Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Trimerization of Enones under Air Enabled by NHC/NaO*t*Bu via a SET Radical Pathway

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Supporting Information

Contents

I: General Information

Commercially available materials purchased from Alfa Aesar, Merk or Aldrich were used as received. Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on a Bruker AV400 (400 MHz) spectrometer. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00). ^{1}H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted are designated as multiplet (m) or broad (br). Carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker AV400 (100 MHz) spectrometer. High resolution mass spectral analysis (HRMS) was performed on Waters Q-TOF Premier mass spectrometer. IR spectra were recorded on a Shimadzu IRPrestige-21 FT-IR spectrometer as neat thinfilms between NaCl plates in case of liquids and as KBr pellets in the case of solids. LC-MS were recorded on ThermoFinnigan LCQ Fleet MS. Melting points were measured on SRS Optimelt Automated Point System SRS. MPA100. Flash chromatography was performed using Merck silica gel 60 with distilled solvents. Analytical thin-layer chromatography (TLC) was carried out on Merck 60 F254 pre-coated silica gel plate (0.2 mm thickness). Visualization was performed using a UV lamp.

The X-ray crystallographic coordinates for structures reported in this article have been deposited at the Cambridge Crystallographic Data Centre (CCDC), under deposition numbers CCDC 1443861 (**2a**), CCDC 1443862 (**3a**).

II: General Procedure for Table 1(entry 2) and Table 2

1: General procedures for Table 1 (entry 2)

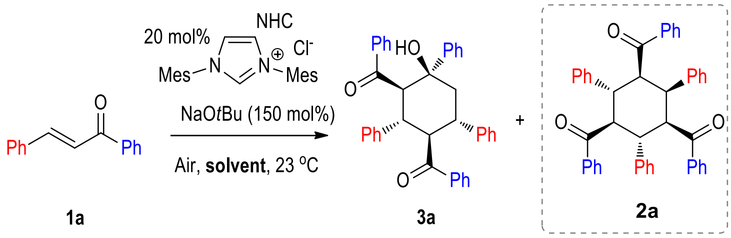
To a 4 mL sample vial equipped with a magnetic stir bar, was added imidazolium-based NHC (**L2**) (6.8 mg, 20 mol %), NaO*t*Bu (15.0 mg, 1.5 equiv) and chalcone **1a** (0.1 mmol). The vial was closed with a plastic stopper after addition of 1.5 mL diethyl ether (purchased from Merk for analysis, used without further purification). The resulting mixture was stirred at room temperature. After the consumption of chalcone **1a** montiored by TLC, solvent was evaporated and the reaction mixture was then applied to silica gel chromatography (hexane/ethyl acetate = 10:1) to obtain product **3a**.

2: General procedures for Table 2

To a 8 mL sample vial equipped with a magnetic stir bar, was added imidazolium-based NHC (**L2**) (13.6 mg, 20 mol %), NaO*t*Bu (29.0 mg, 1.5 equiv) and enone **1** (0.2 mmol). The vial was closed with a plastic stopper after addition of 3.0 mL diethyl ether (purchased from Merk for analysis, used without further purification). In some cases, 200 μL THF was added in order to increase the solubility of enones. The resulting mixture was stirred at room temperature. After the consumption of enone **1** montiored by TLC, solvent was evaporated and the reaction mixture was then applied to silica gel chromatography to obtain product **3**.

III: Summary of Condition Optimization

Table S1. Solvent screening for trimerization of chalcone (**1a**)[a]

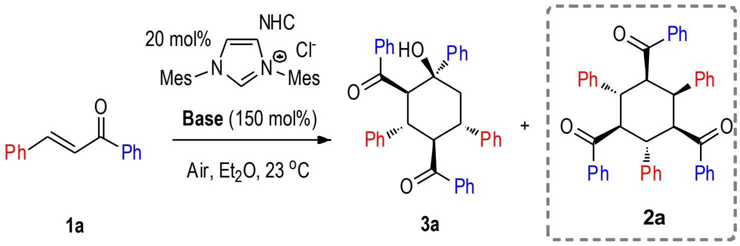


Entry	Solvent	*t* (h)	Yield of **3a** (%)[b]	Yield of **2a** (%)[b]
1	Et_2O	18	80	11
2	toluene	36	65	trace
3	CH_3CN	24	71	trace
4	1,4-dioxane	24	57	20
5	THF	24	55	4
6	hexane	24	45	30
7	DCM	24	29	20

[a] Reaction conditions: NHC (20 mol %), NaO*t*Bu (1.5 equiv.), **1a** (0.1 mmol) and solvent (1.5 mL).

[b] Isolated yield.

Table S2. Base screening for trimerization of chalcone (**1a**)[a]

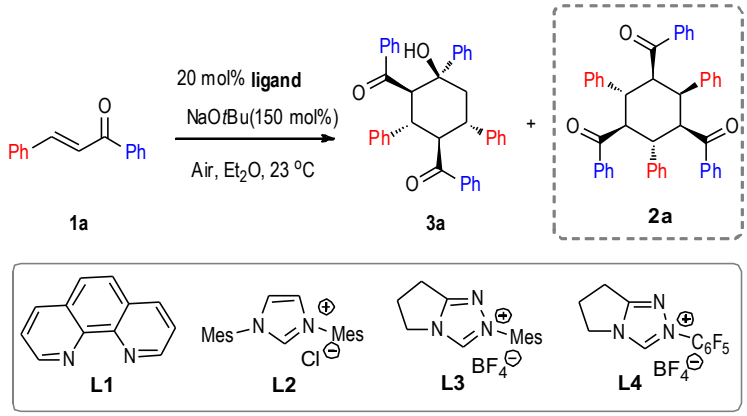


Entry	Base	*t* (h)	Yield of **3a** (%)[b]	Yield of **2a** (%)[b]
1	NaO*t*Bu	18	80	11
2	KO*t*Bu	48	30	17
3[c]	$Mg(OtBu)_2$	48	0	0
4[c]	$Al(OtBu)_3$	48	0	0
5[c]	NaOH	48	0	0
6[c]	NaOMe	48	0	0
7[c]	KOMe	48	0	0
8[c]	K_2CO_3	48	0	0
9[c]	DBU	48	0	0
10[c]	Et_3N	48	0	0

[a] Reaction conditions: NHC (20 mol %), base (1.5 equiv.), **1a** (0.1 mmol) and Et_2O (1.5 mL).

[b] Isolated yield. [c] No reaction.

Table S3. Ligand or additive screening for trimerization of chalcone (**1a**) [a]

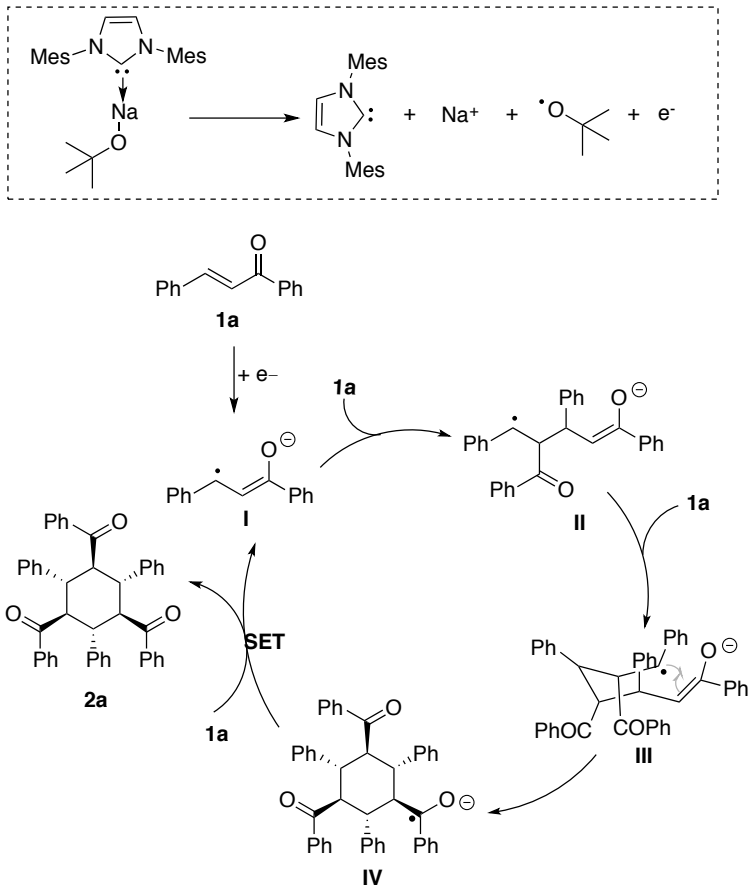


Entry	Ligand	*t* (h)	Yield of **3a** (%)[b]	Yield of **2a** (%)[b]
1	**L1**	24	59	14
2	**L2**	18	80	11
3	**L3**	24	24	14
4	**L4**	24	0	19

[a] Reaction conditions: ligand (20 mol %), NaO*t*Bu (1.5 equiv.), **1a** (0.1 mmol) and Et_2O (1.5 mL).

[b] Isolated yield.

IV: Proposed reaction pathway to 2a



Scheme S1. Proposed reaction pathway to **2a**

A plausible mechanism for **2a** is depicted in Scheme S1. Initially, one electron reduction of chalcone **1a** by NHC/NaO*t*Bu gives radical anion **I**, which then adds to another molecule of **1a** to afford radical anion intermediate **II**. Intermediate **II** reacts with the third molecule of **1a** furtherly to lead to radical anion **III**, which undergoes 6-exo-trig radical cyclization to generate intermediate **IV**. Finally, single-electron transfer from **IV** to **1a** to produce **2a** with the generation of intermediate **I**.

V: Cyclic voltammograms of 1a and 1n

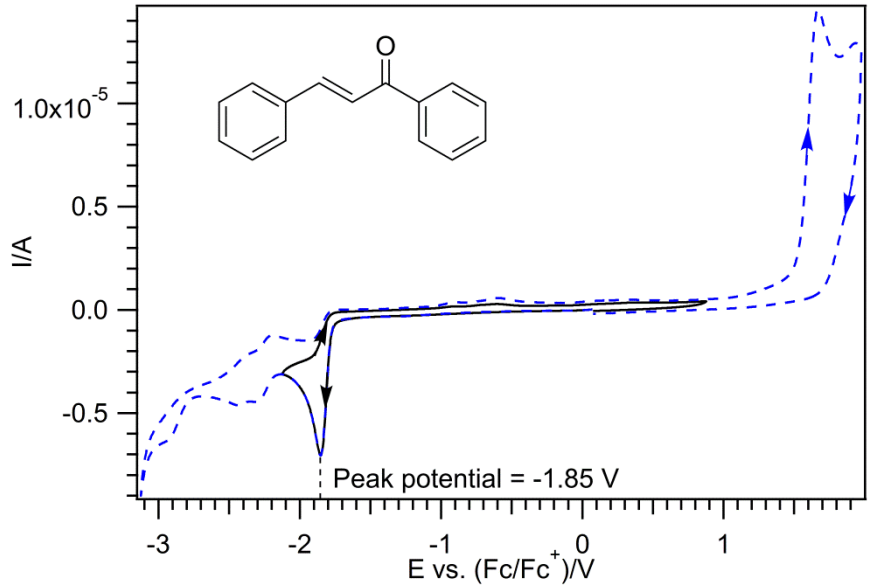


Figure S1. Cyclic voltammograms of 5 mM of analyte recorded at a 1 mm diameter planar circular glassy carbon electrode in acetonitrile containing 0.2 M *n*-Bu_4NPF_6 as the supporting electrolyte, at a scan rate of 0.1 mV s^{-1} and at 22 ± 2 ^oC.

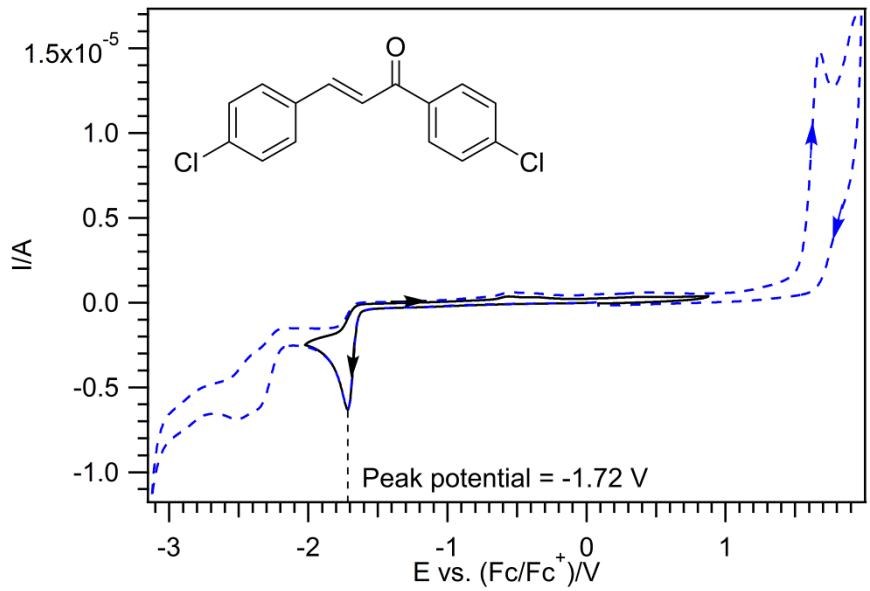
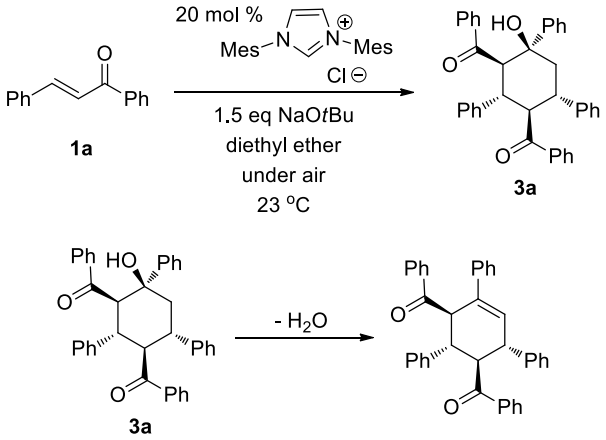
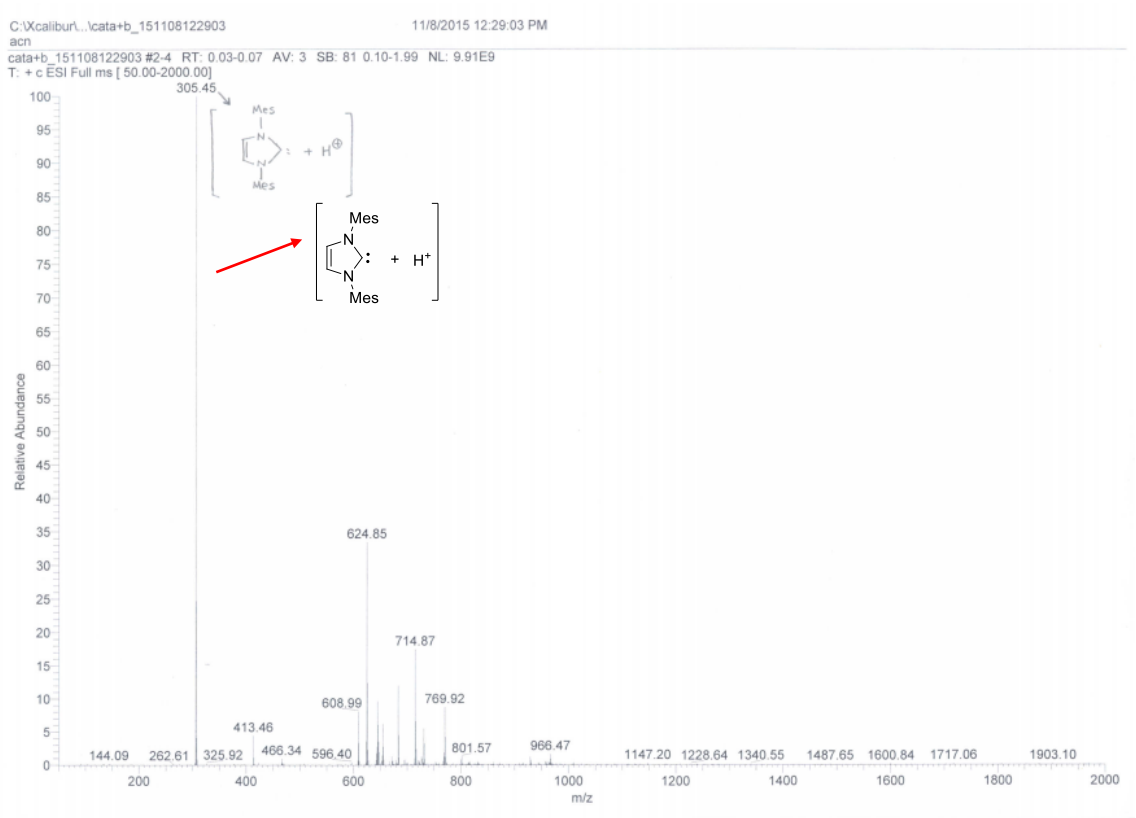


Figure S2. Cyclic voltammograms of 5 mM of analyte recorded at a 1 mm diameter planar circular glassy carbon electrode in acetonitrile containing 0.2 M *n*-Bu_4NPF_6 as the supporting electrolyte, at a scan rate of 0.1 mV s^{-1} and at 22 ± 2 ^oC.

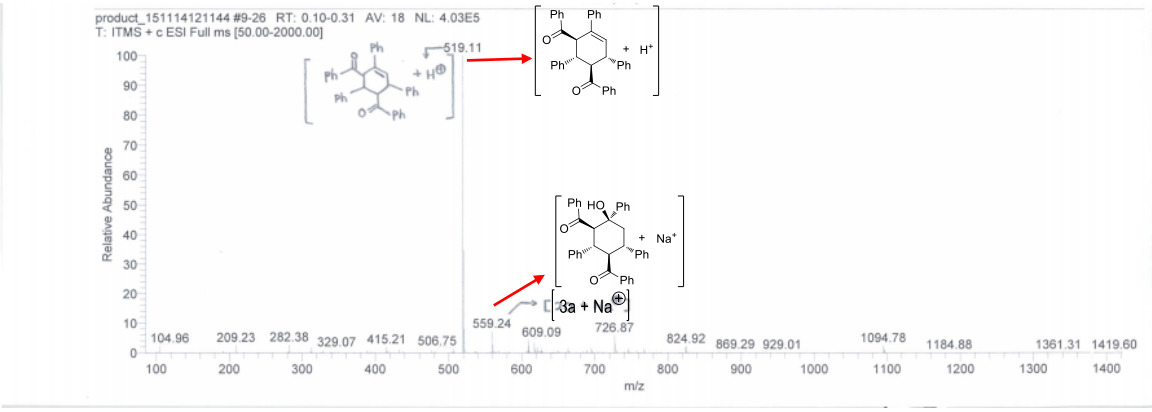
VI: Mechanistic Study with LC-MS



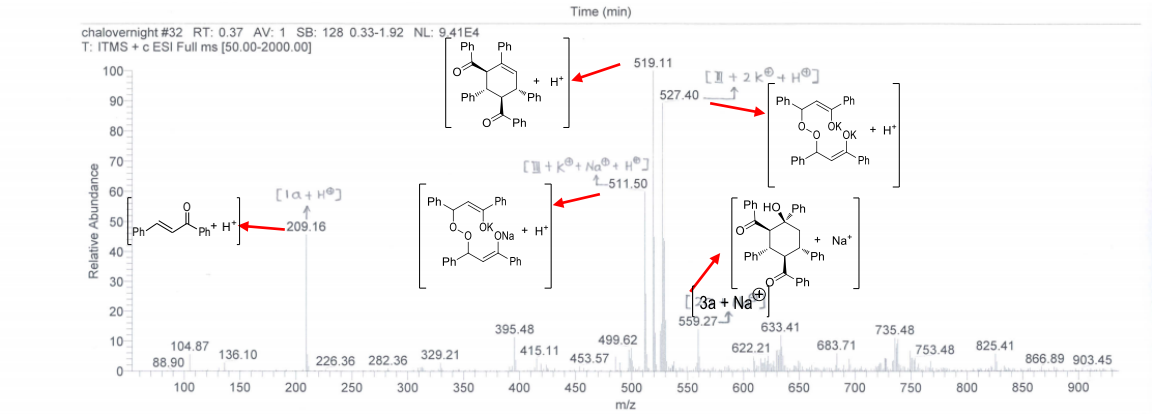
1. 20 mol% NHC precursor L2 and 1.5 equiv. NaO*t*Bu in diethyl ether stirred for 1h



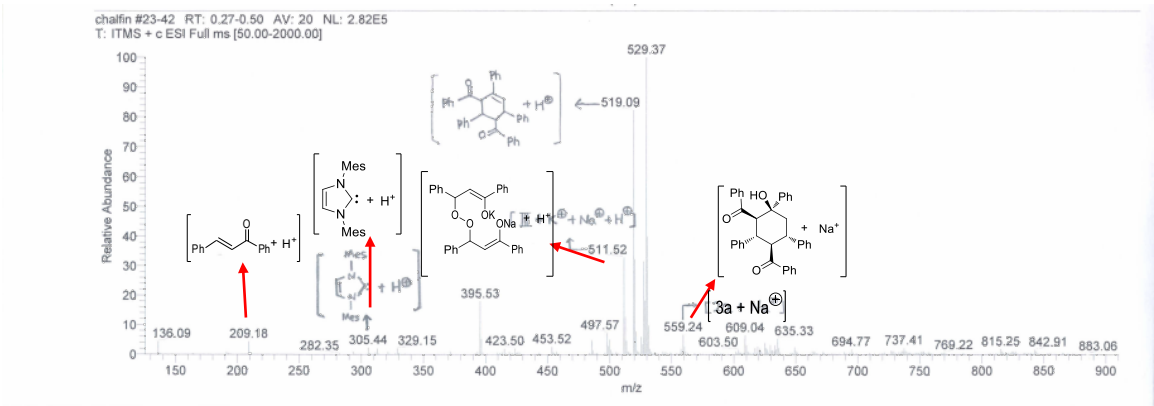
2. product 3a



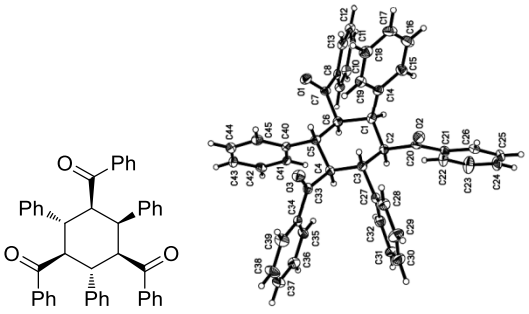
3. chalcone 1a, 20 mol% NHC precursor L2 and 1.5 equiv. of NaO*t*Bu in diethyl ether stirred for 8 h



4. chalcone 1a, 20 mol% NHC precursor L2 and 1.5 equiv. of NaO*t*Bu in diethyl ether stirred for 20 h



VII: Characterization of Products



2a

2,4,6-triphenylcyclohexane-1,3,5-triyl)tris(phenylmethanone)

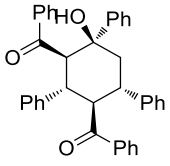
^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.65 – 7.62 (m, 4H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.38 – 7.30 (m, 8H), 7.20 – 7.12 (m, 4H), 7.10 – 6.94 (m, 10H), 6.86 (t, *J* = 7.2 Hz, 2H), 4.77 (dd, *J* = 12.0, 5.2 Hz, 2H), 4.45 (t, *J* = 12.0 Hz, 2H), 4.19 (t, *J* = 11.6 Hz, 1H), 3.99 (t, *J* = 5.2 Hz, 1H);

^{13}C NMR: (100 MHz, $CDCl_3$) δ 204.90, 197.71, 141.36, 138.78, 137.30, 135.88, 132.68, 131.74, 130.31, 128.59, 128.32, 128.21, 128.10, 127.68, 127.63, 127.43, 127.22, 126.63, 58.20, 53.55, 47.30, 43.37.

HRMS: (ESI) $[M+H]^+$ calcd. for $C_{45}H_{37}O_3$, 625.2743 found, 625.2747;

IR (KBr): ν_{max} 3024, 2916, 1681 (C=O), 1667 (C=O), 1589, 1450, 1265, 987, 694 cm^{-1}

mp 302.9-303.4 °C



3a

(4-hydroxy-2,4,6-triphenylcyclohexane-1,3-diyl)bis(phenylmethanone)

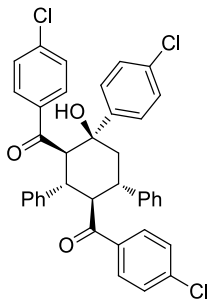
^{1}H NMR: (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.2 Hz, 2H), 7.29 – 6.95 (m, 20H), 6.83 (t, *J* = 7.6 Hz, 2H), 6.71 (t, *J* = 7.2 Hz, 1H), 5.39 (d, *J* = 2.4 Hz, 1H), 4.49 (d, *J* = 11.2 Hz, 1H), 4.26 – 4.14 (m, 2H), 4.10 – 4.02 (m, 1H), 2.56 – 2.48 (m, 1H), 2.26 (dd, *J* = 14.0, 3.6 Hz, 1H);

^{13}C NMR: (100 MHz, $CDCl_3$) δ 207.17, 203.58, 145.88, 142.07, 138.96, 138.61, 138.12, 132.65, 131.76, 128.35, 128.16, 128.06, 127.97, 127.69, 127.66, 127.54, 127.36, 126.98, 126.90, 126.69, 124.80, 75.33, 56.74, 56.72, 48.06, 45.83, 43.34.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{32}O_3Na$, 559.2249 found 559.2252;

IR (KBr): ν_{max} 3402 (OH), 1666 (C=O), 1643 (C=O), 1597, 1439, 1257, 1026, 802, 694 cm^{-1}

mp 244.2-244.7 °C



3b

(4-(4-chlorophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone)

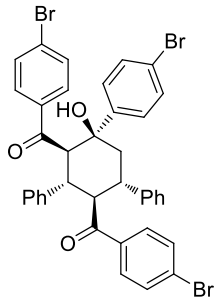
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.8 Hz, 2H), 7.23 (t, *J* = 8.4 Hz, 4H), 7.17 (d, *J* = 8.4 Hz, 4H), 7.11 (t, *J* = 7.6 Hz, 4H), 7.07 – 6.99 (m, 5H), 6.87 (t, *J* = 7.6 Hz, 2H), 6.78 (t, *J* = 7.6 Hz, 1H), 5.36 (d, *J* = 2.4 Hz, 1H), 4.40 – 4.35 (m, 1H), 4.18 – 4.10 (m, 2H), 4.05 – 3.99 (m, 1H), 2.47 – 2.39 (m, 1H), 2.22 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.48, 202.13, 144.40, 141.57, 139.68, 138.35, 138.22, 136.98, 135.95, 133.02, 129.07, 128.72, 128.53, 128.41, 128.35, 128.25, 127.95, 127.81, 127.33, 127.02, 126.22, 75.09, 56.51, 56.30, 47.96, 45.65, 43.25.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{29}O_3NaCl_3$, 661.1080 found, 661.1118;

IR (KBr): ν_{max} 3425 (OH), 1672 (C=O), 1651 (C=O), 1589, 1489, 1396, 1087, 833, 702, 532 cm^{-1}

mp 237.9-238.5 °C



3c

(4-(4-bromophenyl)-4-hydroxy-2,6-diphenylcyclohexane-1,3-diyl)bis((4-bromophenyl)methanone)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.8 Hz, 2H), 7.32 (d, *J* = 8.8 Hz, 2H), 7.24 – 7.21(m, 4H), 7.18 – 7.06 (m, 10H), 7.02 (t, *J* = 7.2 Hz, 1H), 6.87 (t, *J* = 7.6 Hz, 2H), 6.79 (t, *J* = 7.6 Hz, 1H), 5.35 (d, *J* = 2.4 Hz, 1H), 4.40 – 4.33 (m, 1H), 4.17 – 4.09 (m, 2H), 4.04 – 3.98 (m, 1H), 2.46 – 2.37 (m, 1H), 2.22 (dd, *J* = 14.0, 3.6 Hz, 1H).

