# Supporting Information for

# Palladium Catalyzed Enantioselective [5+4] Annulation of *ortho*-Quinone Methides and Vinylethylene Carbonates

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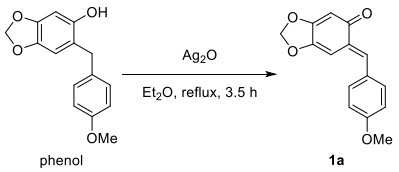
## 1. General information

$^{1}H$ NMR and $^{13}C$ NMR spectra were recorded on Bruker Avance III HD 600 or Avance 400 MHz spectrometer. Chemical shifts are recorded in ppm relative to tetramethylsilane with the solvent resonance as the internal standard. Data are represented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, m = multiplet), coupling constants (*J*) are in Hertz (Hz), and integration. Enantiomer excesses were determined by chiral HPLC analysis on Chiralcel IA/AS-H/ID/OD-H/IE in comparison with the authentic racemates. Chiral HPLC analysis was recorded on Thermo Scientific Dionex Ultimate 3000 and Agilent Technologies 1260 Infinity. Optical rotations were recorded on Autopol Automatic Polarimeter, and were reported as follows: $[\alpha]_D^T$ (c: g/100 mL, in $CH_2Cl_2$). High resolution mass spectra (HRMS) was recorded on an ABI/Sciex QStar Mass Spectrometer (ESI). Single crystal X-ray crystallography data were obtained on Supernova Atlas S2 CCD detector. Melting point (m.p.) data were obtained on X-5 micro melting point apparatus. For column chromatography, silica gel (200-300 mesh) was used as the stationary phase. Unless stated otherwise, all the solvent and reagents were purchased from commercial suppliers and used without further purification.

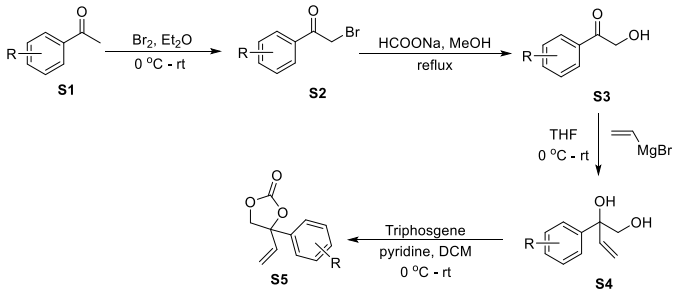
## 2. General Procedure for the Synthesis of Substrates

### 2.1 Synthesis of *ortho*-Quinone Methides (o-QMs) 1[1]




A solution of 2-(4-methoxybenzyl)-4,5-methylenedioxyphe-nol (3.88 g, 15 mmol) in ether (300 mL) was heated under reflux with silver oxide (11.6 g, 50 mmol) for 3.5 h and filtered. The orange crystals were separated by filtration. The solution was concentrated to 150 mL, cooled, and the colored product was collected. The ether filtrate was diluted to 200 mL and treated once again with silver oxide (5.8 g, 25 mmol) for 2 h to give an additional quantity of the orange product (1.56 g, 41% yield). $^1$H NMR (600 MHz, $CDCl_3$): δ 7.89 (s, 1H), 7.49 (d, *J* = 8.4 Hz, 2H), 6.96 (d, J = 8.8 Hz, 2H), 6.71 (s, 1H), 5.97 (s, 1H), 5.89 (s, 2H), 3.86 (s, 3H). Other substrates 1 were synthesized according to the similar procedure.

### 2.2 Synthesis of Substituted Vinylethylene Carbonates (VECs) 2[2]




Bromine (5.0 mmol, 1eq) was added to a solution of the respective ketone (5.0 mmol, 1.0 equiv) in $Et_2O$ (6 mL) at 0 °C. And then 15 minutes later, the solution was stirred for 6-12 h (until the color of the solution changed from red to light-yellow) at r.t.. The reaction progress was monitored by TLC. When the starting material disappeared, the reaction was quenched with ice water (6 mL), and extracted with $Et_2O$ (3 × 5 mL). The combined organic phases were washed with saturated aqueous $NaHCO_3$ solution (10 mL), brine (10 mL), and dried over $Na_2SO_4$. After being filtered and

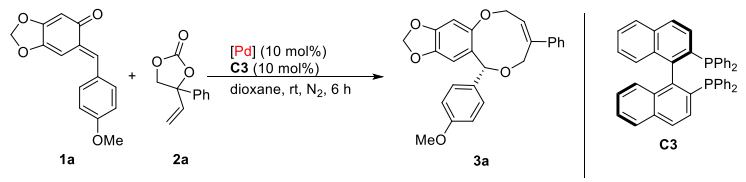
concentrated, the residue was used for the next step without further purification (the crude **S2**).

To a solution of resultant crude **S2** (1.0equiv) in methanol (3 mL/mmol) was added sodium formate (3.0 equiv) at r.t.. The reaction mixture was refluxed until TLC shows the end, and then was filtered. The filtrate was concentrated under reduced pressure. The residue was dissolved with ethyl acetate, washed with water and brine solution, dried over $Na_2SO_4$ and evaporated. The crude material was purified by column chromatography to get **S3**.

To a solution of the **S3** (1.0 equiv) in THF (4 mL/mmol) was added vinylmagnesium bromide (1.0 M in THF, 2.5 equiv) at 0 °C. The reaction was stirred under an $N_2$ atmosphere at room temperature for 2-3 h. The reaction mixture was then quenched with saturated aqueous $NH_4Cl$, and extracted with ethyl acetate. The combined organic layers were dried over anhydrous $Na_2SO_4$, filtered and concentrated. Then the crude product was purified by column chromatography to get the **S4**. To a solution of **S4** in DCM (1.5 mL/mmol) added pyridine (4.0 equiv) and triphosgene (0.5 equiv) at 0 °C. The reaction was stirred under an $N_2$ atmosphere at room temperature for 1-2 h. The reaction mixture was then quenched with saturated aqueous $NH_4Cl$, washed with $H_2O$, and extracted with DCM. The combined organic layers were dried over anhydrous $Na_2SO_4$, filtered and concentrated. The residue was purified by flash chromatography (petroleum ether/ethyl acetate =10:1) on silica to afford the corresponding carbonate (**S5**).

## 3. The Optimization of Reaction Conditions

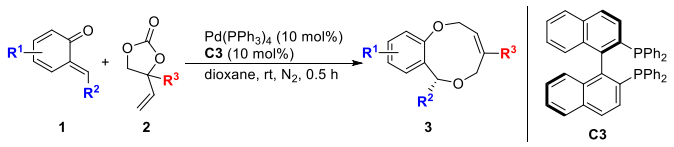
### 3.1 The Screening of Palladium[a]




| entry | [Pd] | yield (%) | ee (%) |
|---|---|---|---|
| 1[b] | $Pd(PPh_3)_4$ | 86 | 95 |
| 2 | $Pd(dppe)_2$ | 22 | 93 |
| 3 | $Pd(P(4\text{-}Me\text{-}Ph)_3)_2$ | 50 | 97 |
| 4 | $Pd(PCy_3)_2$ | NR | -- |
| 5 | $Pd(P^tBu_3)_2$ | 37 | 95 |

[a]Reaction conditions: **1a** (0.1mmol), **2a** (0.15mmo), [Pd] (10 mol%) and **C3** (10 mol%) in dioxane (1 mL) at rt under $N_2$ for 6h. Yields refer to isolated products. The ee values were determined by chiral HPLC analysis. [b]Reaction time was 0.5h.

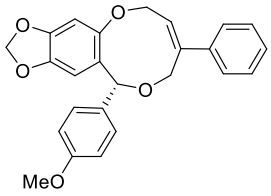
## 4. General procedure for the asymmetric [5+4] Annulations Reactions




A dried Schlenck flask was charged with all solid substances like **1** (0.1 mmol), $Pd(PPh_3)_4$ (11.6 mg, 0.01 mmol, 10 mol %) and **C3** (6.2 mg, 0.01 mmol, 10 mol %). The reaction tube was placed under vacuum and backfilled with argon three times. Dioxane (1.0 mL) followed by **2** (0.15 mmol, 1.5 equiv) were added via syringe under argon. The resulting mixture was stirred at rt for 0.5 h, and the solvent was removed under vacuum. The residue was purified by flash column chromatography on silica gel (PE:EA:$Et_3N$ = 150:10:1–100:10:1 as eluent) to give the corresponding annulation products **3** (the products **3** were acid-sensitive).

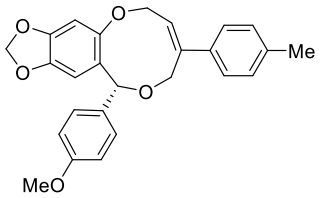
## 5.Characterization Data of Products 3

**(*R*)-11-(4-methoxyphenyl)-8-phenyl-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3a)**



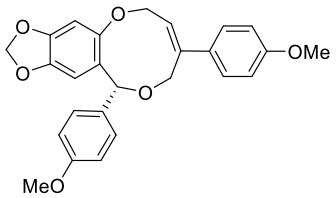

White solid; m.p. 125.3-128.2 °C; 34.6 mg, 86% yield, 95% ee; $[\alpha]_D^{20}$ = -122.65 (c = 1.07, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 16.410 min, 17.243 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.55-7.53 (m, 2H), 7.33-7.31 (m, 4H), 7.29-7.27 (m, 1H), 6.89 (d, *J* = 8.4 Hz, 2H), 6.64 (s, 1H), 6.43 (t, *J* = 7.8 Hz, 1H), 6.35 (s, 1H), 5.91 (s, 1H), 5.90 (s, 2H), 4.90-4.87 (m, 1H), 4.55-4.51 (m, 2H), 4.29 (d, *J* = 13.8 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.7, 155.1, 148.3, 144.3, 143.0, 141.0, 133.5, 128.5, 128.0, 127.9, 127.8, 126.7, 126.6, 113.7, 108.4, 102.9, 101.6, 73.2, 70.4, 66.0, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{23}O_5$ $(M+H)^+$ requires m/z 403.1540, found m/z 403.1541.

**(*R*)-11-(4-methoxyphenyl)-8-(p-tolyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3b)**




White solid; m.p. 51.3-54.6 °C; 36.6 mg, 88% yield, 91% ee; $[\alpha]_D^{20}$ = -89.33 (c = 1.22, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 13.973 min, 15.002 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.64 (s, 1H), 6.41 (t, *J* = 7.6 Hz, 1H), 6.34 (s, 1H), 5.90 (d, *J* = 3.2 Hz, 2H), 5.88 (s, 1H), 4.87 (q, *J* = 7.6 Hz, 1H), 4.54-4.51 (m, 1H), 4.49 (s, 1H), 4.27 (d, *J* = 13.6 Hz, 1H), 3.81 (s, 3H), 2.33 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.7, 155.2, 148.3, 144.3, 142.7, 138.0, 137.8, 133.6, 129.2, 127.9, 127.8, 126.5, 125.8, 113.7, 108.5, 102.9, 101.6, 73.1, 70.5, 66.0, 55.4, 21.2; **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{25}O_5$ $(M+H)^+$ requires m/z 417.1697, found m/z 417.1693.

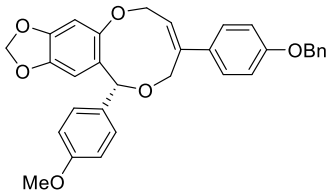
**(*R*)-8,11-bis(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3c)**




White solid; m.p. 54.6-57.9 °C; 29.0 mg, 67% yield, 96% ee; $[\alpha]_D^{20}$ = -73.70 (c = 0.72, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 20.250 min, 22.823 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.8 Hz, 2H), 7.31 (d, *J* = 8.8 Hz, 2H), 6.89-6.84 (m, 4H), 6.63 (s, 1H), 6.37 (t, *J* = 7.6 Hz, 1H), 6.34 (s, 1H), 5.90 (d, *J* = 4.4 Hz, 2H), 5.87 (s, 1H), 4.87 (q, *J* = 7.2 Hz, 1H), 4.51-4.46 (m, 2H), 4.23 (d, *J* = 13.6 Hz, 1H), 3.80 (s, 3H), 3.79 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 159.6, 158.7, 155.2, 148.3, 144.3, 142.3, 133.7, 133.4, 127.9, 127.8, 127.7, 125.2, 113.9, 113.7, 108.4, 102.9, 101.6, 72.9, 70.6, 65.8, 55.4, 55.3; **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{25}O_6$ $(M+H)^+$ requires m/z 433.1646, found m/z 433.1642.

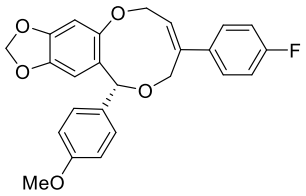
**(*R*)-8-(4-(benzyloxy)phenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-**

**[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3d)**




White solid; m.p. 129.1-131.2 °C; 42.7 mg, 84% yield, 94% ee; $[\alpha]_D^{20}$ = -47.07 (c = 0.99, $CH_2Cl_2$); **HPLC** CHIRALCEL IA, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 22.827 min, 34.500 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.49 (d, *J* = 9.0 Hz, 2H), 7.43-7.42 (m, 2H), 7.38 (t, *J* = 7.2 Hz, 2H), 7.34-7.30 (m, 3H), 6.90 (dd, *J* = 26.4, 9.0 Hz, 4H), 6.63 (s, 1H), 6.38 (t, *J* = 8.4 Hz, 1H), 6.34 (s, 1H), 5.91-5.89 (m, 2H), 5.87 (s, 1H), 5.06 (s, 2H), 4.87 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.51-4.47 (m, 2H), 4.24 (d, *J* = 13.2 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.8, 158.7, 155.2, 148.3, 144.3, 142.2, 137.0, 133.7, 133.6, 128.7, 128.1, 127.9, 127.8, 127.7, 127.6, 125.2, 114.8, 113.7, 108.4, 102.9, 101.6, 72.9, 70.6, 70.1, 65.8, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{32}H_{28}O_6Na$ $(M+Na)^+$ requires m/z 531.1778, found m/z 531.1783.

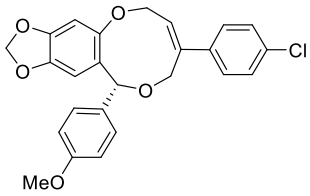
**(*R*)-8-(4-fluorophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3e)**




Colorless oil; 26.9 mg, 64% yield, 92% ee; $[\alpha]_D^{20}$ = -29.89 (c = 0.60, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 14.518 min, 16.385 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.49 (m, 2H), 7.32-7.29 (m, 2H), 7.02-6.97 (m, 2H), 6.89-6.87 (m, 2H), 6.63 (s, 1H), 6.37 (t, *J* = 7.6 Hz, 1H), 6.34 (s, 1H), 5.91-5.89 (m, 2H), 5.86 (s, 1H), 4.87 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.50-4.45 (m, 2H), 4.23 (d, *J* = 13.2 Hz, 1H), 3.80 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 163.9, 161.5, 158.7, 155.1, 148.4, 144.4, 142.0, 137.1 ($J_{C\text{-}F}$ = 4.0 Hz), 133.6, 128.4 ($J_{C\text{-}F}$ = 8.0 Hz), 127.8, 127.6, 126.8, 115.5, 115.3, 113.7, 108.4, 102.8, 101.6, 73.0, 70.3, 65.8, 55.4; **$^{19}$F NMR** (564 MHz, $CDCl_3$) δ –112.3 (s); **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}FO_5$ $(M+H)^+$ requires m/z 421.1446, found m/z 421.1445.

**(*R*)-8-(4-chlorophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-**

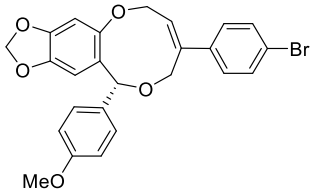
**[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3f)**




White solid; m.p. 58.2-62.3 °C; 31.4 mg, 72% yield, 89% ee; $[\alpha]_D^{20}$ = -63.37 (c = 1.03, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 14.428 min, 16.697 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.4 Hz, 2H), 7.31-7.26 (m, 4H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.63 (s, 1H), 6.41 (t, *J* = 8.0 Hz, 1H), 6.34 (s, 1H), 5.92 (d, *J* = 5.6 Hz, 2H), 5.85 (s, 1H), 4.88 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.50-4.45 (m, 2H), 4.23 (d, *J* = 13.6 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.7, 155.0, 148.4, 144.4, 141.9, 139.3, 133.9, 133.5, 128.7, 128.0, 127.8, 127.5, 127.2, 113.7, 108.4, 102.7, 101.6, 73.0, 70.3, 65.6, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{21}ClO_5Na$ $(M+Na)^+$ requires m/z 459.0971, found m/z 459.0970.

**(*R*)-8-(4-bromophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-**

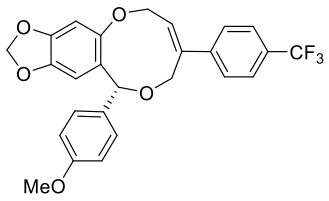
**[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3g)**




White solid; m.p. 55.6-58.9 °C; 38.9 mg, 81% yield, 92% ee; $[\alpha]_D^{20}$ = -37.13 (c = 1.16, $CH_2Cl_2$), $[\alpha]_D^{17}$ = -83.32 (c = 0.55, $CHCl_3$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 14.550 min, 16.913 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.45-7.41(m, 4H), 7.30 (d, *J* = 8.4 Hz, 2H), 6.90-6.87 (m, 2H), 6.62 (s, 1H), 6.41 (t, *J* = 7.8 Hz, 1H), 6.34 (s, 1H), 5.90 (dd, *J* = 8.4, 1.2 Hz, 2H), 5.85 (s, 1H), 4.88 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.47 (dd, *J* = 12.0, 9.0 Hz, 2H), 4.22 (d, *J* = 13.8 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.7, 155.0, 148.4, 144.4, 141.9, 139.8, 133.5, 131.6, 128.3, 127.8, 127.5, 127.3, 122.1, 113.7, 108.4, 102.7, 101.6, 73.0, 70.2, 65.6, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}BrO_5$ $(M+H)^+$ requires m/z 481.0645, found m/z 481.0642.

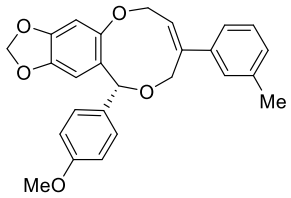
**(*R*)-11-(4-methoxyphenyl)-8-(4-(trifluoromethyl)phenyl)-6,9-dihydro-11H-**

**[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3h)**



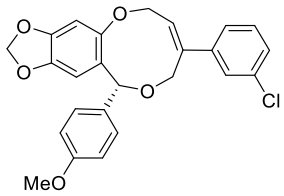

White solid; m.p. 55.5-59.3 °C; 24.4 mg, 52% yield, 72% ee; $[\alpha]_D^{20}$ = -42.87 (c = 0.72, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 10.183 min, 11.792 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 32.4, 8.4 Hz, 4H), 7.32-7.30 (m, 2H), 6.91-6.87 (m, 2H), 6.64 (s, 1H), 6.48 (t, *J* = 8.0 Hz, 1H), 6.35 (s, 1H), 5.92-5.90 (m, 2H), 5.87 (s, 1H), 4.91 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.53-4.51 (m, 1H), 4.50-4.48 (m, 1H), 4.26 (d, *J* = 13.6 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.8, 154.9, 148.5, 144.5, 141.9, 133.4, 128.7, 127.8, 127.4, 127.0, 125.6 ($J_{C\text{-}F}$ = 3.0 Hz), 125.4 ($J_{C\text{-}F}$ = 4.0 Hz), 113.7, 108.4, 102.7, 101.6, 73.1, 70.1, 65.6, 55.4; **$^{19}$F NMR** (564 MHz, $CDCl_3$) δ –62.6 (s); **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{22}F_3O_5$ $(M+H)^+$ requires m/z 471.1414, found m/z 471.1415.

**(*R*)-11-(4-methoxyphenyl)-8-(m-tolyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3i)**



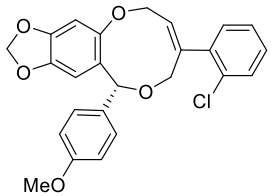

White solid; m.p. 112.5-115.3 °C; 22.9 mg, 55% yield, 98% ee; $[\alpha]_D^{20}$ = -65.92 (c = 1.43, $CH_2Cl_2$); **HPLC** CHIRALCEL IE, *n*-hexane/2-propanol = 90/10, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 14.375 min, 15.567 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.31 (m, 4H), 7.21 (t, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.4 Hz, 2H), 6.64 (s, 1H), 6.40 (t, *J* = 8.0 Hz, 1H), 6.34 (s, 1H), 5.90 (t, *J* = 3.6 Hz, 1H), 4.87 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.56-4.49 (m, 2H), 4.30 (d, *J* = 13.6 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.7, 155.2, 148.3,144.3, 143.1, 140.9, 138.0, 133.5, 128.7, 128.4, 128.0, 127.8, 127.4, 126.4, 123.7, 113.7, 108.5, 102.9, 101.6, 73.3, 70.5, 66.2, 55.4, 21.7; **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{25}O_5$ $(M+H)^+$ requires m/z 417.1697, found m/z 417.1696.

**(*R*)-8-(3-chlorophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3j)**



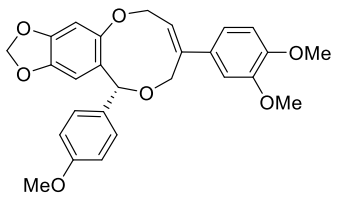

White solid; m.p. 125.6-130.0 °C; 38.4 mg, 88% yield, 93% ee; $[\alpha]_D^{20}$ = -71.86 (c = 1.22, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 13.498 min, 14.978 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.43-7.41 (m, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.25-7.24 (m, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 6.63 (s, 1H), 6.42 (t, *J* = 7.6 Hz,1H), 6.33 (s, 1H), 5.90 (d, *J* = 4.4 Hz, 2H), 5.86 (s, 1H), 4.88 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.52-4.45 (m, 2H), 4.24 (d, *J* = 13.6 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.8, 155.0, 148.4, 144.4, 142.8, 141.9, 134.4, 133.4, 129.8, 128.0, 127.9, 127.8, 127.6, 126.8, 124.8, 113.7, 108.4, 102.7, 101.6, 73.2, 70.2, 65.8, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}ClO_5$ $(M+H)^+$ requires m/z 437.1150, found m/z 437.1146.

**(*R*)-8-(2-chlorophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3k)**



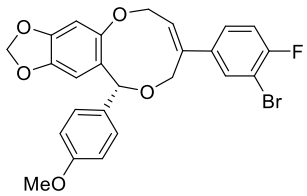

White solid; m.p. 125.5-130.2 °C; 35.8 mg, 82% yield, 89% ee; $[\alpha]_D^{20}$ = -34.64 (c = 0.97, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 13.730 min, 18.912 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.34-7.30 (m, 3H), 7.23-7.16 (m, 2H), 7.12-7.10 (m, 1H), 6.90 (d, *J* = 8.4 Hz, 2H), 6.66 (s, 1H), 6.30 (s, 1H), 6.07-6.03 (m, 2H), 5.90 (s, 2H), 4.83-4.71 (m, 2H), 4.34 (s, 2H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.9, 155.2, 148.1, 144.1, 143.4, 140.2, 132.8, 132.3, 130.9, 129.6, 129.0, 128.6, 128.2, 128.1, 126.9, 113.8, 108.6, 103.3, 101.5, 74.6, 70.2, 67.6, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}ClO_5$ $(M+H)^+$ requires m/z 437.1150, found m/z 437.1153.

**(*R*)-8-(3,4-dimethoxyphenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3l)**



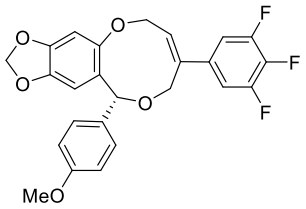

Colorless oil; 24.0 mg, 52% yield, 92% ee; $[\alpha]_D^{20}$ = -77.25 (c = 0.88, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 46.305 min, 51.677 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.33-7.30 (m, 2H), 7.16 (d, *J* = 2.0 Hz, 1H), 7.11 (dd, *J* = 8.0, 4.0 Hz, 1H), 6.89-6.85 (m, 2H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.63 (s, 1H), 6.40 (t, *J* = 8.0 Hz, 1H), 6.35 (s, 1H), 5.90 (dd, *J* = 5.2, 1.6 Hz, 2H), 5.88 (s, 1H), 4.88 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.51-4.44 (m, 2H), 4.23 (d, *J* = 13.6 Hz, 1H), 3.87 (s, 6H), 3.80 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.7, 155.2, 149.0, 148.8, 148.4, 144.4, 142.4, 133.9, 133.7, 127.8, 127.6, 125.7, 119.0, 113.6, 111.1, 110.0, 108.4, 102.9, 101.6, 72.7, 70.5, 65.7, 56.0, 55.9, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{27}H_{27}O_7$ $(M+H)^+$ requires m/z 463.1751, found m/z 463.1757.

**(*R*)-8-(3-bromo-4-fluorophenyl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3m)**



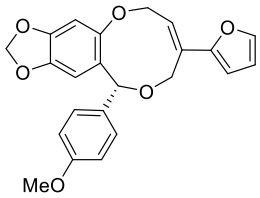

White solid; m.p. 57.7-60.3 °C; 27.9 mg, 56% yield, 74% ee; $[\alpha]_D^{20}$ = -32.08 (c = 1.01, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 13.655 min, 16.295 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 6.8, 2.4 Hz, 1H), 7.49-7.45 (m, 1H), 7.30 (d, *J* = 8.8 Hz, 2H), 7.06 (t, *J* = 8.4 Hz, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 6.62 (s, 1H), 6.37 (t, *J* = 8.0 Hz, 1H), 6.33 (s, 1H), 5.91 (dd, *J* = 5.6, 1.2 Hz, 2H), 5.84 (s, 1H), 4.88 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.49-4.43 (m, 2H), 4.20 (d, *J* = 13.2 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 160.1, 158.8, 157.6, 154.9, 148.5, 144.4, 141.0, 138.6 ($J_{C\text{-}F}$ = 3.0 Hz), 133.4, 131.8, 127.8, 127.4, 127.3 ($J_{C\text{-}F}$ = 7.0 Hz), 116.5 ($J_{C\text{-}F}$ = 22.0 Hz), 113.7, 109.2 ($J_{C\text{-}F}$ = 21.0 Hz), 108.4, 102.7, 101.6, 73.1, 70.1, 65.6, 55.4; **$^{19}$F NMR** (564 MHz, $CDCl_3$) δ –108.7 (s); **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{21}BrFO_5$ $(M+H)^+$ requires m/z 499.0551, found m/z 499.0544.

**(*R*)-11-(4-methoxyphenyl)-8-(3,4,5-trifluorophenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3n)**



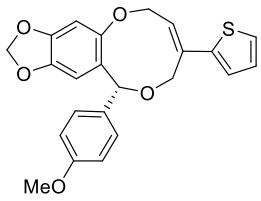
Yellow oil; 21.9 mg, 48% yield, 46% ee; $[\alpha]_D^{20}$ = -25.21 (c = 0.78, $CH_2Cl_2$); **HPLC** CHIRALCEL xx, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 11.523 min, 13.055 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.22-7.18 (m, 2H), 6.89 (d, *J* = 8.8 Hz, 2H), 6.61 (s, 1H), 6.40 (t, *J* = 7.6 Hz, 1H), 6.33 (s, 1H), 5.91 (dd, *J* = 6.4, 1.2 Hz, 2H), 5.81 (s, 1H), 4.90 (dd, *J* = 12.8, 7.2 Hz, 1H), 4.46-4.40 (m, 2H), 4.16 (d, *J* = 13.2 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.8, 154.7, 148.6, 144.5, 140.4, 133.4, 128.7, 127.7, 127.4, 127.2, 113.8, 110.1 ($J_{C\text{-}F}$ = 6.0 Hz), 110.8 ($J_{C\text{-}F}$ = 6.0 Hz), 108.4, 102.5, 101.7, 73.0, 69.8, 65.2, 55.4; **$^{19}$F NMR** (564 MHz, $CDCl_3$) δ –134.3 (d, *J* = 31.0 Hz), -161.4 (t, *J* = 30.5 Hz); **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{20}F_3O_5$ $(M+H)^+$ requires m/z 457.1257, found m/z 457.1252.

**(*R*)-8-(furan-2-yl)-11-(4-methoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3o)**



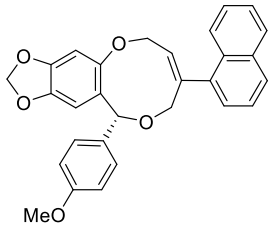
Colorless oil; 19.6 mg, 50% yield, 51% ee; $[\alpha]_D^{20}$ = -115.66 (c = 1.12, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 16.213 min, 20.438 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.36 (s, 1H), 7.29-7.27 (m, 2H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.67 (t, *J* = 8.0 Hz, 1H), 6.63 (s, 1H), 6.52 (d, *J* = 3.6 Hz, 1H), 6.39 (s, 1H), 6.32 (s, 1H), 5.90 (d, *J* = 5.2 Hz, 2H), 5.80 (s, 1H), 4.90 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.52-4.47 (m, 2H), 4.14 (d, *J* = 13.6 Hz, 1H), 3.80 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.7, 155.3, 153.5, 148.4, 144.3, 142.5, 133.5, 132.6, 127.9, 127.4, 122.7, 113.6, 111.7, 108.4, 108.3, 102.8, 101.6, 73.2, 70.3, 63.4, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{23}H_{21}O_6$ $(M+H)^+$ requires m/z 393.1333, found m/z 393.1330.

**(*R*)-11-(4-methoxyphenyl)-8-(thiophen-2-yl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3p)**




White solid; m.p. 58.3-63.1 °C; 18.4 mg, 45% yield, 34% ee; $[\alpha]_D^{20}$ = -64.98 (c = 0.75, $CH_2Cl_2$); **HPLC** CHIRALCEL IA, *n*-hexane/2-propanol = 80/20, flow rate = x.x mL/min, λ = 250 nm, retention time: 17.447 min, 19.548 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.31-7.29 (m, 2H), 7.26-7.25 (m, 1H), 7.18 (d, *J* = 5.4 Hz, 1H), 6.99-6.98 (m, 1H), 6.88-6.87 (m, 2H), 6.63 (s, 1H), 6.54 (t, *J* = 7.8 Hz, 1H), 6.34 (s, 1H), 5.90 (dd, *J* = 7.2, 1.8 Hz, 2H), 5.84 (s, 1H), 4.86 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.56 (d, *J* = 13.8 Hz, 1H), 4.47 (dd, *J* = 12.6, 8.4 Hz, 1H), 4.21 (d, *J* = 13.2 Hz, 1H), 3.80 (s, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.7, 155.3, 148.4, 144.4, 144.0, 137.0, 133.5, 127.9, 127.8, 127.4, 125.2, 125.1, 124.9, 113.6, 108.5, 102.8, 101.6, 73.1, 70.5, 65.2, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{23}H_{21}O_5S$ $(M+H)^+$ requires m/z 409.1104, found m/z 409.1101.

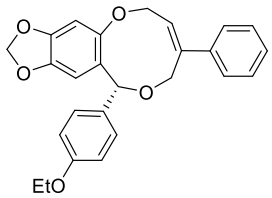
**(*R*)-11-(4-methoxyphenyl)-8-(naphthalen-1-yl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3q)**




White solid; m.p. 126.6-130.2 °C; 38.4 mg, 85% yield, 95% ee; $[\alpha]_D^{20}$ = -65.17 (c = 1.20, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 16.818 min, 18.850 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.84-7.78 (m, 3H), 7.66 (dd, *J* = 9.0, 1.8 Hz, 1H), 7.48-7.44 (m, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 9.0 Hz, 2H), 6.67 (s, 1H), 6.58 (t, *J* = 7.8 Hz, 1H), 6.38 (s, 1H), 5.95 (s, 1H), 5.91 (dd, *J* = 7.2, 1.2 Hz, 2H), 4.94 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.66, (d, 13.8 Hz, 1H), 4.58 (dd, *J* = 12.0, 8.4 Hz, 1H), 4.38 (d, *J* = 13.8 Hz, 1H), 3.81 (s, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.7, 155.2, 148.4, 144.3, 142.9, 138.1, 133.5, 133.4, 133.0, 128.5, 128.1, 127.9, 127.8, 127.7, 127.1, 126.3, 126.2, 125.8, 124.6, 113.7, 108.5, 102.9, 101.6, 73.2, 70.5, 66.0, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{29}H_{25}O_5$ $(M+H)^+$ requires m/z 453.1697, found m/z 453.1692.

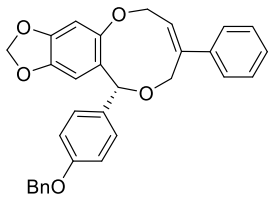
**(*R*)-11-(4-ethoxyphenyl)-8-phenyl-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-**

**b][1,5]dioxonine (3r)**



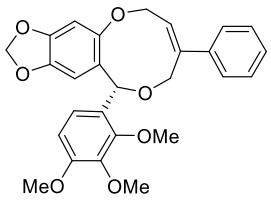
White solid; m.p. 45.5-48.9 °C; 25.0 mg, 60% yield, 90% ee; $[\alpha]_D^{20}$ = -72.81 (c = 0.89, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 16.292 min, 20.288 min; **$^1$H NMR** (600 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.8 Hz, 2H), 7.33-7.27 (m, 5H), 6.87 (d, *J* = 7.8 Hz, 2H), 6.63 (s, 1H), 6.42 (t, *J* = 7.8 Hz, 1H), 6.34 (s, 1H), 5.91 (s, 1H), 5.89 (d, *J* = 7.2 Hz, 2H), 4.88 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.55-4.50 (m, 2H), 4.29 (d, *J* = 13.2 Hz, 1H), 4.03 (q, *J* = 7.2 Hz, 2H), 1.41 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (150 MHz, $CDCl_3$) δ 158.1, 155.1, 148.3, 144.3, 143.0, 141.0, 133.4, 128.5, 128.0, 127.9, 127.8, 126.7, 126.6, 114.3, 108.5, 102.9, 101.6, 73.2, 70.5, 66.1, 63.6, 15.0; **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{25}O_5$ $(M+H)^+$ requires m/z 417.1697, found m/z 417.1692.

**(*R*)-11-(4-(benzyloxy)phenyl)-8-phenyl-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3s)**



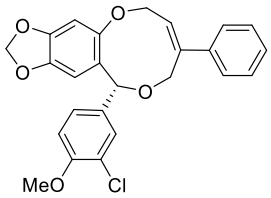
White solid; m.p. 110.3-113.6 °C; 29.6 mg, 62% yield, 94% ee; $[\alpha]_D^{20}$ = -72.62 (c = 1.13, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 22.567 min, 24.882 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 2H), 7.45-7.28 (m, 10H), 6.98-6.96 (m, 2H), 6.64 (s, 1H), 6.43 (t, *J* = 8.0 Hz, 1H), 6.35 (s, 1H), 5.91-5.89 (m, 3H), 5.07 (s, 2H), 4.88 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.55-4.50 (m, 2H), 4.28 (d, *J* = 14.0 Hz, 1H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.0, 155.1, 148.3, 144.3, 143.0, 141.0, 137.3, 133.9, 128.7, 128.5 128.1, 128.0, 127.9, 127.7, 126.7, 126.6, 114.6, 108.5, 102.9, 101.6, 73.1, 70.5, 70.2, 66.0; **HRMS** (ESI-TOF): exact mass calcd for $C_{31}H_{27}O_5$ $(M+H)^+$ requires m/z 479.1853, found m/z 479.1854.

**(*S*)-8-phenyl-11-(2,3,4-trimethoxyphenyl)-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3t)**



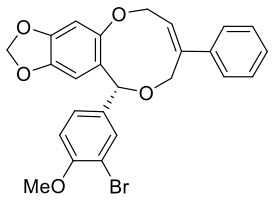

White solid; m.p. 52.6-55.9 °C; 30.5 mg, 66% yield, 86% ee; $[\alpha]_D^{20}$ = -62.04 (c = 1.34, $CH_2Cl_2$); **HPLC** CHIRALCEL ID, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 15.883 min, 17.445 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.53-7.50 (m, 2H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.32-7.23 (m, 3H), 6.78 (d, *J* = 8.8 Hz, 1H), 6.65 (s, 1H), 6.44 (t, *J* = 8.0 Hz, 1H), 6.37 (s, 1H), 6.06 (s, 1H), 5.88 (dd, *J* = 5.6, 1.2 Hz, 2H), 4.90 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.55-4.48 (m, 2H), 4.28 (d, *J* = 13.2 Hz, 1H), 3.88 (s, 3H), 3.79 (s, 3H), 3.41 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 154.7, 153.0, 150.8, 148.1, 144.1, 142.9, 142.3, 141.1, 128.5, 128.1, 128.0, 127.9, 127.0, 126.6, 121.0, 107.7, 106.9, 103.0, 101.5, 70.4, 69.2, 65.8, 60.8, 60.2, 56.1; **HRMS** (ESI-TOF): exact mass calcd for $C_{27}H_{27}O_7$ $(M+H)^+$ requires m/z 463.1751, found m/z 463.1760.

**(*R*)-11-(3-chloro-4-methoxyphenyl)-8-phenyl-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3u)**



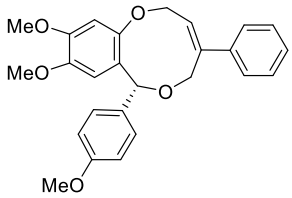

Colorless oil; 24.9 mg, 57% yield, 80% ee; $[\alpha]_D^{20}$ = -19.78 (c = 1.05, $CH_2Cl_2$); **HPLC** CHIRALCEL IA, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 12.222 min, 13.732 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.55-7.54 (m, 2H), 7.45-7.44 (m, 1H), 7.36-7.29 (m, 3H), 7.24-7.21 (m, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 6.64 (s, 1H), 6.44 (t, *J* = 8.0 Hz, 1H), 6.32 (s, 1H), 5.92 (d, *J* = 2.8 Hz, 2H), 5.85 (s, 1H), 4.89 (dd, *J* = 12.4, 7.2 Hz, 1H), 4.53-4.49 (m, 1H), 4.47-4.44 (m, 1H), 4.24 (d, *J* = 11.2 Hz, 1H), 3.89 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 155.1, 154.0, 148.6, 144.4, 142.7, 140.8, 134.9, 128.6, 128.5, 128.1, 127.0, 126.9, 126.6, 126.1, 122.3, 111.9, 108.3, 102.9, 101.7, 73.2, 70.4, 65.7, 56.3; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}ClO_5$ $(M+H)^+$ requires m/z 437.1150, found m/z 437.1154.

**(*R*)-11-(3-bromo-4-methoxyphenyl)-8-phenyl-6,9-dihydro-11H-[1,3]dioxolo[4',5':4,5]benzo[1,2-b][1,5]dioxonine (3v)**



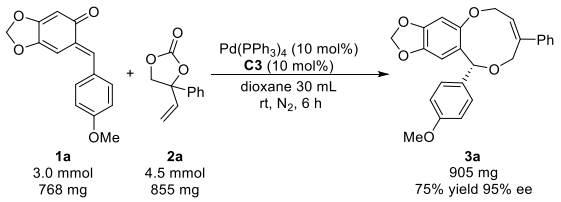

Colorless oil; 33.6 mg, 70% yield, 90% ee; $[\alpha]_D^{20}$ = -66.17 (c = 1.07, $CH_2Cl_2$); **HPLC** CHIRALCEL IA, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 13.638 min, 14.900 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.61-7.60 (m, 1H), 7.55-7.53 (m, 2H), 7.36-7.27 (m, 4H), 6.87 (d, *J* = 8.8 Hz, 1H), 6.63 (s, 1H), 6.44 (t, *J* = 8.0 Hz, 1H), 6.32 (s, 1H), 5.92 (dd, *J* = 4.0, 1.2 Hz, 2H), 5.85 (s, 1H), 4.89 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.53-4.40 (m, 2H), 4.24 (d, *J* = 13.4 Hz, 1H), 3.89 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 155.1, 154.9, 148.6, 144.4, 142.8, 140.8, 135.4, 131.6, 128.6, 128.1, 127.1, 127.0, 126.9, 126.6, 111.7, 111.6, 108.3, 102.9, 101.7, 76.9, 72.3, 70.4, 65.8, 56.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{22}BrO_5$ $(M+H)^+$ requires m/z 481.0645, found m/z 481.0650.

