Electronic Supplementary Material (ESI) for RSC Advances.


# Ruthenium-catalysed *C*-alkylation of 1,3-dicarbonyl compounds with primary alcohols and synthesis of 3-keto-quinolines

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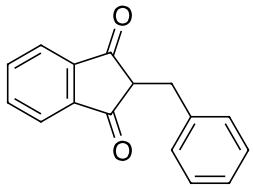
**Supporting Information**

**GENERAL EXPERIMENTAL DETAILS**

All reagents were used as purchased from commercial suppliers without further purification. The reactions were carried out in oven dried or flamed vessels. Solvents were dried and purified by conventional methods prior use. Toluene was distilled from sodium. Flash column chromatography was performed with Merck silica gel 60, 0.040-0.063 mm (230-400 mesh). Merck aluminium backed plates pre-coated with silica gel 60 (UV254) were used for analytical and preparative thin layer chromatography and were visualized by staining with a $KMnO_4$ solution. NMR spectra were recorded at 25 °C and 400 MHz for $^1H$ and 100 MHz for $^{13}C$. The solvent is specified for each spectrum. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. Chemical shifts (δ) are given in ppm relative to the resonance of their respective residual solvent peak. GC/MS analyses were performing using a ion-trap detector equipped with a 30 m OV-101 capillary column, splitting injector at 280 °C, method: 80 °C - 200 °C 30 min. High and low resolution mass spectroscopy analyses were recorded by electronspray ionization. Melting points were determined in open capillary tubes and are uncorrected. MW heated reaction were carried out with a CEM Discover oven, the temperature recorder through the IR sensor. The internal pressure was monitored during the reaction and the max value is reported.

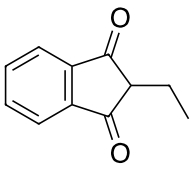
**2-Benzyl-1,3-indandione** [1]




**12**

**Yield:** 64% Solid that decomposes on heating. $^1H$-NMR (400 MHz, $CDCl_3$): δ 7.87 – 7.82 (m, 2H), 7.71 (d, *J* = 5.7 Hz, 2H), 7.15 – 7.03 (m, 5H), 3.30 (s, 3H). $^{13}C$-NMR (100 MHz, $CDCl_3$): δ 199.6, 142.1, 136.7, 135.1, 129.1, 127.8, 126.2, 122.6, 54.6, 31.8**.** GC/MS: $R_t$ 20.23 min; m/z 236 ($C_{16}H_{12}O_2$). HRMS (EI): Calcd for $C_{16}H_{12}O_2Na$ $[M+Na]^+$ 259.0735, found 259.0738.

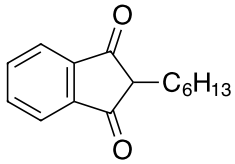
**2-Ethyl-1,3-indandione**




**13**

**Yield:** 61% Mp 53-54 °C (Lit mp: 55 °C).[2] $^{1}$H-NMR (400 MHz, $CDCl_3$): δ 8.16 – 7.49 (m, 4H), 2.98 – 2.90 (m, 1H), 2.07 – 1.94 (m, 2H), 0.95 (t, *J* = 7.6 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 200.8, 142.3, 133.9, 127.6, 51.1, 17.0, 11.5. GC/MS: $R_t$ 14.01 min; m/z 174 ($C_{11}H_{10}O_2$). HRMS (EI): Calcd for $C_{11}H_{10}O_2Na$ $[M+Na]^+$ 197.0579, found 197.0576.

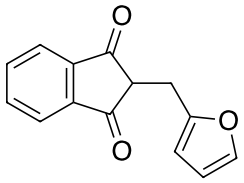
**2-Hexyl-1,3-indandione**




**14**

**Yield:** 57% Dense waxy material. $^{1}$H-NMR (400 MHz, $CDCl_3$): δ 8.23 – 7.51 (m, 4H), 2.97 (t, *J* = 6.0 Hz, 1H), 1.95 – 1.75 (m, 2H), 1.36 – 1.10 (m, 8H), 0.79 (d, *J* = 16.9 Hz, 3H). $^{13}$C-NMR (100 MHz, $CDCl_3$): δ 201.0, 140.9, 132.6, 127.2, 53.0, 31.9, 29.5, 29.0, 22.3, 15.1. GC/MS: $R_t$ 18.81 min; m/z 230 ($C_{15}H_{18}O_2$). HRMS (EI): Calcd for $C_{15}H_{18}O_2Na$ $[M+Na]^+$ 253.1204, found 253.1206.

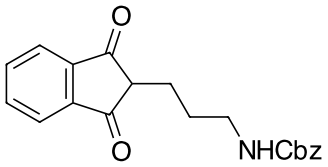
**2-Furfuryl-1,3-indandione**




**15**

**Yield:** 66% Mp 96-98 °C (Lit. mp 97.5-98.0 °C)[3] $^{1}$H-NMR (400 MHz, $CDCl_3$) δ 7.91 (dt, *J* = 6.8, 3.4 Hz, 2H), 7.81 – 7.73 (m, 2H), 7.05 (d, *J* = 1.9 Hz, 1H), 6.10 (d, *J* = 2.7 Hz, 1H), 5.96 (d, *J* = 3.2 Hz, 1H), 3.34 (d, *J* = 5.4 Hz, 2H), 3.31 – 3.26 (m, 1H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 199.6, 151.9, 142.4, 141.3, 134.6, 123.7, 110.3, 107.5, 53.0, 24.5. HRMS (EI): Calcd for $C_{14}H_{10}O_3Na$ $[M+Na]^+$ 249.0528, found 249.0527.

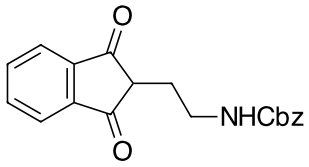
**2-(3-Benzyloxycarbonylaminopropyl)-1,3-indandione**




**16**

**Yield:** 76% Dense material that tends to solidify. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.09 – 7.69 (m, 4H), 7.42 – 6.98 (m, 5H), 4.98 (m, 3H), 3.18 (q, *J* = 6.3 Hz, 2H), 2.98 (t, *J* = 6.1 Hz, 1H), 1.98 – 1.82 (m, 2H), 1.64 (dq, *J* = 14.3, 6.9 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 204.1, 145.8, 139.3, 132.0, 131.6, 126.8, 70.2, 56.4, 44.2, 30.5, 27.6. HRMS (EI): Calcd for $C_{20}H_{19}NO_4Na$ $[M+Na]^+$ 360.1212, found 360.1210.

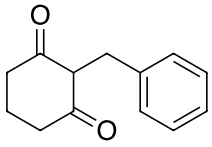
**2-(3-Benzyloxycarbonylaminoethyl)-1,3-indandione**




**17**

**Yield:** 64% M.p. 67-68 °C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.09 – 7.62 (m, 4H), 7.44 – 7.09 (m, 5H), 5.24(bs, 1H) 4.99 (m, 2H), 3.44 (m, 2H), 3.04 (t, *J* = 6.4 Hz, 1H), 2.18 – 1.94 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 199.8, 155.8, 141.6, 136.2, 135.3, 128.0, 127.6, 123.7, 122.9, 66.2, 51.3, 38.6, 29.2, 26.4. HRMS (EI): Calcd for $C_{19}H_{17}NO_4Na$ $[M+Na]^+$ 346.1055, found 346.1053.

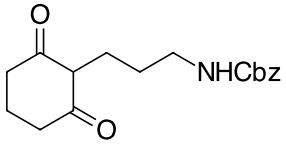
**2-Benzyl-1,3-cyclohexanedione**




**18**

**Yield:** 62% M.p. 179-181 °C (Lit m.p. 180 °C)[4]. $^{1}$H NMR (400 MHz, $CD_3OD$) δ 7.37 – 6.86 (m, 5H), 4.83 (s, 2H), 3.30 (s, 1H), 2.43 (t, *J* = 6.2 Hz, 4H), 1.94 (p, *J* = 5.4, 4.6 Hz, 2H). $^{13}$C NMR (100 MHz, $CD_3OD$) δ 199.2, 139.7, 126.1, 125.5, 122.9, 113.3, 30.4, 27.4, 25.0, 20.9, 18.6. GC/MS**:** $R_t$ 18.34 min; m/z 202 ($C_{13}H_{14}O_2$). HRMS (EI): Calcd for $C_{13}H_{14}O_2Na$ $[M+Na]^+$ 225.0892, found 225.0895.

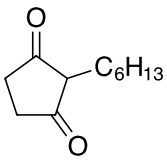
**2-(3-Benzyloxycarbonylaminopropyl)-1,3-cyclohexanedione**




**19**

**Yield:** 52% M.p. 94-96 °C. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.20 (m, 5H), 5.06 (d, *J* = 9.2 Hz, 3H), 3.15 (t, *J* = 10.6 Hz, 1H), 3.06 (q, *J* = 6.2 Hz, 2H), 2.58 (m, 1H), 2.45 (m, 2H), 2.32 (m, 2H), 2.14 (m, 1H) 1.90 (m, 2H), 1.55 (m, 2H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 209.4, 161.4, 132.1, 131.6, 131.5, 70.6, 43.7, 40.7, 39.7, 33.3, 27.3, 24.4, 22.0. HRMS (EI): Calcd for $C_{17}H_{21}NO_4Na$ $[M+Na]^+$ 326.1369, found 326.1365.

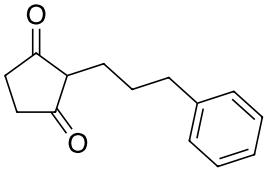
**2-Hexyl-1,3-cyclopentanedione**




**20**

**Yield:** 51% Solid that decomposes on heating (Lit m.p. 240 °C)[5] $^{1}$H NMR (400 MHz, $CD_3OD$) δ 2.47 (s, 4H), 2.09 (t, *J* = 7.4 Hz, 2H), 1.42 – 1.23 (m, 9H), 0.89 (t, *J* = 6.7 Hz, 3H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 203.5, 49.6, 31.2, 30.2, 29.8, 26.9, 22.3, 20.3, 13.2. HRMS (EI): Calcd for $C_{11}H_{18}O_2Na$ $[M+Na]^+$ 205.1205, found 205.1203.

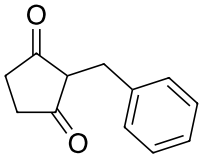
**2-(3-Phenylpropyl)-1,3-cyclopentanedione**



**21**

**Yield:** 47% Gum that tends to solidify on standing. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 7.36 – 6.99 (m, 5H), 2.54 (d, *J* = 33.1 Hz, 7H), 2.22 (t, *J* = 7.6 Hz, 2H), 1.75 (p, *J* = 7.8 Hz, 2H). $^{13}$C NMR (100 MHz, CDCl3) δ 197.8, 142.4, 127.5, 125.2, 43.5, 38.8, 35.6, 28.8, 20.3. HRMS (EI): Calcd for $C_{14}H_{16}O_2Na$ $[M+Na]^+$ 239.1048, found 239.1050.

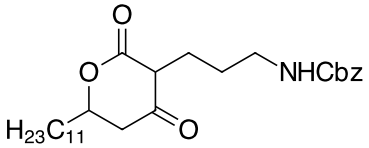
**2-Benzyl-1,3-cyclopentanedione**



**22**

**Yield:** 49% Solid that decomposes on heating . $^{1}$H NMR (400 MHz, $CD_3OD$) δ 7.25 – 6.98 (m, 5H), 3.49 – 3.15 (m, 3H), 2.48 (s, 4H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 198.2, 140.3, 128.2, 127.7, 125.5, 116.6, 36.9, 32.9, 26.1. HRMS (EI): Calcd for $C_{12}H_{12}O_2Na$ $[M+Na]^+$ 211.0735, found 211.0733.

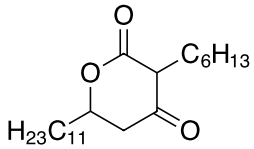
**3-(3-Benzyloxycarbonylaminopropyl)-6-undecyl-5,6-dihydro-3*H*-pyran-2,4-dione**




**23**

**Yield:** 44% Dense waxy material $^{1}$H NMR (400 MHz, $CDCl_3$); the spectrum is refereed to the prevalent enolic form: δ 7.33 (s,5H), 5.09 (d, *J* = 19.3 Hz, 3H), 4.29 (m, 1H), 3.10 (m, 2H), 2.58 – 2.26 (m, 2H), 1.83 – 1.48 (m, 4H), 1.24 (s, 20H), 0.86 (t, *J* = 6.6 Hz, 3H). $^{13}$C NMR (101 MHz, ) δ 174.9, 169.6, 154.9, 132.1, 131.7, 99.8, 81.0, 80.7, 80.3, 79.2, 71.9, 70.6, 70.4, 41.1, 41.1, 40.8, 40.6, 39.8, 38.1, 37.0, 36.1, 33.7, 33.7, 33.3, 33.3, 27.4, 26.7, 26.5, 25.9, 24.4, 24.0, 23.5, 22.5, 22.3, 22.1. A preparative TLC gave the analytical sample. HRMS (EI): Calcd for $C_{27}H_{41}NO_5Na$ $[M+Na]^+$ 482.2883, found 482.2880.

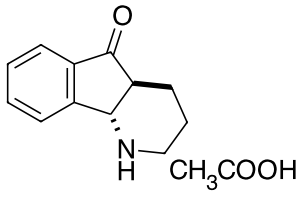
**3-Hexyl-6-undecyl-5,6-dihydro-3*H*-pyran-2,4-dione**




**2**

**Yield:** 50% M.p 110-112 °C (Lit. m.p. 112 °C)[6] $^1$H NMR (400 MHz, $CDCl_3$) enol form: δ 4.64 (m, 1H), 2.74 – 2.59 (m, 1H), 2.38 (dd, *J* = 18.8, 11.8 Hz, 1H), 1.98 – 1.69 (m, 3H), 1.41 – 1.22 (m, 28H), 0.85 (t, *J* = 5.8 Hz, 6H). A preparative TLC gave the analytical sample. HRMS (EI): Calcd for $C_{22}H_{40}O_3Na$ $[M+Na]^+$ 375.2876 100, found 375.2873

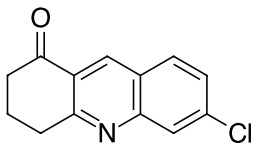
**2,3,4,4a-Tetrahydro-1H-indeno[1,2-b]pyridin-5(9bH)-one acetate**




**28**

In a round bottom flask containing a magnetic stirrer bar, 2-(3-benzyloxycarbonylaminopropyl)-1,3-indandione **16** (67 mg, 0.2 mmol) was solubilized in 20 mL of MeOH under nitrogen. Acetic acid (25 mg, 24 µL, 0.42 mmol) and 20% $Pd(OH)_2$ on charcoal (3 mg, 0.021 mmol) were added and reaction mixture was stirred under $H_2$ for 72 h. The reaction mixture was then filtered on celite and the catalyst washed with MeOH. The solvent was evaporated and the crude purified by flash chromatography ($CH_2Cl_2$ to $CH_2Cl_2$/MeOH, 80:20). Obtained 22 mg, (47 % yield) of a solid that decomposes on heating. *Major diasteroisomer* (**trans**) $^1$H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.71 (m, 1H), 7.23 (d, *J* = 3.8 Hz, 4H), 4.19 (d, *J* = 11.4 Hz, 1H), 3.37 – 3.29 (m, 2H), 2.97 (dd, *J* = 14.7, 6.8 Hz, 1H), 2.78 (s, 3H), 2.52 (dd, *J* = 14.7, 11.3 Hz, 1H), 2.33 (m, 1H), 2.13 (m 1H), 1.94 (m, 2H), 1.67 (m, 1H). $^{13}$C NMR (101 MHz, $CDCl_3$) δ 200.5, 134.3, 128.8, 128.5, 127.4, 127.1, 124.8, 61.6, 55.1, 46.0, 34.2, 27.5. GC/MS: $R_t$ 15.36 min m/z 187 ($C_{12}H_{13}ON$). HRMS (EI) Calcd. for $C_{12}H_{14}NO$ $[M+H]^+$ 188.1076, found 188.1074.

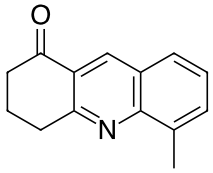
**6-Chloro-3,4-dihydro-2*H*-acridin-1-one**




**37**

**Yield:** 70 % Waxy material $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 7.75 (d, *J* = 9.4 Hz, 1H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.25 (d, *J* = 8.6 Hz, 1H), 3.33 – 2.97 (m, 2H), 2.86 – 2.50 (m, 2H), 2.08 (dt, *J* = 12.5, 6.1 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 197.2, 163.0, 149.6, 138.1, 136.5, 136.3, 130.6, 128.1, 127.5, 127.1, 126.2, 124.9, 38.8, 33.2, 21.5. HRMS (EI): Calcd for $C_{13}H_{11}ClNO$ $[M+H]^+$ 232.0529, found 232.0526.

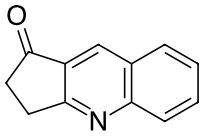
**5-Methyl-3,4-dihydro-2*H*-acridin-1-one**




**38**

Yield 62%. M.p. 76-78 °C. $^{1}$H NMR (400 MHz, $CDCl_3$): δ 8.69 (s, 1H), 7.66 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 6.9 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 3.35 – 3.20 (m, 2H), 2.72 (d, *J* = 9.9 Hz, 5H), 2.19 (p, *J* = 6.4 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 197.7, 160.3, 148.3, 136.5, 136.2, 131.7, 127.2, 126.2, 125.8, 125.4, 38.7, 33.2, 21.5, 17.5. HRMS (EI): Calcd for $C_{14}H_{14}NO$ $[M+H]^+$ 212.1075, found 212.1077.

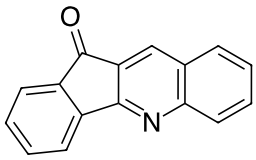
**2-Azatricyclo[7.4.0.0$^{3,7}$]trideca-1(9),2,7,10,12-pentaen-6-one**




**39**

Yileld 49%. Gum $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.53 (s, 1H), 8.09 (d, *J* = 8.6 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.81 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 3.45 – 3.39 (m, 2H), 2.90 – 2.82 (m, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$ ) δ 204.8, 151.7, 133.5, 133.2, 132.6, 130.4, 129.4, 129.0, 127.8, 126.8, 36.3, 28.9. HRMS (EI): Calcd for $C_{12}H_{10}NO$ $[M+H]^+$ 184.0762, found 184.0765.

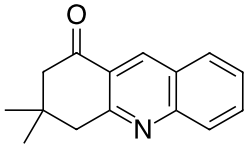
**9-Azatetracyclo[8.7.0.0$^{3,8}$.0$^{11,16}$]heptadeca-1,3(8),4,6,9,11,13,15-octaen-17-one**



**40**

**Yield:** 61% M.p. 172-174°C. Lit m.p. (175-176 °C)[8] $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.30 (s, 1H), 8.05 (t, *J* = 8.7 Hz, 2H), 7.79 (dd, *J* = 14.2, 7.7 Hz, 2H), 7.71 (t, *J* = 7.7 Hz, 1H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.47 (td, *J* = 7.5, 3.6 Hz, 2H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 190.4, 161.6, 150.2, 143.4, 137.0, 135.1, 133.9, 132.0, 131.6, 131.1, 130.1, 129.6, 129.4, 127.8, 127.2, 126.8, 123.7, 121.4. HRMS (EI): Calcd for $C_{16}H_{10}NO$ $[M+H]^+$ 232.0762, found 232.0764.

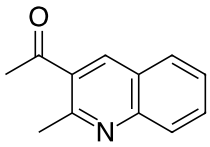
**3,3-Dimethyl-3,4-dihydro-2H-acridin-1-one**



**41**

**Yield:** 67% M.p. 101-102°C. Lit m.p. (101-102 °C)[7] $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.72 (s, 1H), 7.96 (d, *J* = 8.6 Hz, 1H), 7.83 (d, *J* = 8.1 Hz, 1H), 7.75 – 7.65 (m, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 3.11 (s, 2H), 2.56 (s, 2H), 1.06 (s, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 197.4, 160.3, 149.5, 136.0, 131.7, 129.3, 128.1, 126.3, 126.2, 124.8, 52.0, 46.7, 32.3, 29.2, 27.9. HRMS (EI) Calcd for $C_{15}H_{16}NO$ $[M+H]^+$ 226.1232, found 226.1234.

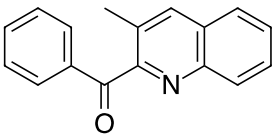
**1-(2-Methyl-3-quinolyl)-1-ethanone**



**42**

**Yield:** 58% M.p. 72-74°C. Lit m.p. (73-74 °C)[7] $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.98 (d, *J* = 8.5 Hz, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.75 – 7.68 (m, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 2.86 (s, 3H), 2.65 (s, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 200.4, 158.0, 148.7, 138.8, 132.2, 131.4, 128.9, 128.7, 127.0, 125.9, 29.7, 26.1. HRMS (EI): Calcd for $C_{12}H_{12}NO$ $[M+H]^+$ 186.0919, found 186.0916.

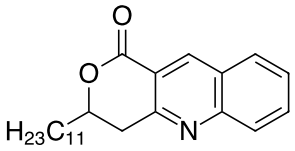
**1-(2-Phenylquinolin-3-yl)ethanone**



**43**

**Yield:** 52% M.p. 131-133 (Lit m.p. 132-133)[9] $^1$H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 12.1 Hz, 1H), 7.86 – 7.70 (m, 2H), 7.70 – 7.39 (m, 5H), 7.22 (dq, *J* = 19.3, 6.8 Hz, 3H), 2.73 (s, 3H). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 12.1 Hz, 1H), 7.86 – 7.70 (m, 3H), 7.70 – 7.39 (m, 5H), 7.22 (dq, *J* = 19.3, 6.8 Hz, 3H), 2.73 (s, 2H). HRMS (EI): Calcd for $C_{17}H_{14}NO$ $[M+H]^+$ 248.1075, found 248.1078.

