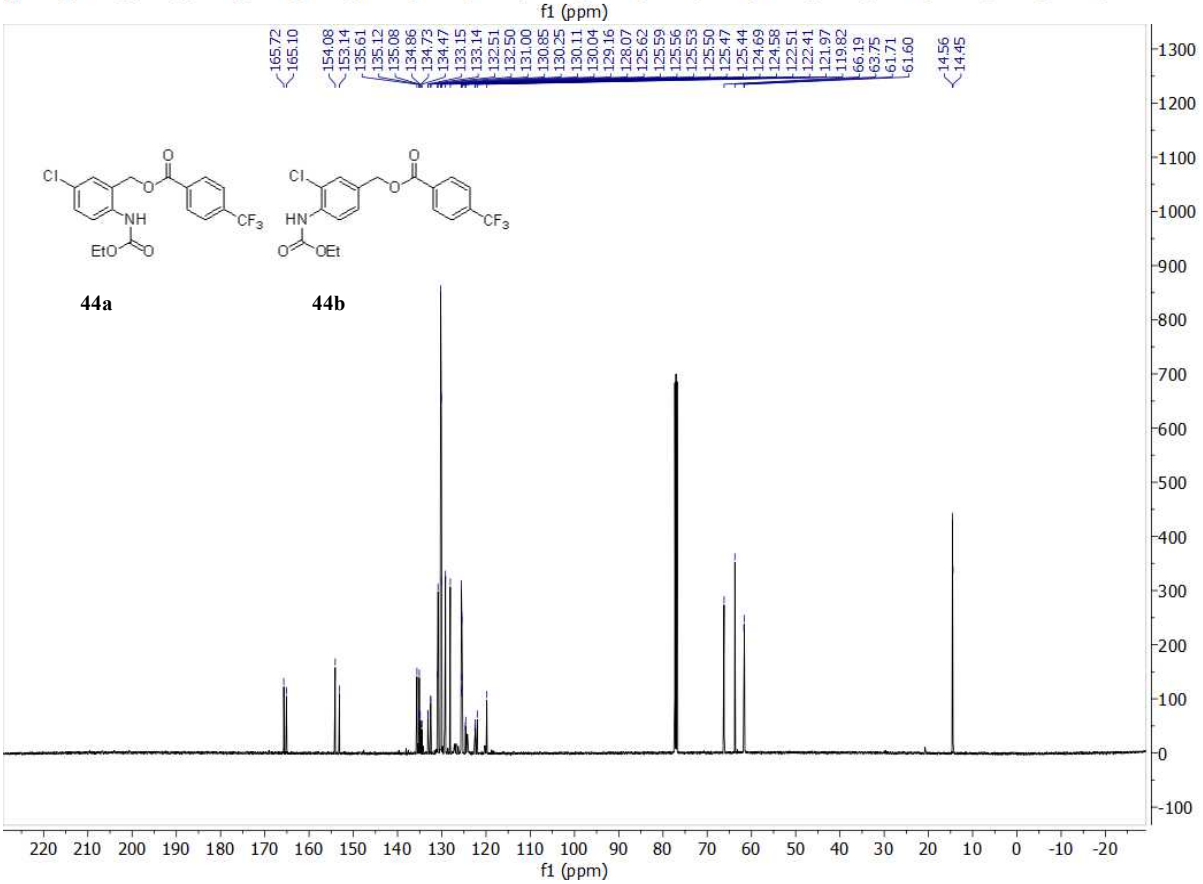

8.16
8.14
7.36
7.36
7.26
7.25
7.24
7.23
7.09
4.28
4.27
4.26
4.24
1.36
1.35
1.33
Cl
Cl
NH
O
OEt
42
1.00
1.03
1.04
0.97
2.04
3.08
f1 (ppm)


153.06
133.65
128.69
128.02
127.89
122.42
120.57
61.76
14.47
Cl
Cl
NH
O
OEt
42
f1 (ppm)

