Journal of the Serbian Chemical Society
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J. Serb. Chem. Soc. 91 (7–8) S174–S191 (2026) *Supplementary material*

SUPPLEMENTARY MATERIAL TO

Synthetic studies of lignanes: Attempted synthesis of arctigenin and enterolactone

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J. Serb. Chem. Soc. 91 (7–8) (2026) 665–676

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GENERAL EXPERIMENTAL

All chromatographic separations[1] were performed on Silica gel 60 (0.063-0.2 mm), Merck. Standard techniques were used for the purification of reagents and solvents.[2] NMR spectra were recorded on Varian/Agilent 400 (^{1}H NMR at 400 MHz, ^{13}C NMR at 100 MHz) and on Bruker Avance III 500 (^{1}H NMR at 500 MHz, ^{13}C NMR at 125 MHz), in deuterated chloroform, if not otherwise stated. Chemical shifts are expressed in ppm (δ) using tetramethylsilane as internal standard, coupling constants (*J*) are in Hz. IR spectra were recorded on Thermo Scientific Nicolet Summit FT-IR instrument, and are expressed in cm^{-1}. Mass spectra were obtained on Orbitrap Exploris 240 spectrometer. Blue light irradiation was performed using KessilPR160L 440 nm lamp (www.kessil.com).

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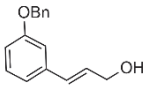
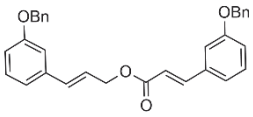
*(E)-3-(3-(benzyloxy) henyl)prop-2-en-1-ol (**12**)*



Ethyl chloroformate (0.54 mL, 5.64 mmol, 1 Eq) was added dropwise to a cold (−7 °C) solution of (*E*)-3-(3-(benzyloxy)phenyl)acrylic acid[3] (1.43 g, 5.64 mmol, 1 Eq) and triethylamine (1.57 mL, 11.27 mmol, 2 Eq) in dry THF (15 mL), under an argon atmosphere. After stirring for 30 minutes, reaction mixture was filtered and washed with additional THF (3 mL). $NaBH_4$ (0.64 g, 16.9 mmol, 3 Eq) was then added to the reaction mixture as one portion at 10 °C and MeOH (8.4 mL) was added dropwise. The suspension was stirred at 10 °C for 1 hour before quenched with water and extracted with DCM. The organic extract was dried over anhydrous $MgSO_4$ and concentrated on rotovap. The residue was purified by dry-flash chromatography (petroleum ether/ethyl acetate = 1:1), to afford 1.22 g (89%) of compound **12**, as a colorless oil. Spectroscopic data in accordance with the literature.[4]

*(E)-3-(3-(benzyloxy)phenyl)allyl (E)-3-(3-(benzyloxy)phenyl)acrylate (**7b**)*



(*E*)-3-(3-(benzyloxy)phenyl)acrylic acid[3] (129.7 mg, 0.52 mmol, 1 Eq), compound **12** (147.0 mg, 0.61 mmol, 1.2 Eq), DCC (126.0 mg, 0.61, 1.2 Eq) and DMAP (12.0 mg, 0.01, 0.2 Eq) were suspended in DCM (5.4 mL) and stirred for 30 minutes. The reaction mixture was filtered through Celite and purified by dry-flash chromatography (benzene/ethyl acetate = 99:1), to afford 243.0 g (100%) of pure **7b**, as a colorless oil.

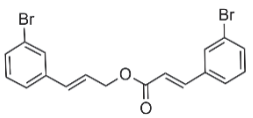
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, *J*=16.0, 1H), 7.49–7.24 (m, 12H), 7.18–7.14 (m, 2H), 7.08–7.00 (m, 3H), 6.92 (ddd, *J*= 8.2, 2.6, 1.0 Hz, 1H), 6.73–6.66 (m, 1H), 6.48 (dd, *J*= 16.0, 0.7 Hz, 1H), 6.37 (dt, *J* = 15.9, 6.4 Hz, 1H), 5.09 (d, *J*= 3.6 Hz, 4H), 4.89 (dd, *J* = 6.4, 1.3 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.7, 159.2, 159.1, 145.1, 137.8, 137.0, 136.7, 135.9, 134.2, 130.1, 129.7, 128.8, 128.7, 128.2, 128.1, 127.6, 123.7, 121.2, 119.7, 118.3, 117.2, 114.7, 114.1, 113.0, 70.2, 70.1, 65.2.

IR (ATR): ν = 3064, 3033, 2934, 2873, 1713, 1638, 1597, 1580, 1490, 1445, 1381, 1309, 1293, 1260, 1162, 1110, 1081, 1027, 979, 910 cm^{-1}.

R_f=0.8 (PhH/EtOAc=95:5).

*(E)-3-(3-bromophenyl)allyl (E)-3-(3-bromophenyl)acrylate (**7c**)*



(*E*)-3-(3-bromophenyl)acrylic acid[5] (100.0 mg, 0.44 mmol, 1 Eq), (*E*)-3-(3-bromophenyl)prop-2-en-1-ol[6] (112.5 mg, 0.53 mmol, 1.2 Eq), DCC (109.0 mg, 0.53, 1.2 Eq) and DMAP (11.0 mg, 0.09, 0.2 Eq) were dissolved in DCM (4.6 mL) and was stirred for 30 minutes. The reaction mixture was filtered through Celite and purified by dry-flash

chromatography (benzene/ethyl acetate = 99:1), to afford 168.0 g (90.4%) of pure **7c**, as a colorless oil.

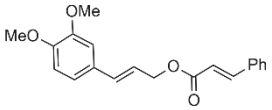
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69–7.61 (m, 2H), 7.57–7.54 (m, 1H), 7.51 (d, *J*= 8 Hz, 1H), 7.44 (d, *J*= 7.7 Hz, 1H), 7.39 (d, *J*= 8 Hz, 1H), 7.31 (d, *J*= 8 Hz, 1H), 7.29–7.23 (m, 1H), 7.19 (t, *J* = 7.8 Hz, 4H), 6.63 (d, *J* = 15.9 Hz, 3H), 6.47 (d, *J* = 16.0 Hz, 4H), 6.35 (dt, *J* = 15.9, 6.2 Hz, 4H), 4.87 (dd, *J* = 6.3, 1.4 Hz, 7H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 143.6, 138.4, 136.5, 133.3, 132.6, 131.0, 130.9, 130.5, 130.2, 129.6, 126.8, 125.4, 124.9, 123.2, 122.9, 119.3, 119.3, 65.0.

IR (ATR): $\tilde{\nu}$= 3060, 2925, 2854, 1714, 1639, 1591, 1473, 1424, 1374, 1312, 1268, 1198, 1166, 1111, 1089, 1072, 995 cm^{-1}.