**3-(5-Undecene-1,3-diynyl)-2-oxa-10-aza-3,4-dihydroanthracen-1-one**



**44**

**Yield:** 44% Waxy material $^1$H NMR (400 MHz, CDCl3) δ 8.89 (s, 1H), 8.03 (d, *J* = 8.6 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.81 (t, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 4.72 – 4.60 (m, 1H), 3.36 – 3.16 (m, 2H), 1.97 – 1.84 (m, 1H), 1.85 – 1.71 (m, 1H), 1.57 – 1.43 (m, 2H), 1.25 (d, *J* = 21.4 Hz, 16H), 0.84 (t, *J* = 6.7 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 164.6, 156.6, 149.3, 139.8, 132.2, 128.9, 128.3, 126.7, 118.5, 78.0, 36.6, 34.6, 31.5, 29.2, 29.1, 28.9, 24.5, 22.2, 13.7. HRMS (EI): Calcd for $C_{23}H_{32}NO_2$ $[M+H]^+$ 354.2433, found 354.2436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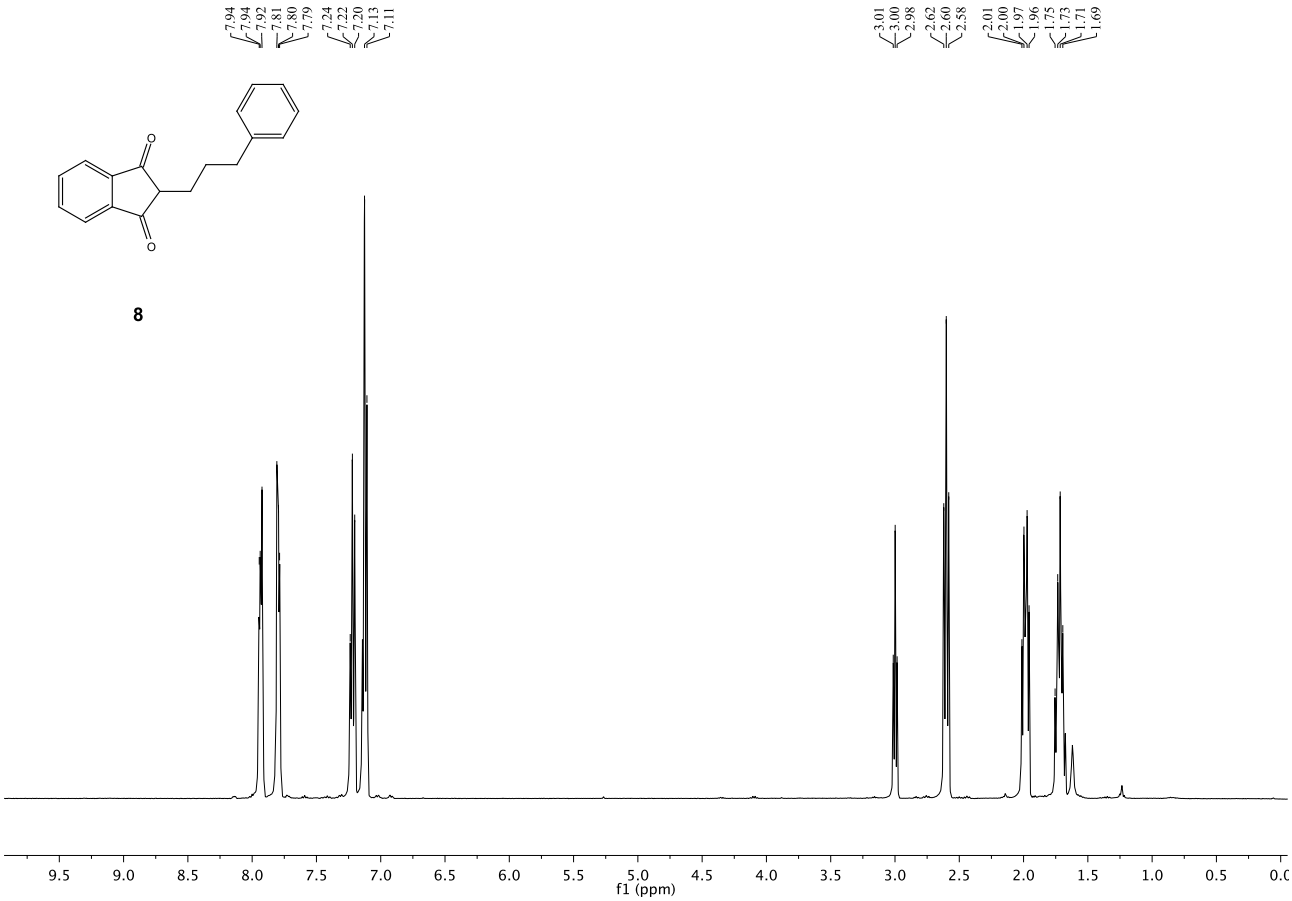
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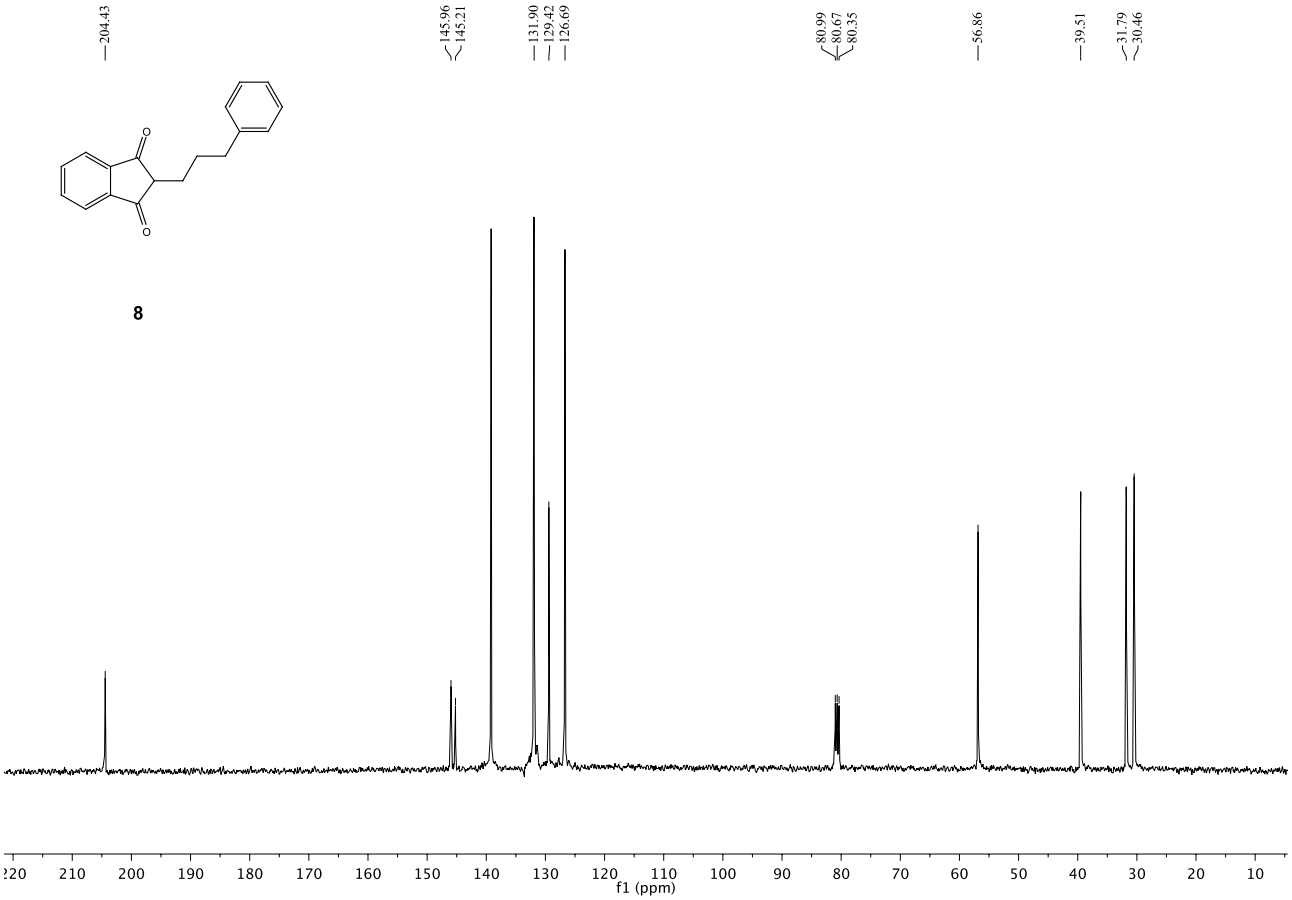
7.94
7.92
7.81
7.80
7.79
7.24
7.22
7.20
7.13
7.11
3.01
3.00
2.98
2.62
2.60
2.58
2.01
2.00
1.97
1.96
1.75
1.73
1.71
1.69
O
O
8
f1 (ppm)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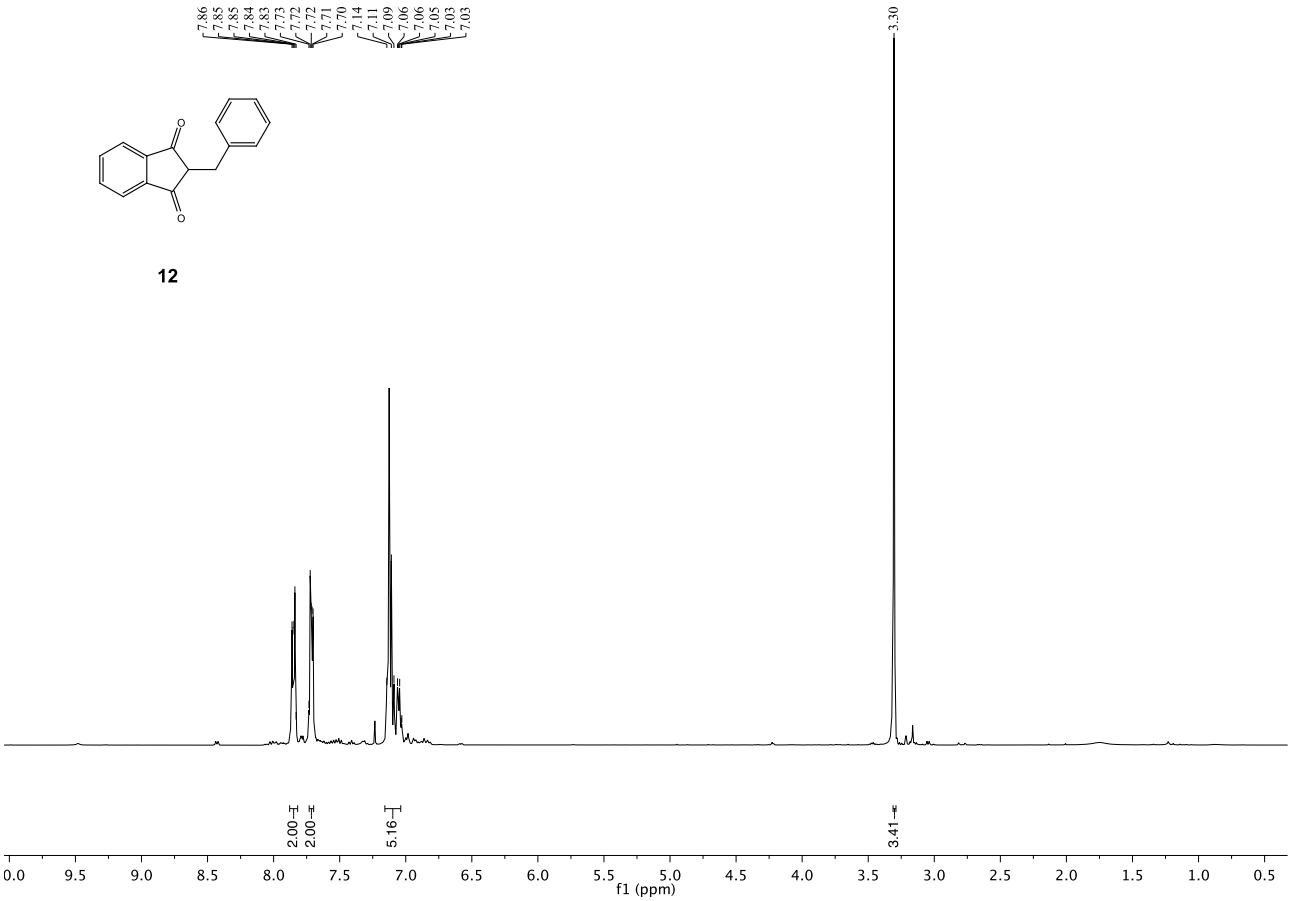
145.96
145.21
131.90
129.42
126.69
80.99
80.67
80.35
56.86
39.51
31.79
30.46
O
O
8
f1 (ppm)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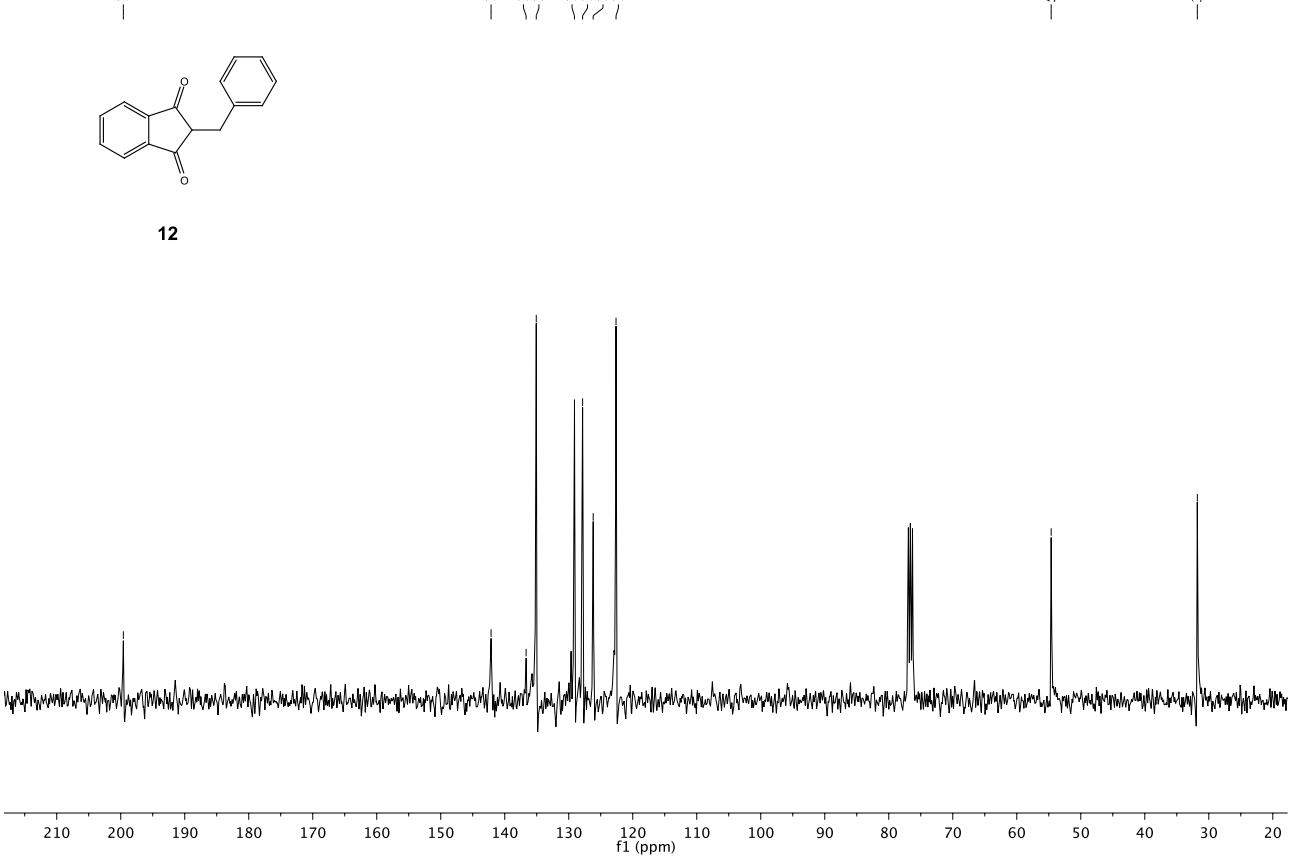
7.85
7.85
7.84
7.83
7.73
7.72
7.72
7.71
7.70
7.14
7.11
7.09
7.06
7.06
7.05
7.03
7.03
3.30
2.00
2.00
5.16
3.41
f1 (ppm)

**12**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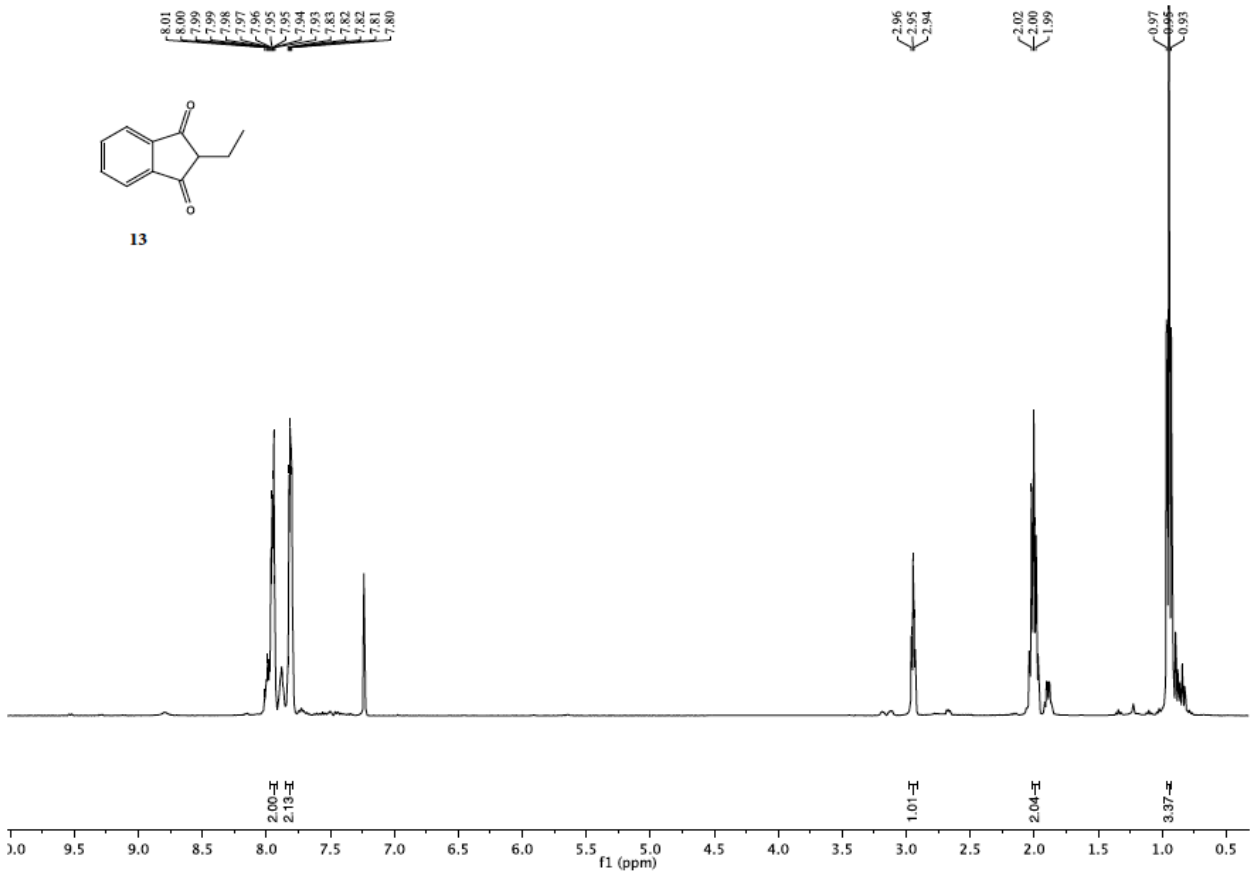
129.11
127.84
126.19
122.62
54.63
31.79
f1 (ppm)