**(*R*)-9,10-dimethoxy-7-(4-methoxyphenyl)-4-phenyl-2,5-dihydro-7H-benzo[b][1,5]dioxonine (3w)**



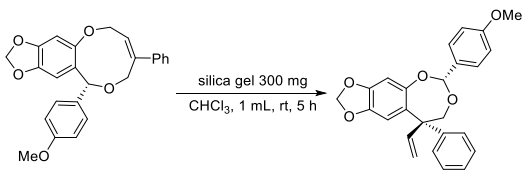

Colorless oil; 31.4 mg, 75% yield, 92% ee; $[\alpha]_D^{20}$ = -71.21 (c = 0.72, $CH_2Cl_2$); **HPLC** CHIRALCEL IE, *n*-hexane/2-propanol = 80/20, flow rate = 0.6 mL/min, λ = 250 nm, retention time: 18.075 min, 21.323 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.57-7.55 (m, 2H), 7.35-7.28 (m, 5H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.68 (s, 1H), 6.48 (t, *J* = 8.0 Hz, 1H), 6.40 (s, 1H), 5.91 (s, 1H), 4.90 (dd, *J* = 12.0, 7.2 Hz, 1H), 4.55-4.50 (m, 2H), 4.26 (d, *J* = 13.6 Hz, 1H), 3.90 (s, 3H), 3.81 (s, H), 3.66 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 158.6, 154.5, 150.1, 146.0, 142.7, 141.0, 133.6, 128.5, 128.0, 127.9, 127.1, 126.6, 126.0, 113.6, 111.7, 105.2, 72.7, 70.4, 65.7, 56.2, 56.1, 55.4; **HRMS** (ESI-TOF): exact mass calcd for $C_{26}H_{27}O_5$ $(M+H)^+$ requires m/z 419.1853, found m/z 419.1847.

## 6. Scale-up synthesis of product 3a




In a 50 mL reaction tube, **1a** (768mg, 3.0 mmol), $Pd(PPh_3)_4$ (347mg, 0.3 mmol, 10 mol%) and **C3** (187 mg, 0.3 mmol, 10 mol%) were added. The reaction tube was placed under vacuum and backfilled with argon three times. **2a** (855 mg, 4.5 mmol) dissolved in dioxane (30 mL), and this solution was added via syringe under $N_2$. The resulting mixture was stirred at rt for 5 h, and the solvent was removed under vacuum. The residue was purified by flash column chromatography on silica gel (PE:EA:$Et_3N$ = 150:10:1–100:10:1 as eluent) to give the products **3a** (acid-sensitive) as white solid (905 mg, 75% yield, 95% ee).

## 7. Derivatization of product 3a

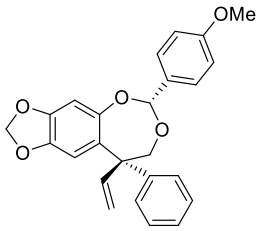



**3a**
95% ee

**4a**
95 yield, 94% ee

To a solution of the compound **3a** (19.5 mg, 0.049 mmol) in $CHCl_3$ (1.0 mL), silica gel (300 mg) (200-300 meshes) was added. After the reaction was stirred at room temperature for 5 h, and the solvent was removed under vacuum. The residue was purified by flash column chromatography on silica gel (PE:EA = 15:1−10:1) to give the product **4a** as white solid (18.6 mg, 95% yield, 94% ee).

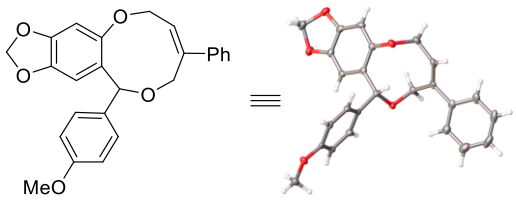
**(*6R,9R*)-6-(4-methoxyphenyl)-9-phenyl-9-vinyl-8,9-dihydro-[1,3]dioxolo[4',5':4,5]benzo[1,2-d][1,3]dioxepine (4a)**




White solid; m.p. 107.3-109.6 °C; $[\alpha]_D^{20}$ = -5.98 (c = 1.02, $CH_2Cl_2$); **HPLC** CHIRALCEL IC, *n*-hexane/2-propanol = 95/5, flow rate = 0.8 mL/min, λ = 250 nm, retention time: 9.277 min, 10.710 min; **$^1$H NMR** (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.31-7.26 (m, 2H), 7.22-7.15 (m, 3H), 6.87 (d, *J* = 8.4 Hz, 2H), 6.73 (s, 1H), 6.57 (s, 1H), 6.24 (dd, *J* = 17.2, 10.4 Hz, 1H), 5.96 (d, *J* = 3.6 Hz, 2H), 5.76 (s, 1H), 5.39 (d, *J* = 10.4 Hz, 1H), 4.79 (d, *J* = 17.2 Hz, 1H), 4.72 (d, *J* = 12.4 Hz, 1H), 4.03 (d, *J* = 12.4 Hz, 1H), 3.79 (s, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$) δ 160.1, 152.3, 146.8, 144.0, 143.0, 142.6, 131.3, 129.7, 128.6, 128.1, 127.5, 126.5, 117.9, 113.7, 110.2, 104.6, 104.1, 101.7, 73.5, 56.6, 55.5; **HRMS** (ESI-TOF): exact mass calcd for $C_{25}H_{23}O_5$ $(M+H)^+$ requires m/z 403.1540, found m/z 403.1536.

## 8. The X-ray data of 3a and 4a

### 8.1 The X-ray data of 3a




rac-3a rac-3a

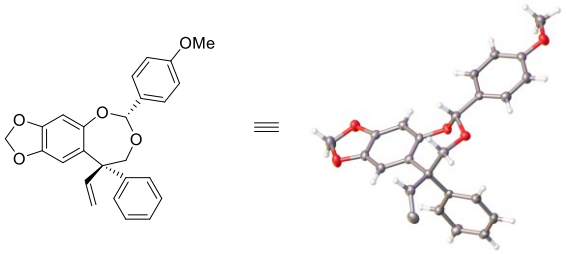
The product rac-**3a** was recrystallized by petroleum ether/diethyl ether (2/1). CCDC 1976904 (rac-**3a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

**Table 1 Crystal data and structure refinement for rac-3a.**

| | |
|---|---|
| Identification code | 3a |
| Empirical formula | $C_{25}H_{22}O_5$ |
| Formula weight | 402.42 |
| Temperature/K | 100.01(10) |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| a/Å | 6.4099(4) |
| b/Å | 16.5738(9) |
| c/Å | 18.3583(11) |
| α/° | 90 |
| β/° | 90.04(4) |
| γ/° | 90 |
| Volume/Å$^3$ | 1950.3(2) |
| Z | 4 |
| $\rho_{calc}$g/cm$^3$ | 1.371 |
| μ/mm$^{-1}$ | 0.095 |
| F(000) | 848.0 |
| Crystal size/mm$^3$ | 0.12 × 0.11 × 0.09 |
| Radiation | MoKα (λ = 0.71073) |
| 2Θ range for data collection/° | 4.438 to 49.986 |
| Index ranges | $-7 \le h \le 7$, $-18 \le k \le 19$, $-17 \le l \le 21$ |

| | |
|---|---|
| Reflections collected | 12558 |
| Independent reflections | 3439 [$R_{int}$ = 0.0337, $R_{sigma}$ = 0.0331] |
| Data/restraints/parameters | 3439/0/272 |
| Goodness-of-fit on $F^2$ | 1.049 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0377, $wR_2$ = 0.0862 |
| Final R indexes [all data] | $R_1$ = 0.0456, $wR_2$ = 0.0910 |
| Largest diff. peak/hole / e $Å^{-3}$ | 0.19/-0.23 |

**8.2 The X-ray data of 4a**




The product rac-**4a** was recrystallized by petroleum ether/diethyl ether (2/1). CCDC 1976905 (rac-**4a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

**Table 1 Crystal data and structure refinement for rac-4a.**

| | |
|---|---|
| Identification code | 4a |
| Empirical formula | $C_{25}H_{22}O_5$ |
| Formula weight | 402.42 |
| Temperature/K | 293(2) |
| Crystal system | monoclinic |
| Space group | $P2_1/c$ |
| a/Å | 14.7117(2) |
| b/Å | 10.94480(10) |
| c/Å | 13.1133(2) |
| α/° | 90 |
| β/° | 112.597(2) |
| γ/° | 90 |
| Volume/$Å^3$ | 1949.36(5) |
| Z | 4 |
| $\rho_{calc}g/cm^3$ | 1.371 |

| | |
|---|---|
| $\mu/mm^{-1}$ | 0.776 |
| F(000) | 848.0 |
| Crystal size/$mm^3$ | 0.150 × 0.120 × 0.110 |
| Radiation | CuKα (λ = 1.54184) |
| 2Θ range for data collection/° | 10.38 to 142.398 |
| Index ranges | $-17 \leq h \leq 17$, $-8 \leq k \leq 13$, $-15 \leq l \leq 15$ |
| Reflections collected | 10459 |
| Independent reflections | 3702 [$R_{int}$ = 0.0206, $R_{sigma}$ = 0.0206] |
| Data/restraints/parameters | 3702/0/280 |
| Goodness-of-fit on $F^2$ | 1.045 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0357, $wR_2$ = 0.0891 |
| Final R indexes [all data] | $R_1$ = 0.0396, $wR_2$ = 0.0922 |
| Largest diff. peak/hole / e $Å^{-3}$ | 0.21/-0.24 |

## 9. Determination of the absolute configuration of compound 3g

The absolute configuration of **3g** was assigned by comparison with literature data (Chem. Eur. J, 2019, DOI: 10.1002/chem.201904903).

By comparing the specific optical [α] values, the absolute configuration of **3g** was determined to be (R) by comparison with literature data.

(1) This work: (**3g**) 92% ee. $[\alpha]_D^{17}$ = -83.32 (c = 0.55 $CHCl_3$).

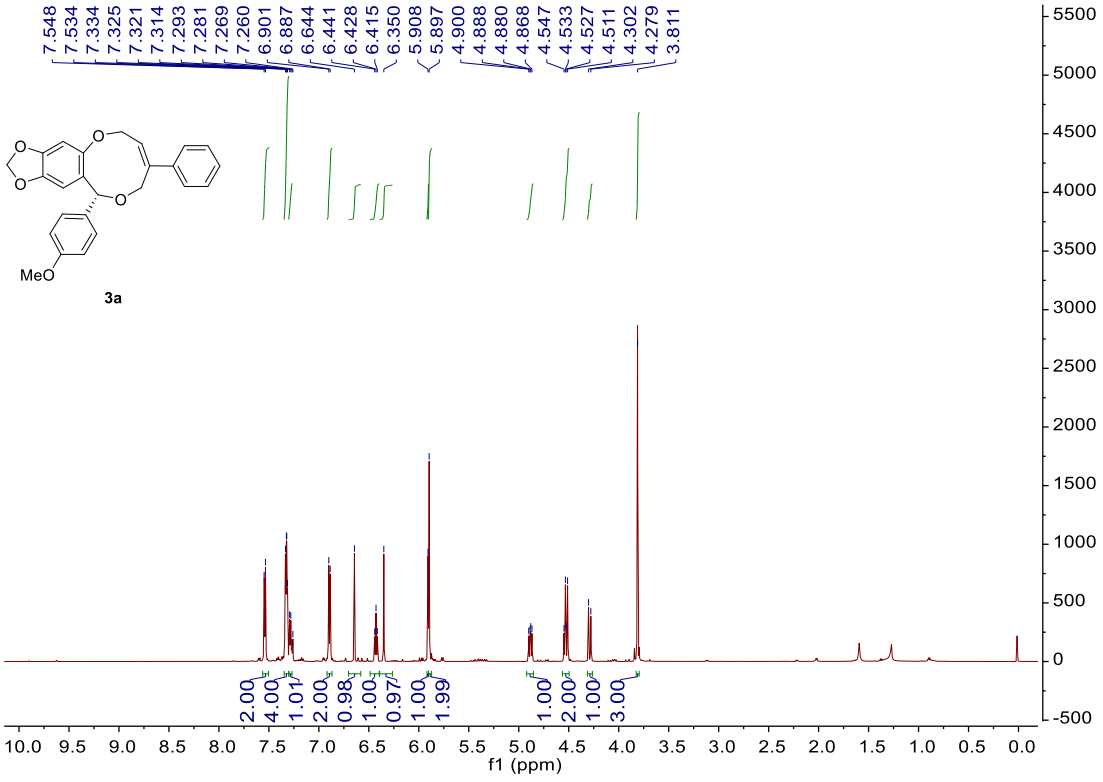
(2) Ref: (**3ag**) 97% ee $[\alpha]_D^{17}$ = -92.0 (c = 0.55, $CHCl_3$).

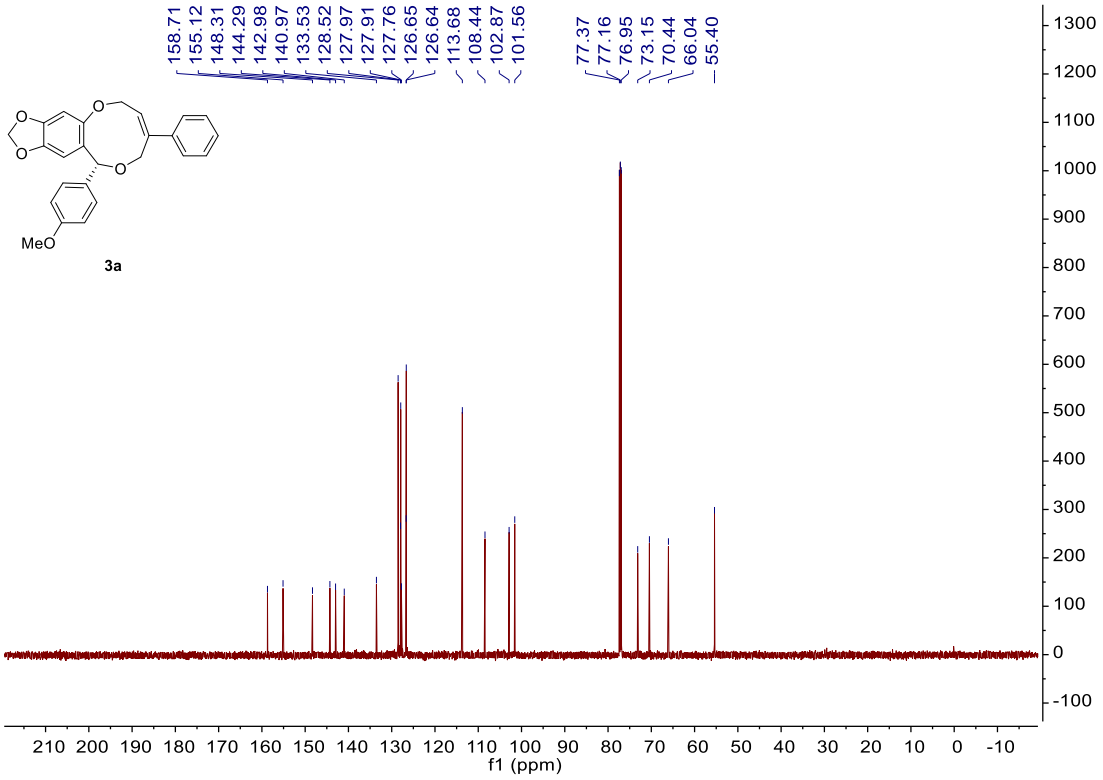
## 10. References

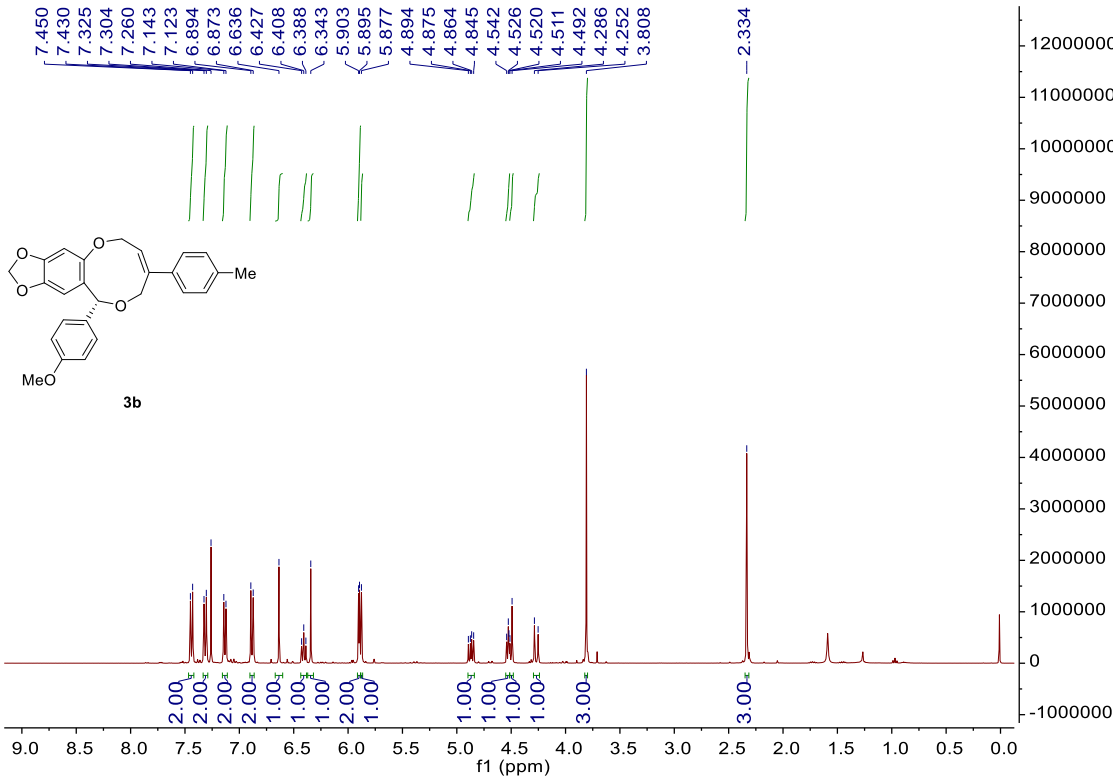
1. L. Jurd, Quinones and quinone-methides-I: Cyclization and dimerisation of crystalline ortho-quinone methides from phenol oxidation reactions, *Tetrahedron*, 1977, **33**, 163-168.

2. Y. Xu, L. Chen, Y.-w. Yang, Z. Zhang and W. Yang, Vinylethylene carbonates as *α,β*-unsaturated aldehyde surrogates for regioselective [3+3] cycloaddition, *Org. Lett*., 2019, **21**, 6674-6678.

