

Supporting Information
for
Cross-coupling of dissimilar ketone enolates via enolonium
species to afford non-symmetrical 1,4-diketones

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Experimental, characterization data and copies of NMR spectra

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General methods: Air and/or moisture-sensitive reactions were carried out in anhydrous solvents under an argon atmosphere in predried glassware. CH₂Cl₂ was freshly distilled from CaH₂. All commercially available reagents were used without purification. Column chromatography was carried out on silica gel (100–200 mesh). NMR spectra were recorded on a Bruker Avance III 400 spectrometer operating at 400 MHz (¹H) and 101 MHz (¹³C). ¹H and ¹³C NMR chemical shifts are reported in ppm relative to chloroform-D (δ = 7.26 for ¹H and 77.16 for ¹³C) or TMS (0.0 ppm) and coupling constants (*J*) are reported in hertz (Hz). The following abbreviations have been used to designate signal multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. IR spectra were recorded as films on a Bruker FT-IR. TOF-ESI-MS (*m/z*) was recorded on a Waters Micromass LCT premier instrument at 70 eV in the positive or negative mode. APCI-HRMS was recorded on a Bruker Maxis Impact QTOF instrument using the APCI solid probe.

General procedure for the synthesis of 1,4-diketones

Under an argon atmosphere, BF₃·OEt₂ (1.25 equiv) was added to a slurry of hydroxy(tosyloxy)iodobenzene (1.25 equiv) in anhydrous CH₂Cl₂ (8 mL) at room temperature resulting in a clear yellow solution. The solution was cooled to –78 °C, and the first trimethylsilyl enol ether (1.0 equiv) dissolved in CH₂Cl₂ (4 mL) was added slowly. After 10 minutes the second trimethylsilyl enol ether (1.2–1.4 equiv) dissolved in CH₂Cl₂ (2 mL) was added dropwise. The reaction mixture was stirred for 10 min at –78 °C and then allowed to warm to room temperature. Stirring was continued for 1 h in total. The reaction mixture was quenched by the addition of H₂O (5 mL) and then extracted with CH₂Cl₂ (3 × 10 mL). The combined organic extracts were dried over anhydrous Na₂SO₄ and concentrated in vacuo. The residue was subjected to silica gel column chromatography to isolate the corresponding 1,4-diketone product(s).

1-(4-Methoxyphenyl)-4-phenylbutane-1,4-dione (8): Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (192 mg, 1.0 mmol) with 1-(4-methoxyphenyl)-1-trimethylsiloxyethylene (311 mg, 1.4 mmol) following the general procedure gave 1-(4-methoxyphenyl)-4-phenylbutane-1,4-dione (193 mg, 72%) as white solid; **R_f**: 0.4 (1:4 v/v EtOAc/hexane). **FT-IR:** *V*_{max} 2979, 1710, 1678, 1599, 1356, 1171, 954 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ 7.97–8.08 (m, 4H), 7.54–7.61 (m, 1H), 7.45–7.52 (m, 2H), 6.92–6.98 (m, 2H), 3.88 (s, 3H), 3.37–3.51 (m, 4H) ppm. **¹³C NMR** (101 MHz, CDCl₃) δ 199.1, 197.4, 163.7, 137.0, 133.3, 130.5, 130.1, 128.7, 128.3, 113.9, 55.6, 32.8, 32.4 ppm; **HRMS** (APCI⁺): *m/z* calculated for C₁₇H₁₇O₃ 269.1178; [M+H]⁺ found 269.1192.

1-(4-Nitrophenyl)-4-phenylbutane-1,4-dione (9): Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (192 mg, 1.0 mmol) with 1-(4-nitrophenyl)-1-trimethylsiloxyethylene (332 mg, 1.4 mmol) following the general procedure gave 1-(4-nitrophenyl)-4-phenylbutane-1,4-dione (184 mg, 65%) as an yellow solid; **R_f**: 0.4 (1:4 v/v EtOAc/hexane). **FT-IR:** *V*_{max} 2968, 1662, 1303, 959, 864 cm⁻¹; **¹H NMR** (400 MHz, CDCl₃): δ 8.28–8.37 (m, 2H), 8.15–8.24 (m, 2H), 7.97–8.09 (m, 2H), 7.56–7.65 (m, 1H), 7.44–7.54 (m, 2H), 3.41–3.56 (m, 4H) ppm. **¹³C NMR** (101 MHz, CDCl₃): δ 198.3, 197.5, 150.5, 141.5, 136.6, 133.6, 129.3, 128.8, 128.3, 124.0, 33.2, 32.8 ppm; **HRMS** (APCI⁺): *m/z* calculated for C₁₆H₁₄N₂O 284.0923; [M+H]⁺ found 284.0949.

1-(4-Fluorophenyl)-4-phenylbutane-1,4-dione (10) [1]: Cross-coupling of 1-(4-fluorophenyl)-1-trimethylsiloxyethylene (262 mg, 1.246 mmol) with 1-phenyl-1-trimethylsiloxyethylene (336 mg, 1.747 mmol) following the general procedure gave 1-(4-fluorophenyl)-4-phenylbutane-1,4-dione (219 mg, 69%) as colorless crystal. **R_f**: 0.4 (1:9 v/v EtOAc/hexane). **¹H NMR** (400 MHz, CDCl₃): δ 8.02–8.09 (m, 4H), 7.58 (tt, *J* = 7.28, 1.2 Hz, 1H), 7.48 (tt, *J* = 7.28, 1.64 Hz, 2H), 7.15 (tt, *J* = 8.62, 2.1 Hz, 2H), 3.42–3.47 (m, 4H) ppm.

¹³C NMR (101 MHz, CDCl₃): 198.7, 197.2, 166.0 (d, *J* = 254.6 Hz), 136.9, 133.4, 130.5 (d, *J* = 9.3 Hz), 128.8, 128.7, 128.3, 115.8 (d, *J* = 21.9 Hz), 32.7, 32.6 ppm.

1-(4-Chlorophenyl)-4-phenylbutane-1,4-dione (11)[2]: Cross-coupling of 1-(4-chlorophenyl)-1-trimethylsiloxyethylene (283 mg, 1.248 mmol) with 1-phenyl-1-trimethylsiloxyethylene (336 mg, 1.747 mmol) following the above general procedure gave 1-(4-chlorophenyl)-4-phenylbutane-1,4-dione (229 mg, 67%) as a colorless solid. **R_f**: 0.4 (1:9 v/v EtOAc/hexane). **¹H NMR** (400 MHz, CDCl₃): δ 8.02–8.05 (m, 2H), 7.96–8.00 (m, 2H), 7.58 (tt, *J* = 7.4, 1.3 Hz, 1H), 7.44–7.50 (m, 4H), 3.40–3.49 (m, 4H) ppm. **¹³C NMR** (101 MHz, CDCl₃): δ 198.6, 197.6, 139.7, 136.8, 135.3, 133.4, 129.7, 129.1, 128.8, 128.3, 32.7, 32.4 ppm.

1-(4-Bromophenyl)-4-phenylbutane-1,4-dione (12): Cross-coupling of 1-(4-bromophenyl)-1-trimethylsiloxyethylene (337 mg, 1.242 mmol) with 1-phenyl-1-trimethylsiloxyethylene (336 mg, 1.747 mmol) following the above general procedure gave 1-(4-bromophenyl)-4-phenylbutane-1,4-dione (246 mg, 62%) as a colorless solid. **R_f**: 0.4 (1:9 v/v EtOAc/hexane). **FT-IR**: *V*_{max} 2905, 1673, 1583, 1319, 991, 742, 689 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃): δ 8.02–8.05 (m, 2H), 7.88–7.92 (m, 2H), 7.63 (td, *J* = 8.64, 1.96 Hz, 2H), 7.58 (tt, *J* = 7.4, 1.96 Hz, 1H), 7.48 (tt, *J* = 7.32, 1.64 Hz, 2H), 3.42–3.47 (m, 4H) ppm. **¹³C NMR**: (101 MHz, CDCl₃): δ 198.6, 197.8, 136.8, 135.7, 133.4, 132.1, 129.82, 129.8, 128.5, 128.3, 32.7, 32.6 ppm. **HRMS (ESI⁺)**: *m/z* calculated for C₁₆H₁₄BrO₂ 317.0177 [M+H]⁺; found 317.0198.

1-(4-Iodophenyl)-4-phenylbutane-1,4-dione (13): Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (192 mg, 1.0 mmol) with 1-(4-iodophenyl)-1-trimethylsiloxyethylene (440 mg, 1.382 mmol) following the general procedure gave 1-(4-iodophenyl)-4-phenylbutane-1,4-dione (248 mg, 68%) as a white solid; **R_f**: 0.5 (1:5 v/v EtOAc/hexane). **FT-IR**: *V*_{max} 2921, 1705, 1678, 1599, 1451, 1350, 922 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃) δ

7.98–8.09 (m, 2H), 7.79–7.91 (m, 2H), 7.69–7.79 (m, 2H), 7.52–7.64 (m, 1H), 7.42–7.52 (m, 2H), 3.36–3.51 (m, 4H) ppm. ^{13}C NMR (101 MHz, CDCl_3) δ 198.6, 198.2, 138.1, 136.8, 136.2, 133.4, 129.7, 129.66, 128.8, 128.3, 101.2, 32.7, 32.6 ppm; HRMS (APCI $^+$) m/z calculated for $\text{C}_{16}\text{H}_{14}\text{IO}_2$ 365.0039; $[\text{M}+\text{H}]^+$ found 365.0061.

2-Methyl-1,4-diphenylbutane-1,4-dione (14):[2] Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (240 mg, 1.248 mmol) with trimethyl((1-phenylprop-1-en-1-yl)oxy)silane (360 mg, 1.747 mmol) following the above general procedure gave 2-methyl-1,4-diphenylbutane-1,4-dione (233 mg, 74%) as a colorless solid; R_f : 0.4 (1:9 v/v EtOAc/hexane). ^1H NMR (400 MHz, CDCl_3): δ 8.06 (dt, J = 8.5, 1.7 Hz, 2H), 7.99 (dt, J = 8.5, 1.7 Hz, 2H), 7.54–7.60 (m, 2H), 7.43–7.51 (m, 4H), 4.19 (ddd, J = 8.4, 7.2, 4.9 Hz, 1H), 3.74 (dd, J = 18.0, 8.5 Hz, 1H), 3.12 (dd, J = 18.0, 4.9 Hz, 1H), 1.29 (d, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 203.5, 198.6, 136.7, 136.2, 133.3, 133.1, 128.8, 128.7, 128.6, 128.2, 42.5, 36.4, 18.1 ppm.

1,2,4-Triphenylbutane-1,4-dione (15):[3] Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (192 mg, 1.00 mmol) with ((1,2-diphenylvinyl)oxy)trimethylsilane (376 mg, 1.400 mmol) following the general procedure gave 1,2,4-triphenylbutane-1,4-dione (173 mg, 55%) as a colorless oil. R_f : 0.5 (1:5 v/v EtOAc/hexane). ^1H NMR (400 MHz, CDCl_3): δ 7.95–8.08 (m, 4H), 7.28–7.60 (m, 10H), 7.18–7.26 (m, 1H), 5.34 (dd, J = 10.0, 3.7 Hz, 1H), 4.23 (dd, J = 18.0, 10.1 Hz, 1H), 3.31 (dd, J = 18.0, 3.7 Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 199.1, 198.2, 138.8, 136.6, 133.4, 133.0, 129.3, 129.1, 128.8, 128.6, 128.59, 128.4, 128.3, 127.5, 48.9, 44.0 ppm.

2-(2-Oxo-2-phenylethyl)cyclohexan-1-one (16):[4] Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (240 mg, 1.248 mmol) with trimethylsilyl enolether of cyclohexanone (255 mg, 1.49 mmol) following the above general procedure gave 2-(2-oxo-2-

phenylethyl)cyclohexan-1-one (103 mg, 38%) as colorless solid. When 3 equivalents (637 mg, 3.744 mmol) of trimethylsilyl enol ether of cyclohexanone was used the yield was 78% (211 mg). **R_f**: 0.24 (1:4 v/v Et₂O/Pentane). **¹H NMR** (400 MHz, CDCl₃): δ 7.98 (d, 2H), 7.55 (t, 1H), 7.45 (t, 2H), 3.60 (dd, *J* = 17.7, 6.6 Hz, 1H), 3.10–3.23 (m, 1H), 2.68 (dd, *J* = 17.7, 5.7 Hz, 1H), 2.43 (dd, *J* = 9.8, 5.0 Hz, 2H), 2.06–2.24 (m, 2H), 1.56–1.94 (m, 3H), 1.45 (qd, *J* = 12.8, 3.8 Hz, 1H) ppm. **¹³C NMR** (101 MHz, CDCl₃): δ 211.7, 198.8, 137.2, 133.13, 128.6, 128.2, 46.6, 42.1, 38.4, 34.4, 28.1, 25.5 ppm.

2-(2-Oxo-2-phenylethyl)-3,4-dihydronaphthalen-1(2H)-one (17)[5]: Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (192 mg, 1.00 mmol) with ((3,4-dihydronaphthalen-1-yl)oxy)trimethylsilane (306 mg, 1.400 mmol) following the general procedure gave 2-(2-oxo-2-phenylethyl)-3,4-dihydronaphthalen-1(2H)-one (133 mg, 50%) as a white solid; **R_f**: 0.6 (1:5 v/v EtOAc/hexane); **¹H NMR** (400 MHz, CDCl₃): δ 7.97–8.10 (m, 3H), 7.52–7.65 (m, 1H), 7.42–7.52 (m, 3H), 7.24–7.34 (m, 2H), 3.87 (dd, *J* = 17.5, 4.6 Hz, 1H), 3.33 (ddt, *J* = 13.3, 7.1, 4.6 Hz, 1H), 3.19 (ddd, *J* = 17.0, 12.7, 4.5 Hz, 1H), 2.99 (ddd, *J* = 17.6, 7.2, 2.9 Hz, 2H), 2.25–2.36 (m, 1H), 1.99 (qd, *J* = 13.0, 4.3 Hz, 1H) ppm. **¹³C NMR** (101 MHz, CDCl₃): δ 199.2, 198.7, 144.3, 137.2, 133.5, 133.3, 132.5, 128.9, 128.8, 128.3, 127.7, 126.8, 44.4, 39.2, 29.7, 29.6 ppm.