**12**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8.00
7.99
7.99
7.98
7.97
7.96
7.95
7.95
7.94
7.93
7.83
7.82
7.82
7.81
7.80
2.96
2.95
2.94
2.02
2.00
1.99
0.97
0.95
0.93
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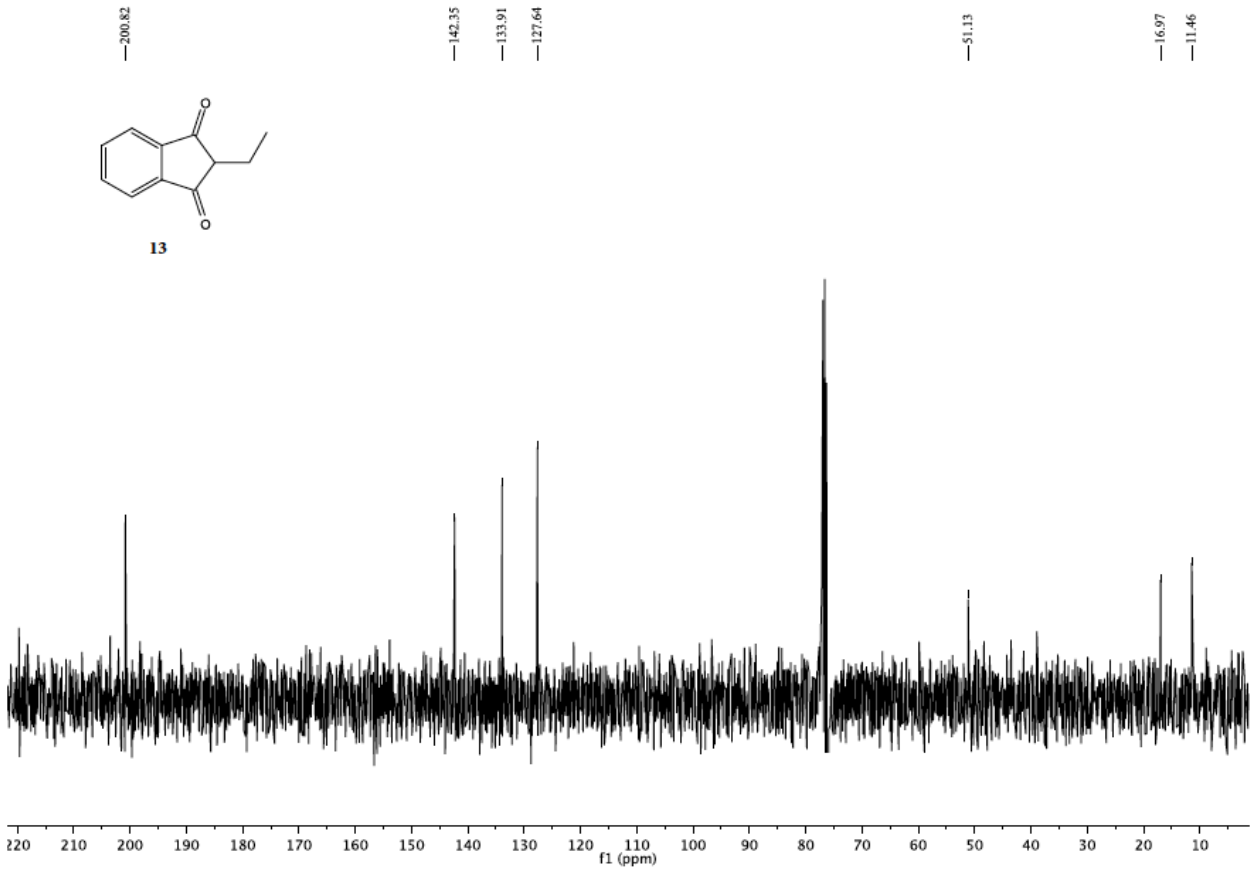
O
13
2.00
2.13
1.01
2.04
3.37
f1 (ppm)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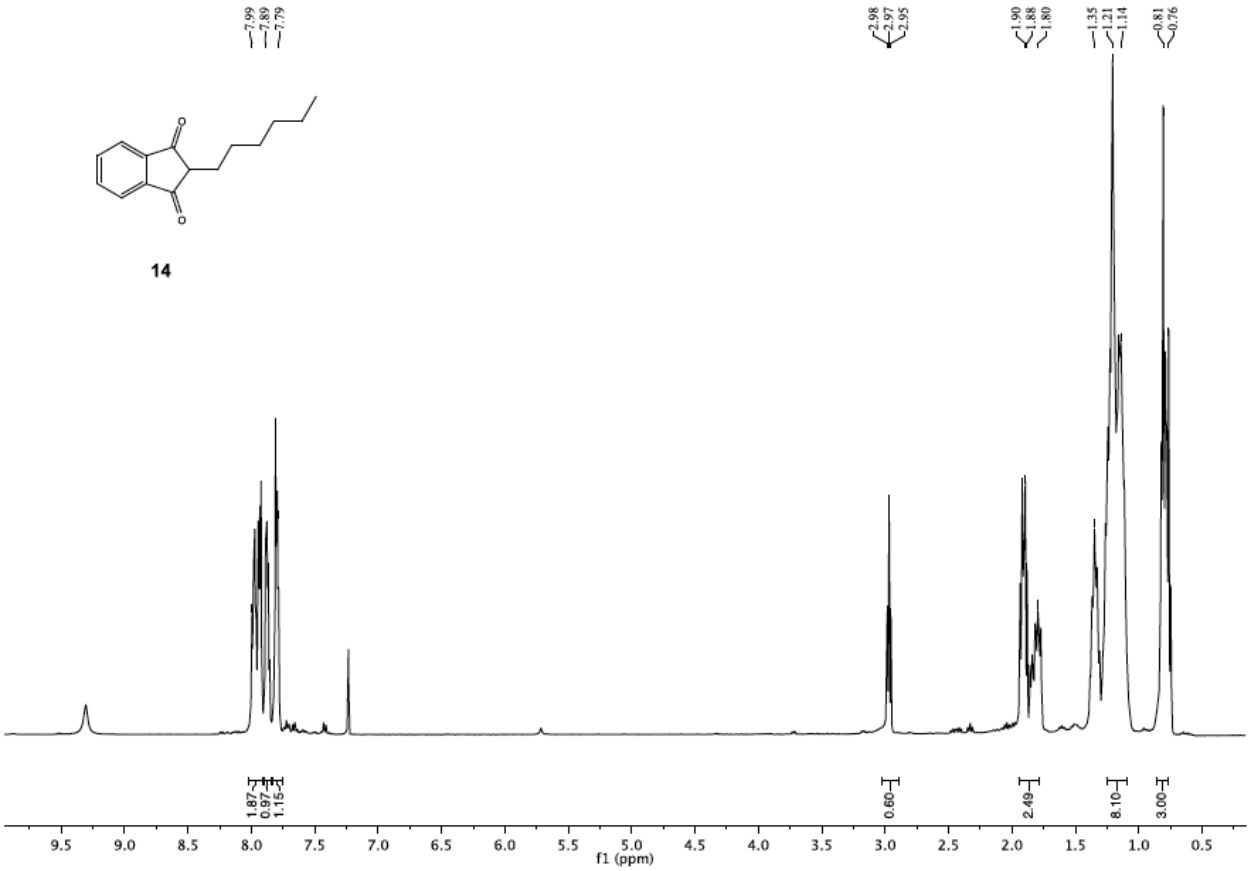
133.91
127.64
51.13
16.97
11.46
O
O
13
f1 (ppm)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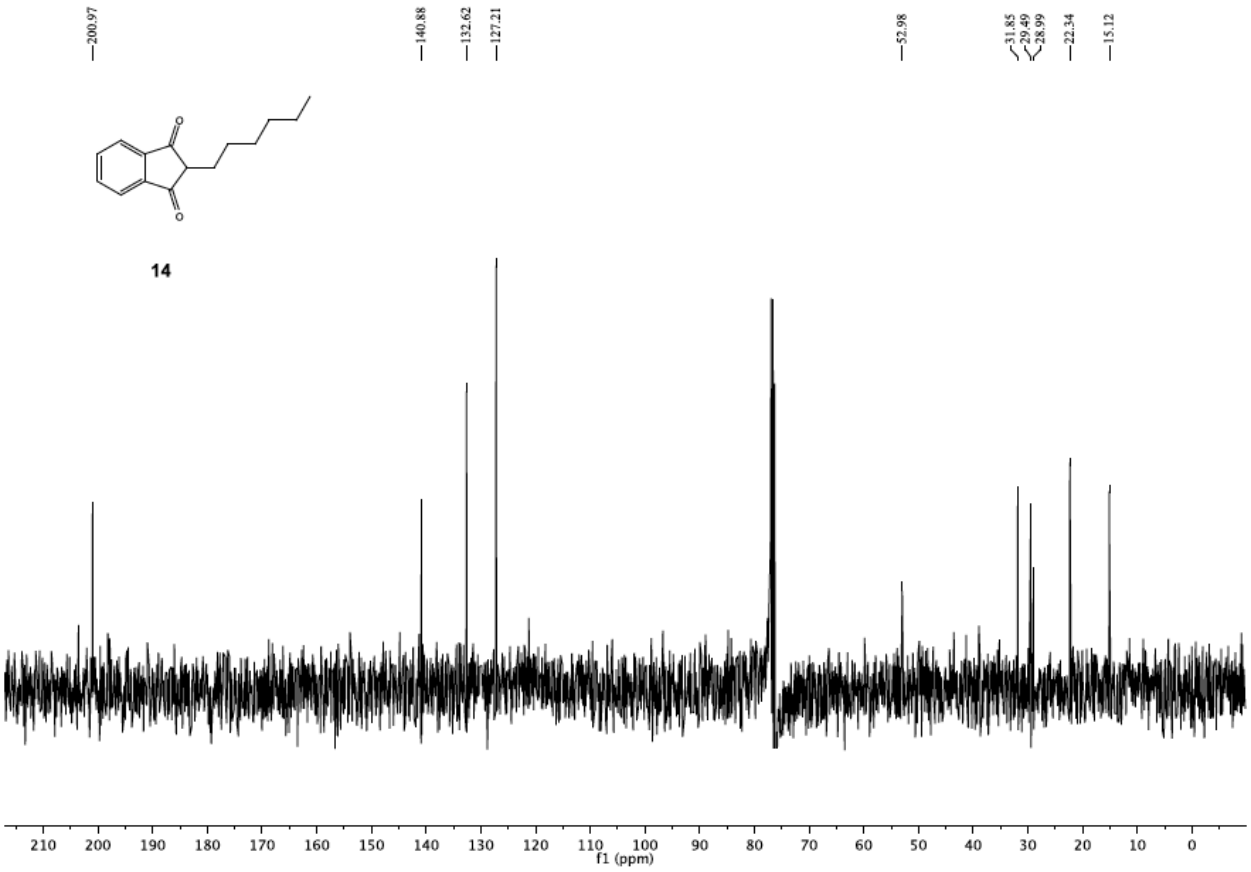
7.89
7.79
2.98
2.97
2.95
1.90
1.88
1.80
1.35
1.21
1.14
0.81
0.76
14
1.87
0.97
1.15
0.60
2.49
8.10
3.00
f1 (ppm)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132.62
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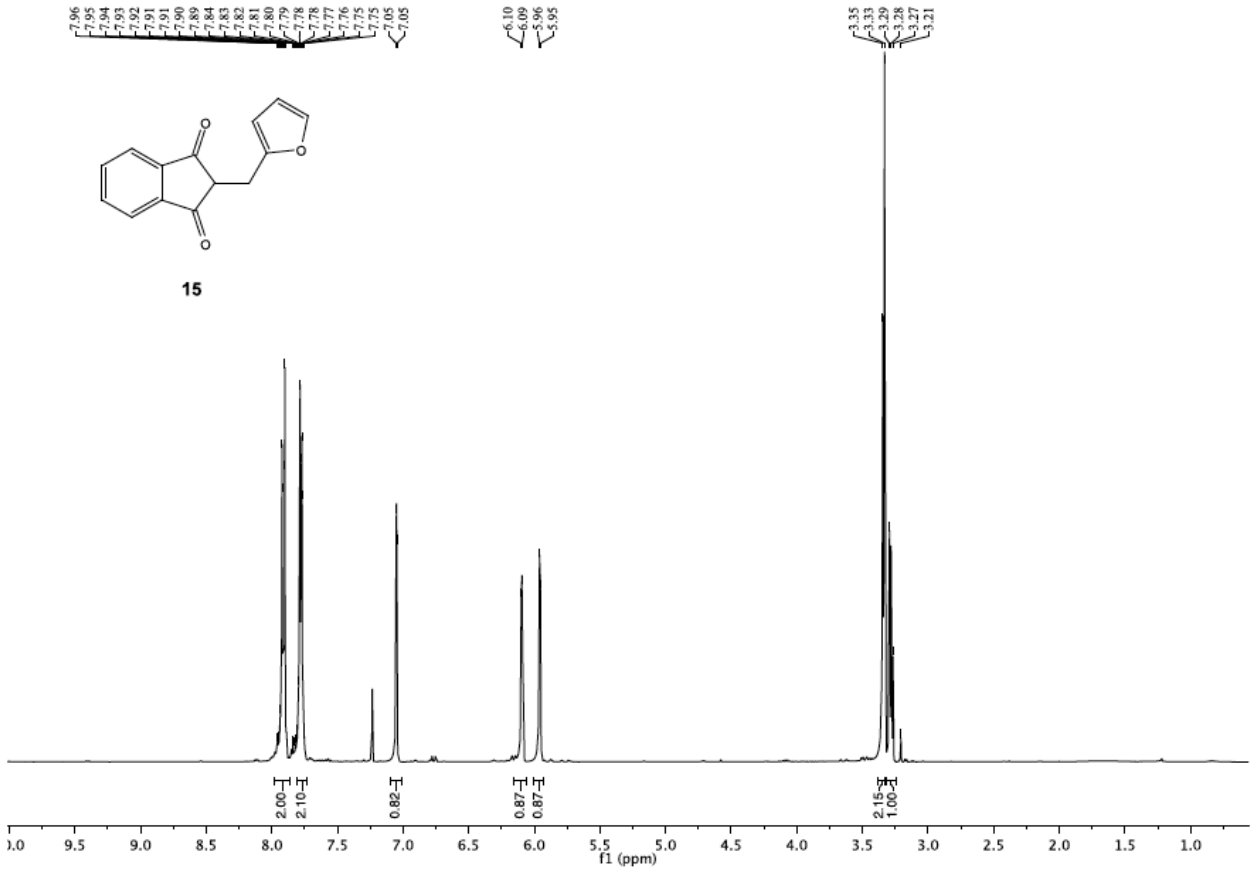
52.98
31.85
29.49
28.99
22.34
15.12
14
f1 (ppm)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7.95
7.94
7.93
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7.91
7.91
7.90
7.89
7.84
7.83
7.82
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7.05
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5.95
3.35
3.33
3.29
3.28
3.27
3.21
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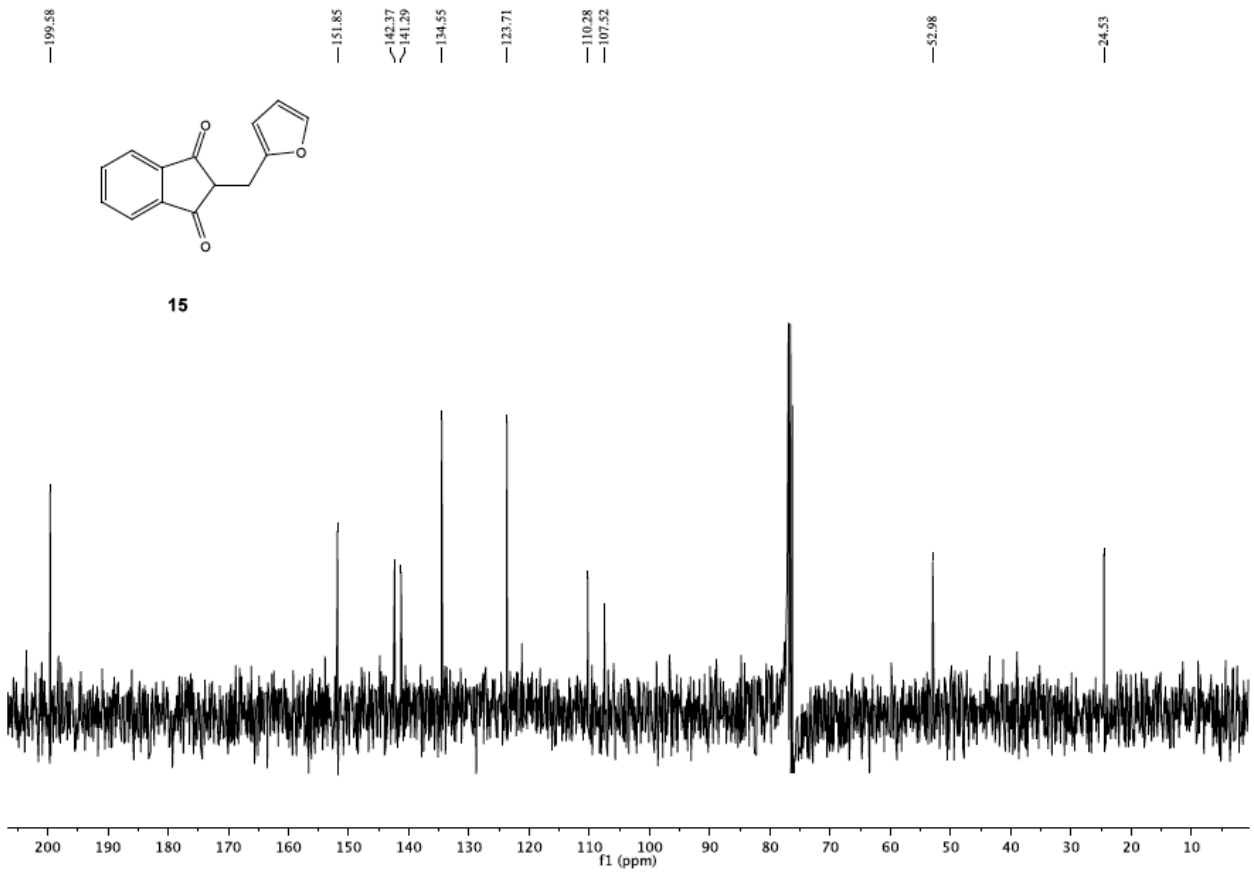
O
O
15
2.00
2.10
0.82
0.87
0.87
2.15
1.00
f1 (ppm)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24.53
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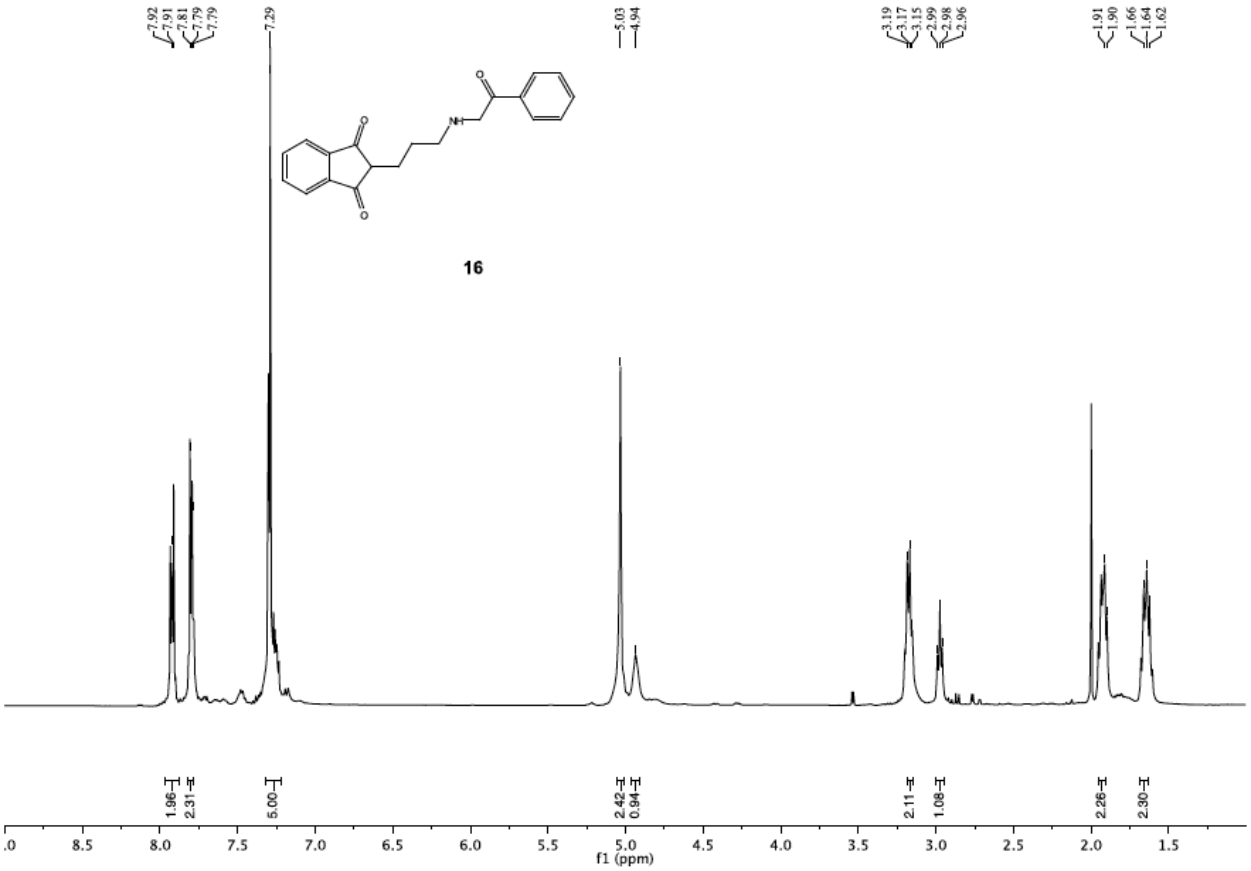
O
15
f1 (ppm)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7.91
7.81
7.79
7.79
7.79
7.29
5.03
4.94
3.19
3.17
3.15
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1.91
1.90
1.66
1.64
1.62
NH
O
O
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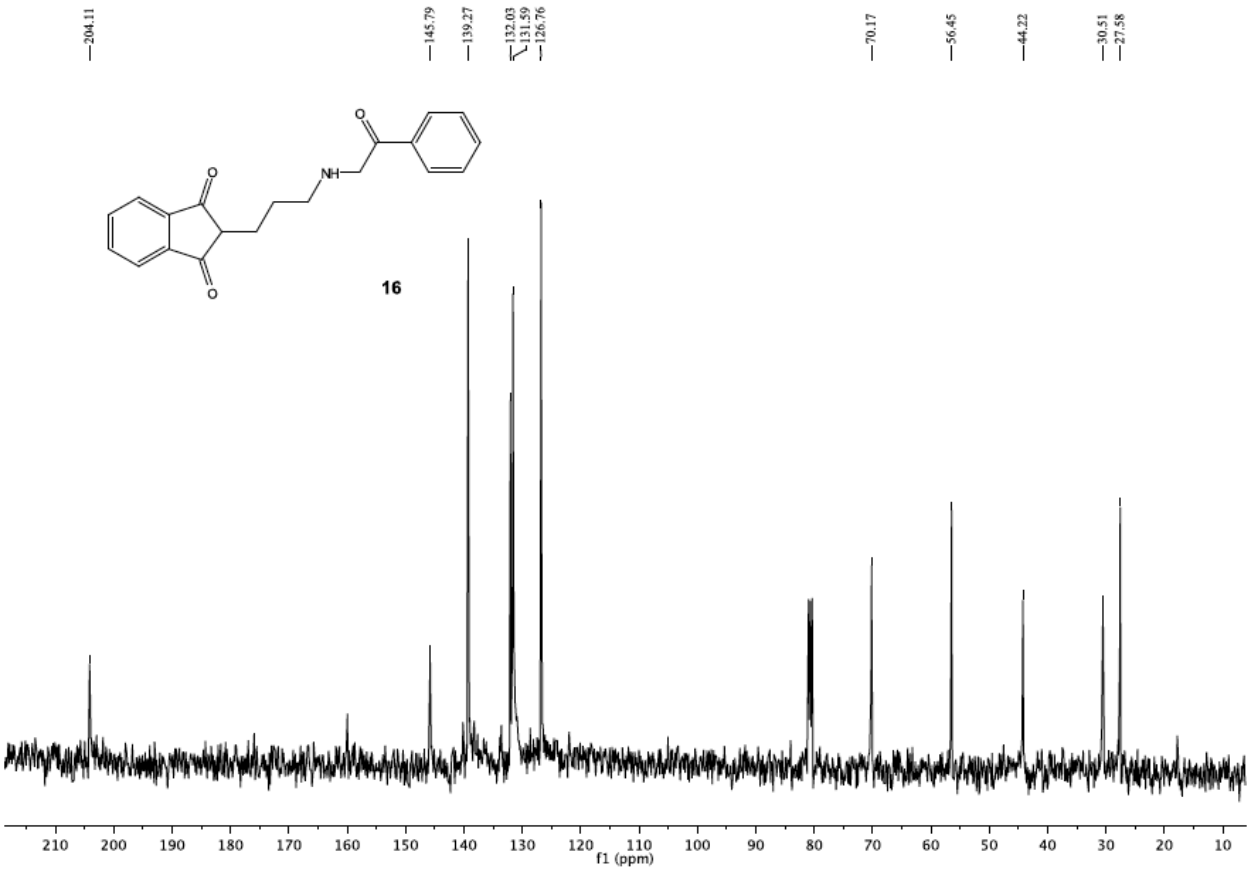
2.11
1.08
2.26
2.30
f1 (ppm)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126.76
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56.45
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30.51
27.58
NH
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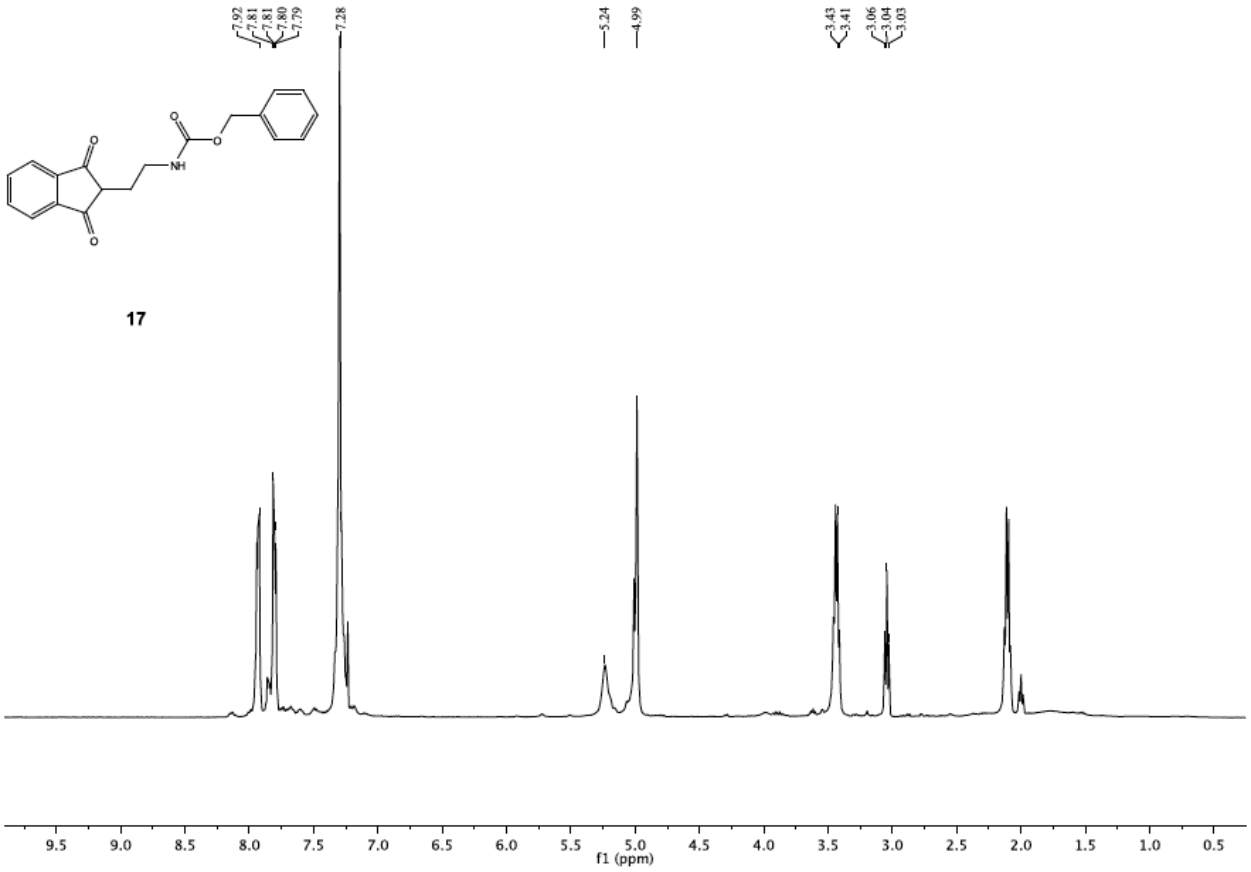
O
O
16
f1 (ppm)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4.99
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3.06
3.04
3.03
NH
O
O
O
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6.5
6.0
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4.0
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2.0
1.5
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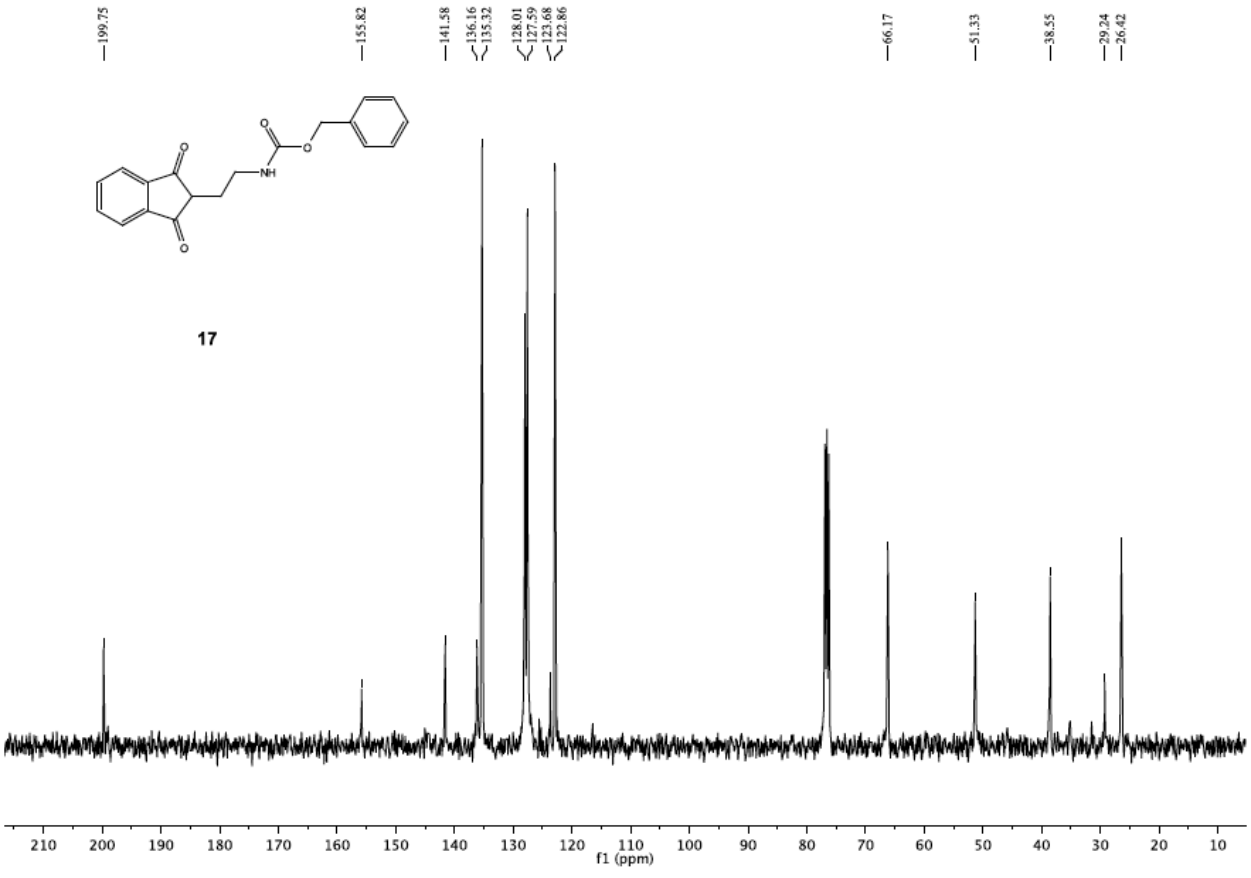
f1 (ppm)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26.42
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O
O
O
O
17
210
200
190
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140
130
120
110
100
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80
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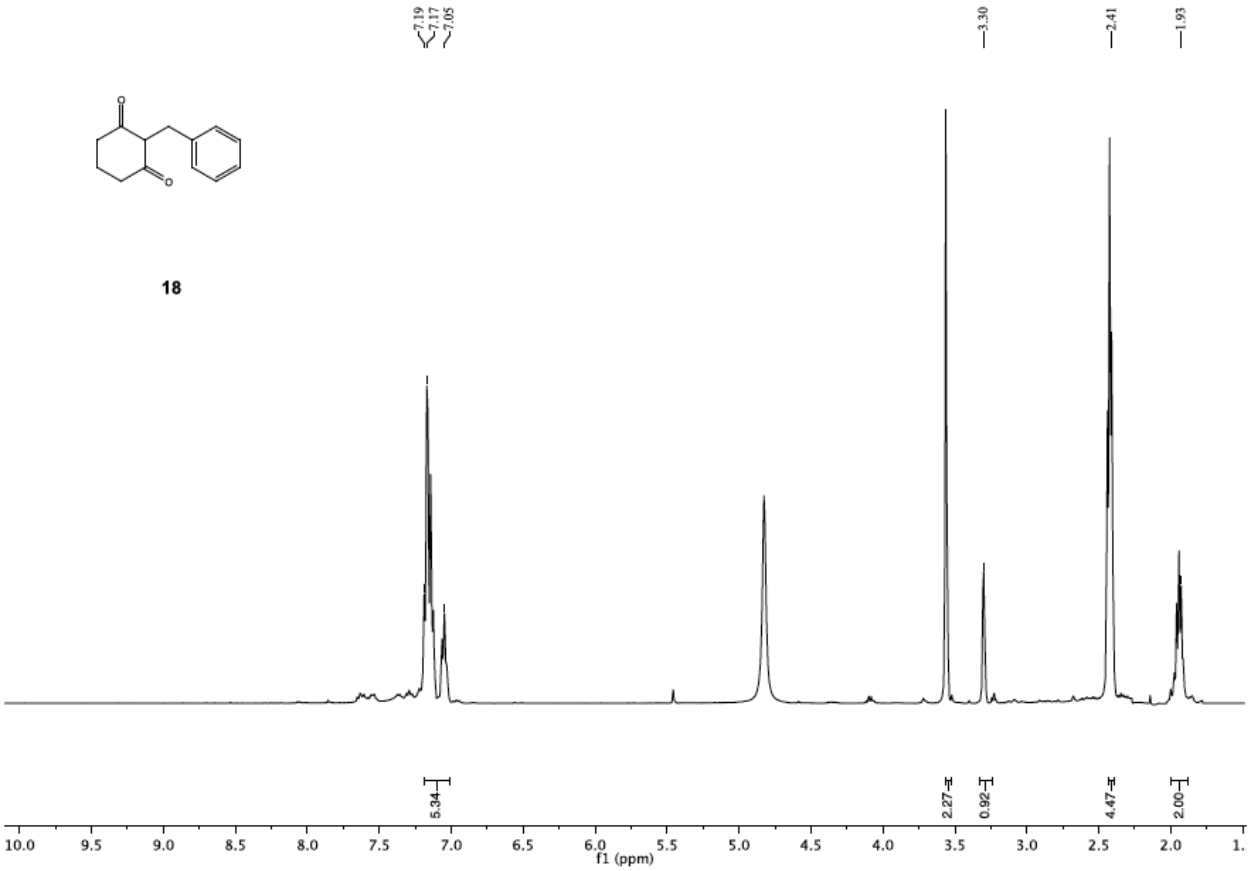
60
50
40
30
20
10
f1 (ppm)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2.41
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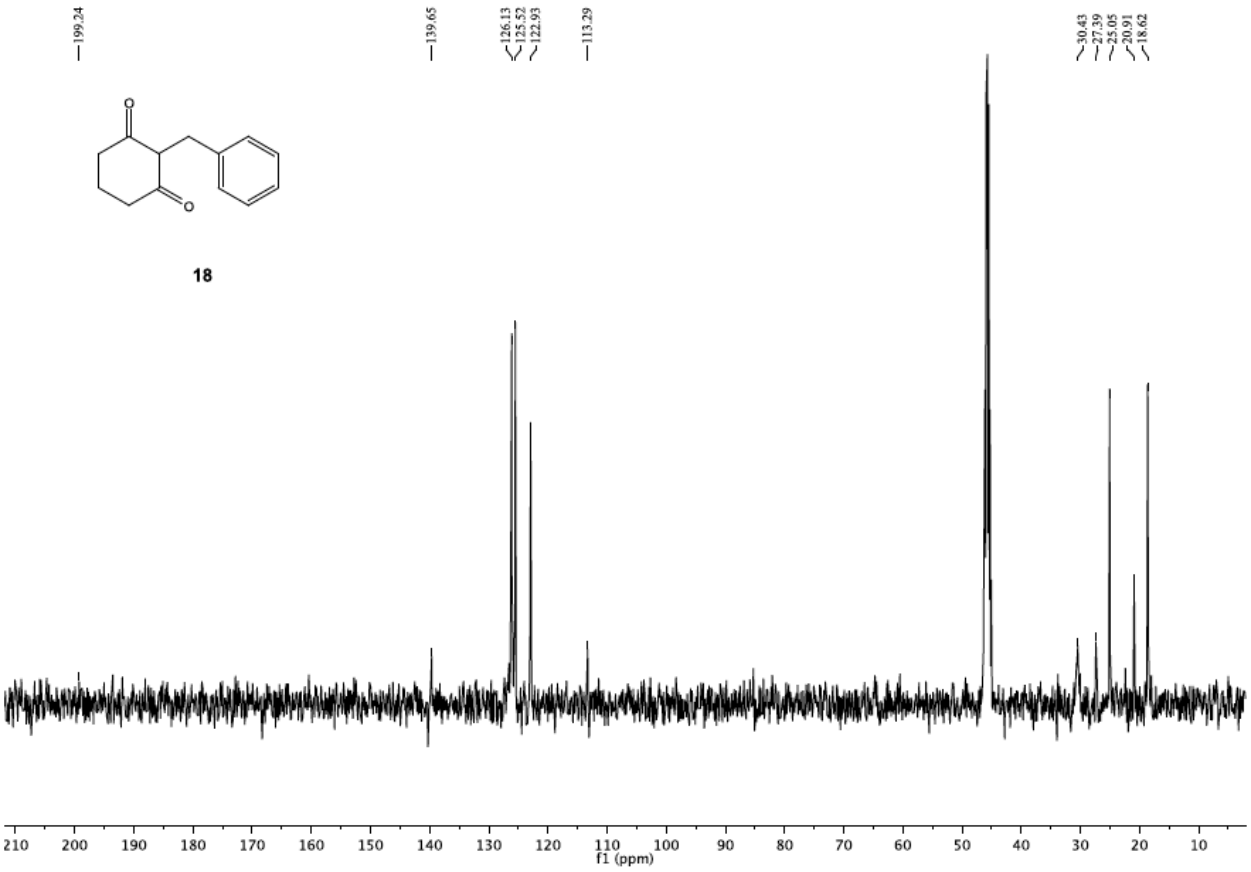
5.34
2.27
0.92
4.47
2.00
f1 (ppm)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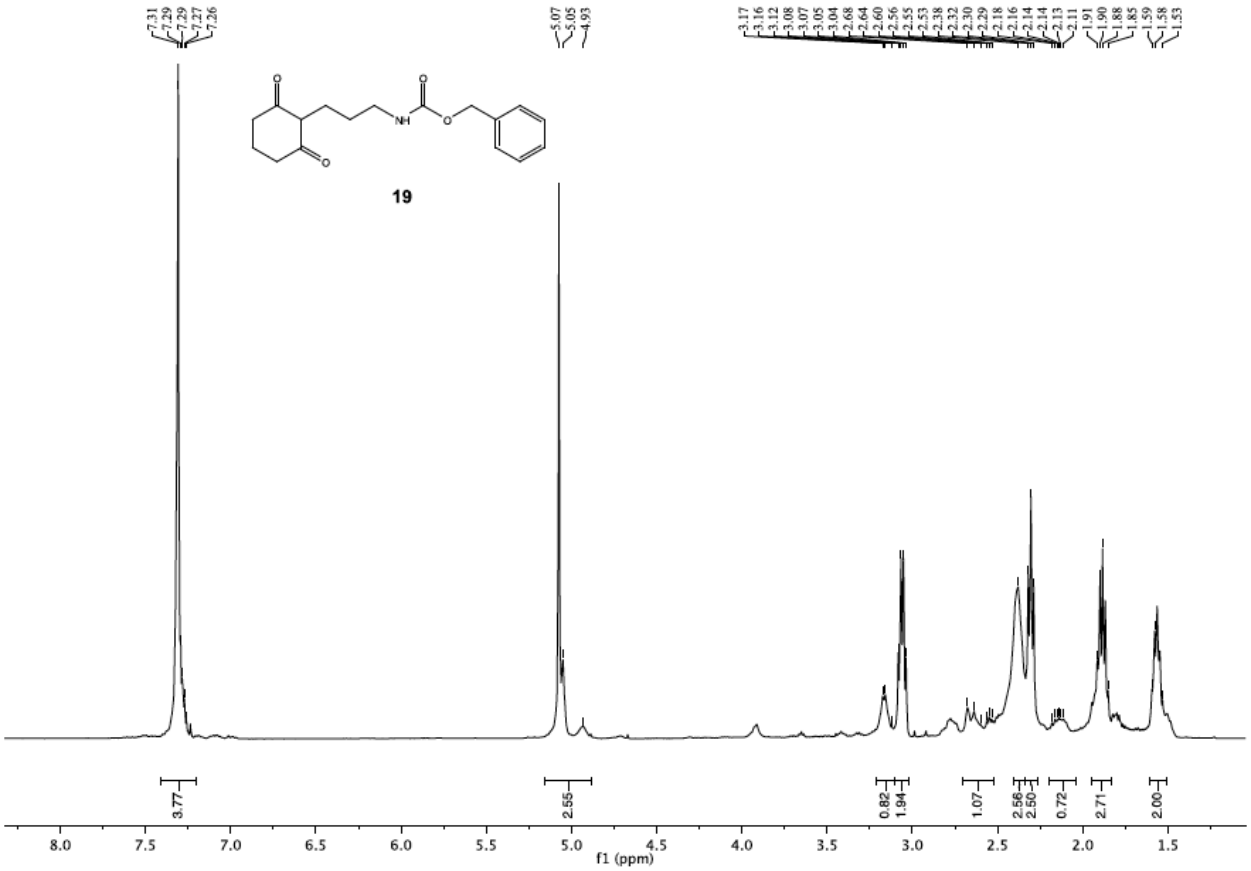
27.39
25.05
20.91
18.62
18
f1 (ppm)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3.16
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3.08
3.07
3.05
3.04
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2.60
2.56
2.55
2.53
2.38
2.32
2.30
2.29
2.18
2.16
2.14
2.14
2.13
2.11
1.91
1.90
1.88
1.85
1.59
1.58
1.53
19
3.77
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1.07
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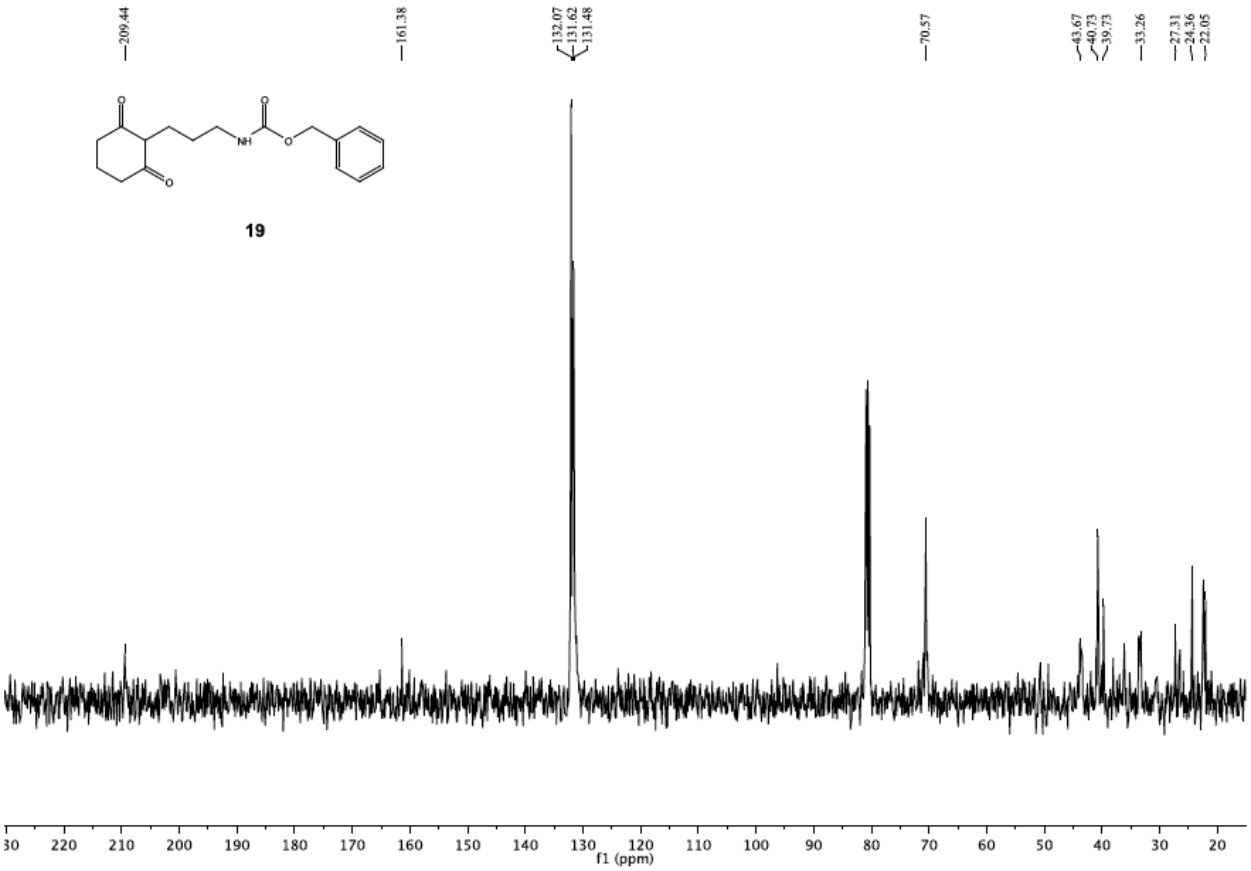
1.5
f1 (ppm)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220
210
200
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70
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30
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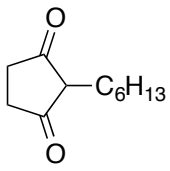
f1 (ppm)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C6H13
O