## 11. Copies of $^{1}H$ and $^{13}C$ NMR spectra

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Me
MeO
3b
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1.00
1.00
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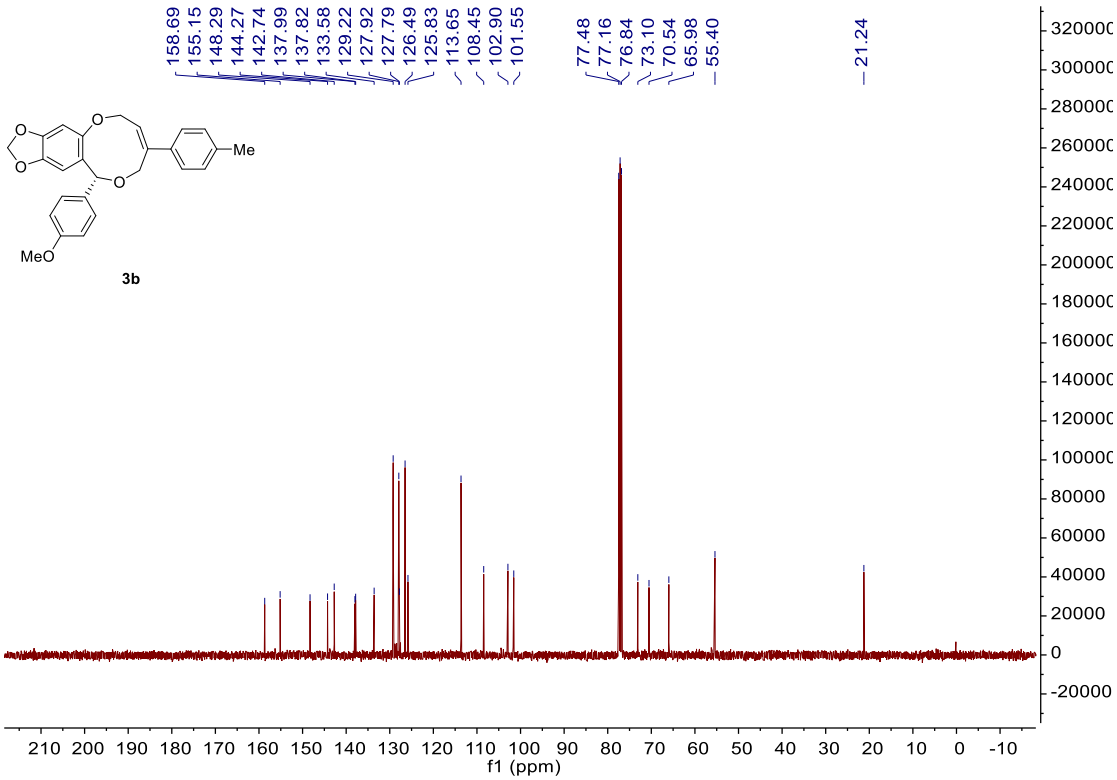
1.00
3.00
3.00
f1 (ppm)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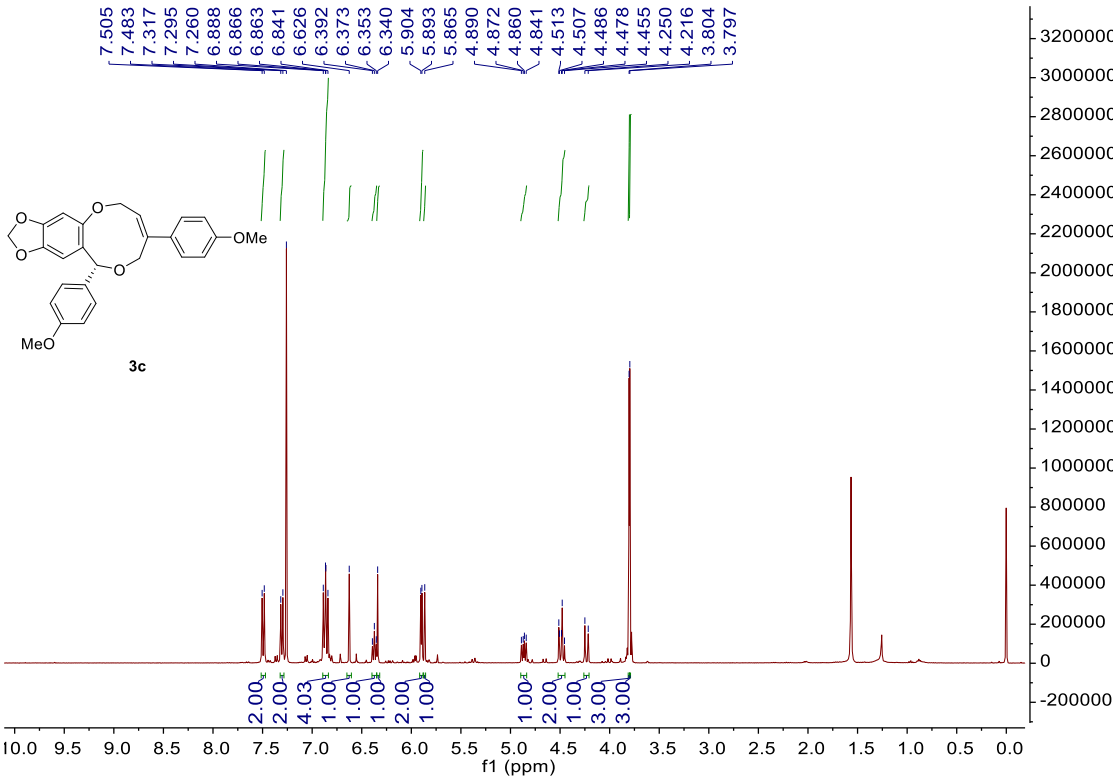
Me
MeO
3b
f1 (ppm)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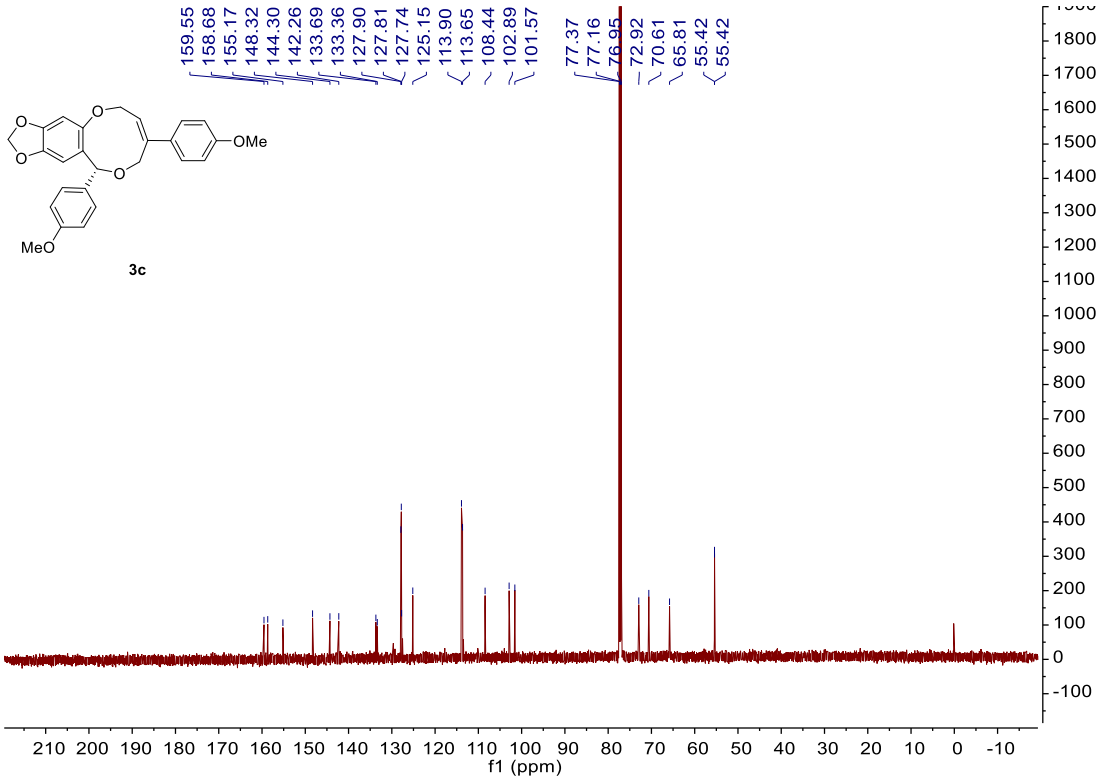
f1 (ppm)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127.74
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55.42
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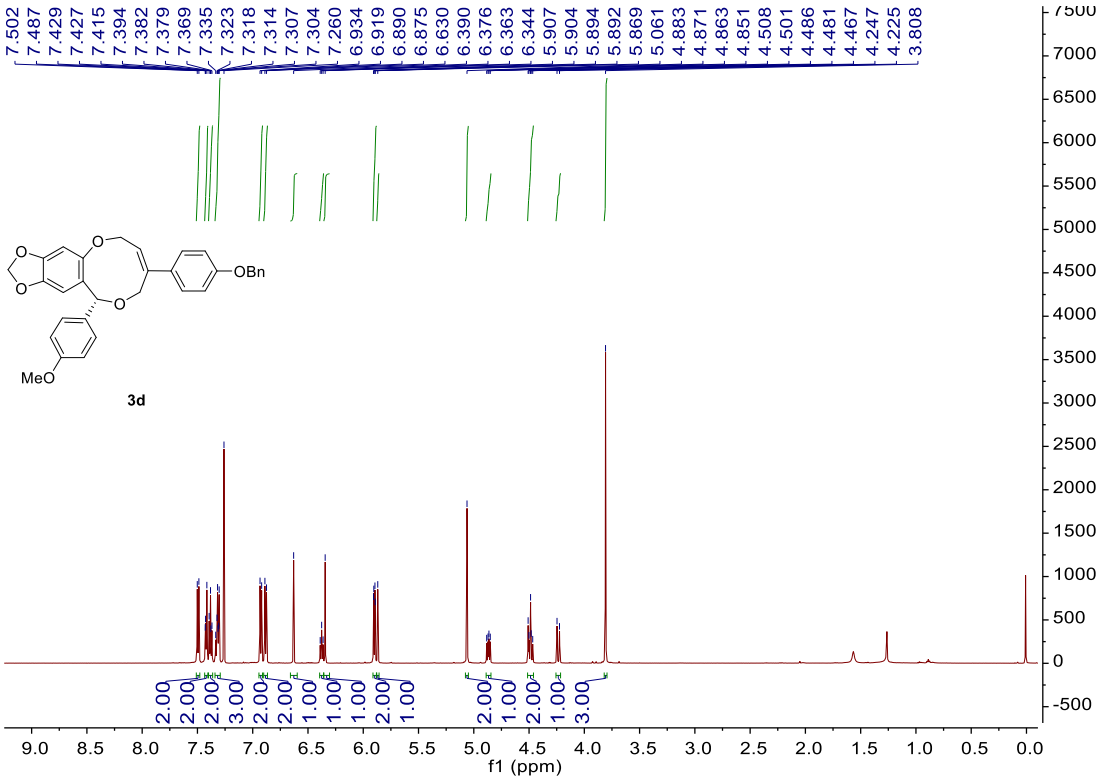
3c
f1 (ppm)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6.363
6.344
5.907
5.904
5.894
5.892
5.869
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4.863
4.851
4.508
4.501
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2.00
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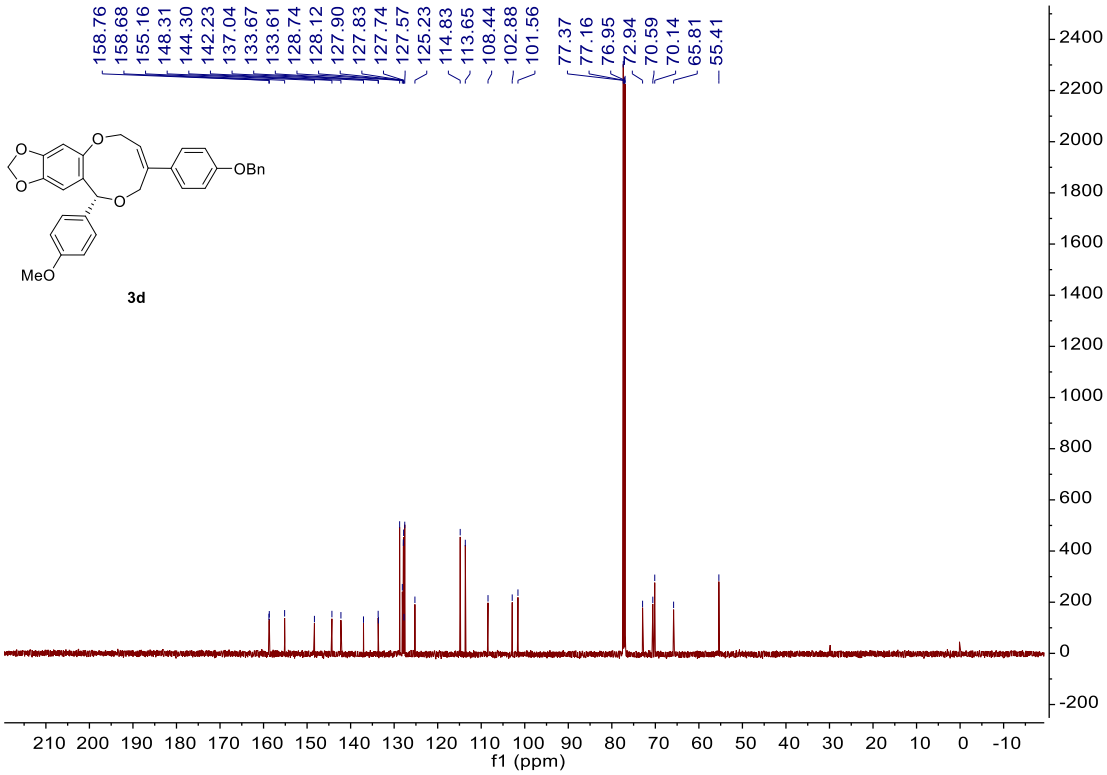
3.00
f1 (ppm)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101.56
77.37
77.16
76.95
72.94
70.59
70.14
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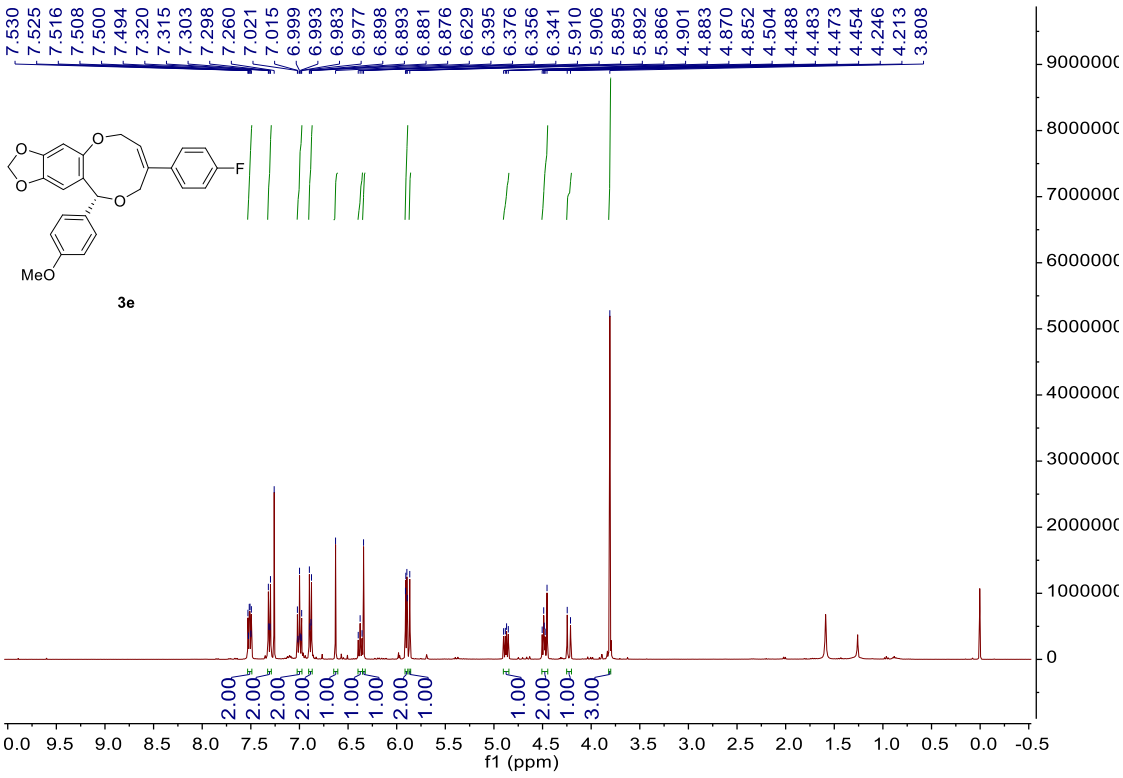
55.41
OBn
MeO
3d
f1 (ppm)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7.260
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7.015
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6.993
6.983
6.977
6.898
6.893
6.881
6.876
6.629
6.395
6.376
6.356
6.341
5.910
5.906
5.895
5.892
5.866
4.901
4.883
4.870
4.852
4.504
4.488
4.483
4.473
4.454
4.246
4.213
3.808
F
O
O
O
O
MeO
3e
2.00
2.00
2.00
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2.00
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2.00
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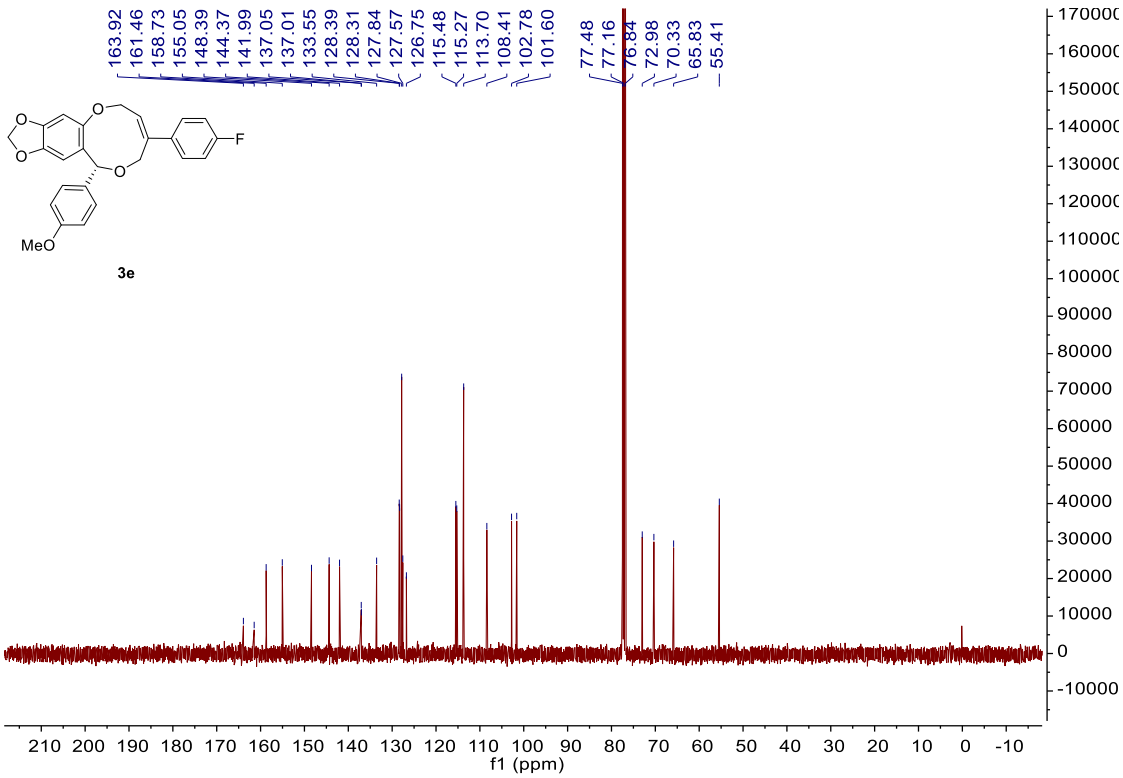
3.00
f1 (ppm)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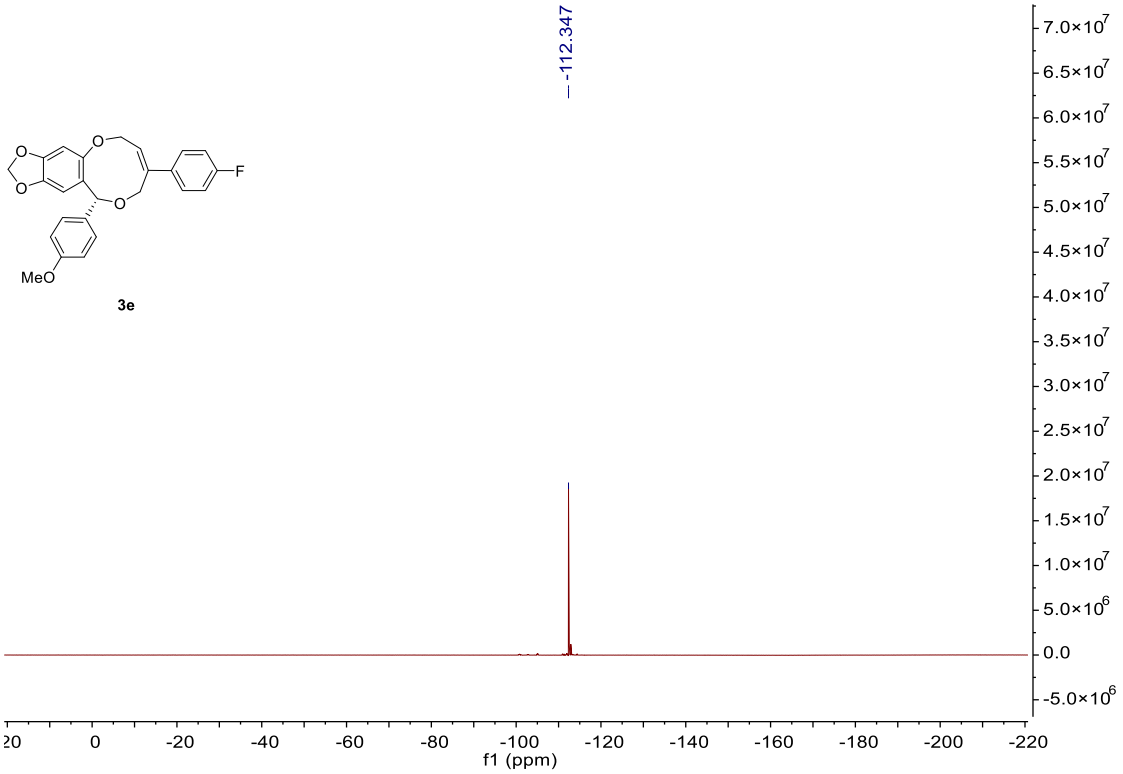
144.37
141.99
137.05
137.01
133.55
128.39
128.31
127.84
127.57
126.75
115.48
115.27
113.70
108.41
102.78
101.60
77.48
77.16
76.84
72.98
70.33
65.83
55.41
F
O
O
O
O
MeO
3e
f1 (ppm)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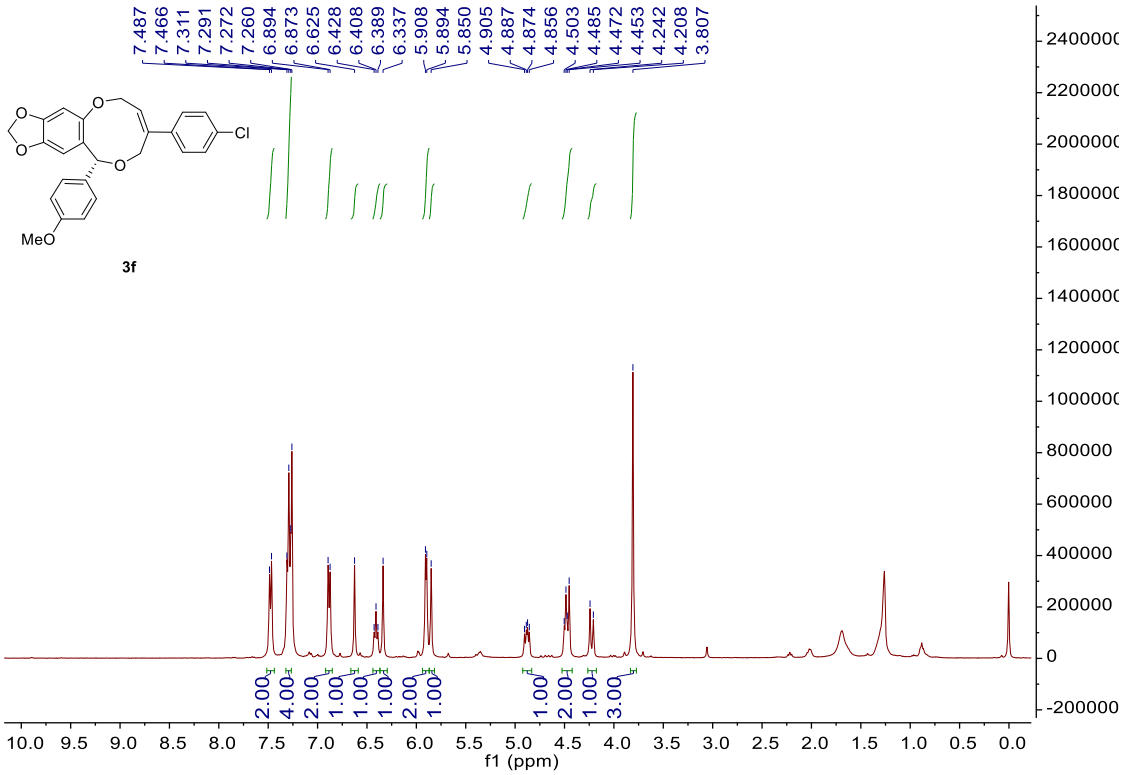
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6.389
6.337
5.908
5.894
5.850
4.905
4.887
4.874
4.856
4.503
4.485
4.472
4.453
4.242
4.208
3.807
Cl
MeO
3f
2.00
4.00
2.00
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1.00
1.00
2.00
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1.00
2.00
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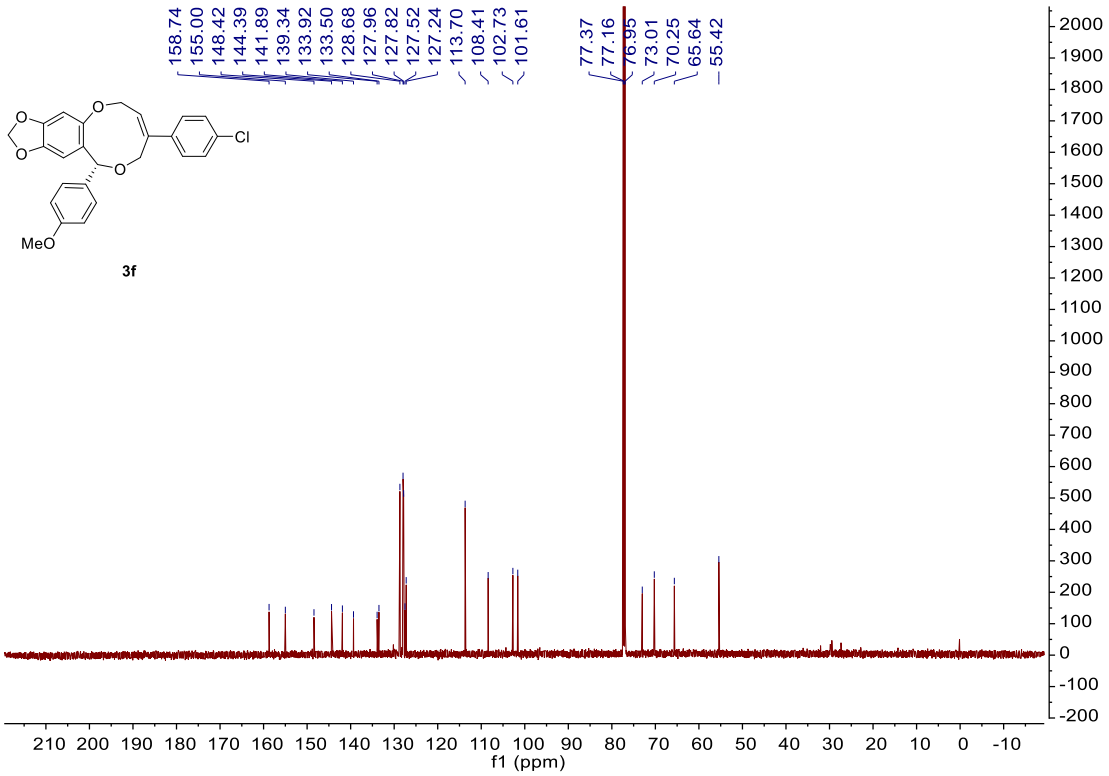
3.00
f1 (ppm)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133.50
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127.82
127.52
127.24
113.70
108.41
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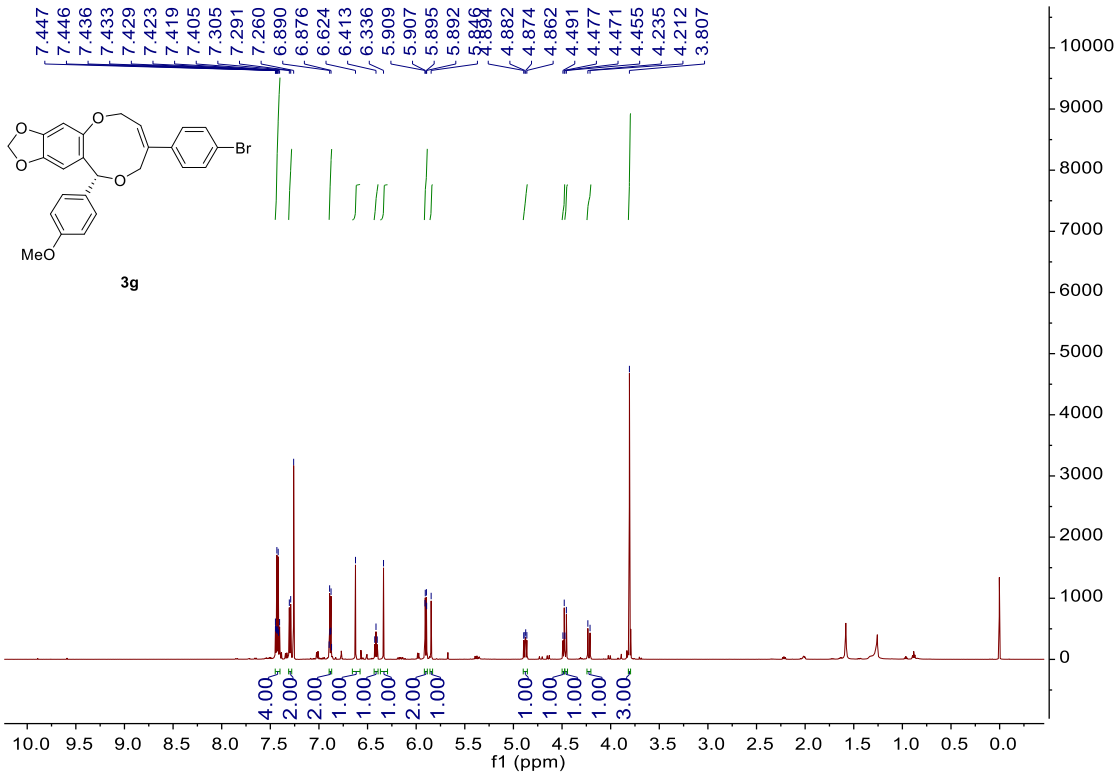
76.95
73.01
70.25
65.64
55.42
Cl
MeO
3f
f1 (ppm)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7.423
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7.291
7.260
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6.876
6.624
6.413
6.336
5.909
5.907
5.895
5.892
4.882
4.874
4.862
4.491
4.477
4.471
4.455
4.235
4.212
3.807
Br
MeO
3g
4.00
2.00
2.00
1.00
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1.00
2.00
1.00
1.00
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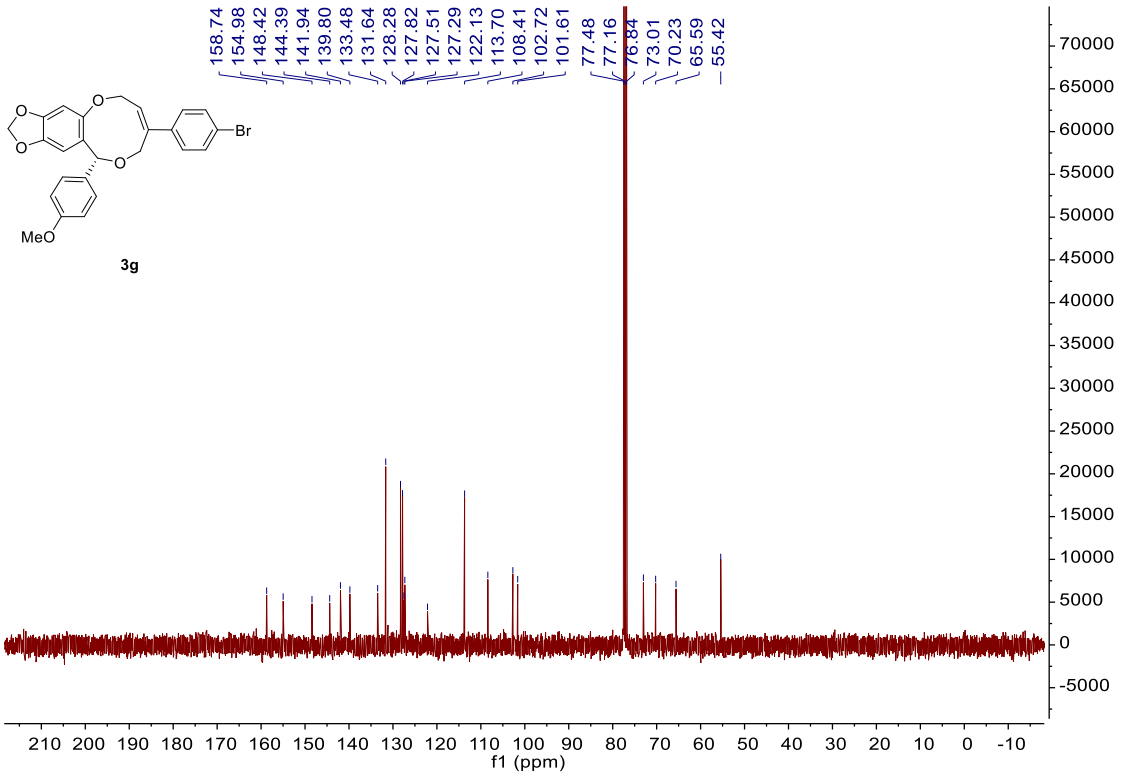
1.00
1.00
3.00
f1 (ppm)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122.13
113.70
108.41
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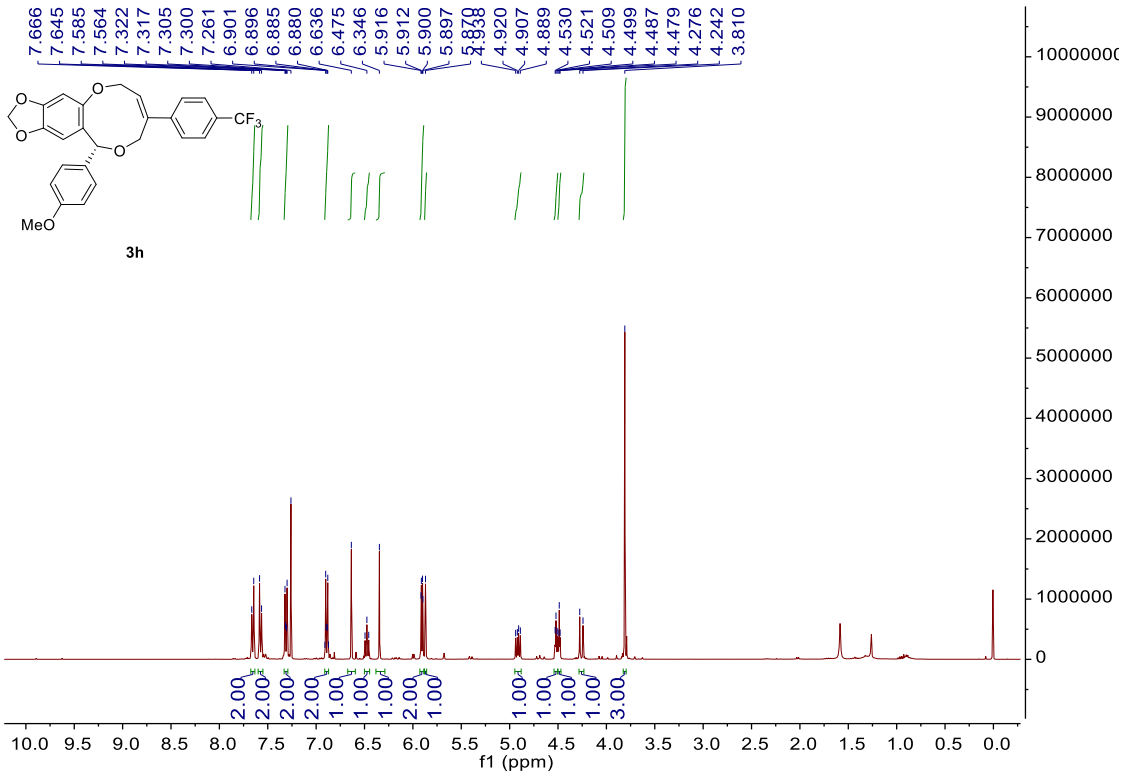
77.16
76.84
73.01
70.23
65.59
55.42
Br
MeO
3g
f1 (ppm)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6.896
6.885
6.880
6.636
6.475
6.346
5.916
5.912
5.900
5.897
4.938
4.920
4.907
4.889
4.530
4.521
4.509
4.499
4.487
4.479
4.276
4.242
3.810
CF3
MeO
3h
2.00
2.00
2.00
2.00
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1.00
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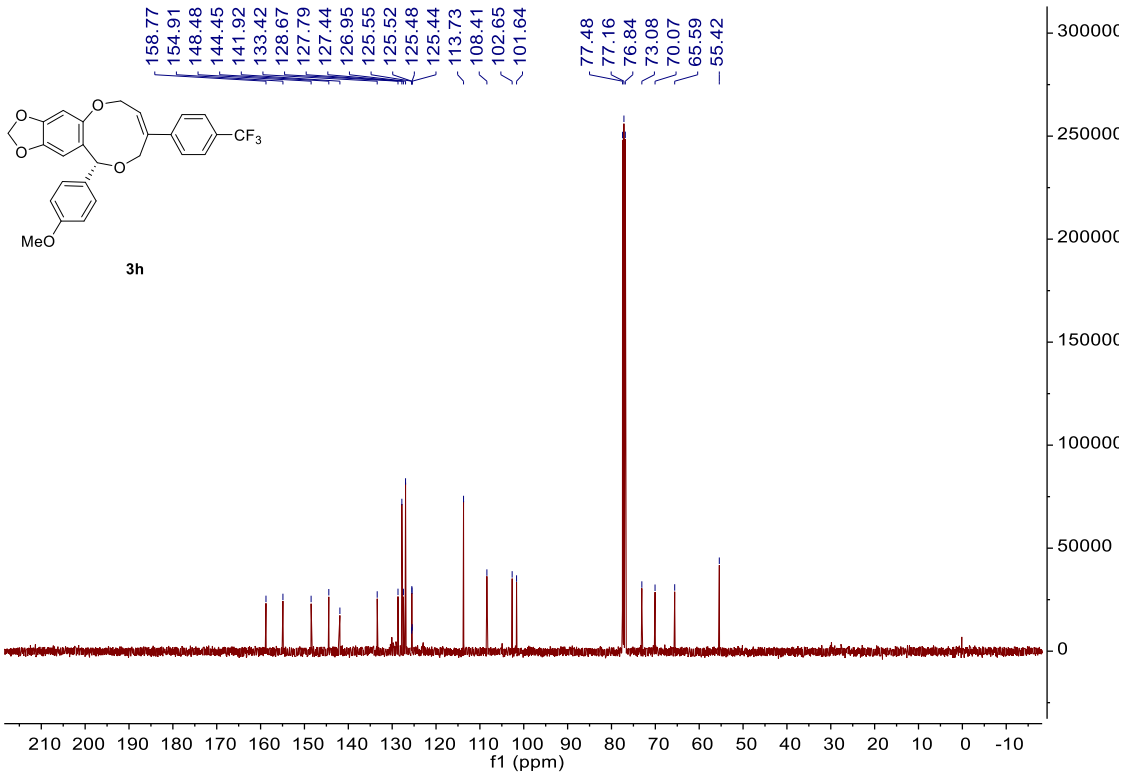
3.00
f1 (ppm)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125.52
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125.44
113.73
108.41
102.65
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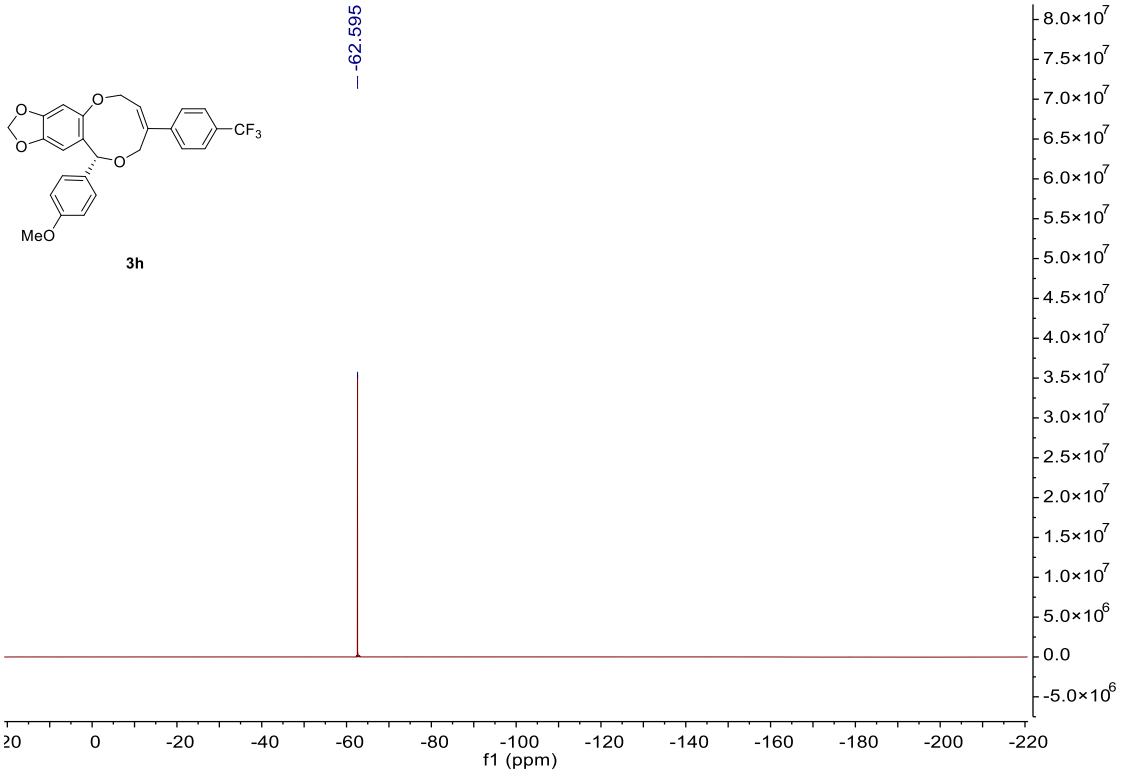
77.16
76.84
73.08
70.07
65.59
55.42
CF3
MeO
3h
f1 (ppm)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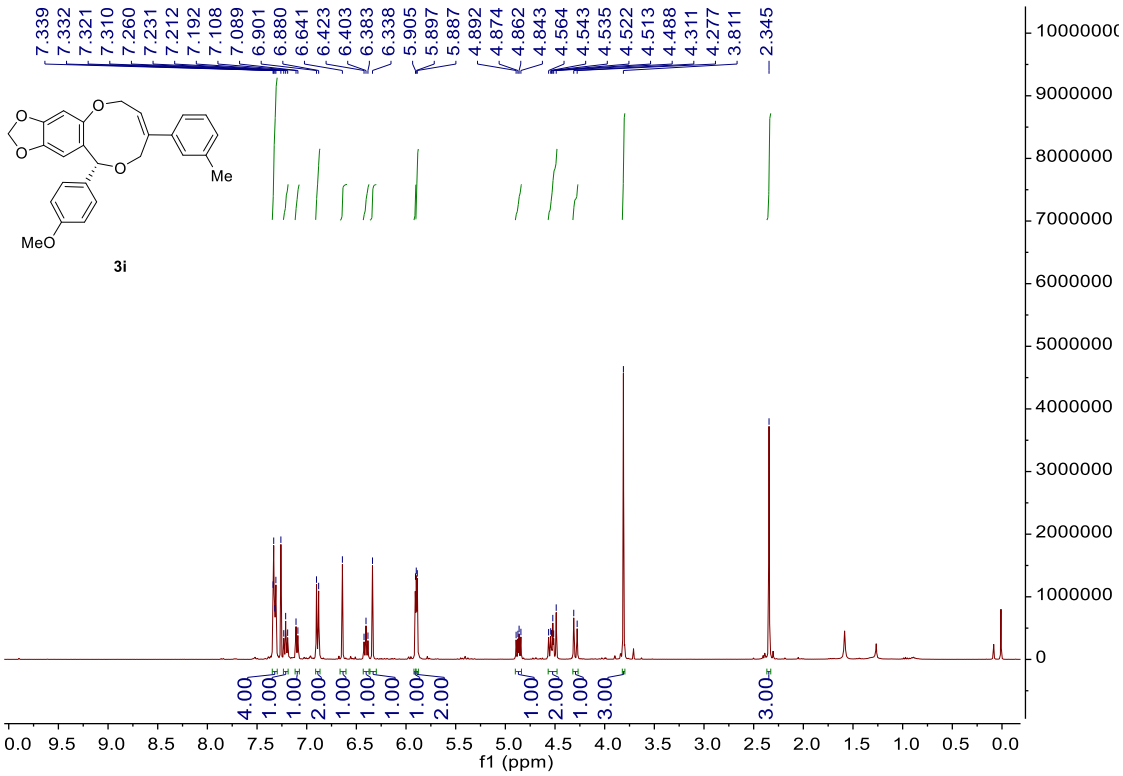
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7.108
7.089
6.901
6.880
6.641
6.423
6.403
6.383
6.338
5.905
5.897
5.887
4.892
4.874
4.862
4.843
4.564
4.543
4.535
4.522
4.513
4.488
4.311
4.277
3.811
2.345
Me
MeO
3i
4.00
1.00
1.00
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1.00
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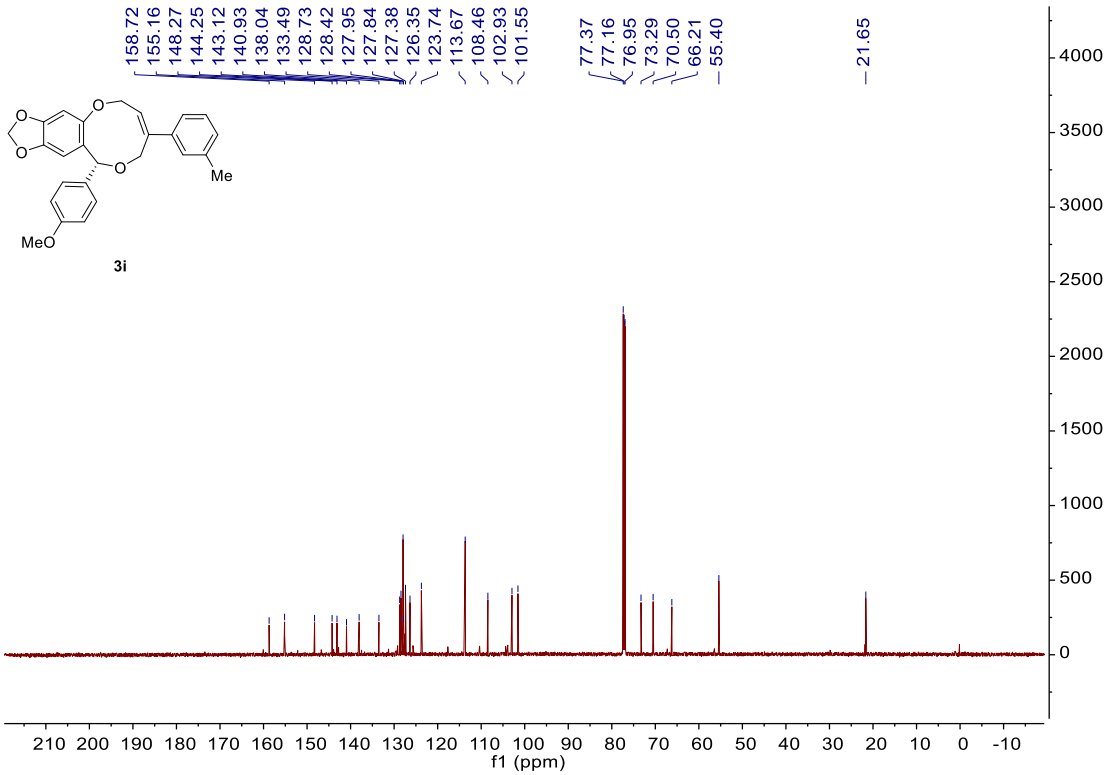
1.00
1.00
2.00
1.00
2.00
1.00
3.00
3.00
f1 (ppm)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126.35
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108.46
102.93
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76.95
73.29
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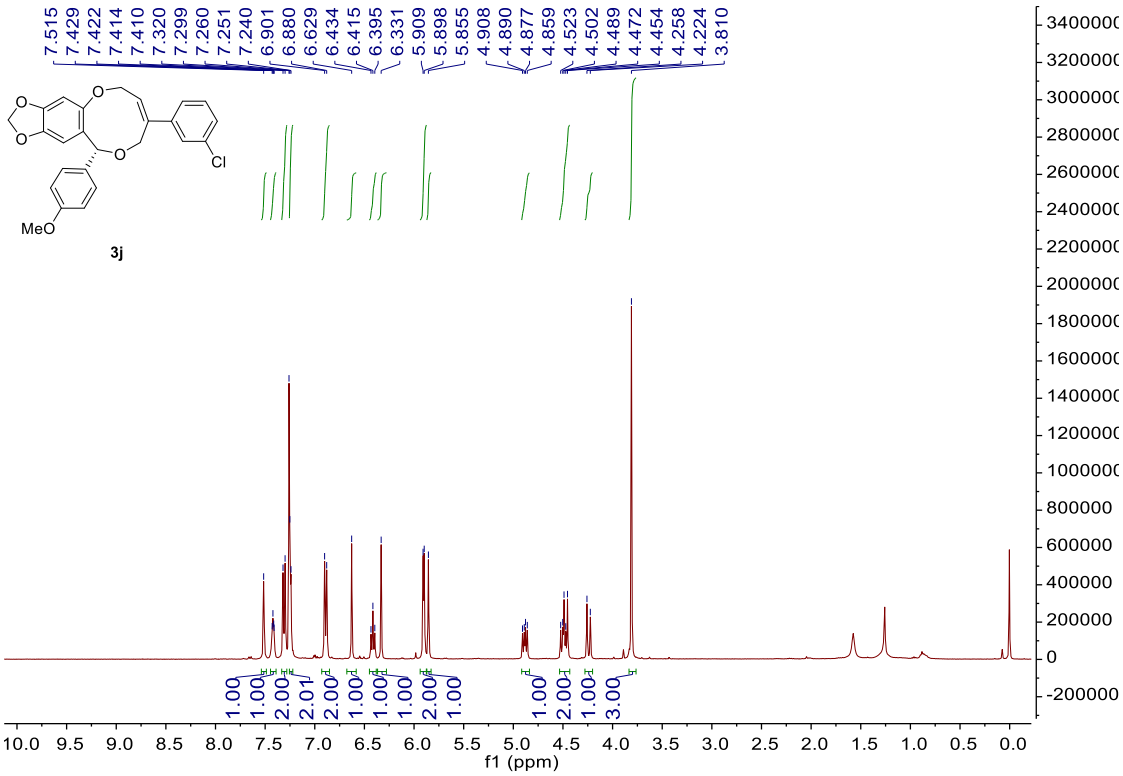
21.65
Me
MeO
3i
f1 (ppm)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7.299
7.260
7.251
7.240
6.901
6.880
6.629
6.434
6.415
6.395
6.331
5.909
5.898
5.855
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4.890
4.877
4.859
4.523
4.502
4.489
4.472
4.454
4.258
4.224
3.810
Cl
MeO
3j
1.00
1.00
2.00
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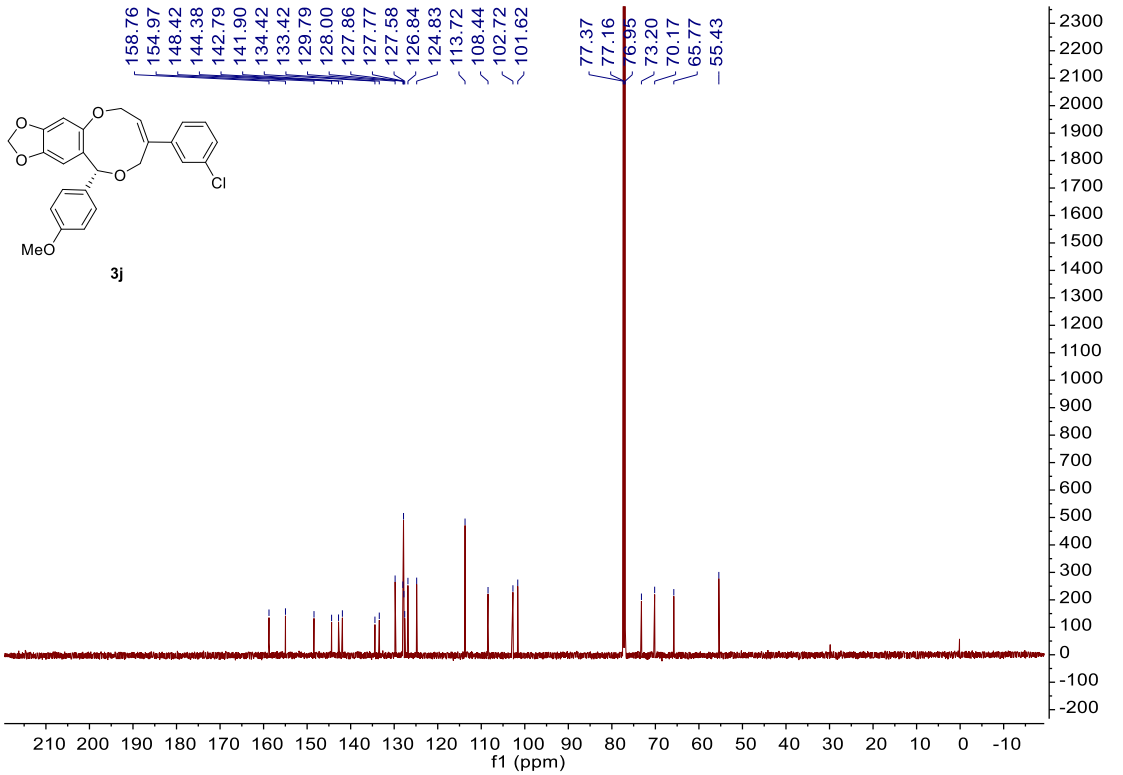
2.00
1.00
1.00
1.00
2.00
1.00
1.00
2.00
1.00
3.00
f1 (ppm)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127.58
126.84
124.83
113.72
108.44
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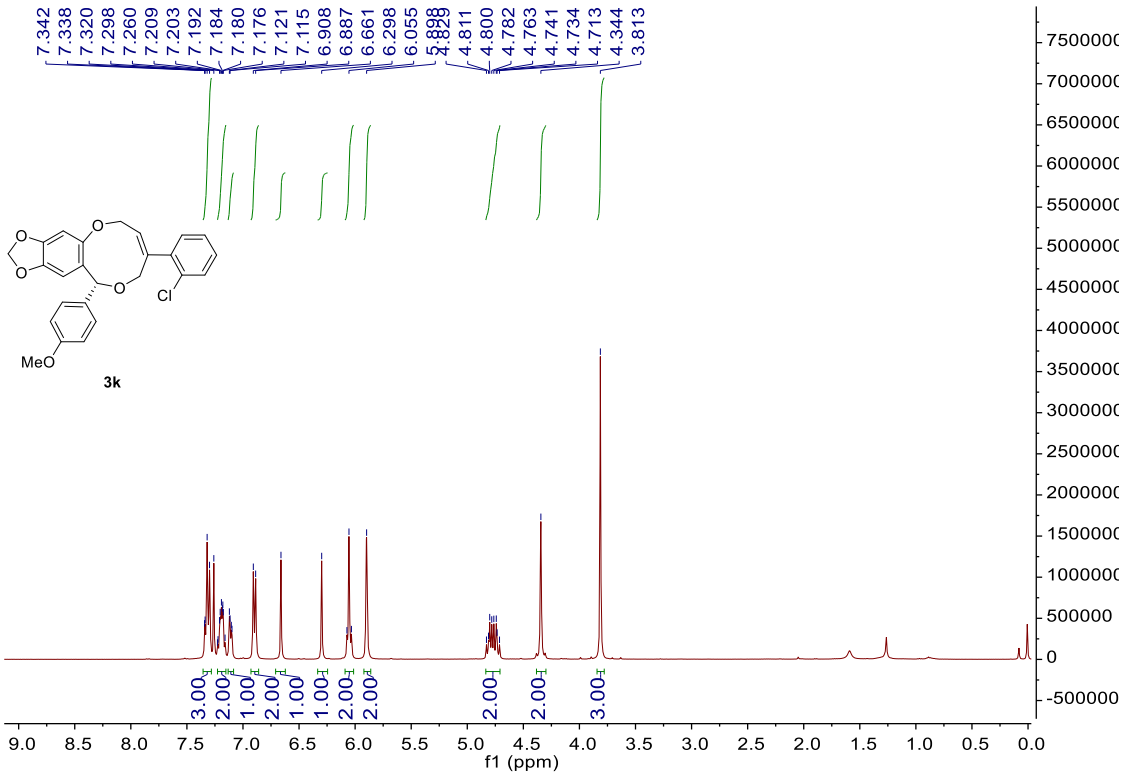
77.16
76.95
73.20
70.17
65.77
55.43
Cl
MeO
3j
f1 (ppm)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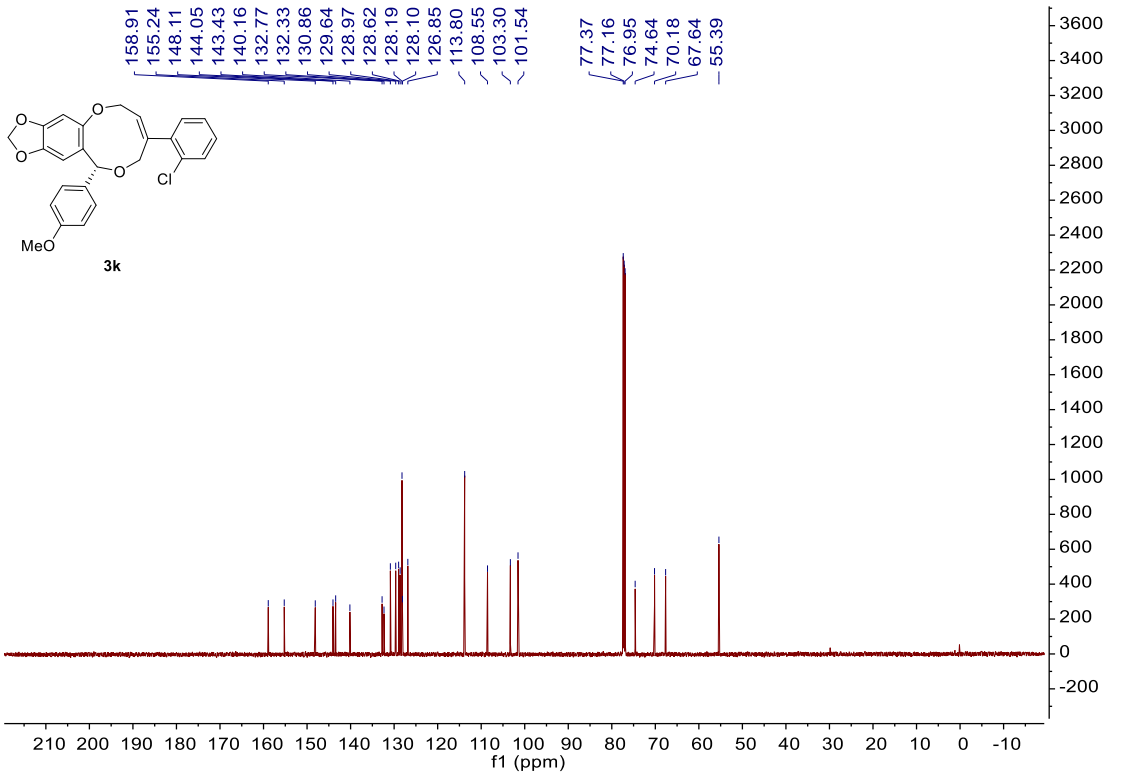
7.121
7.115
6.908
6.887
6.661
6.298
6.055
5.898
5.822
4.811
4.800
4.782
4.763
4.741
4.734
4.713
4.344
3.813
O
O
O
O
Cl
MeO
3k
3.00
2.00
1.00
2.00
1.00
1.00
2.00
2.00
2.00
2.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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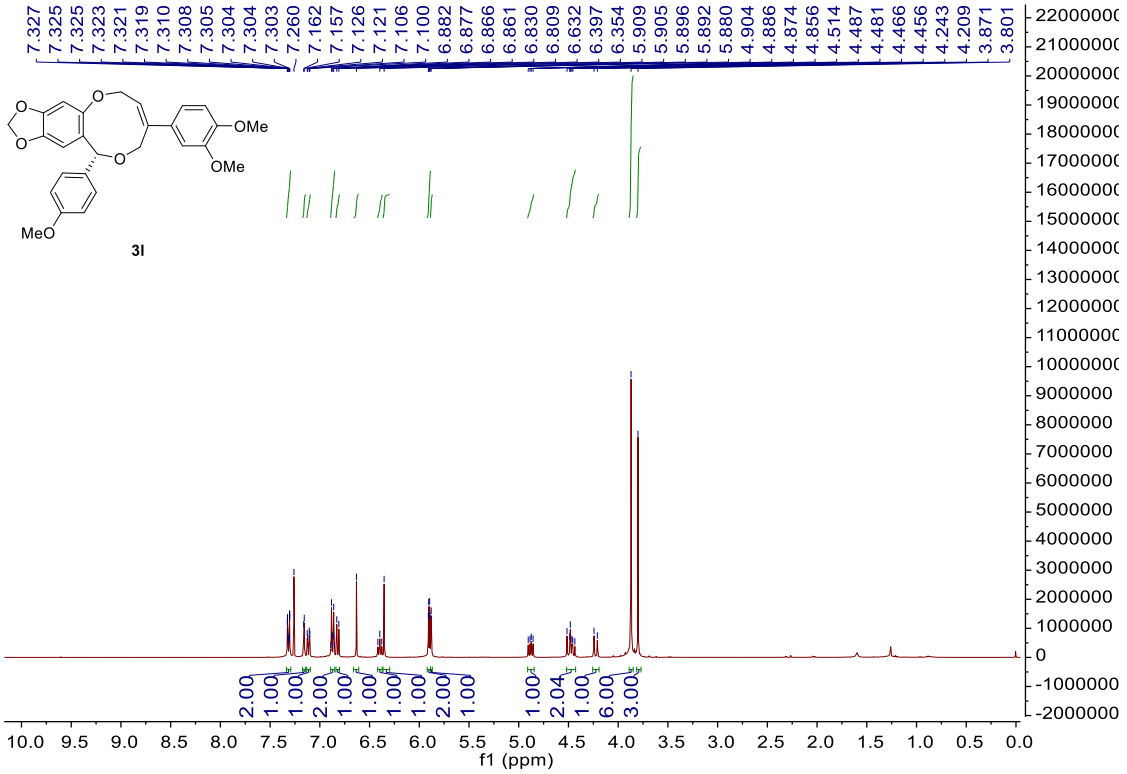
129.64
128.97
128.62
128.19
128.10
126.85
113.80
108.55
103.30
101.54
77.37
77.16
76.95
74.64
70.18
67.64
55.39