1,6-Diphenylhex-5-ene-1,4-dione (18): Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (231 mg, 1.2 mmol) with trimethyl((4-phenylbuta-1,3-dien-2-yl)oxy)silane (366 mg, 1.678 mmol) following the above general procedure gave 1,6-diphenylhex-5-ene-1,4-dione (206 mg, 65%) as colorless solid. **R_f**: 0.26 (1:4 v/v Et₂O/pentane). **FT-IR**: 3041, 1686, 1619, 1395, 1353, 1207, 1103, 749 cm⁻¹. **¹H NMR** (400 MHz, CDCl₃): δ 7.94–8.03 (m, 2H), 7.65 (d, *J* = 16.2 Hz, 1H), 7.57 (dd, *J* = 6.6, 2.9 Hz, 3H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.37–7.43 (m, 3H), 6.83 (d, *J* = 16.2 Hz, 1H), 3.41 (t, *J* = 6.5 Hz,

2H), 3.17 (t, $J = 6.5$ Hz, 2H). ^{13}C NMR: (101 MHz, CDCl_3): δ 198.8, 198.78, 143.0, 136.9, 134.7, 133.3, 130.6, 129.1, 128.7, 128.5, 128.3, 126.3, 34.6, 32.7 ppm. HRMS (APCI+): m/z calculated for $\text{C}_{18}\text{H}_{17}\text{O}_2$ 265.1229 $[\text{M}+\text{H}]^+$; found 297.1517.

6-(2-Oxo-2-phenylethyl)cyclohex-2-enone (19): Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (240 mg, 1.248 mmol) with (cyclohexa-1,5-dien-1-yloxy)trimethylsilane (294 mg, 1.747 mmol) following the above general procedure gave 6-(2-oxo-2-phenylethyl)cyclohex-2-enone (136 mg, 51%) as a colorless liquid; R_f : 0.4 (1:3 v/v EtOAc/hexane). FT-IR: 2931, 2859, 1686, 1452, 1181, 1103, 983, cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 7.99 (dt, $J = 8.5, 1.7$ Hz, 2H), 7.54 (tt, $J = 7.4, 1.3$ Hz, 1H), 7.45 (tt, $J = 7.3, 1.6$ Hz, 2H), 6.97 (dddd, $J = 9.7, 5.6, 2.3, 1.5$ Hz, 1H), 5.05 (ddd, $J = 10.0, 2.9, 1.0$ Hz, 1H), 3.75 (dd, $J = 17.6, 4.4$ Hz, 1H), 3.12 (ddt, $J = 13.5, 7.6, 4.5$ Hz, 1H), 2.82 (dd, $J = 17.6, 7.5$ Hz, 1H), 2.35–2.60 (m, 2H), 2.15–2.20 (m, 1H), 1.82 (tdd, $J = 13.3, 11.5, 5.1$ Hz, 1H) ppm. ^{13}C NMR (101 MHz, CDCl_3): δ 200.3, 198.7, 150.2, 137.1, 133.2, 129.5, 128.7, 128.2, 43.1, 38.6, 29.2, 26.2 ppm. HRMS (APCI+): m/z calculated for $\text{C}_{14}\text{H}_{15}\text{O}_2$ 215.1072 $[\text{M}+\text{H}]^+$; found 215.1066.

1-Phenyl-4-(thiophen-2-yl)butane-1,4-dione (20)[6]: Cross-coupling of 1-phenyl-1-trimethylsiloxyethylene (240 mg, 1.248 mmol) with trimethyl((1-(thiophen-2-yl)vinyl)oxy)silane (346 mg, 1.747 mmol) following the above general procedure gave 1-phenyl-4-(thiophen-2-yl)butane-1,4-dione (156 mg, 51%) as a colorless solid; R_f : 0.4 (1:3 v/v EtOAc/hexane). FT-IR: 2926, 2869, 1681, 1655, 1400, 1348, 1181 cm^{-1} . ^1H NMR (400 MHz, CDCl_3): δ 8.03 (dt, $J = 7.1, 1.0$ Hz, 2H), 7.84 (dd, $J = 3.8, 1.1$ Hz, 1H), 7.65 (dd, $J = 5.0, 1.1$ Hz, 1H), 7.58 (tt, $J = 7.4, 1.2$ Hz, 1H), 7.48 (tt, $J = 7.3, 1.6$ Hz, 2H), 7.16 (dd, $J = 4.9, 3.8$ Hz, 1H), 3.38–3.48 (m, 4H) ppm. ^{13}C NMR: (101 MHz, CDCl_3): δ 198.6, 191.8, 144.1, 136.8, 133.7, 133.4, 132.2, 128.8, 128.3, 33.3, 32.4 ppm.

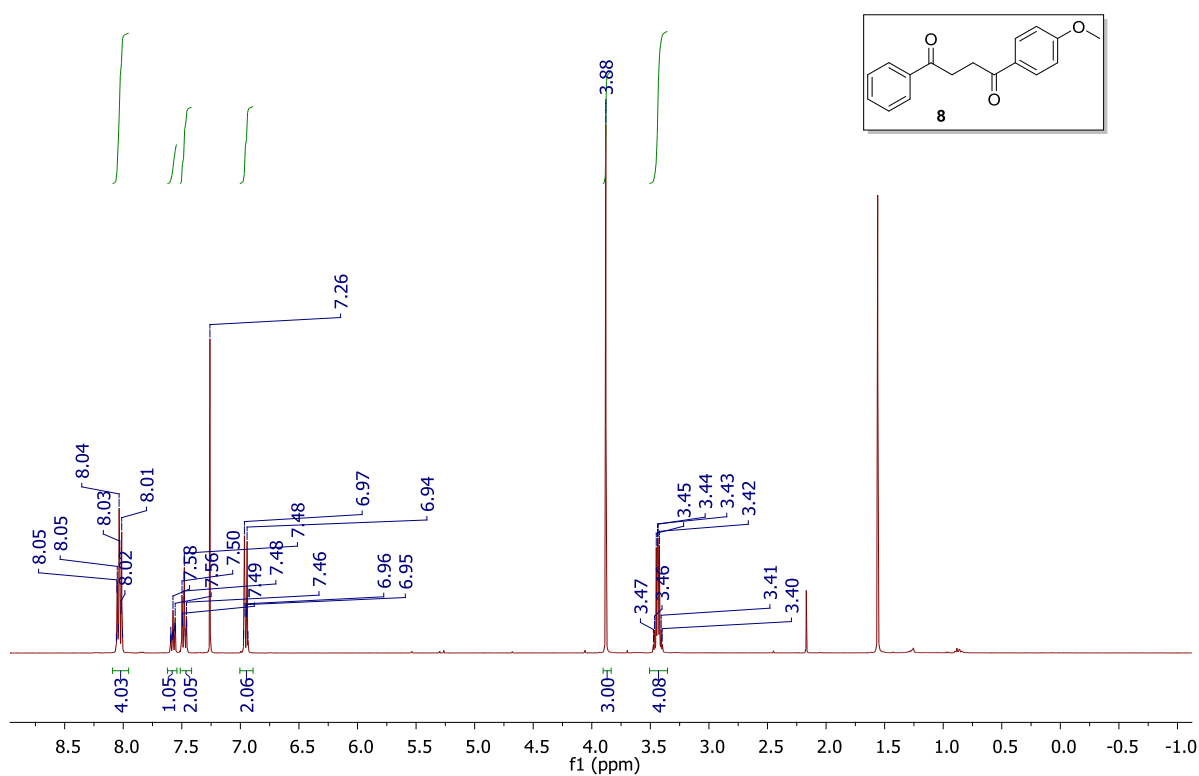
2,3-Dimethyl-1,4-diphenylbutane-1,4-dione (*meso*-23 and *rac*-23): Cross-coupling of trimethyl((1-phenylprop-1-en-1-yl)oxy)silane (257 mg, 1.247 mmol) with trimethyl((1-phenylprop-1-en-1-yl)oxy)silane (360 mg, 1.746 mmol) following the above general procedure gave 2,3-dimethyl-1,4-diphenylbutane-1,4-dione (188 mg, 57%), where *rac:meso* = 1:6.2.

***meso*-2,3-Dimethyl-1,4-diphenylbutane-1,4-dione (*meso*-23)[7]:** Colourless solid. **R_f**: 0.5 (1:9 v/v EtOAc/hexane). **¹H NMR** (400 MHz, CDCl₃): δ 8.05 (dt, *J* = 7.1, 1.4 Hz, 4H), 7.59 (tt, *J* = 7.4, 1.3 Hz, 2H), 7.50 (tt, *J* = 7.3, 1.6, 4H), 4.02–4.08 (m, 2H), 1.14 (dd, *J* = 4.5, 2.0 Hz, 6H) ppm. **¹³C NMR**: (101 MHz, CDCl₃): δ 203.9, 137.0, 133.5, 128.9, 128.6, 43.5, 17.6 ppm.

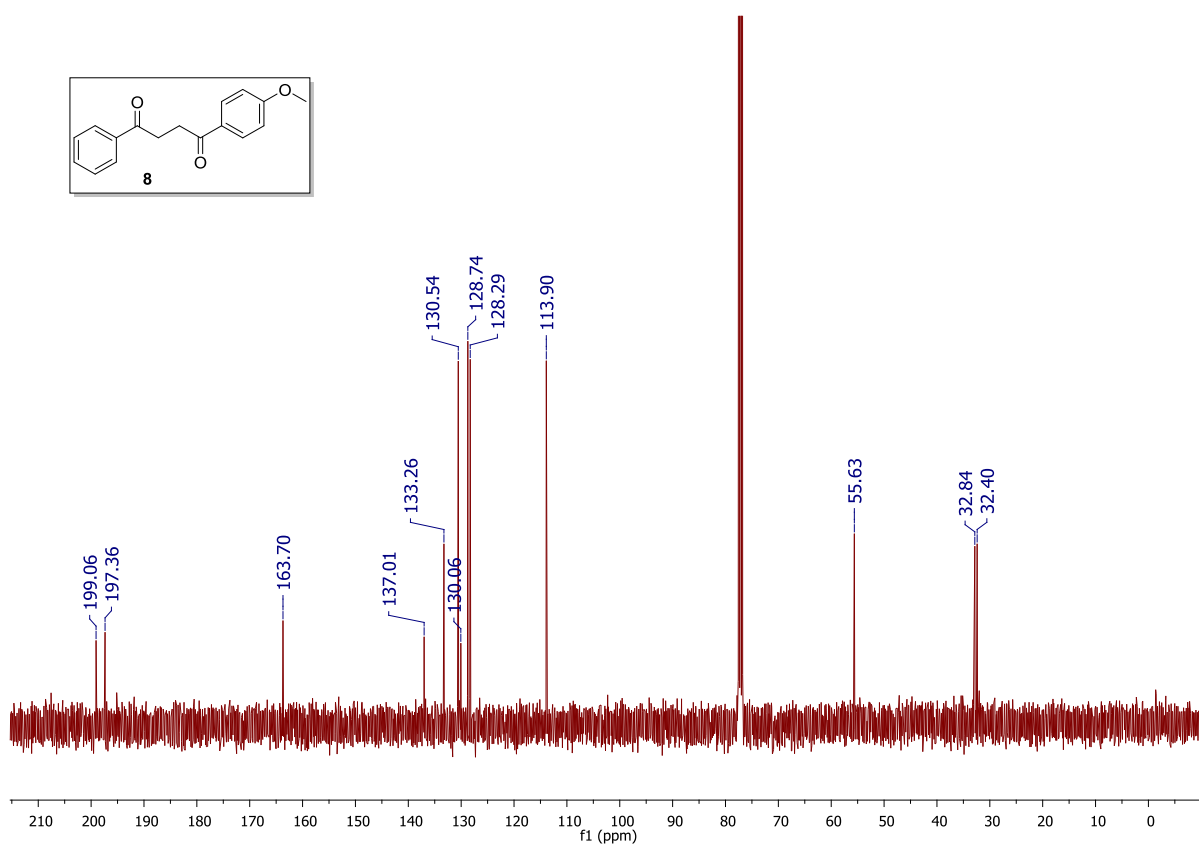
***rac*-2,3-Dimethyl-1,4-diphenylbutane-1,4-dione (*rac*-23)[7]:** Colourless solid. **R_f**: 0.45 (1:9 v/v EtOAc/hexane). **¹H NMR** (400 MHz, CDCl₃): δ 7.99 (dt, *J* = 7.1, 1.4 Hz, 4H), 7.55 (tt, *J* = 7.4, 1.3 Hz, 2H), 7.46 (tt, *J* = 7.3, 1.6, 4H), 3.93–4.01 (m, 2H), 1.29 (dd, *J* = 4.5, 2.0 Hz, 6H) ppm. **¹³C NMR**: (101 MHz, CDCl₃): δ 204.5, 136.3, 133.1, 128.7, 128.6, 43.8, 15.6 ppm.