**20**


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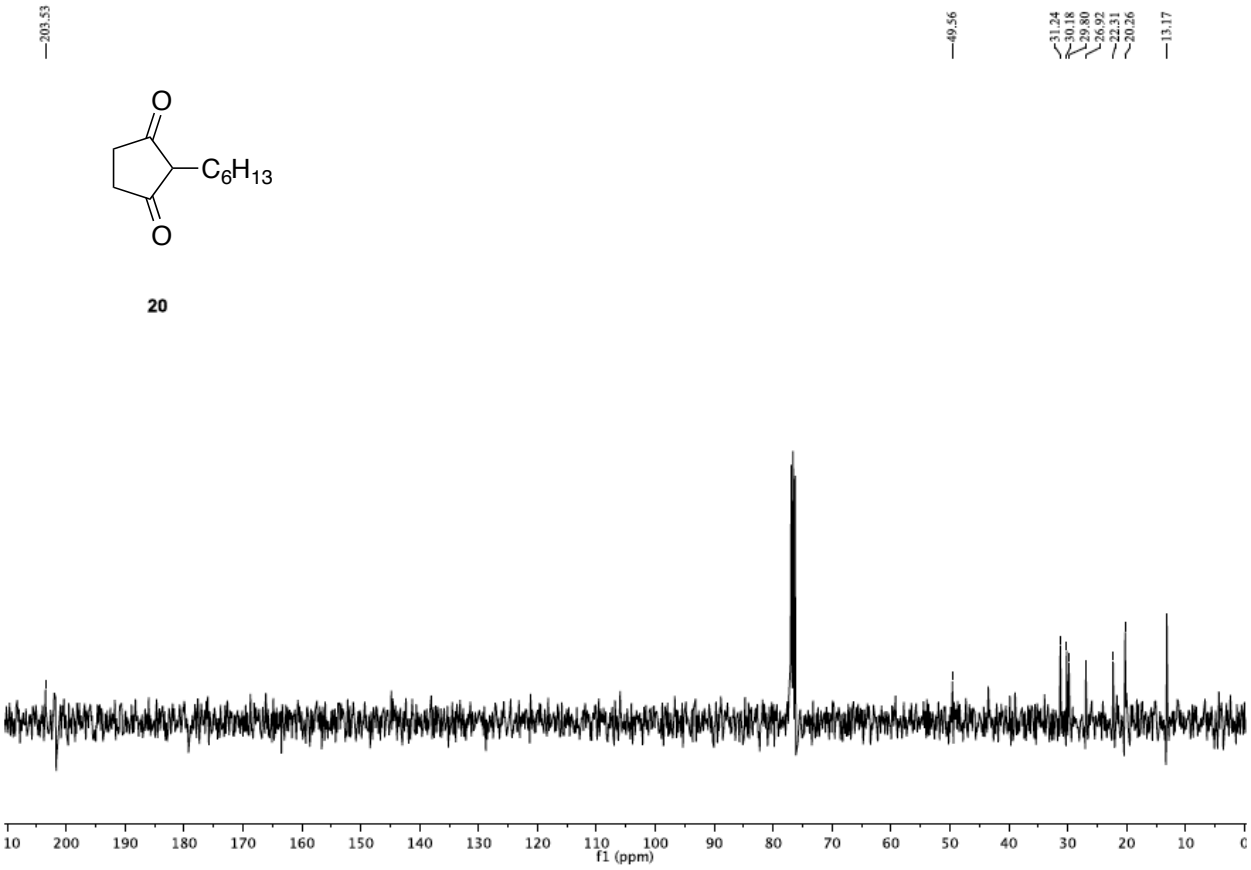
f1 (ppm)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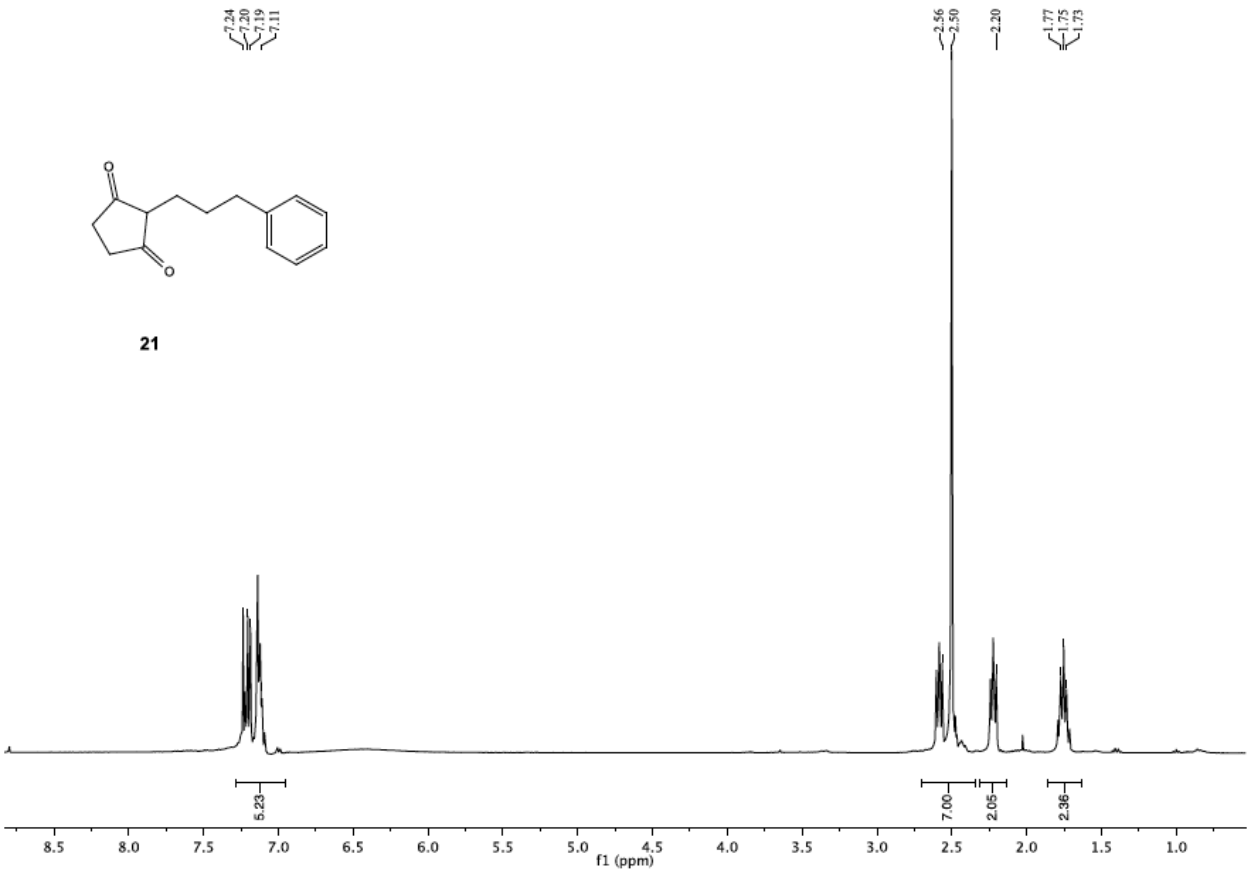
20.26
13.17
f1 (ppm)