O
O
O
O
Cl
MeO
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.106
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6.877
6.866
6.861
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6.809
6.632
6.397
6.354
5.909
5.905
5.896
5.892
5.880
4.904
4.886
4.874
4.856
4.514
4.487
4.481
4.466
4.456
4.243
4.209
3.871
3.801
OMe
OMe
MeO
3l
2.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
2.00
1.00
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1.00
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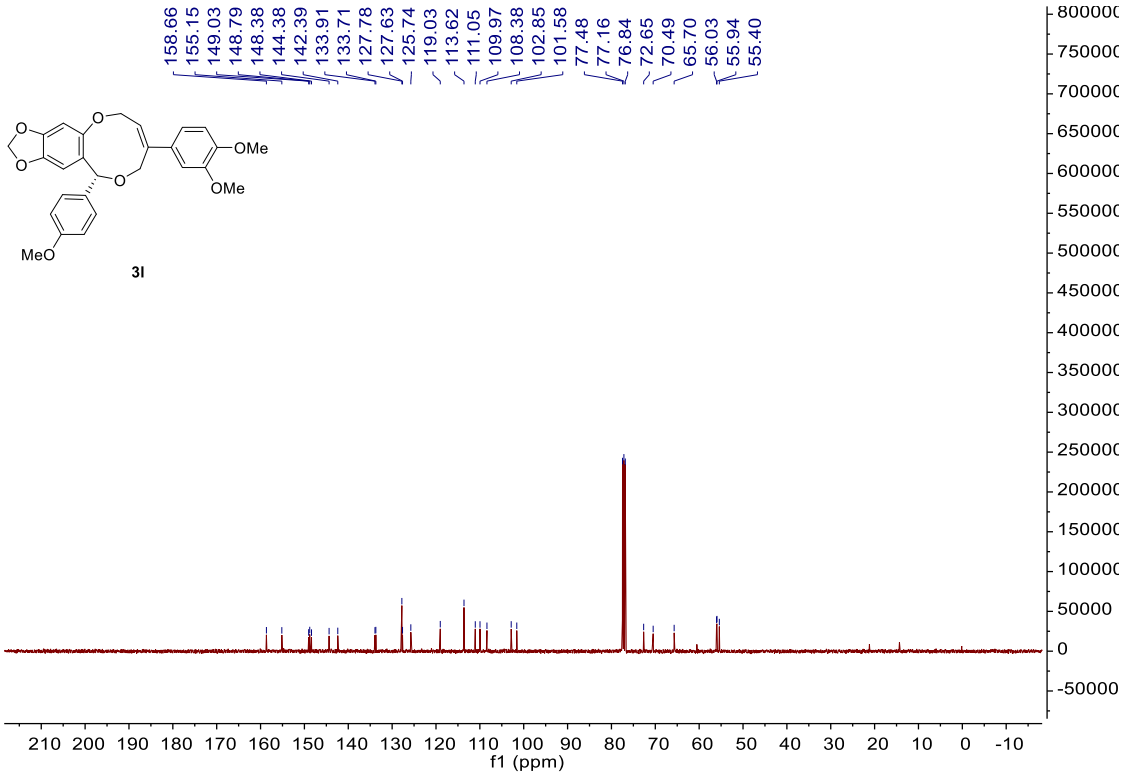
3.00
f1 (ppm)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125.74
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111.05
109.97
108.38
102.85
101.58
77.48
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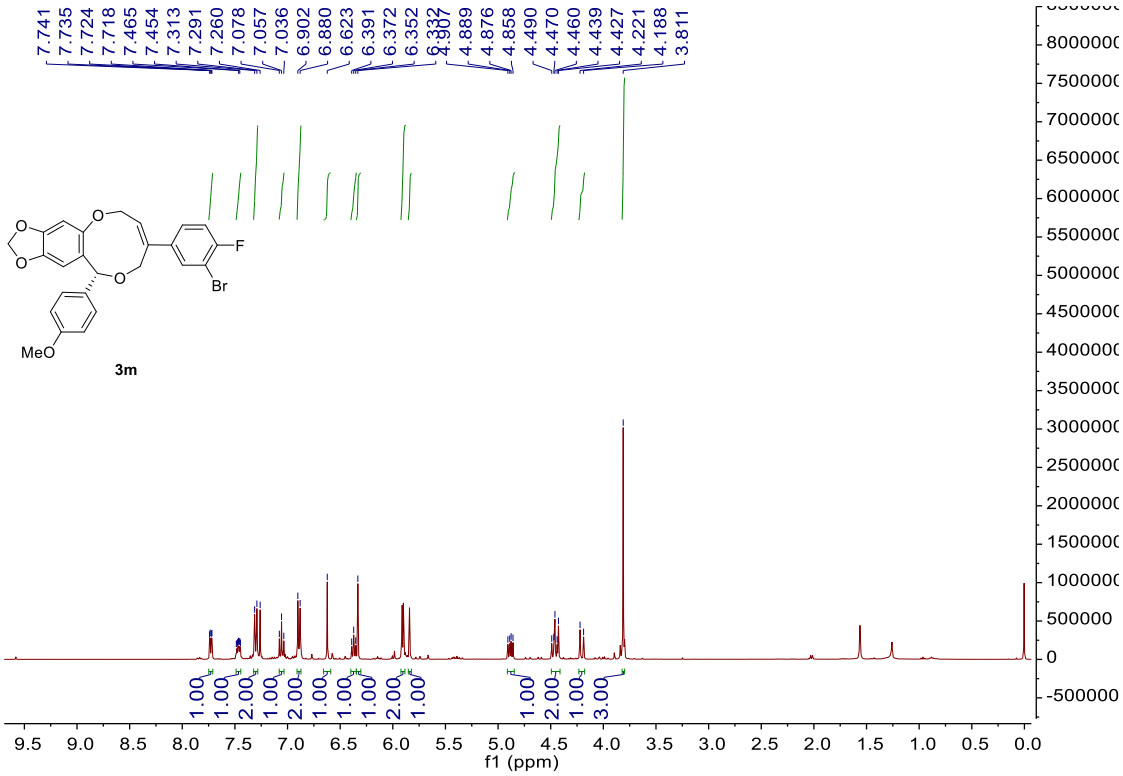
70.49
65.70
56.03
55.94
55.40
OMe
OMe
MeO
3l
f1 (ppm)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7.036
6.902
6.880
6.623
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6.372
6.352
6.332
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4.858
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4.221
4.188
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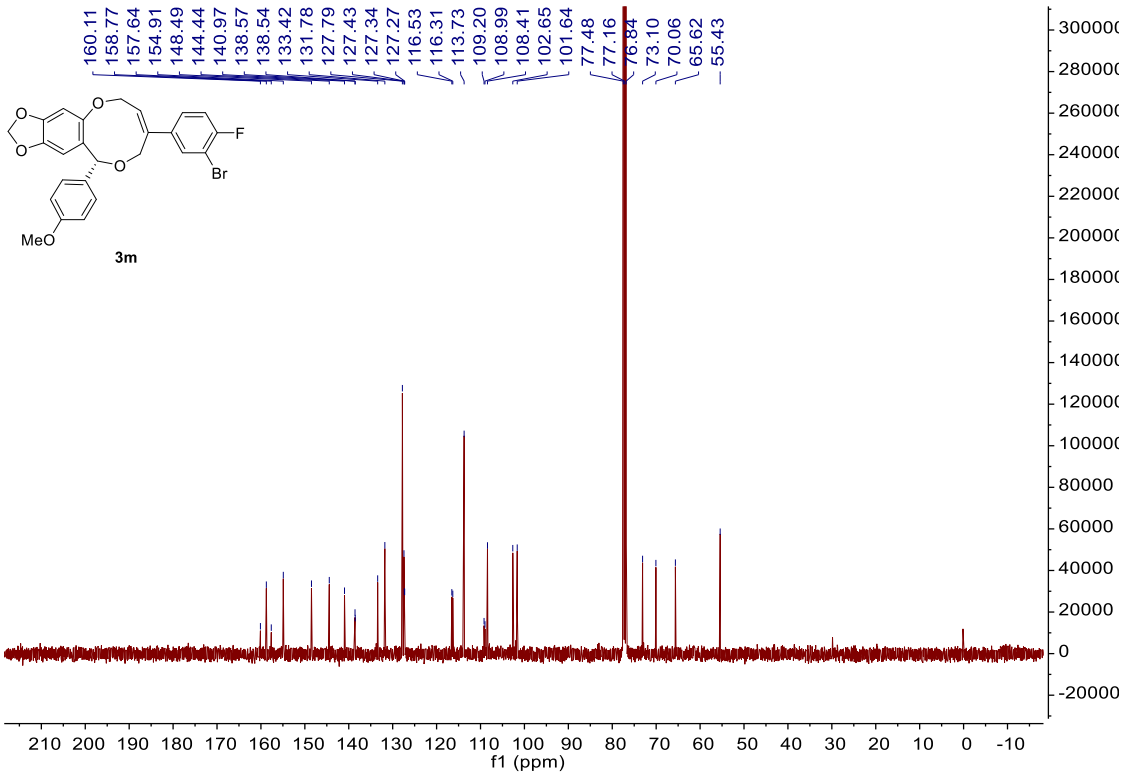
F
Br
MeO
3m
f1 (ppm)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127.43
127.34
127.27
116.53
116.31
113.73
109.20
108.99
108.41
102.65
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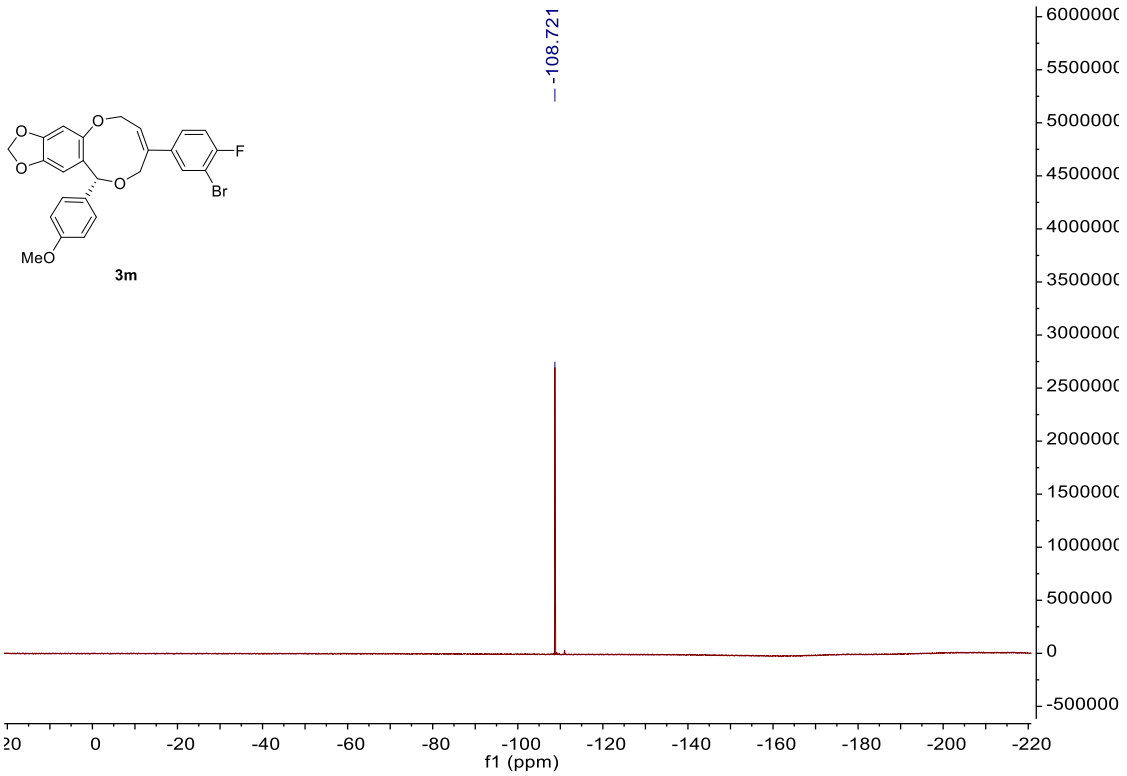
76.84
73.10
70.06
65.62
55.43
F
Br
MeO
3m
f1 (ppm)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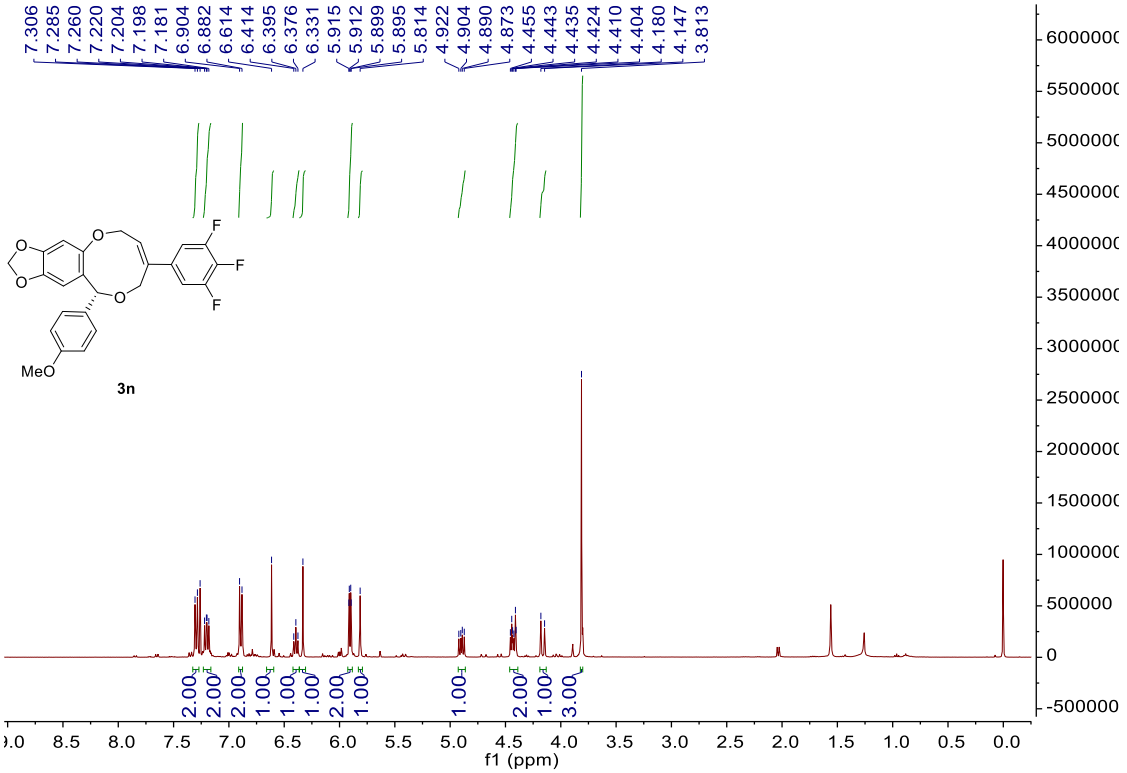
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4.410
4.404
4.180
4.147
3.813
F
F
F
MeO
3n
2.00
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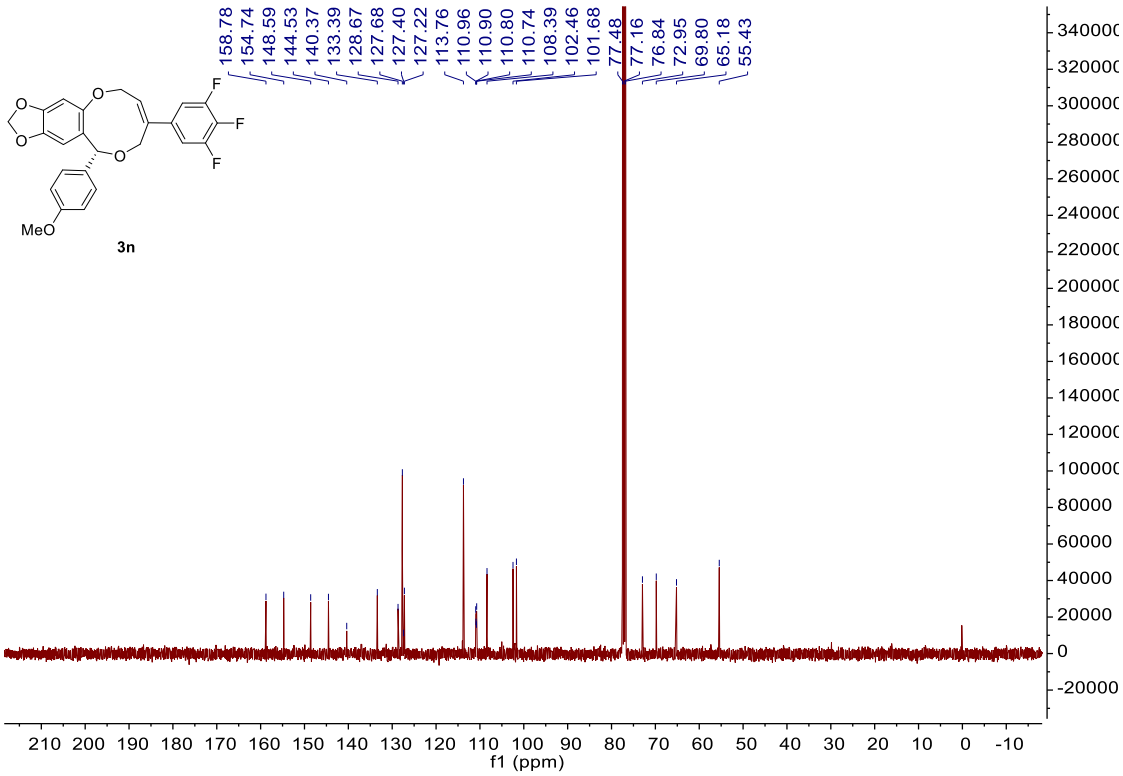
1.00
1.00
2.00
1.00
1.00
2.00
1.00
3.00
f1 (ppm)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110.74
108.39
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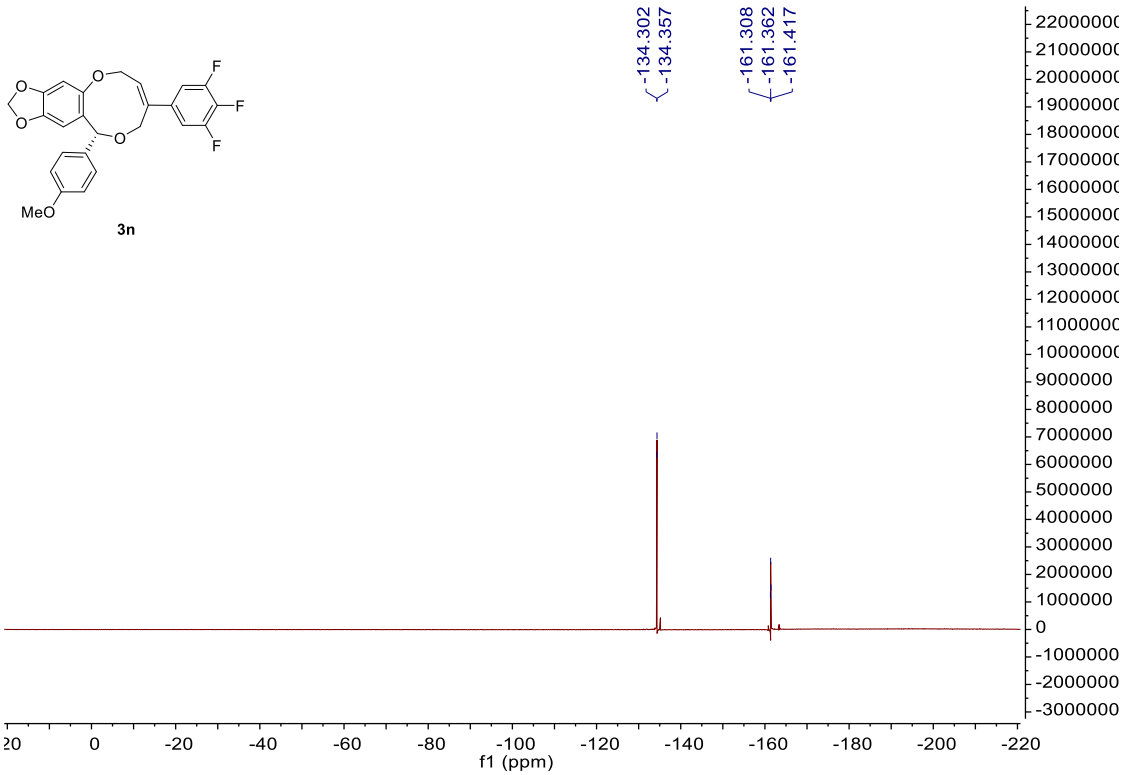
72.95
69.80
65.18
55.43
MeO
3n
f1 (ppm)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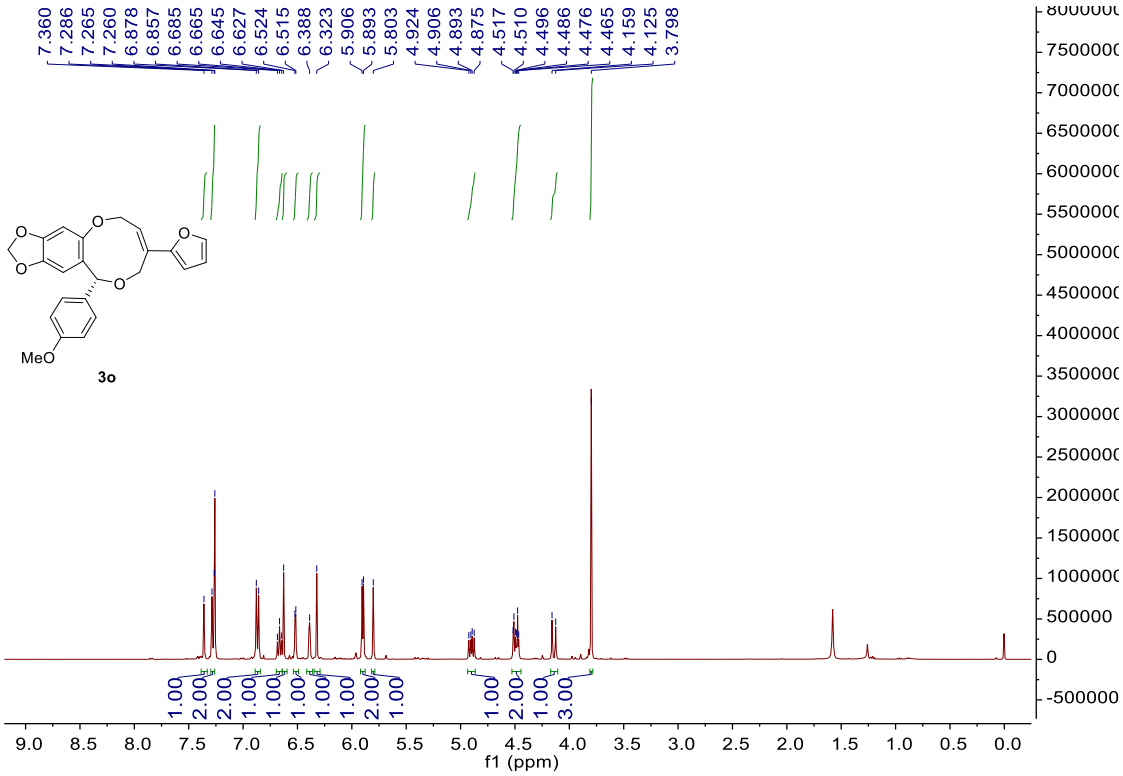
3n
f1 (ppm)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4.125
3.798
MeO
3o
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1.00
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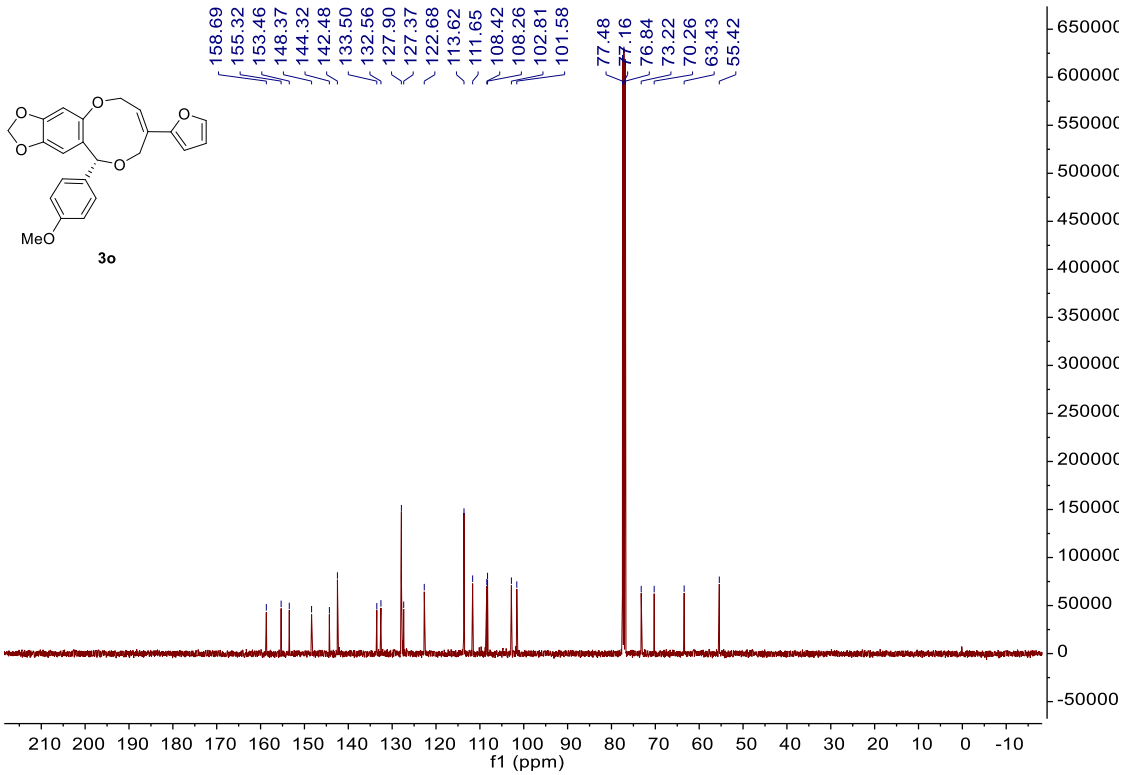
2.00
1.00
3.00
f1 (ppm)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108.42
108.26
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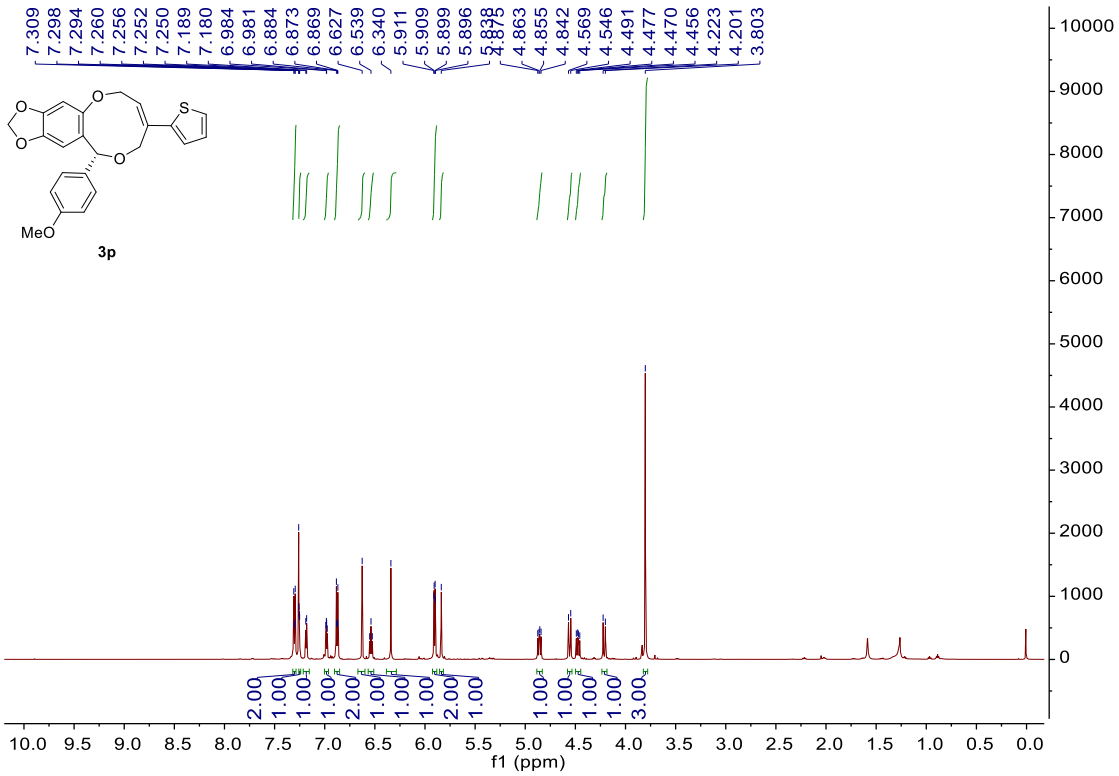
MeO
3o
f1 (ppm)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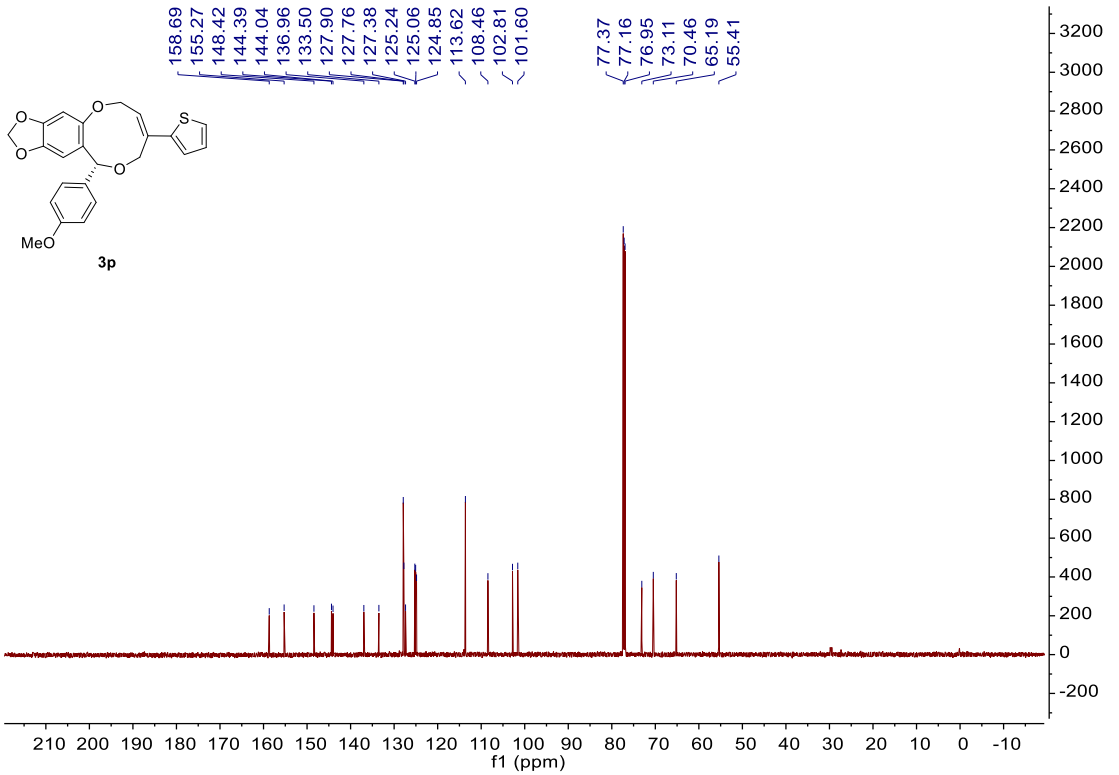
6.539
6.340
5.911
5.909
5.899
5.896
4.863
4.855
4.842
4.569
4.546
4.491
4.477
4.470
4.456
4.223
4.201
3.803
MeO
3p
2.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
1.00
3.00
f1 (ppm)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148.42
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144.04
136.96
133.50
127.90
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127.38
125.24
125.06
124.85
113.62
108.46
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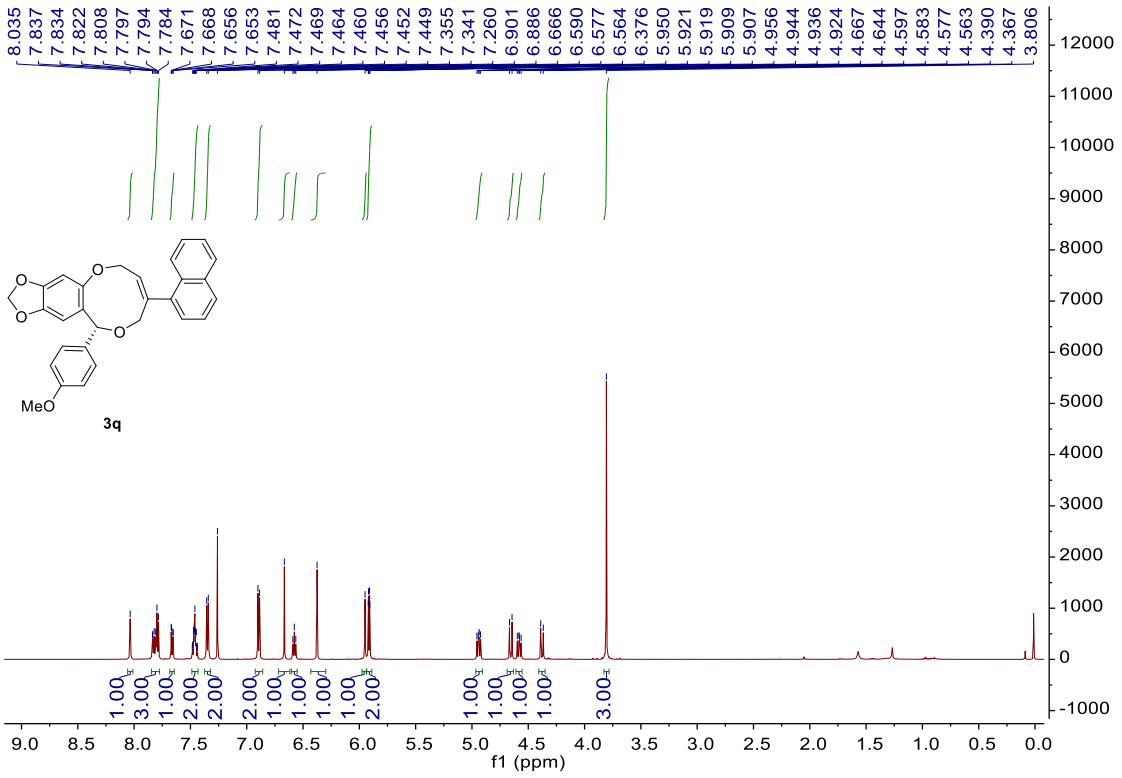
101.60
77.37
77.16
76.95
73.11
70.46
65.19
55.41
MeO
3p
f1 (ppm)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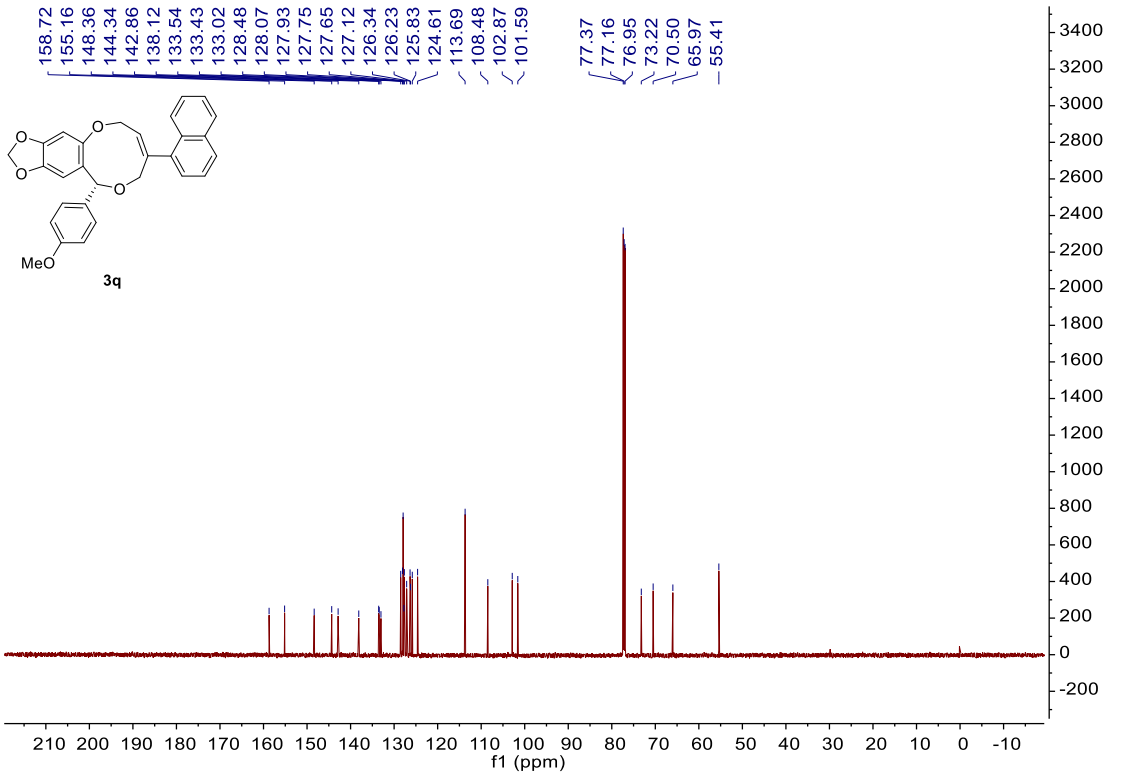
7.668
7.656
7.653
7.481
7.472
7.469
7.464
7.460
7.456
7.452
7.449
7.355
7.341
7.260
6.901
6.886
6.666
6.590
6.577
6.564
6.376
5.950
5.921
5.919
5.909
5.907
4.956
4.944
4.936
4.924
4.667
4.644
4.597
4.583
4.577
4.563
4.390
4.367
3.806
MeO
3q
1.00
3.00
1.00
2.00
2.00
2.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
3.00
f1 (ppm)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144.34
142.86
138.12
133.54
133.43
133.02
128.48
128.07
127.93
127.75
127.65
127.12
126.34
126.23
125.83
124.61
113.69
108.48
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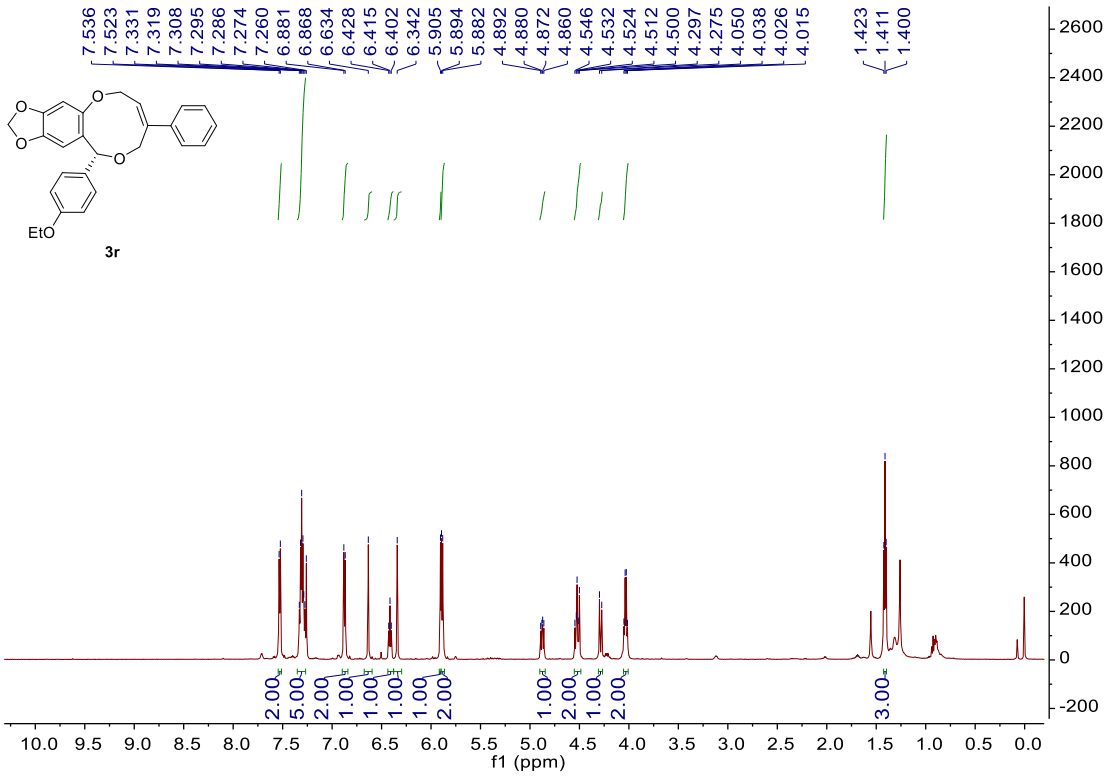
101.59
77.37
77.16
76.95
73.22
70.50
65.97
55.41
MeO
3q
f1 (ppm)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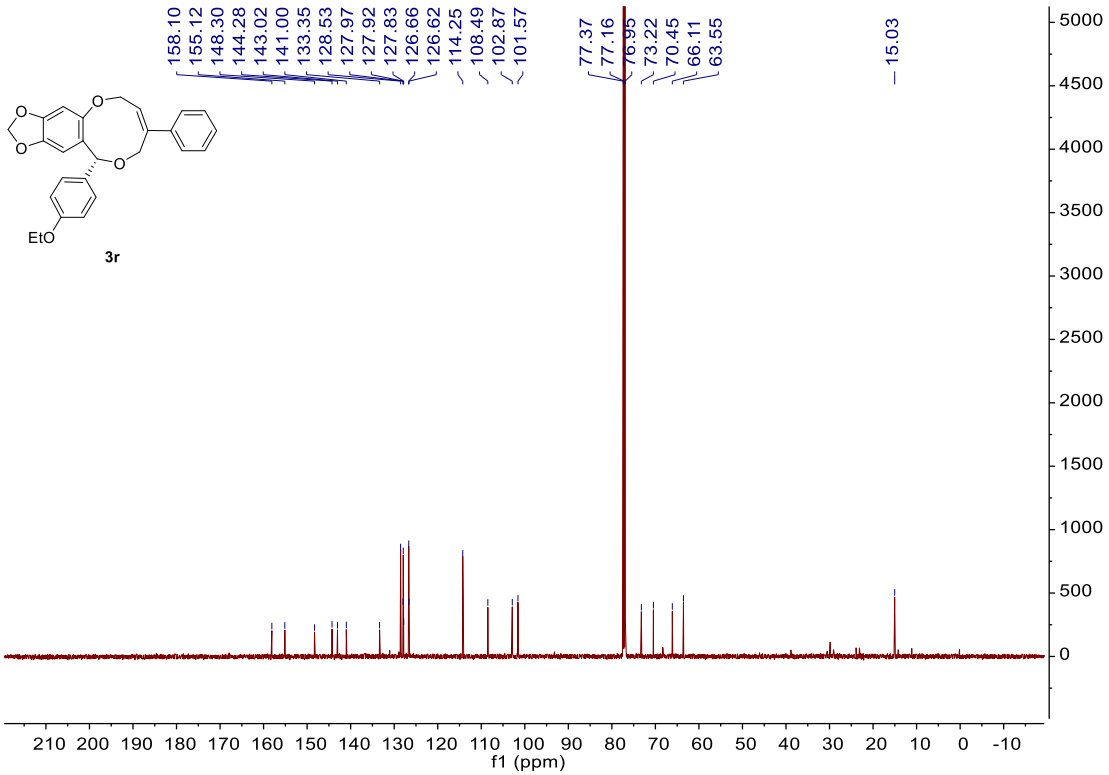
7.295
7.286
7.274
7.260
6.881
6.868
6.634
6.428
6.415
6.402
6.342
5.905
5.894
5.882
4.892
4.880
4.872
4.860
4.546
4.532
4.524
4.512
4.500
4.297
4.275
4.050
4.038
4.026
4.015
1.423
1.411
1.400
EtO
3r
2.00
5.00
2.00
1.00
1.00
1.00
1.00
2.00
1.00
2.00
1.00
2.00
3.00
f1 (ppm)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143.02
141.00
133.35
128.53
127.97
127.92
127.83
126.66
126.62
114.25
108.49
102.87
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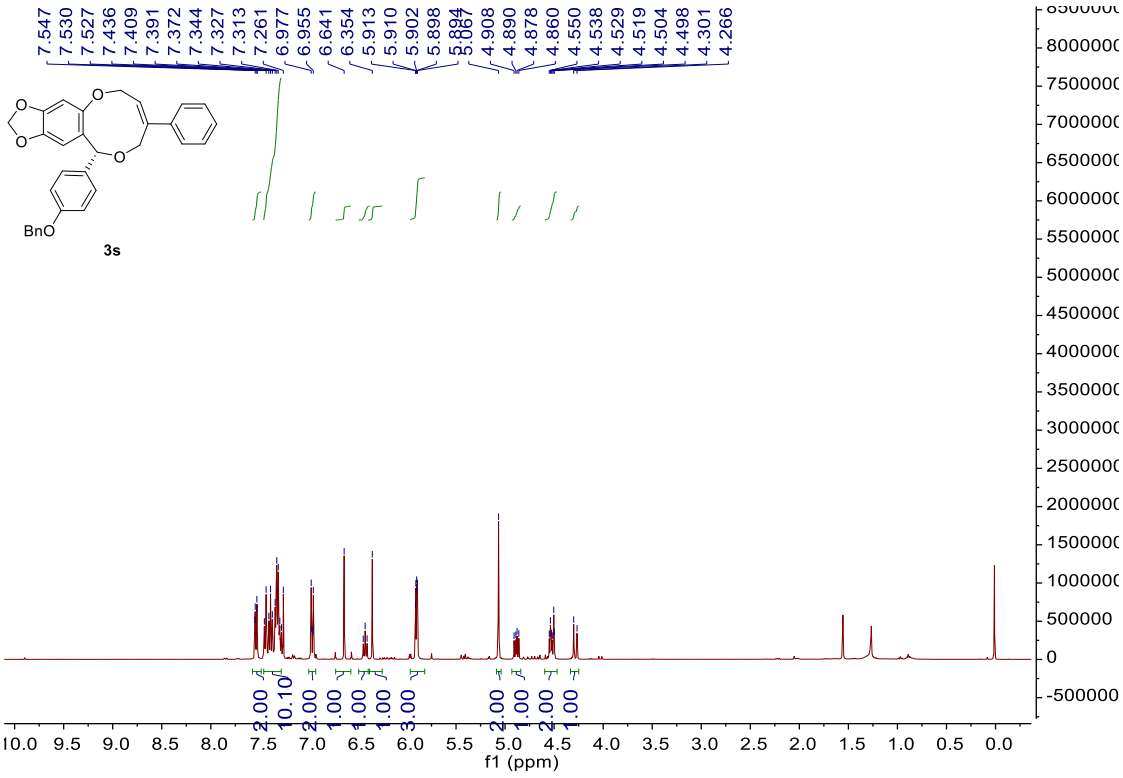
77.37
77.16
76.95
73.22
70.45
66.11
63.55
15.03
EtO
3r
f1 (ppm)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7.344
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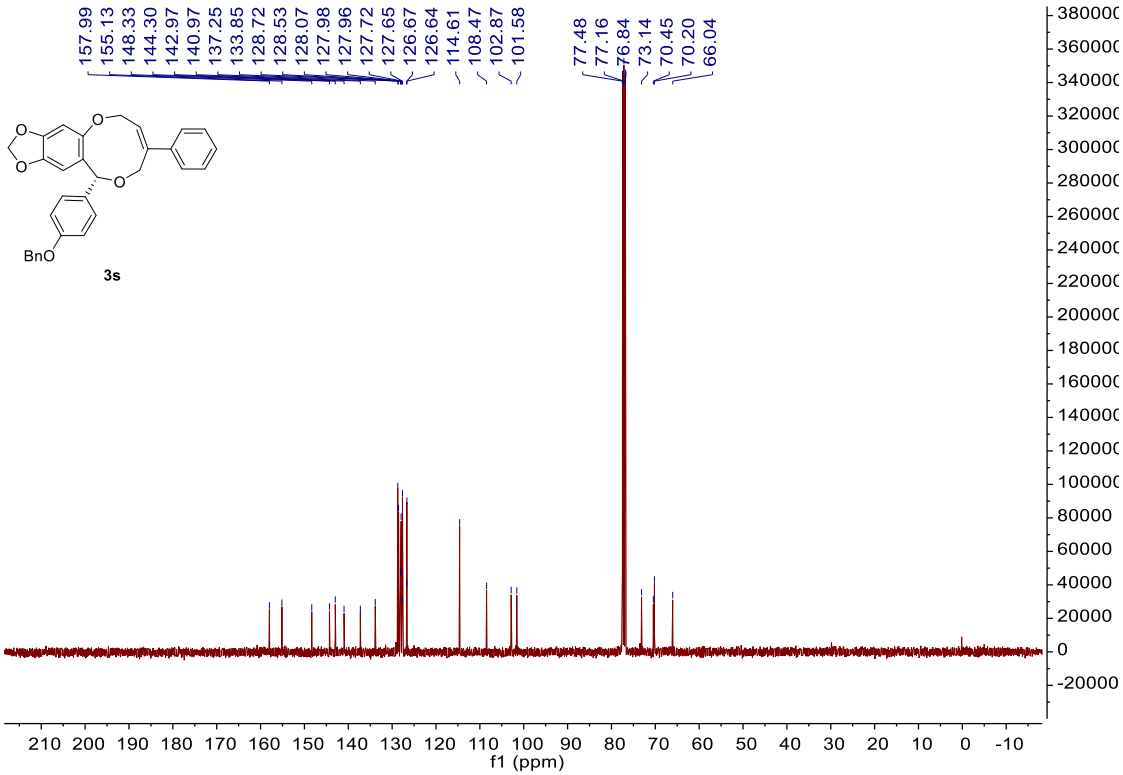
7.313
7.261
6.977
6.955
6.641
6.354
5.913
5.910
5.902
5.898
5.067
4.908
4.890
4.878
4.860
4.550
4.538
4.529
4.519
4.504
4.498
4.301
4.266
BnO
3s
2.00
10.10
2.00
1.00
1.00
1.00
3.00
2.00
1.00
2.00
1.00
f1 (ppm)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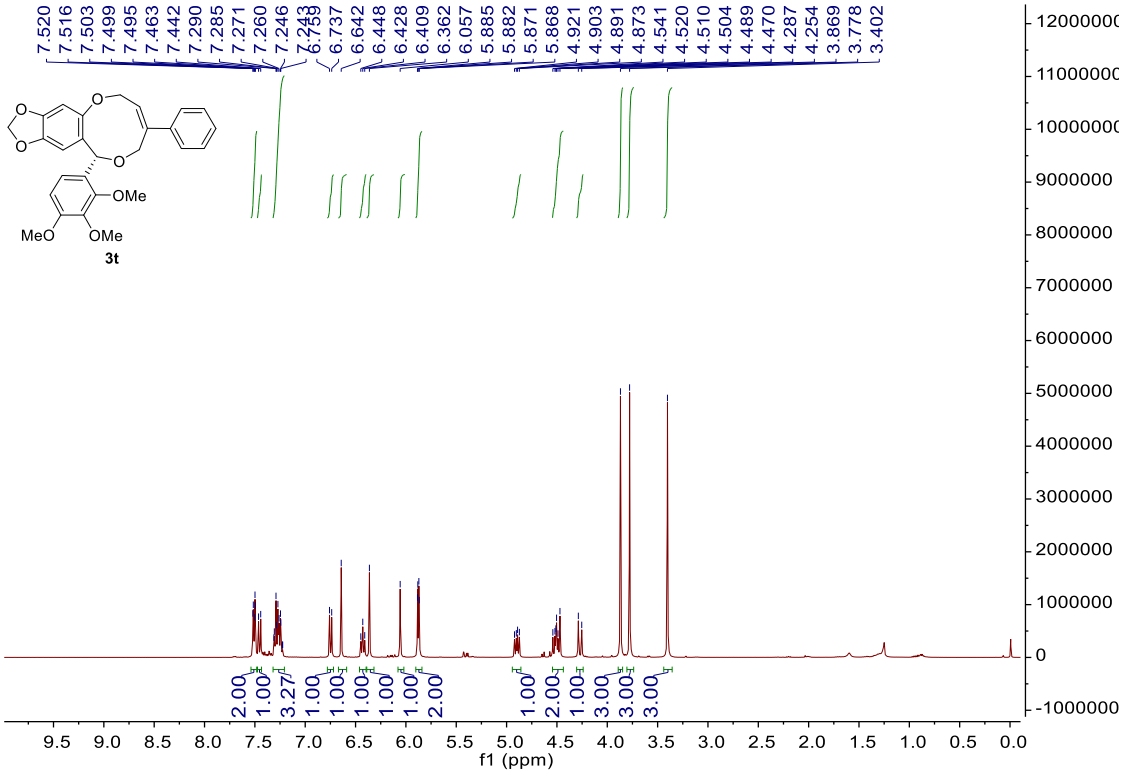
144.30
142.97
140.97
137.25
133.85
128.72
128.53
128.07
127.98
127.96
127.72
127.65
126.67
126.64
114.61
108.47
102.87
101.58
77.48
77.16
76.84
73.14
70.45
70.20
66.04
BnO
3s
f1 (ppm)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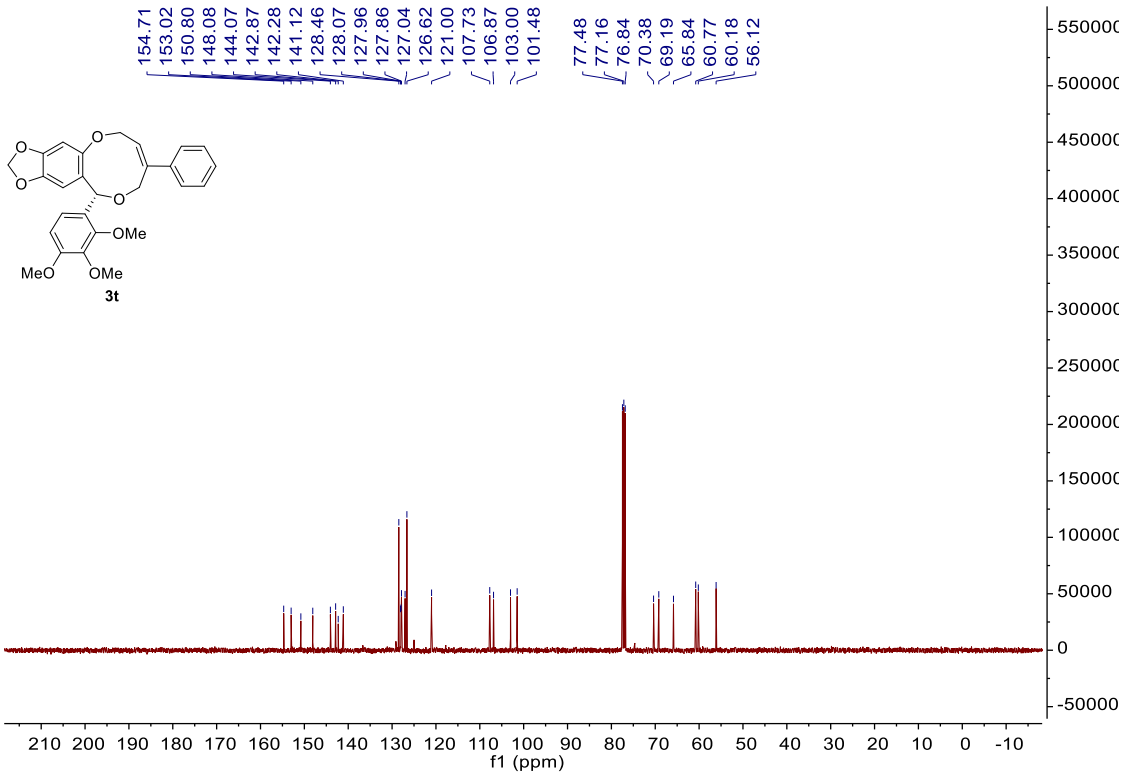
7.503
7.499
7.495
7.463
7.442
7.290
7.285
7.271
7.260
7.246
6.737
6.642
6.448
6.428
6.409
6.362
6.057
5.885
5.882
5.871
5.868
4.921
4.903
4.891
4.873
4.541
4.520
4.510
4.504
4.489
4.470
4.287
4.254
3.869
3.778
3.402
OMe
MeO
OMe
3t
2.00
1.00
3.27
1.00
1.00
1.00
1.00
2.00
1.00
2.00
1.00
3.00
3.00
3.00
f1 (ppm)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144.07
142.87
142.28
141.12
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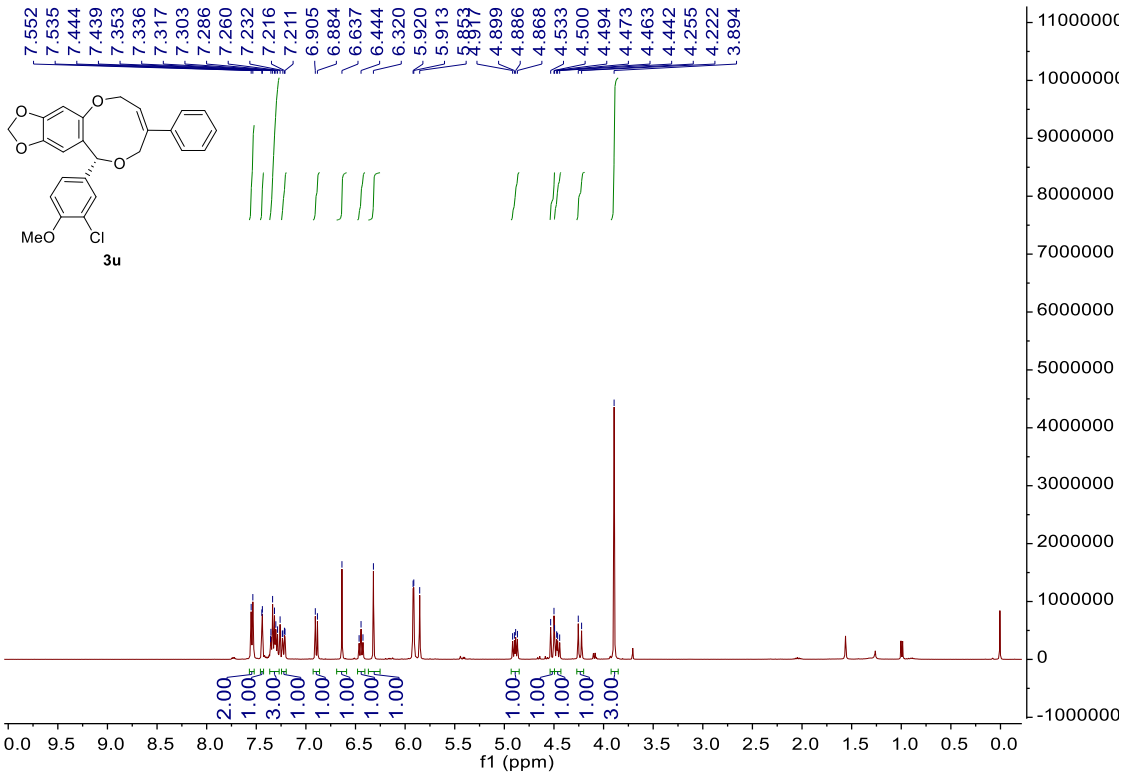
128.07
127.96
127.86
127.04
126.62
121.00
107.73
106.87
103.00
101.48
77.48
77.16
76.84
70.38
69.19
65.84
60.77
60.18
56.12
OMe
MeO
OMe
3t
f1 (ppm)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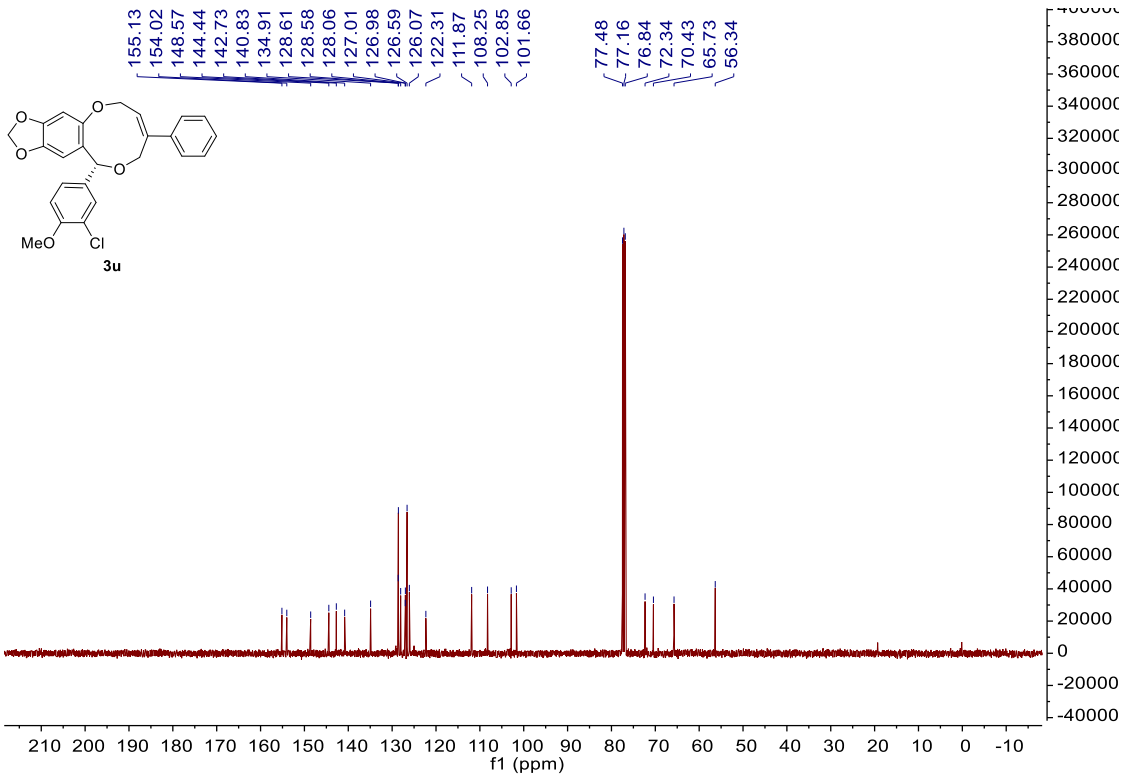
7.260
7.232
7.216
7.211
6.905
6.884
6.637
6.444
6.320
5.920
5.913
4.899
4.886
4.868
4.533
4.500
4.494
4.473
4.463
4.442
4.255
4.222
3.894
MeO
Cl
3u
2.00
1.00
3.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
1.00
3.00
f1 (ppm)