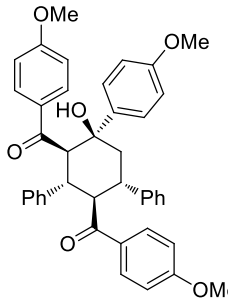
^{13}C NMR (100 MHz, $CDCl_3$) δ 205.70, 202.33, 144.93, 141.53, 138.17, 137.39, 136.36, 131.38, 131.25, 130.93, 129.12, 128.82, 128.55, 128.37, 127.81, 127.36, 127.12, 127.04, 126.57,

121.21, 75.14, 56.53, 56.22, 47.96, 45.61, 43.24.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{29}O_3NaBr_3$, 792.9564 found, 792.9537;

IR (KBr): ν_{max} 3441 (OH), 1666 (C=O), 1643 (C=O), 1581, 1489, 1396, 1072, 1002, 833, 702, 532 cm^{-1}

mp 256.8-258.0 °C



3d

(4-hydroxy-4-(4-methoxyphenyl)-2,6-diphenylcyclohexane-1,3-diyl)bis((4-methoxyphenyl)methanone)

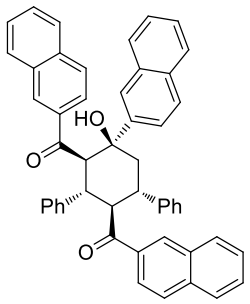
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.8 Hz, 2H), 7.35 – 7.31 (m, 4H), 7.26 (d, *J* = 7.2 Hz, 2H), 7.15 – 7.06 (m, 4H), 6.97 (t, *J* = 7.2 Hz, 1H), 6.84 (t, *J* = 7.6 Hz, 2H), 6.74 – 6.69 (m, 3H), 6.55 – 6.50 (m, 4H), 5.56 (d, *J* = 2.0 Hz, 1H), 4.44 – 4.36 (m, 1H), 4.19 – 4.12 (m, 2H), 4.07– 4.00 (m, 1H), 3.70 (s, 3H), 3.68 (s, 3H), 3.66 (s, 3H), 2.49 – 2.41 (m, 1H), 2.20 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 204.97, 201.47, 163.21, 162.43, 158.26, 142.38, 139.02, 138.48, 131.84, 130.96, 130.36, 129.85, 128.26, 127.97, 127.92, 126.72, 126.56, 125.95, 113.40, 112.93, 112.74, 75.00, 55.95, 55.88, 55.26, 55.16, 55.11, 47.99, 46.28, 43.30.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{41}H_{38}O_6Na$, 649.2566 found, 649.2526;

IR (KBr): ν_{max} 3402 (OH), 1658 (C=O), 1597 (C=O), 1512, 1257, 1174, 1026, 833, 702, 540 cm^{-1}

mp 223.2-224.4 °C



3e

(4-hydroxy-4-(naphthalen-2-yl)-2,6-diphenylcyclohexane-1,3-diyl)bis(naphthalen-2-

ylmethanone)

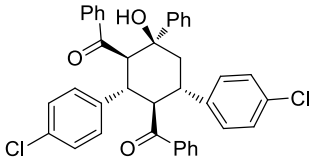
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.78 – 7.56 (m, 10H), 7.49 – 7.27 (m, 14H), 7.08 (t, *J* = 7.6 Hz, 2H), 6.91 (t, *J* = 7.2 Hz, 1H), 6.75 (t, *J* = 8.0 Hz, 2H), 6.55 (t, *J* = 7.2 Hz, 1H), 5.69 (d, *J* = 2.4 Hz, 1H), 4.84 (d, *J* = 11.6 Hz, 1H), 4.48 (t, *J* = 11.2 Hz, 1H), 4.37 (t, *J* = 11.2 Hz, 1H), 4.25 – 4.18 (m, 1H), 2.77– 2.69 (m, 1H), 2.38 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 206.92, 203.39, 143.41, 142.17, 138.74, 136.32, 135.36, 135.03, 134.78, 133.10, 132.30, 131.93, 131.66, 129.86, 129.32, 129.30, 128.98, 128.57, 128.41, 128.35, 128.10, 128.00, 127.91, 127.87, 127.57, 127.48, 127.43, 127.26, 126.96, 126.79, 126.39, 126.19, 126.04, 125.73, 124.10, 123.55, 123.17, 123.00, 75.73, 56.88, 56.49, 48.37, 45.96, 43.53.

HRMS: (ESI) $[M+H]^+$ calcd. for $C_{50}H_{39}O_3$, 687.2899 found, 687.2899;

IR (KBr): ν_{max} 3410 (OH), 1666 (C=O), 1621 (C=O), 1512, 1357, 1180, 748 cm^{-1}

mp 247.6-248.3 °C



3f

(2,6-bis(4-chlorophenyl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

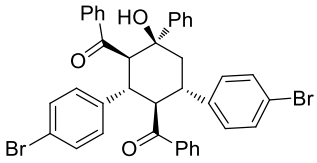
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.2 Hz, 2H), 7.29 – 7.15 (m, 10H), 7.12 – 7.02 (m, 9H), 6.80 (d, *J* = 8.4 Hz, 2H), 5.32 (d, *J* = 2.4 Hz, 1H), 4.48 – 4.40 (m, 1H), 4.21 – 4.10 (m, 2H), 4.08 – 3.99 (m, 1H), 2.51 – 2.39 (m, 1H), 2.22 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 206.68, 202.88, 145.43, 140.40, 138.46, 137.85, 137.11, 133.00, 132.70, 132.43, 132.33, 129.26, 128.51, 128.26, 128.24, 127.87, 127.66, 127.35, 127.18, 124.69, 75.22, 56.51, 56.46, 47.41, 45.69, 42.76.

HRMS: (ESI) $[M+H]^+$ calcd. for $C_{38}H_{31}O_3Cl_2$, 605.1650 found, 605.1650;

IR (KBr): ν_{max} 3394 (OH), 1668 (C=O), 1631 (C=O), 1597, 1489, 1096, 1010, 694, 540 cm^{-1}

mp 250.5-252.9 °C



3g

(2,6-bis(4-bromophenyl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.6 Hz, 2H), 7.31 – 7.16 (m, 10H), 7.15 – 6.92 (m,

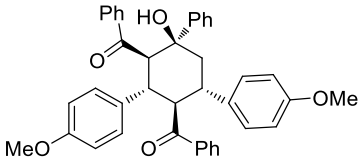
11H), 5.31 (d, *J* = 2.4 Hz, 1H), 4.48 – 4.39 (m, 1H), 4.20 – 4.08 (m, 2H), 4.07 – 3.97 (m, 1H), 2.50 – 2.39 (m, 1H), 2.22 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 206.65, 202.84, 145.39, 140.90, 138.45, 137.85, 137.61, 133.00, 132.34, 131.47, 131.19, 129.63, 128.27, 127.89, 127.87, 127.65, 127.35, 127.19, 124.69, 120.87, 120.54, 75.20, 56.48, 56.34, 47.47, 45.63, 42.82.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{30}O_3NaBr_2$, 715.0459 found, 715.0471;

IR (KBr): ν_{max} 3441 (OH), 1666 (C=O), 1631 (C=O), 1597, 1489, 1072, 1010, 694, 540 cm^{-1}

mp 260.4-261.6 °C



3h

(4-hydroxy-2,6-bis(4-methoxyphenyl)-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

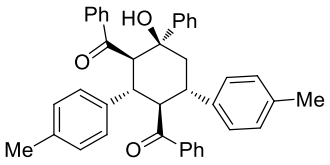
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.6 Hz, 2H), 7.28 – 7.14 (m, 10H), 7.10 – 6.98 (m, 7H), 6.63 (d, *J* = 8.8 Hz, 2H), 6.36 (d, *J* = 8.8 Hz, 2H), 5.35 (d, *J* = 2.4 Hz, 1H), 4.49 – 4.39 (m, 1H), 4.18 – 4.07 (m, 2H), 4.05 – 3.92 (m, 1H), 3.63 (s, 3H), 3.47 (s, 3H), 2.51 – 2.38 (m, 1H), 2.20 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.39, 203.92, 158.16, 145.93, 139.03, 138.17, 134.29, 132.60, 131.72, 130.75, 128.87, 128.13, 127.69, 127.67, 127.59, 127.40, 126.93, 124.78, 113.75, 113.50, 75.39, 57.26, 57.01, 55.11, 54.97, 47.23, 46.10, 42.52.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{40}H_{36}O_5Na$, 619.2460 found, 619.2462;

IR (KBr): ν_{max} 3417 (OH), 1666 (C=O), 1643 (C=O), 1512, 1249, 1033, 825, 694, 540 cm^{-1}

mp 199.8-200.1 °C



3i

(4-hydroxy-4-phenyl-2,6-di-p-tolylcyclohexane-1,3-diyl)bis(phenylmethanone)

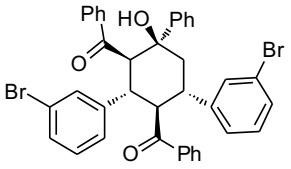
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.2 Hz, 2H), 7.28 – 7.12 (m, 10H), 7.10– 6.94 (m, 7H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.62 (d, *J* = 8.0 Hz, 2H), 5.34 (d, *J* = 2.0 Hz, 1H), 4.45 (d, *J* = 11.2 Hz, 1H), 4.22 – 4.08 (m, 2H), 4.05 – 3.95 (m, 1H), 2.54 – 2.41 (m, 1H), 2.21 (dd, *J* = 14.0, 3.6 Hz, 1H), 2.12 (s, 3H), 1.91 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.34, 203.79, 145.95, 139.09, 138.22, 136.30, 136.06, 135.57, 132.47, 131.54, 128.96, 128.68, 128.10, 127.79, 127.72, 127.59, 127.47, 126.90, 124.81, 75.37, 57.03, 56.98, 47.60, 46.04, 42.93, 20.83, 20.65.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{40}H_{36}O_3Na$, 587.2562 found, 587.2556;

IR (KBr): ν_{max} 3417 (OH), 1666 (C=O), 1643 (C=O), 1597, 1342, 1210, 694, 540 cm^{-1}

mp 270.7-271.9 °C



3j

(2,6-bis(3-bromophenyl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

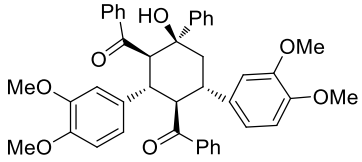
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.51 (m, 2H), 7.47 (t, *J* = 1.6 Hz, 1H), 7.40 – 6.96 (m, 17H), 6.93 (t, *J* = 8.0 Hz, 1H), 6.86 – 6.80 (m, 1H), 6.69 (t, *J* = 8.0 Hz, 1H), 5.38 (d, *J* = 2.4 Hz, 1H), 4.45 (d, *J* = 11.2 Hz, 1H), 4.22 – 4.07 (m, 2H), 4.05 – 3.95 (m, 1H), 2.50 – 2.36 (m, 1H), 2.23 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 206.54, 202.52, 145.44, 144.21, 140.86, 138.44, 137.80, 133.06, 132.37, 130.64, 130.05, 129.99, 129.87, 129.74, 128.29, 127.85, 127.70, 127.39, 127.19, 127.07, 124.68, 122.43, 122.15, 75.20, 56.15, 55.96, 47.61, 45.82, 43.09.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{30}O_3Br_2Na$, 715.0459 found, 715.0447;

IR (KBr): ν_{max} 3402 (OH), 1666 (C=O), 1643 (C=O), 1573, 1188, 1072, 902, 779, 694 cm^{-1}

mp 236.0-237.1 °C



3k

(2,6-bis(3,4-dimethoxyphenyl)-4-hydroxy-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

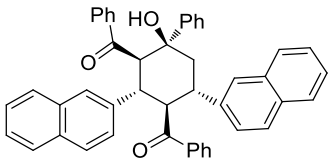
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.6 Hz, 2H), 7.33 – 7.16 (m, 8H), 7.13 – 7.01 (m, 4H), 6.85 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.78 – 6.56 (m, 4H), 6.34 (d, *J* = 8.4 Hz, 2H), 5.40 (d, *J* = 2.0 Hz, 1H), 4.45 (d, *J* = 11.2 Hz, 1H), 4.21 – 4.07 (m, 2H), 4.05 – 3.95 (m, 1H), 3.75 (s, 3H), 3.71 (s, 3H), 3.59 (s, 3H), 3.54 (s, 3H), 2.50 – 2.39 (m, 1H), 2.24 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.29, 203.64, 148.59, 148.31, 147.71, 147.62, 145.88, 139.05, 138.19, 134.81, 132.71, 131.92, 131.11, 128.18, 127.69, 127.66, 127.62, 127.34, 127.01,

124.77, 119.71, 111.86, 111.22, 110.90, 75.45, 56.91, 56.87, 55.86, 55.80, 55.78, 55.62, 47.79, 46.26, 43.06.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{42}H_{40}O_7Na$, 679.2672 found, 679.2667;

IR (film): ν_{max} 3441 (OH), 3024, 1672 (C=O), 1650 (C=O), 1516, 1257, 1141, 1026, 754cm^{-1}



3l

(4-hydroxy-2,6-di(naphthalen-2-yl)-4-phenylcyclohexane-1,3-diyl)bis(phenylmethanone)

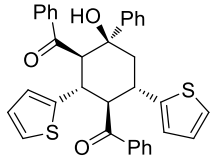
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.15 (m, 22H), 7.13 – 7.01 (m, 2H), 6.99 – 6.86 (m, 3H), 6.80 (t, *J* = 8.0 Hz, 2H), 5.46 (d, *J* = 2.4 Hz, 1H), 4.67 (d, *J* = 11.2 Hz, 1H), 4.54 – 4.37 (m, 2H), 4.35 – 4.25 (m, 1H), 2.75 – 2.55 (m, 1H), 2.35 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 207.13, 203.39, 145.83, 139.55, 138.68, 137.99, 135.99, 133.34, 132.88, 132.56, 132.32, 132.16, 131.66, 128.20, 128.04, 127.88, 127.60, 127.54, 127.38, 127.25, 127.12, 127.04, 126.94, 126.00, 125.78, 125.71, 125.42, 125.33, 124.83, 75.42, 56.79, 56.49, 48.21, 46.17, 43.53.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{46}H_{36}O_3Na$, 659.2562 found, 659.2576;

IR (KBr): ν_{max} 3410 (OH), 1668 (C=O), 1643 (C=O), 1597, 1342, 1256, 817, 694, 478 cm^{-1}

mp 283.5-284.1 ^oC



3m

(4-hydroxy-4-phenyl-2,6-di(thiophen-2-yl)cyclohexane-1,3-diyl)bis(phenylmethanone)

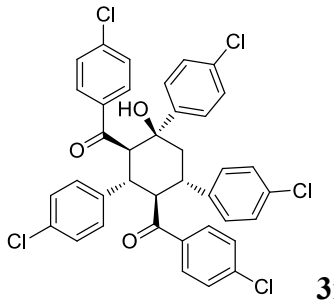
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.6 Hz, 2H), 7.45 (d, *J* = 7.2 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 2H), 7.31 – 7.24 (m, 2H), 7.20 – 7.01 (m, 7H), 6.93 (d, *J* = 5.2 Hz, 1H), 6.74 (d, *J* = 3.2 Hz, 1H), 6.69 – 6.60 (m, 3H), 6.38 (dd, *J* = 4.8, 3.6 Hz, 1H), 5.28 (d, *J* = 2.4 Hz, 1H), 4.55 (t, *J* = 11.2 Hz, 1H), 4.47 – 4.33 (m, 2H), 4.13 (t, *J* = 11.2 Hz, 1H), 2.53 – 2.43 (m, 1H), 2.39 (dd, *J* = 14.0, 4.0 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 206.65, 203.07, 145.46, 145.14, 142.02, 138.59, 137.79, 132.90, 132.14, 128.19, 127.88, 127.72, 127.68, 127.62, 127.11, 126.96, 126.53, 126.23, 125.52, 124.79, 123.88, 123.23, 75.20, 58.89, 57.67, 46.86, 43.30, 39.00.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{34}H_{28}O_3NaS_2$, 571.1378 found, 571.1375;

IR (KBr): ν_{max} 3371 (OH), 1666 (C=O), 1635 (C=O), 1589, 1342, 1249, 694, 524 cm^{-1}

mp 260.1-260.4 ^{o}C



3n

(2,4,6-tris(4-chlorophenyl)-4-hydroxycyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone)

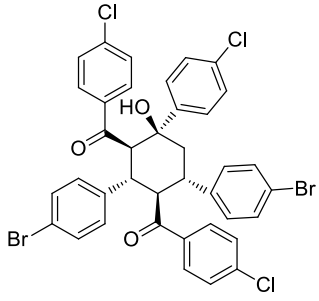
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.8 Hz, 2H), 7.26 – 7.13 (m, 8H), 7.13 – 6.97 (m, 8H), 6.87 (d, *J* = 8.4 Hz, 2H), 5.26 (d, *J* = 2.0 Hz, 1H), 4.33 (d, *J* = 11.2 Hz, 1H), 4.18 – 3.93 (m, 3H), 2.42 – 2.31 (m, 1H), 2.18 (dd, *J* = 14.0, 3.2 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 204.87, 201.34, 143.91, 140.15, 139.91, 139.08, 136.73, 136.46, 135.67, 133.24, 133.20, 132.80, 129.11, 129.03, 128.72, 128.55, 128.50, 128.49, 128.32, 126.13, 74.97, 56.19, 56.06, 47.28, 45.54, 42.67.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{38}H_{27}O_3NaCl_5$, 729.0301 found, 729.0262;

IR (KBr): ν_{max} 3487 (OH), 1666 (C=O), 1635 (C=O), 1589, 1489, 1095, 825, 732, 532 cm^{-1}

mp 259.6-261.2 ^{o}C



3o

(2,6-bis(4-bromophenyl)-4-(4-chlorophenyl)-4-hydroxycyclohexane-1,3-diyl)bis((4-chlorophenyl)methanone)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.8 Hz, 2H), 7.26 – 7.14 (m, 8H), 7.12 – 7.06 (m, 6H), 7.04 – 6.93 (m, 4H), 5.25 (d, *J* = 2.4 Hz, 1H), 4.32 (d, *J* = 11.2 Hz, 1H), 4.17 – 3.91 (m, 3H), 2.41 – 2.29 (m, 1H), 2.18 (dd, *J* = 14.0, 3.6 Hz, 1H).

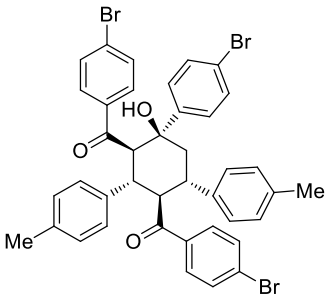
^{13}C NMR (100 MHz, $CDCl_3$) δ 204.85, 201.30, 143.87, 140.41, 140.15, 139.10, 137.23, 136.44, 135.67, 133.24, 131.66, 131.50, 129.46, 129.03, 128.72, 128.50, 128.48, 128.33, 126.12,

121.32, 120.87, 74.94, 56.06, 56.02, 47.33, 45.47, 42.73.

HRMS: (ESI) $[M+H]^+$ calcd. for $C_{38}H_{28}Br_2Cl_3O_3$, 794.9471 found, 794.9457;

IR (KBr): ν_{max} 3441 (OH), 1666 (C=O), 1643 (C=O), 1589, 1489, 1087, 817, 532 cm^{-1}

mp 257.5-259.1 °C



3p

(4-(4-bromophenyl)-4-hydroxy-2,6-di-p-tolylcyclohexane-1,3-diyl)bis((4-bromophenyl)methanone)

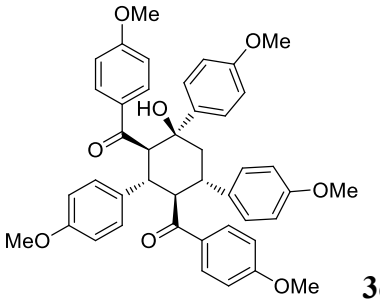
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.8 Hz, 2H), 7.30 (d, *J* = 8.8 Hz, 2H)，7.25 – 7.07 (m, 10H), 6.97 (d, *J* = 7.2 Hz, 2H), 6.90 (d, *J* = 7.6 Hz, 2H), 6.67 (d, *J* = 8.0 Hz, 2H), 5.30 (d, *J* = 2.4 Hz, 1H), 4.37 – 4.27 (m, 1H), 4.13 – 4.02 (m, 2H), 3.99 – 3.89 (m, 1H), 2.41 – 2.31 (m, 1H), 2.20 – 2.10 (m, 4H), 1.97 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.86, 202.49, 145.02, 138.56, 137.52, 136.93, 136.52, 136.48, 135.14, 131.33, 131.18, 130.86, 129.18, 129.16, 128.97, 128.33, 127.64, 126.90, 126.59, 121.12, 75.17, 56.71, 56.50, 47.48, 45.83, 42.84, 20.86, 20.72.

HRMS: (ESI) $[M+H]^+$ calcd. for $C_{40}H_{34}Br_3O_3$, 801.0038 found, 801.0092;

IR (KBr): ν_{max} 3448 (OH), 1666 (C=O), 1643 (C=O), 1581, 1396, 1072, 810 cm^{-1}

mp 282.2-282.8 °C



3q

(4-hydroxy-2,4,6-tris(4-methoxyphenyl)cyclohexane-1,3-diyl)bis((4-methoxyphenyl)methanone)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.8 Hz, 2H), 7.35 (d, *J* = 9.2 Hz, 4H), 7.17 (d, *J* = 8.8 Hz, 2H), 7.05 (d, *J* = 7.2 Hz, 2H), 6.69 (d, *J* = 8.8 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 6.54

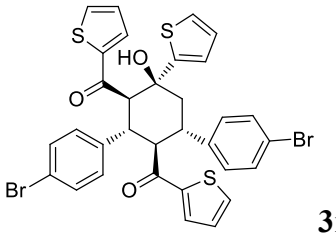
(d, *J* = 8.8 Hz, 4H), 6.37 (d, *J* = 8.8 Hz, 2H), 5.52 (d, *J* = 2.0 Hz, 1H), 4.40 – 4.30 (m, 1H), 4.18 – 4.03 (m, 2H), 4.03 – 3.90 (m, 1H), 3.70 (s, 3H), 3.69 (s, 3H), 3.65 (s, 3H), 3.62 (s, 3H), 3.47 (s, 3H), 2.46 – 2.33 (m, 1H), 2.16 (dd, *J* = 14.0, 3.6 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 205.17, 201.80, 163.19, 162.42, 158.21, 158.01, 157.95, 138.51, 134.61, 131.87, 131.20, 130.97, 130.36, 129.88, 128.80, 125.93, 113.65, 113.36, 112.95, 112.80, 75.06, 56.10, 55.24, 55.16, 55.09, 55.06, 54.91, 47.16, 46.55, 42.48.

HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{43}H_{42}O_8Na$, 709.2777 found, 709.2783;

IR (KBr): ν_{max} 3387 (OH), 1658 (C=O), 1604 (C=O), 1512, 1257, 1172, 1033, 833, 547 cm^{-1}

mp 147.8-248.8 °C



3r

(2,6-bis(4-bromophenyl)-4-hydroxy-4-(thiophen-2-yl)cyclohexane-1,3-diyl)bis(thiophen-2-ylmethanone)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 4.4 Hz, 1H), 7.36 (d, *J* = 4.8 Hz, 1H), 7.31 (d, *J* = 3.3 Hz, 1H), 7.25 (d, *J* = 7.6 Hz, 2H), 7.20 – 7.01 (m, 8H), 6.98 – 6.94 (m, 1H), 6.88 – 6.73 (m, 3H), 5.61 (s, 1H), 4.22 – 4.06 (m, 2H), 4.05 – 3.75 (m, 2H), 2.48 – 2.26 (m, 2H).

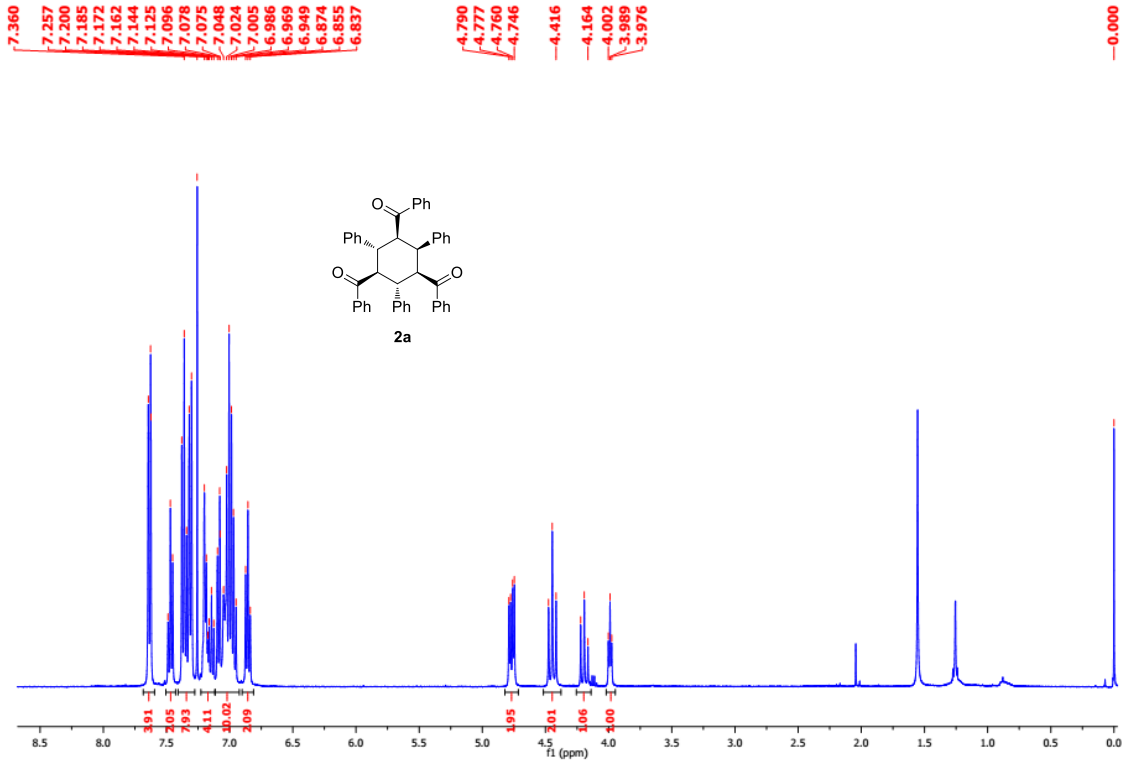
^{13}C NMR (100 MHz, $CDCl_3$) δ 196.92, 193.60, 151.08, 144.43, 140.48, 137.45, 135.48, 134.03, 133.39, 131.72, 131.52, 131.39, 129.58, 127.77, 127.58, 126.82, 124.05, 122.98, 121.06, 120.70, 74.51, 59.48, 57.95, 47.16, 46.81, 42.44.