R_f=0.9 (PhH/EtOAc=95:5).

*(E)-3-(3,4-dimethoxyphenyl)allyl cinnamate (**7e**)*



Cinnamic acid (32.0 mg, 0.21 mmol, 1 Eq), (*E*)-3-(3,4-dimethoxyphenyl)prop-2-en-1-ol (50.0 mg, 0.26 mmol, 1.2 Eq), DCC (53.0 mg, 0.26, 1.2 Eq) and DMAP (5.0 mg, 0.04, 0.2 Eq) were dissolved in DCM (2.5 mL) and was stirred for 30 minutes. The reaction mixture was filtered through Celite and purified by dry-flash chromatography (benzene/ethyl acetate = 9:1), to afford 31.6 g (45%) of pure **7e**, as colorless oil.

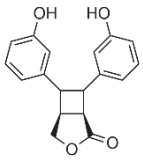
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 16.0 Hz, 1H), 7.54 – 7.49 (m, 2H), 7.40 – 7.37 (m, 3H), 6.99 – 6.93 (m, 2H), 6.83 – 6.79 (m, 1H), 6.68 – 6.62 (m, 1H), 6.48 (dd, *J* = 16.0, 0.6 Hz, 1H), 6.24 (dt, *J* = 15.8, 6.6 Hz, 1H), 4.86 (dd, *J* = 6.6, 1.3 Hz, 2H), 3.88 (dd, *J* = 8.8, 1.4 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ166.8, 149.3, 149.1, 145.1, 134.4, 134.4, 130.4, 129.3, 129.0, 128.1, 121.3, 120.1, 118.0, 111.1, 108.9, 65.4, 55.9, 55.9.

IR (ATR): $\tilde{\nu}$= 3060, 3001, 2936, 2836, 1712, 1637, 1602, 1582, 1515, 1464, 1451, 1419, 1376, 1328, 1310, 1202, 1165, 1141, 1073, 1027, 970 cm^{-1}.

Rf=0.6 (PhH/EtOAc=9:1).

*6,7-bis(3-hydroxyphenyl)-3-oxabicyclo[3.2.0]heptan-2-one (**13**)*



To a solution of **6b** (30.7 mg, 0.064 mmol, 1 Eq) in EtOAc (1.2 mL) was added $Pd(OH)_2$ on carbon (12 mg, 0.013 mmol, 0.2 Eq) and stirred overnight under H_2 atmosphere (1 bar). After reaction was completed, reaction mixture was filtred throught pad of Celite and purified by dry-flash chromatography (petroleum ether/ethyl acetate = 1:1), to afford 13.2 g (70%) of **13** as a mixture of isomers in a ratio 2.7:1, as a colorless oil.

Major isomer: **^{1}H NMR** (500 MHz, $CDCl_3$) δ6.95 (t, J = 7.8 Hz, 1H), 6.89 (t, J = 7.8 Hz, 1H), 6.56 (d, J = 7.5 Hz, 1H), 6.53 – 6.48 (m, 2H), 6.47 – 6.42 (m, 2H), 6.42 – 6.39 (m, 1H), 4.58 (dd, J = 9.5, 6.3 Hz, 1H), 4.42 (dd, J = 9.5, 1.4 Hz, 1H), 4.03 – 3.97 (m, 1H), 3.95 (dd, J = 9.5, 3.2 Hz, 1H), 3.70 (q, J = 7.1 Hz, 1H), 3.39 (ddd, J = 7.9, 3.2, 1.1 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 182.4, 158.1, 158.0, 141.9, 141.5, 130.0, 129.8, 120.4, 120.2, 116.1, 115.8, 114.4, 114.2, 74.7, 42.3, 39.9.

IR (ATR): ν~= 3370, 2968, 2927, 2854, 1740, 1588, 1493, 1454, 1376, 1313, 1257, 1228, 1172, 1086, 1047 999 cm^{-1}.

HRMS (ESI-Orbitrap) *m/z*: $[M+Na]^+$ calcd. for $C_{18}H_{16}O_4$: 319.0941, found: 319.0940.

R_f=0.5 (PE /EtOAc=6:4).

Experimental setup for photocatalyzed [2+2] cycloaddition

Borosilicate culture tube (8 mL, round bottom, screw cap finish 13-415; height 100 mm, diameter 13 mm) was used as the reaction vessel. The distance between reaction vessel and light sourse was 3 cm. The lamp was cooled using Sunon 92x92x25 12VDC (1.68 W) fan. With this experimental setup, temperature reaches 55 °C, as measured using thermometer placed next to the reaction vessel.

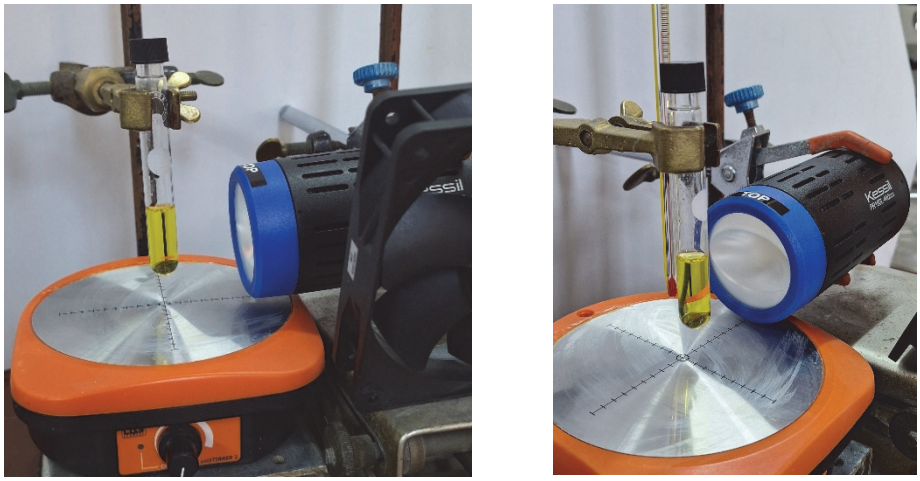


Figure S2 Blue LED setup

*6,7-Diphenyl-3-oxabicyclo[3.2.0]heptan-2-one (**6a**)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42−7.27, 7.22−7.01, 7.00−6.95, 6.88−6.84 (4 x m, 10H major and 10H minor), 4.56 (dd, J = 9.7, 6.1 Hz, 1H), 4.48−4.37 (m, 1H major and 1H minor), 4.27 (dd, J = 10.2, 4.9 Hz, 1H minor), 4.20−4.07 (m, 2H major and 2H minor), 3.71 (q, J = 6.7 Hz, 1H major), 3.60−3.50 (m, 1H minor), 3.47−3.41 (m, 1H major), 3.35−3.28 (m, 1H minor). Major and minor: **^{13}C NMR** (101 MHz, $CDCl_3$) δ 179.5, 178.4, 141.7, 138.7, 138.5, 138.1, 129.0, 128.9, 128.2, 128.1, 128.0, 127.7, 127.3, 127.2, 127.1, 126.7, 126.6, 126.4, 48.2, 47.7, 46.7, 45.9, 42.6, 40.6, 39.0, 35.4.