155.13
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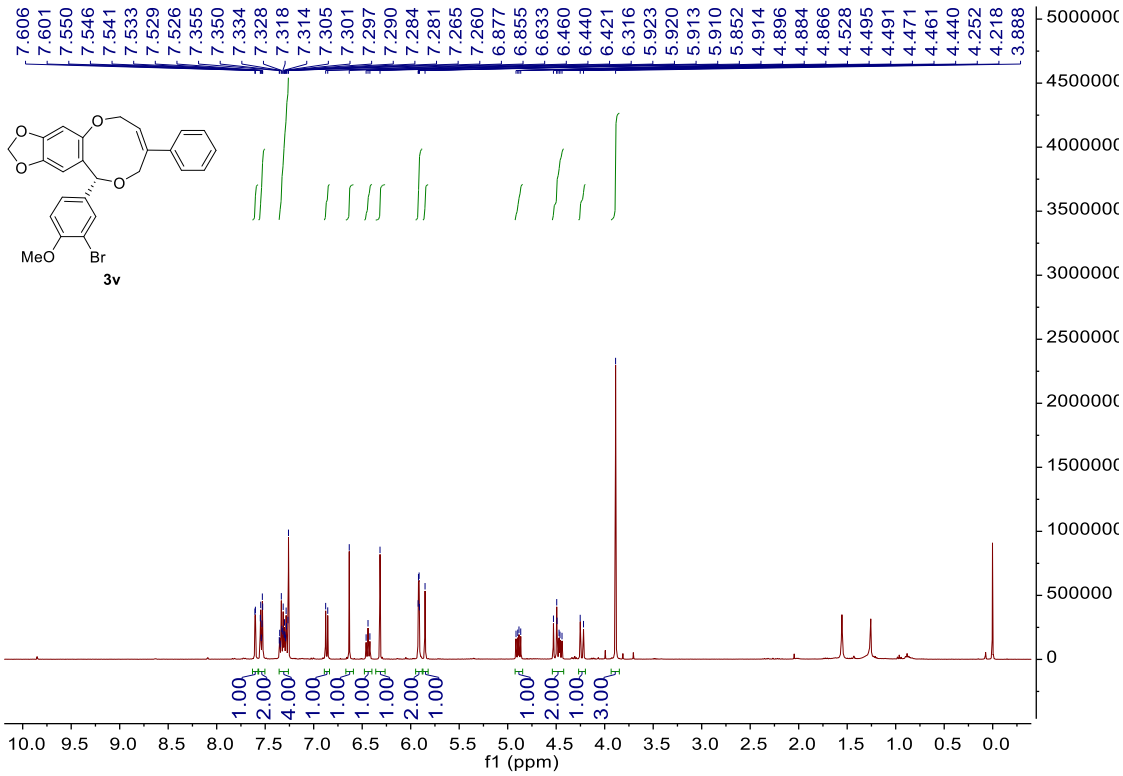
142.73
140.83
134.91
128.61
128.58
128.06
127.01
126.98
126.59
126.07
122.31
111.87
108.25
102.85
101.66
77.48
77.16
76.84
72.34
70.43
65.73
56.34
MeO
Cl
3u
f1 (ppm)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7.334
7.328
7.318
7.314
7.305
7.301
7.297
7.290
7.284
7.281
7.265
7.260
6.877
6.855
6.633
6.460
6.440
6.421
6.316
5.923
5.920
5.913
5.910
5.852
4.914
4.896
4.884
4.866
4.528
4.495
4.491
4.471
4.461
4.440
4.252
4.218
3.888
MeO
Br
3v
1.00
2.00
4.00
1.00
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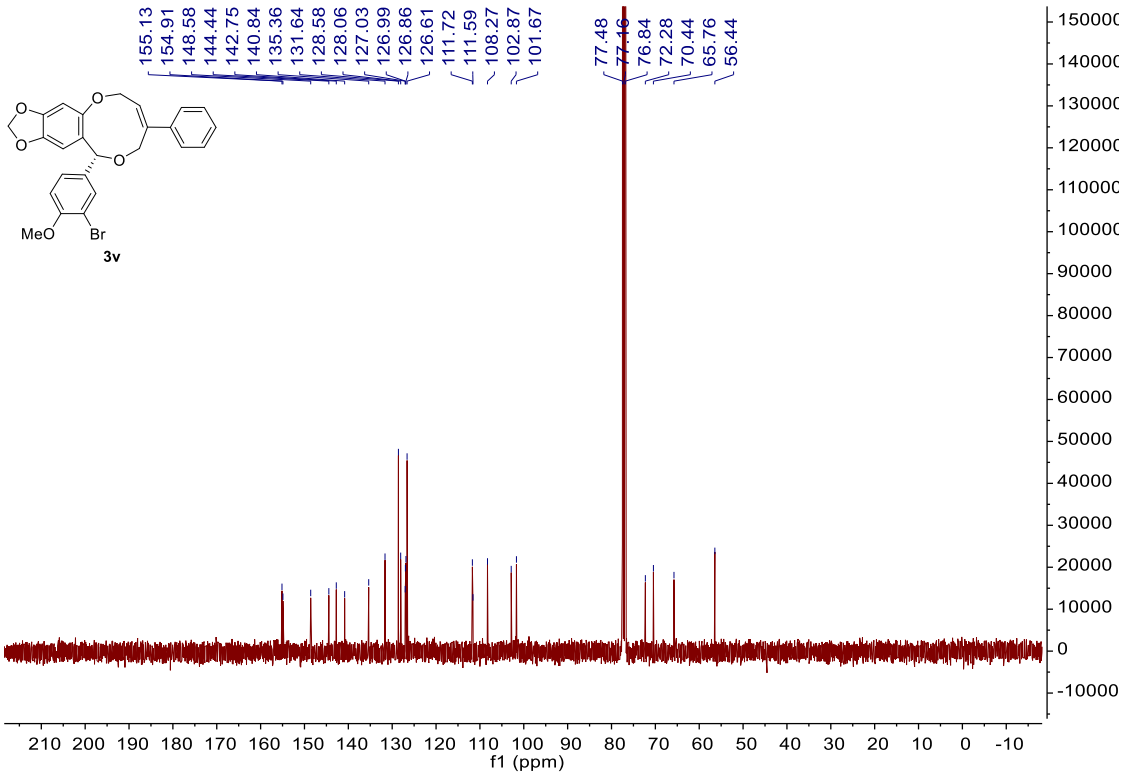
1.00
1.00
2.00
1.00
1.00
2.00
1.00
3.00
f1 (ppm)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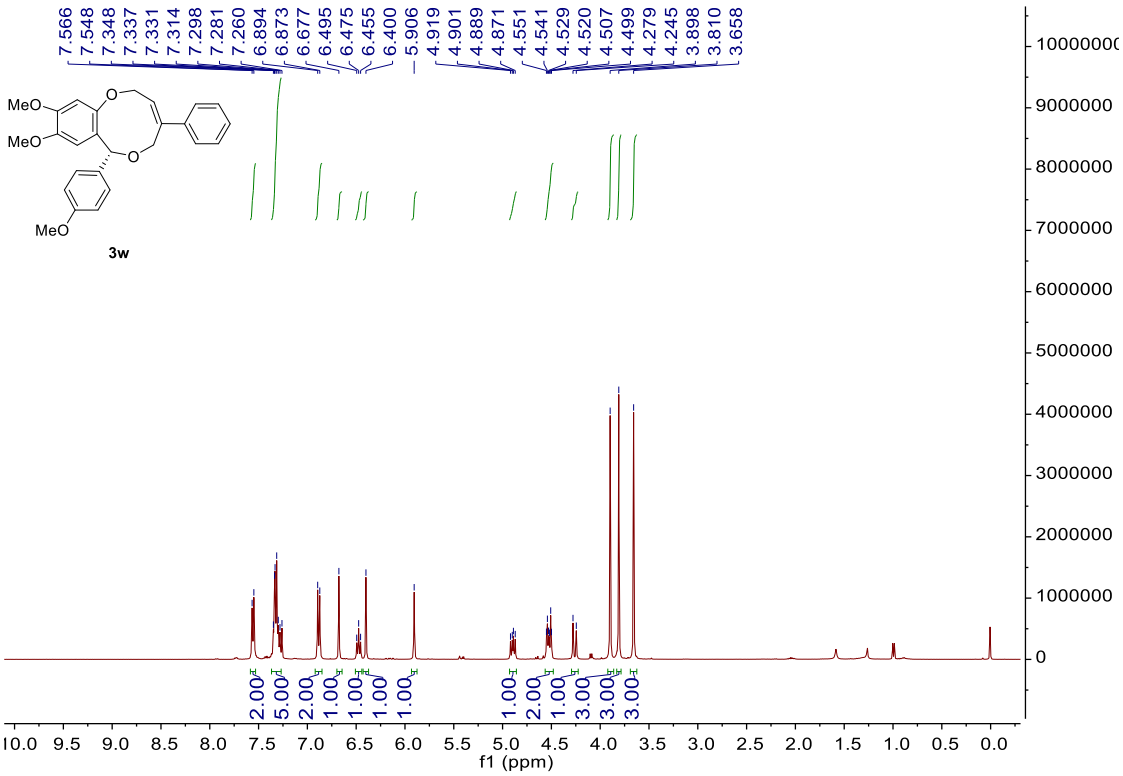
148.58
144.44
142.75
140.84
135.36
131.64
128.58
128.06
127.03
126.99
126.86
126.61
111.72
111.59
108.27
102.87
101.67
77.48
77.16
76.84
72.28
70.44
65.76
56.44
MeO
Br
3v
f1 (ppm)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7.314
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7.281
7.260
6.894
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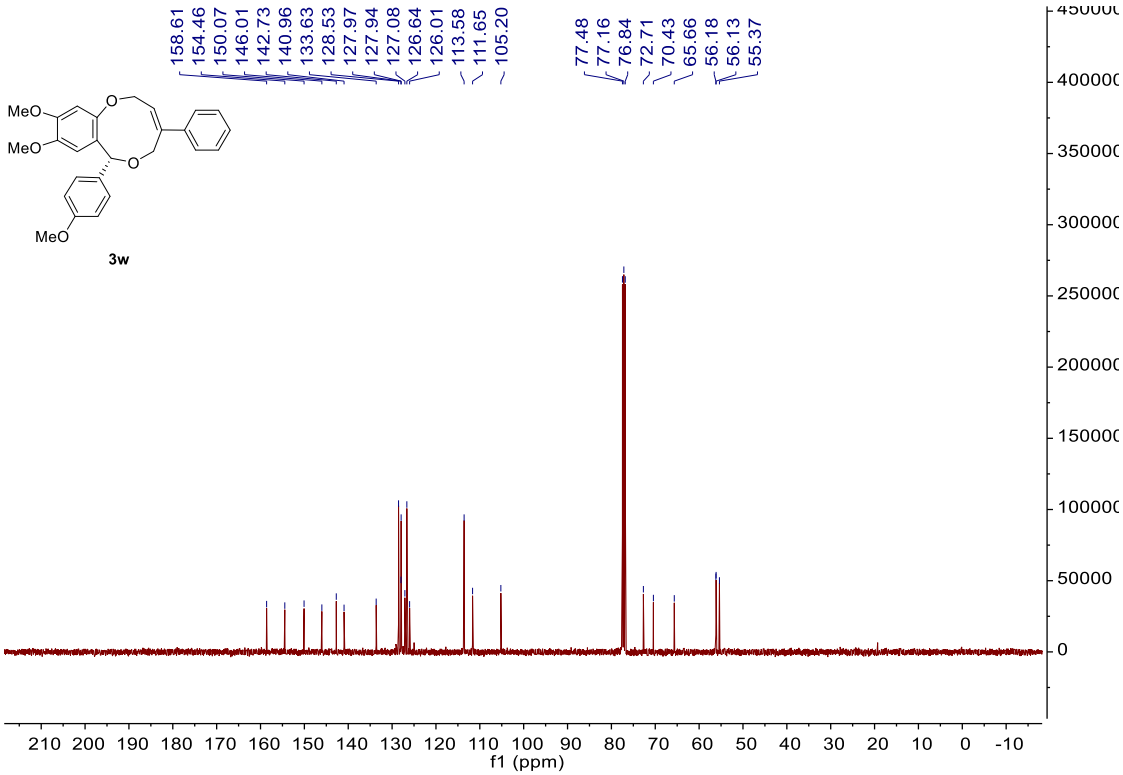
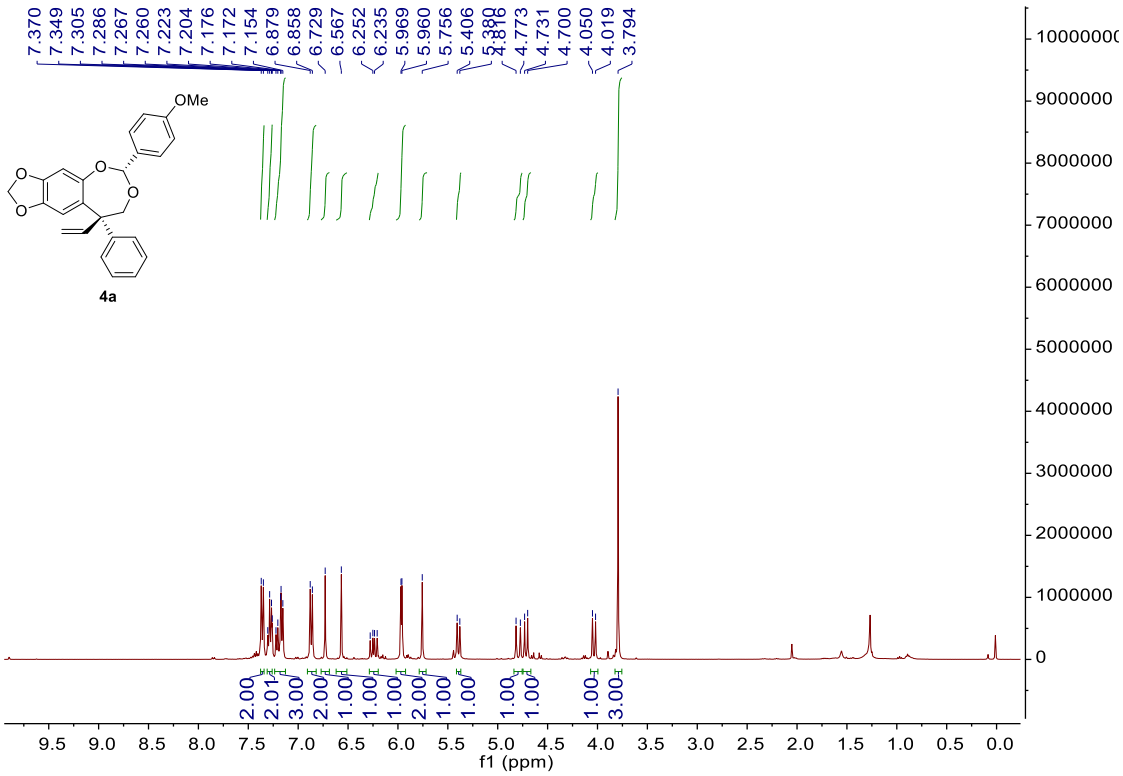
6.677
6.495
6.475
6.455
6.400
5.906
4.919
4.901
4.889
4.871
4.551
4.541
4.529
4.520
4.507
4.499
4.279
4.245
3.898
3.810
3.658
MeO
MeO
MeO
3w
2.00
5.00
2.00
1.00
1.00
1.00
1.00
1.00
2.00
1.00
3.00
3.00
3.00
f1 (ppm)