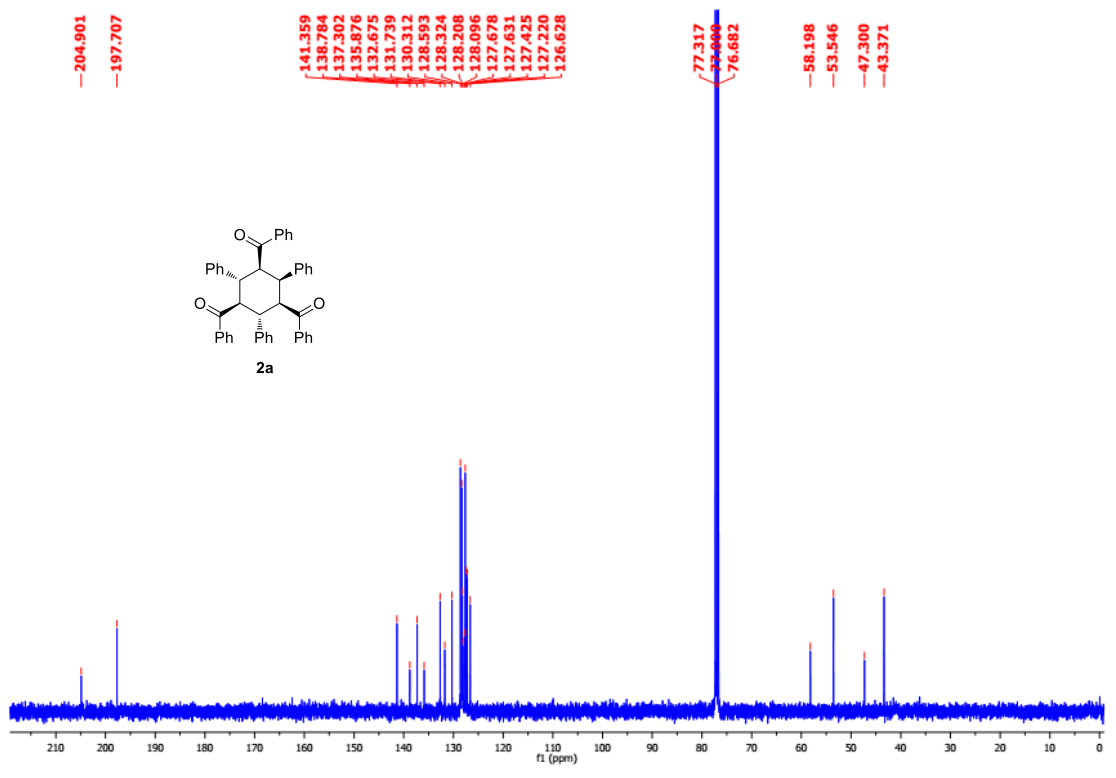
HRMS: (ESI) $[M+Na]^+$ calcd. for $C_{32}H_{24}O_3NaS_3Br_2$, 732.9152 found, 732.9152;

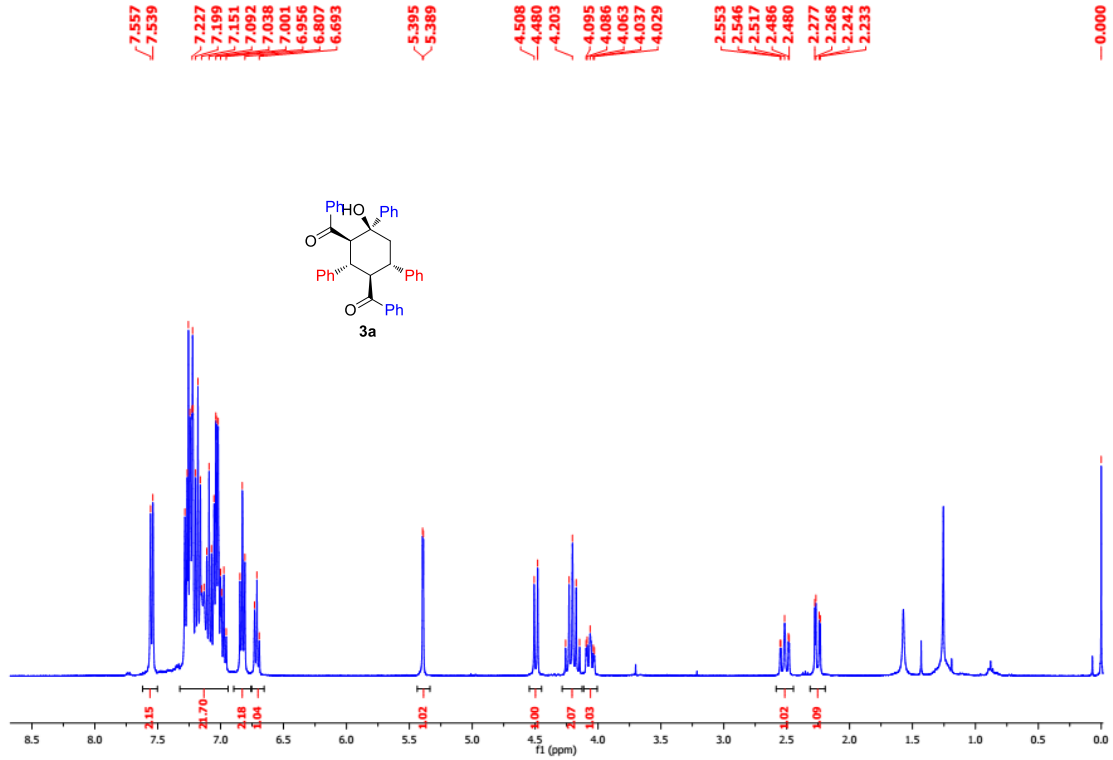
IR (KBr): ν_{max} 3433 (OH), 1643 (C=O), 1604 (C=O), 1411, 1257, 1072, 817, 709, 532 cm^{-1}