References

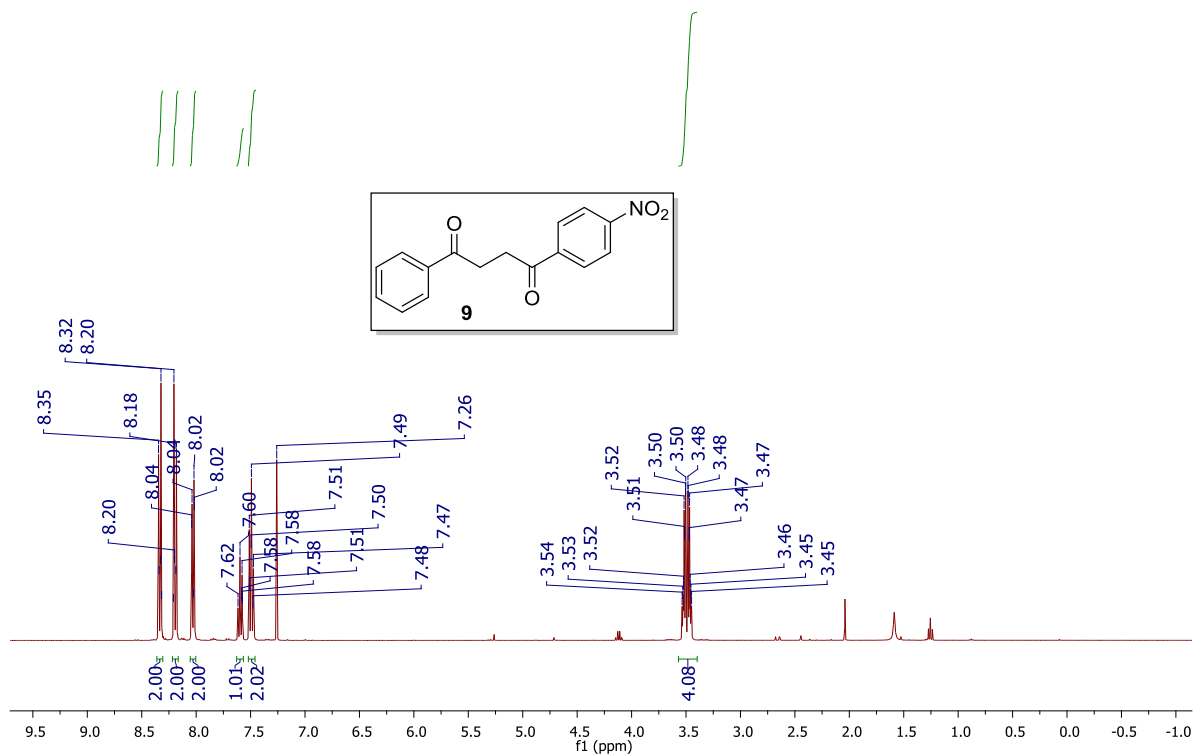
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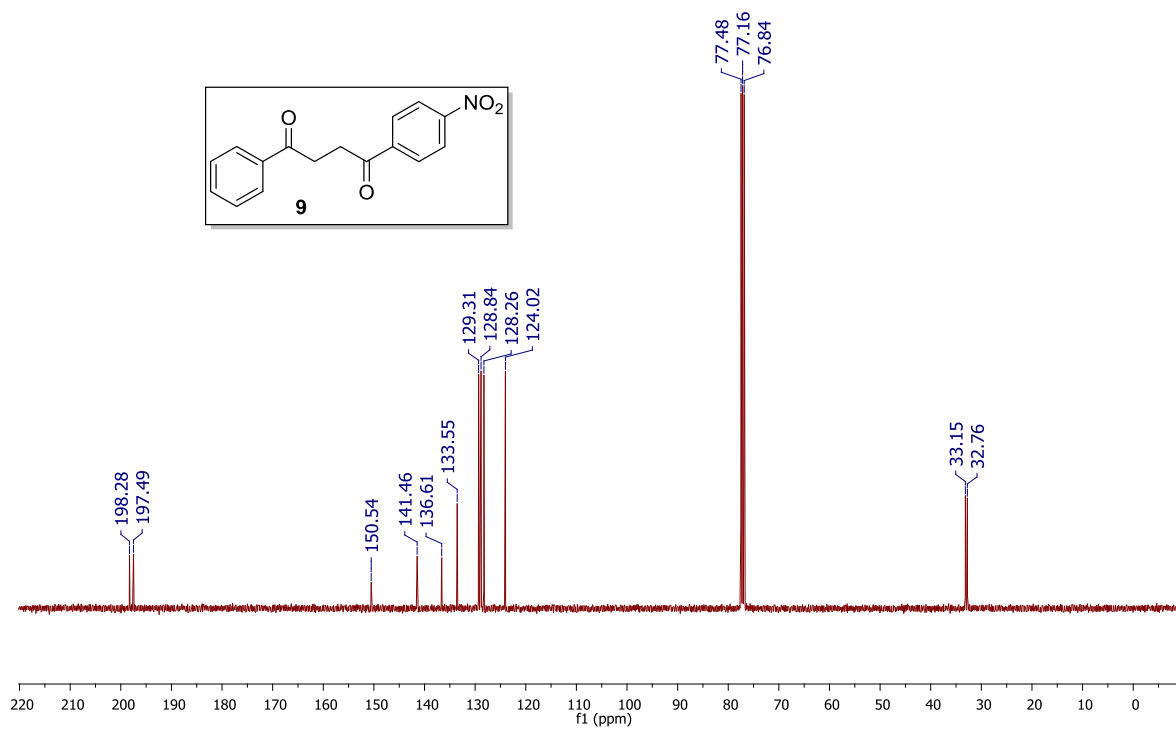
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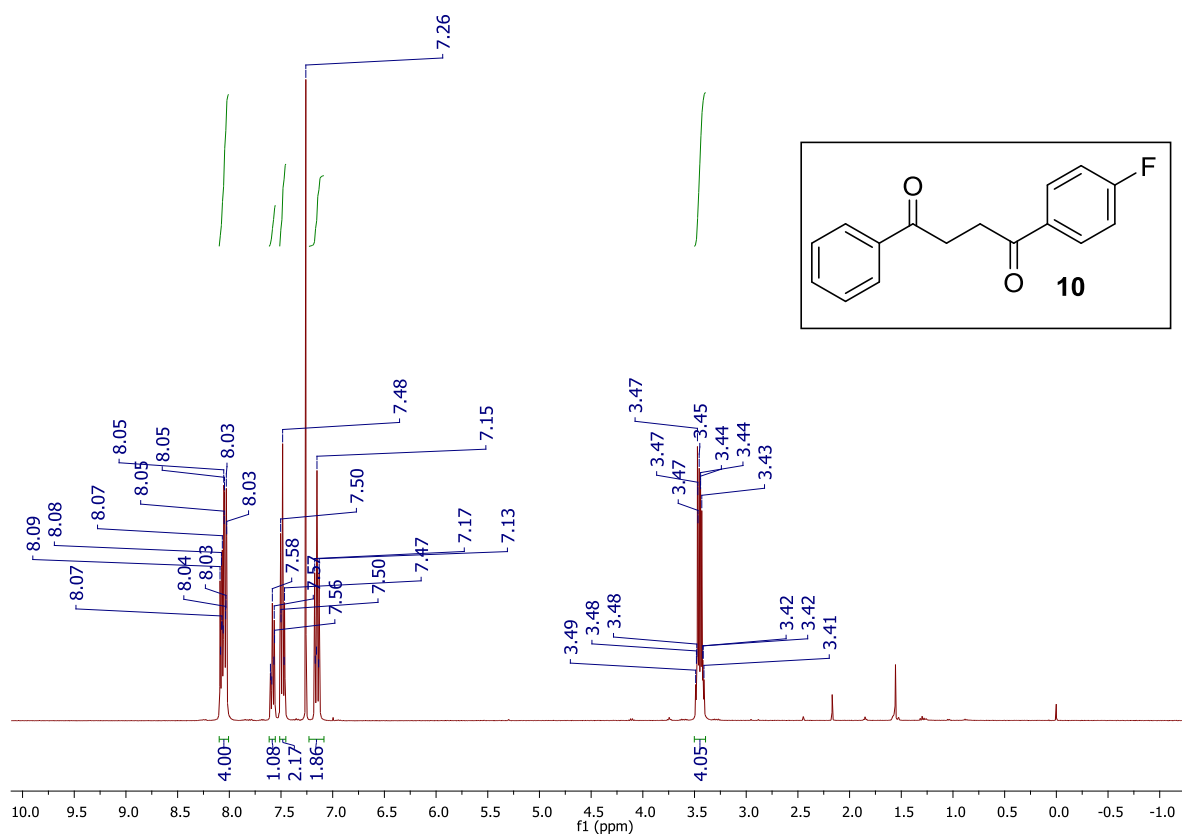
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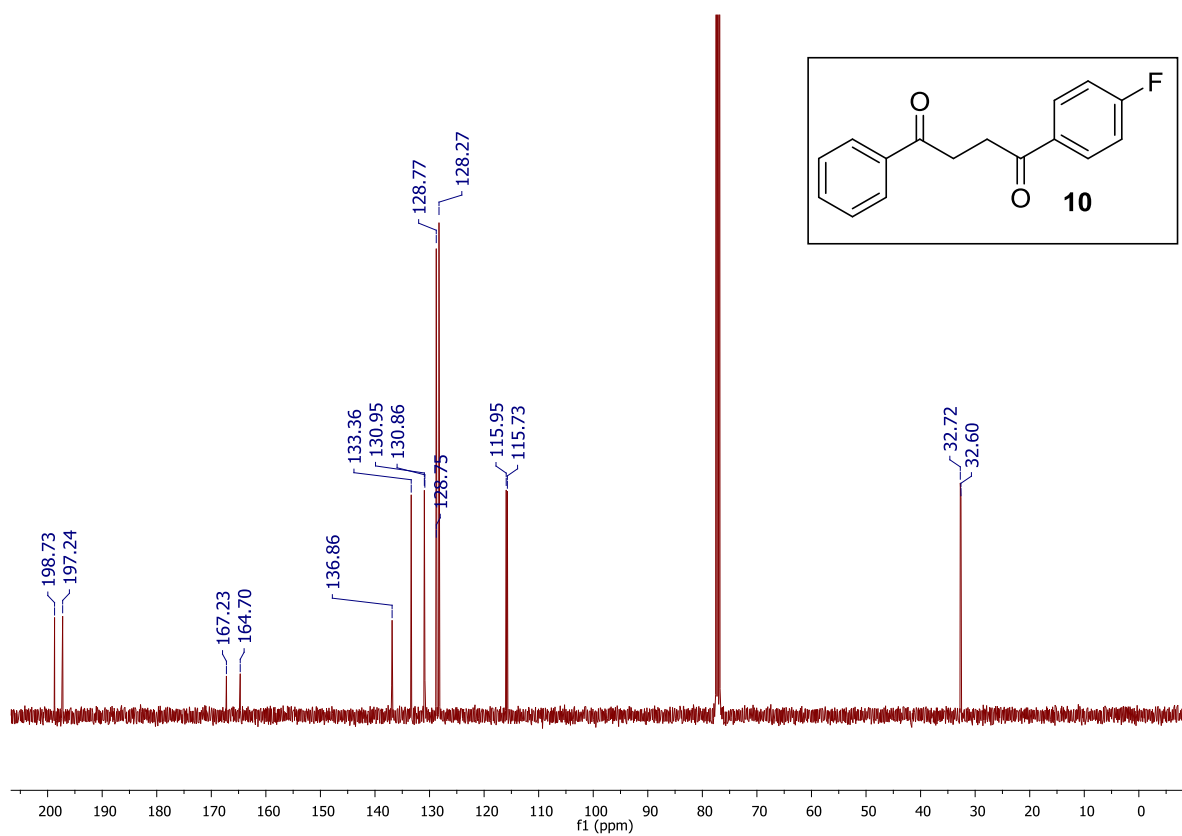
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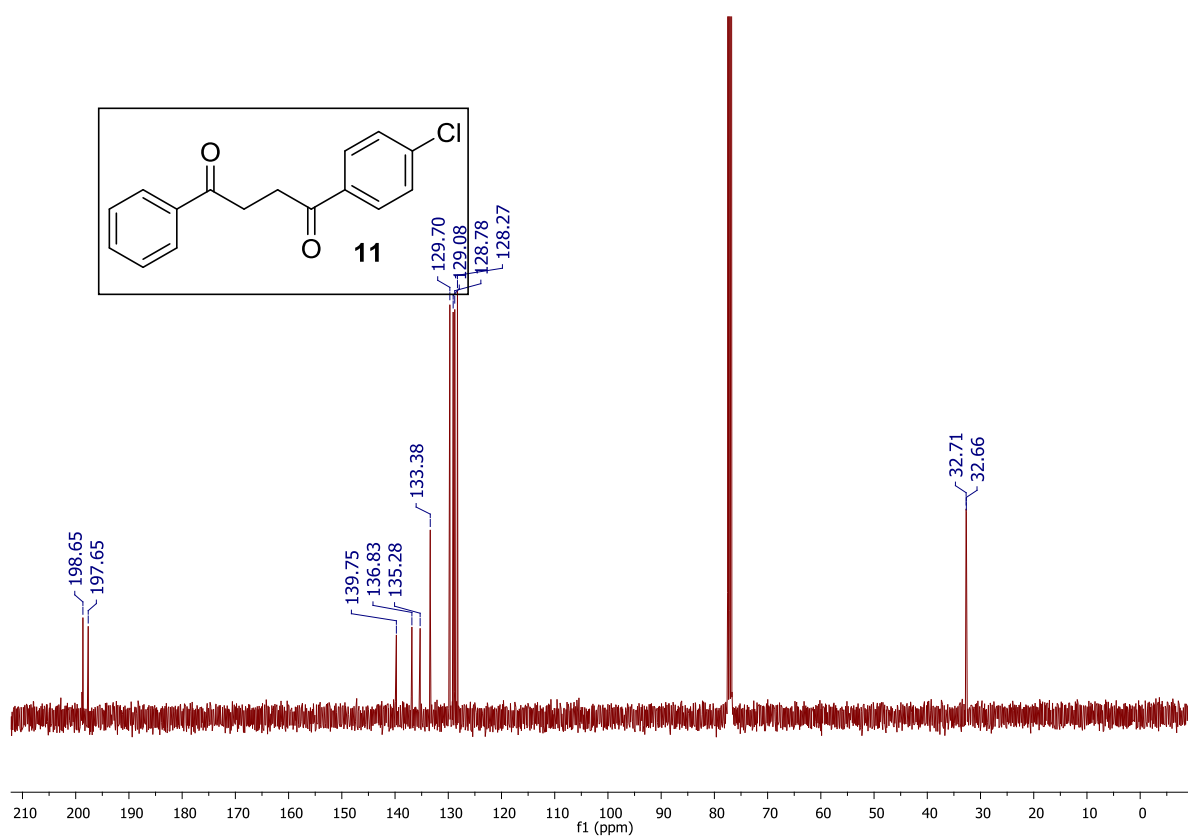
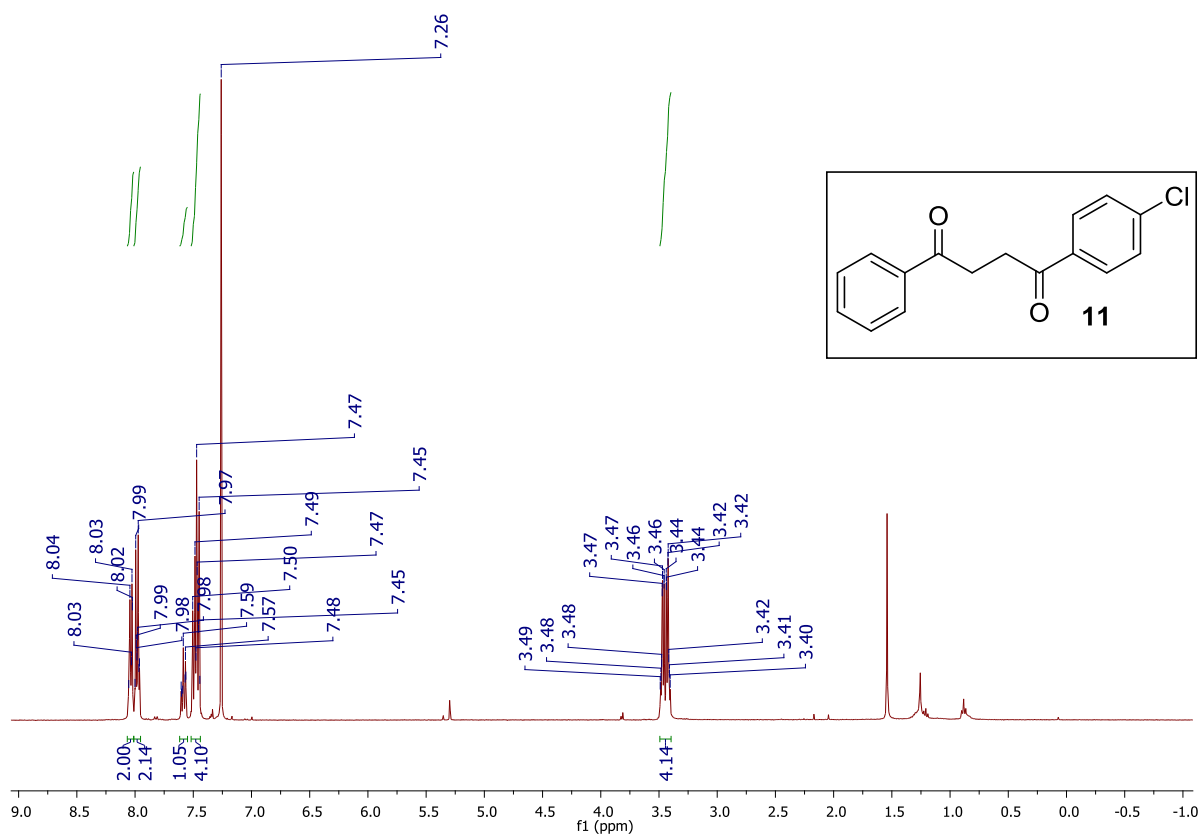
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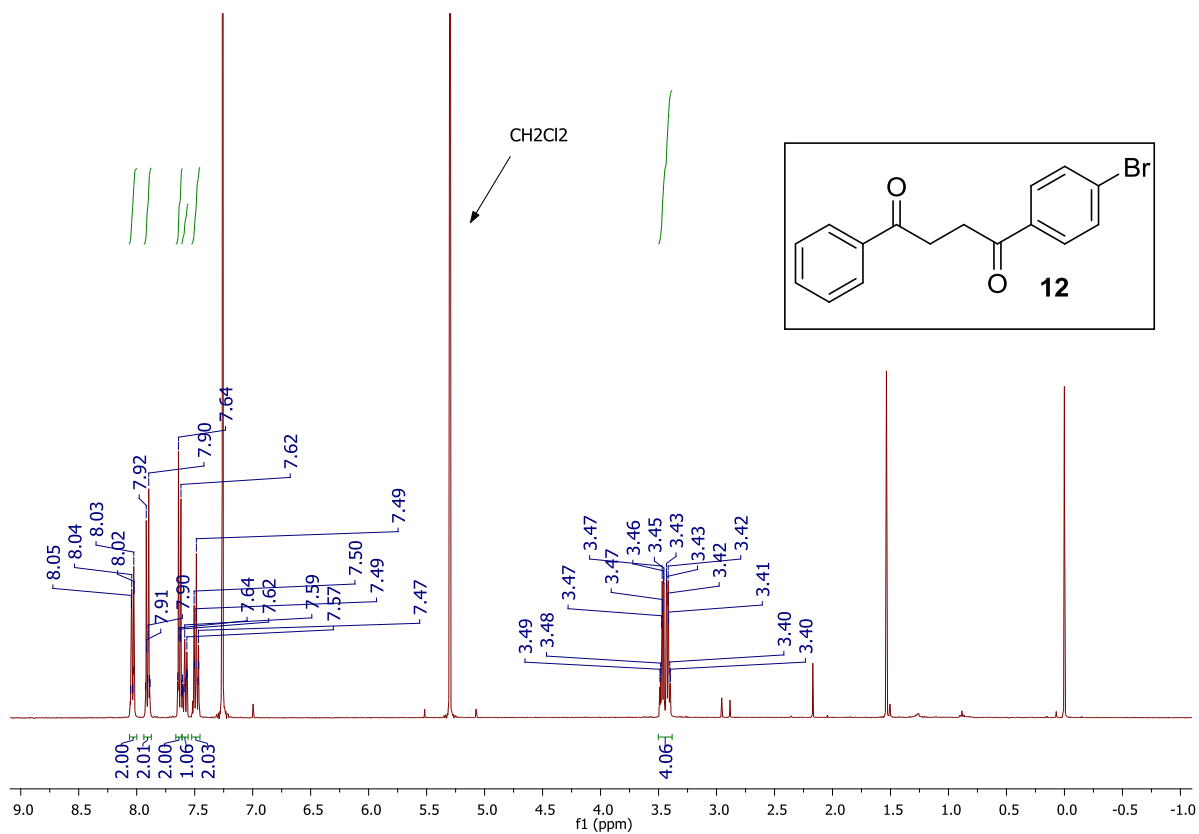