O
C6H13
O

**20**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2.36
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7.5
7.0
6.5
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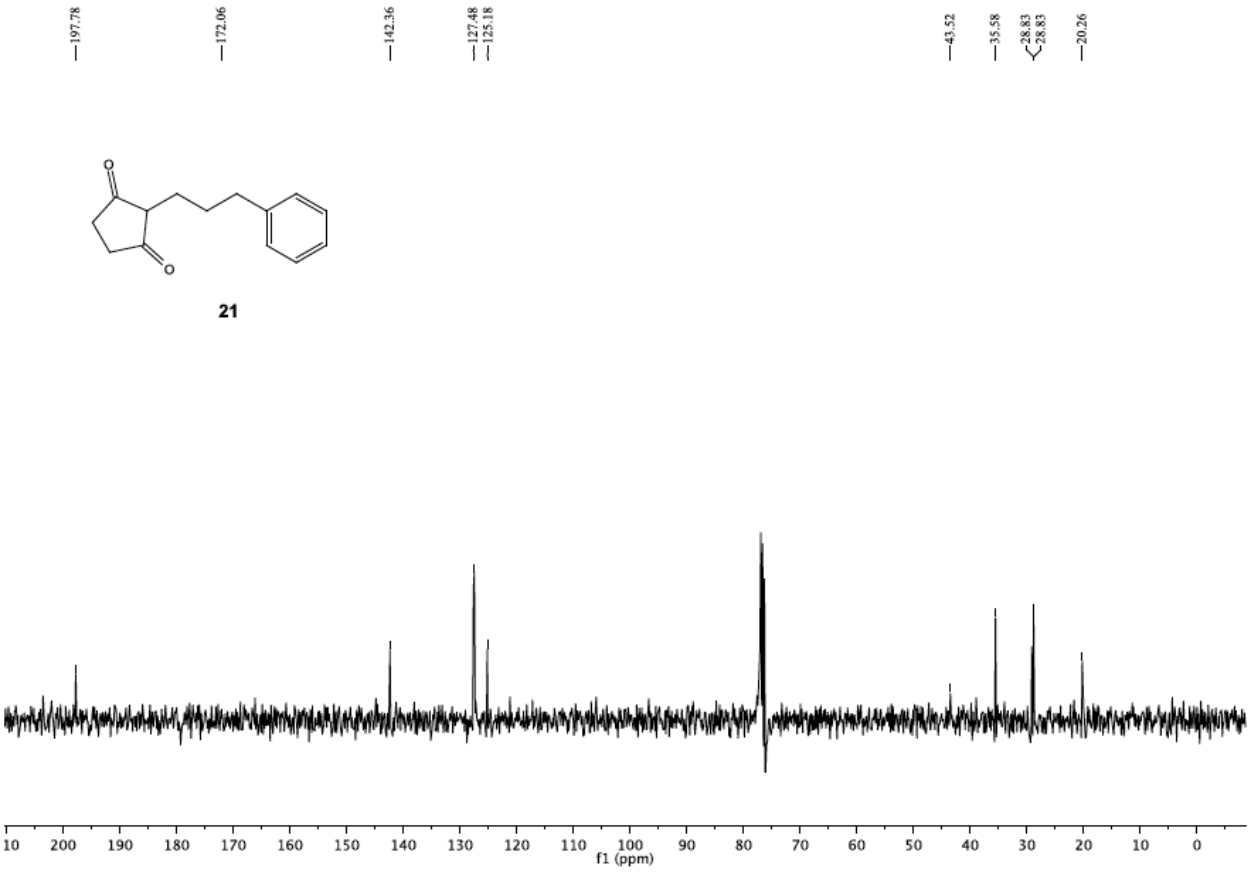
1.0
f1 (ppm)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28.83
20.26
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180
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90
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50
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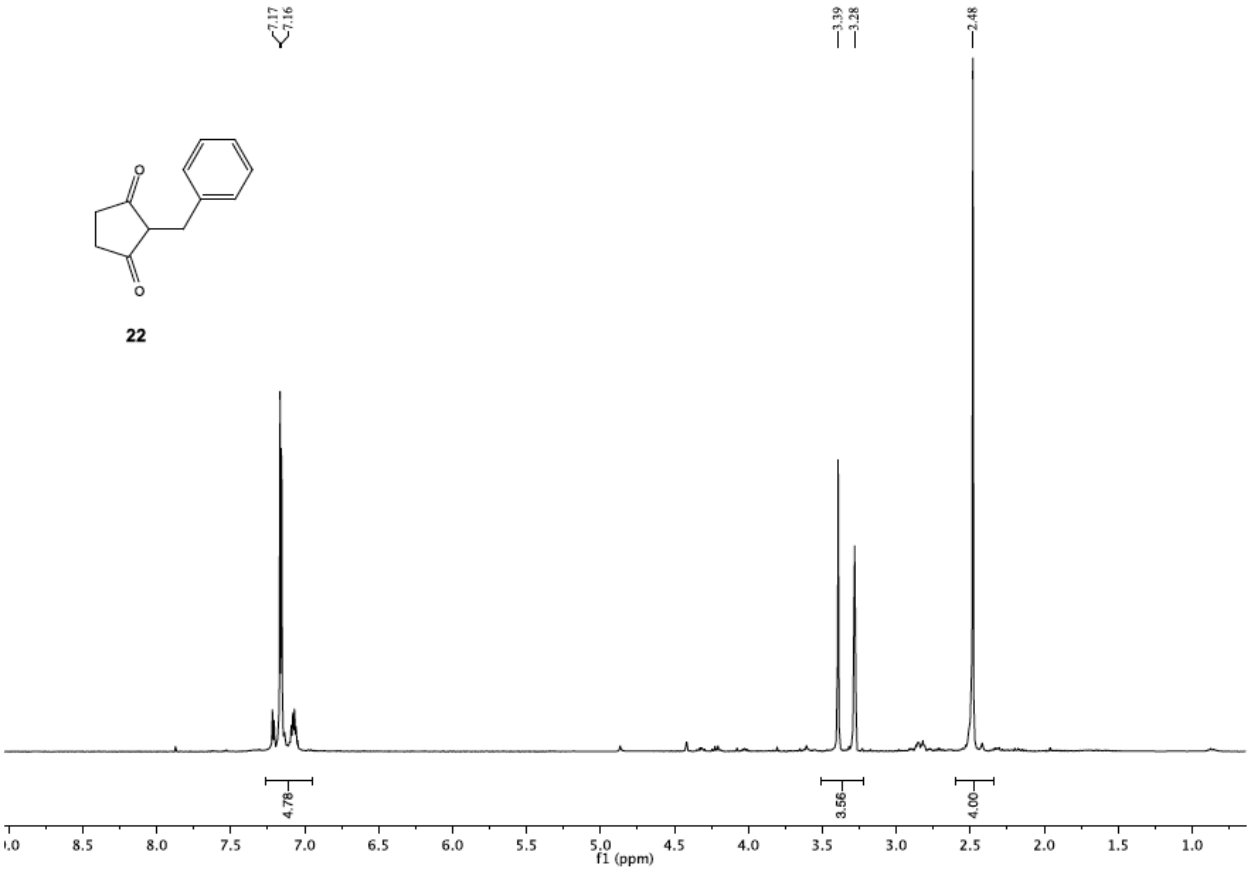
f1 (ppm)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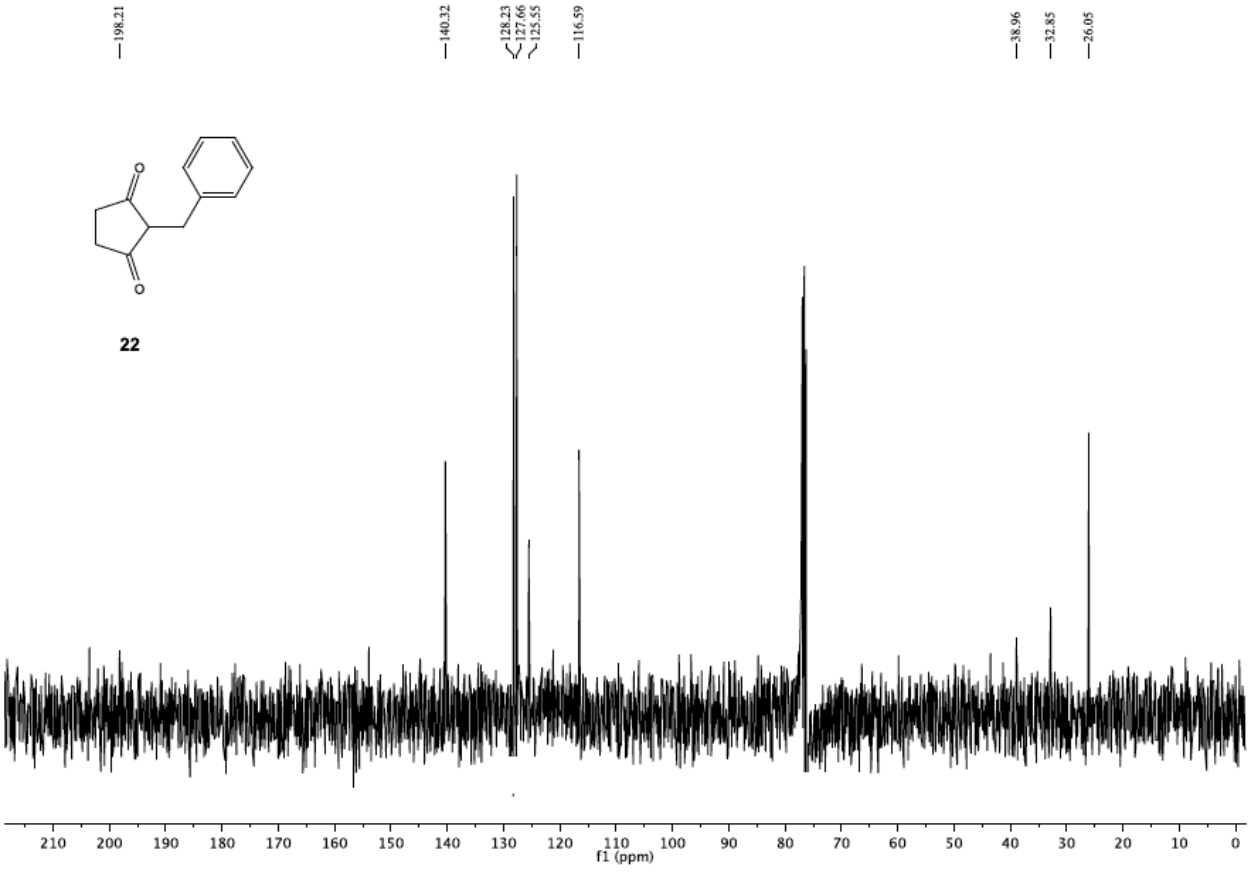
4.00
f1 (ppm)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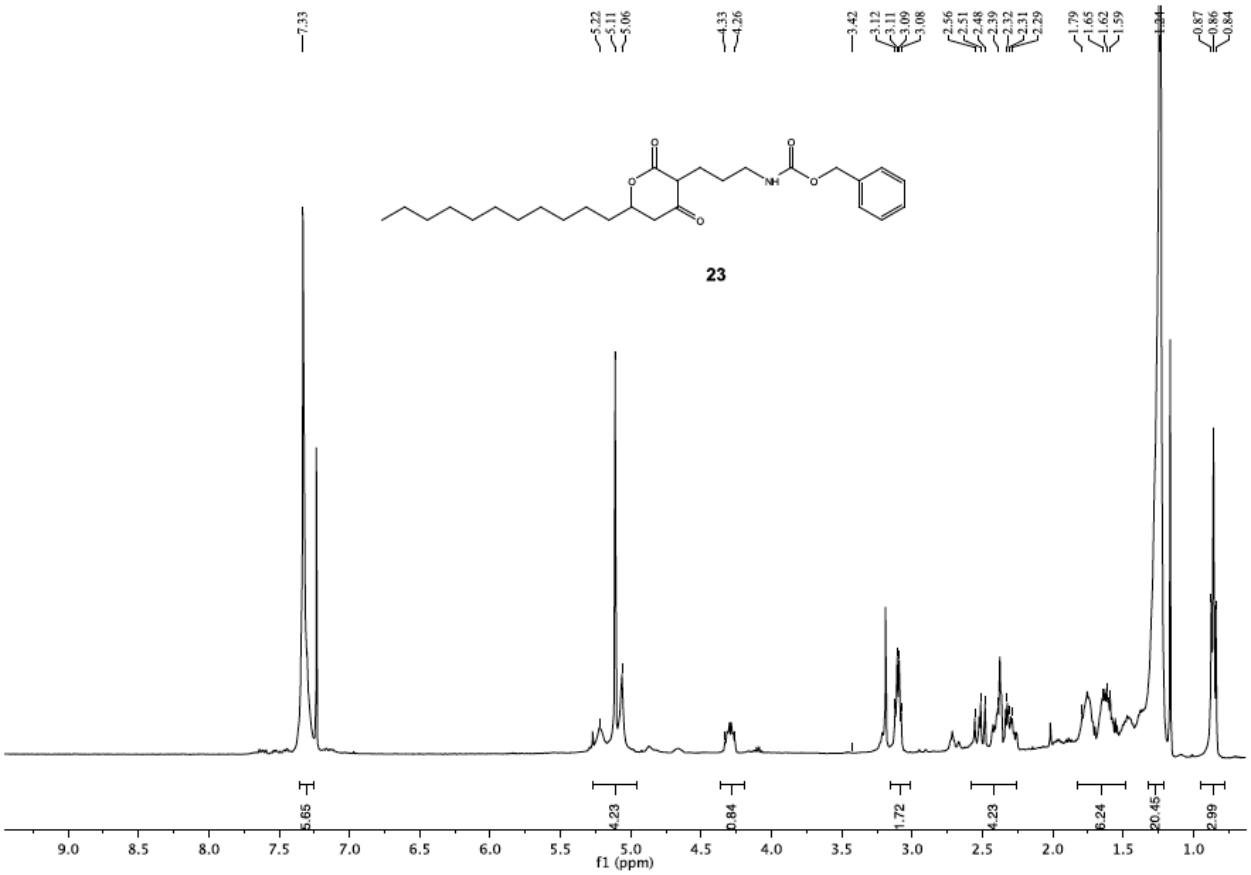
38.96
32.85
26.05
22
f1 (ppm)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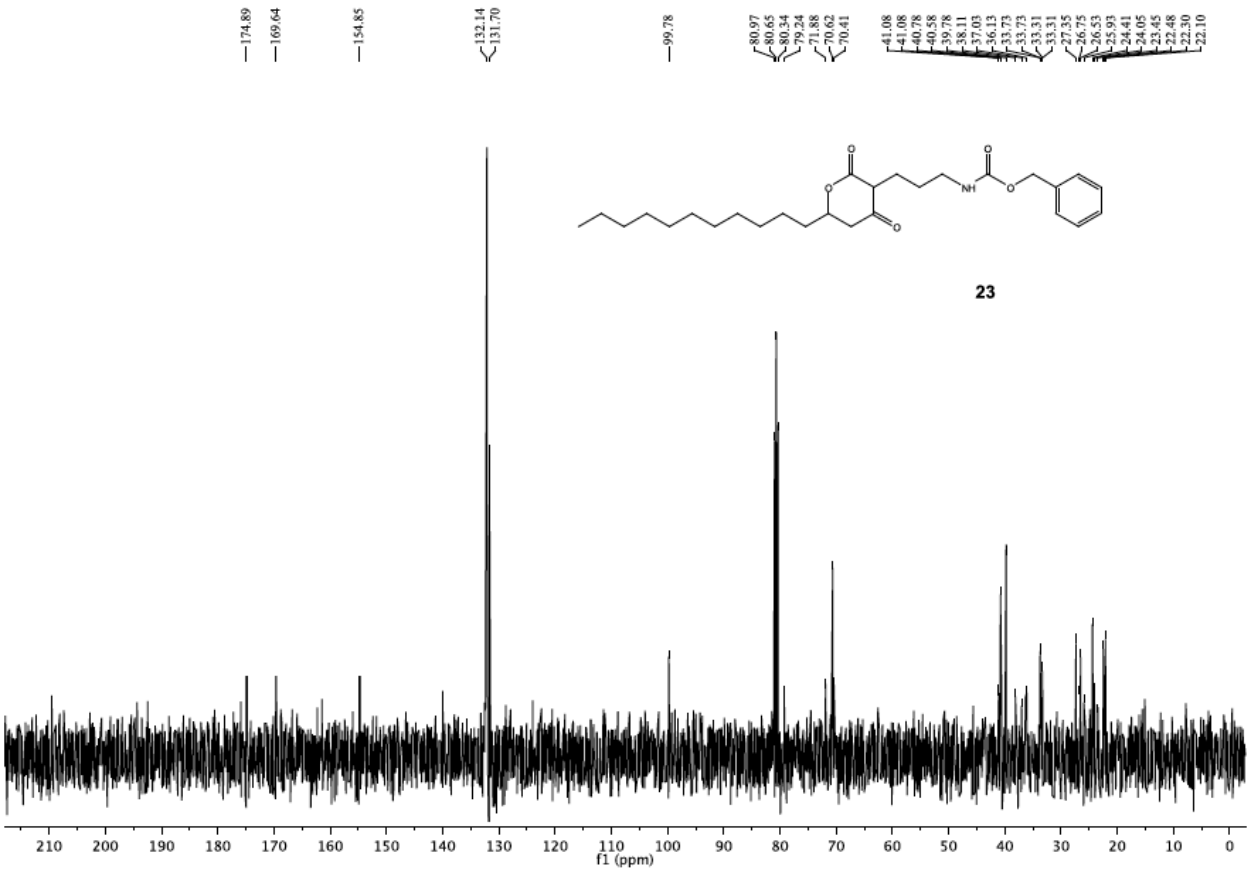
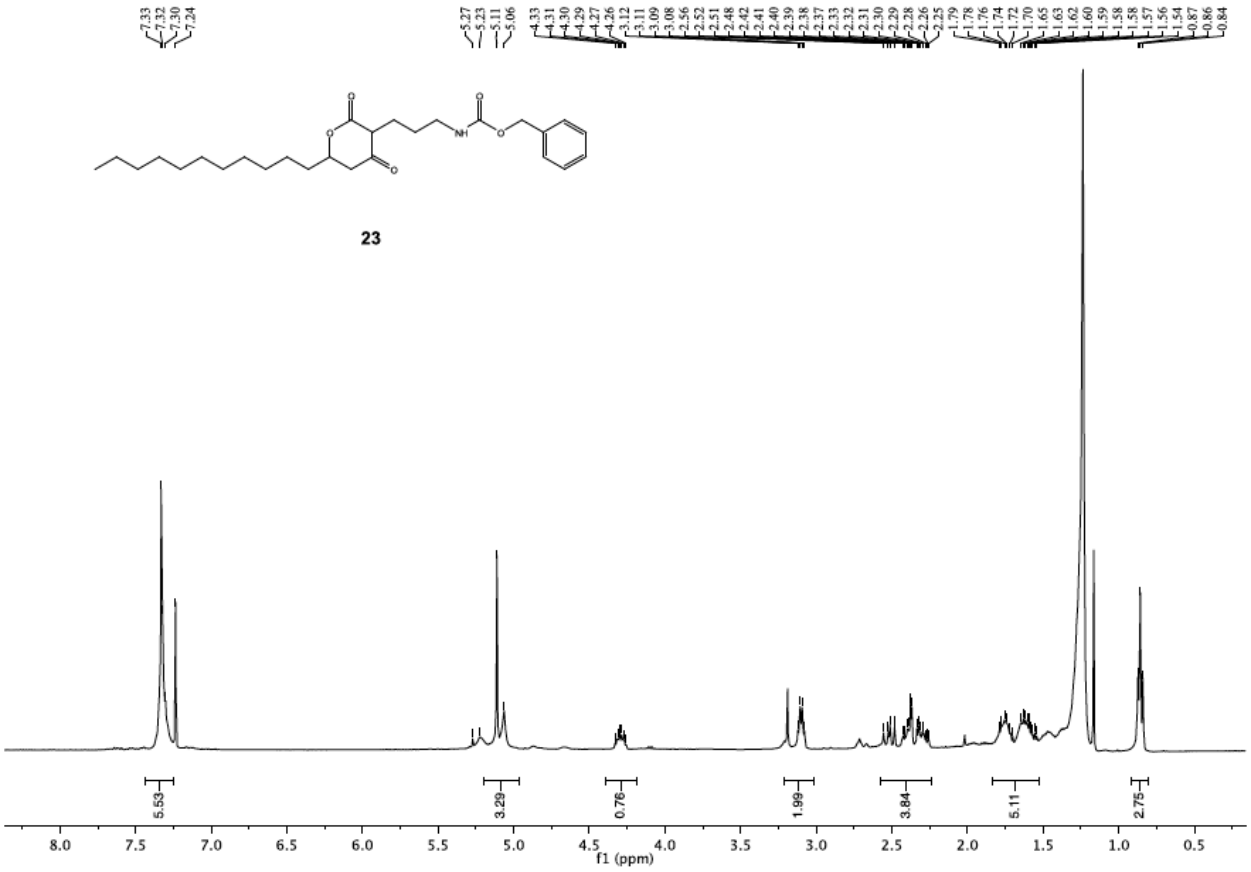
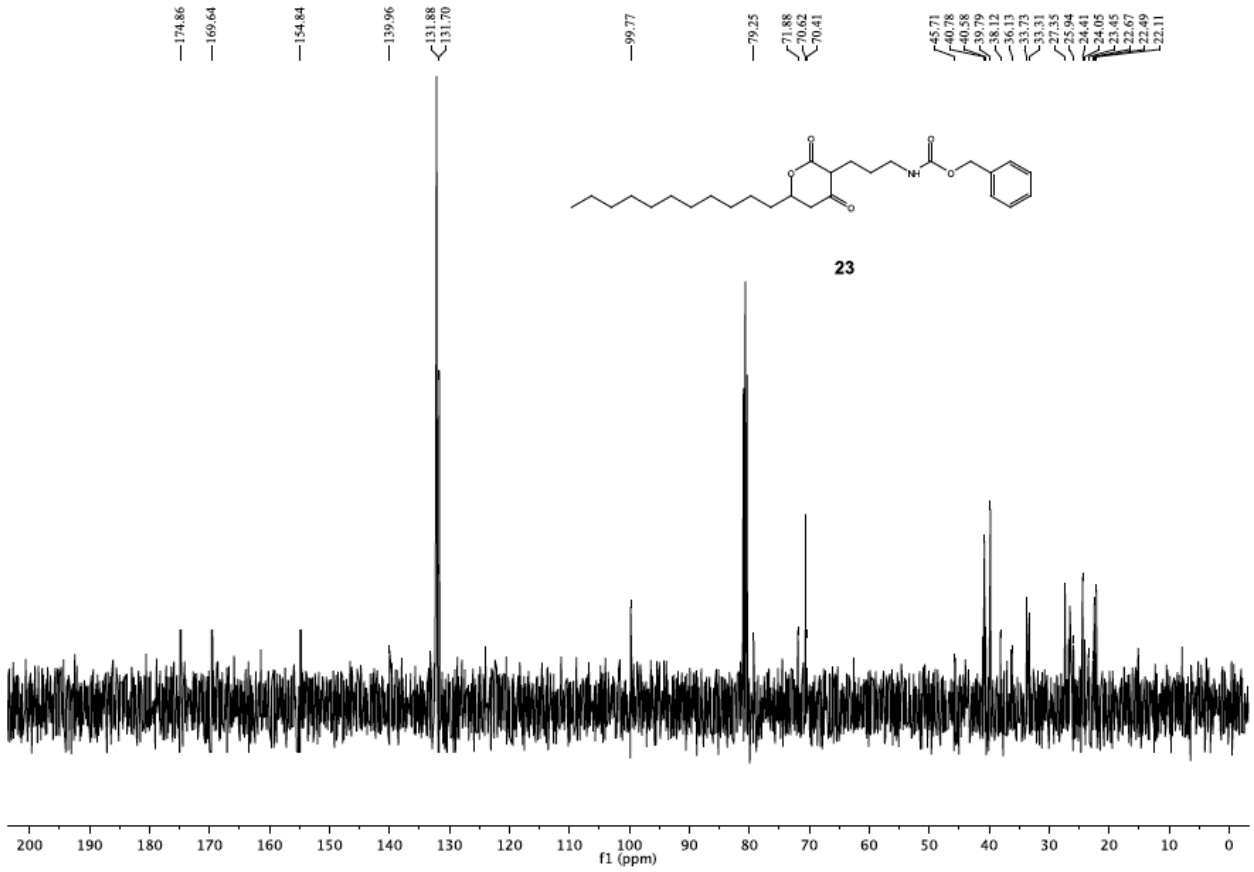
4.33
4.26
3.42
3.12
3.11
3.09
3.08
2.56
2.51
2.48
2.39
2.32
2.31
2.29
1.79
1.65
1.62
1.59
1.24
0.87
0.86
0.84
5.65
4.23
0.84
1.72
4.23
6.24
20.45
2.99
f1 (ppm)