Cis-*(3*R*,4*R*)-dihydro-3,4-bis(phenylmethyl)-2(3H)-furanone (**5a**)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40−7.19 (m, 8H), 7.05−7.01 (m, 2H), 4.07−3.98 (m, 2H), 3.35 (dd, J = 15.0, 4.7 Hz, 1H), 3.13 (ddd, J = 11.4, 7.1, 4.6 Hz, 1H), 3.00 (dd, J = 13.6, 3.9 Hz, 1H), 2.85 (dd, J = 14.9, 10.9 Hz, 1H), 2.73 – 2.63 (m, 1H), 2.44 – 2.34 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 178.1, 138.7, 138.6, 129.1, 129.0, 128.9, 128.5, 126.9, 126.8, 69.5, 45.4, 40.0, 33.0, 31.0.

*Trans-(3R,4R)-dihydro-3,4-bis(phenylmethyl)-2(3H)-furanone (**4a**)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34−7.14 (m, 8H), 7.02−6.97 (m, 2H), 4.11−4.05 (m, 1H), 3.89−3.83 (m, 1H), 3.12−3.06 (m, 1H), 2.96 (dd, J = 14.0, 7.1 Hz, 1H), 2.70–2.57 (m, 2H), 2.56–2.46 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 178.6, 138.1, 137.8, 129.4, 129.1, 128.9, 128.7, 128.5, 127.0, 126.9, 77.5, 77.4, 77.2, 76.8, 71.3, 46.6, 46.4, 41.4, 41.3, 38.6, 35.2, 35.1.

*6,7-Bis(3-(benzyloxy)phenyl)-3-oxabicyclo[3.2.0]heptan-2-one (**6b**)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46−7.28, 7.11−7.02, 6.98−6.86, 6.80−6.68, 6.64−6.59, 6.51−6.46 (6 x m, 18H major and 18H minor), 5.06, 4.90, 4.89, 4.86 (4 x bs, 4H major and 4H minor), 4.51 (dd, J = 9.7, 6.1 Hz, 1H major), 4.40 (d, J = 9.7 Hz, 1H major), 4.48−4.32 (m, 1H minor), 4.25 (dd, J = 10.3, 5.0 Hz, 1H minor), 4.17−4.02 (m, 2H major and 2H minor), 3.60 (q, J = 6.9 Hz, 1H), 3.52−3.44 (m, 1H minor), 3.39 (dd, J = 8.0, 2.6 Hz, 1H), 3.28 (t, J = 7.2 Hz, 1H minor). Major and minor: **^{13}C NMR** (101 MHz, $CDCl_3$) δ 179.4, 178.2, 159.3, 159.2, 158.8, 158.7, 143.3, 140.4, 140.2, 139.9, 137.0, 130.1, 130.0, 129.3, 129.1, 128.7, 128.2, 128.1, 127.7, 127.6, 127.5, 120.7, 120.5, 119.6, 119.0, 115.0, 114.6, 114.1, 113.3, 113.2, 72.7, 70.1, 70.1, 69.1, 48.1, 47.6, 46.7, 45.7, 42.5, 40.6, 39.2, 35.3. **IR** (ATR): ν = 3372, 3063, 3032, 2907, 2871, 1765, 1602, 1583, 1491, 1452, 1378, 1316, 1293, 1263, 1162, 1081, 1045, 1027, 915 cm^{-1}. **HRMS** (ESI-Orbitrap) *m/z*: $[M+K]^+$ calcd. for $C_{32}H_{28}O_4K$: 515.1619, found: 515.1622. R_f=0.5 (PE /EtOAc=6:4).

*6,7-Bis(3-bromophenyl)-3-oxabicyclo[3.2.0]heptan-2-one (**6c**)*

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46−7.39, 7.31−7.28, 7.26−7.19, 7.18−7.14, 7.13−7.09, 7.05−6.95, 6.89−6.84, 6.77−6.72 (8 x m, 8H major and 8H minor), 4.56 (dd, J = 9.8, 6.1 Hz, 1H), 4.45−4.37 (m, 1H major and 1H minor), 4.22 (dd, J = 10.4, 4.7 Hz, 1H minor), 4.15–4.03 (m, 2H major and 2H minor), 3.73 – 3.63 (m, 1H major), 3.54 (m, 1H minor), 3.40 (dd, J = 8.0, 1.7 Hz, 1H major), 3.29 (dd, J = 7.9, 6.5 Hz, 1H minor). Major and minor: **^{13}C NMR** (101 MHz, $CDCl_3$) δ 178.7, 143.5, 140.4, 140.0, 130.9, 130.8, 130.7, 130.1, 130.0, 129.9, 129.8, 129.3, 126.7, 126.4, 125.2, 122.6, 122.5, 72.6, 68.9, 47.7, 47.3, 46.1, 45.4, 42.4, 40.3, 38.7, 35.3. **IR** (ATR): ν = 3373, 3061, 2967, 2906, 1764, 1594, 1566, 1476, 1428, 1372, 1332, 1251, 1165, 1094, 1047, 997 cm^{-1}. **HRMS** (ESI-Orbitrap) *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{14}Br_2O_2$: 420.9433, found: 420.9433. R_f=0.4 (PE/EtOAc=7:3).