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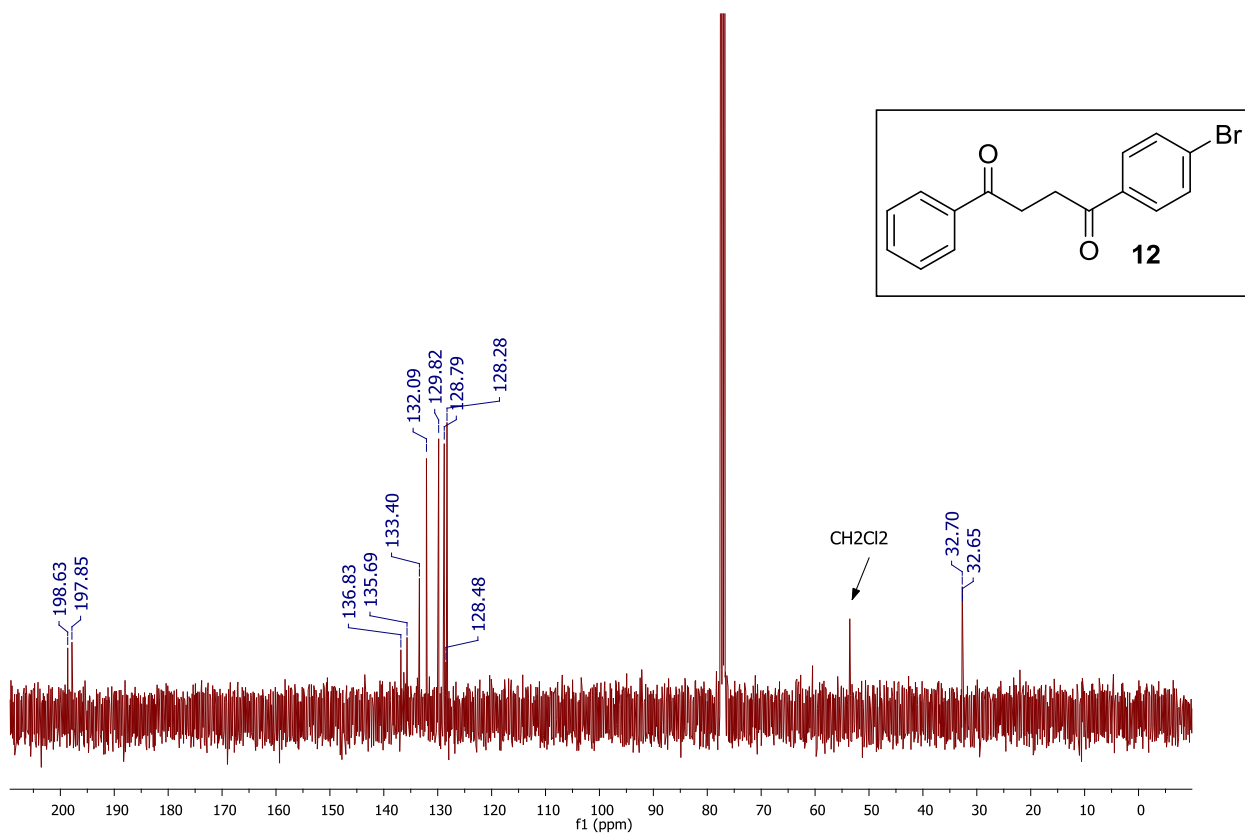