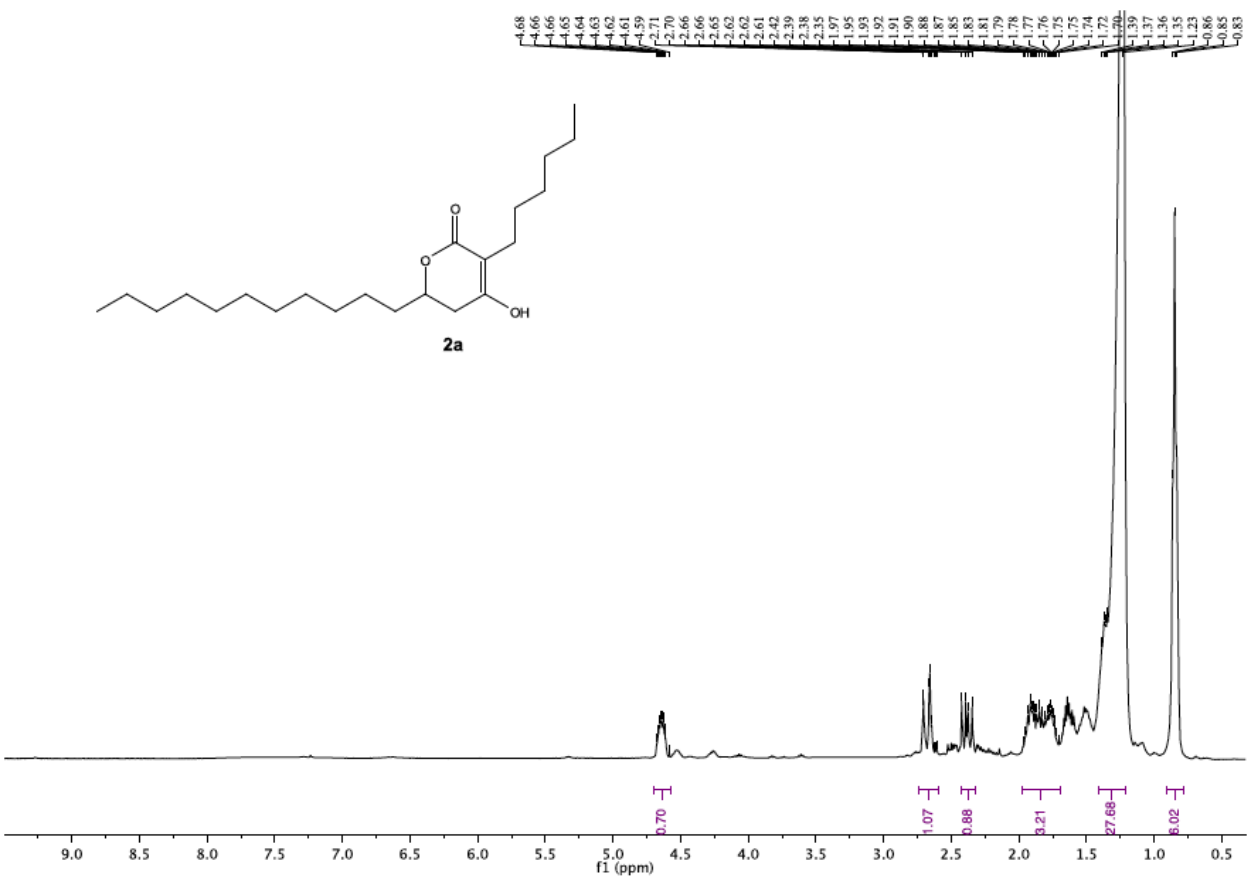
23

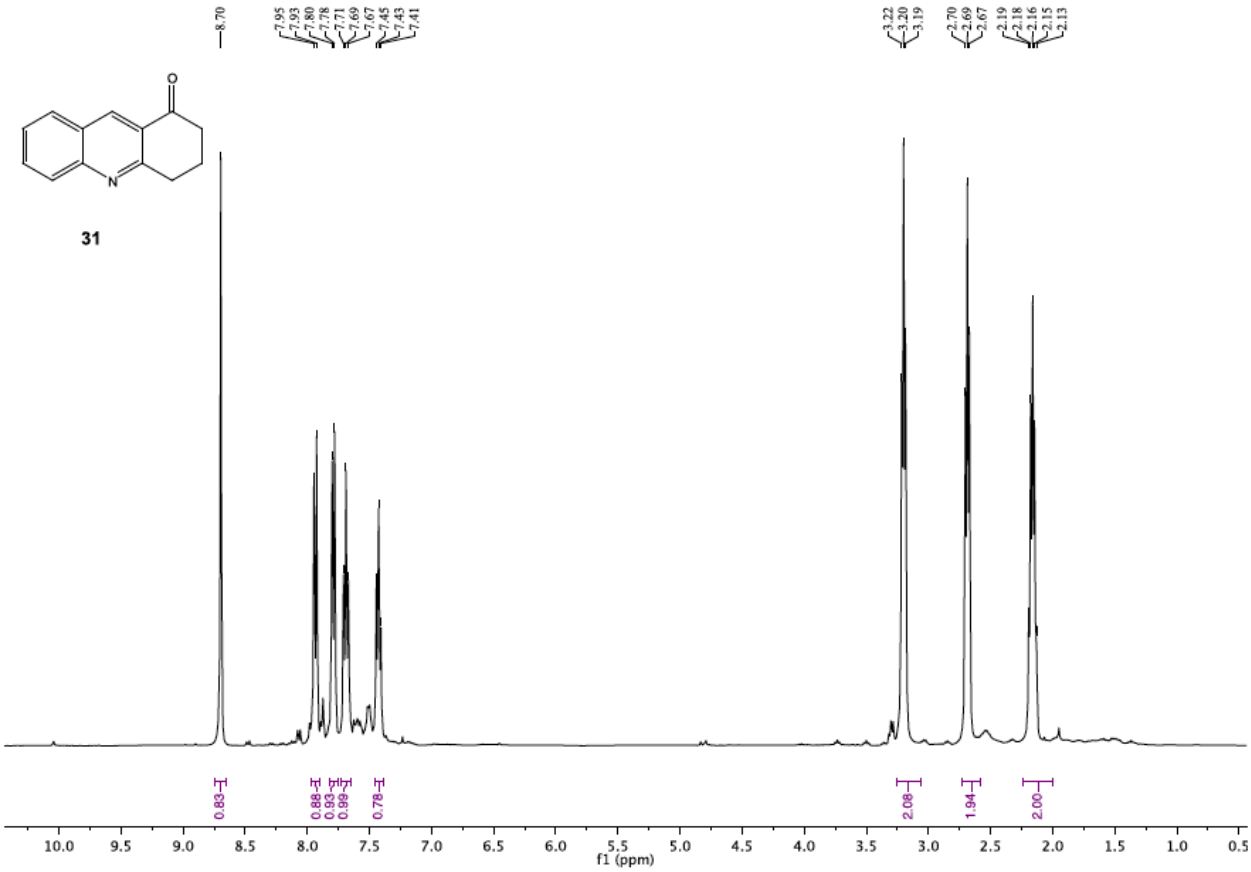

174.89
169.64
154.85
132.14
131.70
99.78
80.97
80.65
80.34
79.24
71.88
70.62
70.41
41.08
41.08
40.78
40.58
39.78
38.11
37.03
36.13
33.73
33.73
33.31
33.31
27.35
26.75
26.53
25.93
24.41
24.05
23.45
22.48
22.30
22.10
f1 (ppm)

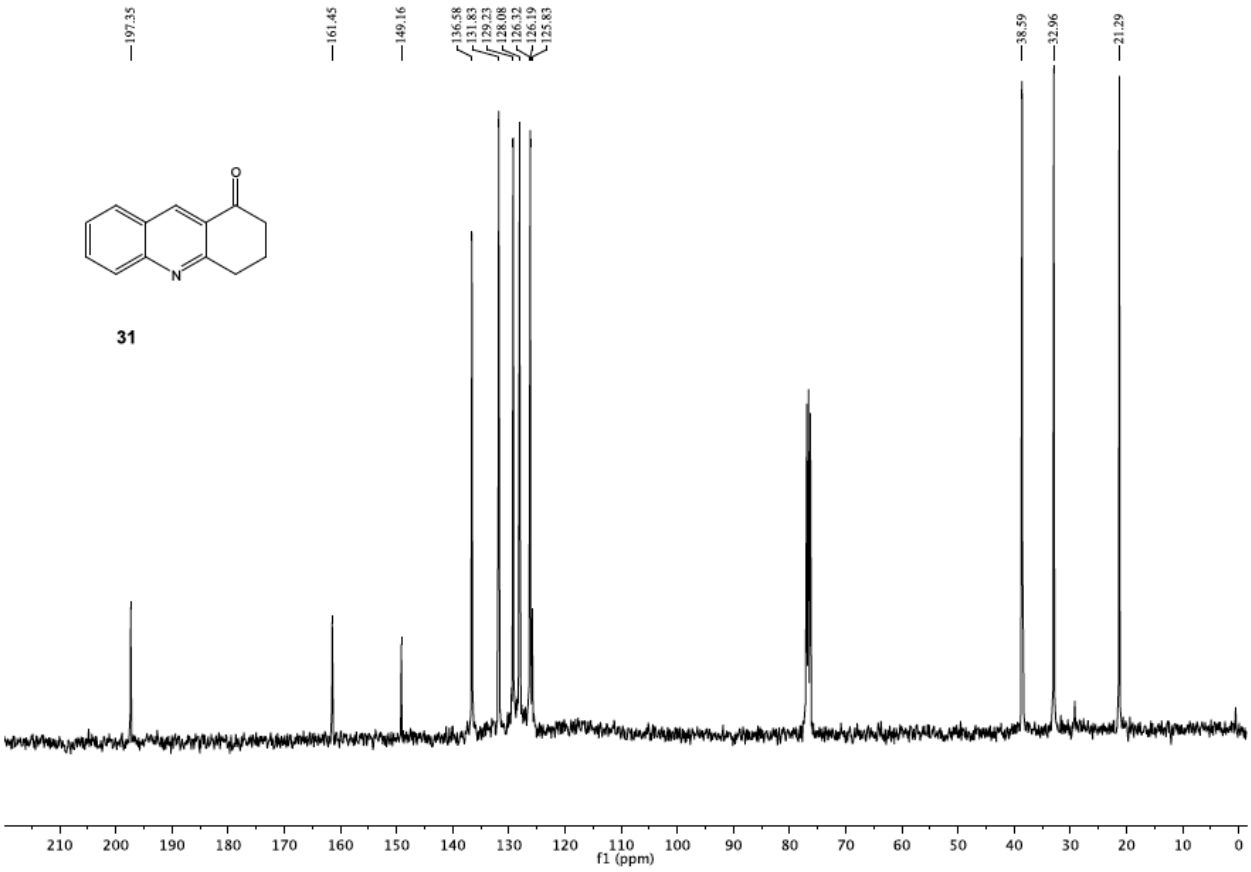
23

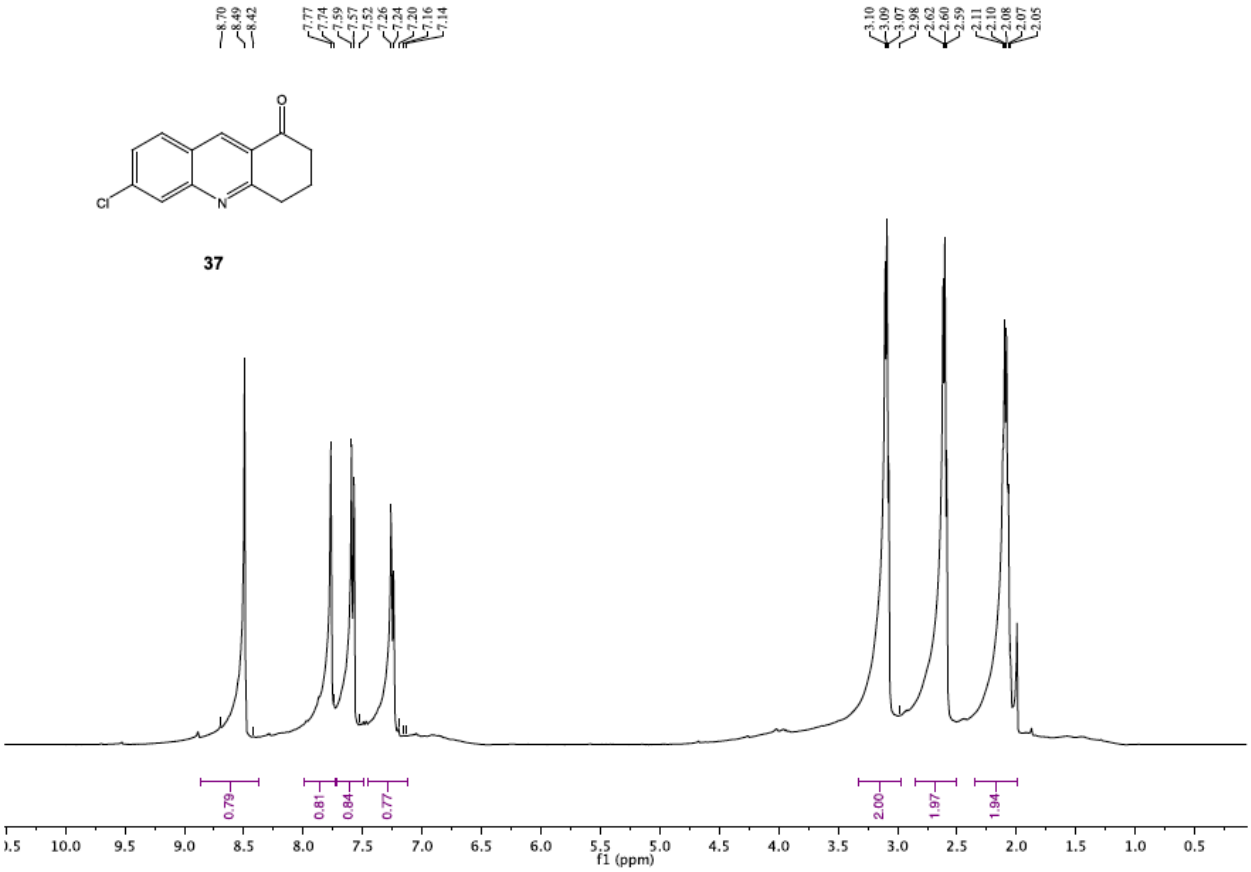

7.33
7.32
7.30
7.24
5.27
5.23
5.11
5.06
4.33
4.31
4.30
4.29
4.27
4.26
3.12
3.11
3.09
3.08
2.56
2.52
2.51
2.48
2.42
2.41
2.40
2.39
2.38
2.37
2.33
2.32
2.31
2.30
2.29
2.28
2.26
2.25
1.79
1.78
1.76
1.74
1.72
1.70
1.65
1.63
1.62
1.60
1.59
1.58
1.58
1.57
1.56
1.54
0.87
0.86
0.84
NH
O
O
O
O
O
23
5.53
3.29
0.76
1.99
3.84
5.11
2.75
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
f1 (ppm)

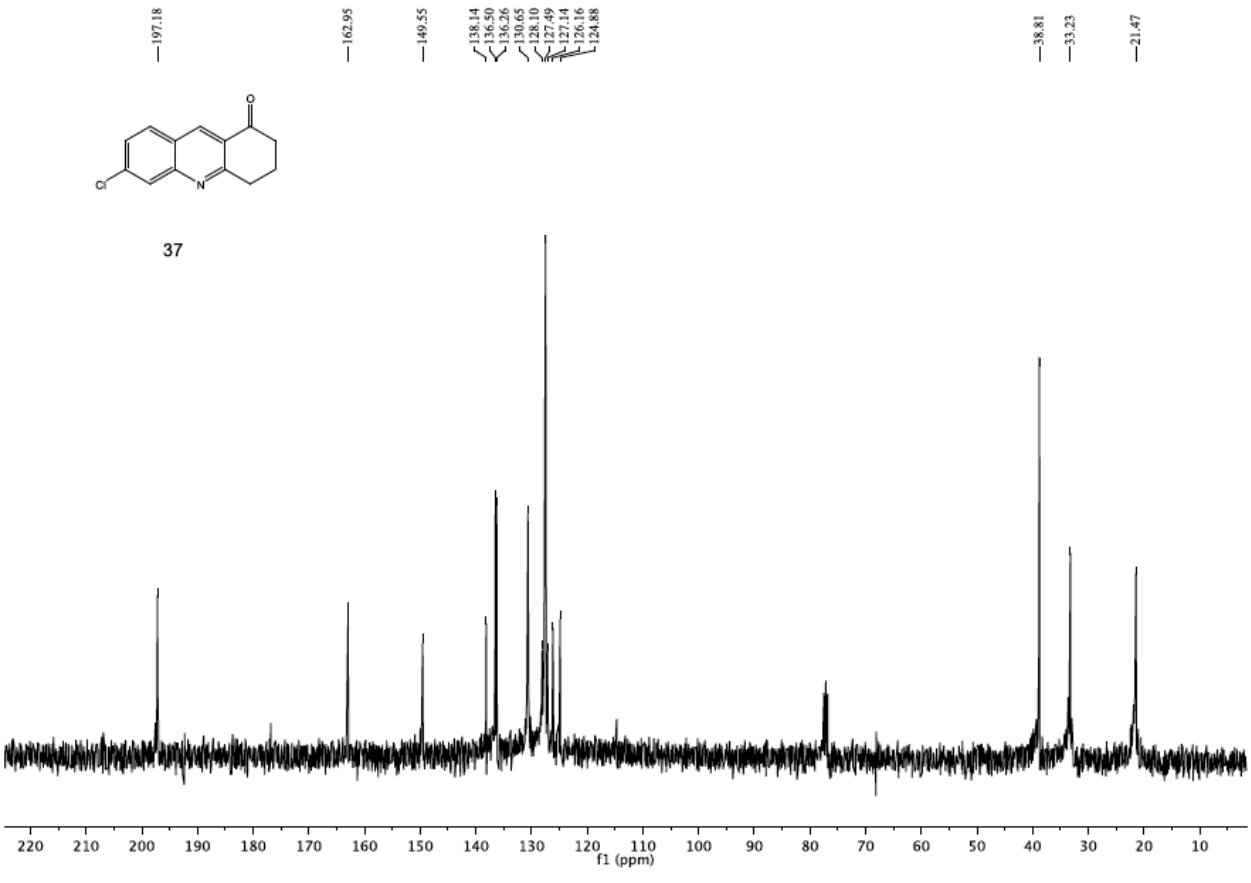

174.86
169.64
154.84
139.96
131.88
131.70
99.77
79.25
71.88
70.62
70.41
45.71
40.78
40.58
39.79
38.12
36.13
33.73
33.31
27.35
25.94
24.41
24.05
23.45
22.67
22.49
22.11
NH
O
O
O
O
O
23
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

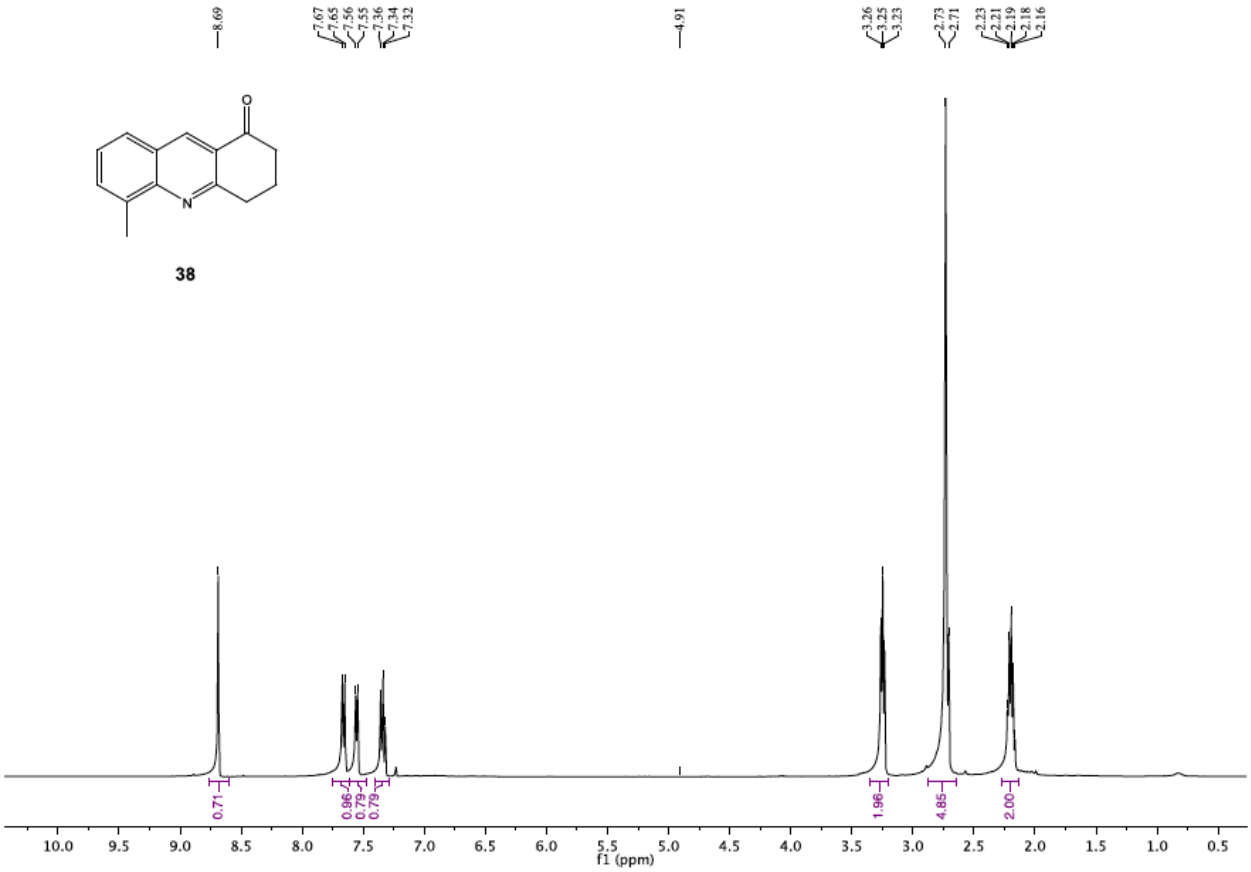

O
O
OH
2a
4.68
4.66
4.66
4.65
4.64
4.63
4.62
4.61
4.59
2.71
2.70
2.66
2.66
2.65
2.62
2.62
2.61
2.42
2.39
2.38
2.35
1.97
1.95
1.93
1.92
1.91
1.90
1.88
1.87
1.85
1.83
1.81
1.79
1.78
1.77
1.76
1.75
1.75
1.74
1.72
1.70
1.39
1.37
1.36
1.35
1.23
0.86
0.85
0.83
0.70
1.07
0.88
3.21
27.68
6.02
9.0
8.5
8.0
7.5
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
f1 (ppm)