mp 251.3-252.6 °C

VIII: ^{1}H NMR, ^{13}C NMR, ^{13}C DEPT-135 spectra of products



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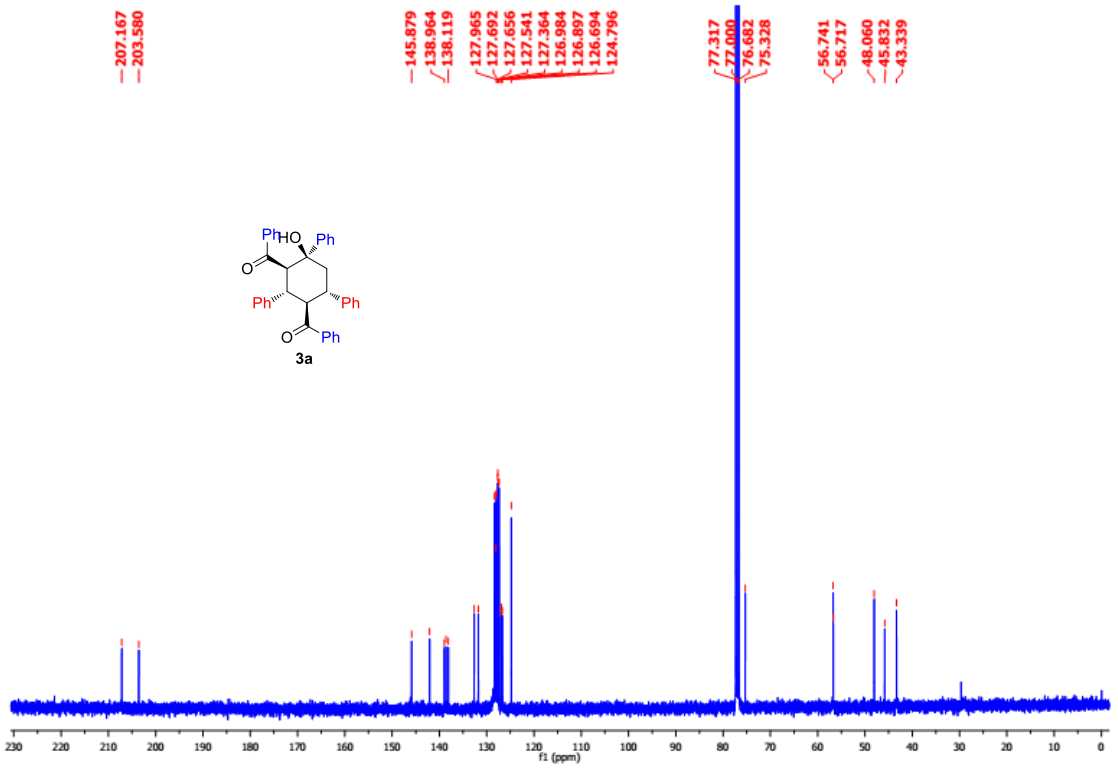
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77.317
77.000
76.682
75.328
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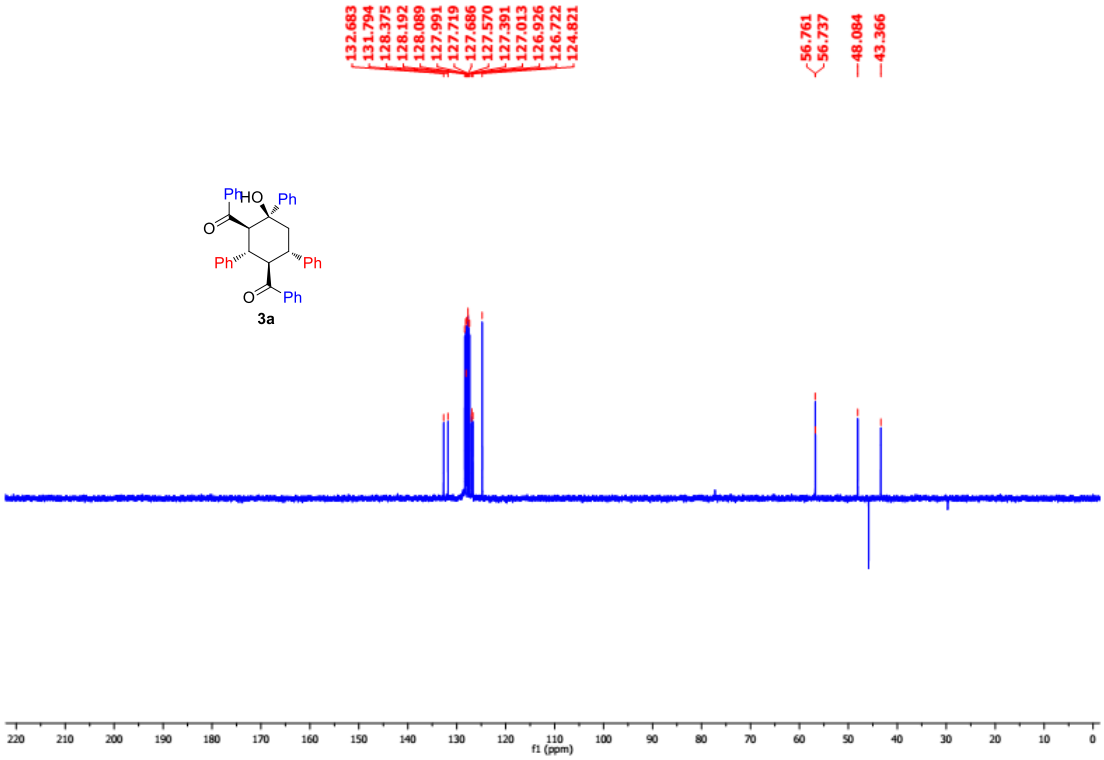
Ph
O
Ph
Ph
O
Ph
3a
f1 (ppm)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126.722
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56.737
48.084
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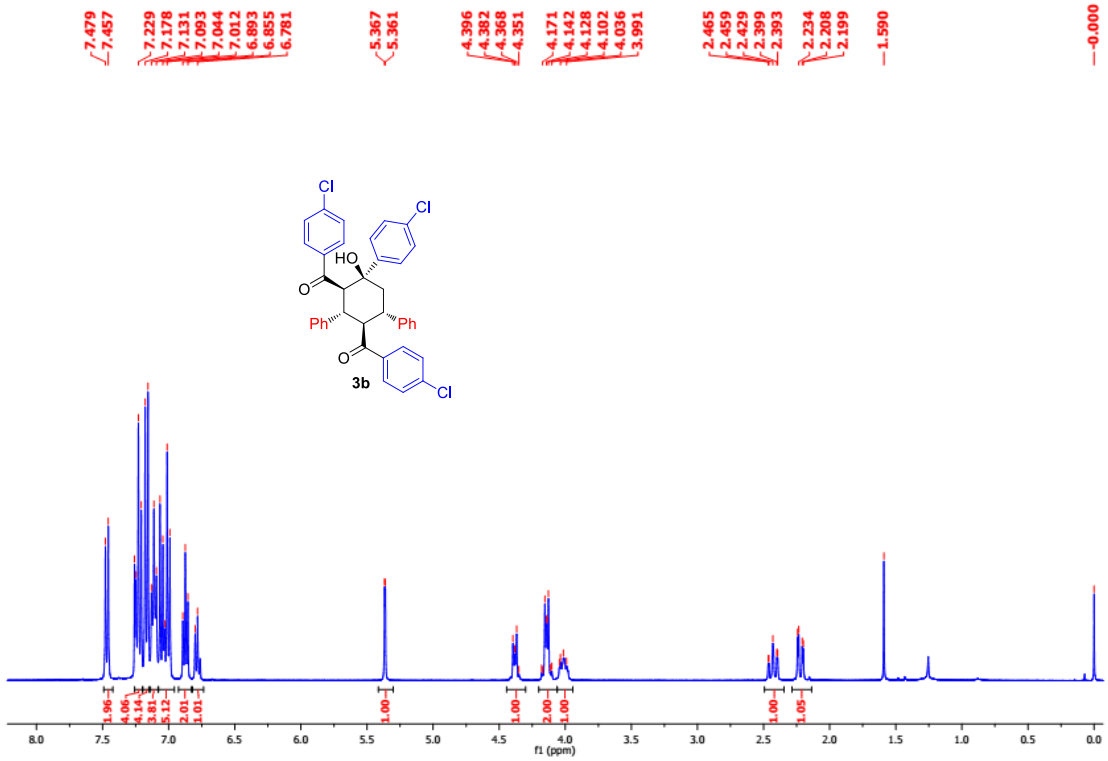
HO
Ph
O
Ph
Ph
O
Ph
3a
f1 (ppm)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7.012
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5.367
5.361
4.396
4.382
4.368
4.351
4.171
4.142
4.128
4.102
4.036
3.991
2.465
2.459
2.429
2.399
2.393
2.234
2.208
2.199
1.590
0.000
Cl
Cl
HO
O
Ph
Ph
O
Cl
3b
1.96
4.08
4.14
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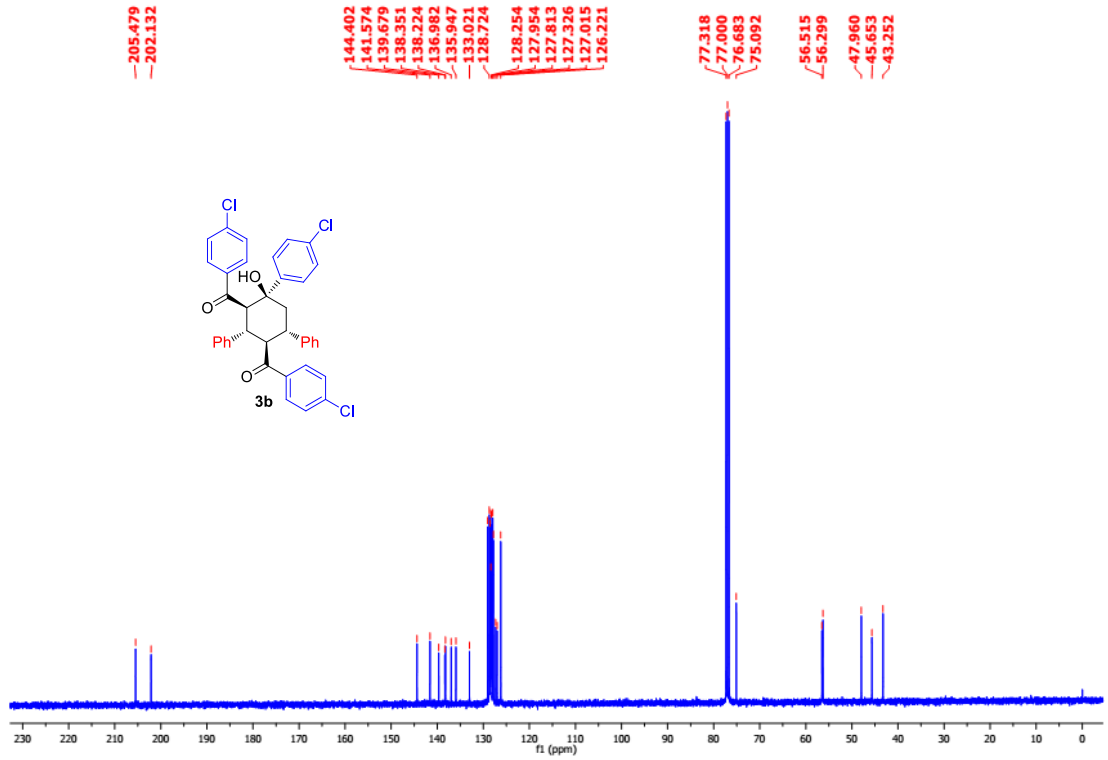
2.01
1.01
1.00
1.00
2.00
1.00
1.00
1.05
f1 (ppm)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128.254
127.954
127.813
127.326
127.015
126.221
77.318
77.000
76.683
75.092
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56.299
47.960
45.653
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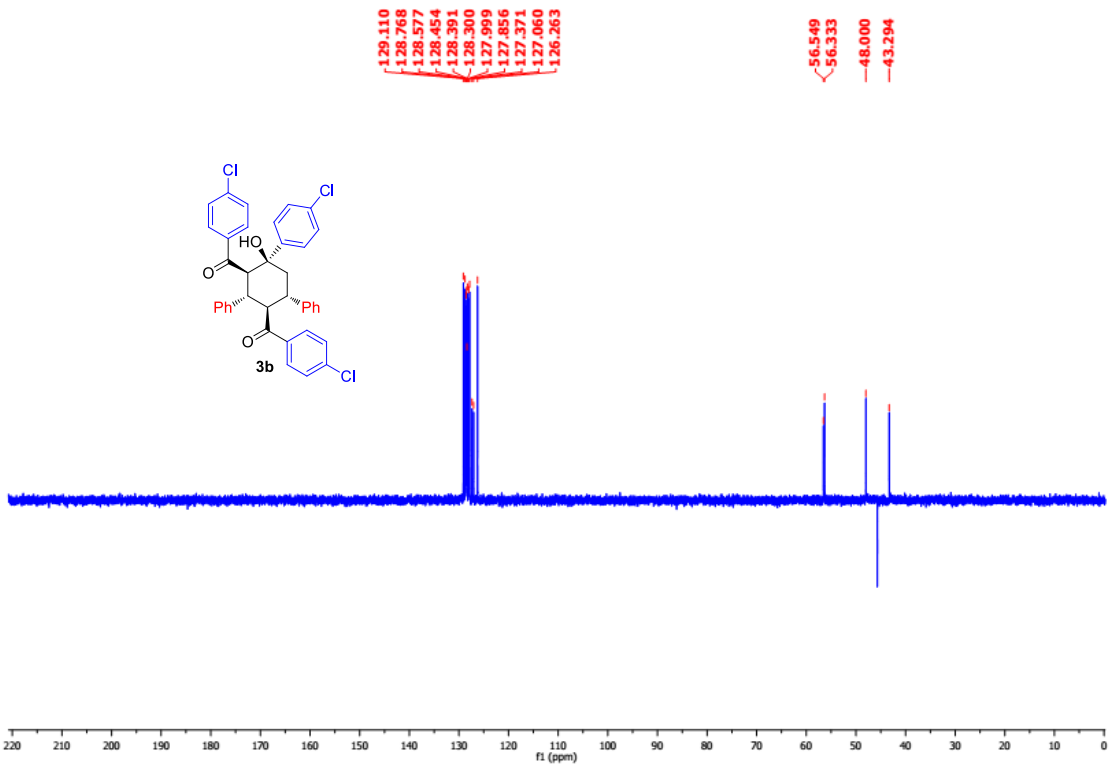
Cl
HO
O
Ph
Ph
O
Cl
3b
f1 (ppm)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127.060
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56.333
48.000
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Cl
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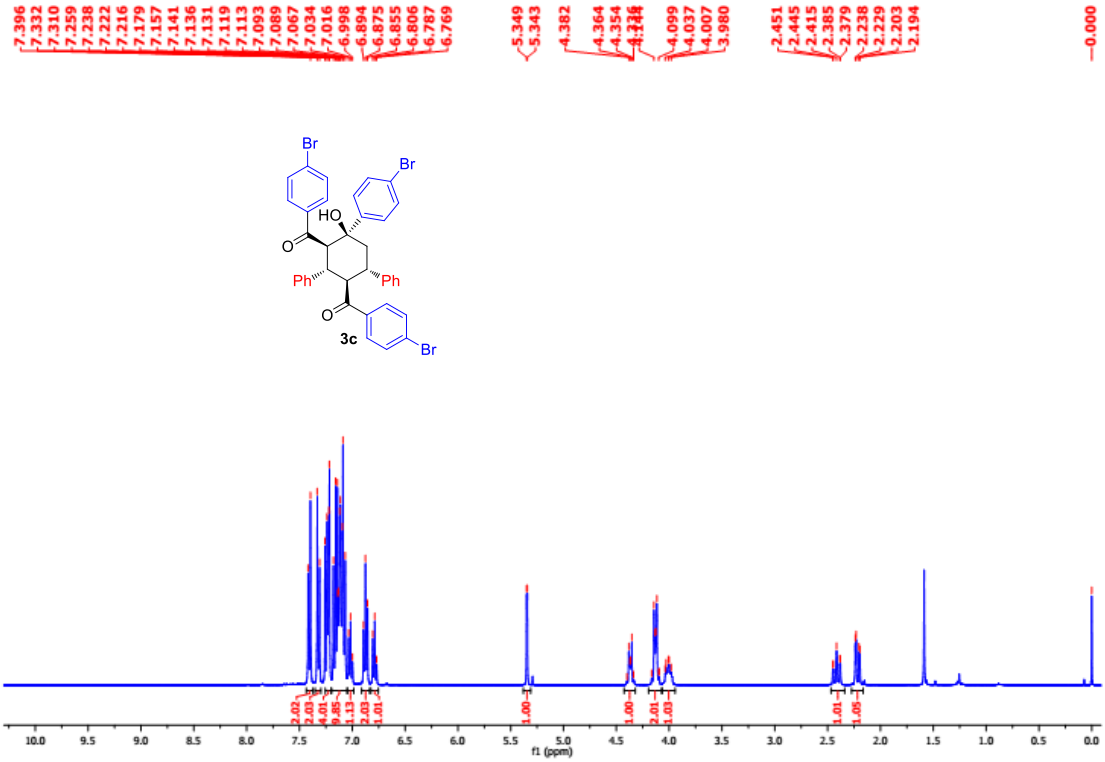
Ph
Ph
O
Cl
3b
f1 (ppm)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7.179
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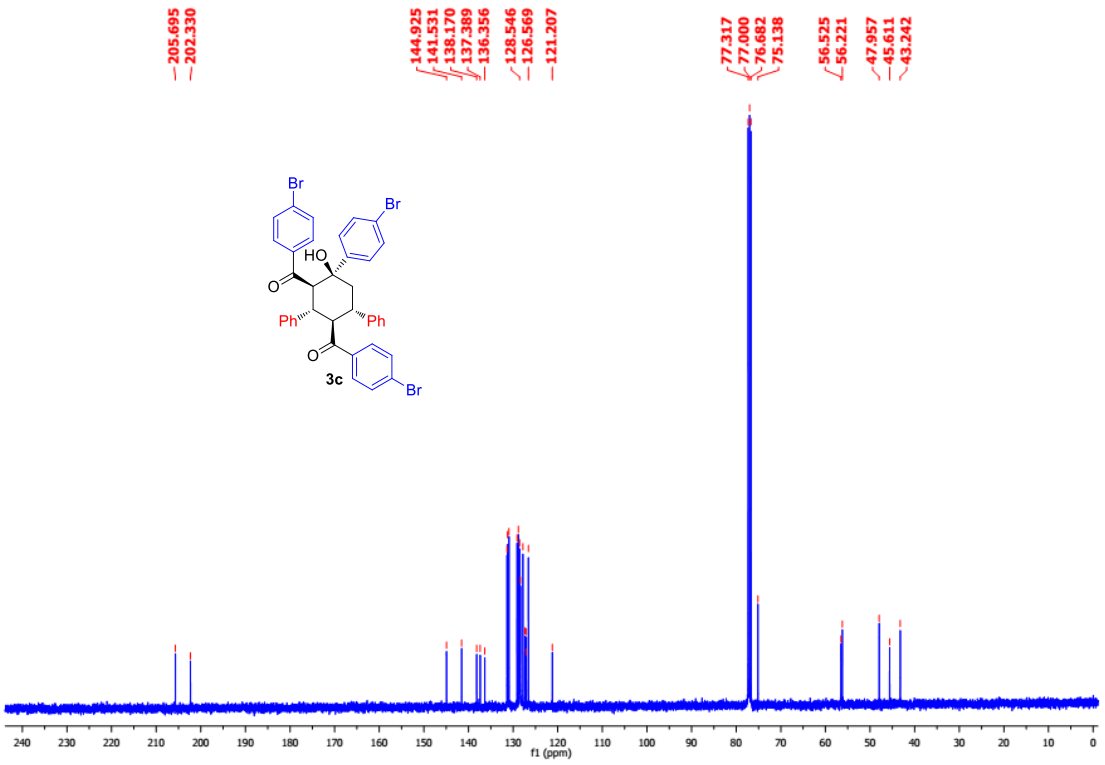
7.136
7.131
7.119
7.113
7.093
7.089
7.067
7.034
7.016
6.998
6.894
6.875
6.855
6.806
6.787
6.769
5.349
5.343
4.382
4.364
4.354
4.099
4.037
4.007
3.980
2.451
2.445
2.415
2.385
2.379
2.238
2.229
2.203
2.194
0.000
Br
Br
HO
O
Ph
Ph
O
Br
3c
f1 (ppm)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128.546
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77.000
76.682
75.138
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45.611
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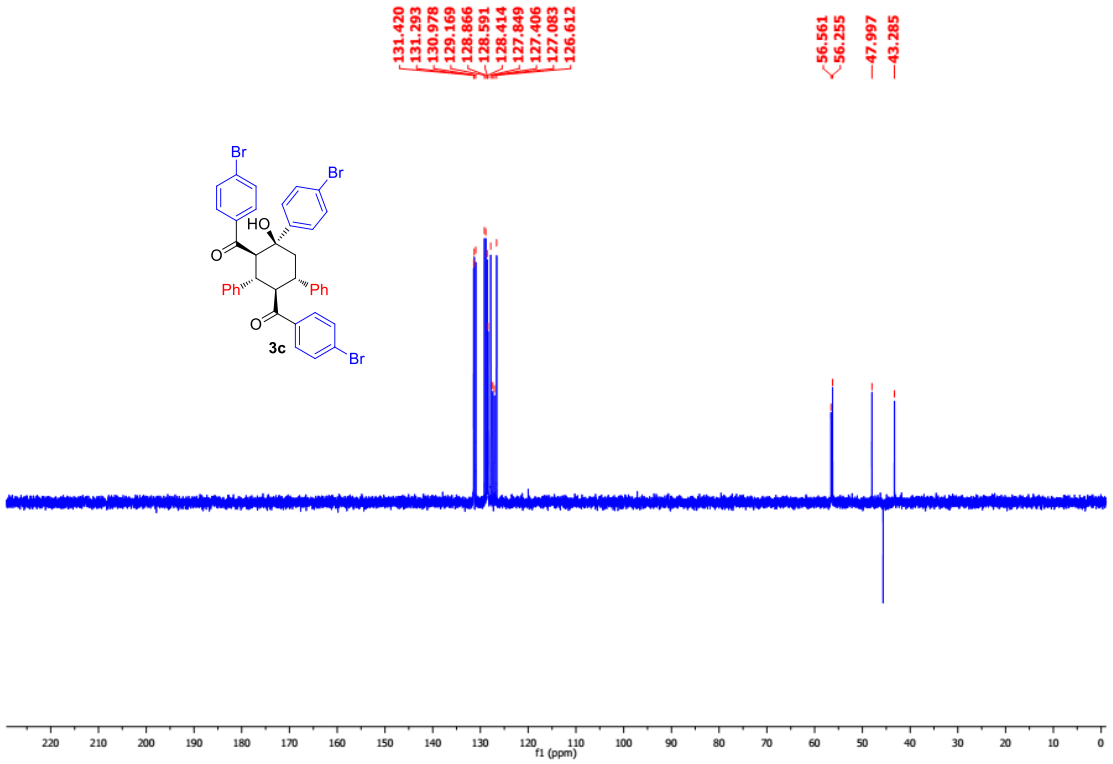
HO
O
Ph
Ph
O
Br
3c
f1 (ppm)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129.169
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128.591
128.414
127.849
127.406
127.083
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56.255
47.997
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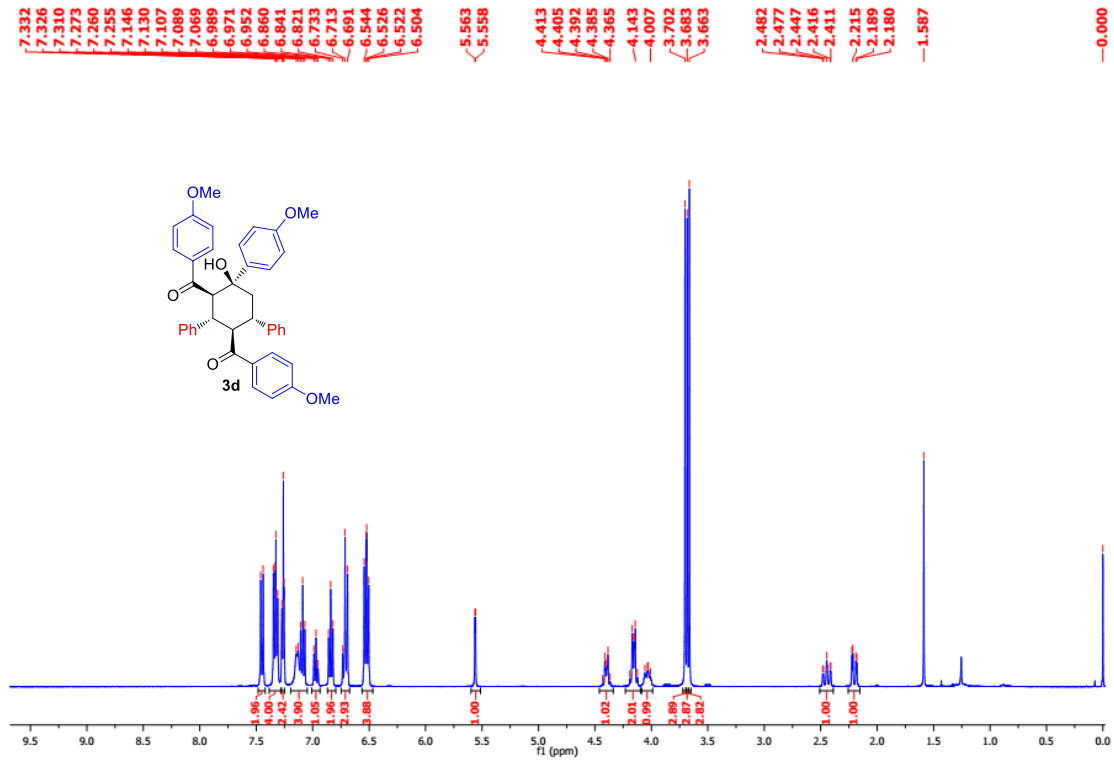
Br
HO
O
Ph
Ph
O
Br
3c
f1 (ppm)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7.107
7.089
7.069
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6.971
6.952
6.860
6.841
6.821
6.733
6.713
6.691
6.544
6.526
6.522
6.504
5.563
5.558
4.413
4.405
4.392
4.385
4.365
4.143
4.007
3.702
3.683
3.663
2.482
2.477
2.447
2.416
2.411
2.215
2.189
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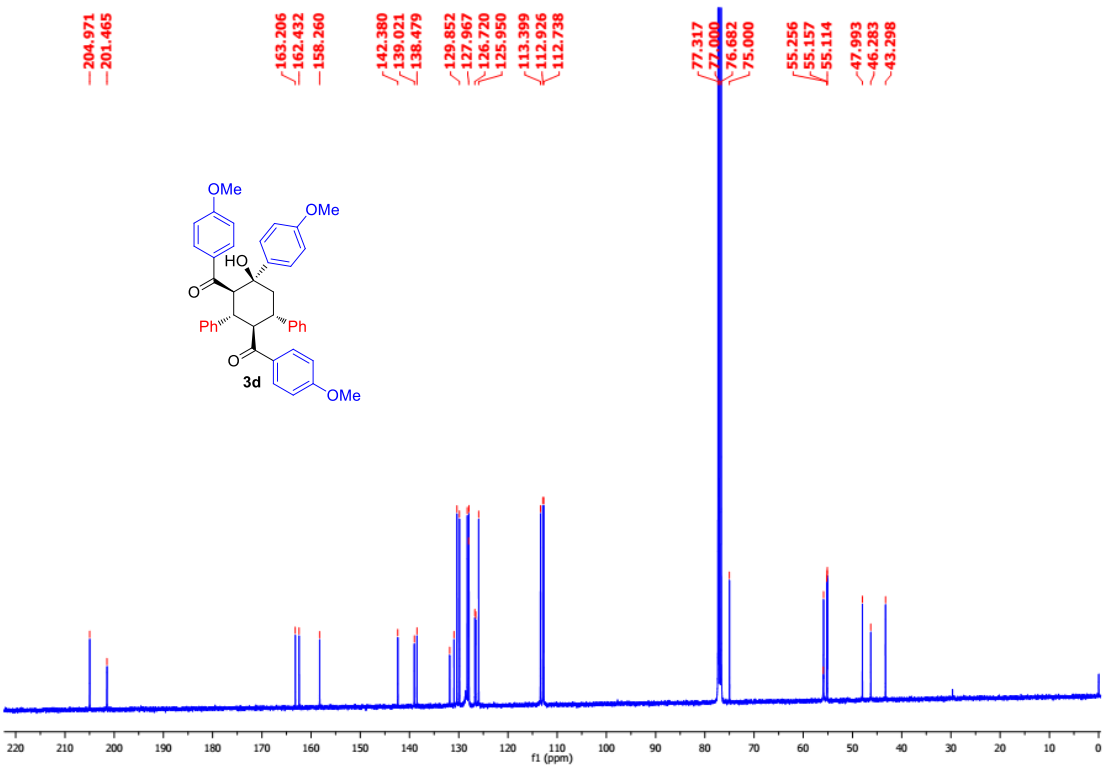
1.587
0.000
OMe
OMe
HO
O
Ph
Ph
O
3d
OMe
f1 (ppm)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138.479
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125.950
113.399
112.926
112.738
77.317
77.000
76.682
75.000
55.256
55.157
55.114
47.993
46.283
43.298
OMe
OMe
HO
O
Ph
Ph
O
3d
OMe
220
210
200
190
180
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160
150
140
130
120
110
100
90
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50
40
30
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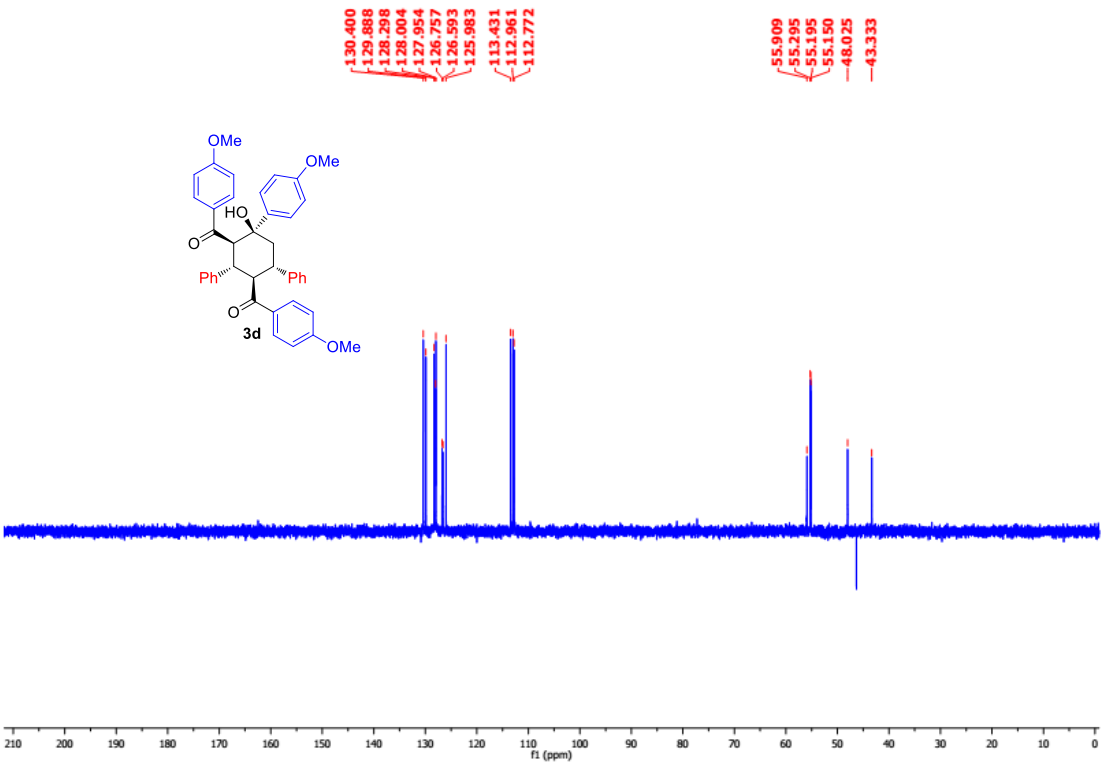
f1 (ppm)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55.295
55.195
55.150
48.025
43.333
OMe
OMe
HO
O
Ph
Ph
O
3d
OMe
210
200
190
180
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150
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130
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110
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50
40
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20
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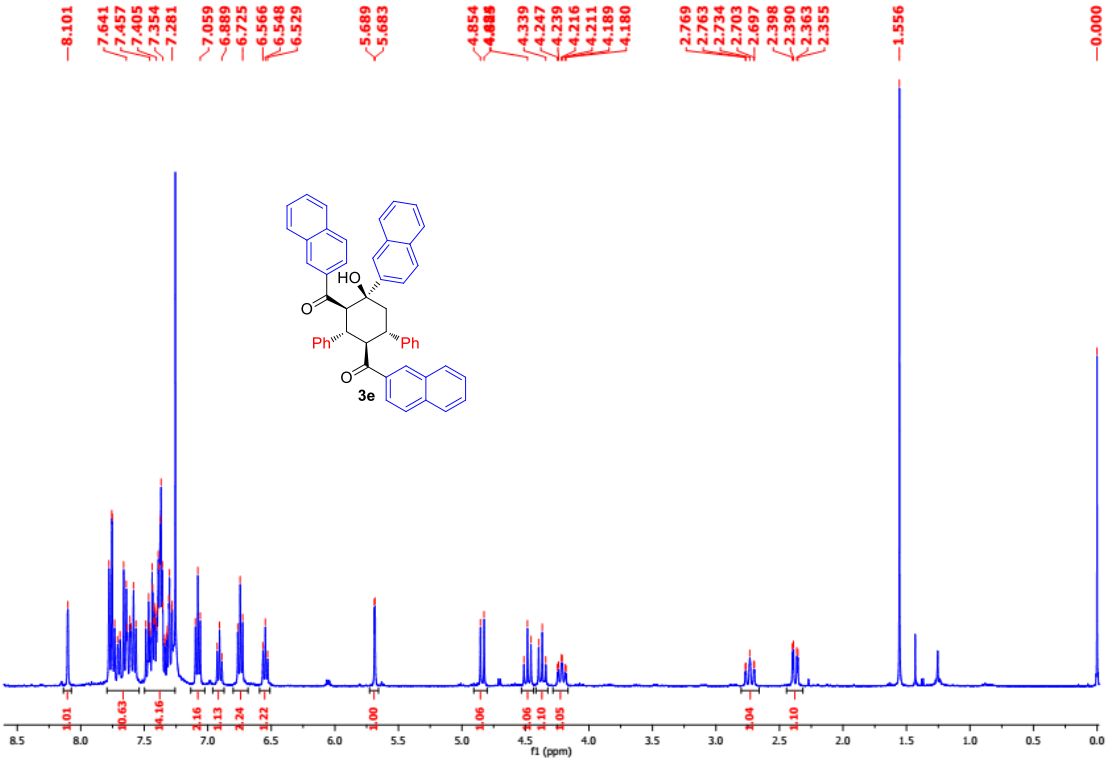
f1 (ppm)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6.548
6.529
5.689
5.683
4.339
4.247
4.239
4.216
4.211
4.189
4.180
2.769
2.763
2.734
2.703
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2.363
2.355
1.556
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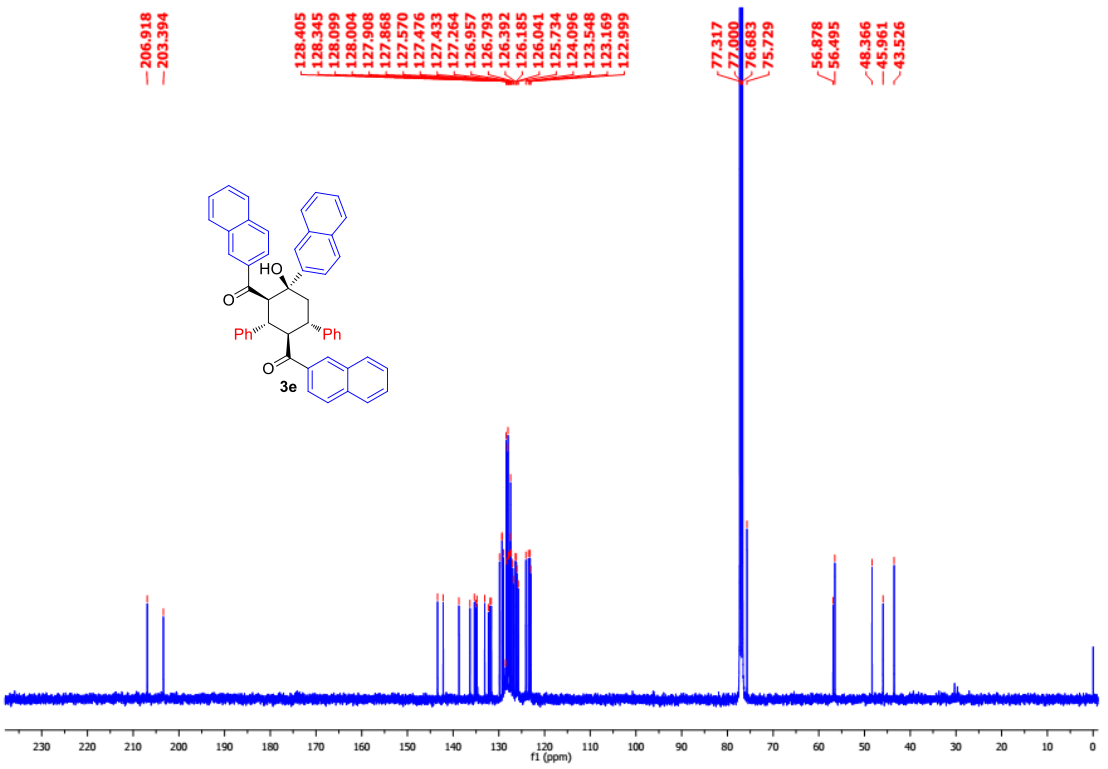
O
Ph
Ph
O
3e
f1 (ppm)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126.957
126.793
126.392
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126.041