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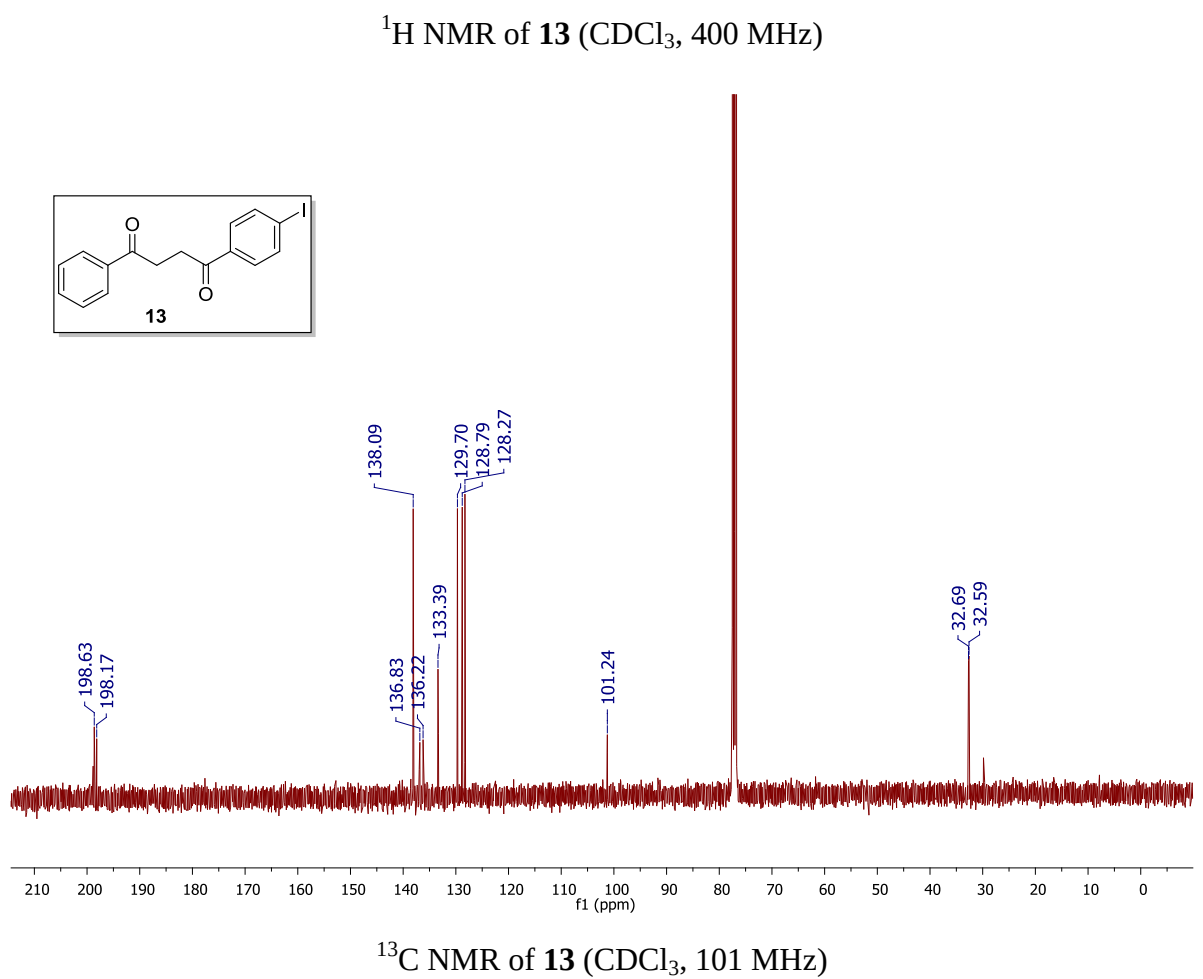
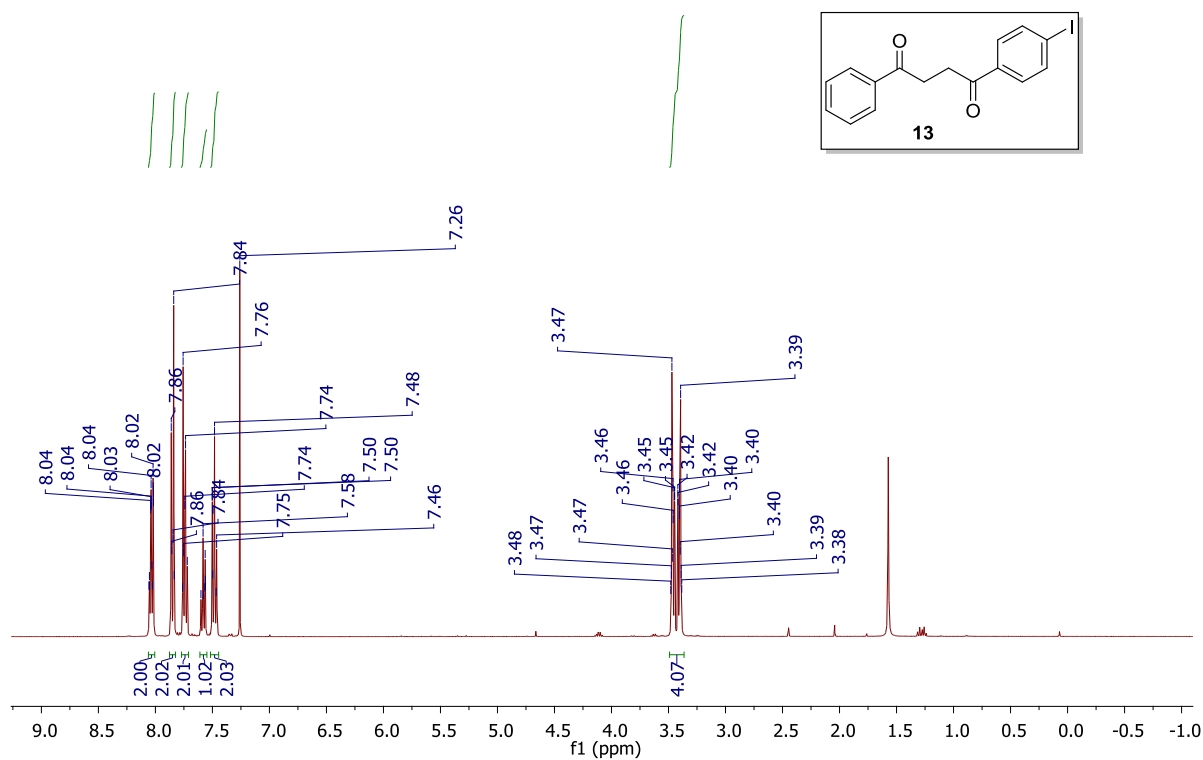


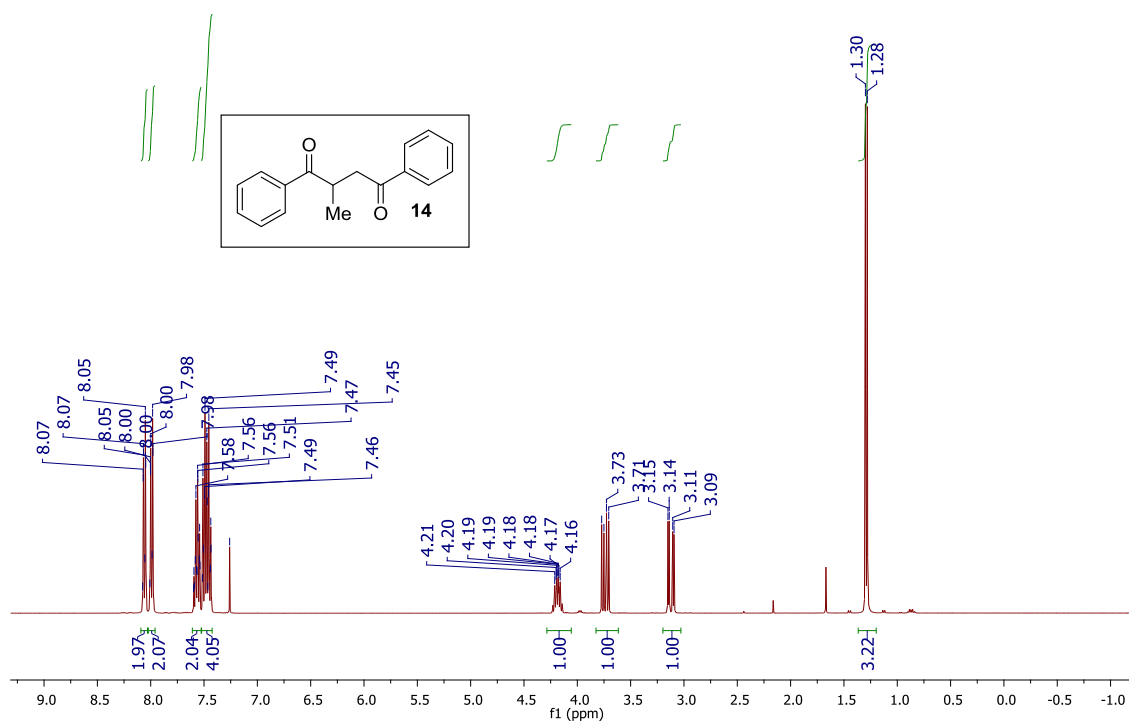


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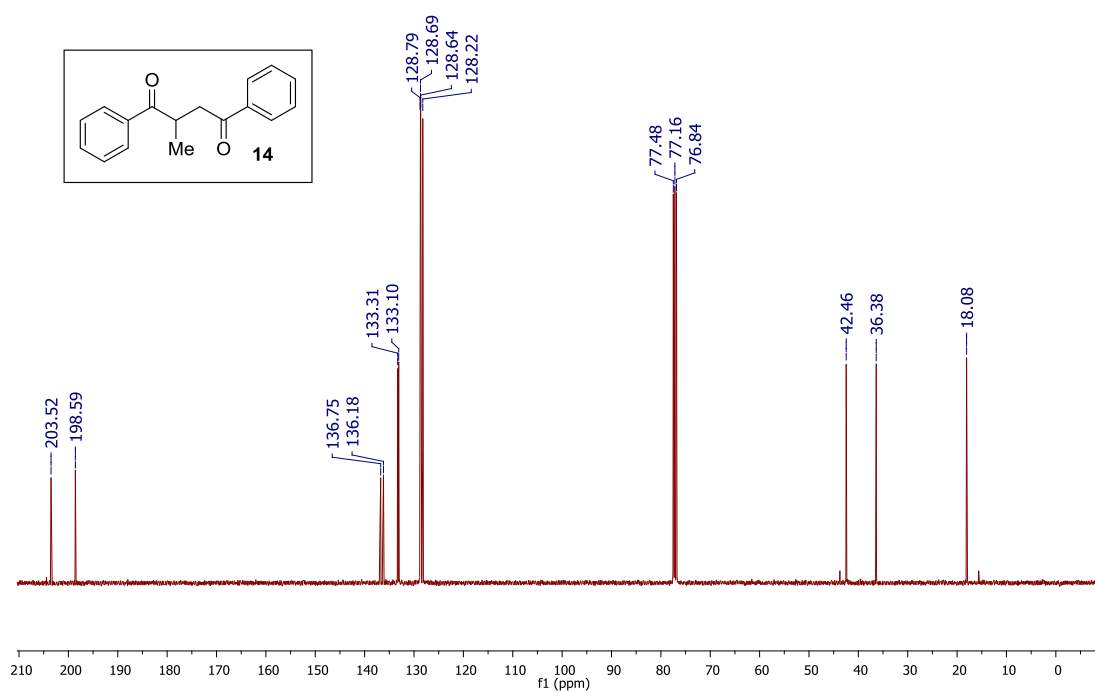


¹³C NMR of **12** (CDCl₃, 101 MHz)

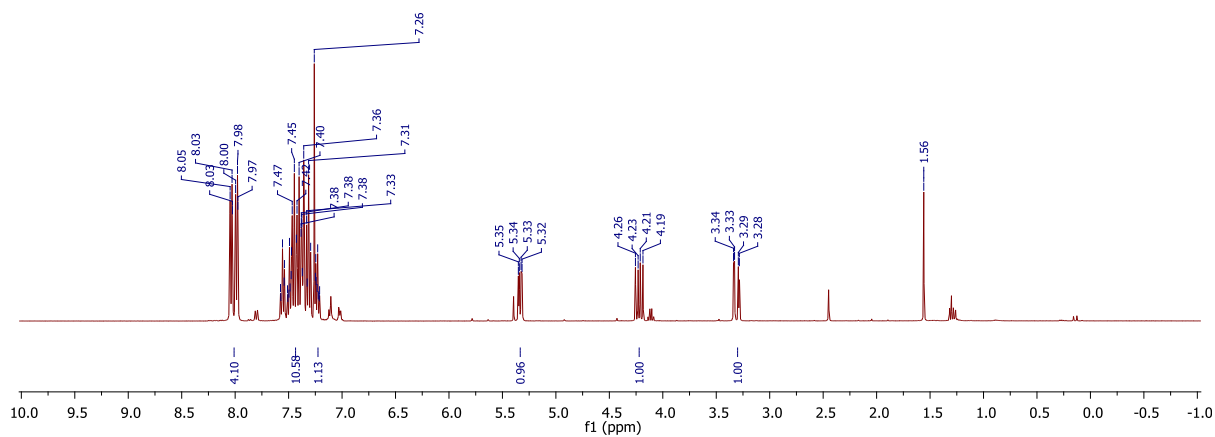
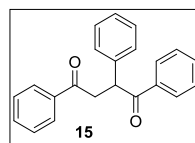




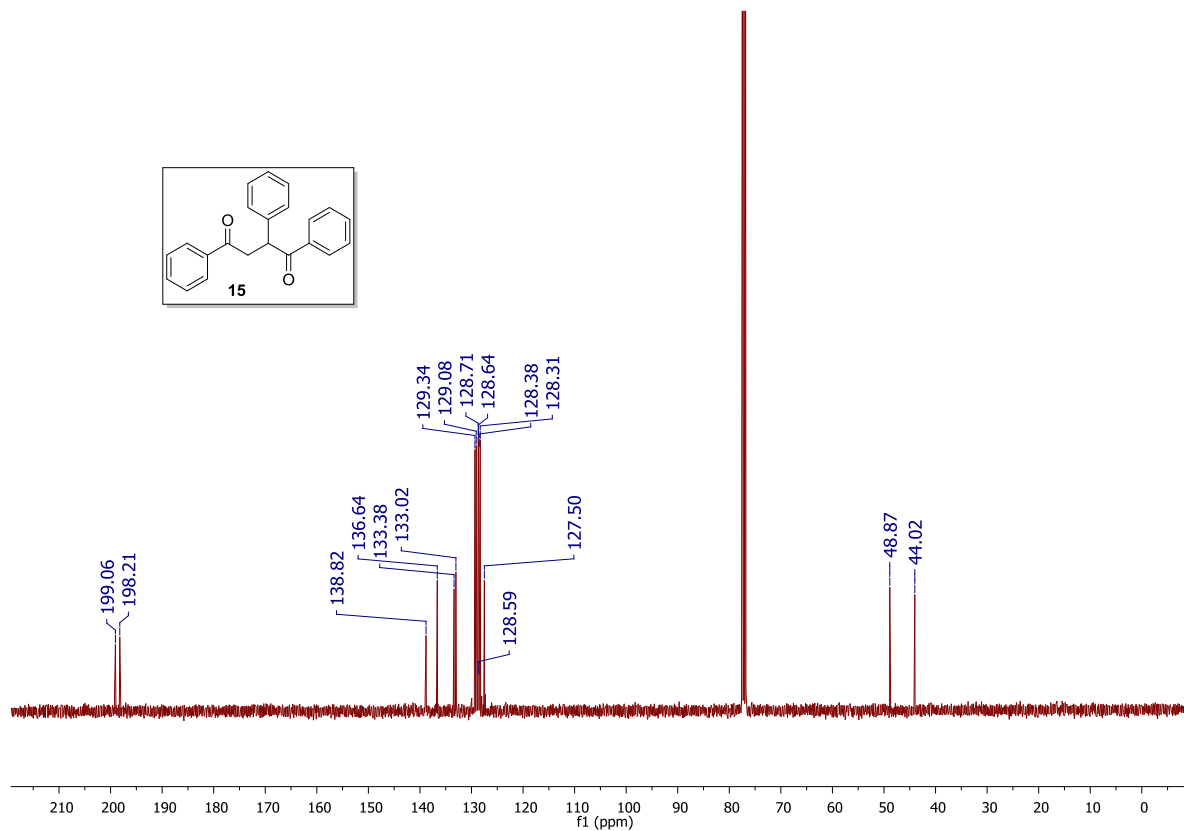
¹H NMR of **14** (CDCl₃, 400 MHz)