158.61
154.46
150.07
146.01
142.73
140.96
133.63
128.53
127.97
127.94
127.08
126.64
126.01
113.58
111.65
105.20
77.48
77.16
76.84
72.71
70.43
65.66
56.18
56.13
55.37
MeO
MeO
MeO
3w
f1 (ppm)


7.370
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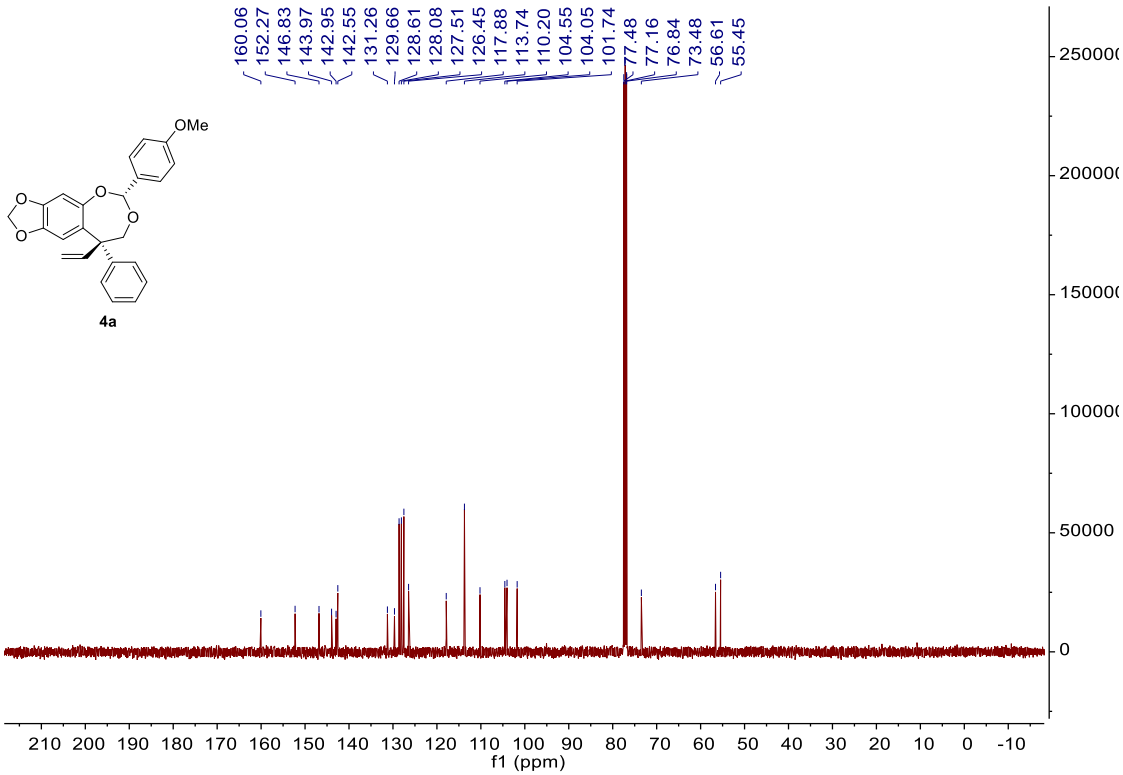
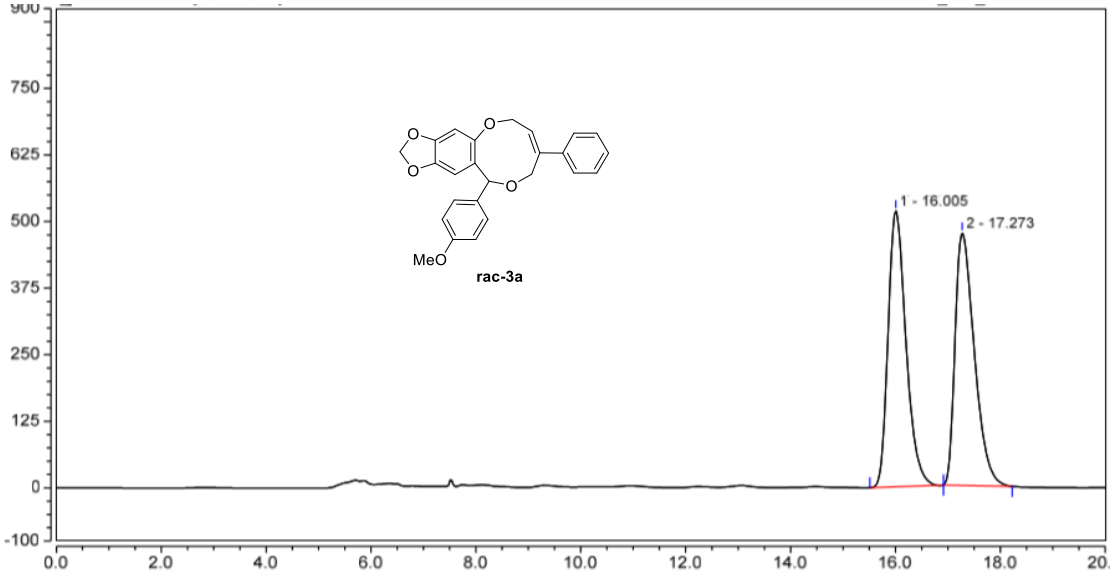
7.267
7.260
7.223
7.204
7.176
7.172
7.154
6.879
6.858
6.729
6.567
6.252
6.235
5.969
5.960
5.756
5.406
5.380
5.318
4.773
4.731
4.700
4.050
4.019
3.794
OMe
4a
2.00
2.01
3.00
2.00
1.00
1.00
1.00
2.00
1.00
1.00
1.00
1.00
3.00
f1 (ppm)

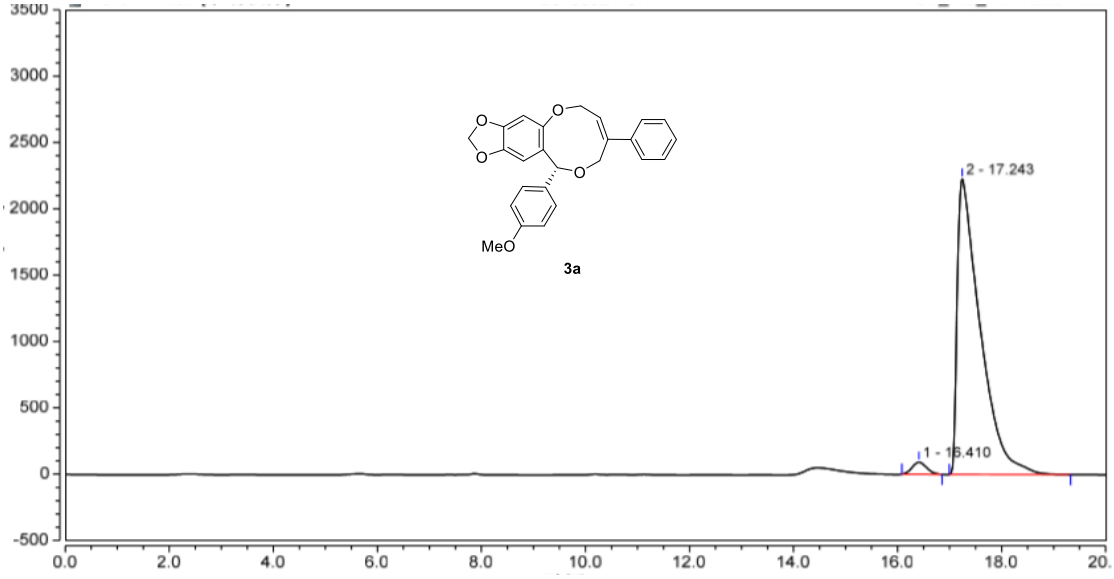

160.06
152.27
146.83
143.97
142.95
142.55
131.26
129.66
128.61
128.08
127.51
126.45
117.88
113.74
110.20
104.55
104.05
101.74
77.48
77.16
76.84
73.48
56.61
55.45
OMe
4a
f1 (ppm)

## 12. Copies of HPLC spectra for racemic and chiral products



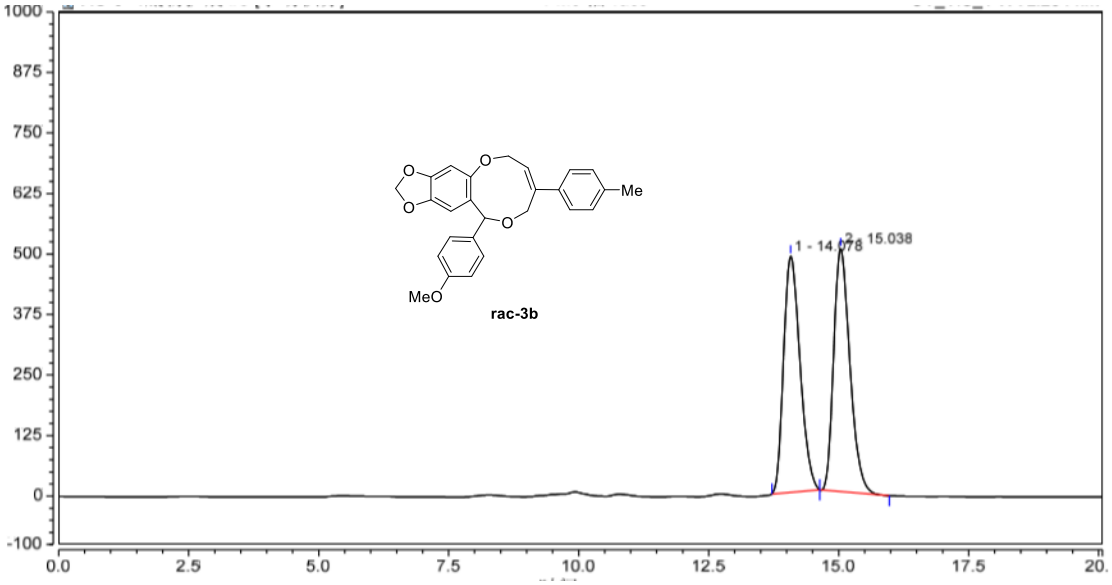

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 16.005 | 204.628 | 518.396 | 50.36 | 52.22 |
| 2 | 17.273 | 201.716 | 474.264 | 49.64 | 47.78 |
| **Total:** | | **406.344** | **992.660** | **100.00** | **100.00** |



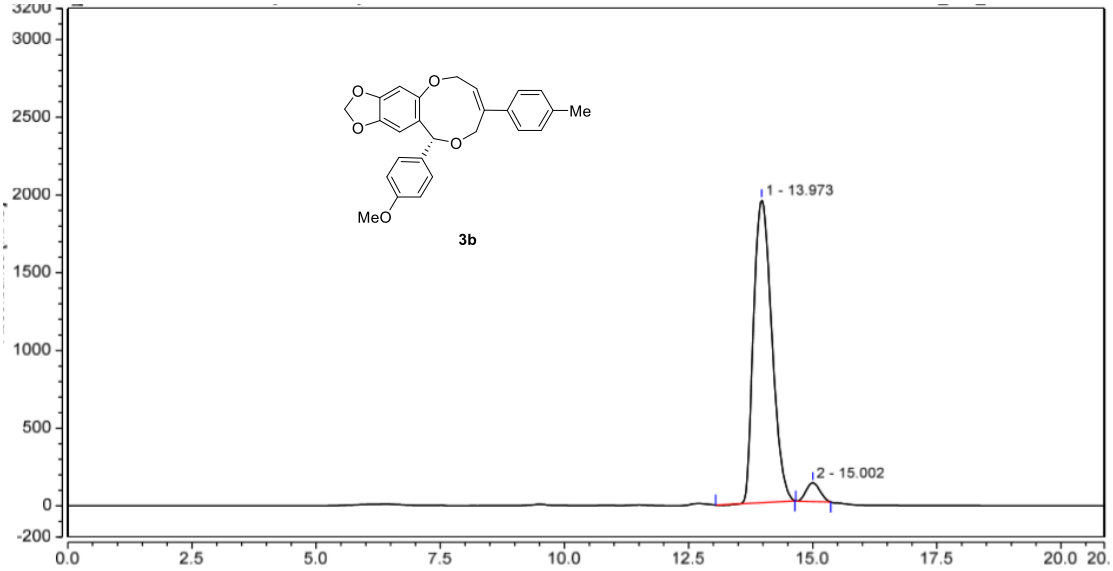

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 16.410 | 29.440 | 90.951 | 2.53 | 3.92 |
| 2 | 17.243 | 1133.114 | 2226.541 | 97.47 | 96.08 |
| **Total:** | | **1162.554** | **2317.492** | **100.00** | **100.00** |



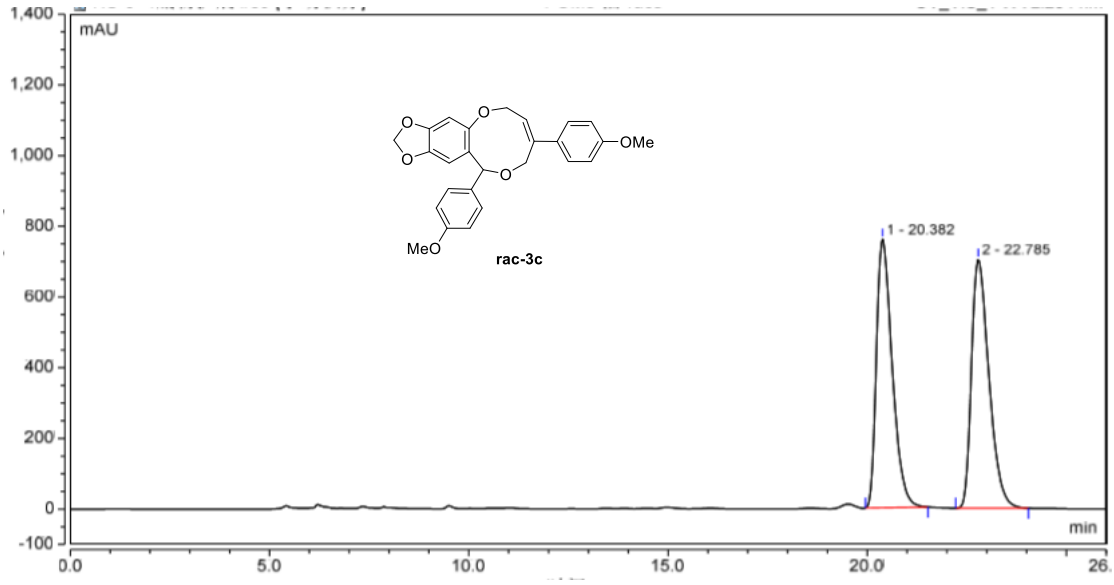

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.078 | 178.660 | 488.425 | 49.59 | 49.29 |
| 2 | 15.038 | 181.637 | 502.410 | 50.41 | 50.71 |
| **Total:** | | **360.297** | **990.834** | **100.00** | **100.00** |



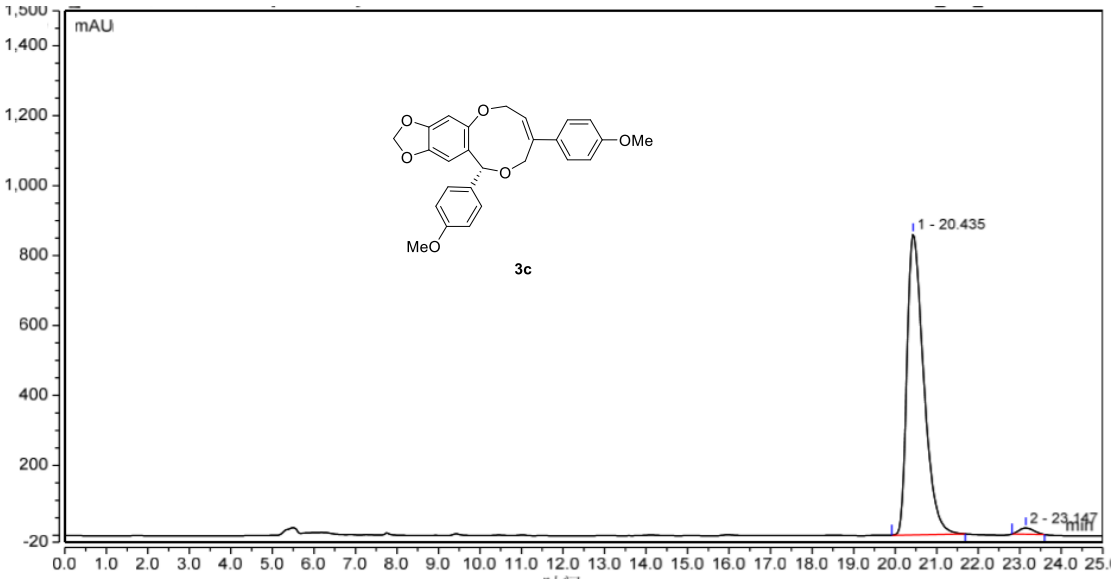

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.973 | 828.947 | 1946.949 | 95.55 | 94.15 |
| 2 | 15.002 | 38.637 | 120.936 | 4.45 | 5.85 |
| **Total:** | | **867.584** | **2067.884** | **100.00** | **100.00** |



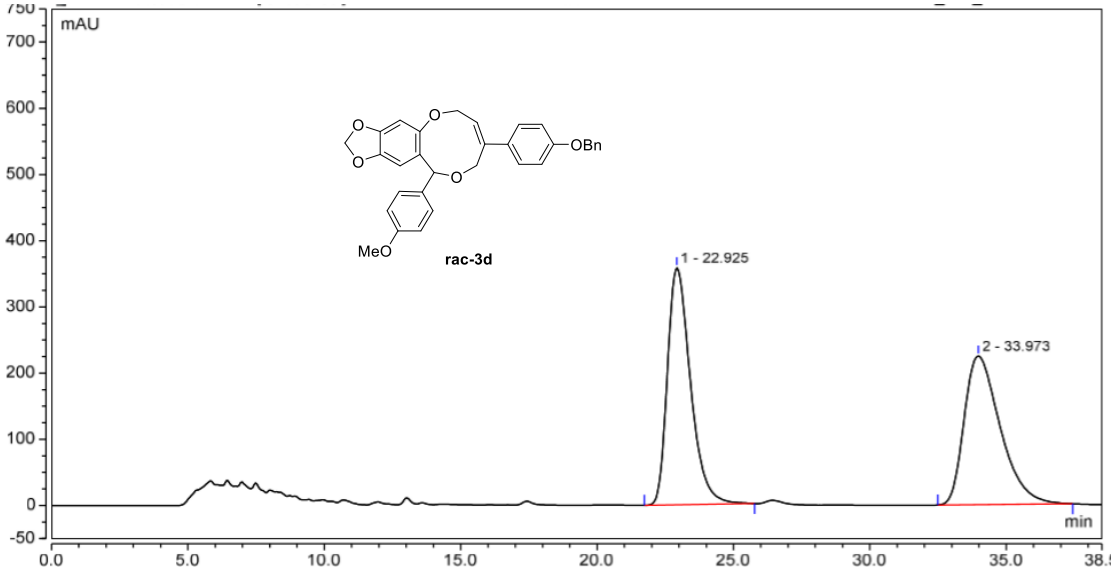

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 20.382 | 349.850 | 759.673 | 49.38 | 51.89 |
| 2 | 22.785 | 358.580 | 704.221 | 50.62 | 48.11 |
| **Total:** | | **708.430** | **1463.895** | **100.00** | **100.00** |

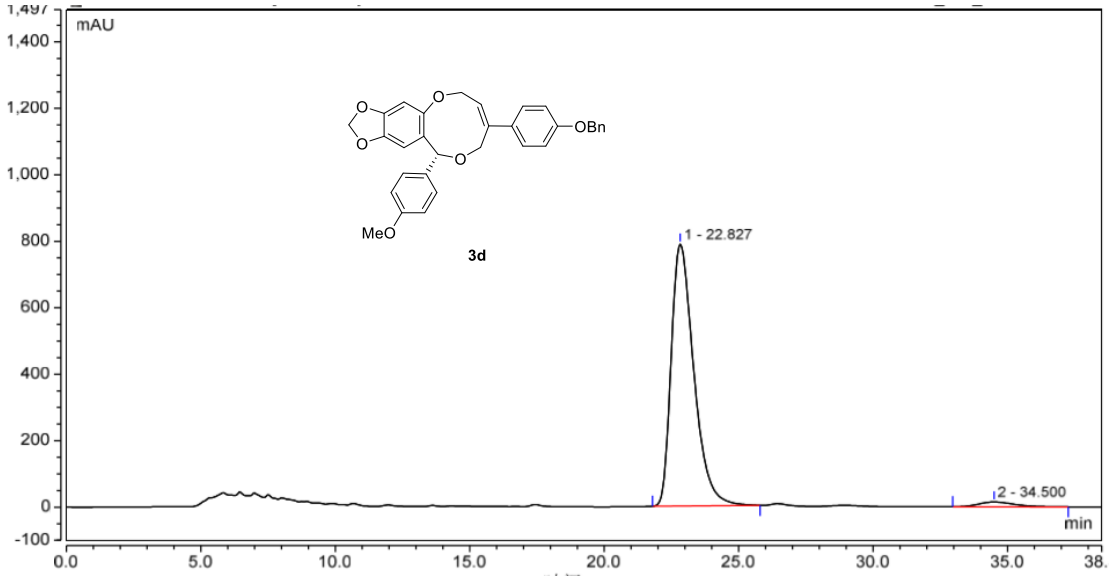



积分结果

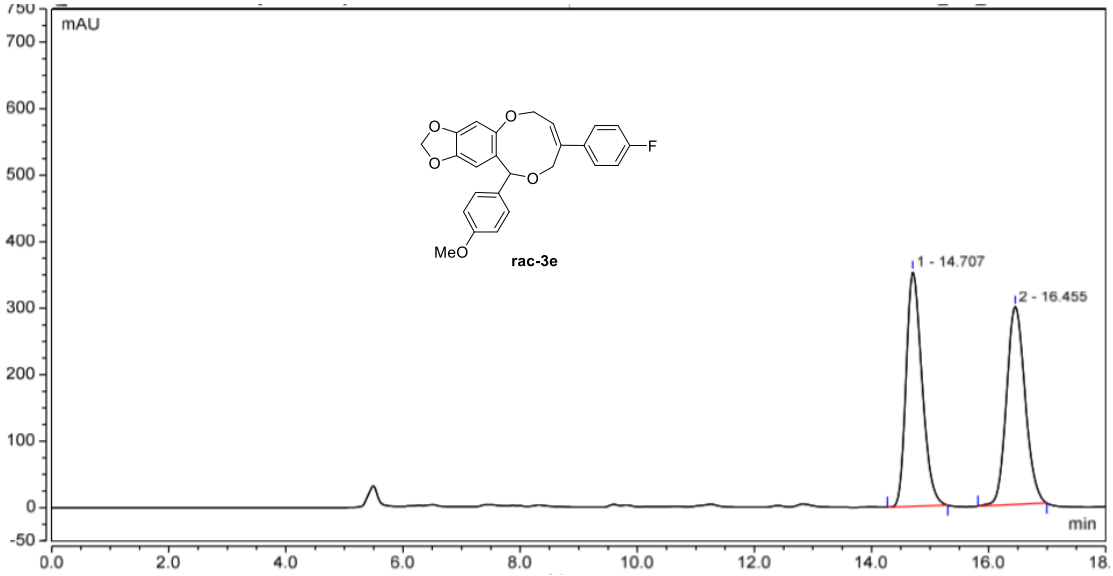
| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 20.435 | 400.790 | 860.441 | 98.19 | 97.96 |
| 2 | 23.147 | 7.392 | 17.916 | 1.81 | 2.04 |
| **Total:** | | **408.182** | **878.356** | **100.00** | **100.00** |




| 积分结果 | | | | | |
|---|---|---|---|---|---|
| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
| 1 | 22.925 | 351.052 | 358.107 | 50.49 | 61.46 |
| 2 | 33.973 | 344.218 | 224.534 | 49.51 | 38.54 |
| **Total:** | | **695.271** | **582.641** | **100.00** | **100.00** |

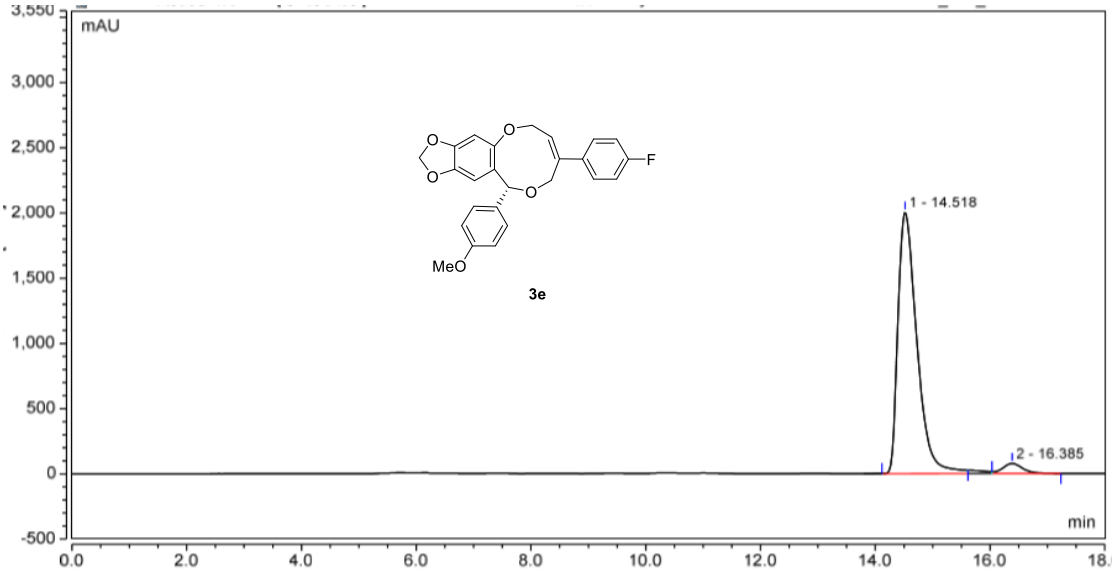



| 积分结果 | | | | | |
|---|---|---|---|---|---|
| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
| 1 | 22.827 | 769.259 | 788.218 | 97.19 | 98.18 |
| 2 | 34.500 | 22.275 | 14.571 | 2.81 | 1.82 |
| **Total:** | | **791.534** | **802.790** | **100.00** | **100.00** |



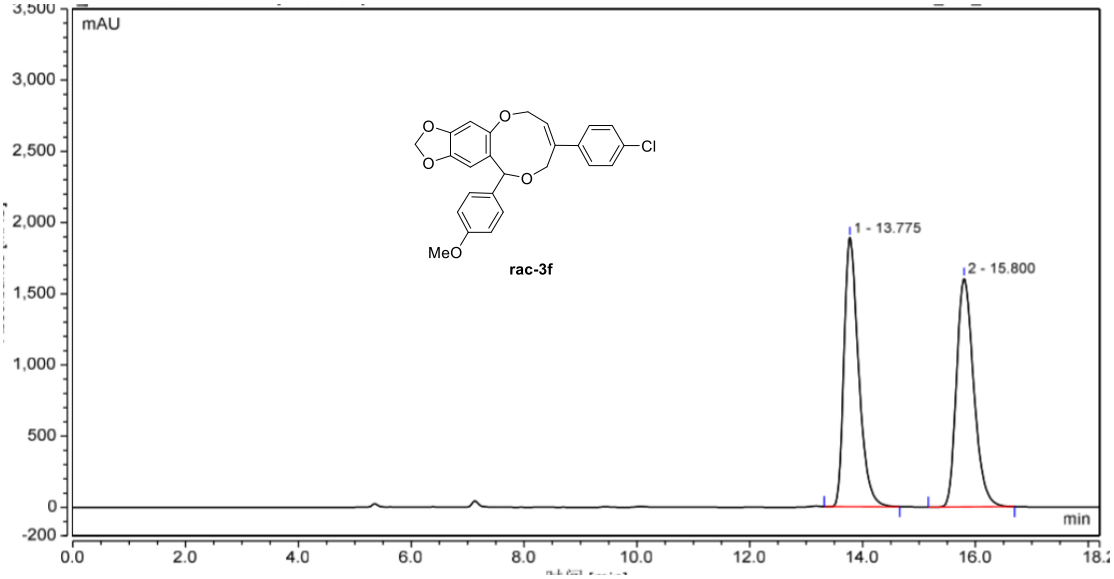

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.707 | 111.157 | 353.367 | 50.34 | 54.26 |
| 2 | 16.455 | 109.644 | 297.835 | 49.66 | 45.74 |
| **Total:** | | **220.801** | **651.202** | **100.00** | **100.00** |



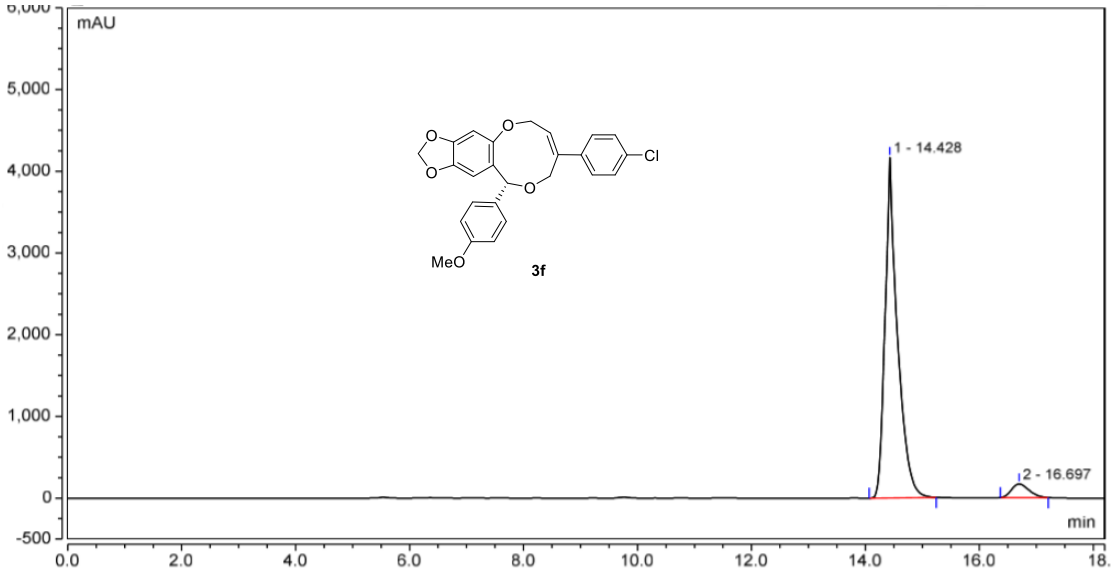

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.518 | 765.823 | 2005.472 | 96.08 | 96.30 |
| 2 | 16.385 | 31.273 | 77.004 | 3.92 | 3.70 |
| **Total:** | | **797.097** | **2082.476** | **100.00** | **100.00** |



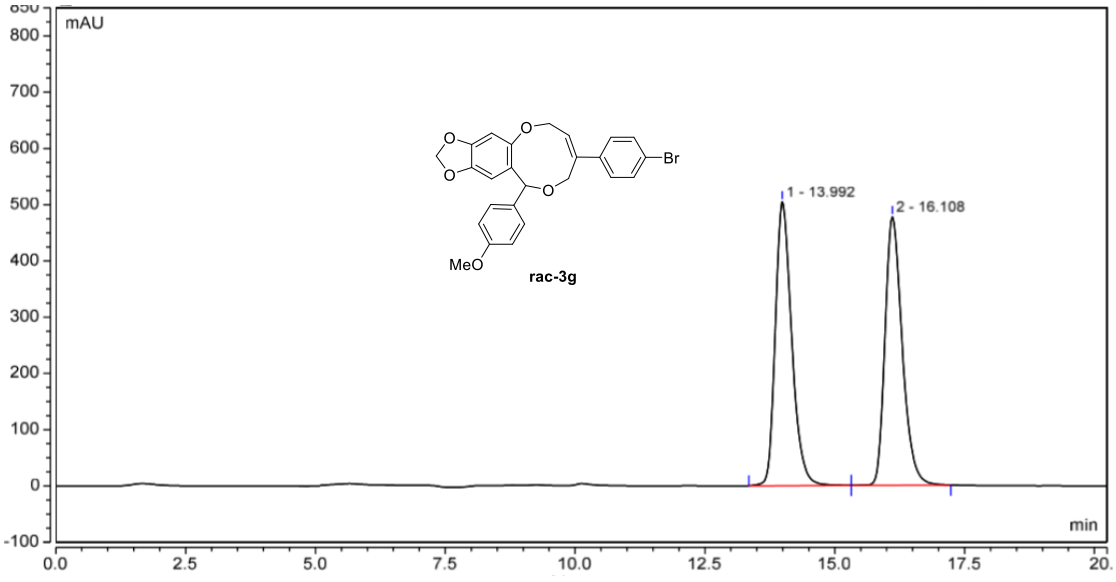

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.775 | 564.911 | 1890.454 | 49.85 | 54.05 |
| 2 | 15.800 | 568.245 | 1607.126 | 50.15 | 45.95 |
| **Total:** | | **1133.156** | **3497.579** | **100.00** | **100.00** |



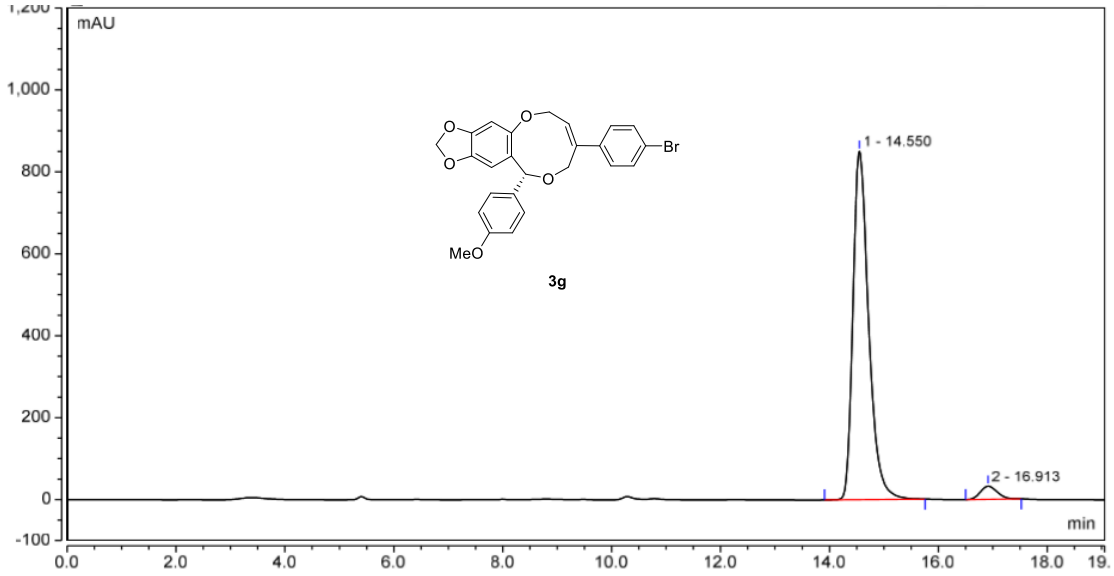

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.428 | 1044.707 | 4164.707 | 94.56 | 96.14 |
| 2 | 16.697 | 60.050 | 167.054 | 5.44 | 3.86 |
| **Total:** | | **1104.758** | **4331.761** | **100.00** | **100.00** |