125.734
124.096
123.548
123.169
122.999
77.317
77.000
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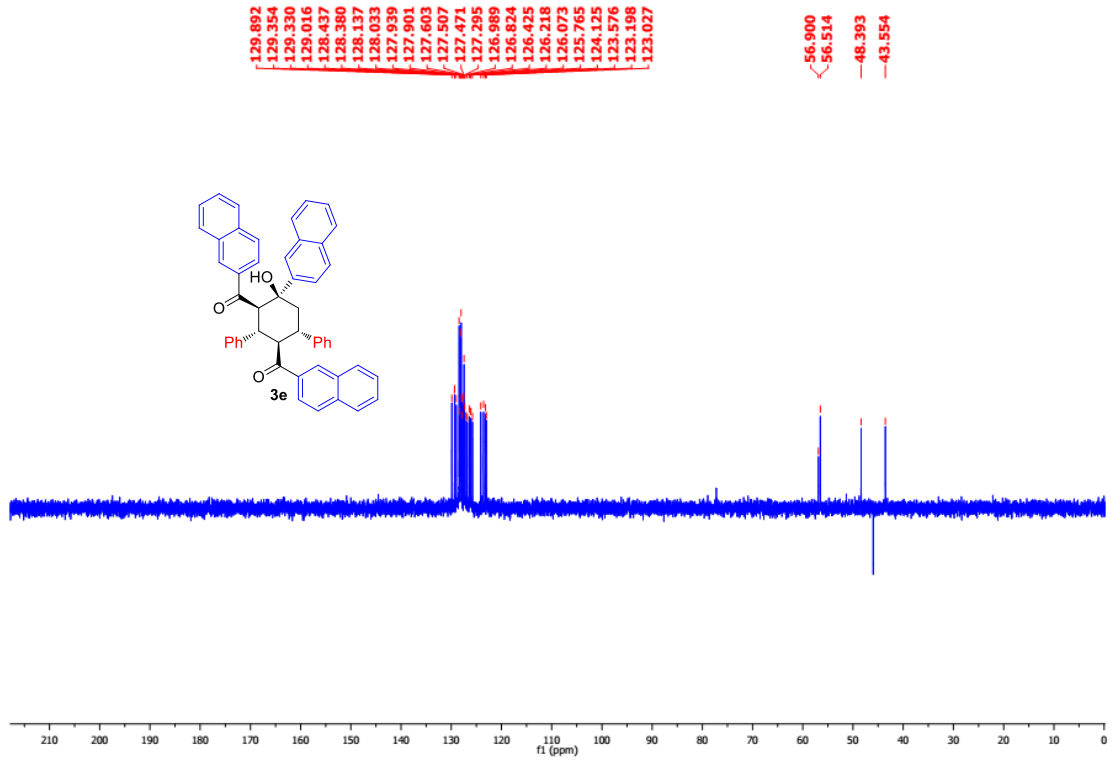
Ph
O
3e
f1 (ppm)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127.471
127.295
126.989
126.824
126.425
126.218
126.073
125.765
124.125
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48.393
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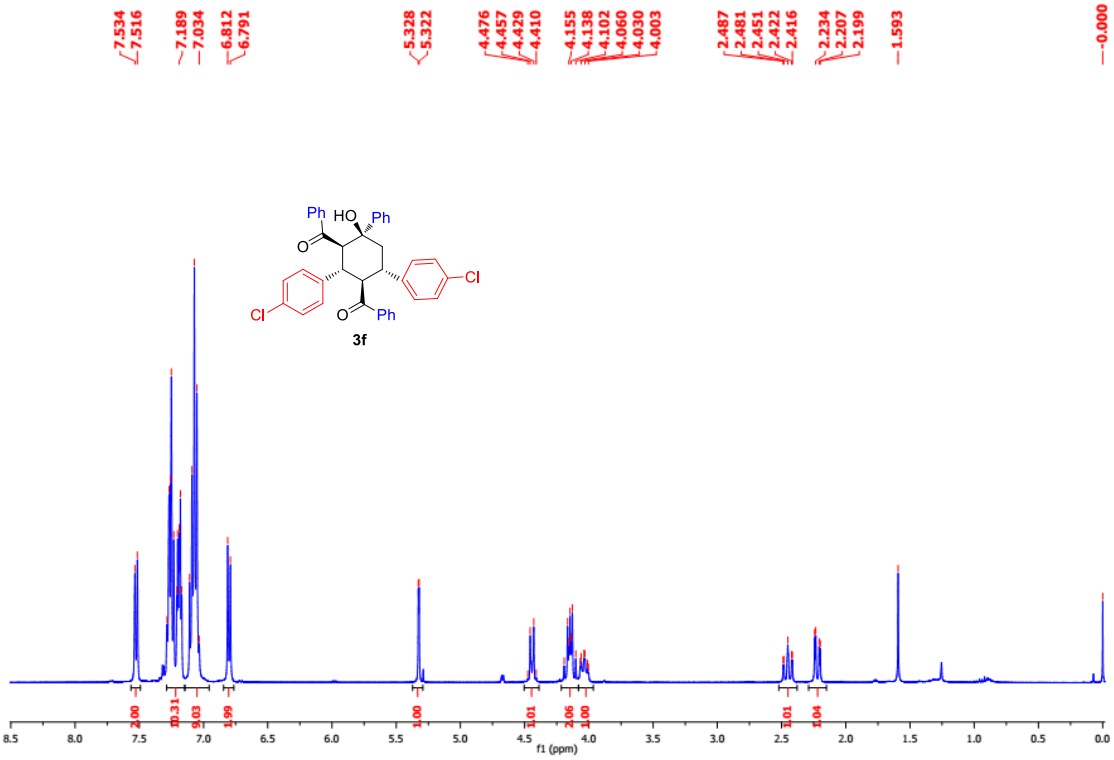
Ph
O
3e
f1 (ppm)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6.812
6.791
5.328
5.322
4.476
4.457
4.429
4.410
4.155
4.138
4.102
4.060
4.030
4.003
2.487
2.481
2.451
2.422
2.416
2.234
2.207
2.199
1.593
0.000
Ph
HO
Ph
O
Cl
Cl
O
Ph
3f
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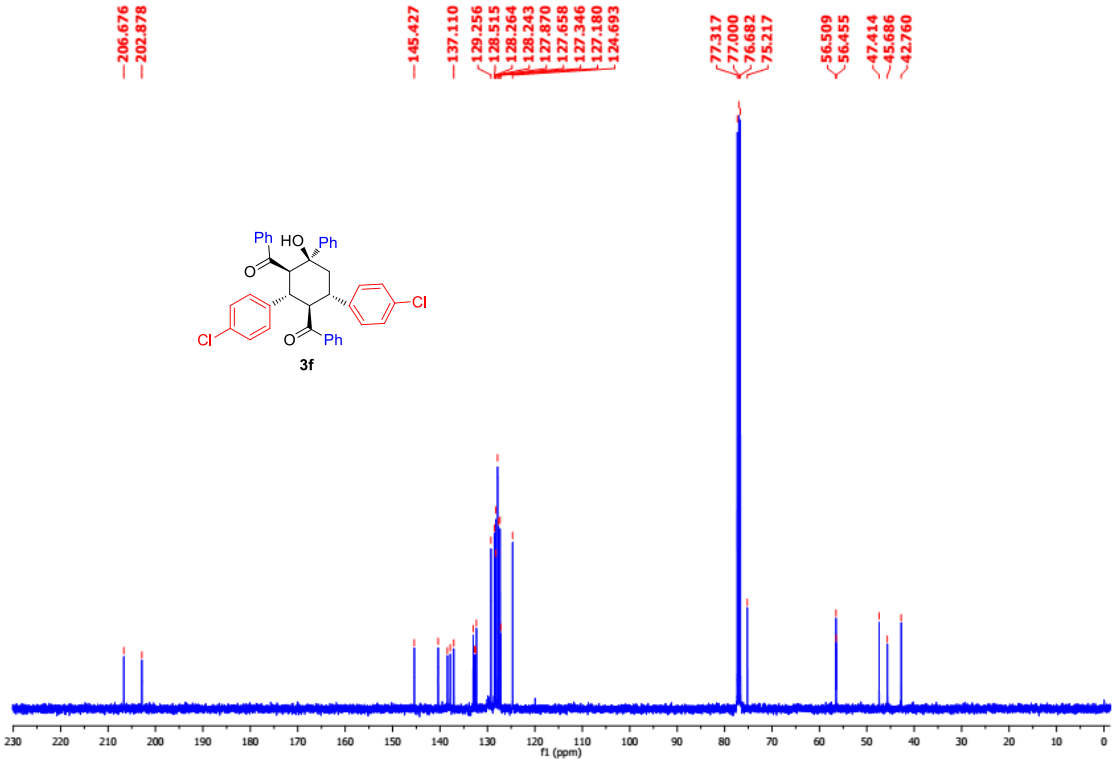
0.31
9.03
1.99
1.00
1.01
2.06
1.00
1.01
1.04
f1 (ppm)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77.317
77.000
76.682
75.217
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45.686
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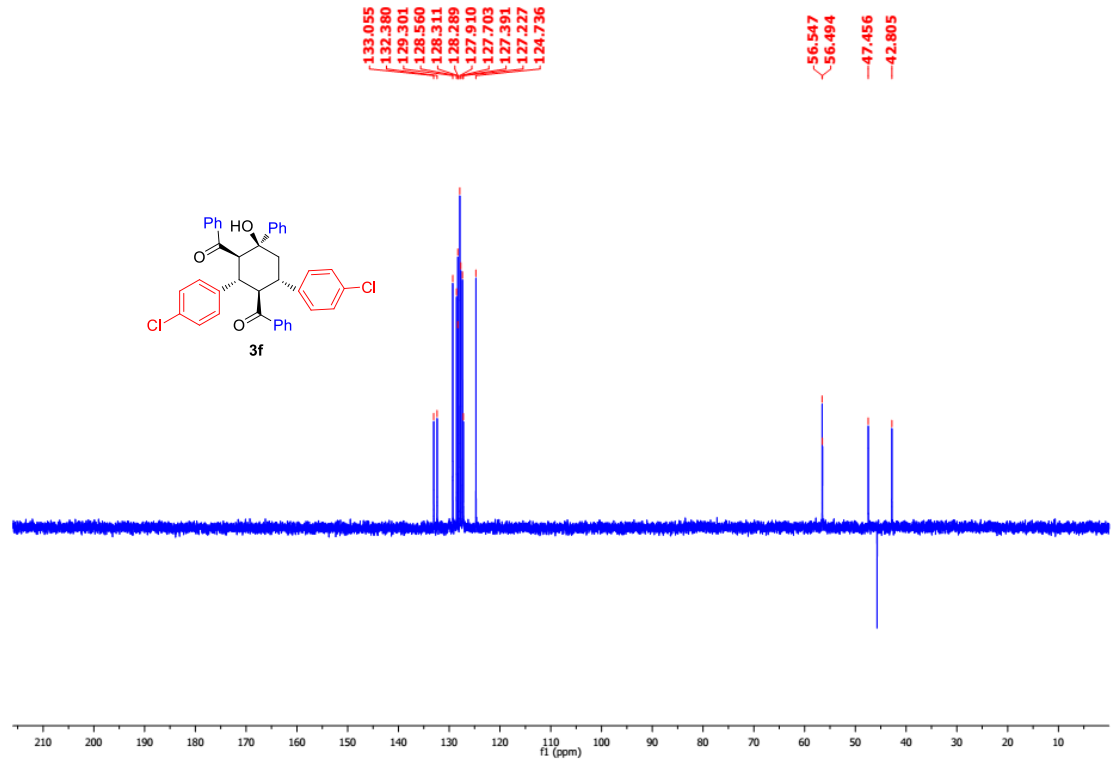
Ph
HO
Ph
O
Cl
Cl
O
Ph
3f
f1 (ppm)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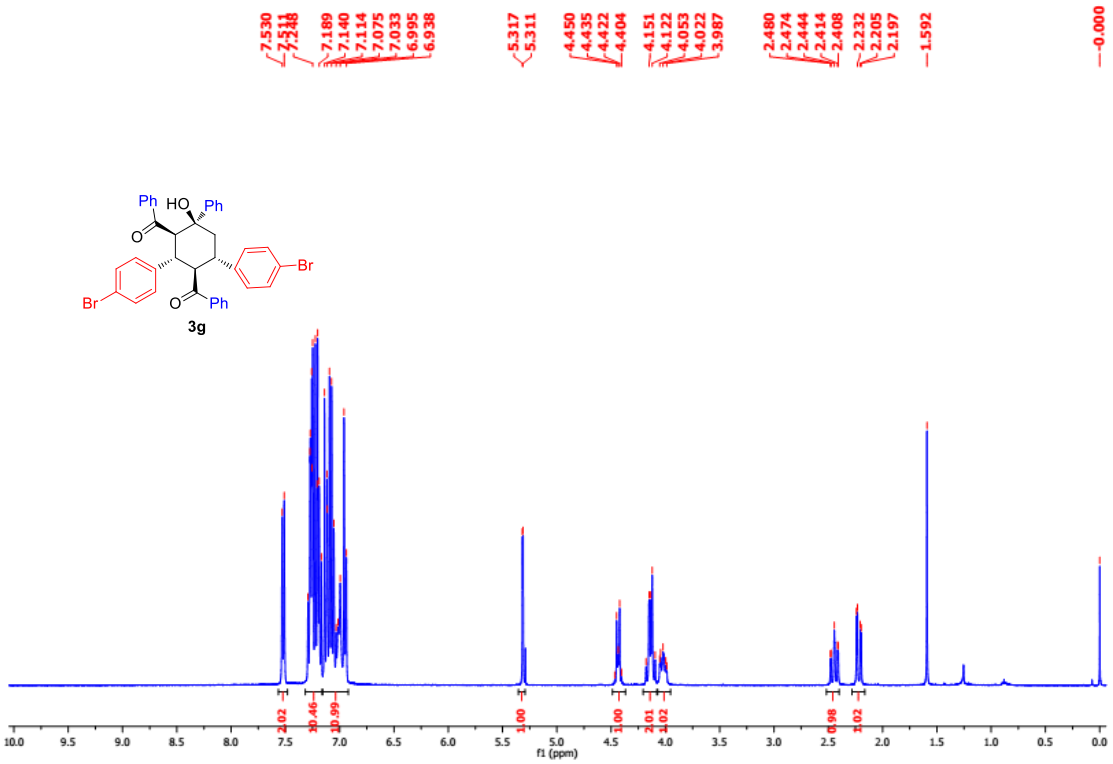
Ph
O
Cl
Cl
O
Ph
3f
f1 (ppm)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4.450
4.435
4.422
4.404
4.151
4.122
4.053
4.022
3.987
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2.444
2.414
2.408
2.232
2.205
2.197
1.592
0.000
Ph
HO
Ph
O
Br
Br
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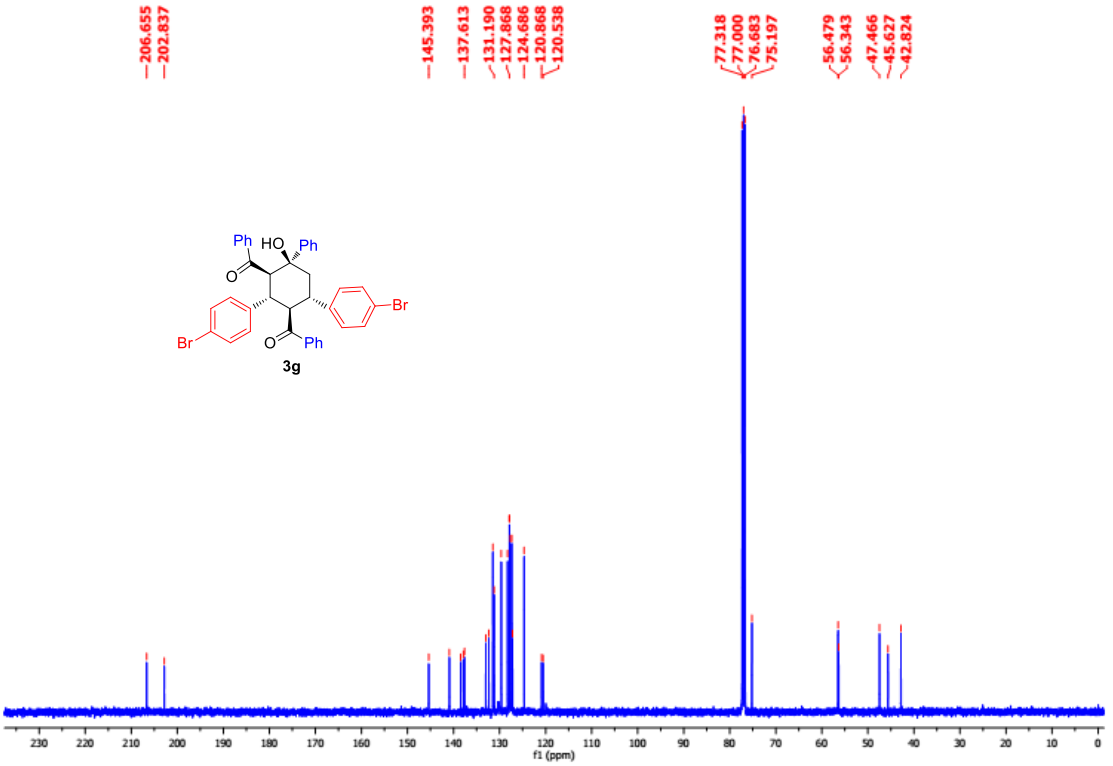
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45.627
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Br
Br
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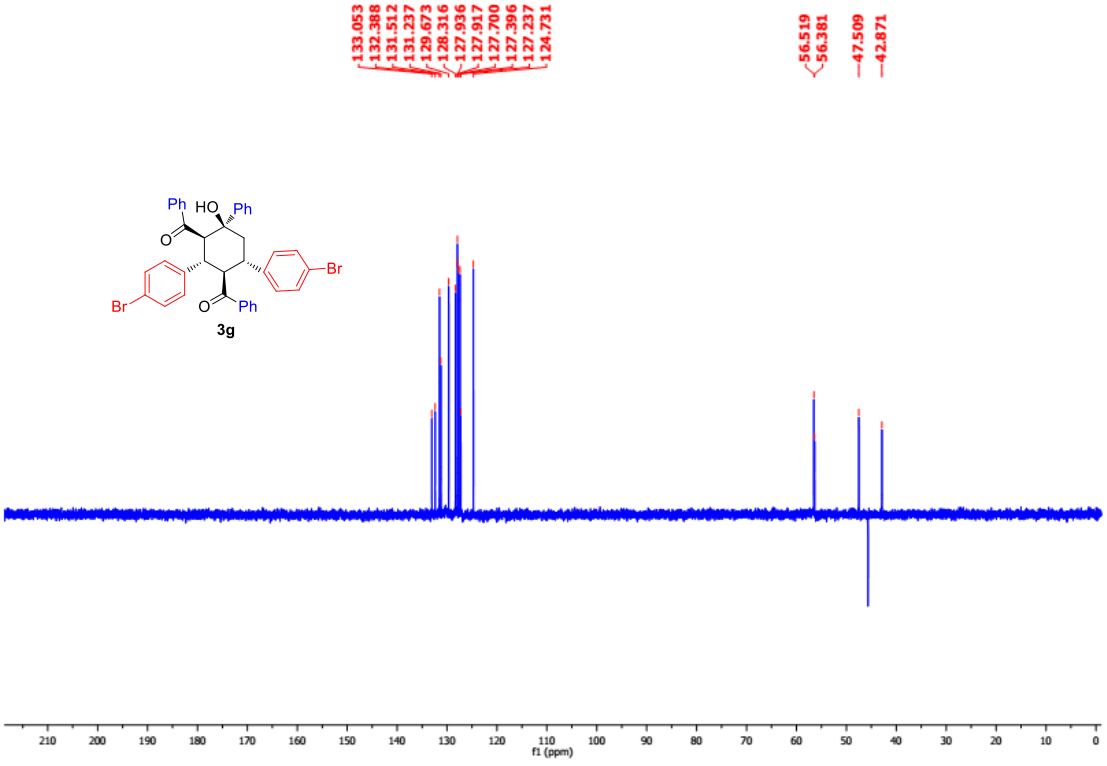
Ph
3g
f1 (ppm)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47.509
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Ph
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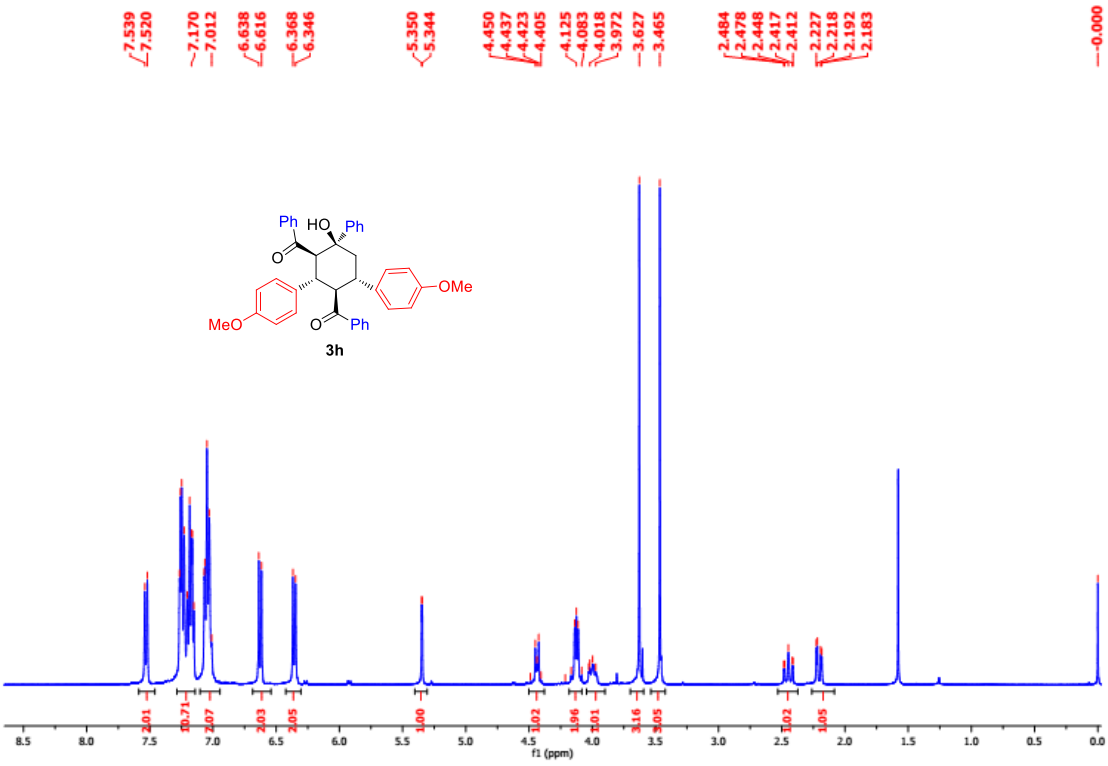
Br
Br
O
Ph
3g
f1 (ppm)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5.344
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4.437
4.423
4.405
4.125
4.083
4.018
3.972
3.627
3.465
2.484
2.478
2.448
2.417
2.412
2.227
2.218
2.192
2.183
0.000
Ph
HO
Ph
O
OMe
MeO
O
Ph
3h
2.01
10.71
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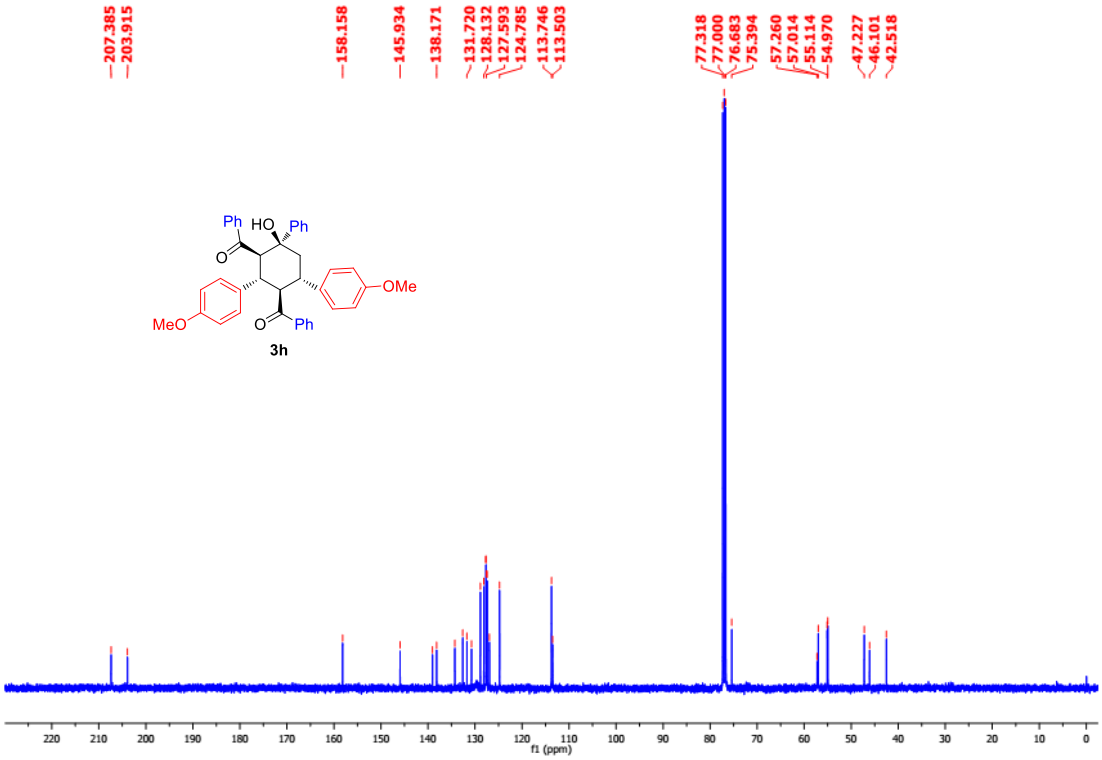
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77.000
76.683
75.394
57.260
57.014
55.114
54.970
47.227
46.101
42.518
Ph
HO
Ph
O
OMe
MeO
O
Ph
3h
220
210
200
190
180
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90
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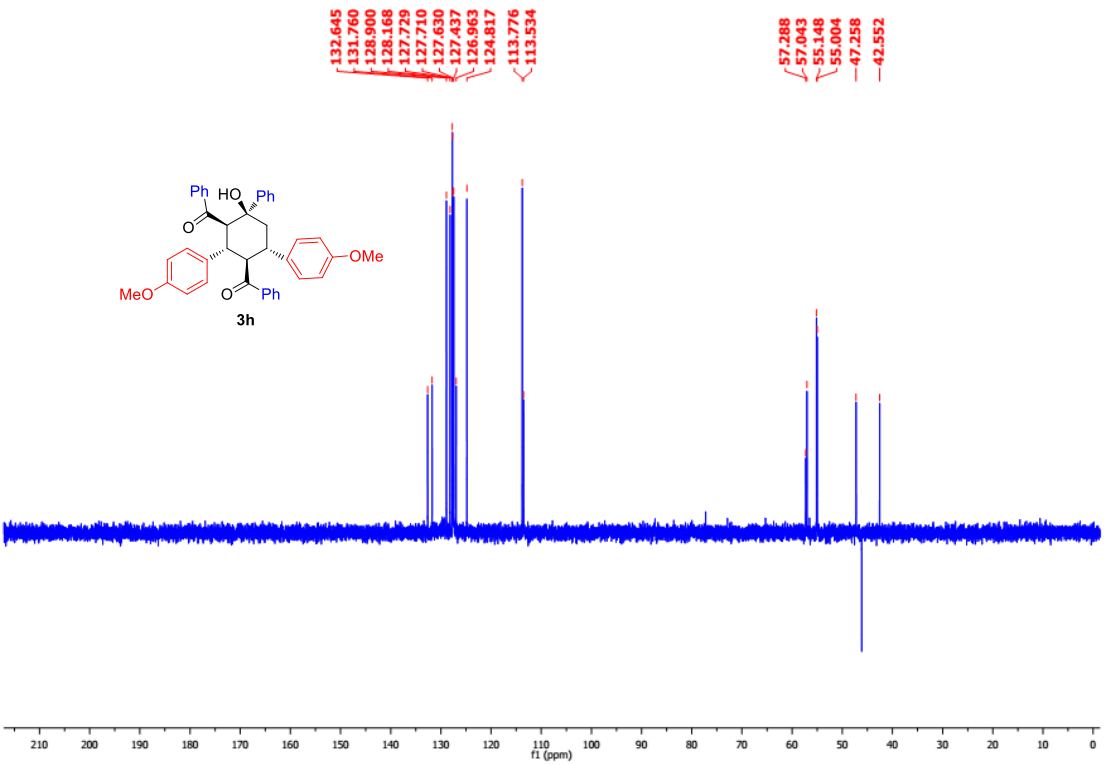
f1 (ppm)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57.043
55.148
55.004
47.258
42.552
Ph
HO
Ph
O
OMe
MeO
O
Ph
3h
210
200
190
180
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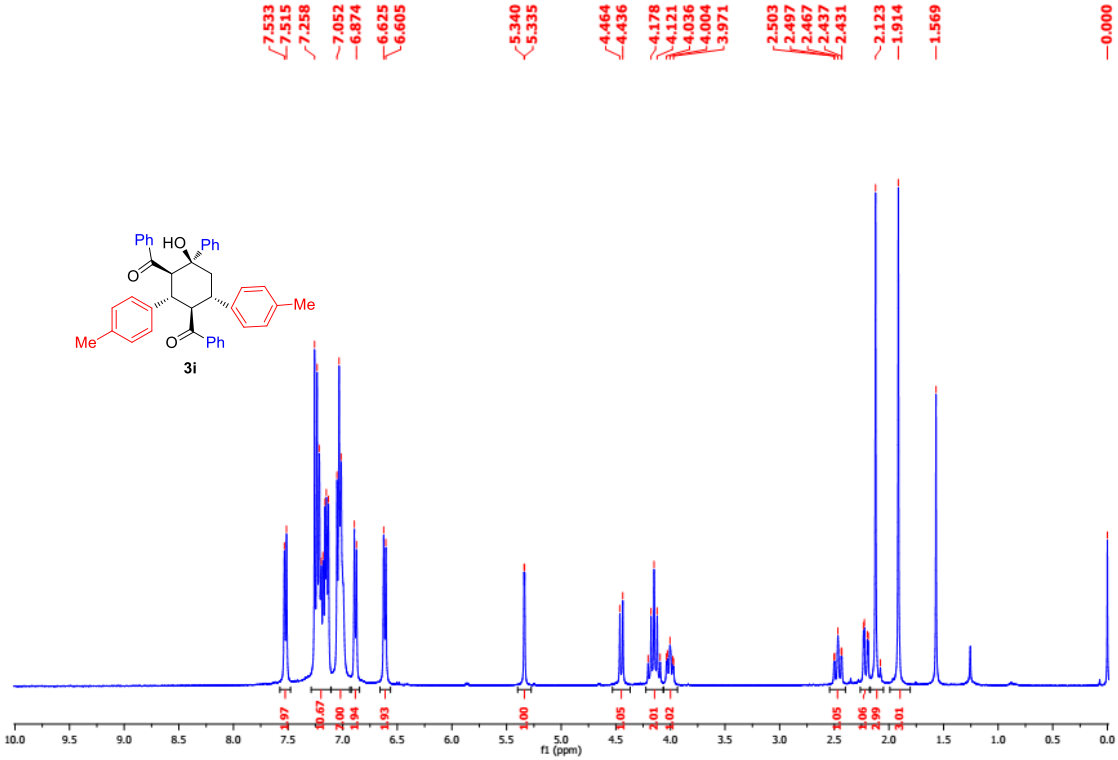
f1 (ppm)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4.036
4.004
3.971
2.503
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2.467
2.437
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2.123
1.914
1.569
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Ph
HO
Ph
O
Me
Me
O
Ph
3i
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10.67
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1.06
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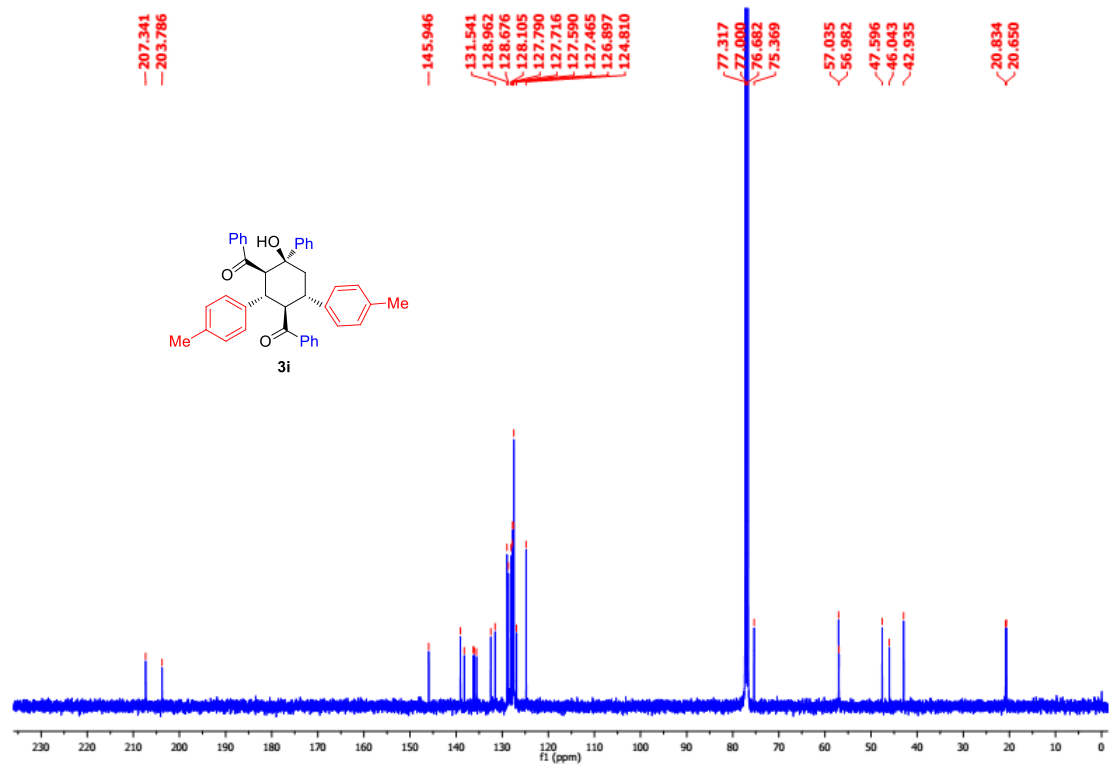
3.01
f1 (ppm)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77.000
76.682
75.369
57.035
56.982
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46.043
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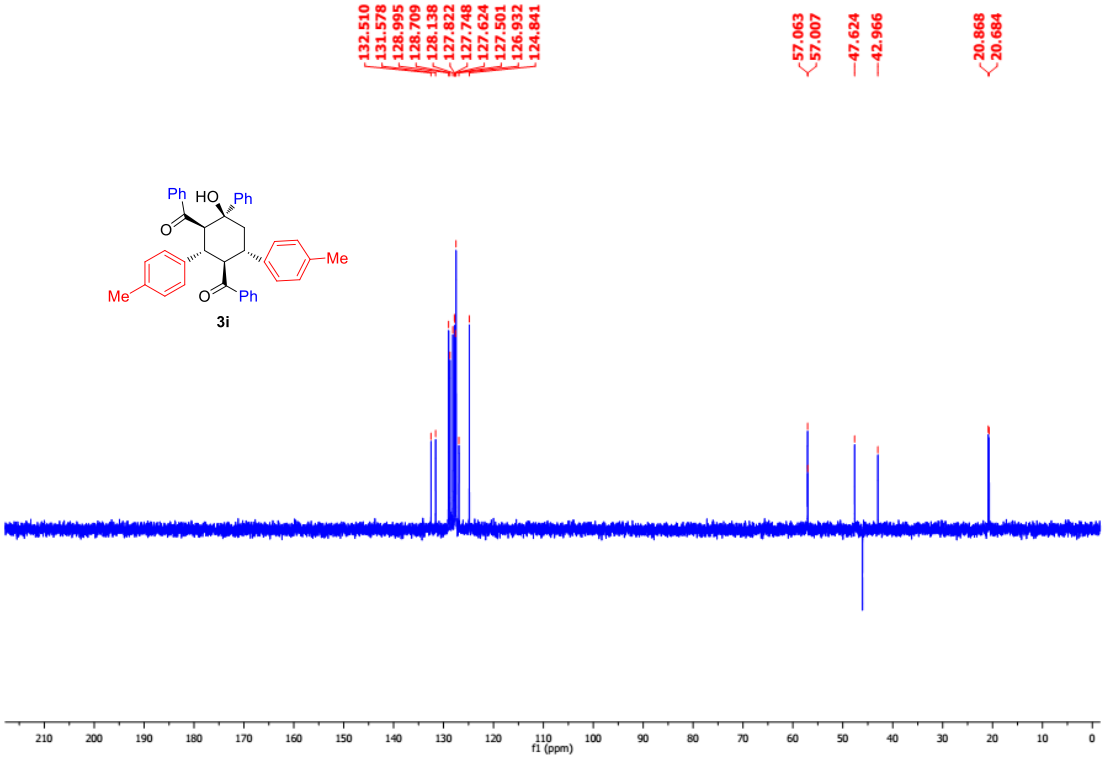
20.834
20.650
Ph
HO
Ph
O
Me
Me
O
Ph
3i
f1 (ppm)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Me
Me
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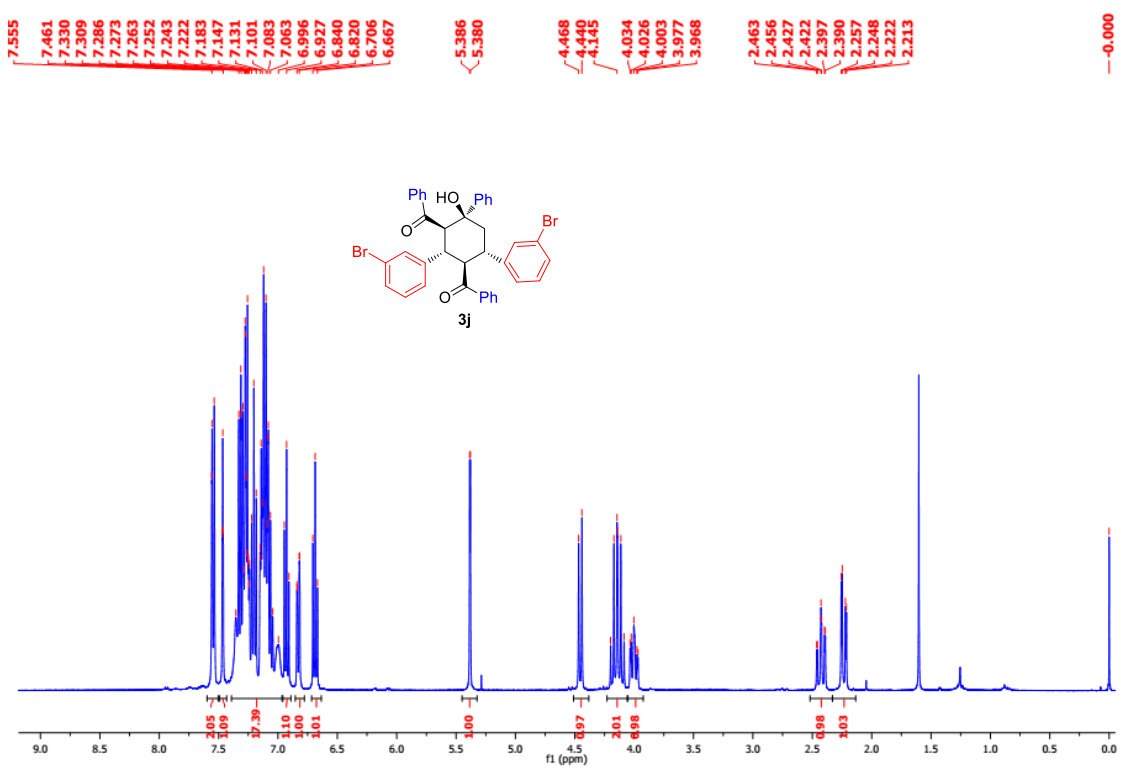
3i
f1 (ppm)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2.397
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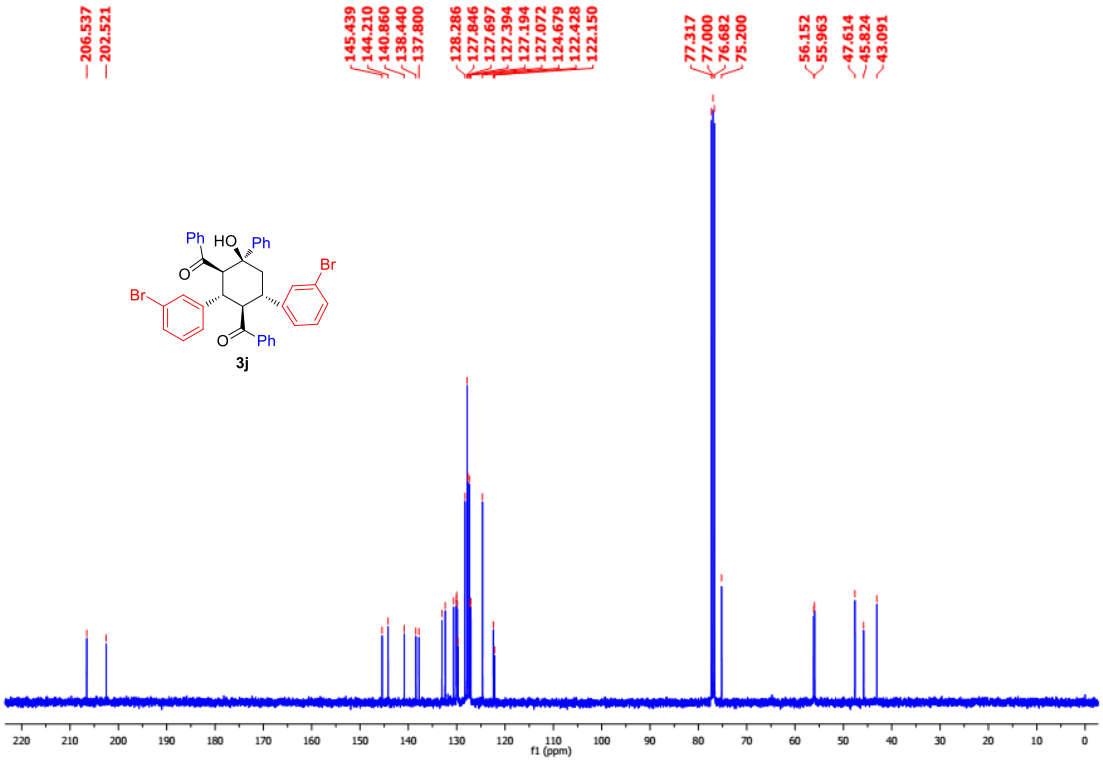
2.257
2.248
2.222
2.213
-0.000
Ph
HO
Ph
Br
Br
O
Ph
3j
f1 (ppm)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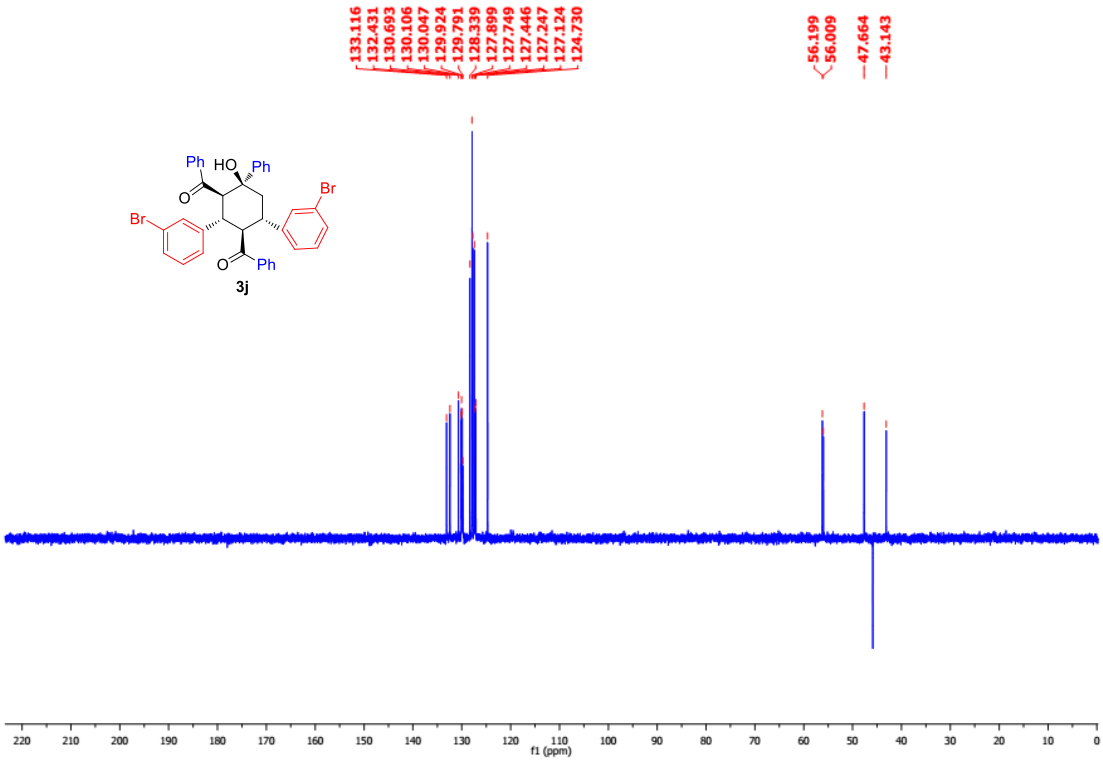
137.800
128.286
127.846
127.697
127.394
127.194
127.072
124.679
122.428
122.150
77.317
77.000
76.682
75.200
56.152
55.963
47.614
45.824
43.091
Ph
HO
Ph
Br
Br
O
O
Ph
3j
f1 (ppm)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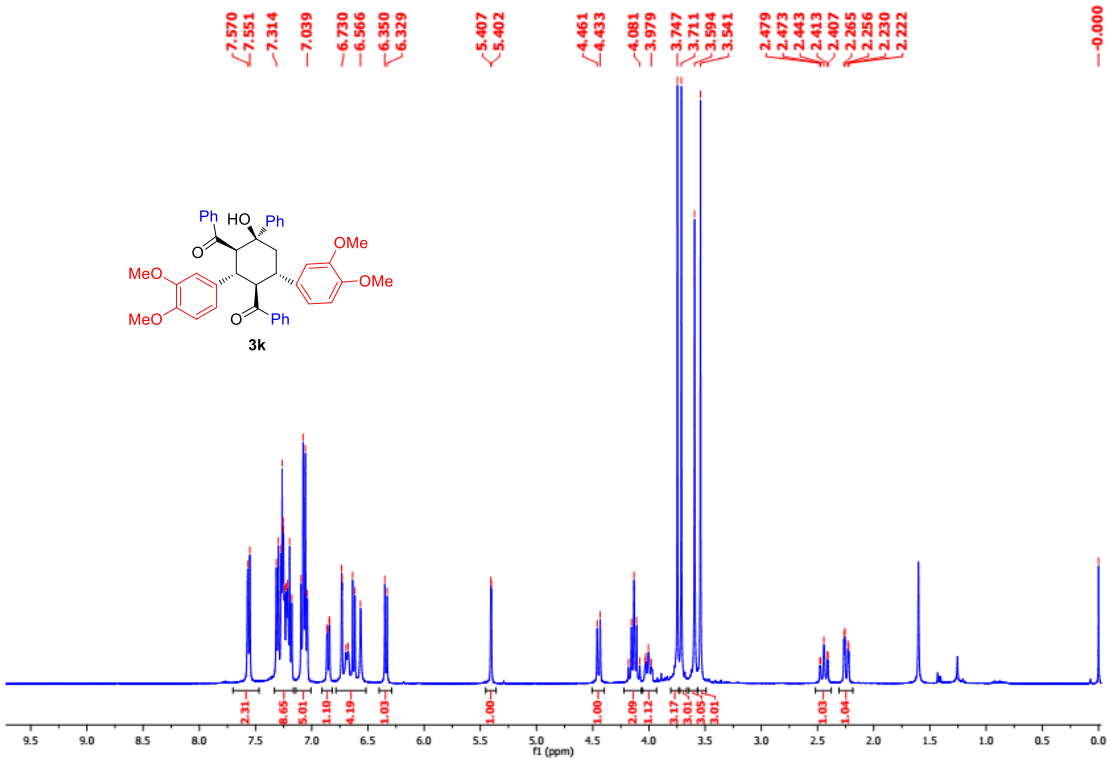
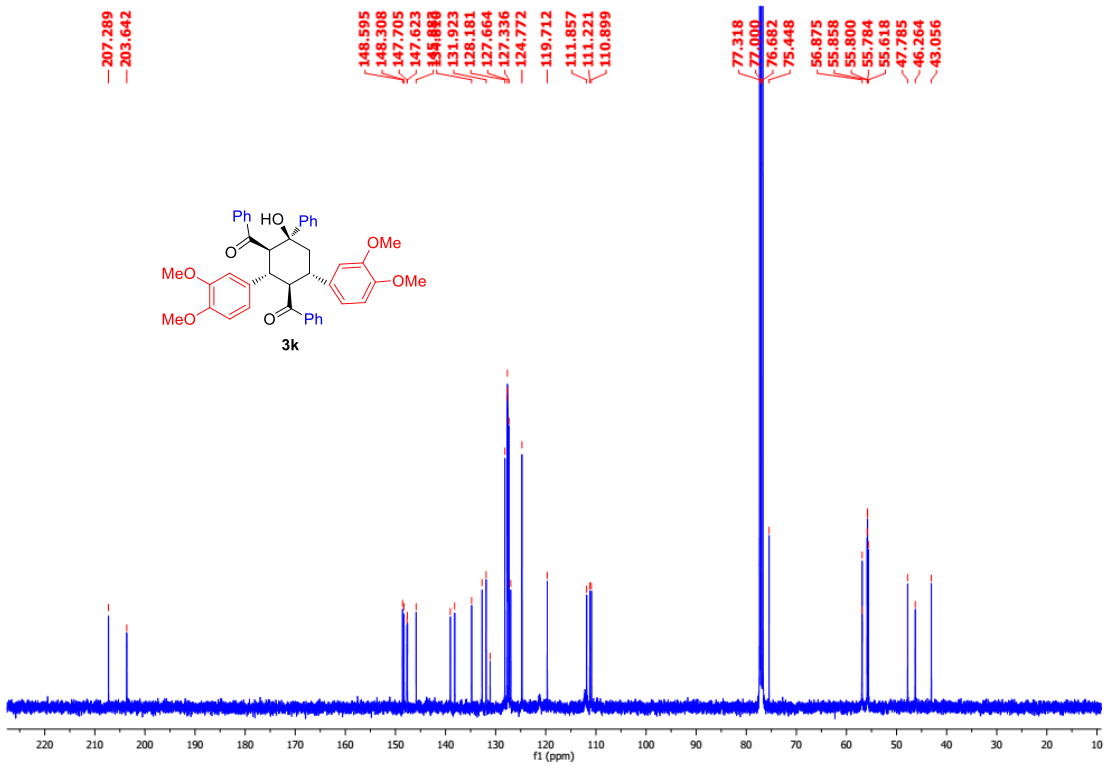
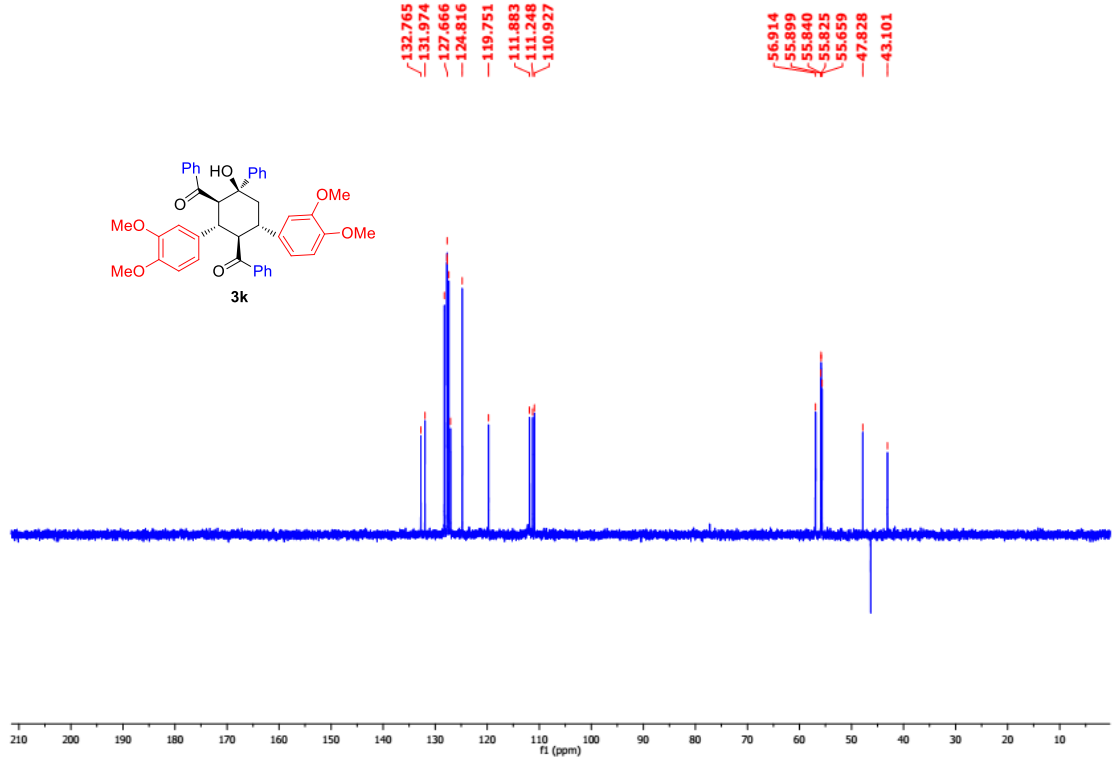
130.693
130.106
130.047
129.924
129.791
128.339
127.899
127.749
127.446
127.247
127.124
124.730
56.199
56.009
47.664
43.143
Ph
HO
Ph
Br
Br
O
O
Ph
3j
f1 (ppm)