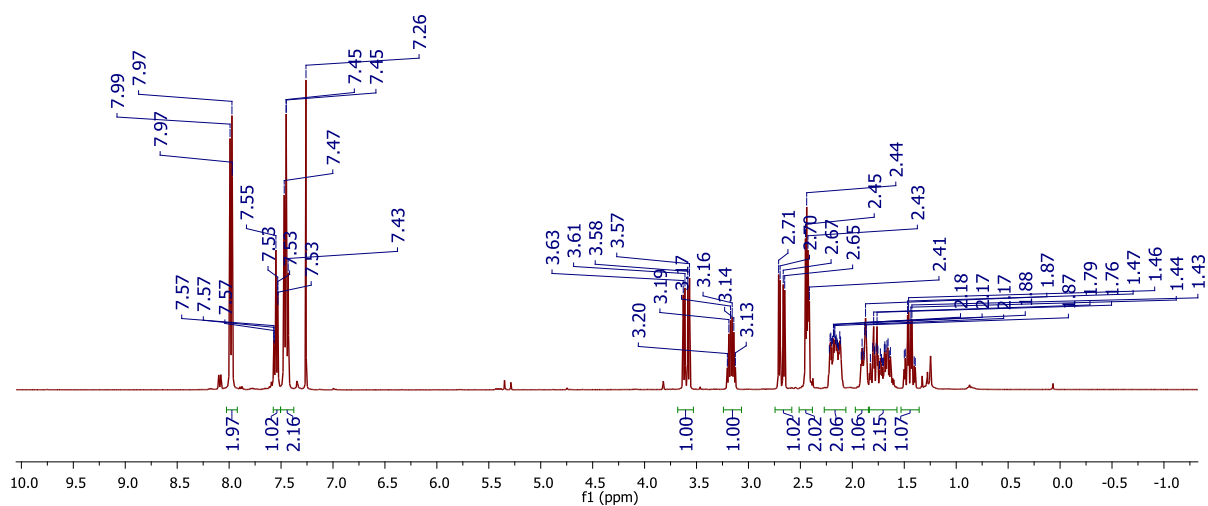
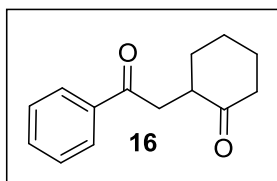
¹³C NMR of **14** (CDCl₃, 101 MHz)



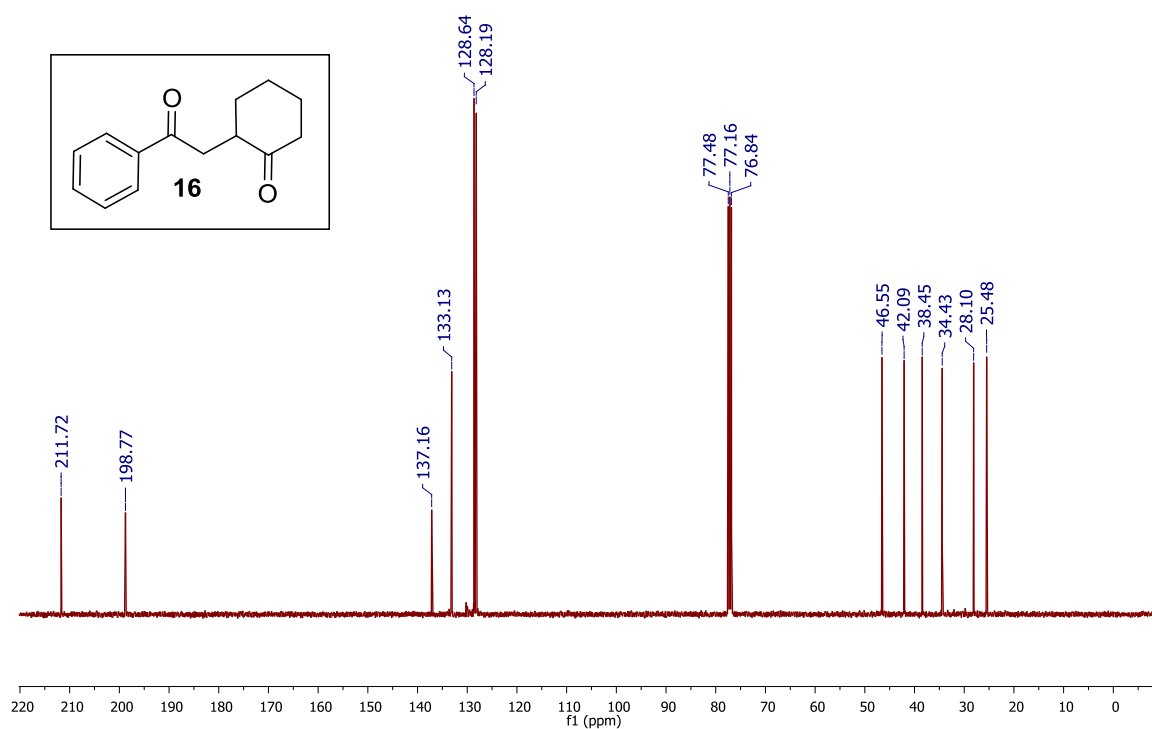
¹H NMR of **15** (CDCl₃, 400 MHz)



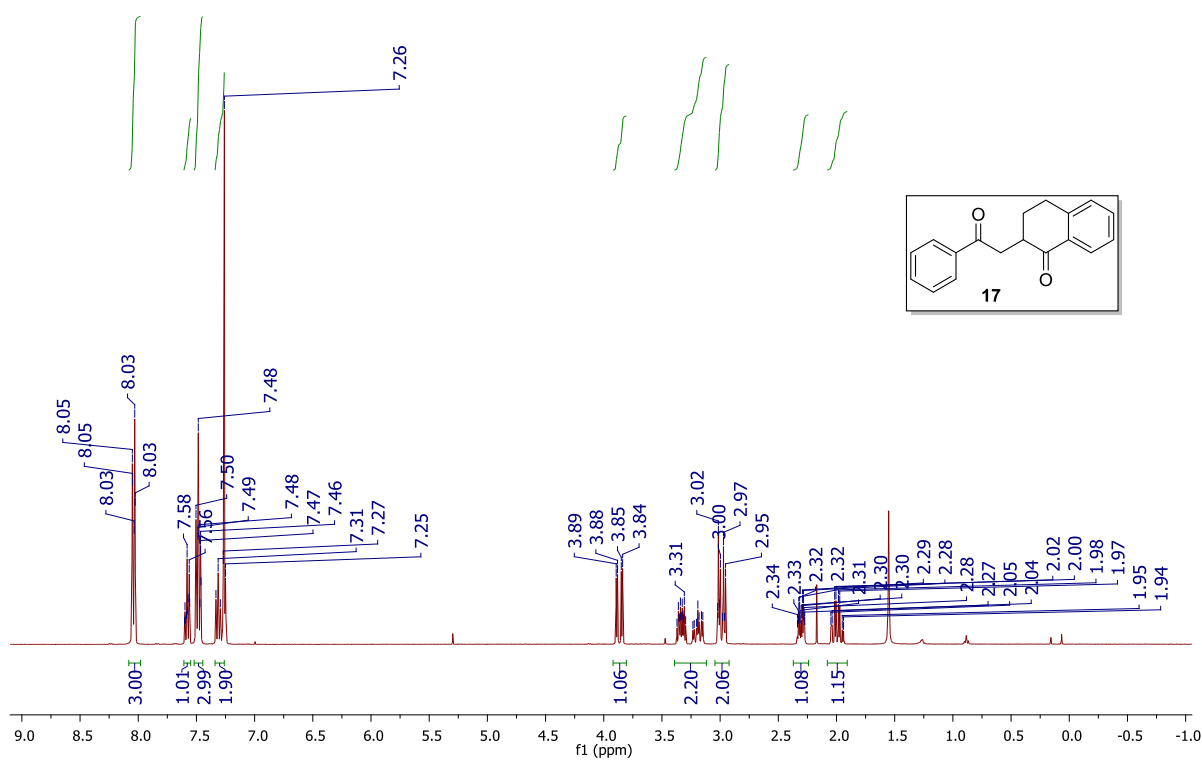
¹³C NMR of **15** (CDCl₃, 101 MHz)



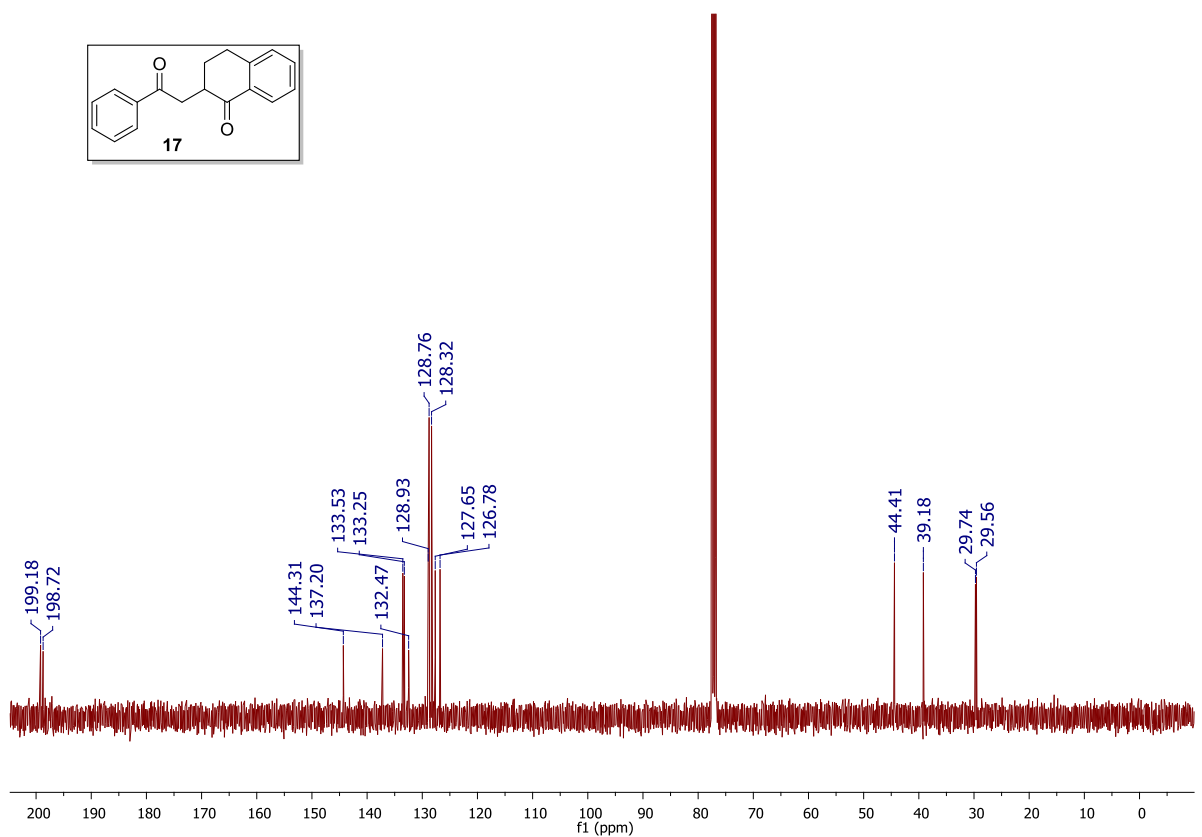
¹H NMR of **16** (CDCl₃, 400 MHz)



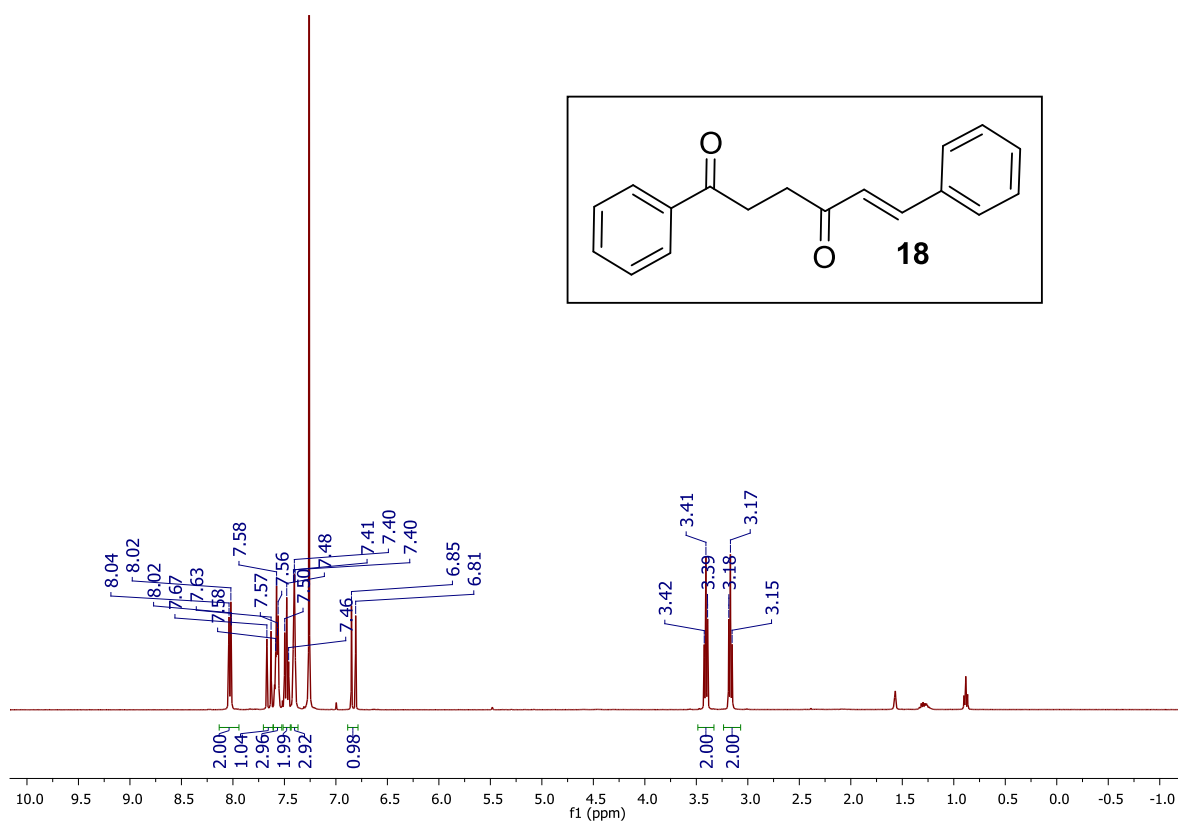
¹³C NMR of **16** (CDCl₃, 101 MHz)