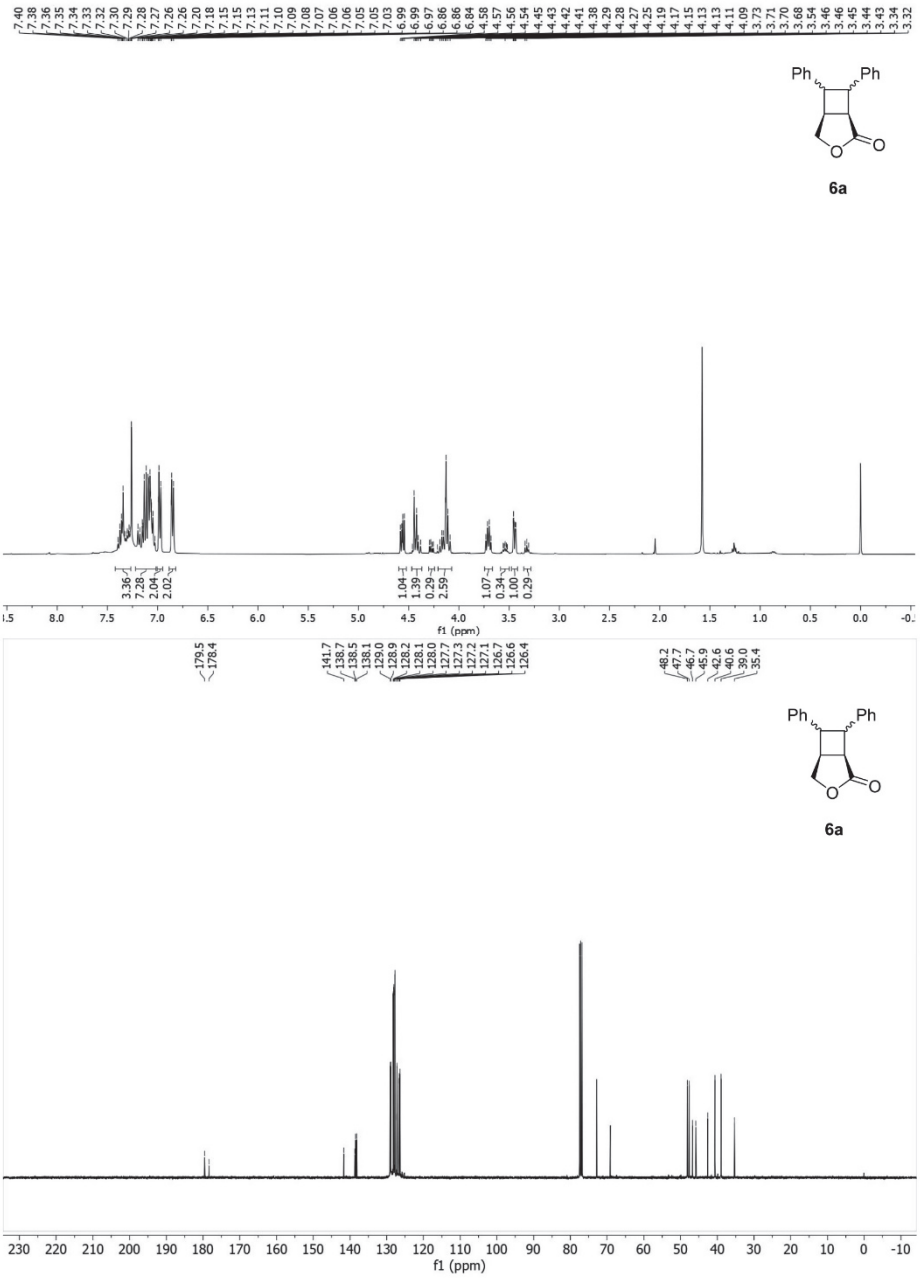
*6-(3,4-Dimethoxyphenyl)-7-phenyl-3-oxabicyclo[3.2.0]heptan-2-one (**6e**)*

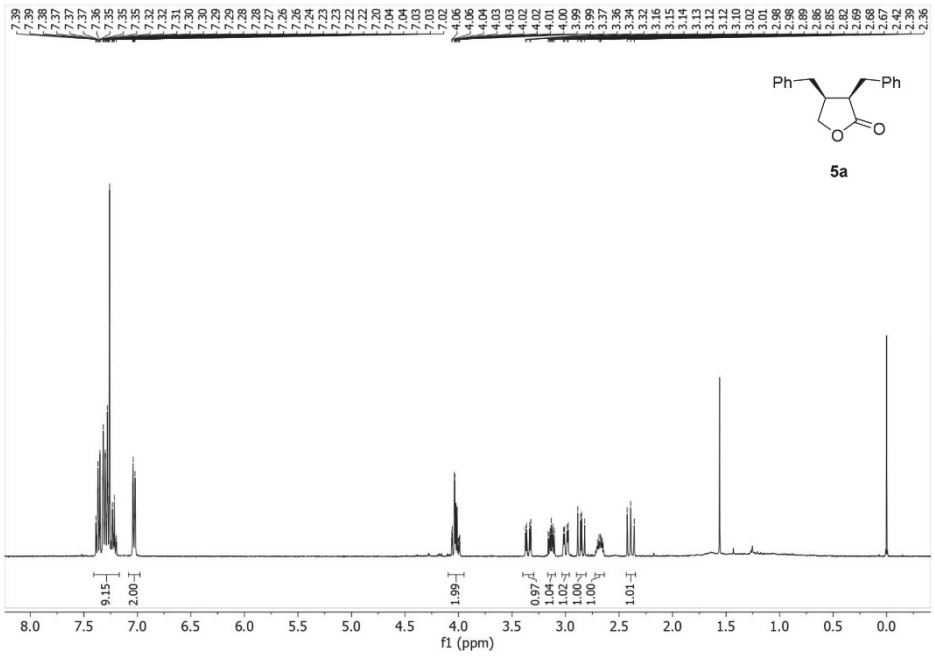
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.31 (m, 5H), 7.19–7.14 (m, 2H), 7.13–7.10 (m, 1H), 7.02–6.98 (m, 2H), 6.89–6.86 (m, 1H), 6.77 (dd, J = 8.3, 2.1 Hz, 1H), 6.66 (d, J = 2.1 Hz, 1H), 6.63 (d, J = 8.2 Hz, 1H), 6.50 (dd, J = 8.4, 2.1 Hz, 1H), 6.16 (d, J = 2.1 Hz, 1H), 4.53 (dd, J = 9.6, 6.0 Hz, 1H), 4.44 – 4.39 (m, 1H), 4.39 – 4.36 (m, 1H), 4.29 (dd, J = 10.2, 4.7 Hz, 1H), 4.12 – 4.09 (m, 3H), 4.02 (t, J = 8.5 Hz, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 3.77 (s, 3H), 3.63 – 3.57 (m, 1H), 3.56 (s, 3H), 3.53 – 3.44 (m, 2H), 3.34 – 3.29 (m, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ179.6, 178.6, 149.5, 148.6, 148.4, 147.9, 141.9, 138.6, 131.1, 130.9, 128.9, 128.7, 128.4, 128.0, 127.3, 126.8, 126.4, 120.1, 119.9, 119.4, 111.6, 111.4, 110.6, 110.5, 72.7, 69.1, 66.0, 56.1, 55.9, 55.8, 48.0, 47.8, 47.0, 46.1, 42.4, 40.3, 40.1, 35.6, 15.4, 0.1. **IR** (ATR): ν~= 3060, 2959, 2935, 2908, 2836, 1763, 1670, 1641, 1590, 1517, 1464, 1419, 1372, 1333, 1258, 1163, 1080, 1027, 912 cm^{-1}. **HRMS** (ESI-Orbitrap) *m/z*: $[M+K]^+$ calcd. for $C_{20}H_{20}O_4K$: 363.0993, found: 363.0997. **R_f**=0.6 (PhH/EtOAc=8:2).