7.570
7.551
7.314
7.039
6.730
6.566
6.350
6.329
5.407
5.402
4.461
4.433
4.081
3.979
3.747
3.711
3.594
3.541
2.479
2.473
2.443
2.413
2.407
2.265
2.256
2.230
2.222
0.000
Ph
HO
Ph
OMe
OMe
MeO
MeO
O
O
Ph
3k
f1 (ppm)


207.289
203.642
148.595
148.308
147.705
147.623
131.923
128.181
127.664
127.336
124.772
119.712
111.857
111.221
110.899
77.318
77.000
76.682
75.448
56.875
55.858
55.800
55.784
55.618
47.785
46.264
43.056
Ph
HO
Ph
OMe
OMe
MeO
MeO
O
O
Ph
3k
f1 (ppm)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131.974
127.666
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119.751
111.883
111.248
110.927
56.914
55.899
55.840
55.825
55.659
47.828
43.101
Ph
HO
Ph
OMe
OMe
MeO
MeO
O
O
Ph
3k
f1 (ppm)


7.268
7.254
7.234
7.230
7.219
7.200
7.196
7.185
7.175
7.096
7.078
7.060
7.042
7.024
6.970
6.951
6.933
6.922
6.903
6.883
6.822
6.802
6.783
5.461
5.455
4.655
4.442
4.414
4.387
4.332
4.324
4.301
4.275
4.266
2.698
2.692
2.662
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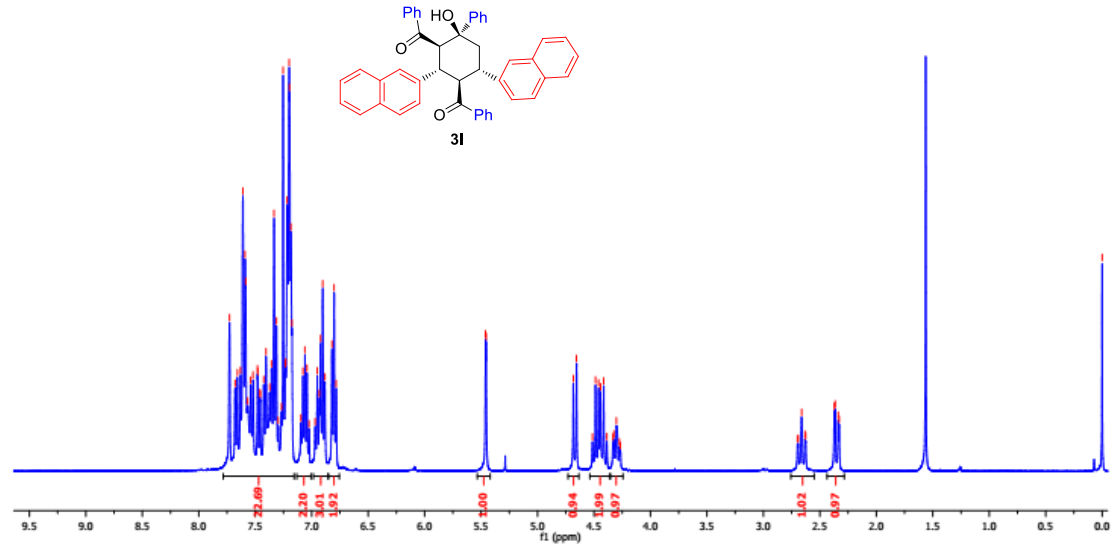
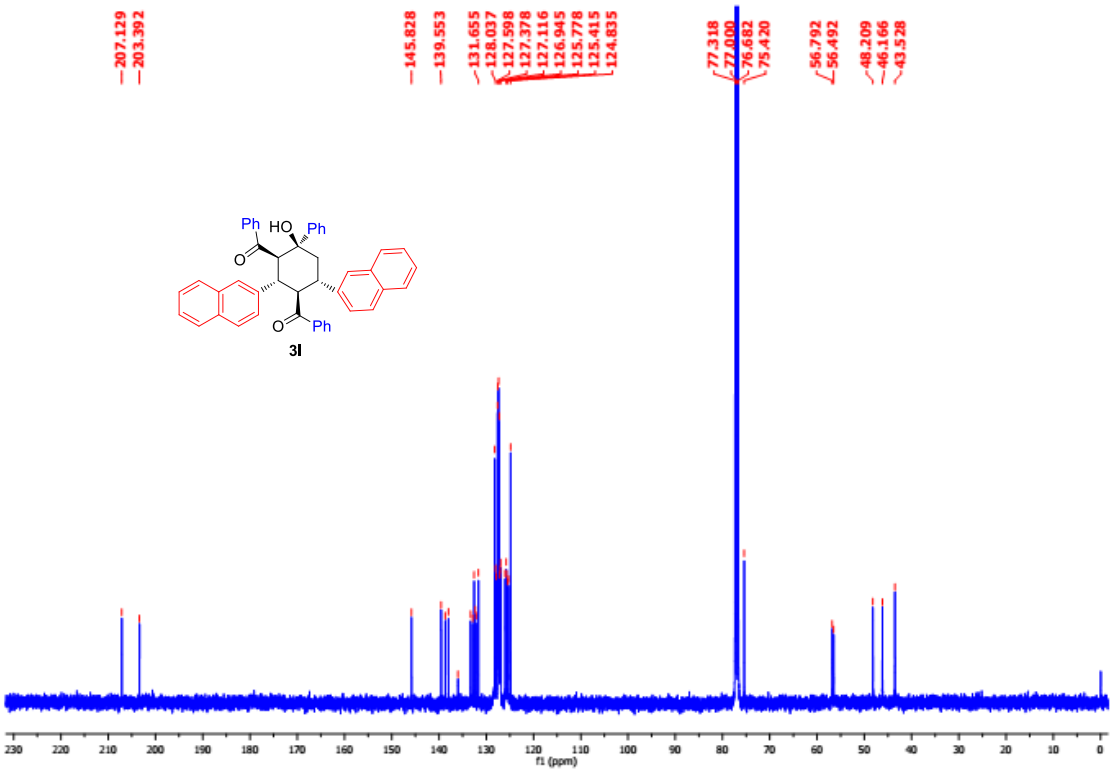
2.625
2.373
2.364
2.338
2.329
0.000


Ph
HO
Ph
O
O
Ph
3l
22.69
3.20
3.01
1.92
1.00
0.94
1.99
0.97
1.02
0.97
f1 (ppm)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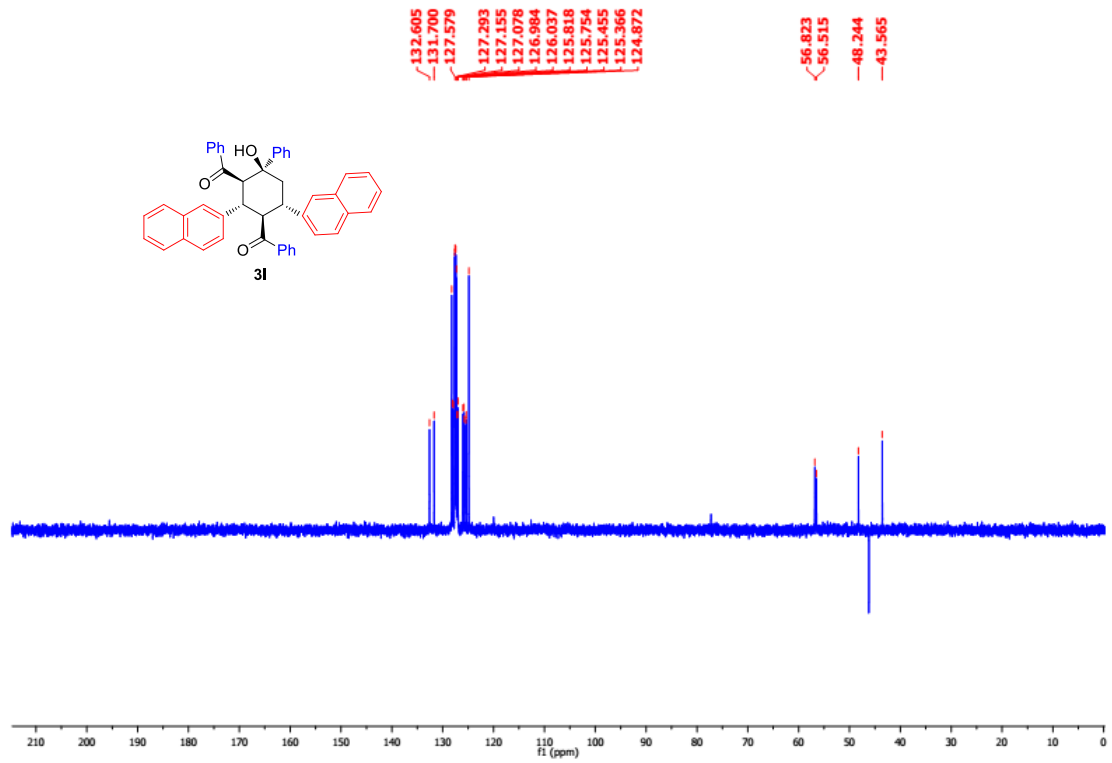
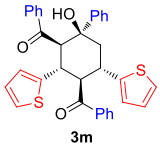
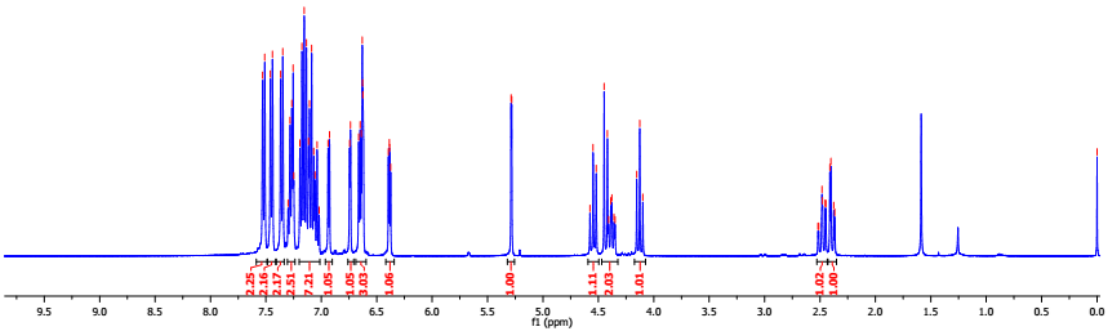
145.828
139.553
131.655
128.037
127.598
127.378
127.116
126.945
125.778
125.415
124.835
77.318
77.000
76.682
75.420
56.792
56.492
48.209
46.166
43.528
Ph
HO
Ph
O
O
Ph
3l
f1 (ppm)