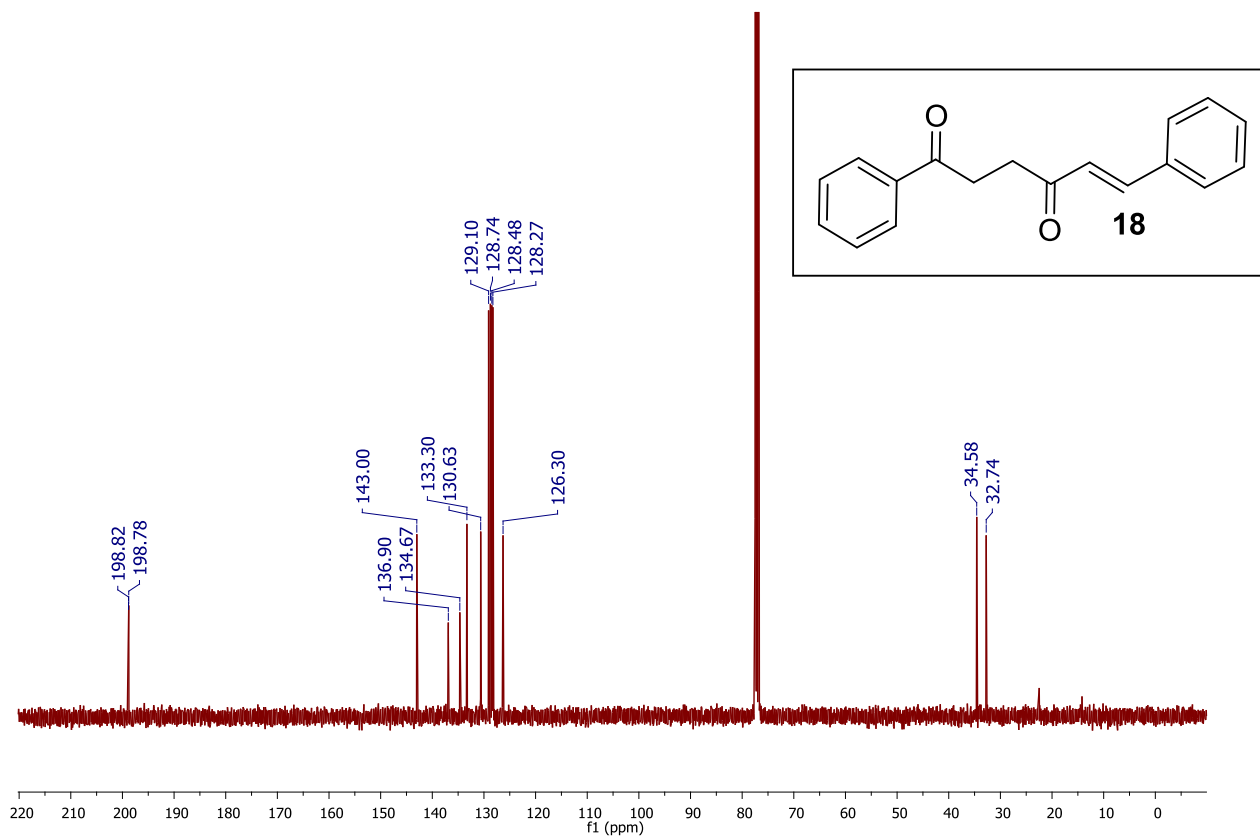
¹H NMR of 17 (CDCl₃, 400 MHz)



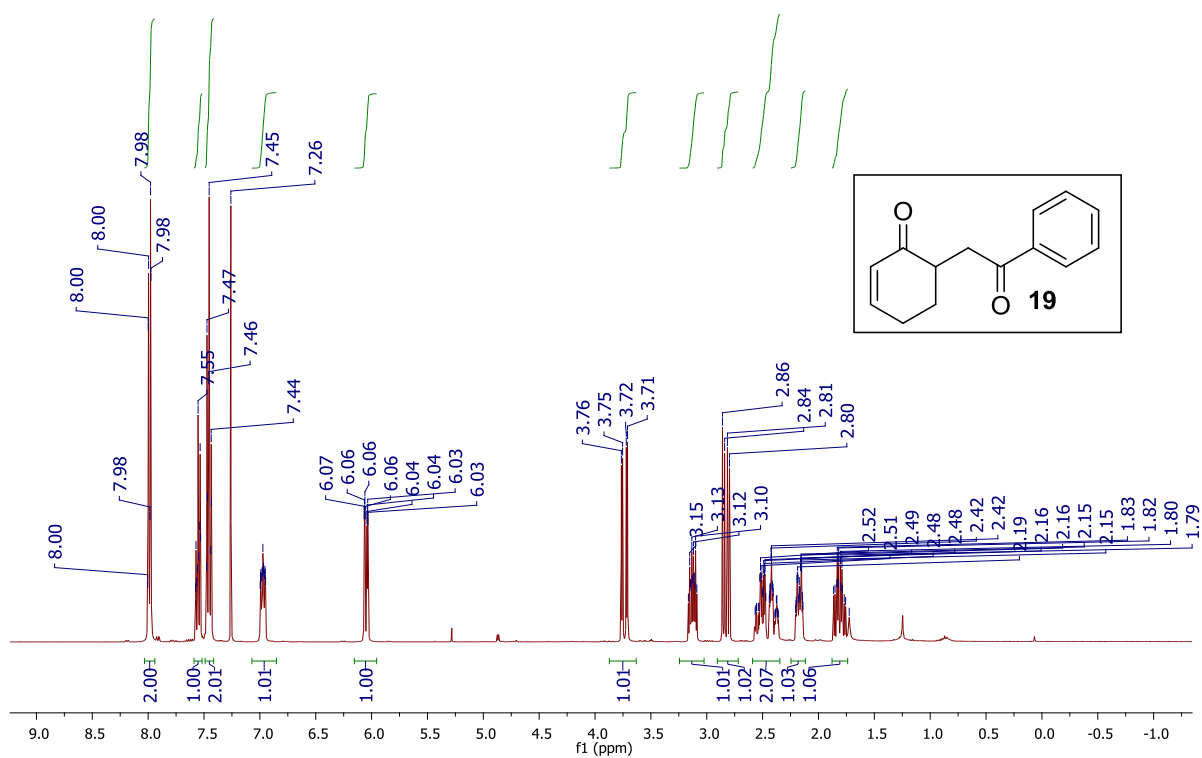
¹³C NMR of 17 (CDCl₃, 101 MHz)



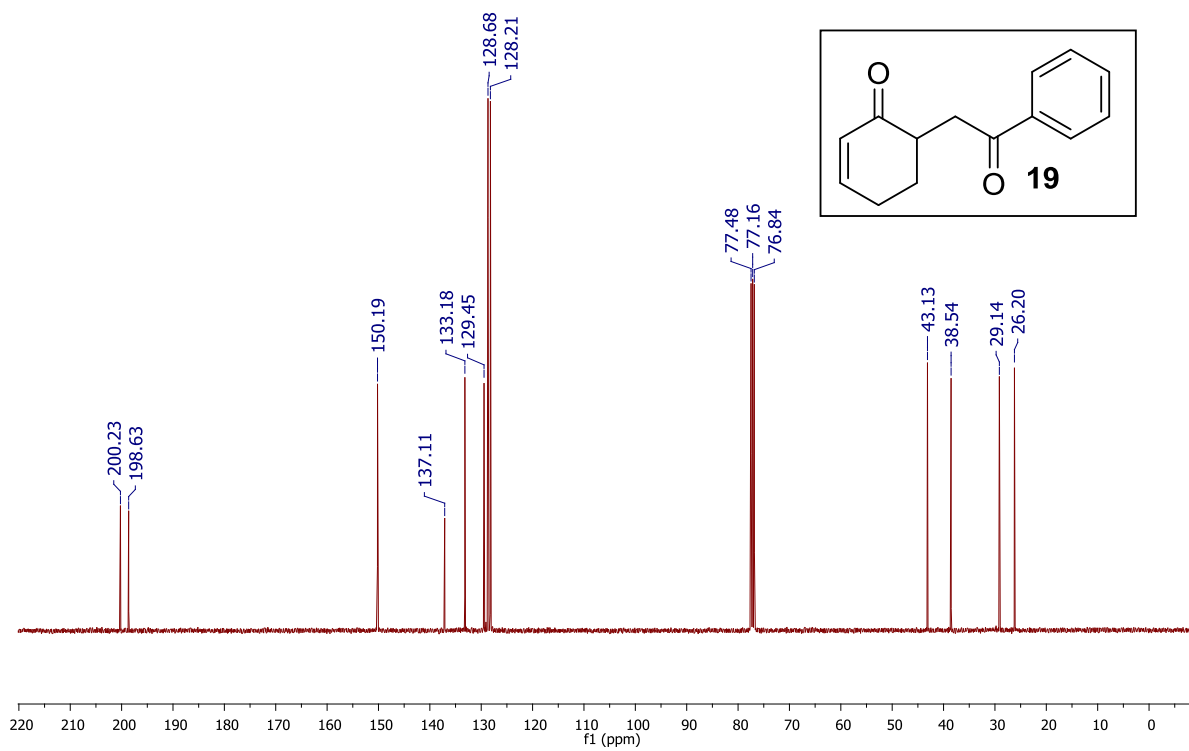
¹H NMR of **18** (CDCl₃, 400 MHz)



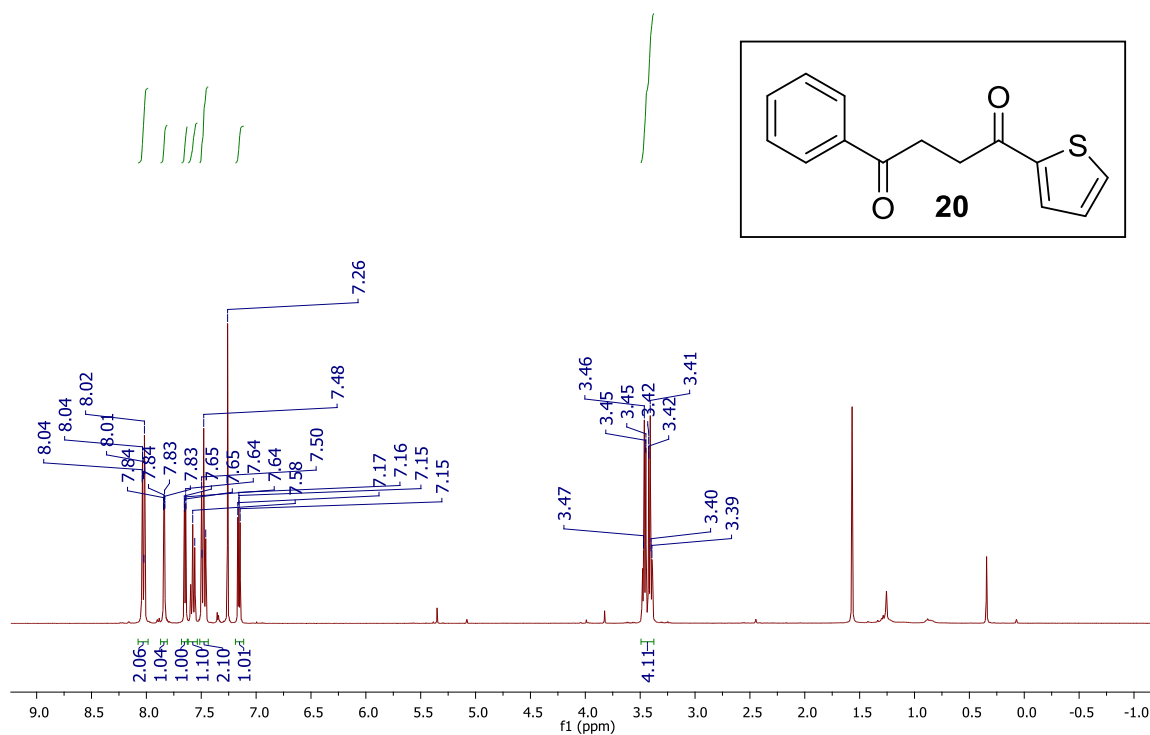
¹³C NMR of **18** (CDCl₃, 101 MHz)