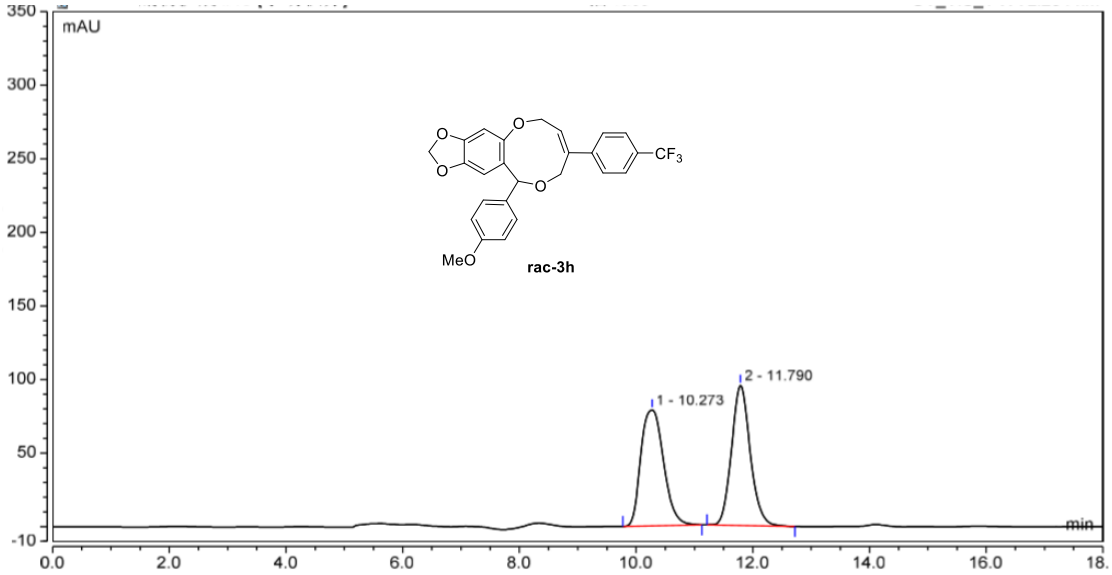

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.992 | 185.195 | 504.688 | 50.16 | 51.38 |
| 2 | 16.108 | 183.991 | 477.584 | 49.84 | 48.62 |
| **Total:** | | **369.186** | **982.271** | **100.00** | **100.00** |



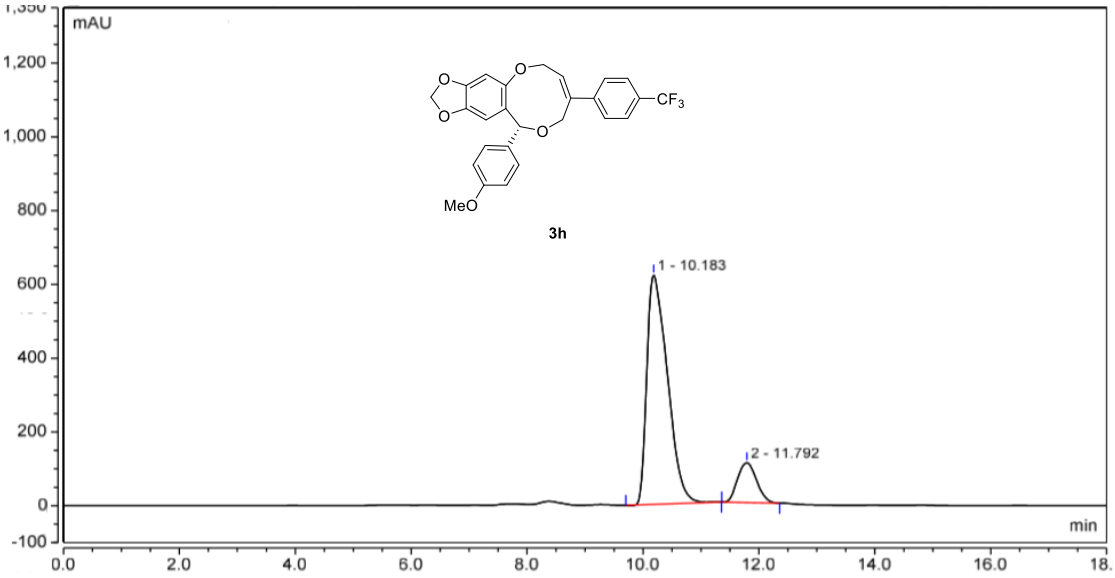

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.550 | 284.546 | 850.755 | 96.07 | 96.37 |
| 2 | 16.913 | 11.651 | 32.013 | 3.93 | 3.63 |
| **Total:** | | **296.197** | **882.768** | **100.00** | **100.00** |



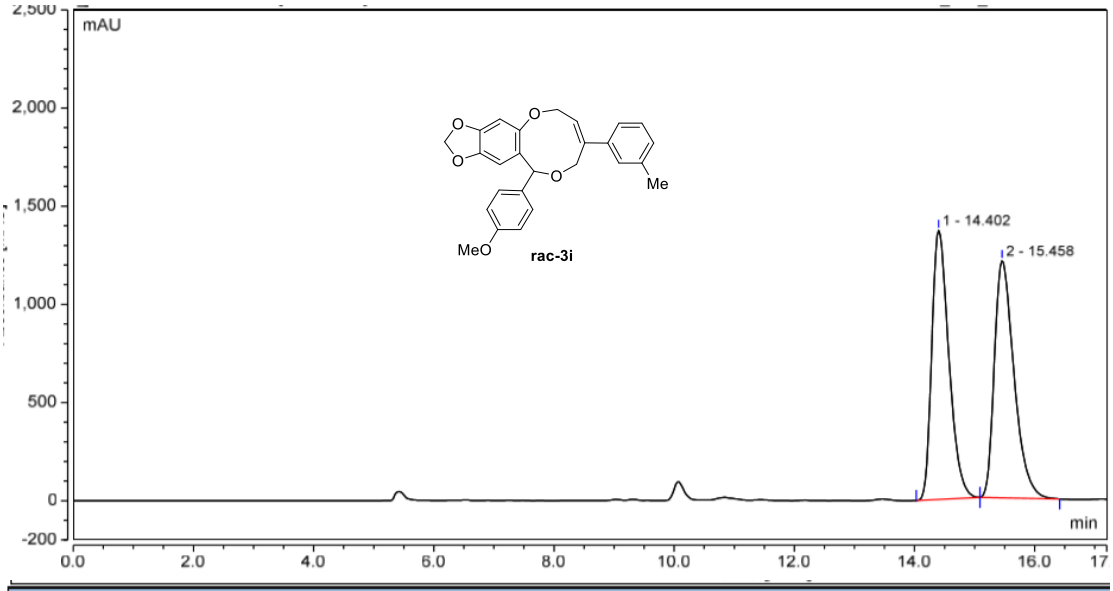

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 10.273 | 35.560 | 78.822 | 50.08 | 45.29 |
| 2 | 11.790 | 35.443 | 95.224 | 49.92 | 54.71 |
| **Total:** | | **71.003** | **174.046** | **100.00** | **100.00** |



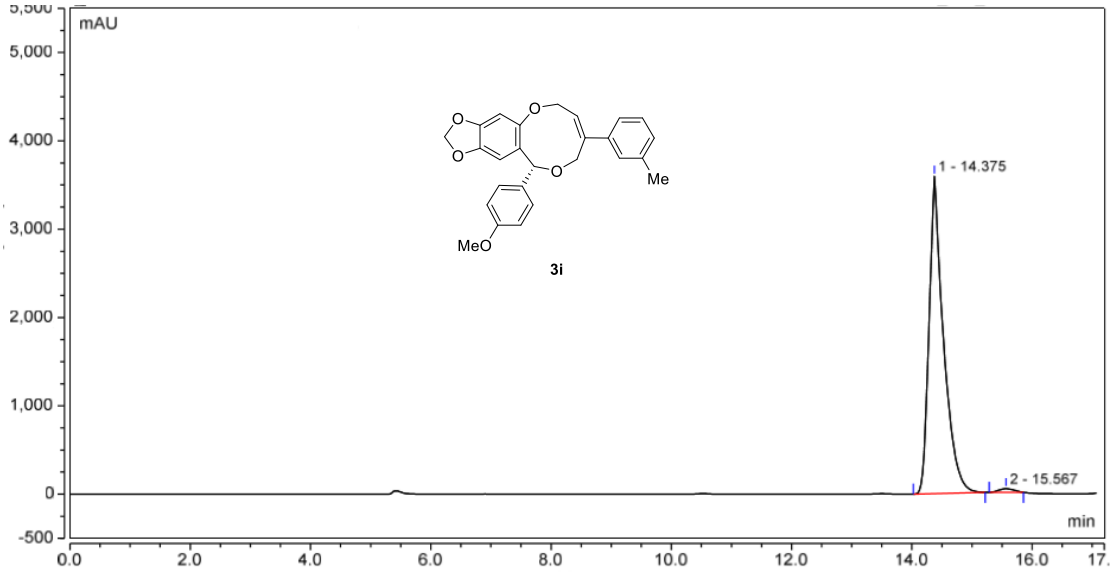

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 10.183 | 256.188 | 621.655 | 86.18 | 85.26 |
| 2 | 11.792 | 41.096 | 107.434 | 13.82 | 14.74 |
| **Total:** | | **297.284** | **729.089** | **100.00** | **100.00** |



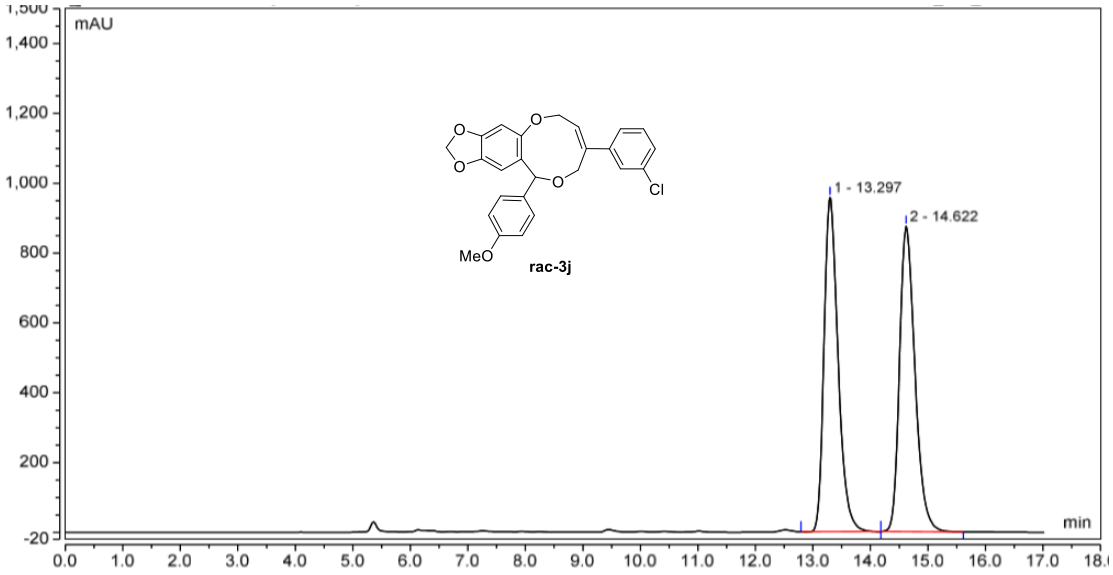

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.402 | 451.544 | 1371.245 | 49.60 | 53.17 |
| 2 | 15.458 | 458.827 | 1207.617 | 50.40 | 46.83 |
| **Total:** | | **910.370** | **2578.862** | **100.00** | **100.00** |



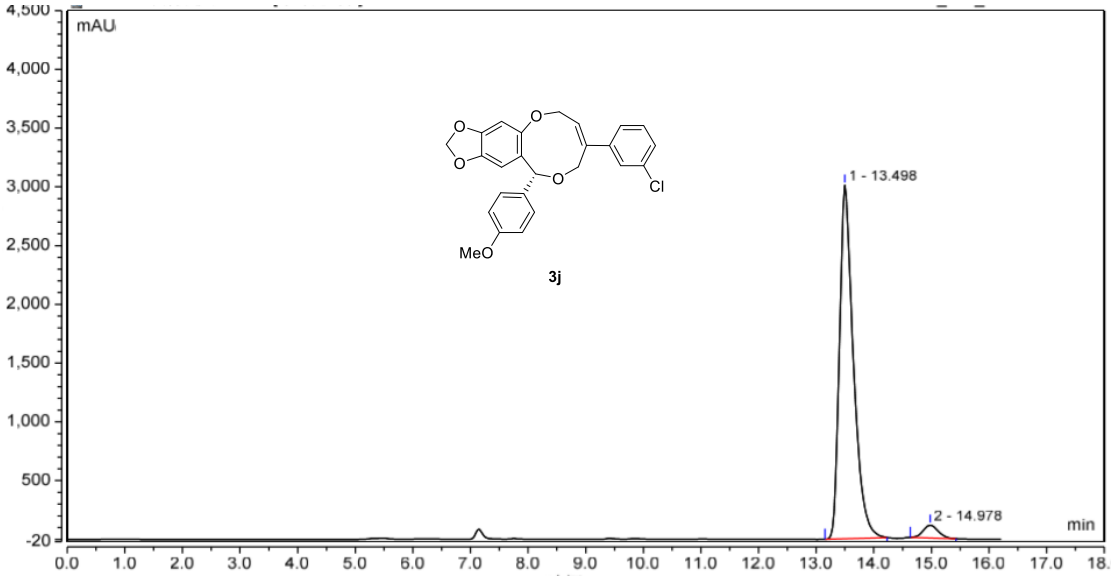

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 14.375 | 995.274 | 3594.609 | 98.78 | 98.87 |
| 2 | 15.567 | 12.311 | 41.092 | 1.22 | 1.13 |
| **Total:** | | **1007.585** | **3635.701** | **100.00** | **100.00** |



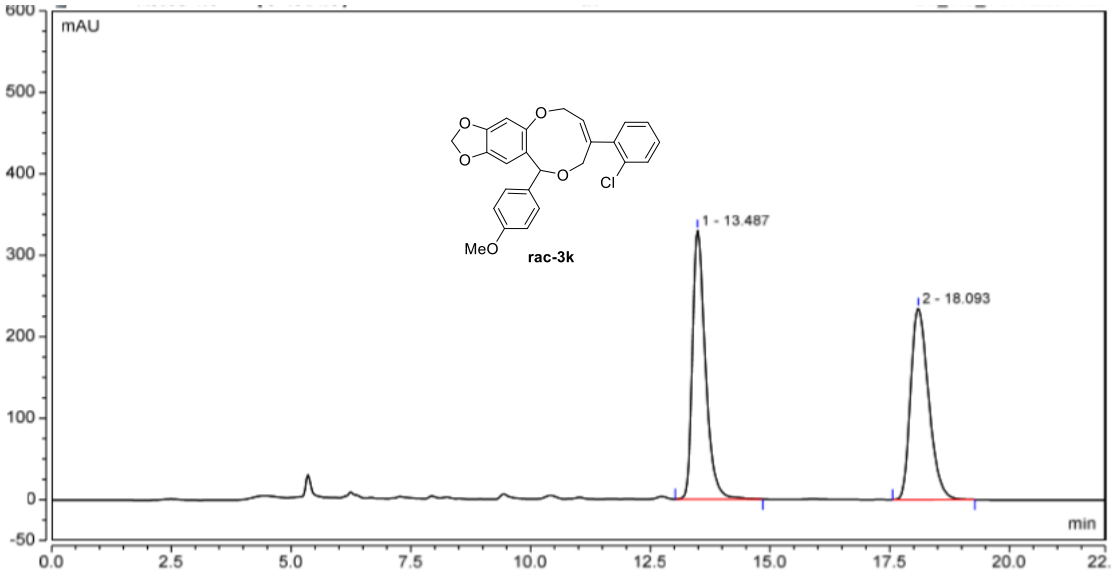

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 13.297 | 270.165 | 957.483 | 49.81 | 52.26 |
| 2 | 14.622 | 272.259 | 874.715 | 50.19 | 47.74 |
| **Total:** | | **542.424** | **1832.198** | **100.00** | **100.00** |



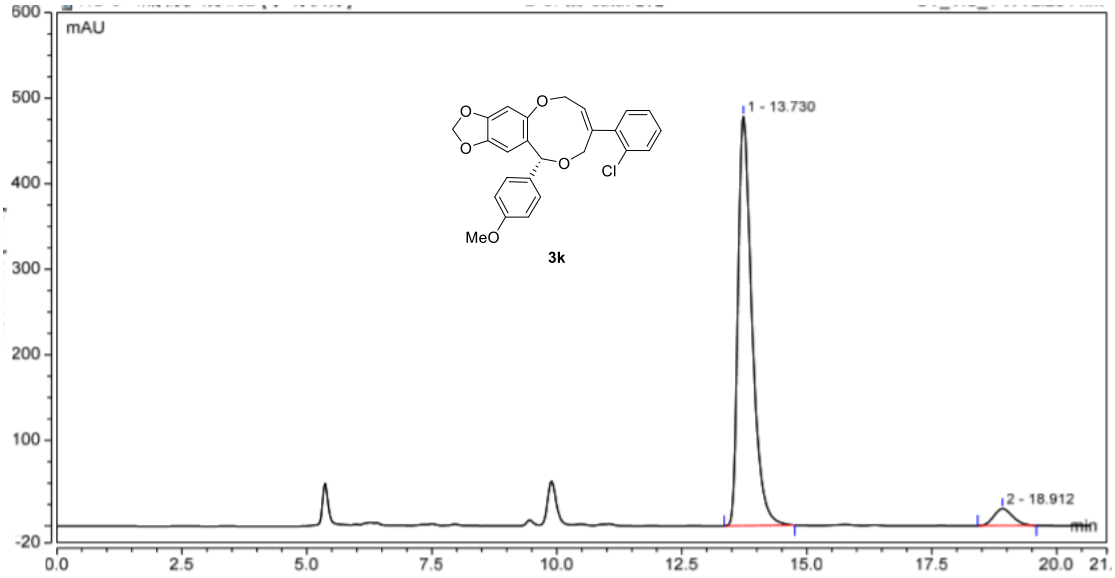

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 13.498 | 840.390 | 3008.749 | 96.24 | 96.55 |
| 2 | 14.978 | 32.790 | 107.593 | 3.76 | 3.45 |
| **Total:** | | **873.180** | **3116.342** | **100.00** | **100.00** |



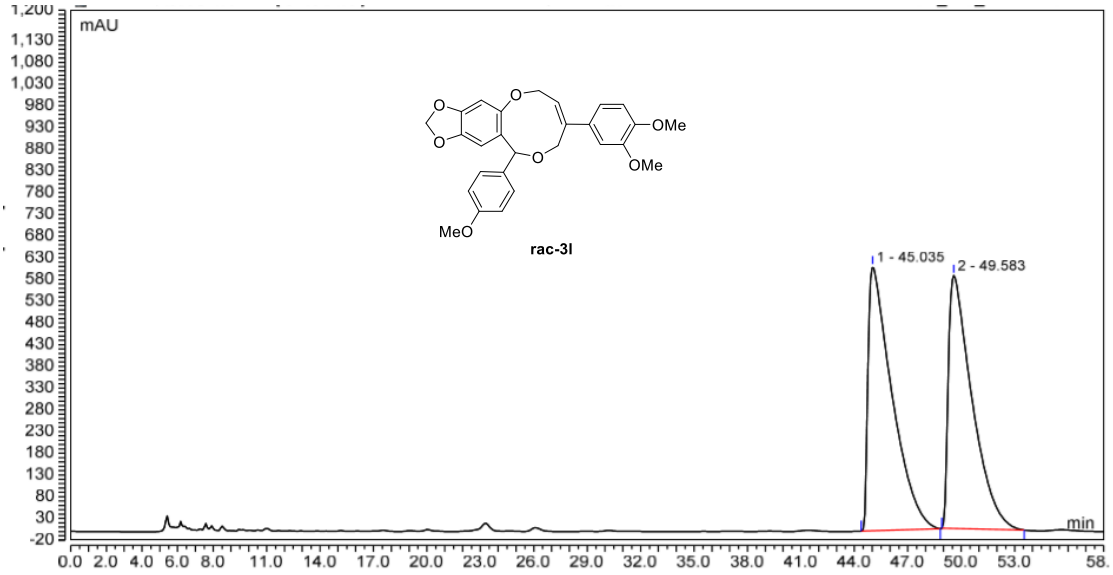

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.487 | 103.987 | 330.274 | 50.31 | 58.44 |
| 2 | 18.093 | 102.714 | 234.850 | 49.69 | 41.56 |
| **Total:** | | **206.701** | **565.123** | **100.00** | **100.00** |



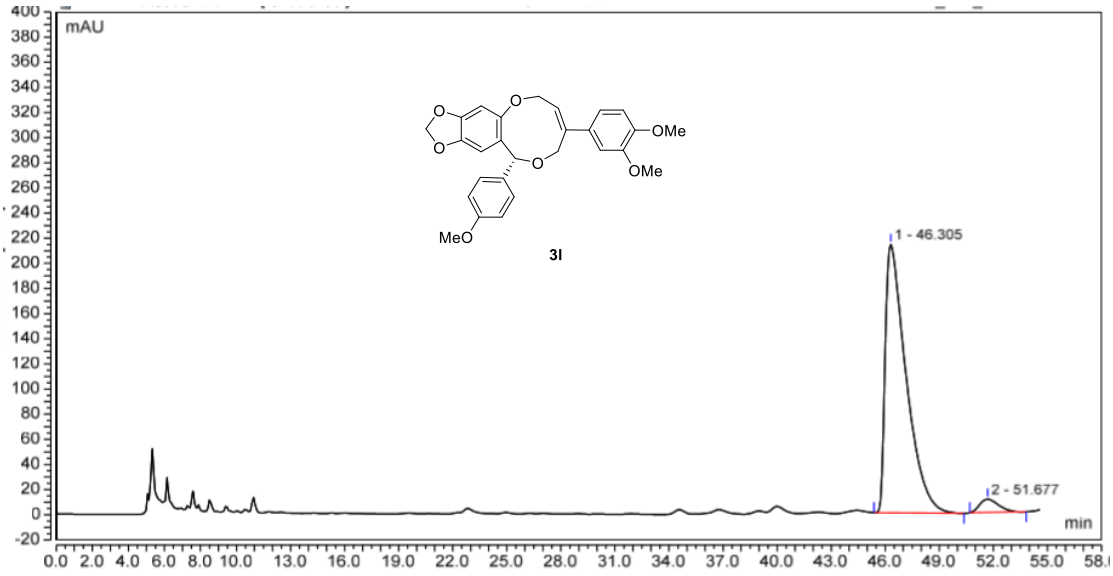

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.730 | 154.974 | 478.644 | 94.66 | 96.03 |
| 2 | 18.912 | 8.746 | 19.798 | 5.34 | 3.97 |
| **Total:** | | **163.720** | **498.442** | **100.00** | **100.00** |



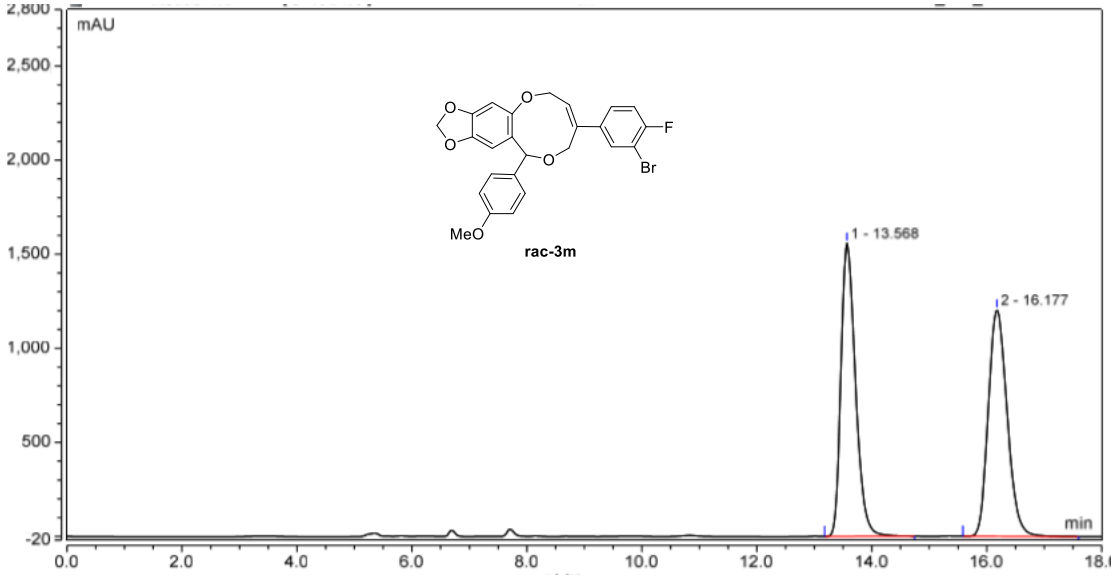

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 45.035 | 945.478 | 608.014 | 51.34 | 51.06 |
| 2 | 49.583 | 896.138 | 582.701 | 48.66 | 48.94 |
| **Total:** | | **1841.616** | **1190.715** | **100.00** | **100.00** |



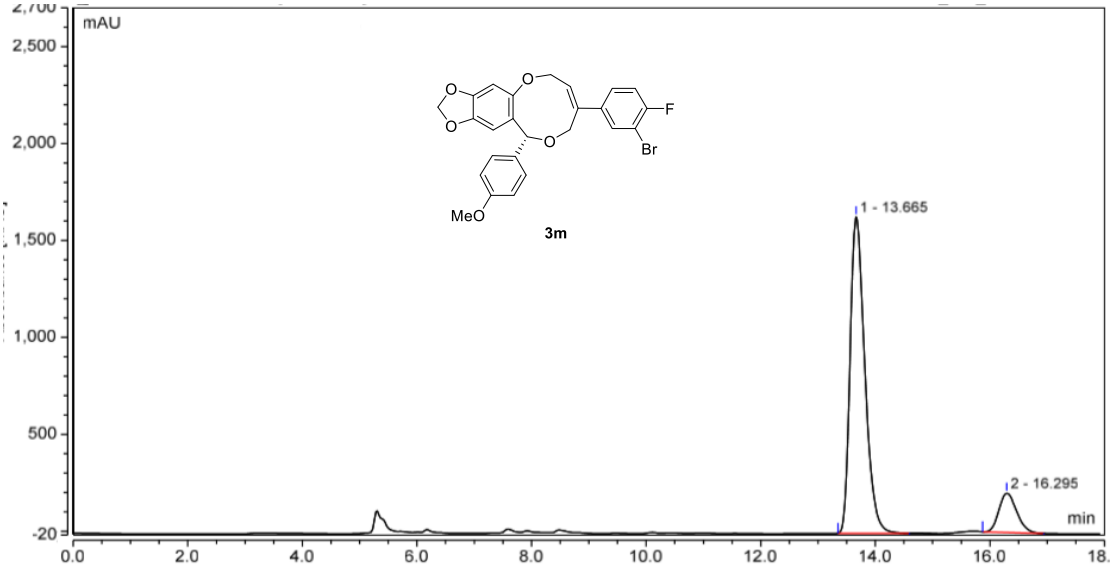

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 46.305 | 285.974 | 213.723 | 95.73 | 95.29 |
| 2 | 51.677 | 12.761 | 10.565 | 4.27 | 4.71 |
| **Total:** | | **298.735** | **224.288** | **100.00** | **100.00** |



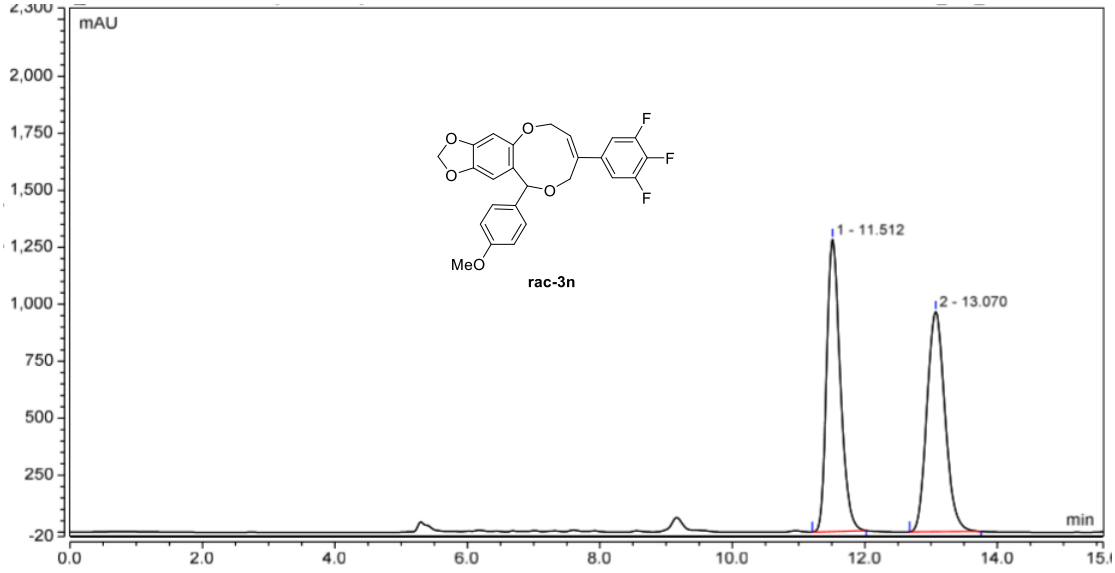

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.568 | 457.299 | 1557.787 | 49.89 | 56.42 |
| 2 | 16.177 | 459.240 | 1203.286 | 50.11 | 43.58 |
| **Total:** | | **916.538** | **2761.072** | **100.00** | **100.00** |



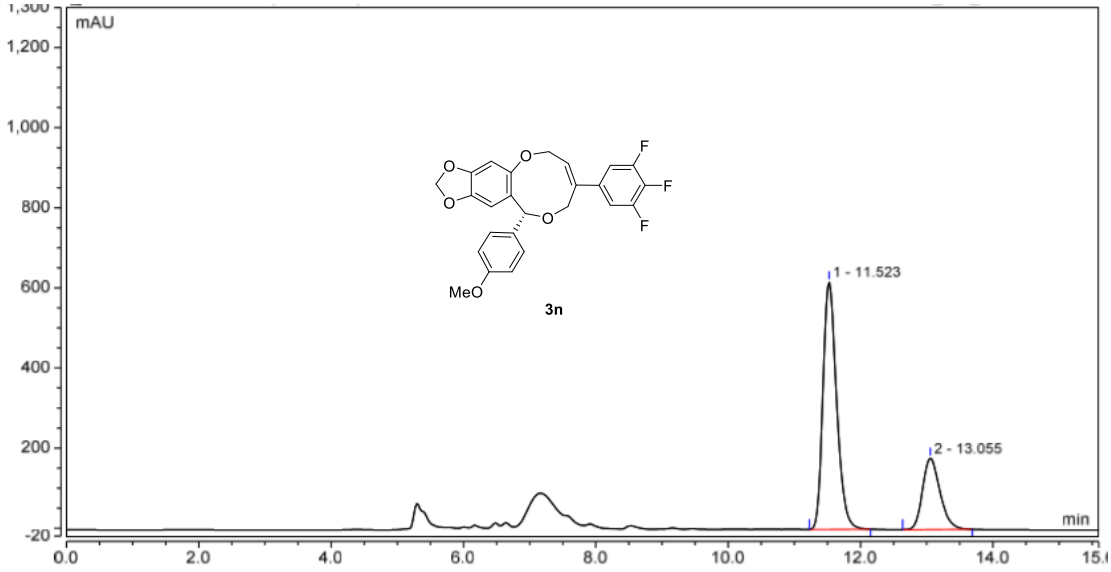

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.665 | 486.127 | 1632.714 | 87.02 | 88.98 |
| 2 | 16.295 | 72.514 | 202.222 | 12.98 | 11.02 |
| **Total:** | | **558.641** | **1834.936** | **100.00** | **100.00** |



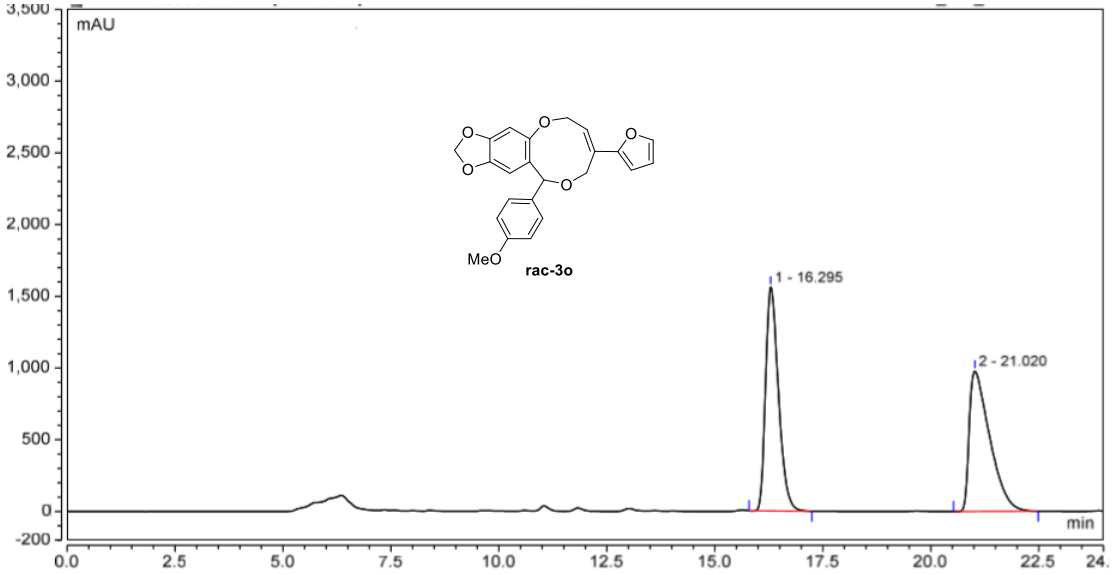

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 11.512 | 305.093 | 1281.231 | 49.93 | 57.00 |
| 2 | 13.070 | 305.930 | 966.477 | 50.07 | 43.00 |
| **Total:** | | **611.023** | **2247.707** | **100.00** | **100.00** |



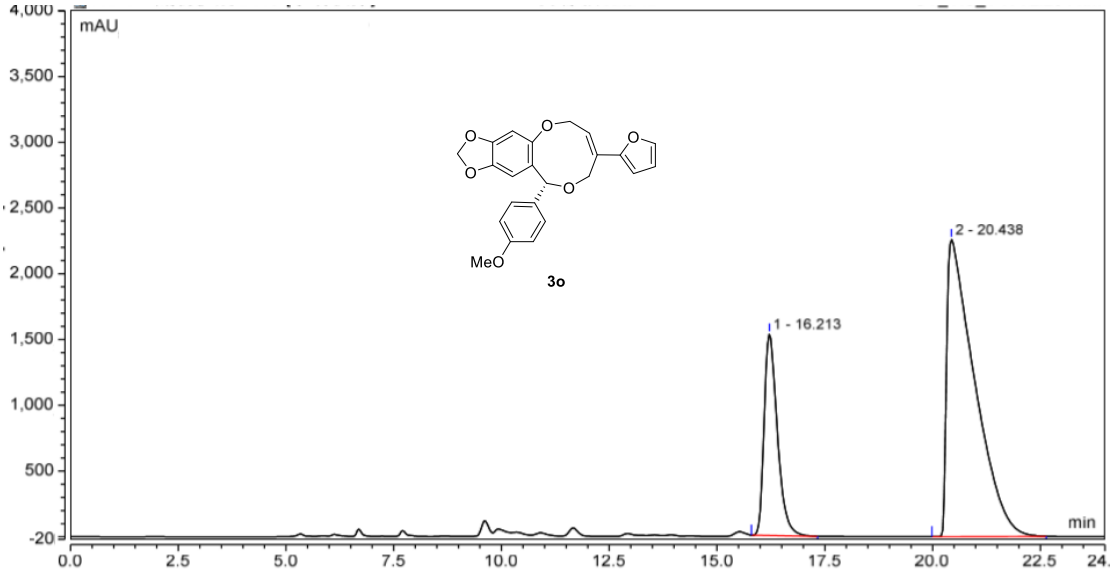

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 11.523 | 147.775 | 618.532 | 72.97 | 77.65 |
| 2 | 13.055 | 54.733 | 177.985 | 27.03 | 22.35 |
| **Total:** | | **202.509** | **796.517** | **100.00** | **100.00** |



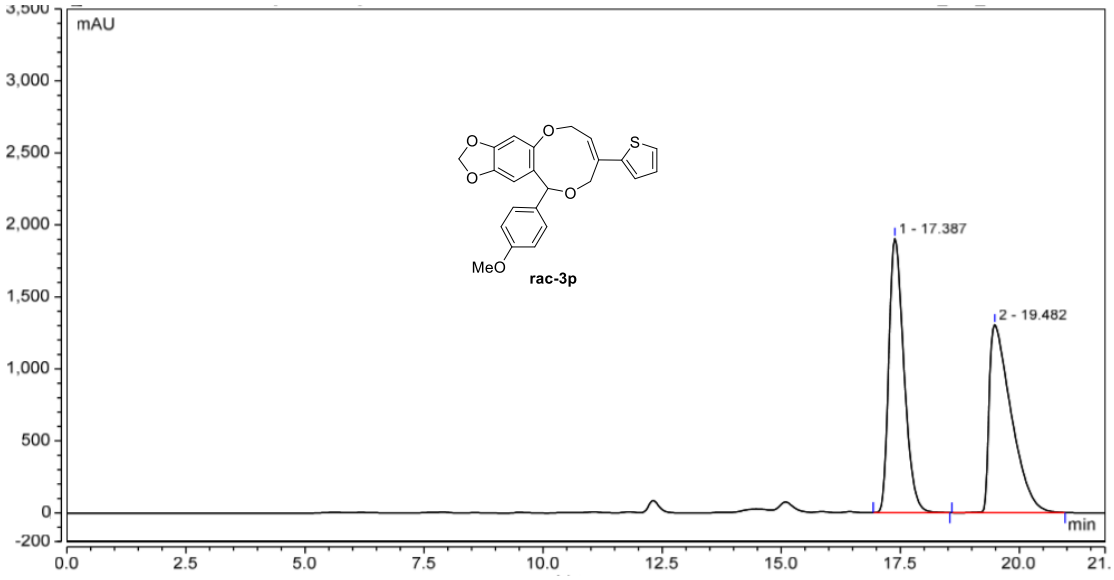

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 16.295 | 538.189 | 1560.676 | 49.99 | 61.42 |
| 2 | 21.020 | 538.439 | 980.418 | 50.01 | 38.58 |
| **Total:** | | **1076.628** | **2541.093** | **100.00** | **100.00** |