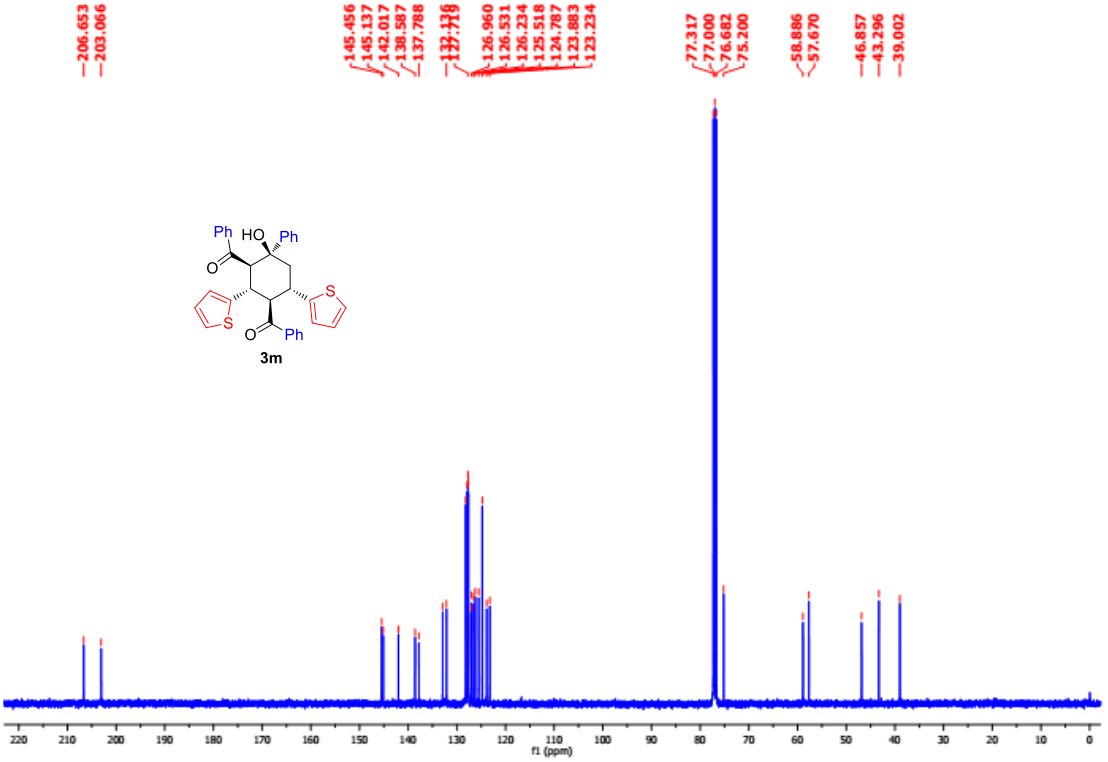

132.605
131.700
127.579
127.293
127.155
127.078
126.984
126.037
125.818
125.754
125.455
125.366
124.872
56.823
56.515
48.244
43.565
Ph
HO
Ph
O
O
Ph
3l
f1 (ppm)


7.348
7.300
7.282
7.267
7.255
7.249
7.194
7.175
7.154
7.134
7.115
7.107
7.087
7.068
7.056
7.038
7.020
6.939
6.926
6.624
6.395
6.386
6.383
6.374
5.288
5.282
4.448
4.407
4.379
4.349
4.154
4.127
4.099
2.518
2.512
2.483
2.477
2.453
2.447
2.411
2.401
2.376
2.366
0.000


Ph
HO
Ph
O
S
S
O
Ph

3m


2.25
2.16
2.17
2.51
7.21
1.05
1.05
3.03
1.06
1.00
1.11
2.03
1.01
1.02
1.00
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
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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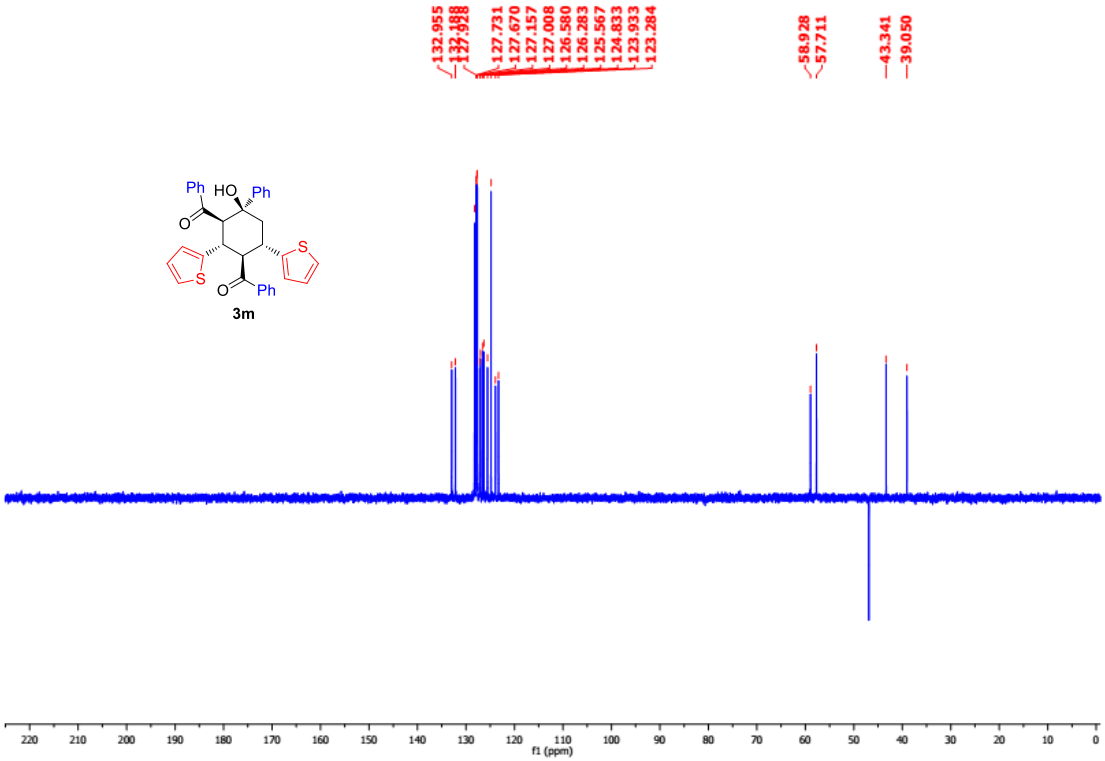
203.066
145.456
145.137
142.017
138.587
137.788
126.960
126.531
126.234
125.518
124.787
123.883
123.234
77.317
77.000
76.682
75.200
58.886
57.670
46.857
43.296
39.002
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Ph
HO
Ph
O
S
S
O
Ph

3m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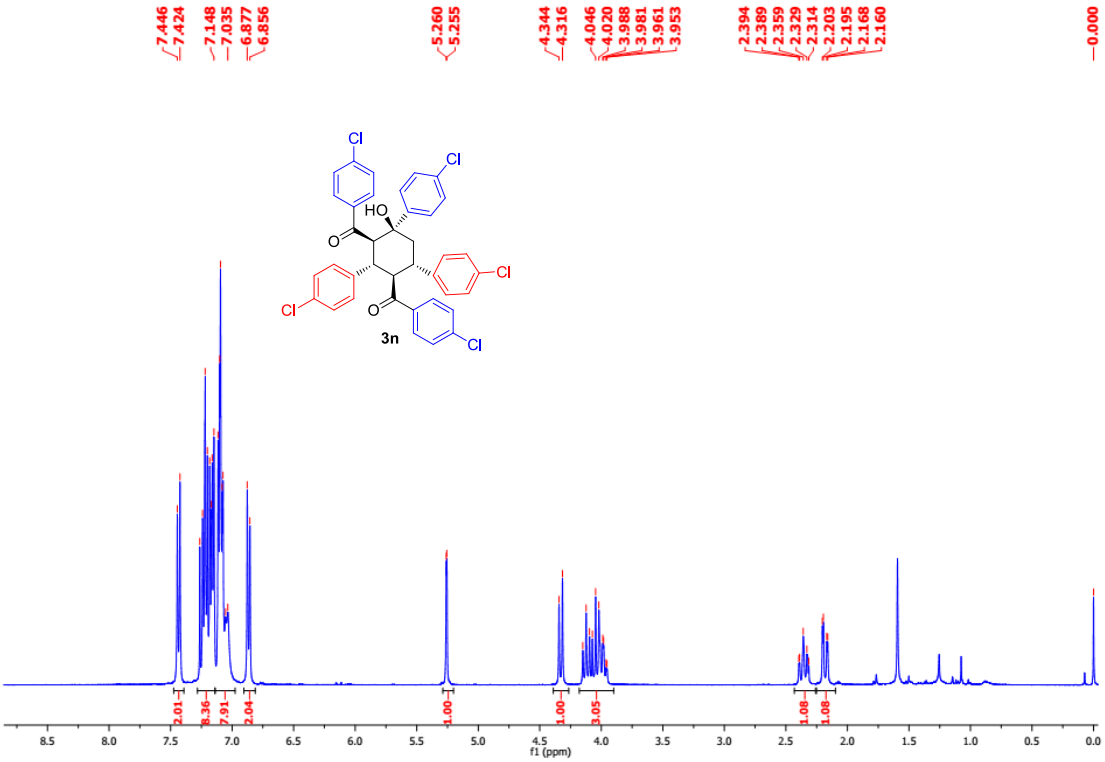
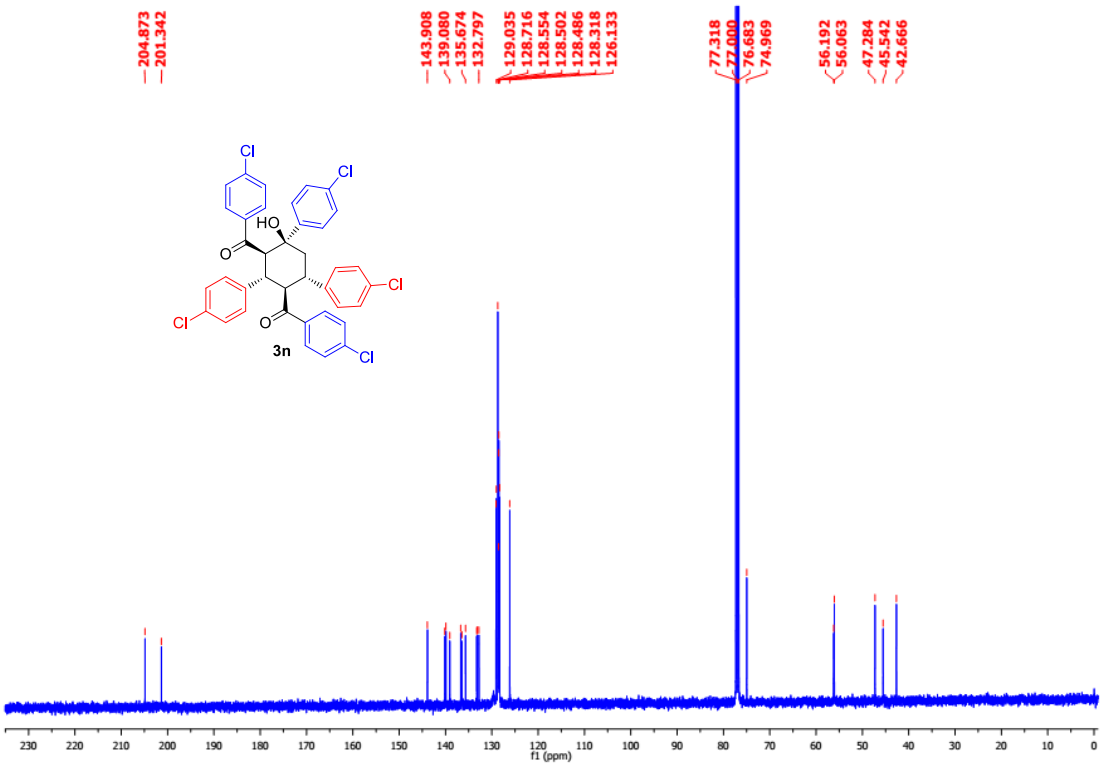
132.185
131.958
131.938
127.731
127.670
127.157
127.008
126.580
126.283
125.567
124.833
123.933
123.284
58.928
57.711
43.341
39.050
Ph
HO
Ph
O
S
S
O
Ph
3m
f1 (ppm)


7.446
7.424
7.148
7.035
6.877
6.856
5.260
5.255
4.344
4.316
4.046
4.020
3.988
3.981
3.961
3.953
2.394
2.389
2.359
2.329
2.314
2.203
2.195
2.168
2.160
0.000
Cl
Cl
HO
O
Cl
Cl
O
Cl
3n
2.01
8.36
7.91
2.04
1.00
1.00
3.05
1.08
1.08
f1 (ppm)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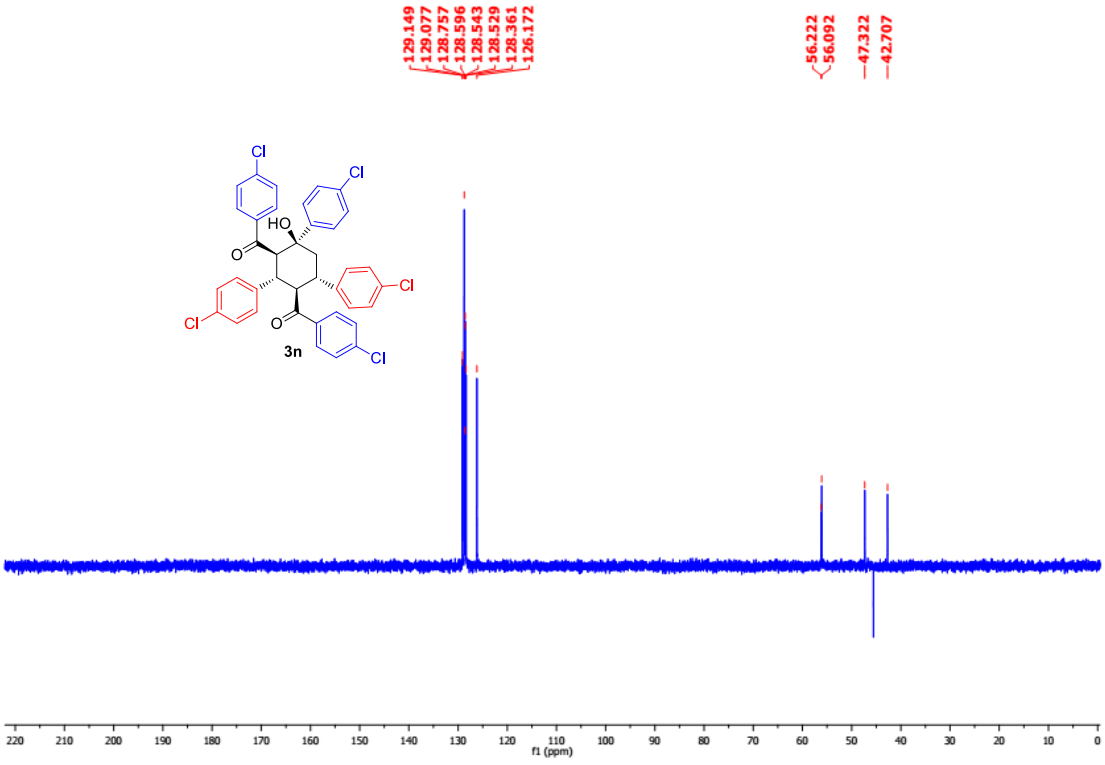
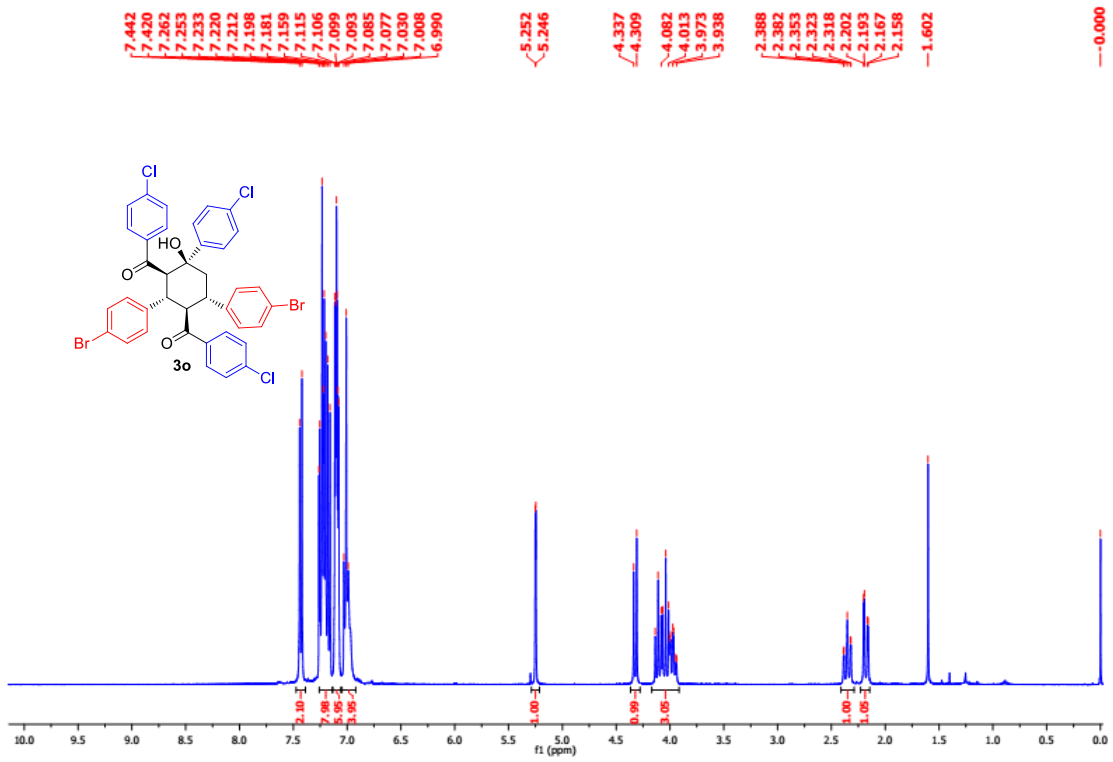
201.342
143.908
139.080
135.674
132.797
129.035
128.716
128.554
128.502
128.486
128.318
126.133
77.318
77.000
76.683
74.969
56.192
56.063
47.284
45.542
42.666
Cl
Cl
HO
O
Cl
Cl
O
Cl
3n
f1 (ppm)