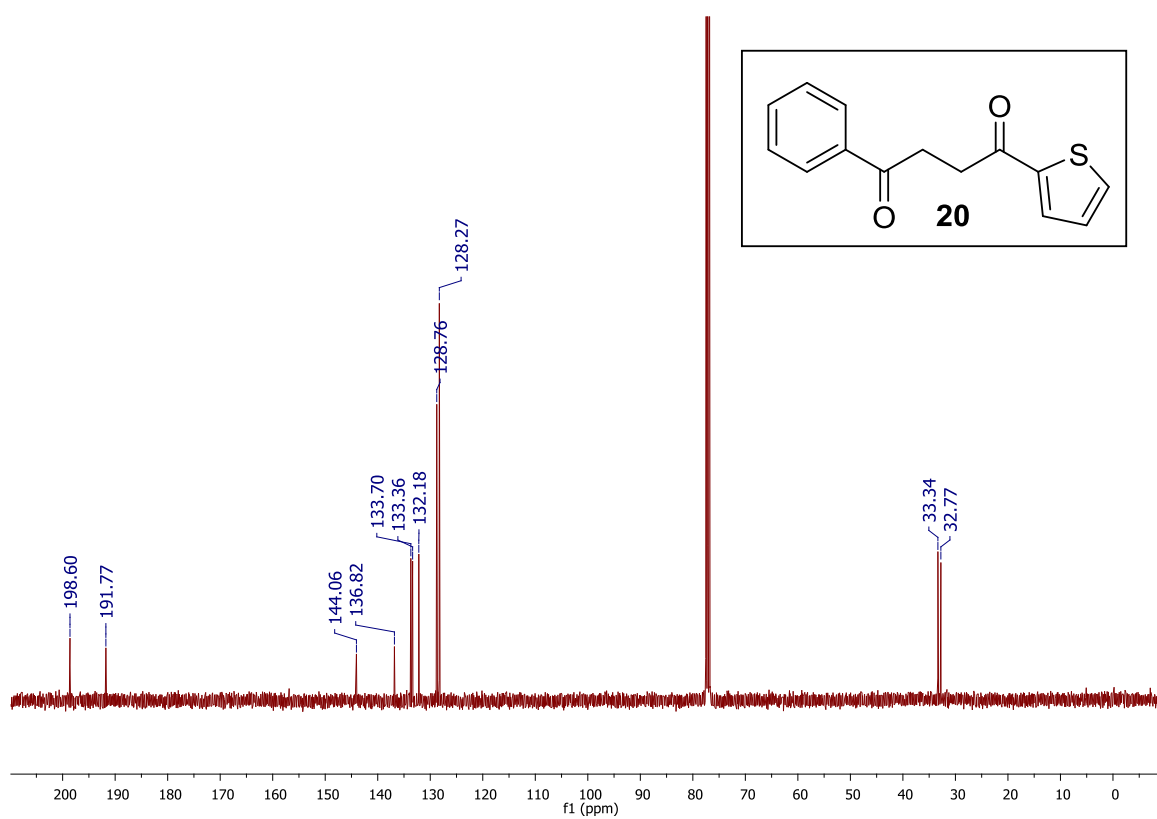
¹H NMR of **19** (CDCl₃, 400 MHz)



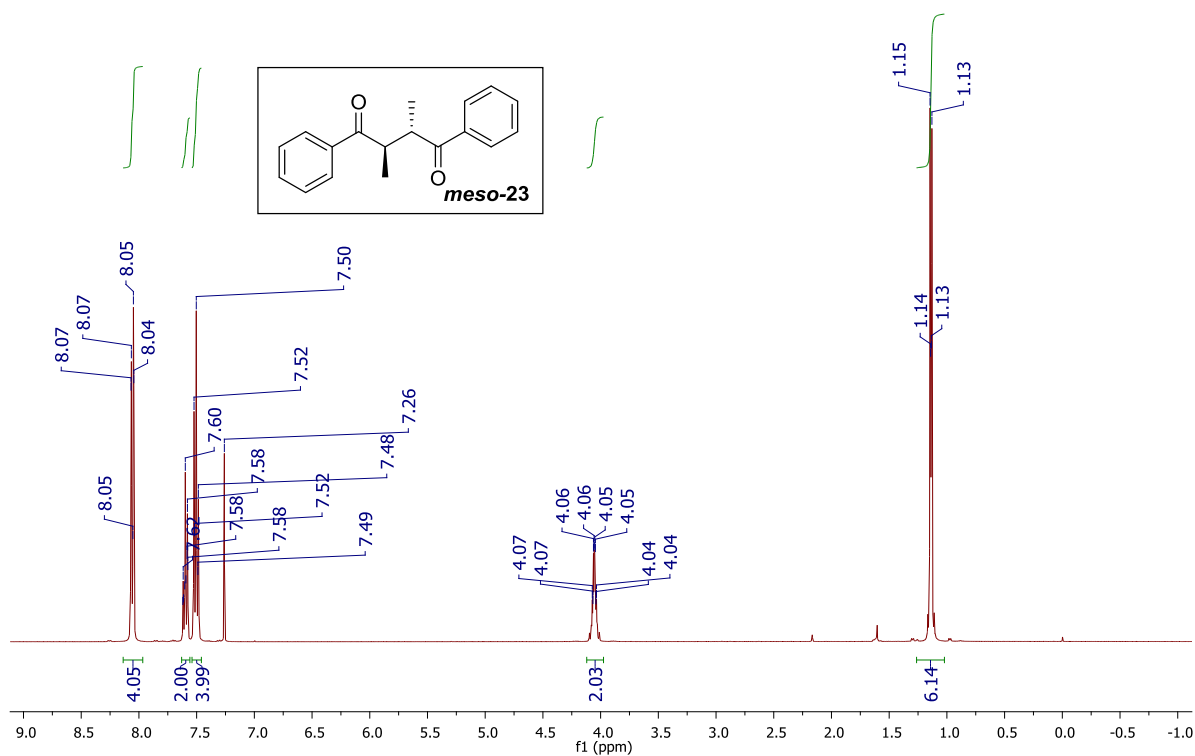
¹³C NMR of **19** (CDCl₃, 101 MHz)



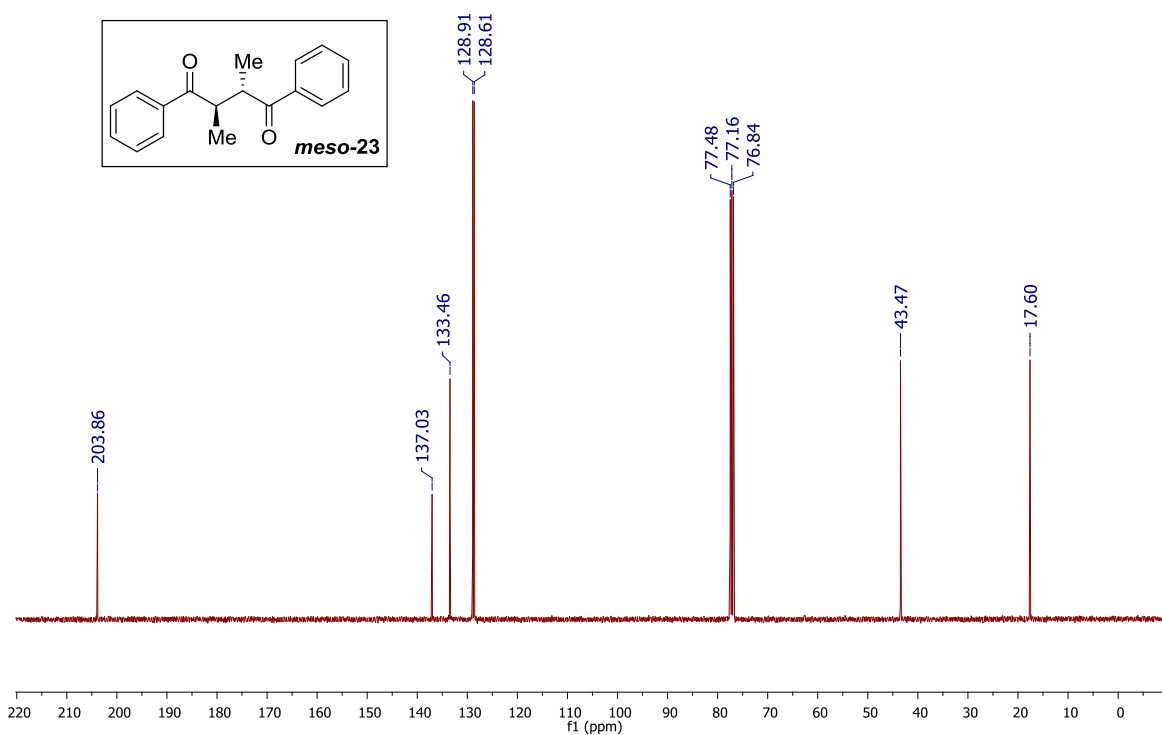
¹H NMR of **20** (CDCl₃, 400 MHz)



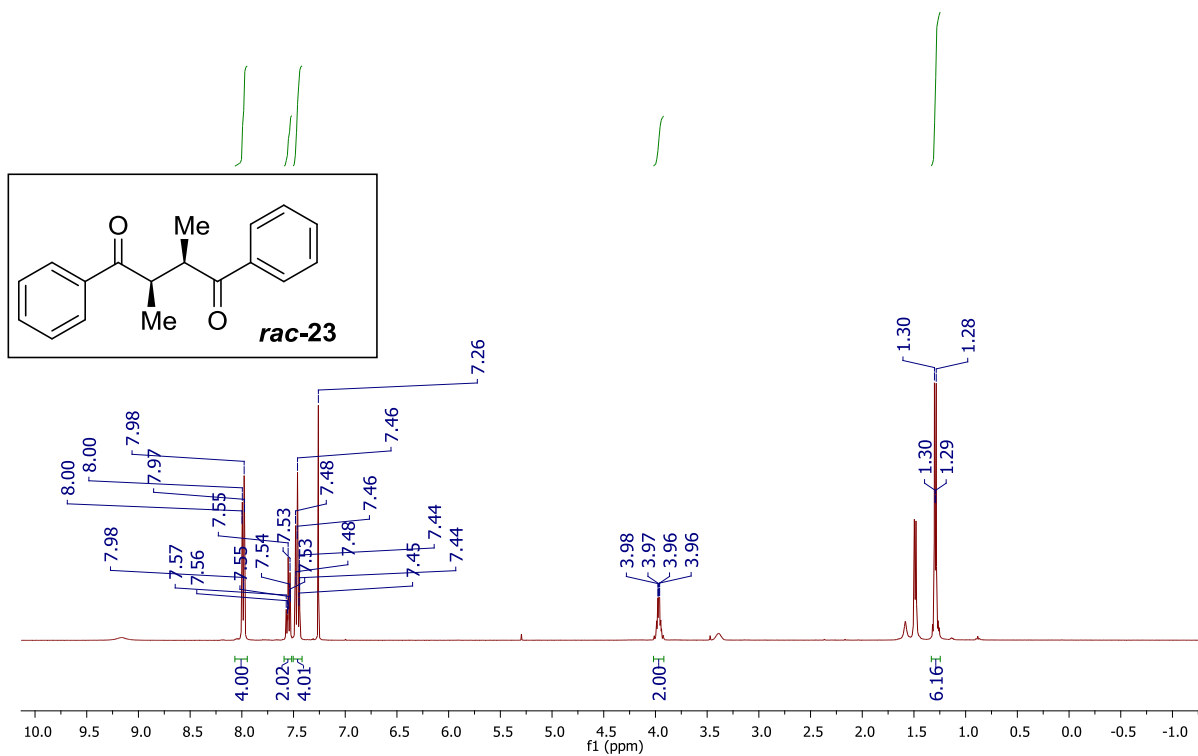
¹³C NMR of **20** (CDCl₃, 101 MHz)



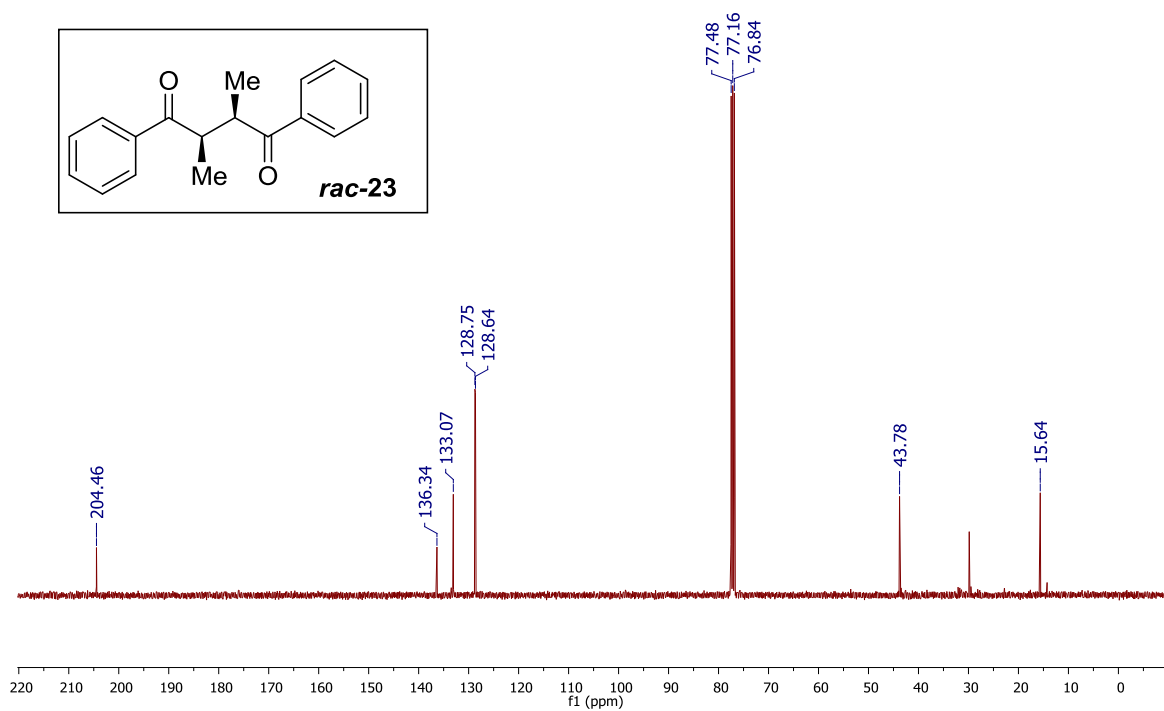
¹H NMR of **23** (CDCl₃, 400 MHz)



¹³C NMR of **23** (CDCl₃, 101 MHz)



¹H NMR of **23** (CDCl₃, 400 MHz)



¹³C NMR of **23** (CDCl₃, 101 MHz)