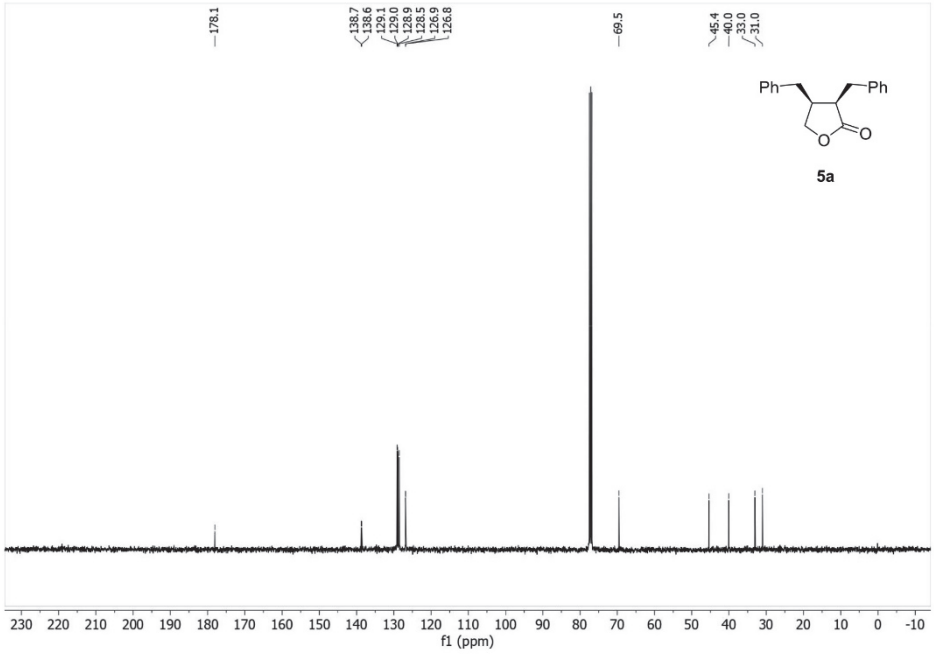
*(2-Oxo-3-oxabicyclo[3.2.0]heptane-6,7-diyl)bis(3,1-phenylene) diacetate (**6o**)*

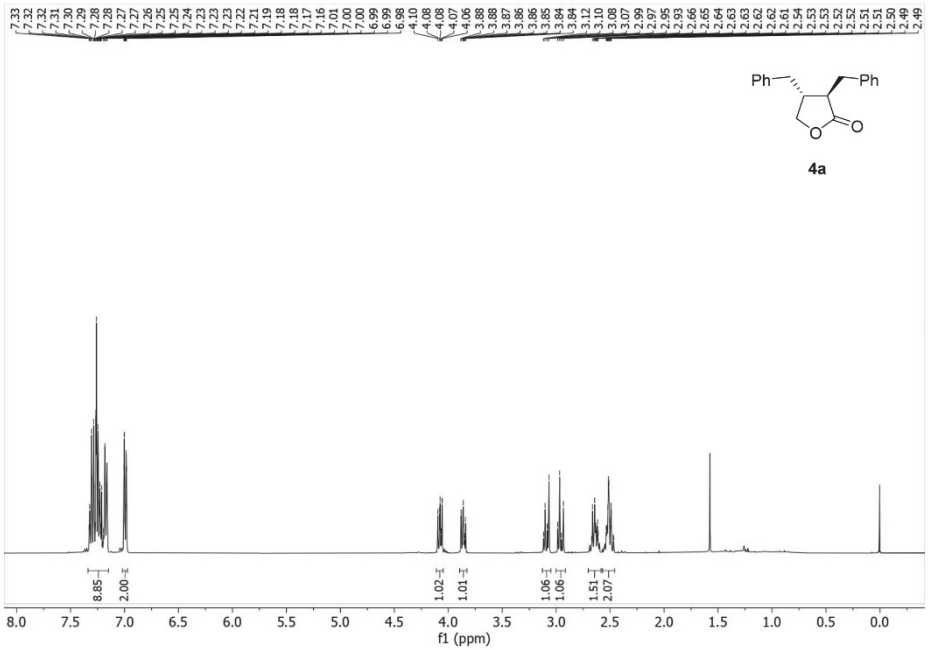
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42−7.32, 7.20−7.25, 7.15−7.07, 7.07−6.87, 6.85−6.74, 6.73−6.68, 6.65−6.63 (7 x m, 8H major and 8H minor), 4.55−4.48, 4.43−4.37, 4.28−4.23, 4.19−4.06 (4 x m, 4H major and 4H minor), 3.70−3.62 (m, 1H major), 3.56−3.48 (m, 1H minor), 3.44−3.38 (m, 1H major), 3.34−3.28 (m, 1H minor), 2.31−2.27 (m, 6H minor), 2.26−2.22 (m, 6H major). Major and minor: **^{13}C NMR** (101 MHz, CDCl3) {delta} 179.0, 178.0, 169.4, 169.3, 151.3, 151.2, 150.8, 150.7, 143.2, 140.2, 139.9, 139.5, 130.2, 130.0, 129.4, 129.2, 125.6, 125.3, 124.5, 123.9, 121.1, 121.0, 120.7, 120.4, 120.0, 119.5, 72.6, 69.0, 47.9, 47.4, 46.3, 45.3, 42.4, 40.4, 39.1, 35.4, 21.3, 21.2. **IR** (ATR): ν~= 3060, 2927, 2853, 1765, 1609, 1587, 1444, 1370, 1208, 1154, 1088, 1046, 1015, 921 cm^{-1}. **HRMS** (ESI-Orbitrap) *m/z*: $[M+H]^+$ calcd. for $C_{22}H_{20}O_6$: 381.1332, found: 381.1326. ***R_f***=0.4 (PE /EtOAc=1:1).