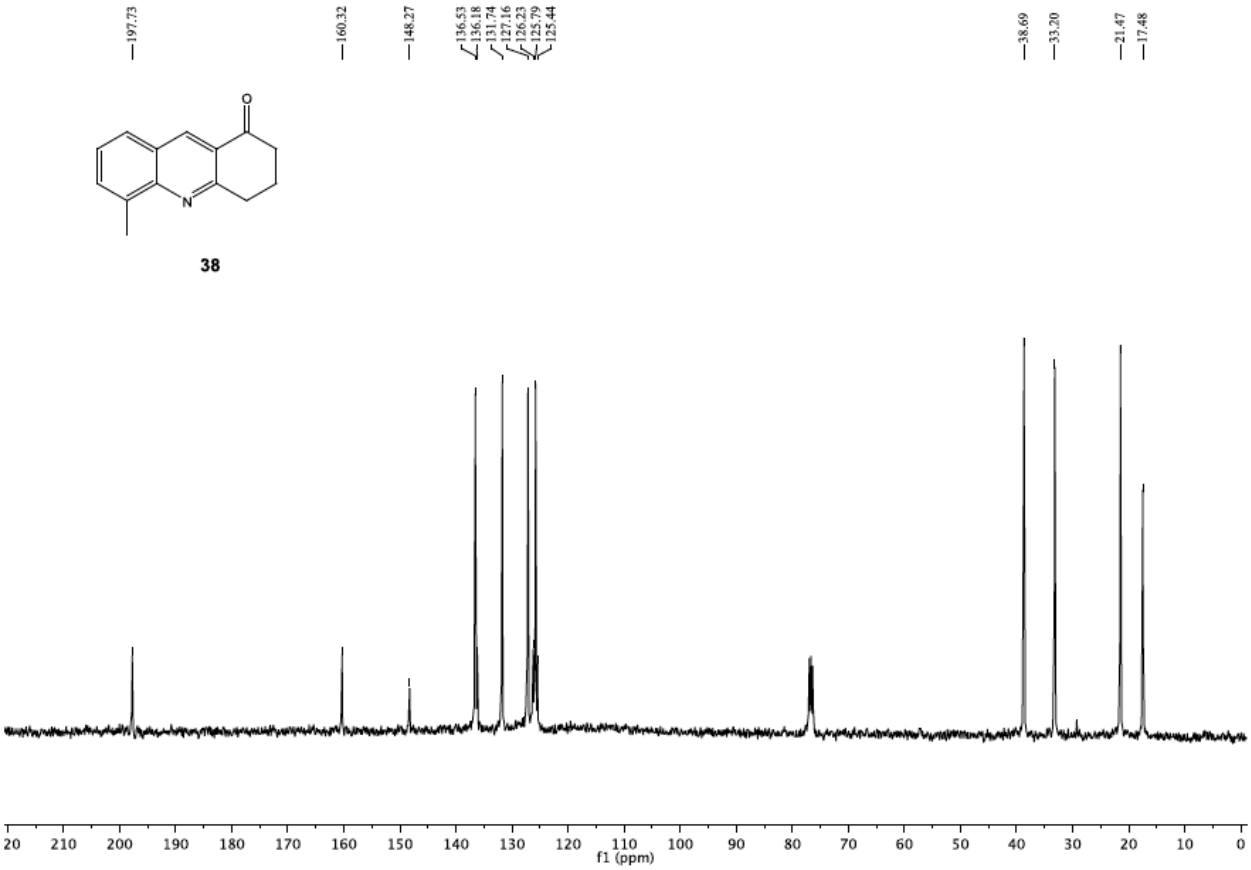

8.70
7.95
7.93
7.80
7.78
7.71
7.69
7.67
7.45
7.43
7.41
3.22
3.20
3.19
2.70
2.69
2.67
2.19
2.18
2.16
2.15
2.13
O
N
31
0.83
0.88
0.93
0.99
0.78
2.08
1.94
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

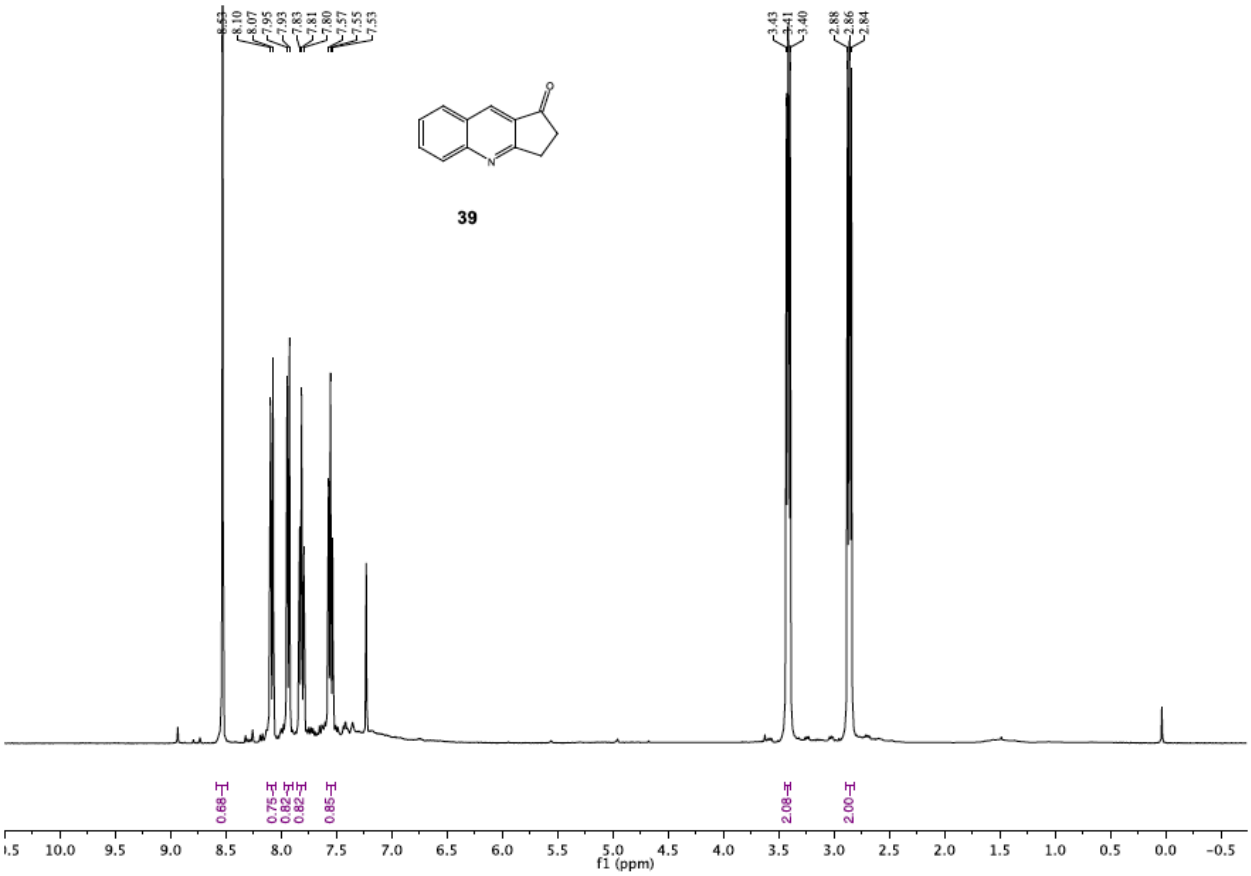

197.35
161.45
149.16
136.58
131.83
129.23
128.08
126.32
126.19
125.83
38.59
32.96
21.29
O
N
31
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.70
8.49
8.42
7.77
7.74
7.59
7.57
7.52
7.26
7.24
7.20
7.16
7.14
3.10
3.09
3.07
2.98
2.62
2.60
2.59
2.11
2.10
2.08
2.07
2.05
O
Cl
N
37
0.79
0.81
0.84
0.77
2.00
1.97
1.94
f1 (ppm)

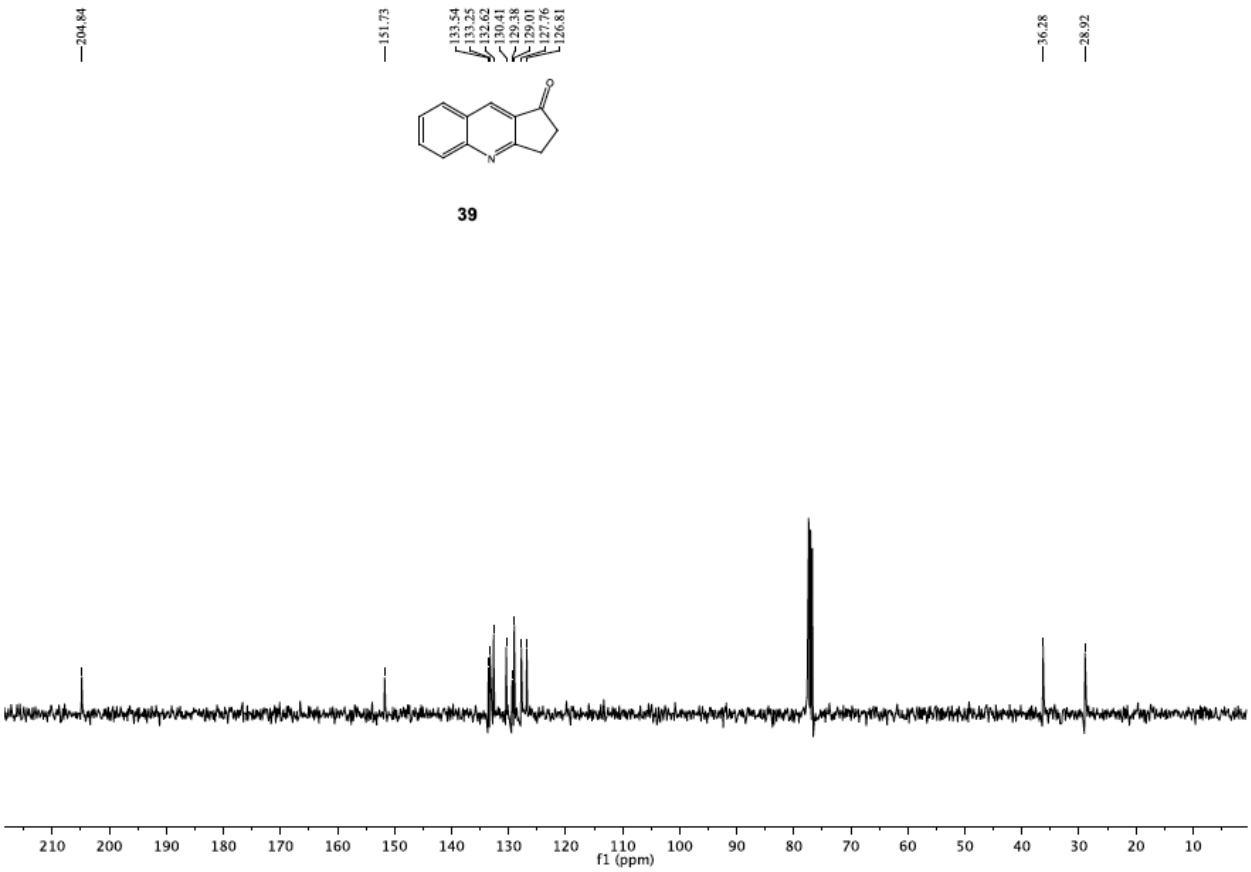

197.18
162.95
149.55
138.14
136.50
136.26
130.65
128.10
127.49
127.14
126.16
124.88
38.81
33.23
21.47
O
Cl
N
37
f1 (ppm)


8.69
7.67
7.65
7.56
7.55
7.36
7.34
7.32
4.91
3.26
3.25
3.23
2.73
2.71
2.23
2.21
2.19
2.18
2.16
O
N
38
0.71
0.96
0.79
0.79
1.96
4.85
2.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

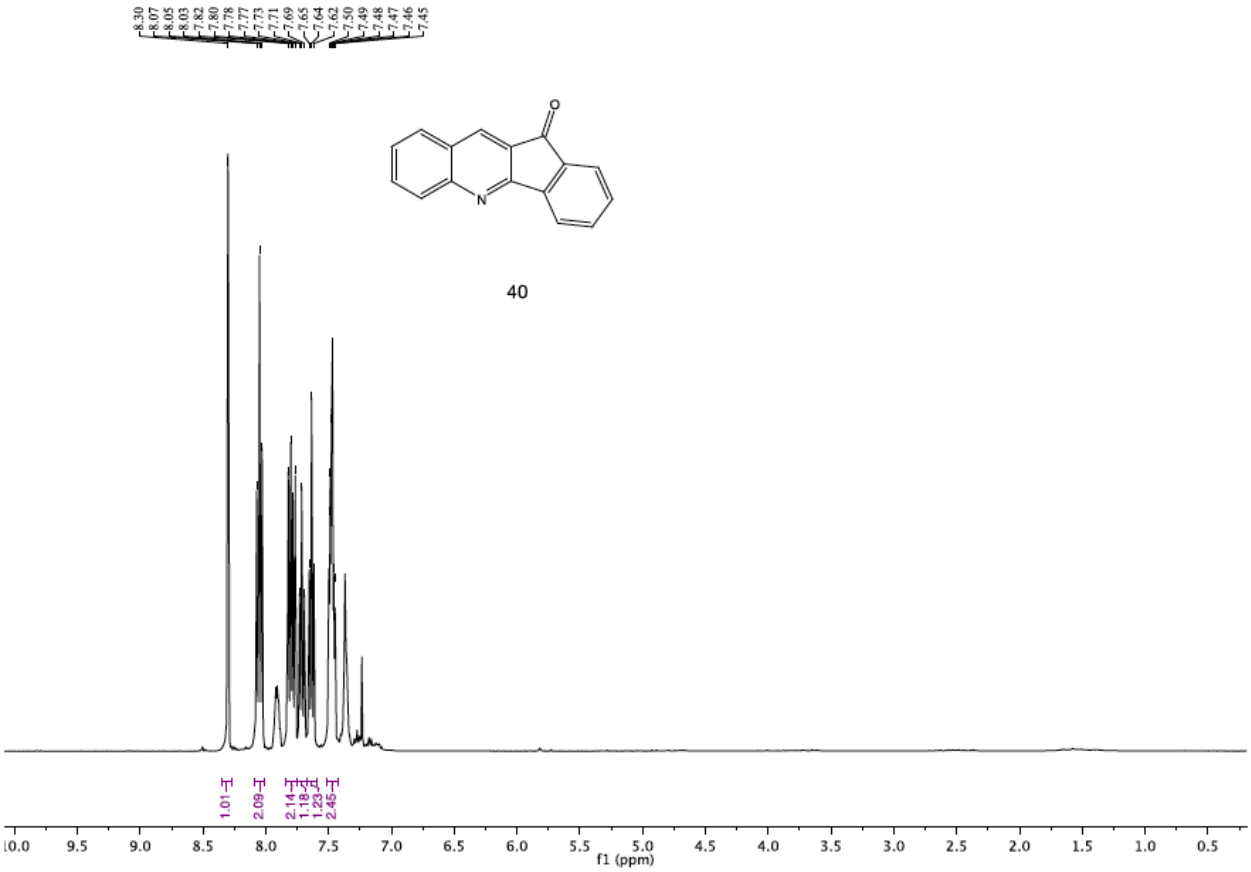

197.73
160.32
148.27
136.53
136.18
131.74
127.16
126.23
125.79
125.44
38.69
33.20
21.47
17.48
O
N
38
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

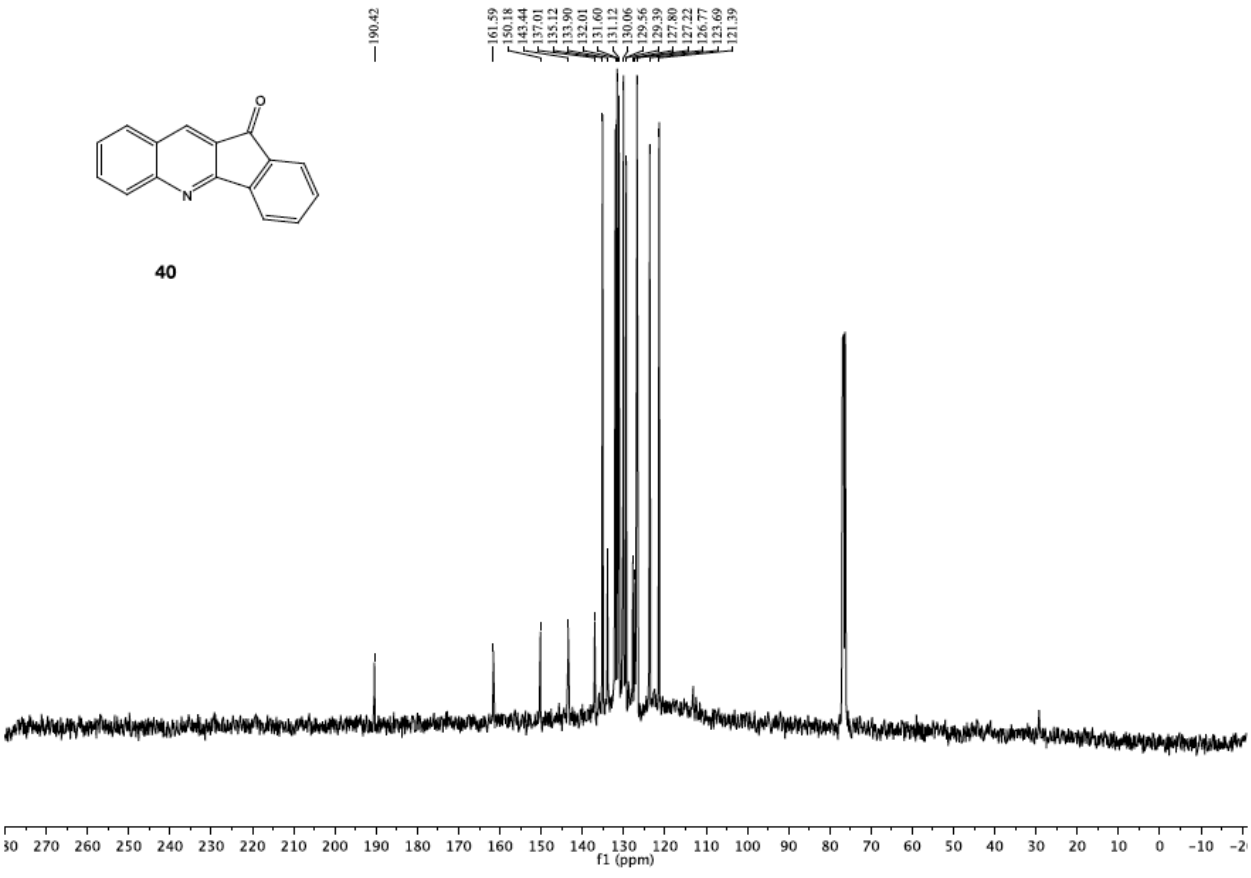

8.63
8.10
8.07
7.95
7.93
7.83
7.81
7.80
7.57
7.55
7.53
3.43
3.41
3.40
2.88
2.86
2.84
O
N
0.68
0.75
0.82
0.82
0.85
2.08
2.00
f1 (ppm)

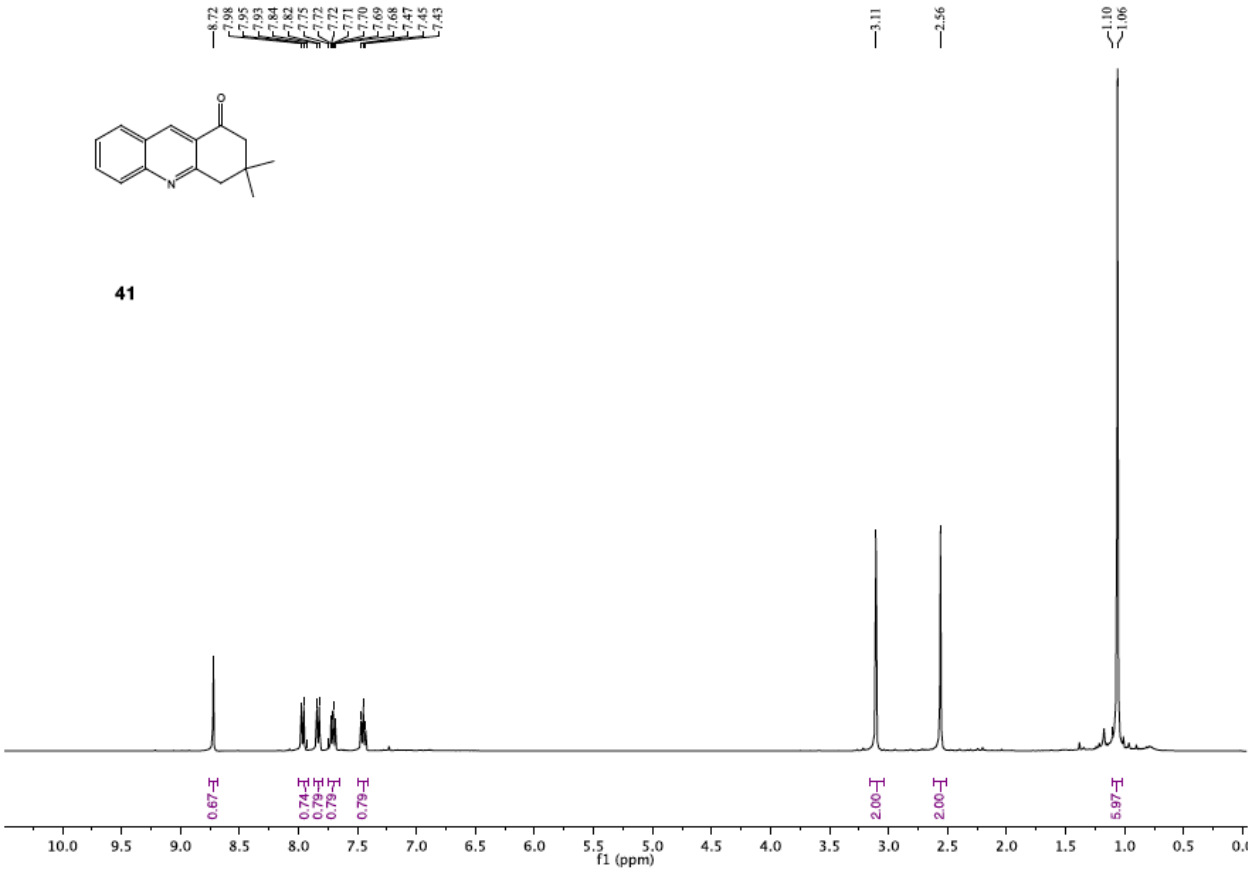
39

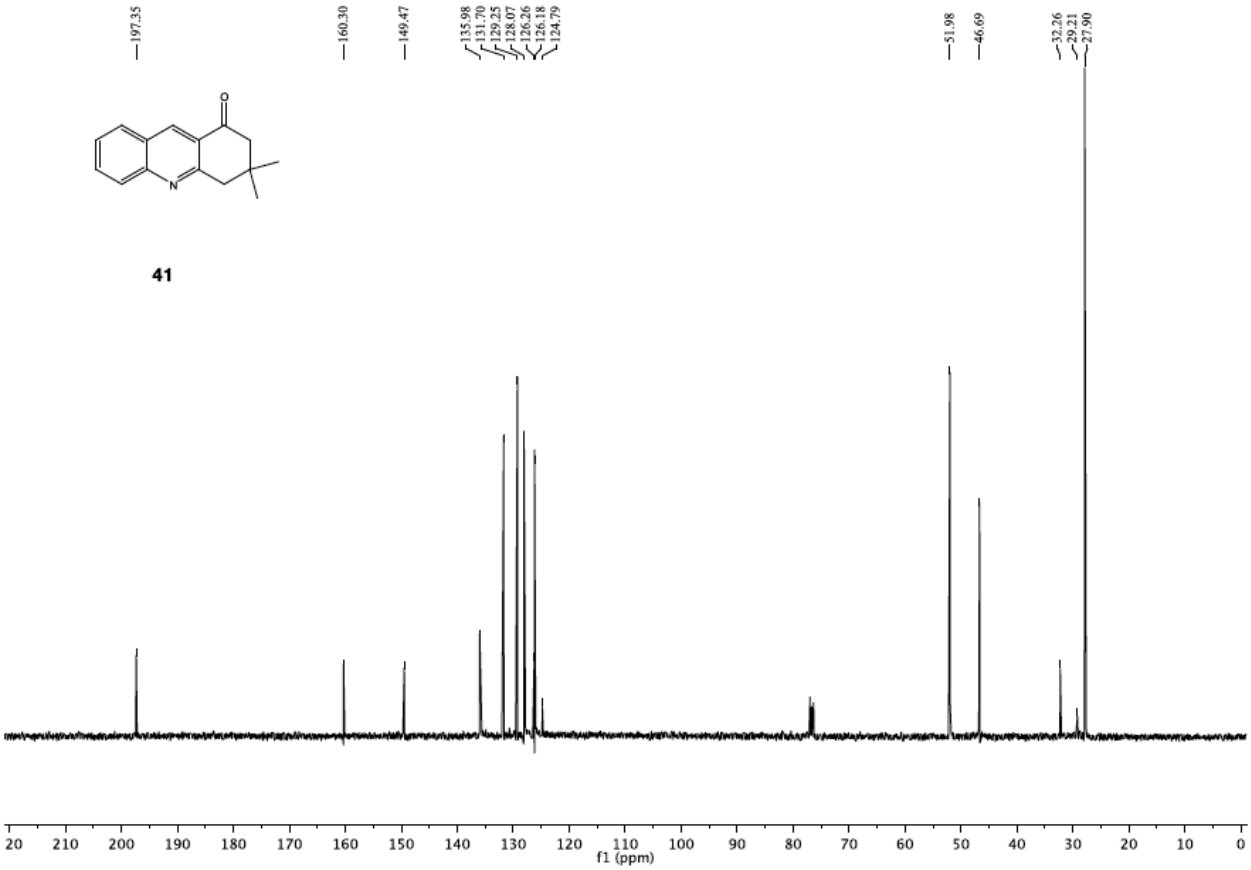

204.84
151.73
133.54
133.25
132.62
130.41
129.38
129.01
127.76
126.81
36.28
28.92
O
N
f1 (ppm)