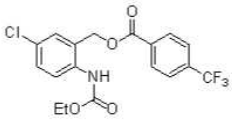

7.74
7.47
7.46
7.45
7.35
7.35
7.33
7.33
7.32
7.32
7.21
7.20
7.08
7.07
7.05
6.34
4.27
4.26
4.24
4.23
2.36
2.25
1.35
1.34
1.33
Br
Me
NH
O
OEt
Br
Me
HN
EtO
O
43a
43b
0.77
0.14
1.73
0.16
0.14
0.85
2.00
3.01
3.03
f1 (ppm)


153.67
135.15
133.00
130.25
129.81
129.07
122.25
116.57
61.50
19.10
17.48
14.54
Br
Me
NH
O
OEt
Br
Me
HN
EtO
O
43a
43b
f1 (ppm)

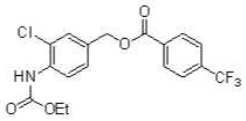

8.24
8.22
8.19
8.18
8.17
8.16
7.84
7.83
7.79
7.73
7.72
7.72
7.71
7.47
7.47
7.44
7.43
7.38
7.37
7.36
7.36
7.34
7.34
7.19
5.36
5.32
4.29
4.29
4.28
4.28
4.26
4.26
4.25
4.25
1.37
1.36
1.35
1.35
1.34
1.33
Cl
O
O
NH
EtO
O
CF3
Cl
HN
O
OEt
CF3
44a
44b
2.51
1.03
2.12
2.10
0.46
1.16
0.92
2.00
3.05
f1 (ppm)


165.72
165.10
154.08
153.14
135.61
135.12
135.08
134.86
134.73
134.47
133.15
133.14
132.51
132.50
131.00
130.85
130.25
130.11
130.04
129.16
128.07
125.62
125.59
125.56
125.53
125.50
125.47
125.44
124.69
124.58
122.51
122.41
121.97
119.82
66.19
63.75
61.71
61.60
14.56
14.45
Cl
O
O
NH
EtO
O
CF3
Cl
HN
O
OEt
CF3
44a
44b
f1 (ppm)

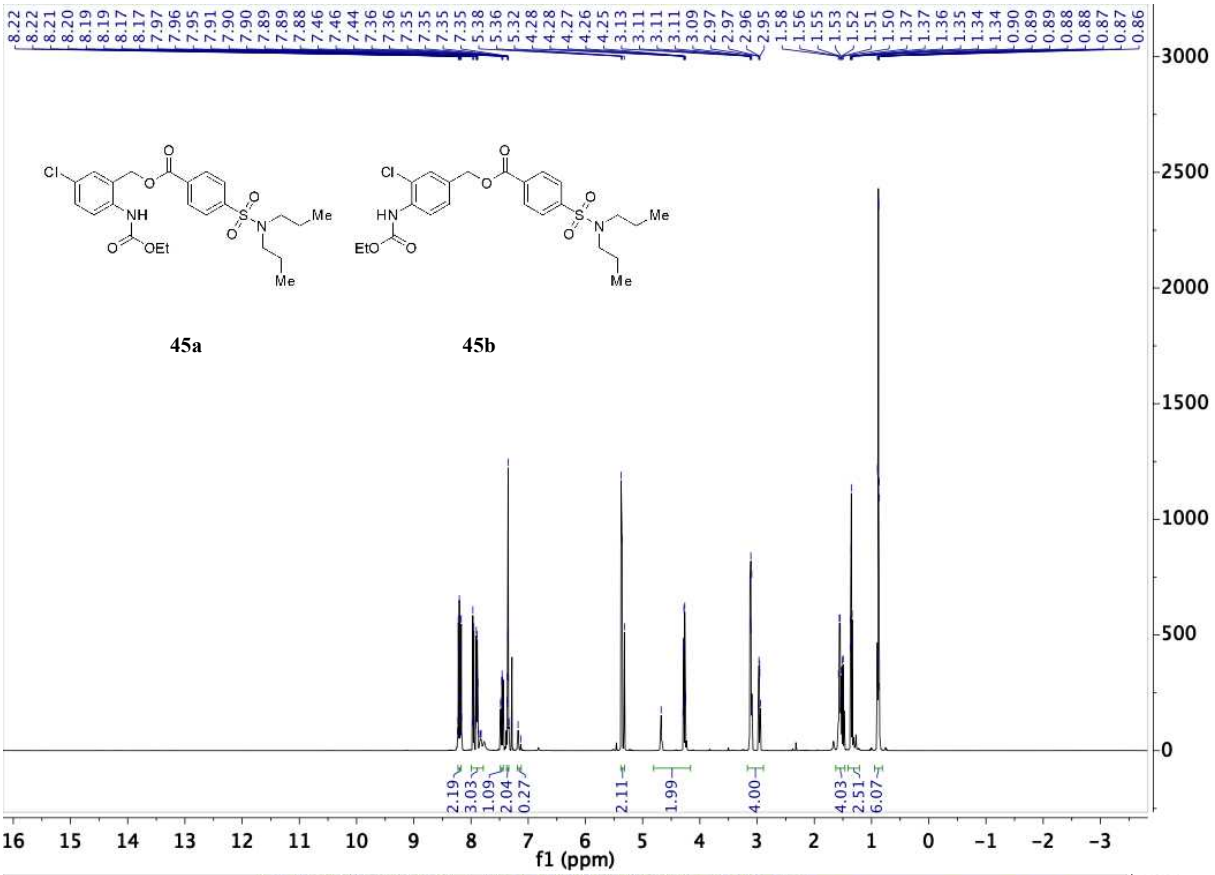
^{19}F NMR (470 MHz)