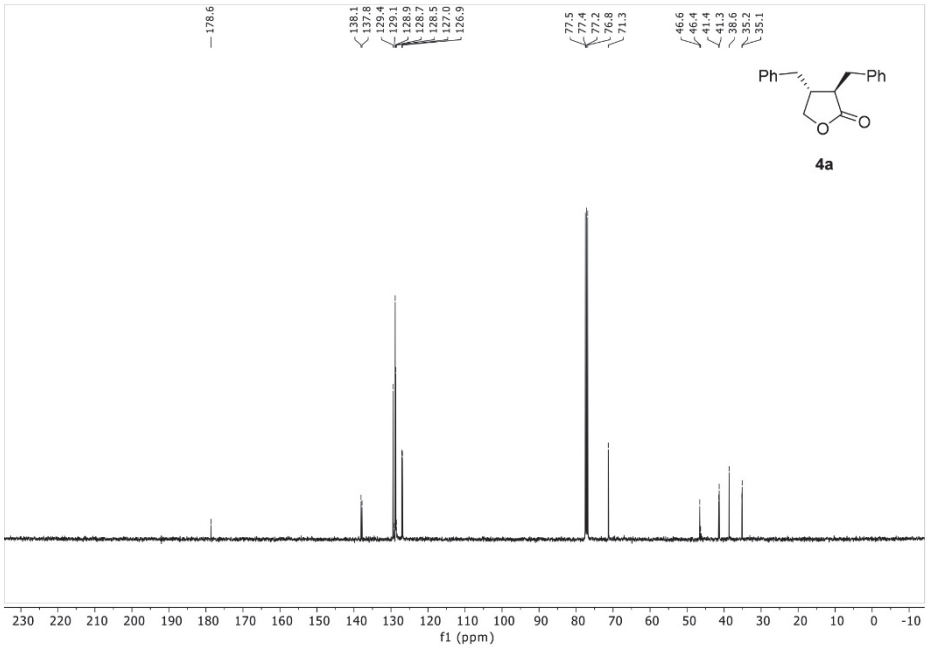
COPIES OF NMR SPECTRA

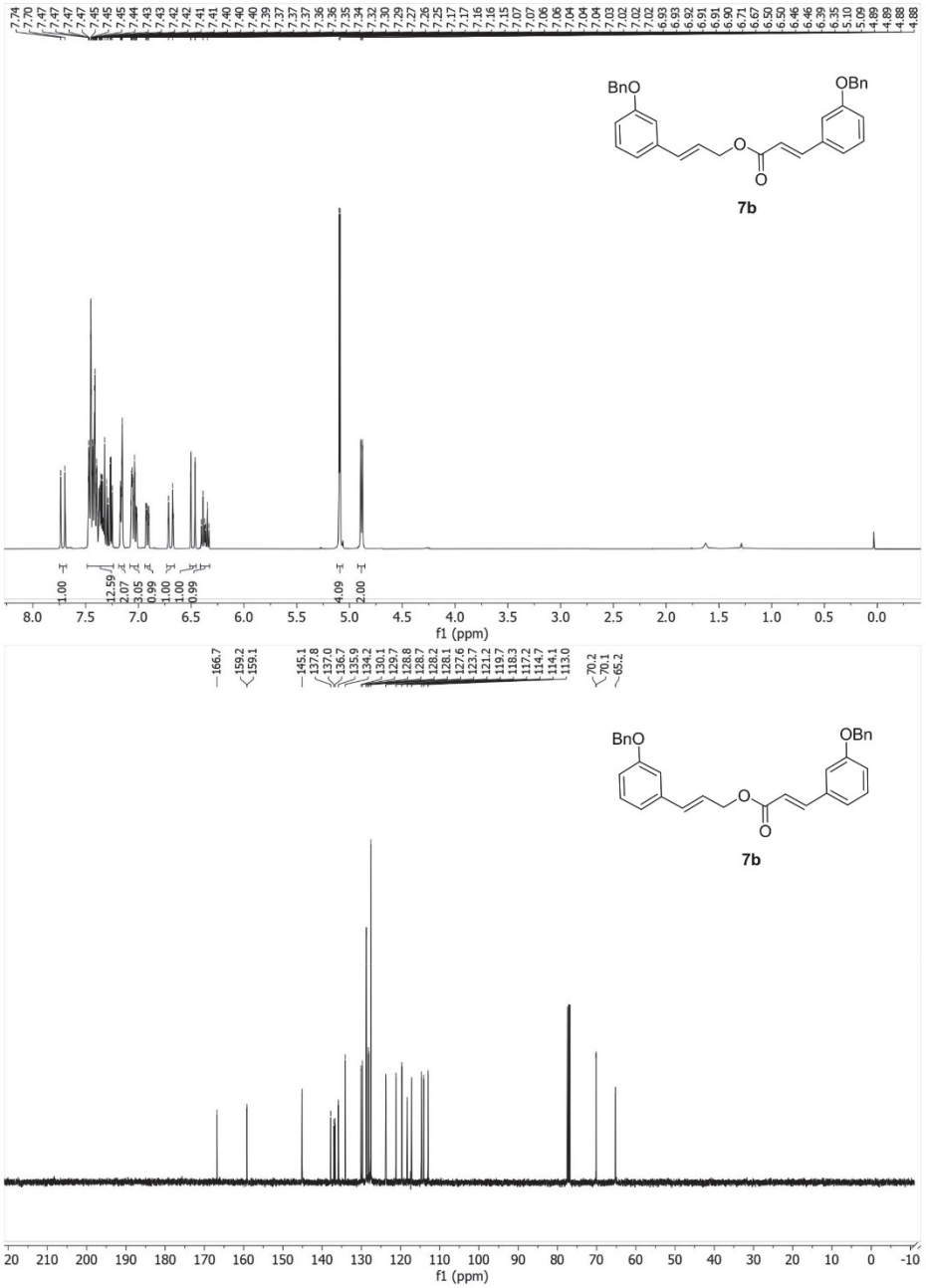