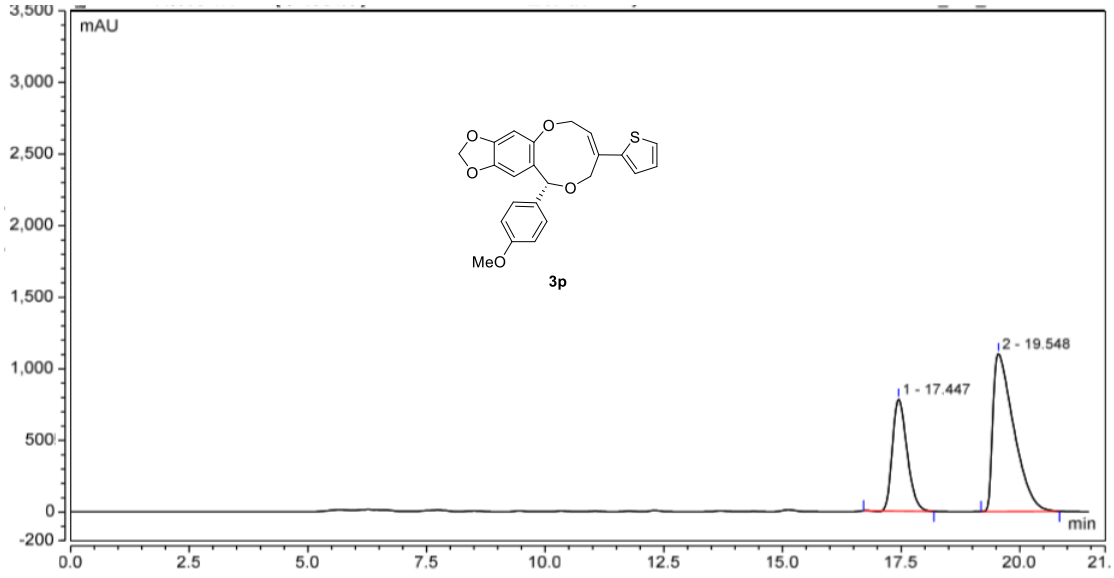

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 16.213 | 545.824 | 1532.145 | 24.41 | 40.41 |
| 2 | 20.438 | 1690.366 | 2259.707 | 75.59 | 59.59 |
| **Total:** | | **2236.190** | **3791.852** | **100.00** | **100.00** |



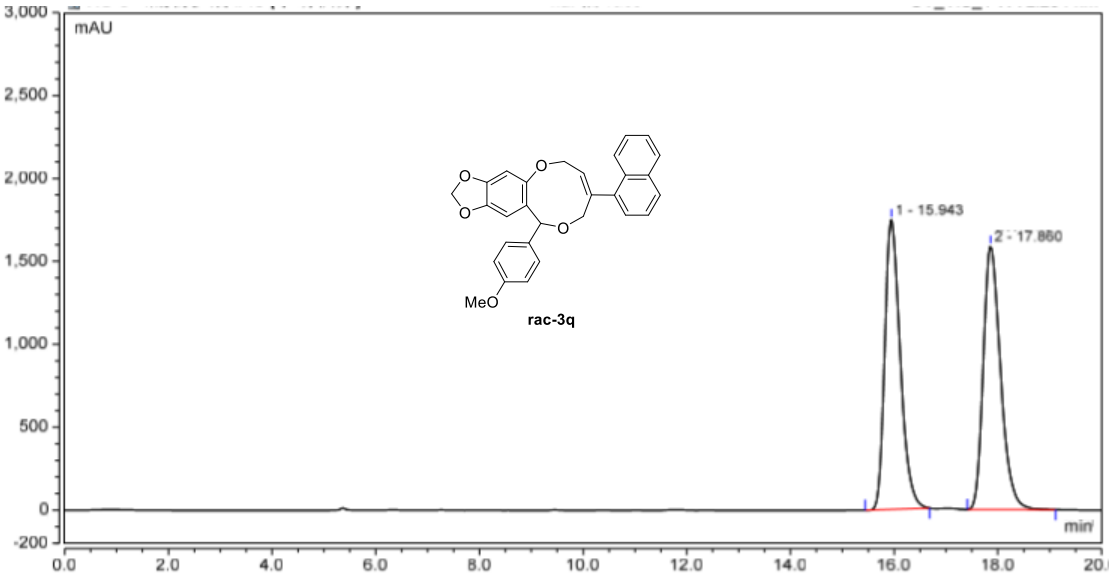

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 17.387 | 700.913 | 1904.940 | 50.04 | 59.30 |
| 2 | 19.482 | 699.715 | 1307.224 | 49.96 | 40.70 |
| **Total:** | | **1400.628** | **3212.164** | **100.00** | **100.00** |



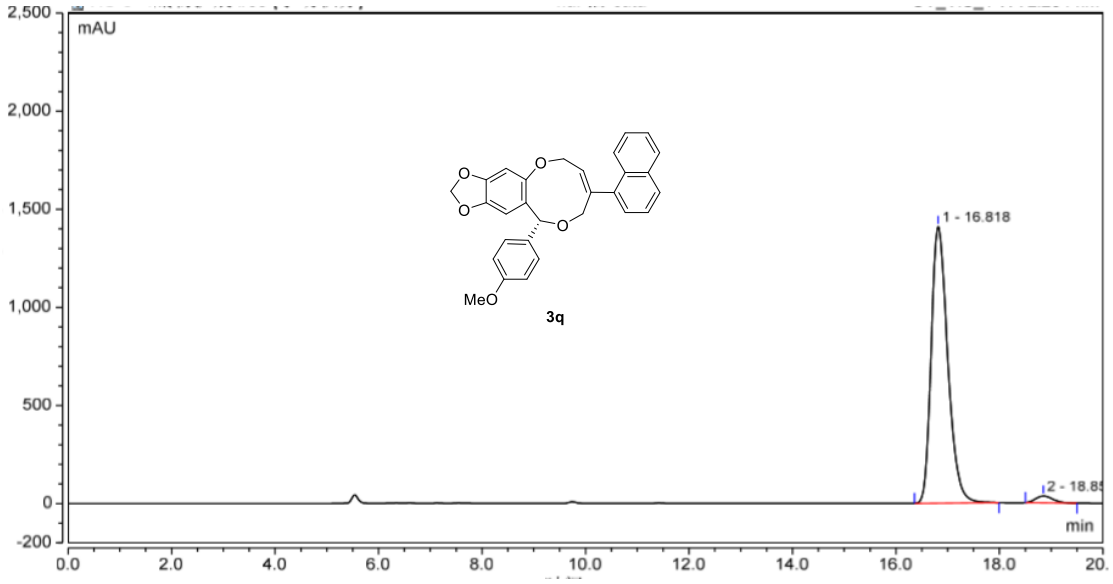

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 17.447 | 280.286 | 781.476 | 33.23 | 41.48 |
| 2 | 19.548 | 563.233 | 1102.302 | 66.77 | 58.52 |
| **Total:** | | **843.518** | **1883.778** | **100.00** | **100.00** |



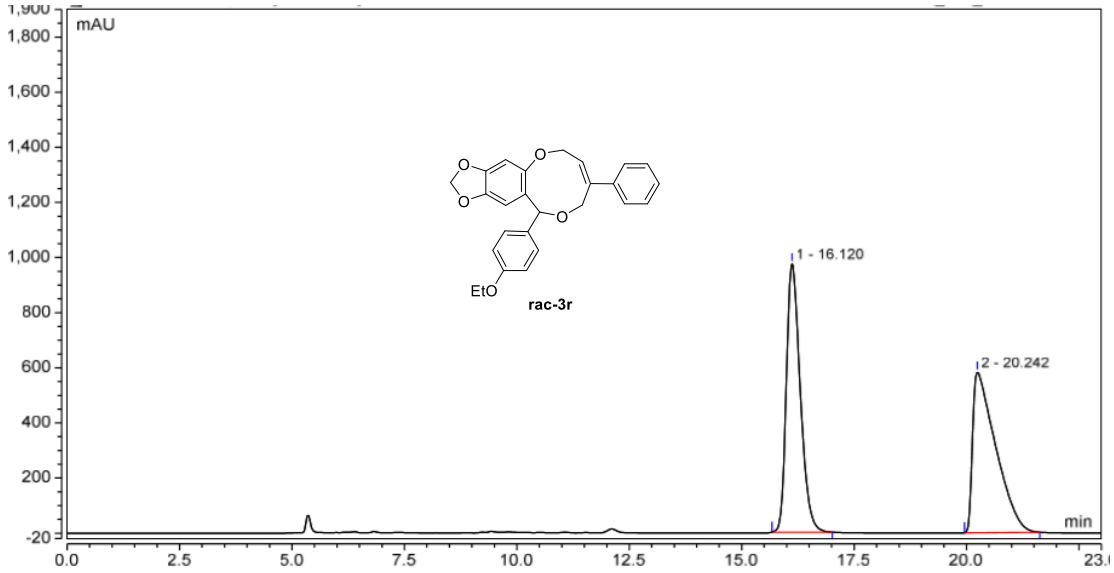

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 15.943 | 604.006 | 1747.246 | 49.57 | 52.38 |
| 2 | 17.860 | 614.556 | 1588.267 | 50.43 | 47.62 |
| **Total:** | | **1218.563** | **3335.513** | **100.00** | **100.00** |



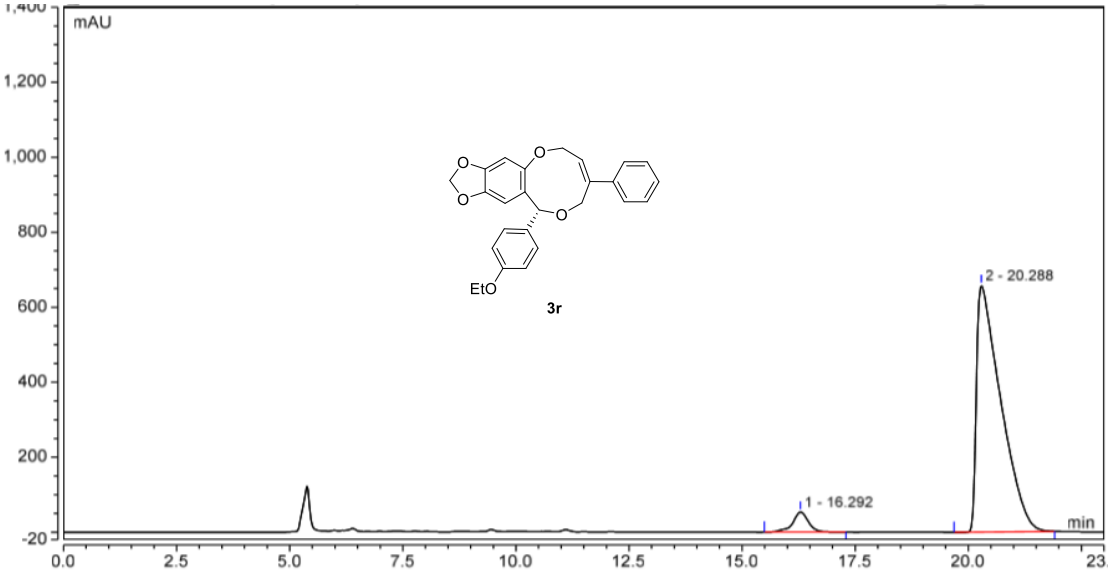

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 16.818 | 522.249 | 1409.789 | 97.49 | 97.59 |
| 2 | 18.850 | 13.465 | 34.864 | 2.51 | 2.41 |
| **Total:** | | **535.714** | **1444.653** | **100.00** | **100.00** |



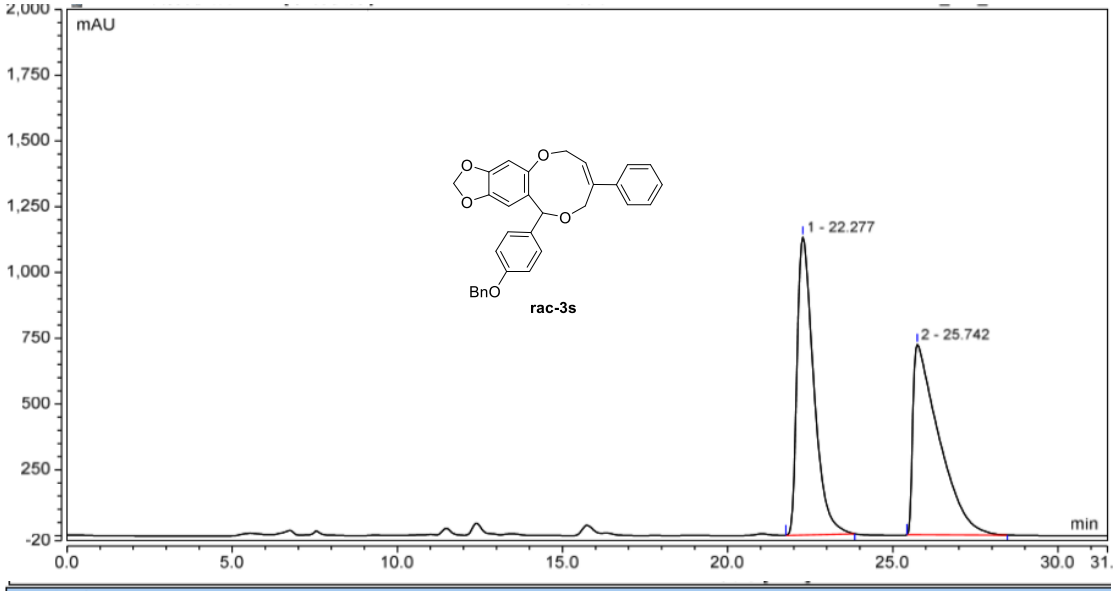

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 16.120 | 349.296 | 973.500 | 50.16 | 62.55 |
| 2 | 20.242 | 347.044 | 582.967 | 49.84 | 37.45 |
| **Total:** | | **696.340** | **1556.467** | **100.00** | **100.00** |



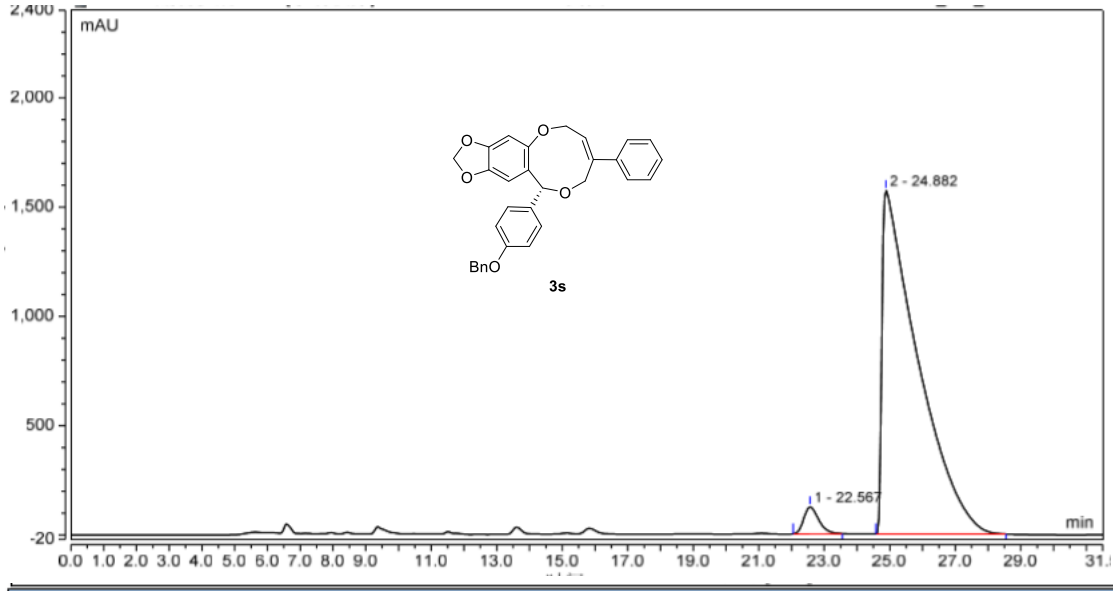

积分结果

| Peak | Retention Time<br>min | Area<br>mAU*min | Height<br>mAU | Area<br>% | Height<br>% |
|---|---|---|---|---|---|
| 1 | 16.292 | 21.595 | 53.342 | 5.02 | 7.50 |
| 2 | 20.288 | 408.976 | 657.663 | 94.98 | 92.50 |
| **Total:** | | **430.570** | **711.005** | **100.00** | **100.00** |



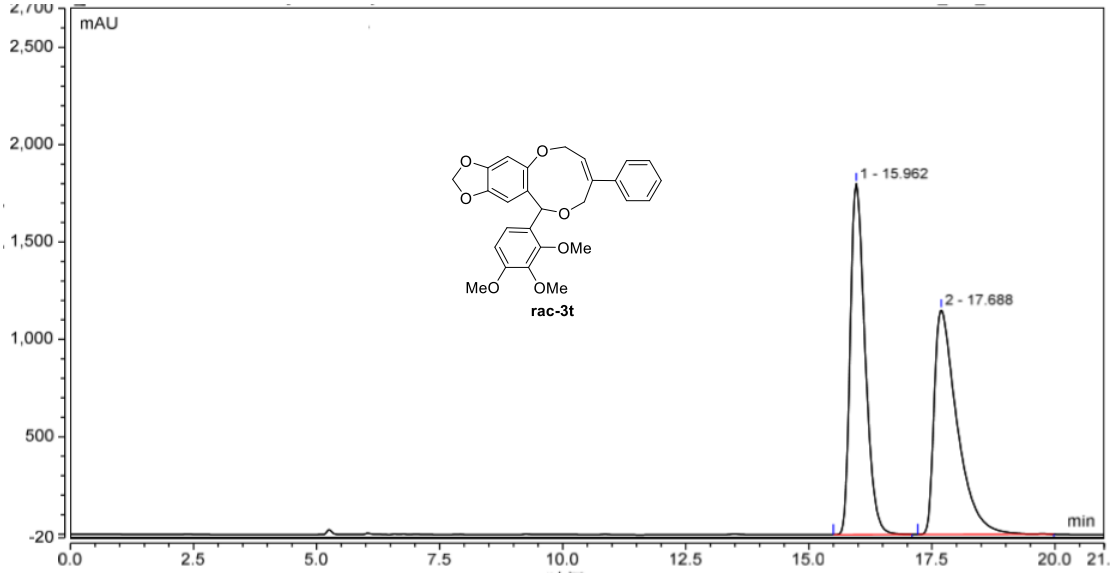

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 22.277 | 661.477 | 1132.494 | 50.00 | 61.00 |
| 2 | 25.742 | 661.537 | 723.989 | 50.00 | 39.00 |
| **Total:** | | **1323.015** | **1856.484** | **100.00** | **100.00** |



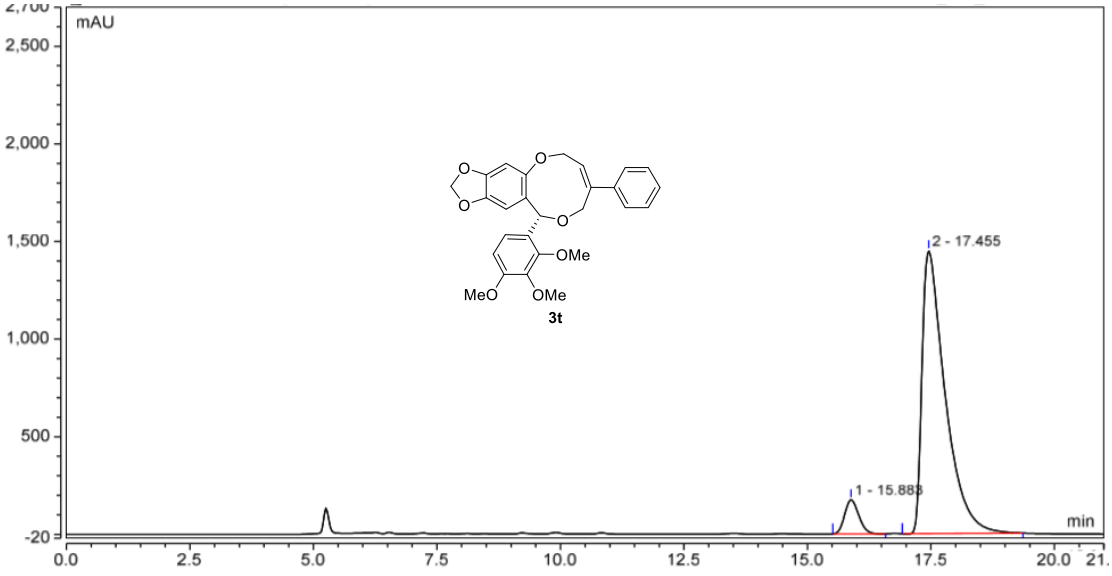

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 22.567 | 65.396 | 123.036 | 3.19 | 7.26 |
| 2 | 24.882 | 1986.972 | 1570.858 | 96.81 | 92.74 |
| **Total:** | | **2052.367** | **1693.894** | **100.00** | **100.00** |



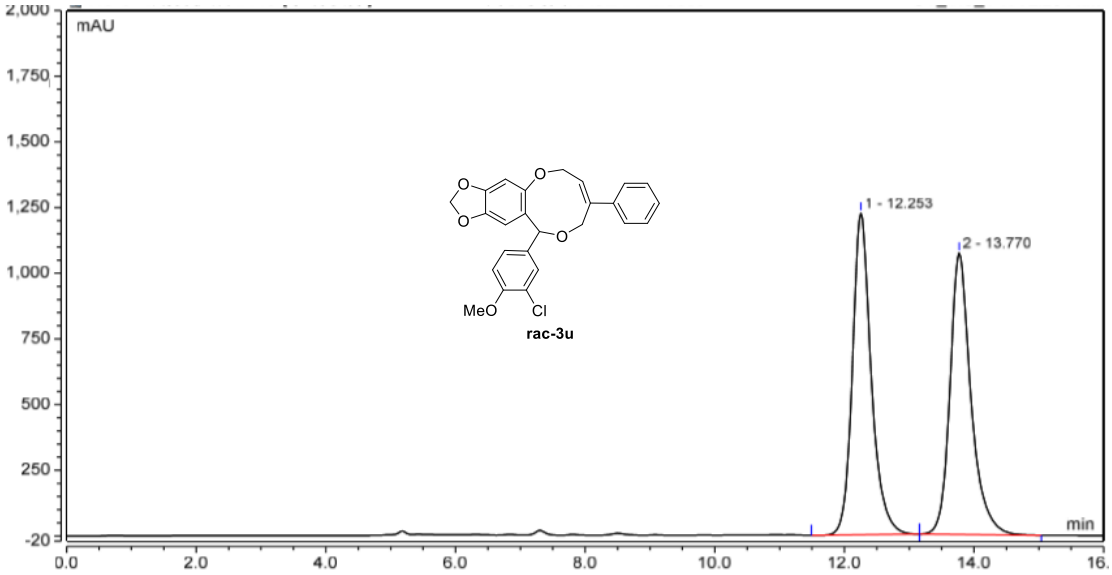

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 15.962 | 628.663 | 1803.516 | 50.16 | 60.98 |
| 2 | 17.688 | 624.605 | 1153.926 | 49.84 | 39.02 |
| **Total:** | | **1253.268** | **2957.441** | **100.00** | **100.00** |



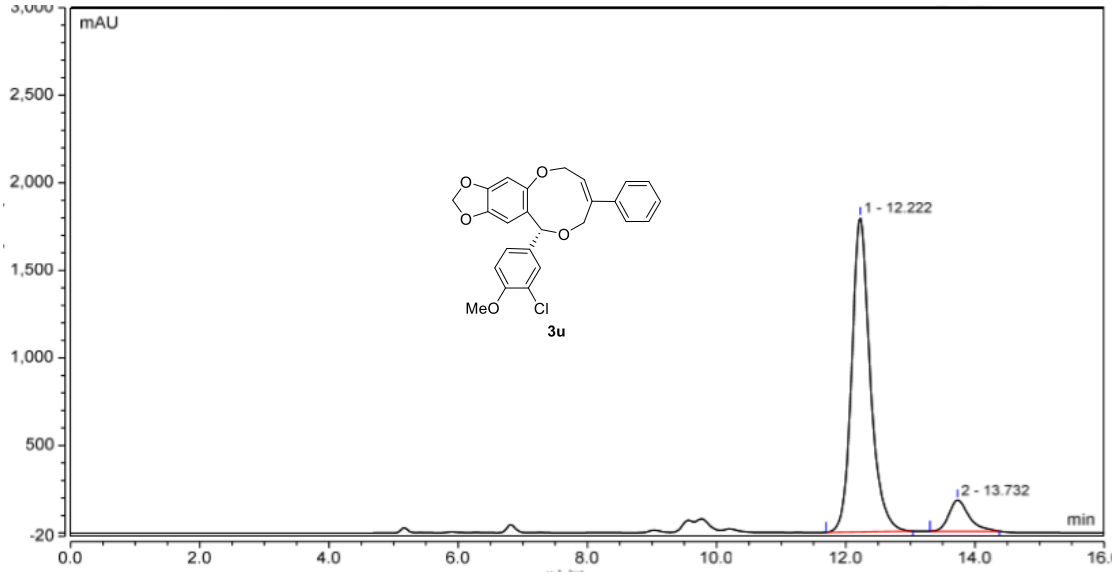

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 15.883 | 57.986 | 174.785 | 6.87 | 10.77 |
| 2 | 17.455 | 785.659 | 1448.086 | 93.13 | 89.23 |
| **Total:** | | **843.646** | **1622.870** | **100.00** | **100.00** |



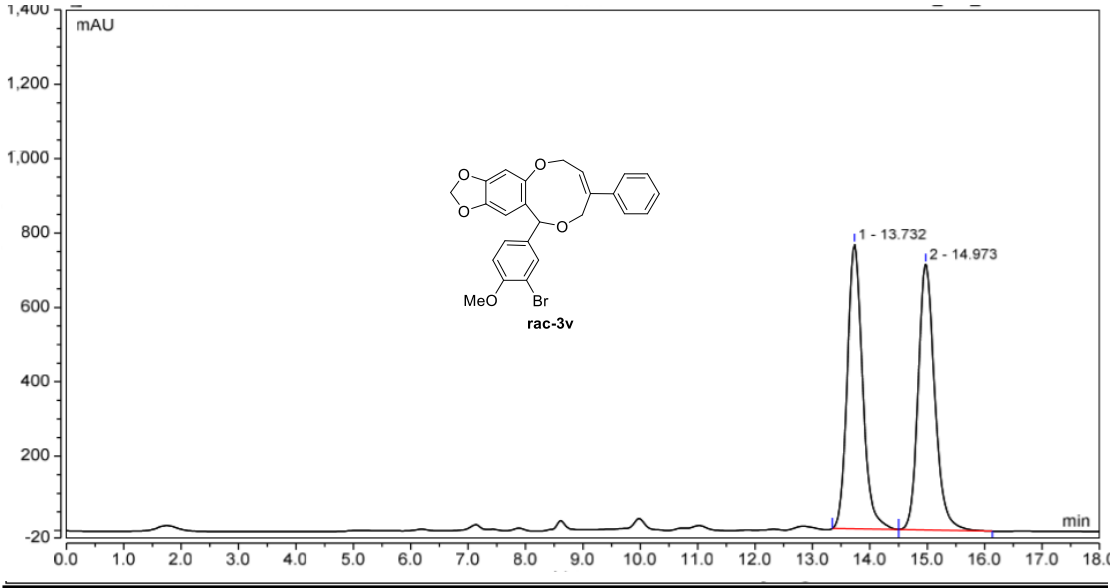

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 12.253 | 412.630 | 1224.975 | 50.59 | 53.33 |
| 2 | 13.770 | 403.018 | 1072.049 | 49.41 | 46.67 |
| **Total:** | | **815.648** | **2297.025** | **100.00** | **100.00** |



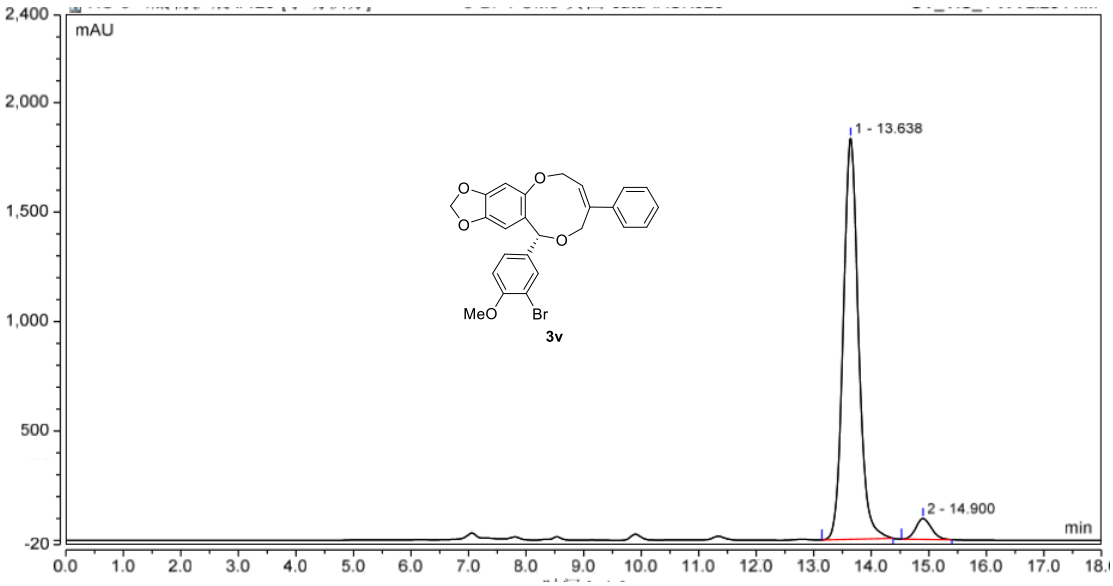

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 12.222 | 595.349 | 1795.024 | 90.24 | 90.99 |
| 2 | 13.732 | 64.423 | 177.767 | 9.76 | 9.01 |
| **Total:** | | **659.772** | **1972.791** | **100.00** | **100.00** |



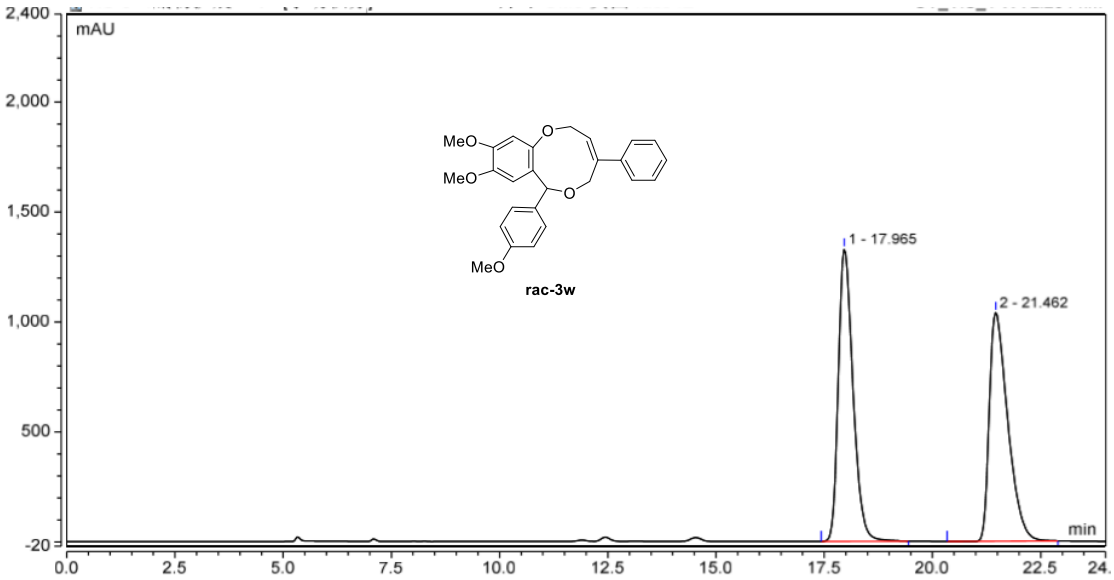

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.732 | 236.750 | 765.486 | 49.70 | 51.71 |
| 2 | 14.973 | 239.584 | 714.862 | 50.30 | 48.29 |
| **Total:** | | **476.334** | **1480.348** | **100.00** | **100.00** |



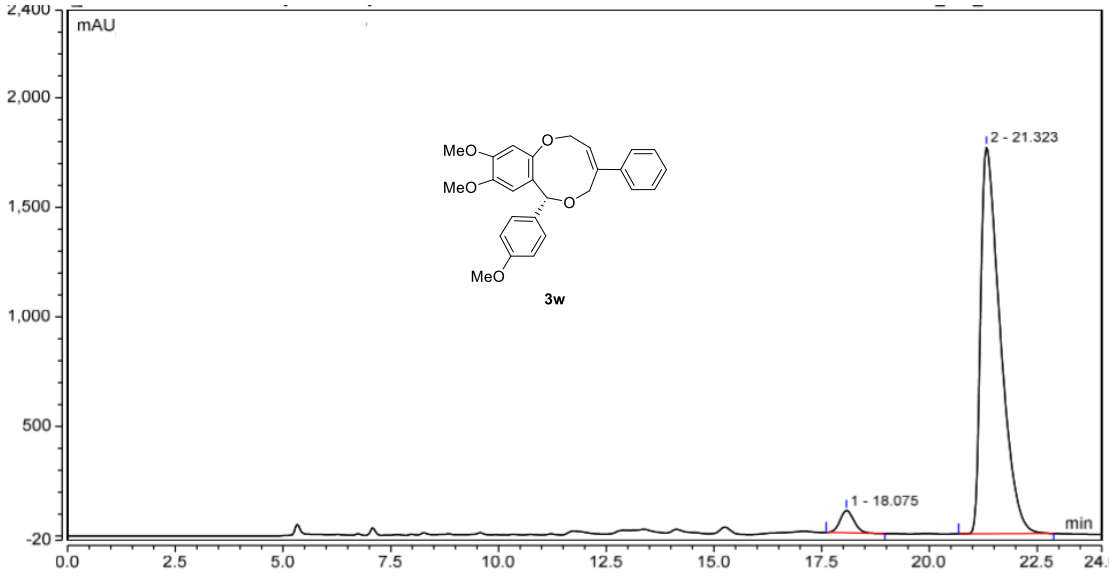

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 13.638 | 560.905 | 1832.550 | 94.91 | 95.05 |
| 2 | 14.900 | 30.059 | 95.525 | 5.09 | 4.95 |
| **Total:** | | **590.964** | **1928.075** | **100.00** | **100.00** |



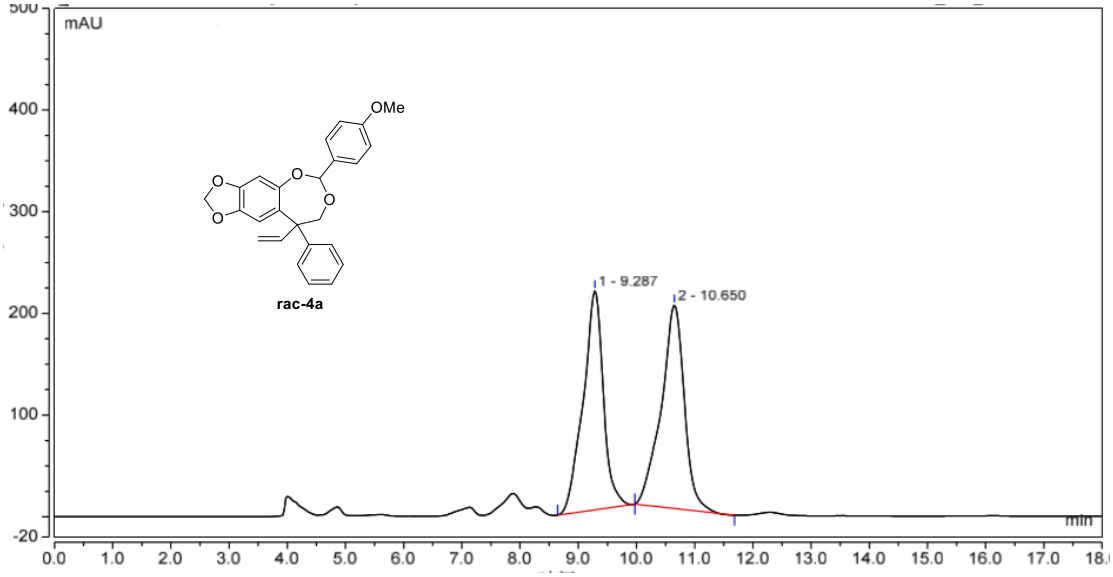

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 17.965 | 522.714 | 1329.919 | 50.41 | 56.12 |
| 2 | 21.462 | 514.203 | 1039.819 | 49.59 | 43.88 |
| **Total:** | | **1036.917** | **2369.737** | **100.00** | **100.00** |



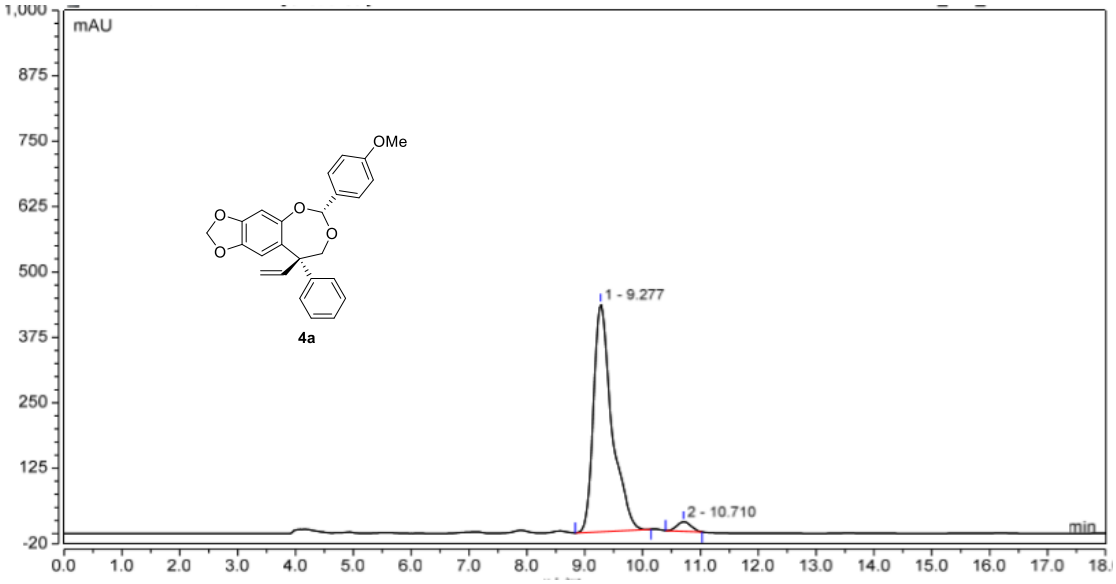

积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 18.075 | 38.558 | 101.979 | 3.99 | 5.46 |
| 2 | 21.323 | 928.870 | 1766.508 | 96.01 | 94.54 |
| **Total:** | | **967.428** | **1868.487** | **100.00** | **100.00** |




积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 9.287 | 86.832 | 215.288 | 48.49 | 51.83 |
| 2 | 10.650 | 92.226 | 200.051 | 51.51 | 48.17 |
| **Total:** | | **179.058** | **415.339** | **100.00** | **100.00** |




积分结果

| Peak | Retention Time min | Area mAU*min | Height mAU | Area % | Height % |
|---|---|---|---|---|---|
| 1 | 9.277 | 160.240 | 434.461 | 96.85 | 95.97 |
| 2 | 10.710 | 5.205 | 18.260 | 3.15 | 4.03 |
| **Total:** | | **165.445** | **452.721** | **100.00** | **100.00** |