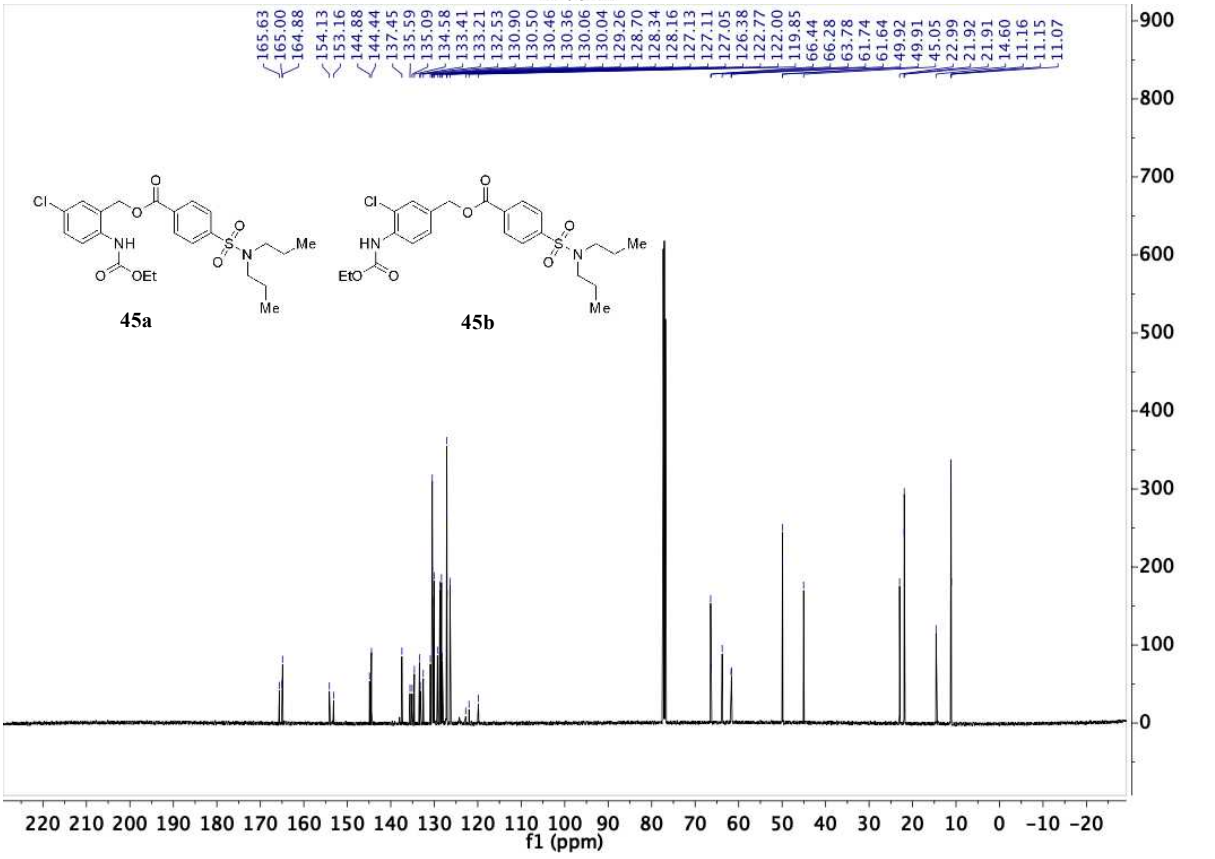


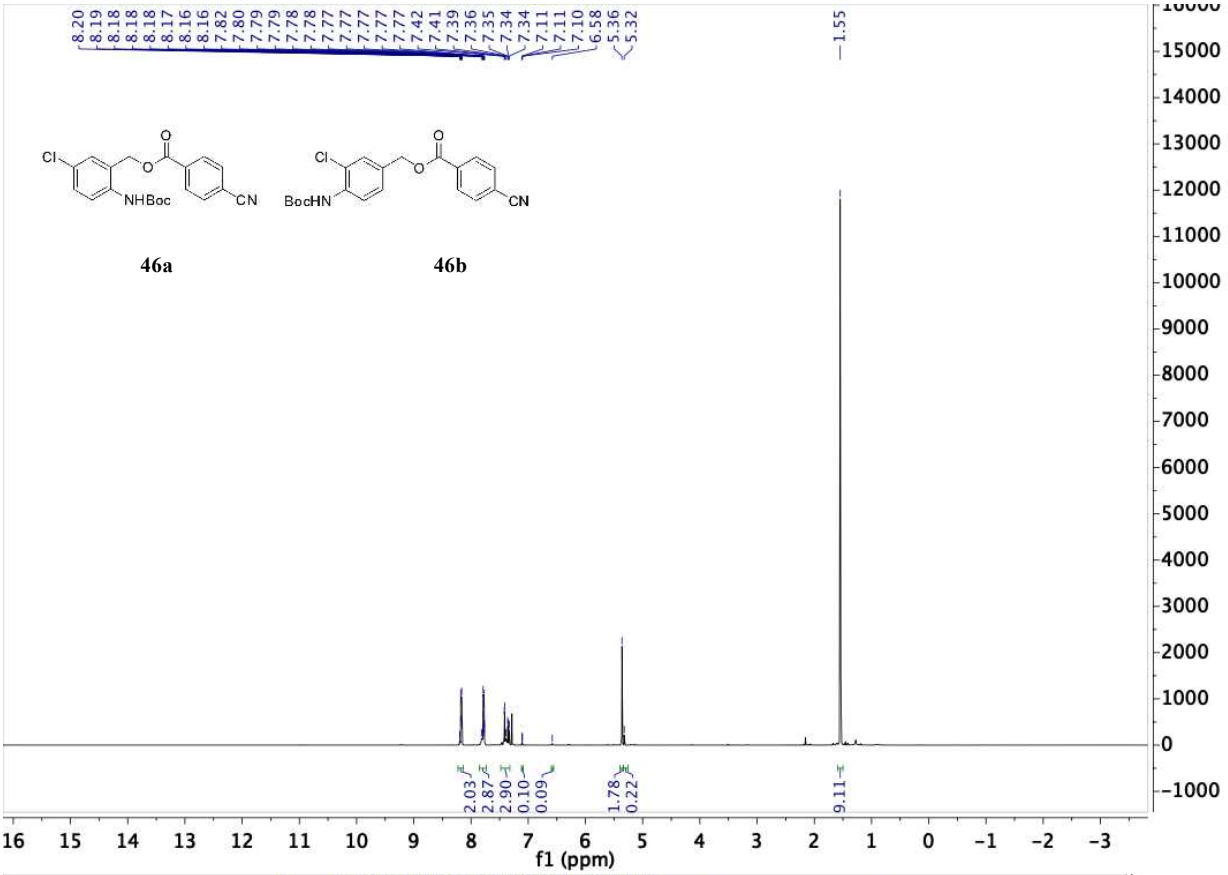
44a

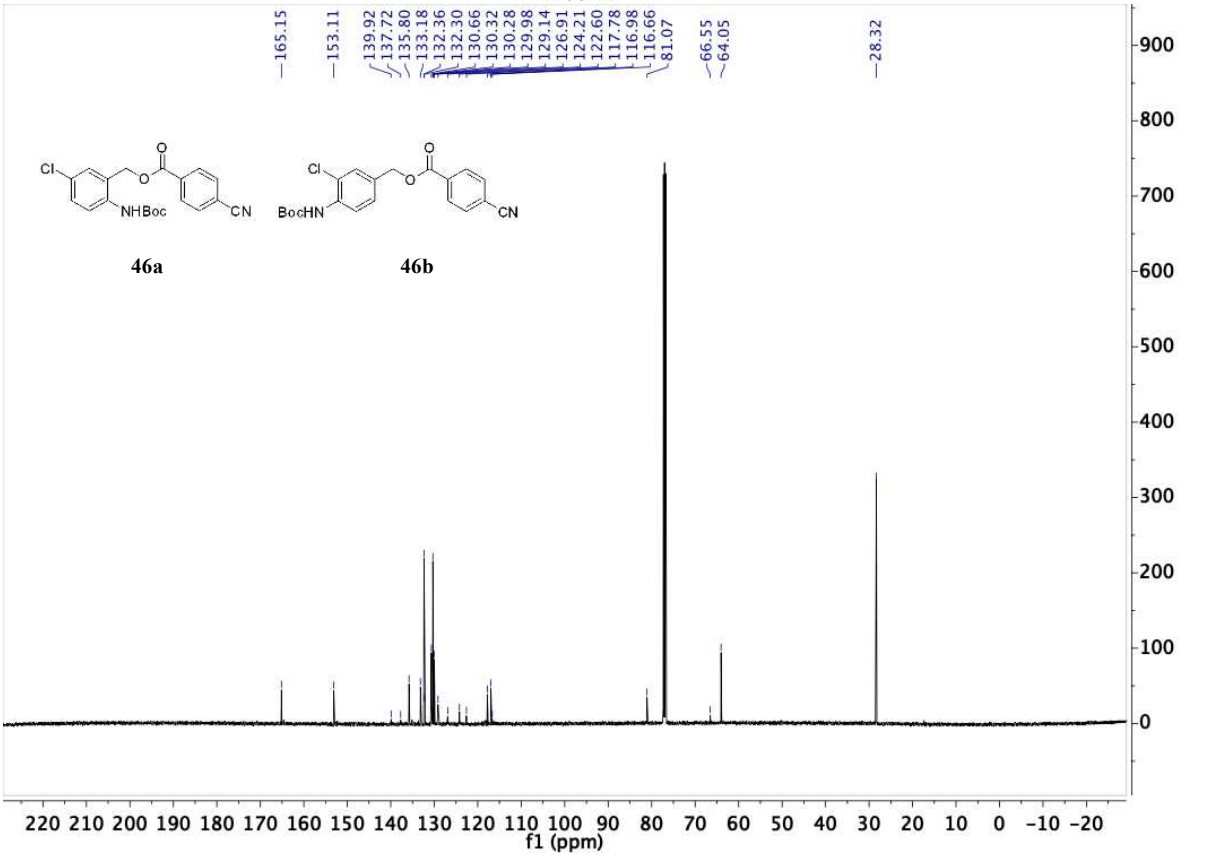


44b


45a
45b
f1 (ppm)


45a
45b
f1 (ppm)


8.20
8.19
8.18
8.18
8.18
8.17
8.16
8.16
7.82
7.80
7.79
7.79
7.78
7.78
7.77
7.77
7.77
7.77
7.77
7.42
7.41
7.39
7.36
7.35
7.34
7.34
7.11
7.11
7.10
6.58
5.36
5.32
1.55
Cl
O
O
NHBoc
CN
46a
Cl
O
O
BocHN
CN
46b
2.03
2.87
2.90
0.10
0.09
1.78
0.22
9.11
f1 (ppm)


165.15
153.11
139.92
137.72
135.80
133.18
132.36
132.30
130.66
130.32
130.28
129.98
129.14
126.91
124.21
122.60
117.78
116.98
116.66
81.07
66.55
64.05
28.32
Cl
O
O
NHBoc
CN
46a
Cl
O
O
BocHN
CN
46b
f1 (ppm)