129.149
129.077
128.757
128.596
128.543
128.529
128.361
126.172
56.222
56.092
47.322
42.707
Cl
Cl
HO
O
Cl
Cl
O
Cl
3n
f1 (ppm)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7.420
7.262
7.253
7.233
7.220
7.212
7.198
7.181
7.159
7.115
7.106
7.099
7.093
7.085
7.077
7.030
7.008
6.990
5.252
5.246
4.337
4.309
4.082
4.013
3.973
3.938
2.388
2.382
2.353
2.323
2.318
2.202
2.193
2.167
2.158
1.602
0.000
Cl
Cl
HO
O
Br
Br
O
Cl
3o
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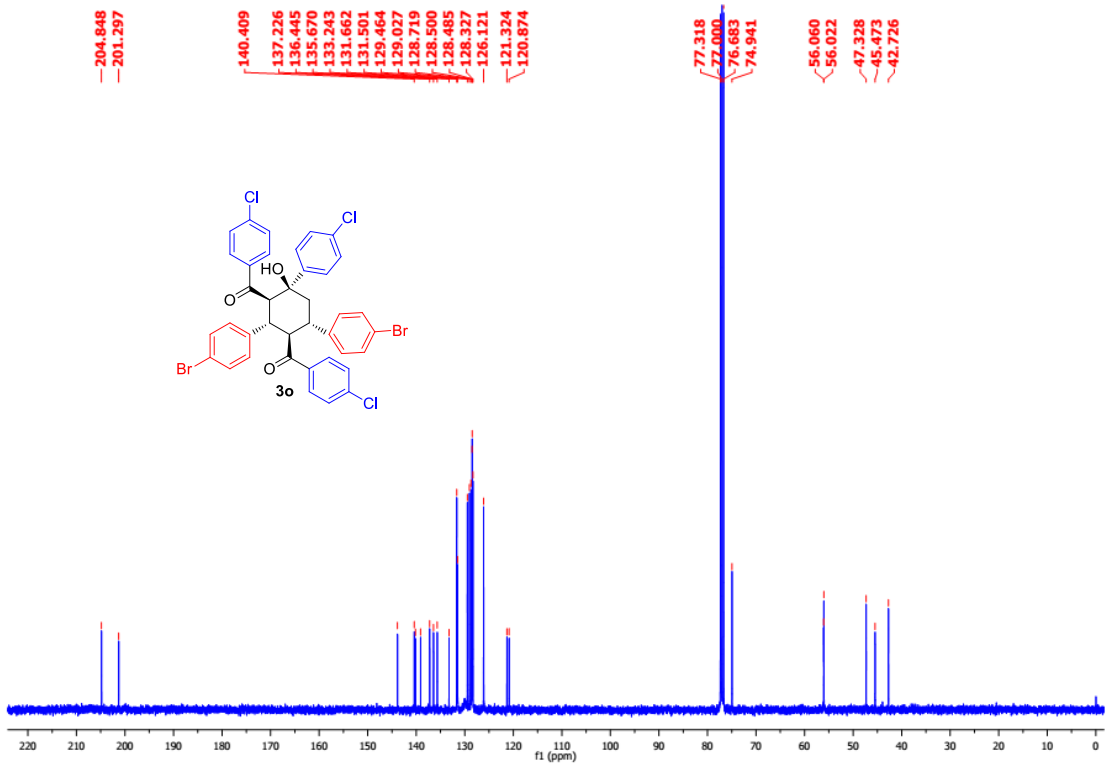
7.98
5.95
3.99
1.00
0.99
3.05
1.00
1.05
f1 (ppm)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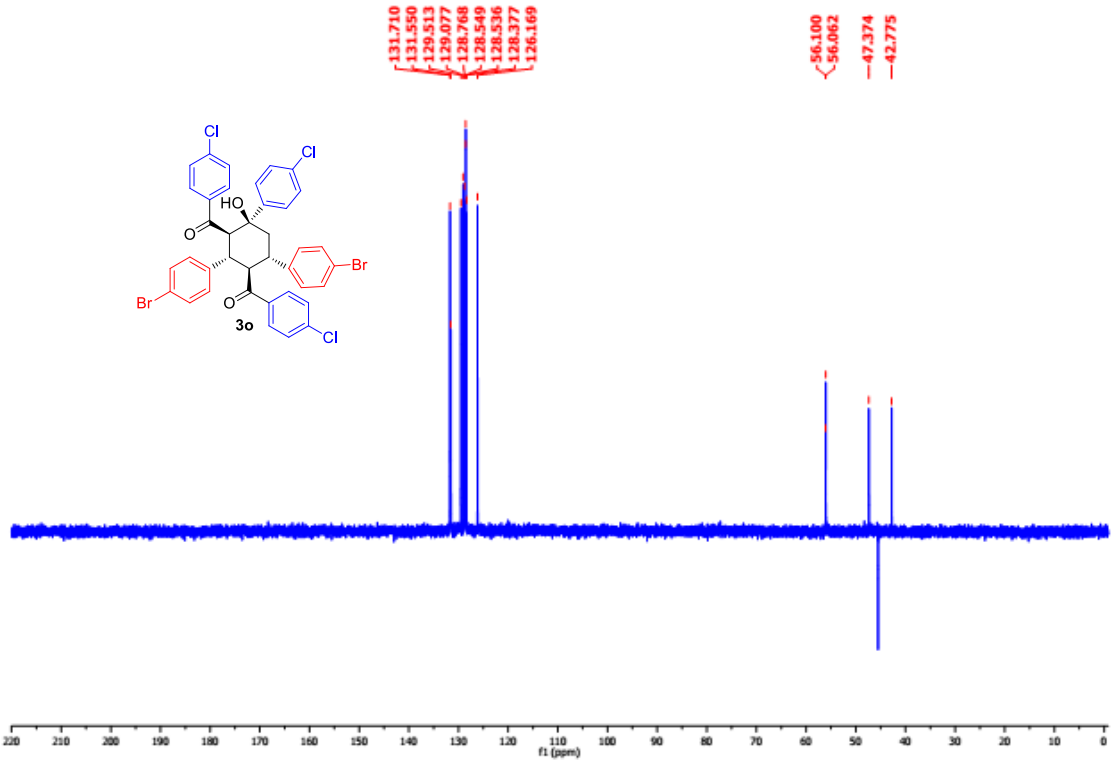
135.670
133.243
131.662
131.501
129.464
129.027
128.719
128.500
128.485
128.327
126.121
121.324
120.874
77.318
77.000
76.683
74.941
56.060
56.022
47.328
45.473
42.726
Cl
Cl
HO
O
Br
Br
O
Cl
3o
f1 (ppm)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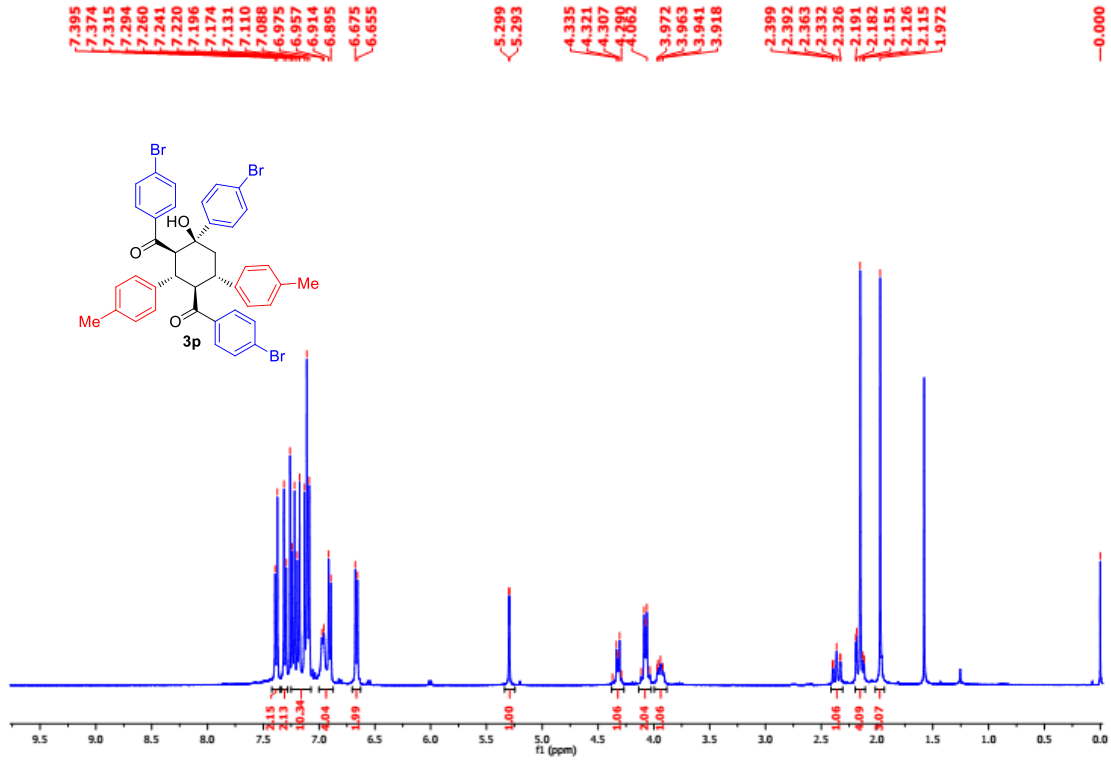
131.550
129.513
129.077
128.768
128.549
128.536
128.377
126.169
56.100
56.062
47.374
42.775
Cl
Cl
HO
O
Br
Br
O
Cl
3o
f1 (ppm)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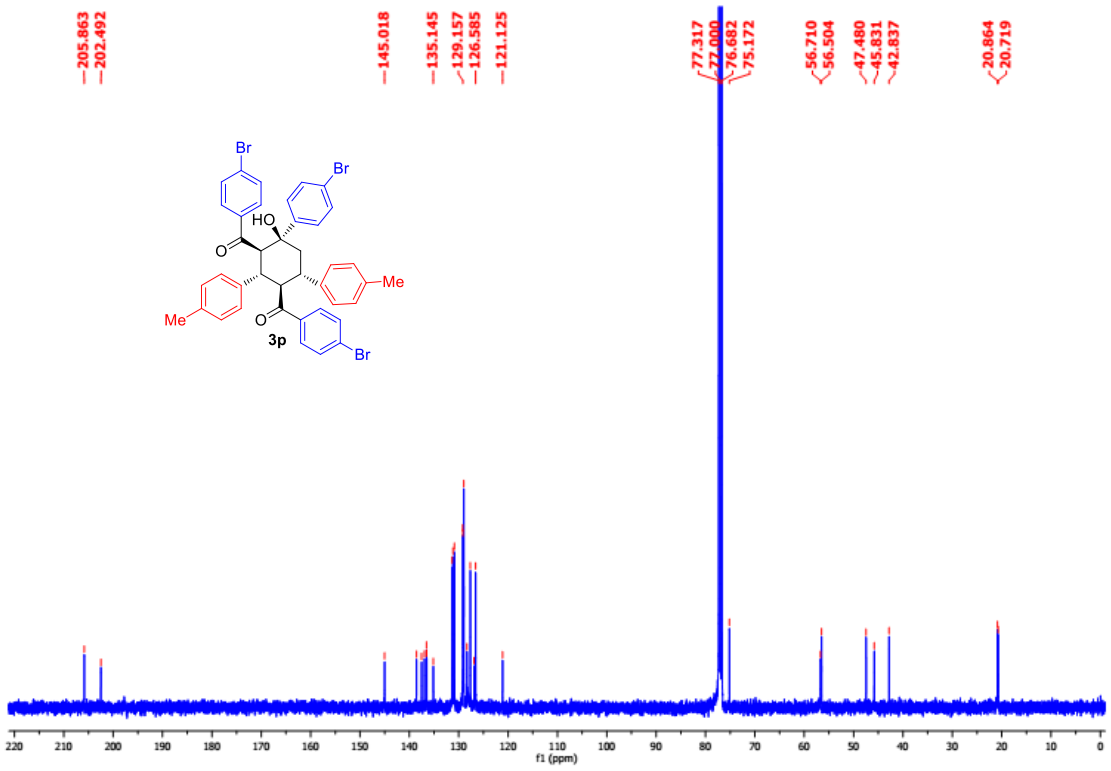
7.374
7.315
7.294
7.260
7.241
7.220
7.196
7.174
7.131
7.110
7.088
6.975
6.957
6.914
6.895
6.675
6.655
5.299
5.293
4.335
4.321
4.307
4.280
4.082
3.972
3.963
3.941
3.918
2.399
2.392
2.363
2.332
2.326
2.191
2.182
2.151
2.126
2.115
1.972
0.000
Br
Br
HO
O
Me
Me
O
3p
Br
f1 (ppm)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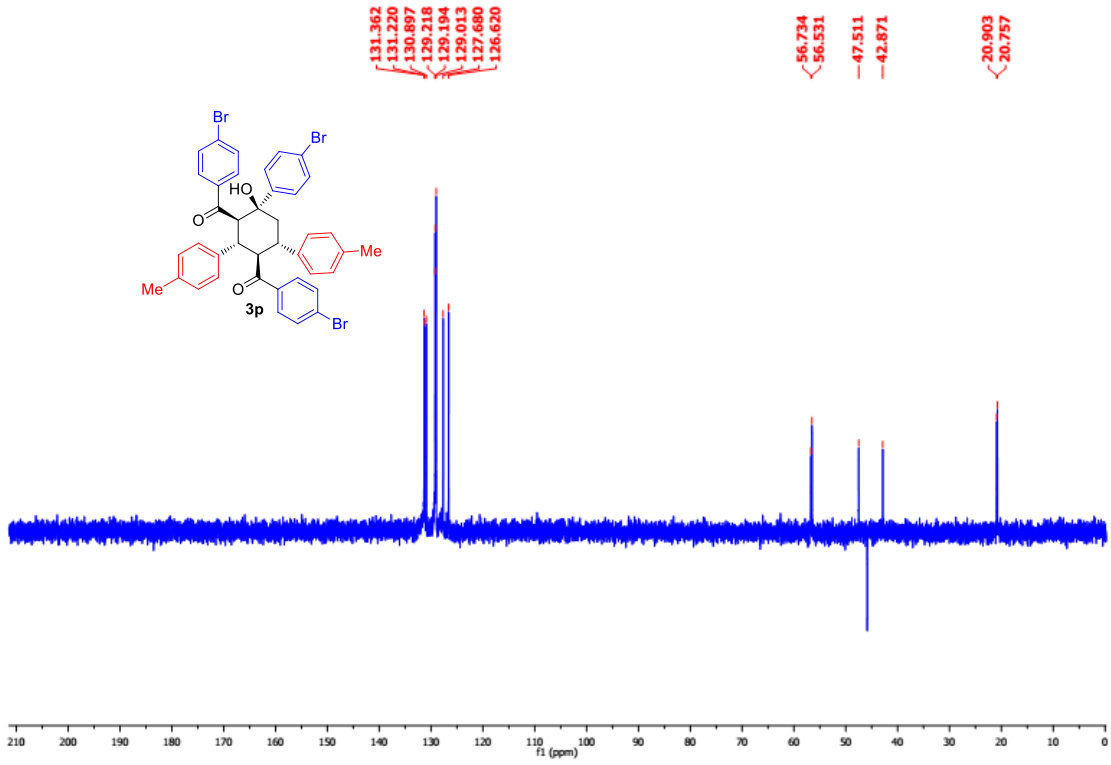
202.492
145.018
135.145
129.157
126.585
121.125
77.317
77.000
76.682
75.172
56.710
56.504
47.480
45.831
42.837
20.864
20.719
Br
Br
HO
O
Me
Me
O
3p
Br
f1 (ppm)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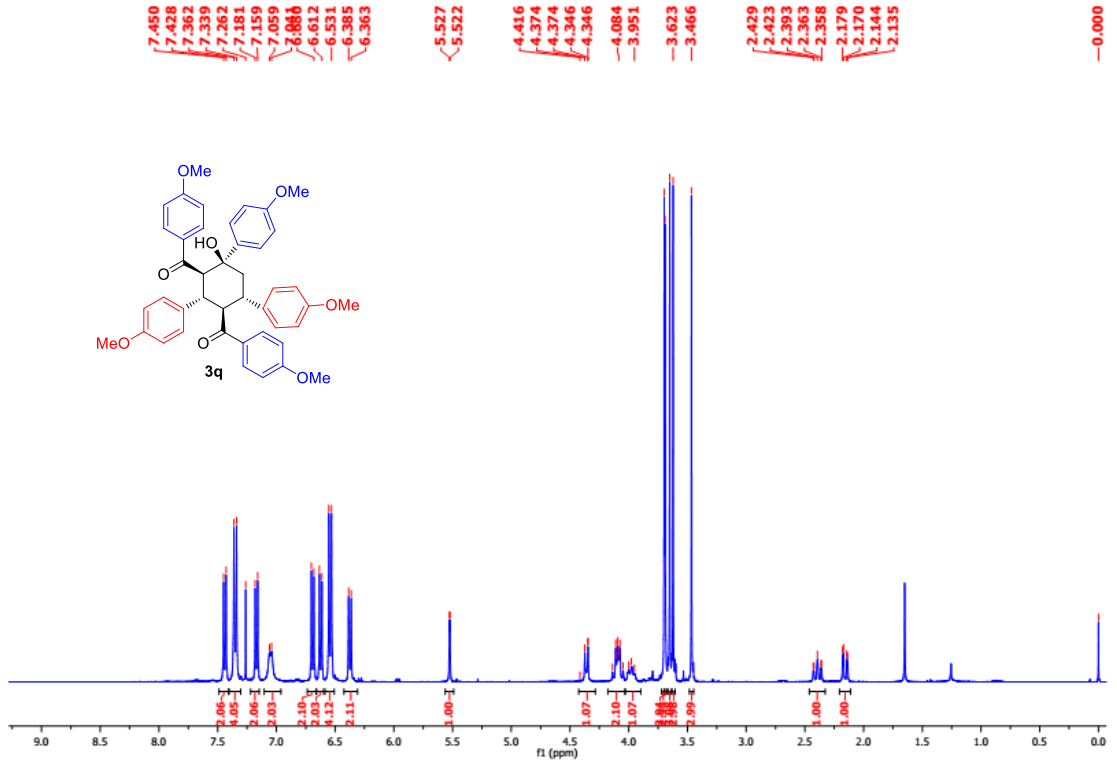
131.220
130.897
129.218
129.194
129.013
127.680
126.620
56.734
56.531
47.511
42.871
20.903
20.757
Br
Br
HO
O
Me
Me
O
3p
Br
f1 (ppm)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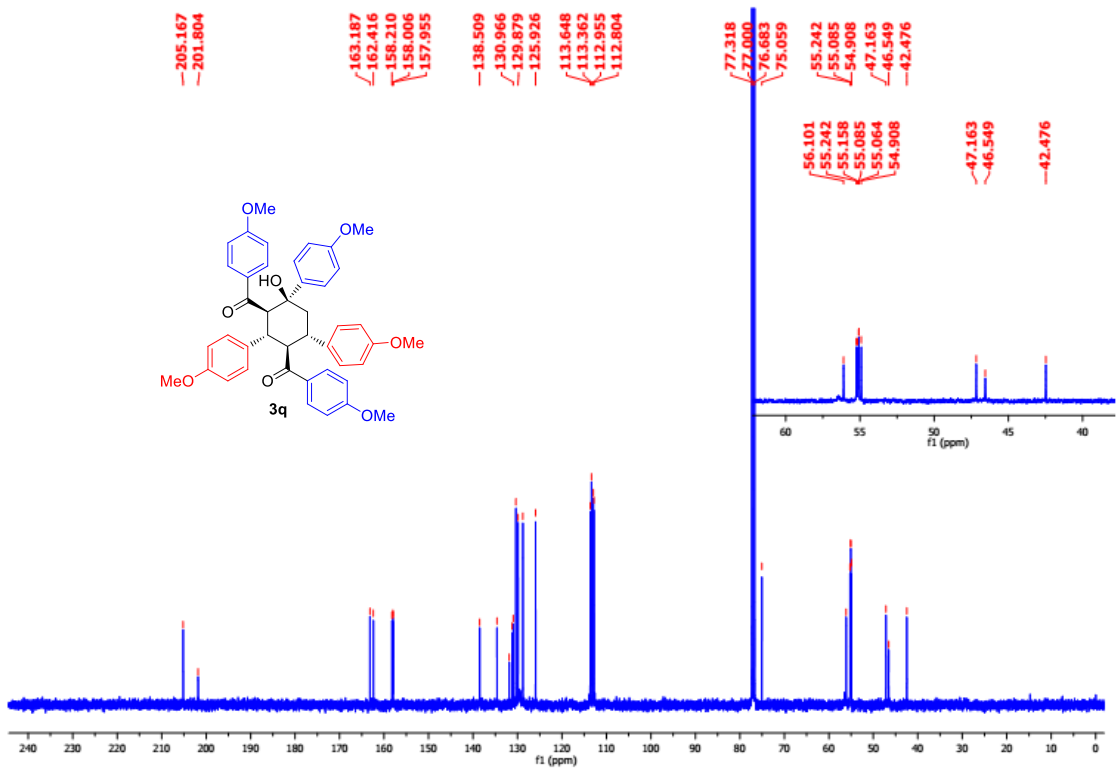
7.428
7.362
7.339
7.262
7.181
7.159
7.059
6.612
6.531
6.385
6.363
5.527
5.522
4.416
4.374
4.374
4.346
4.346
4.084
3.951
3.623
3.466
2.429
2.423
2.393
2.363
2.358
2.179
2.170
2.144
2.135
0.000
OMe
OMe
HO
O
OMe
MeO
O
OMe
3q
f1 (ppm)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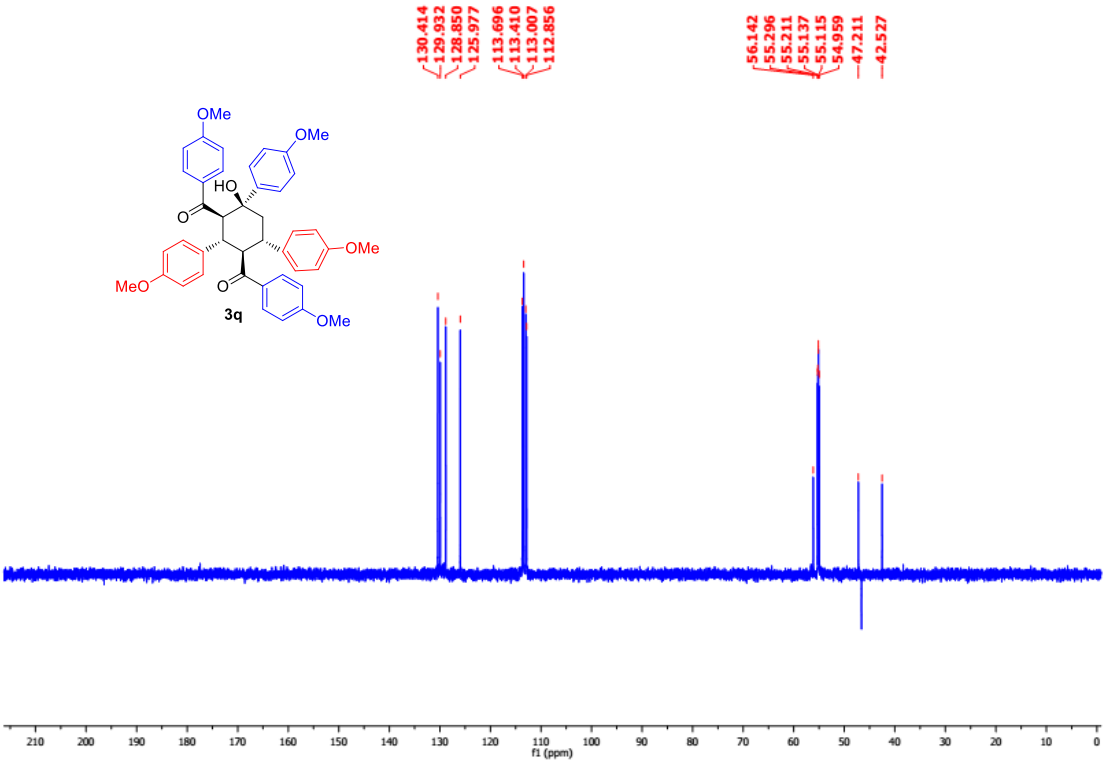
138.509
130.966
129.879
125.926
113.648
113.362
112.955
112.804
77.318
77.000
76.683
75.059
55.242
55.085
54.908
47.163
46.549
42.476
56.101
55.242
55.158
55.085
55.064
54.908
47.163
46.549
42.476
OMe
OMe
HO
O
OMe
MeO
O
OMe
3q
f1 (ppm)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128.850
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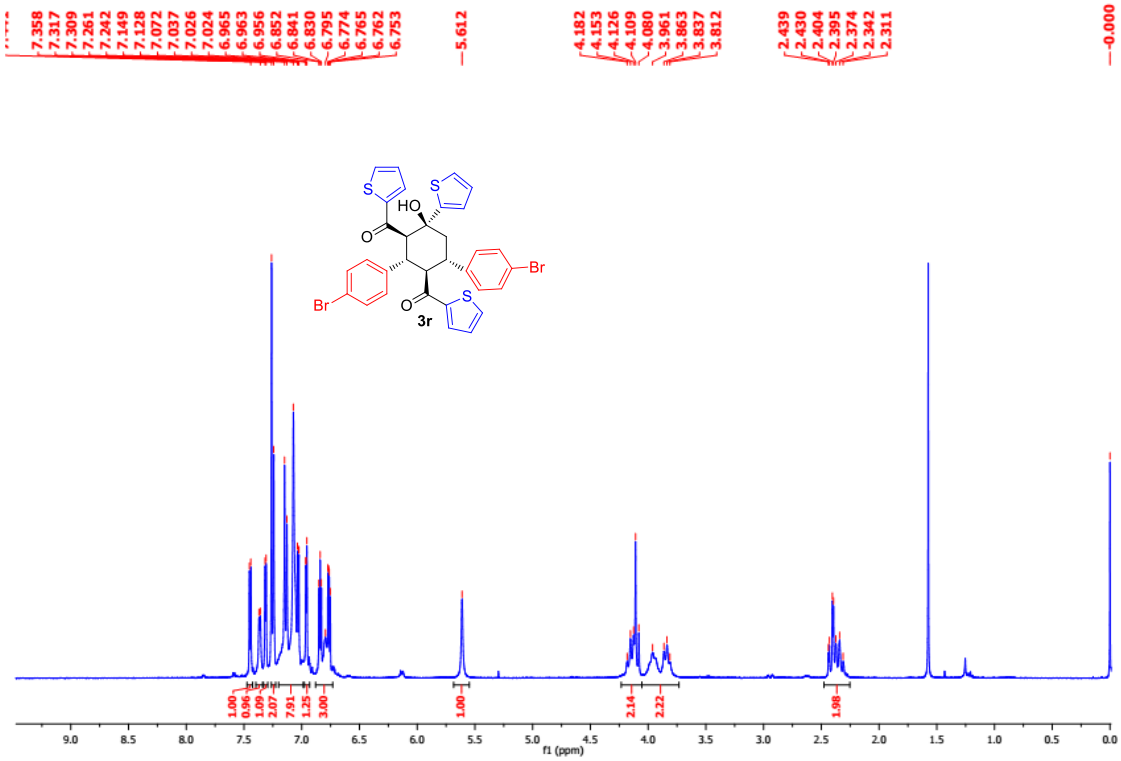
113.410
113.007
112.856
56.142
55.296
55.211
55.137
55.115
54.959
47.211
42.527
OMe
OMe
HO
O
OMe
MeO
O
OMe
3q
f1 (ppm)


7.358
7.317
7.309
7.261
7.242
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7.128
7.072
7.037
7.026
7.024
6.965
6.963
6.956
6.852
6.841
6.830
6.795
6.774
6.765
6.762
6.753
5.612
4.182
4.153
4.126
4.109
4.080
3.961
3.863
3.837
3.812
2.439
2.430
2.404
2.395
2.374
2.342
2.311
0.000
S
HO
S
O
Br
Br
O
S
3r
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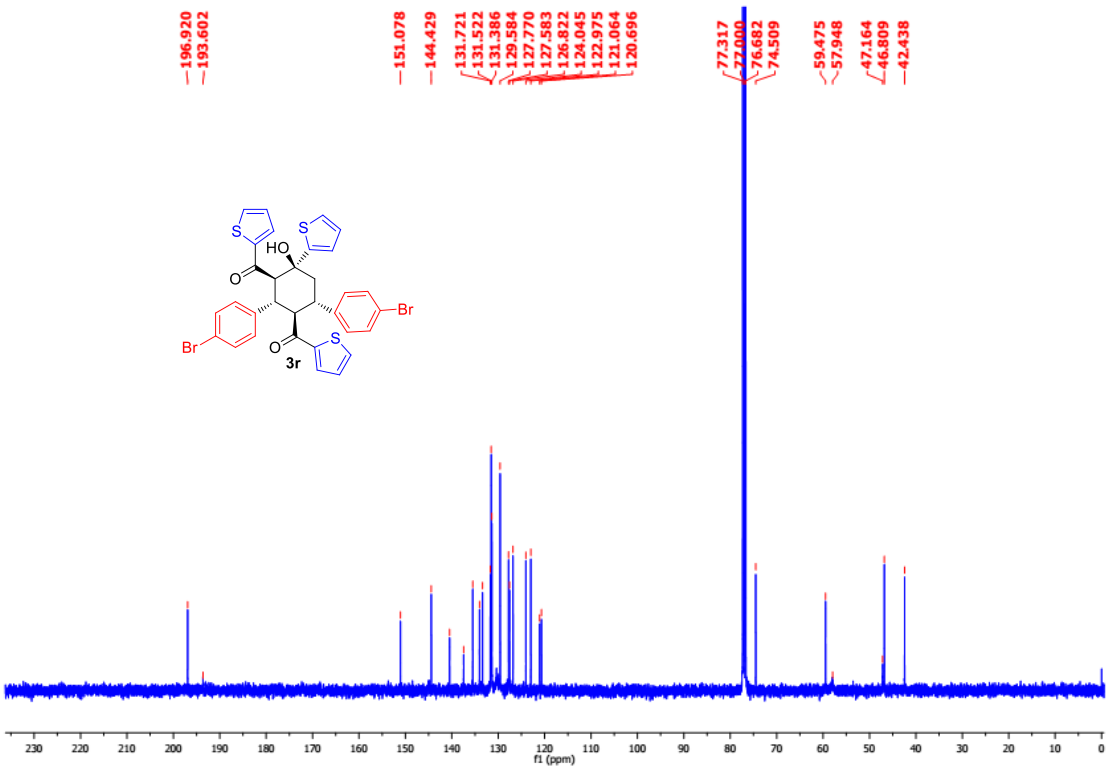
0.96
1.09
2.07
7.91
1.25
3.00
1.00
2.14
2.22
1.98
f1 (ppm)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193.602
151.078
144.429
131.721
131.522
131.386
129.584
127.770
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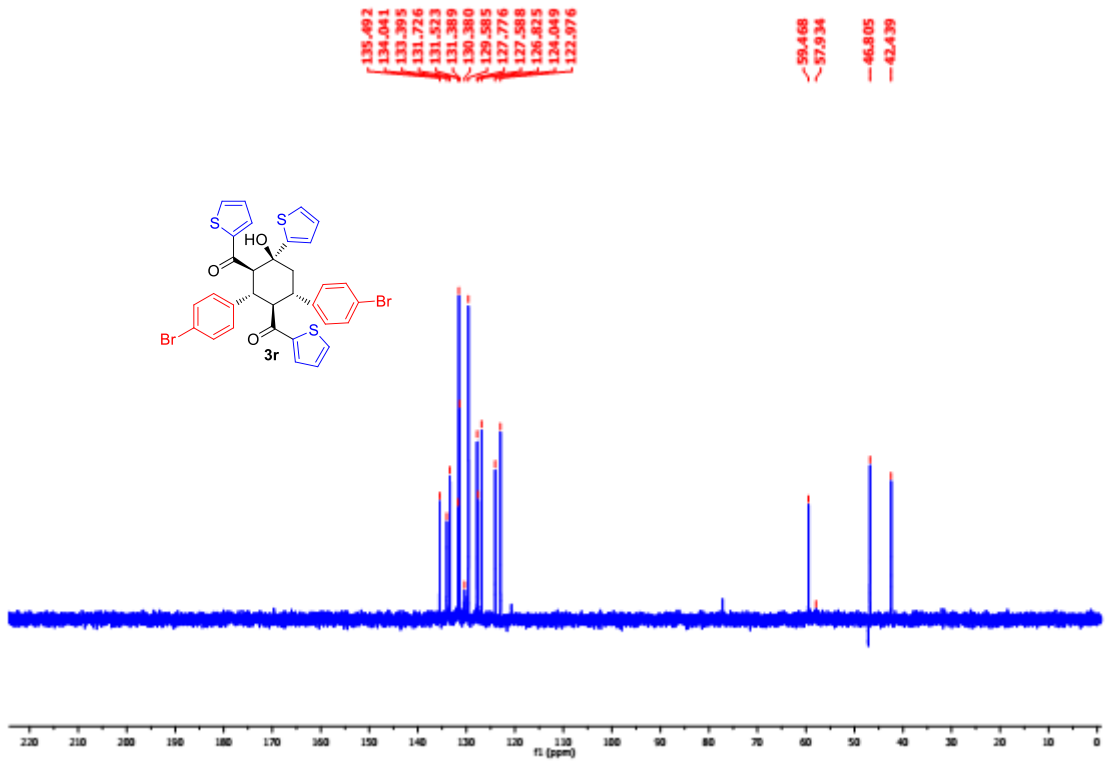
126.822
124.045
122.975
121.064
120.696
77.317
77.000
76.682
74.509
59.475
57.948
47.164
46.809
42.438
HO
O
S
S
Br
Br
O
S
3r
f1 (ppm)


135.492
134.041
133.395
131.726
131.523
131.389
130.380
129.585
127.776
127.588
126.825
124.049
122.976
59.468
57.934
46.805
42.439
HO
O
S
S
Br
Br
O
S
3r
f1 (ppm)