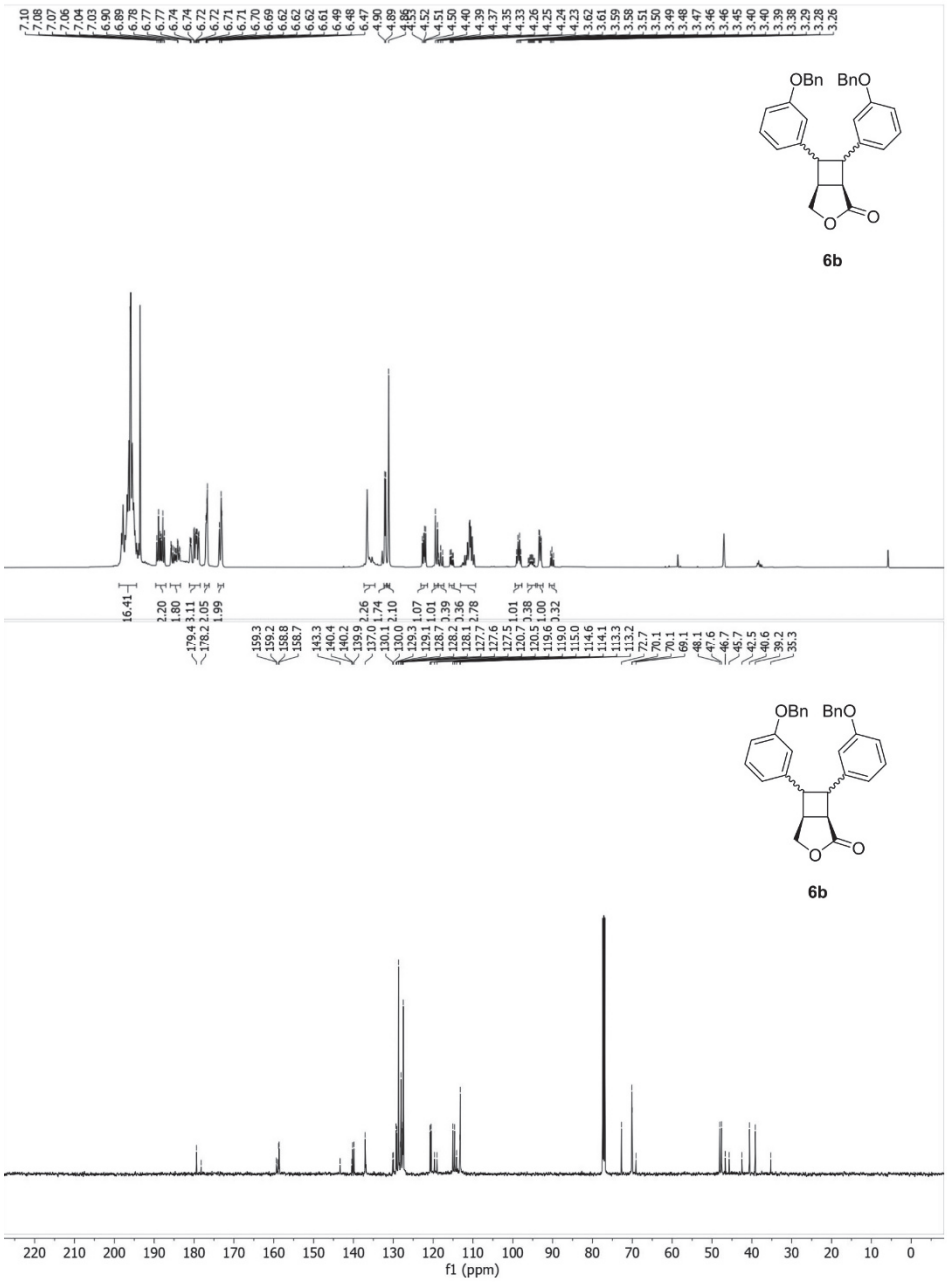


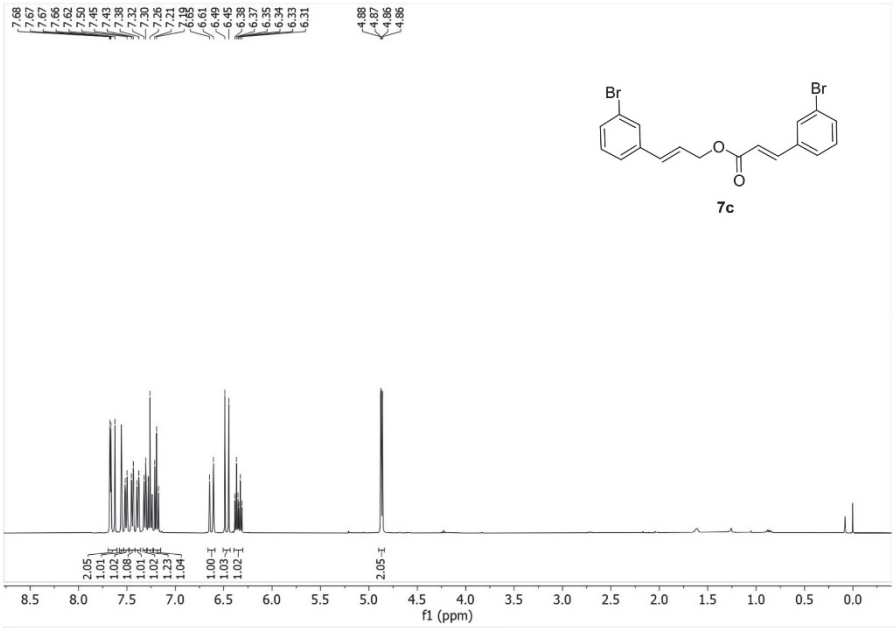


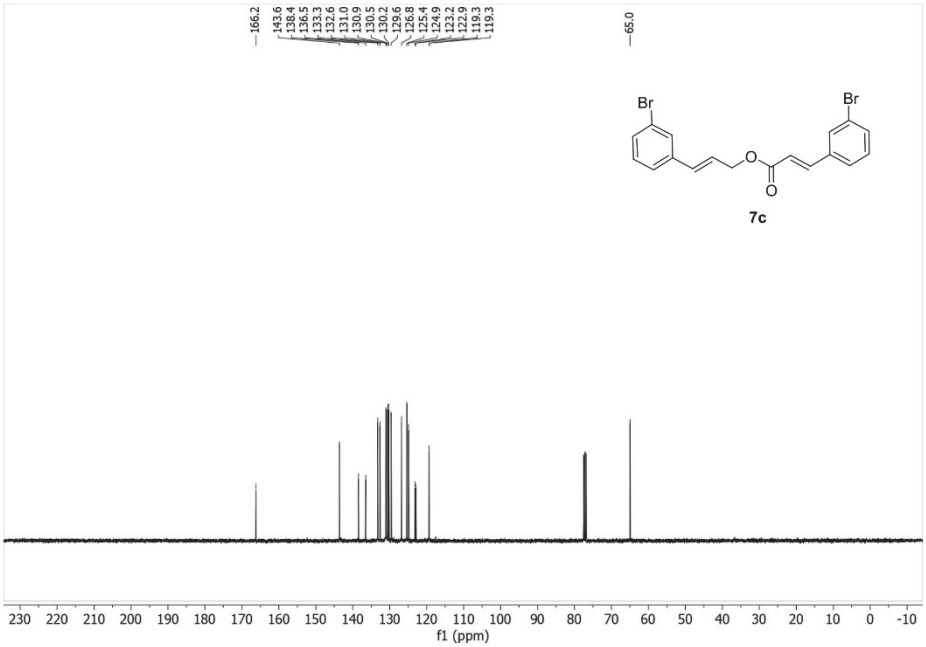