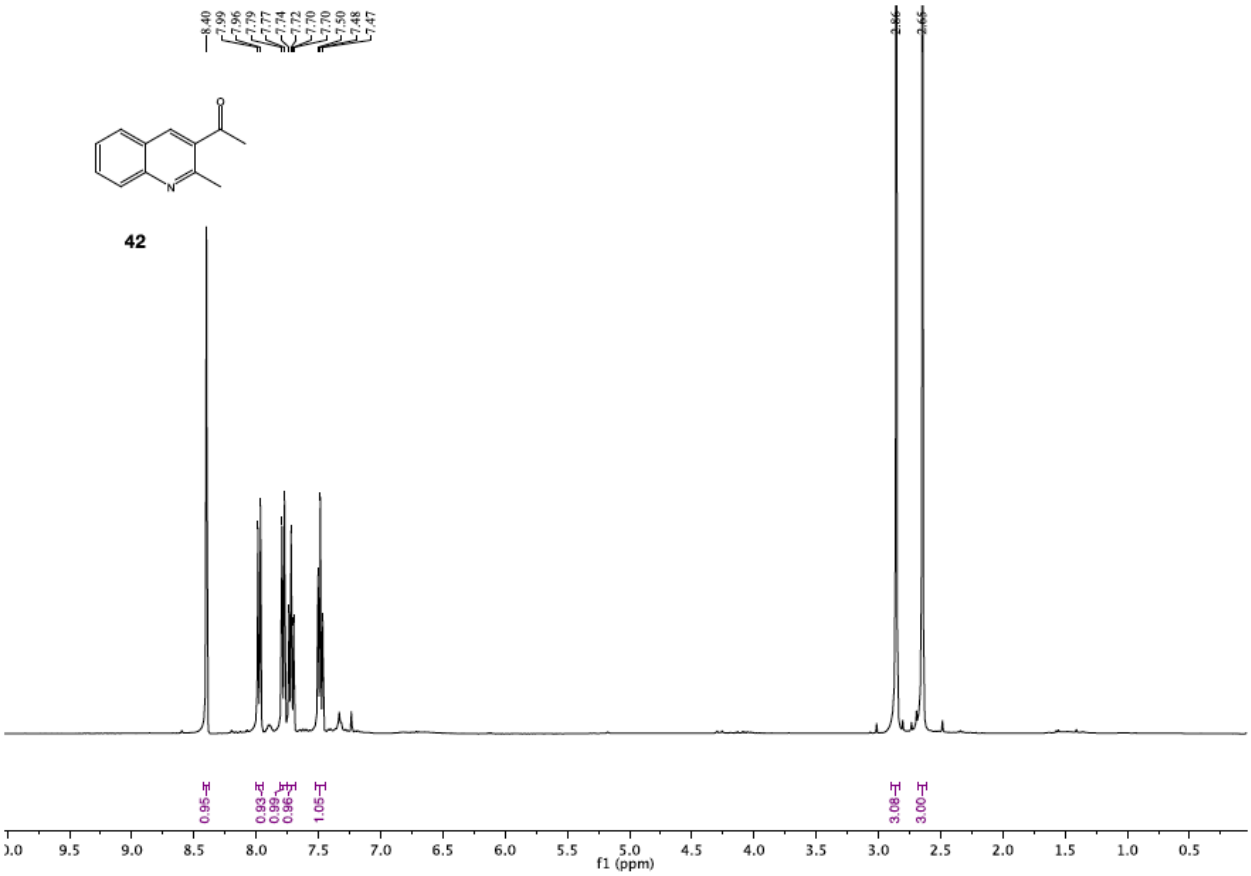
39


8.30
8.07
8.05
8.03
7.82
7.80
7.78
7.77
7.73
7.71
7.69
7.65
7.64
7.62
7.50
7.49
7.48
7.47
7.46
7.45
O
N
40
1.01
2.09
2.14
1.18
1.23
2.45
f1 (ppm)


190.42
161.59
150.18
143.44
137.01
135.12
133.90
132.01
131.60
131.12
130.06
129.56
129.39
127.80
127.22
126.77
123.69
121.39
O
N
40
f1 (ppm)


8.72
7.98
7.95
7.93
7.84
7.82
7.75
7.72
7.72
7.71
7.70
7.69
7.68
7.47
7.45
7.43
3.11
2.56
1.10
1.06
O
N
41
0.67
0.74
0.79
0.79
0.79
2.00
2.00
5.97
f1 (ppm)


197.35
160.30
149.47
135.98
131.70
129.25
128.07
126.26
126.18
124.79
51.98
46.69
32.26
29.21
27.90
O
N
41
f1 (ppm)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7.99
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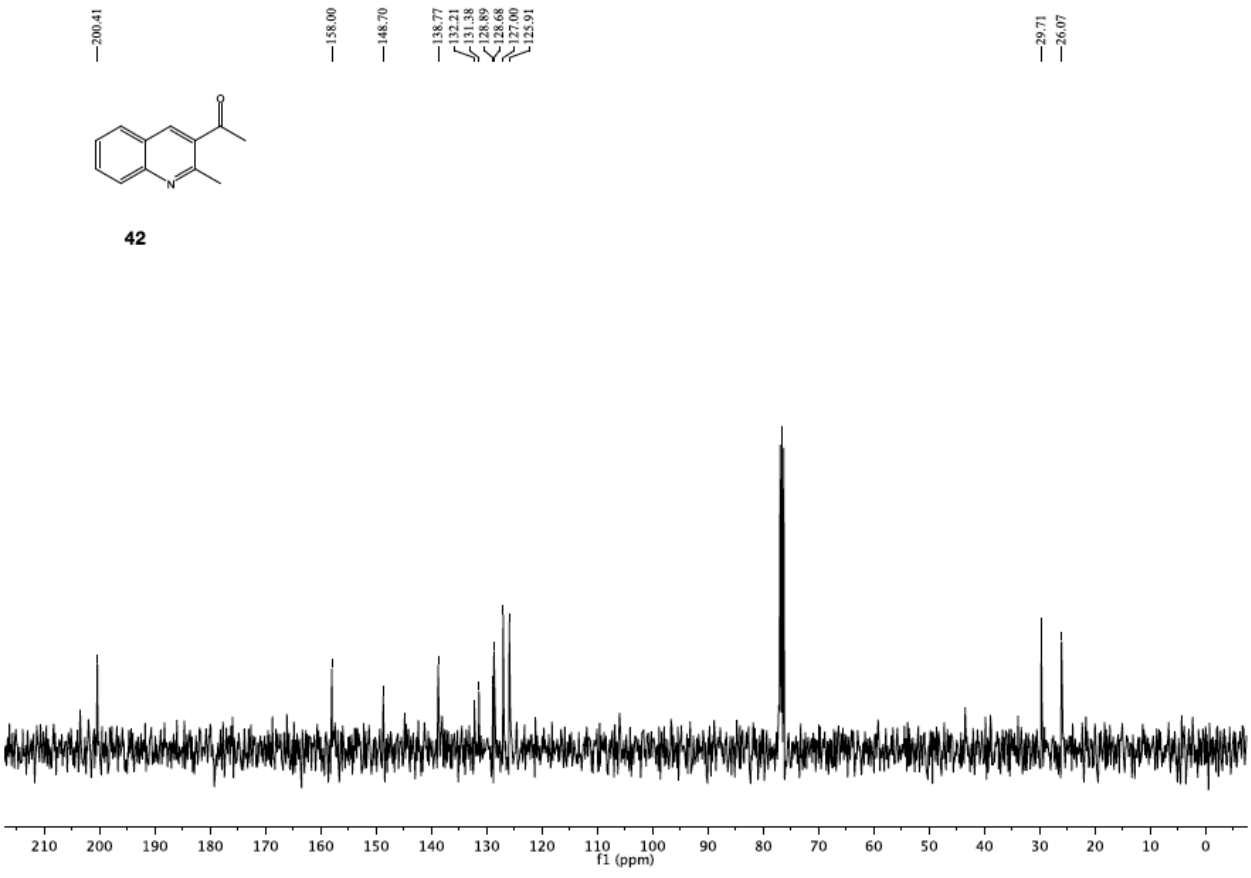
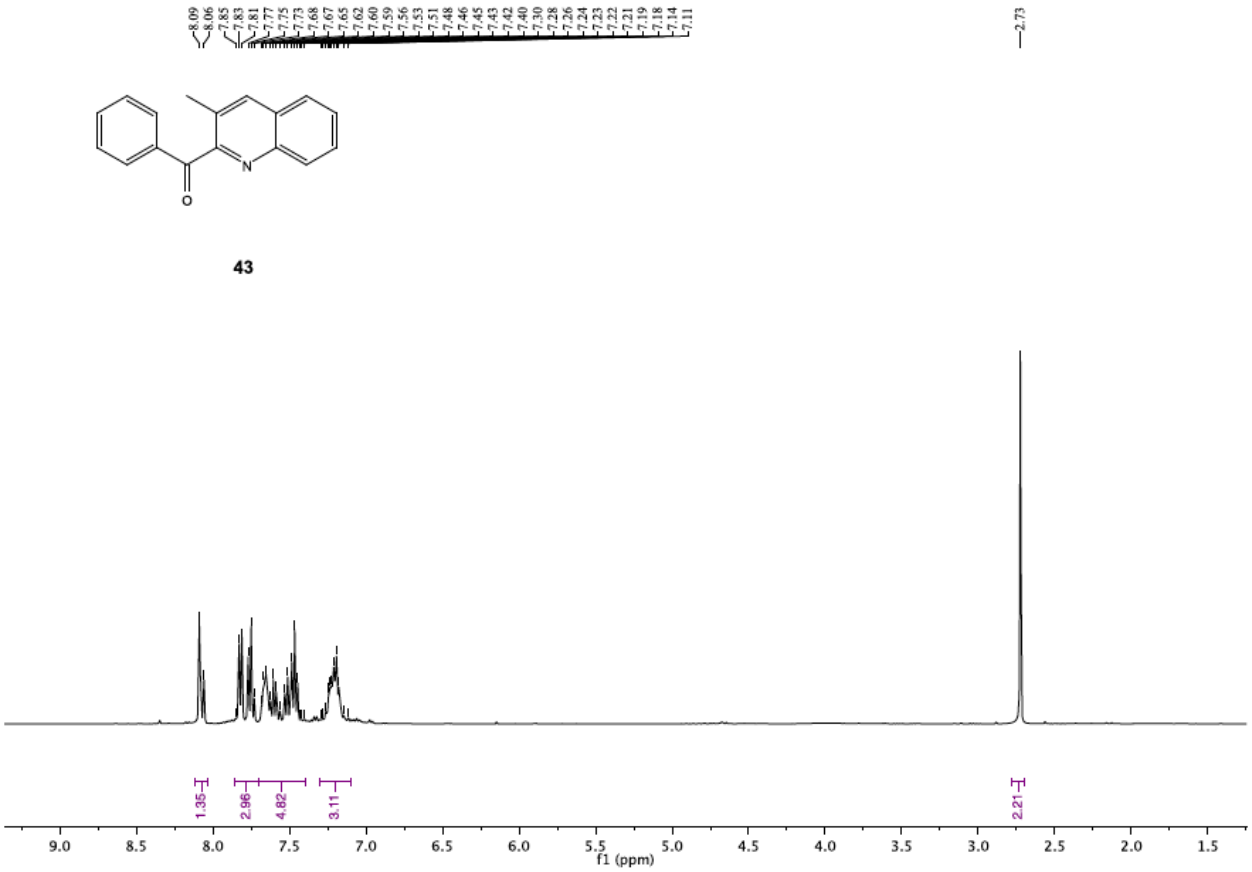
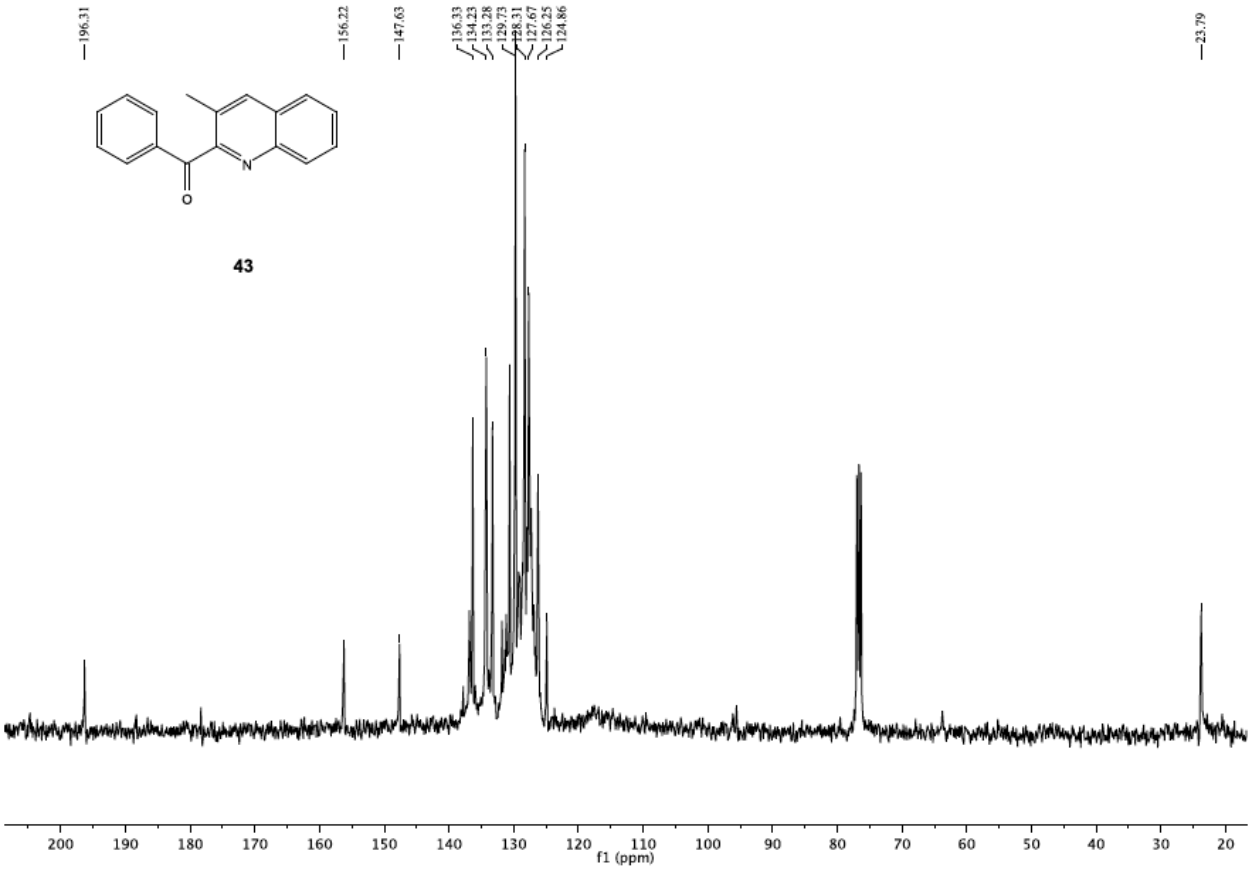
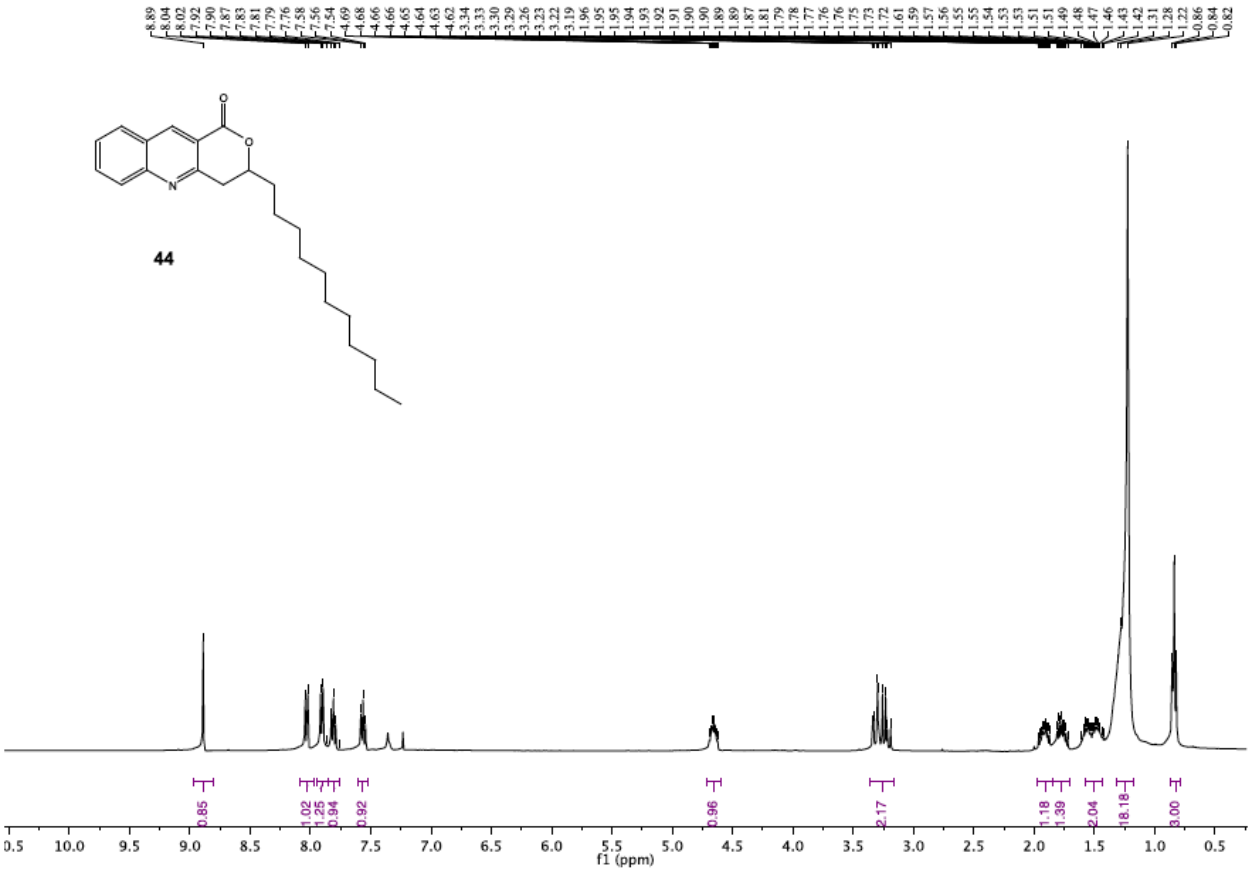
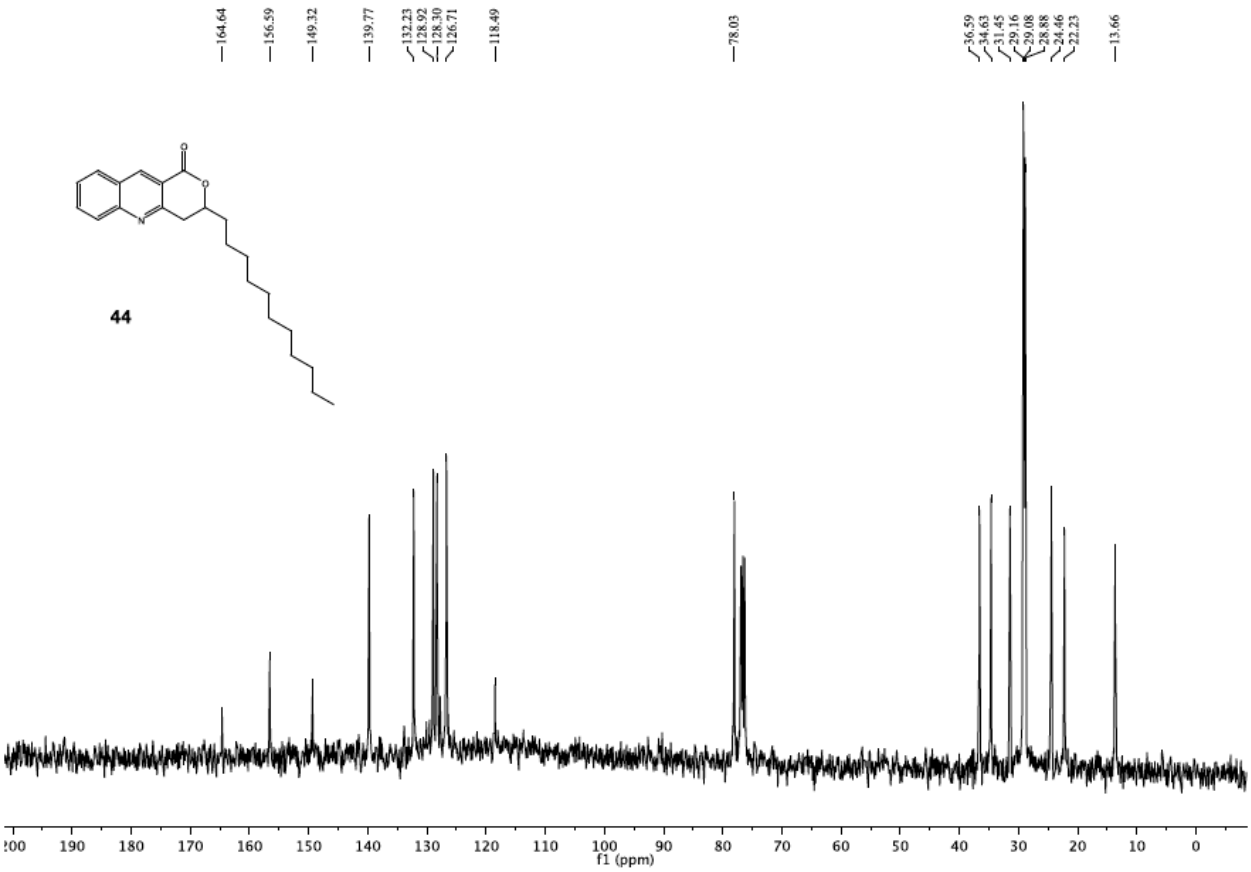
7.79
7.77
7.74
7.72
7.70
7.70
7.50
7.48
7.47
3.66
3.65
42
0.95
0.93
0.99
0.96
1.05
3.08
3.00
f1 (ppm)


200.41
158.00
148.70
138.77
132.21
131.38
128.89
128.68
127.00
125.91
29.71
26.07
42
f1 (ppm)


8.09
8.06
7.85
7.83
7.81
7.77
7.75
7.73
7.68
7.67
7.65
7.62
7.60
7.59
7.56
7.53
7.51
7.48
7.46
7.45
7.43
7.42
7.40
7.30
7.28
7.26
7.24
7.23
7.22
7.21
7.19
7.18
7.14
7.11
2.73
N
O
43
1.35
2.96
4.82
3.11
2.21
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
f1 (ppm)


196.31
156.22
147.63
136.33
134.23
133.28
129.73
128.31
127.67
126.25
124.86
23.79
N
O
43
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


44
f1 (ppm)


44
f1 (ppm)