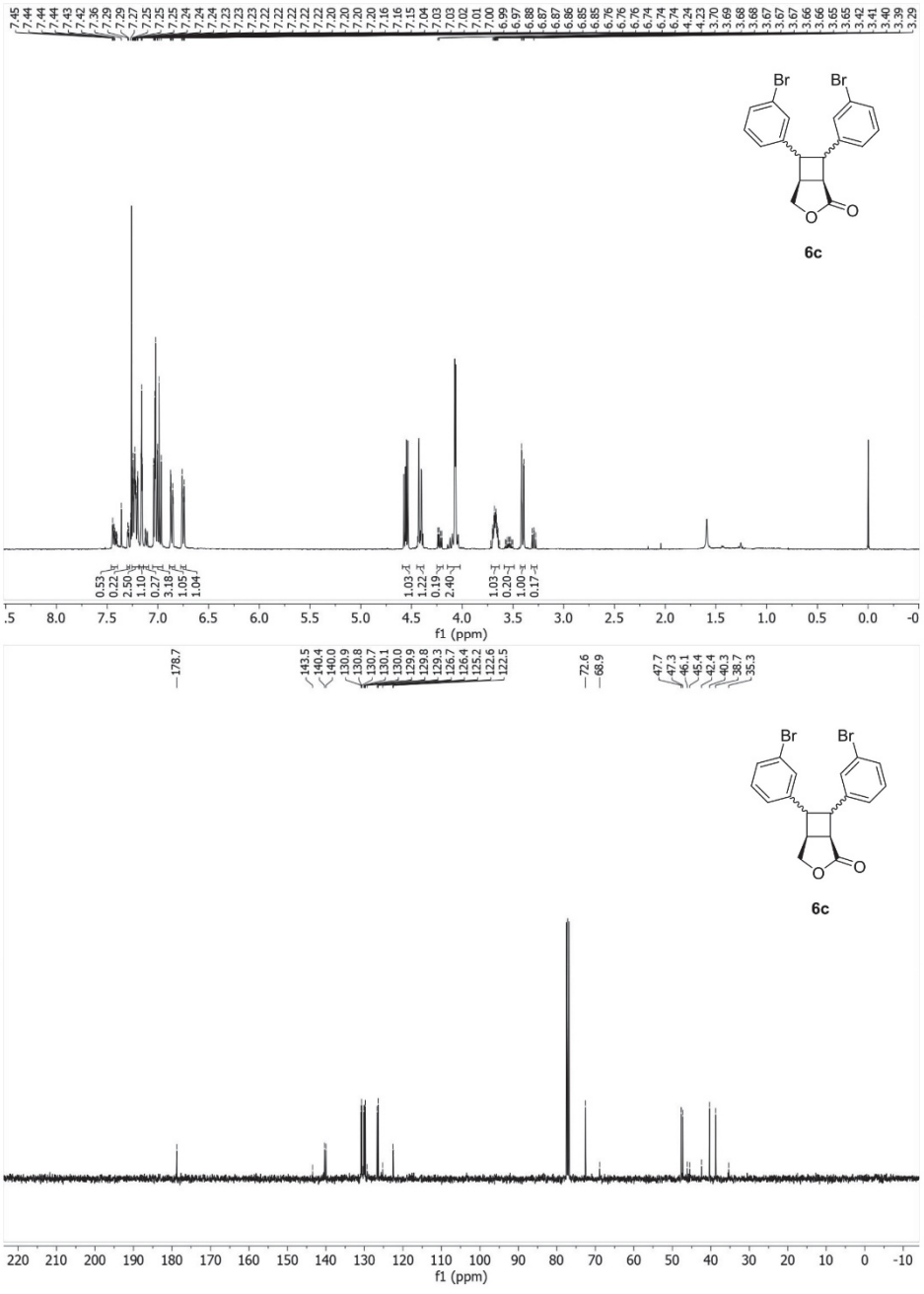


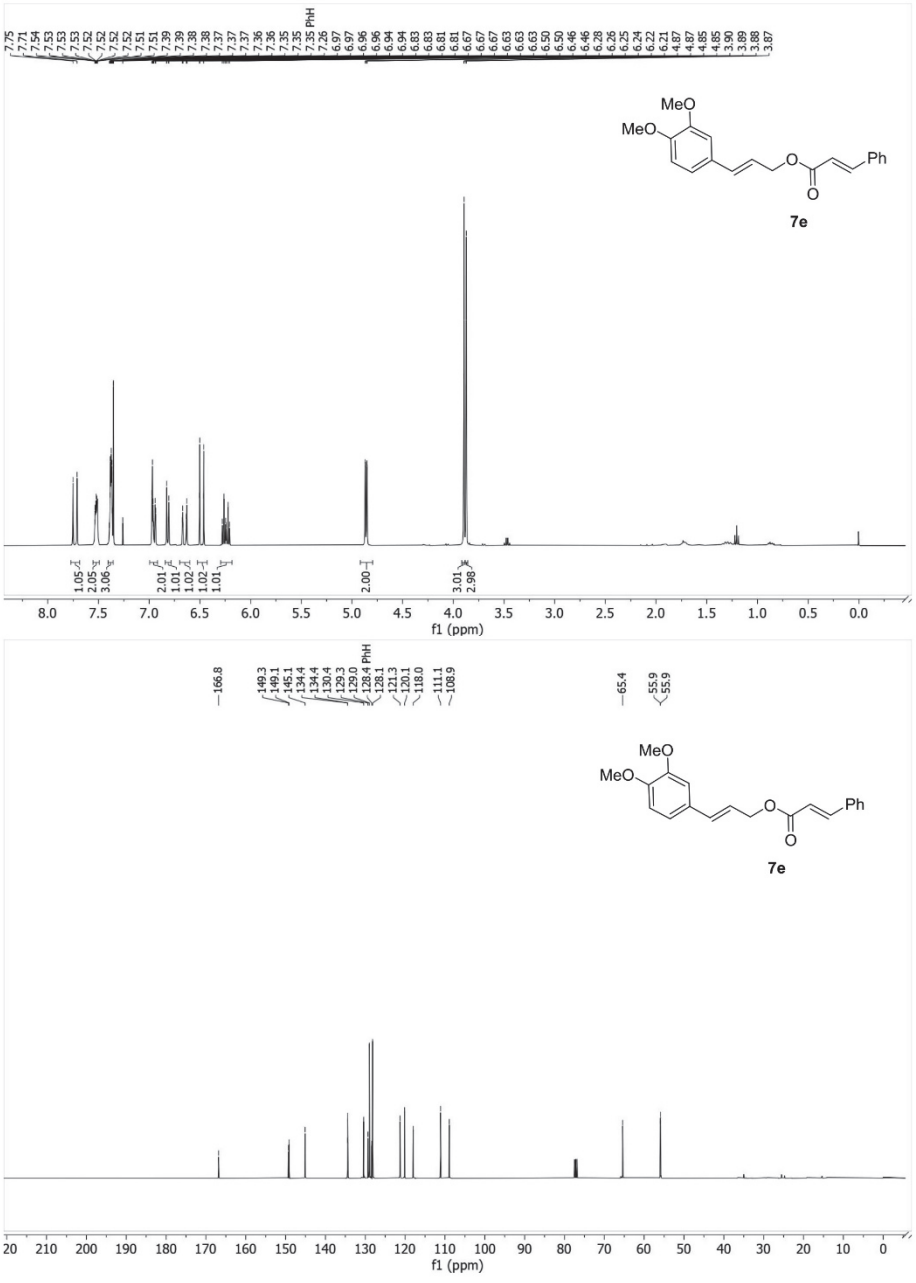


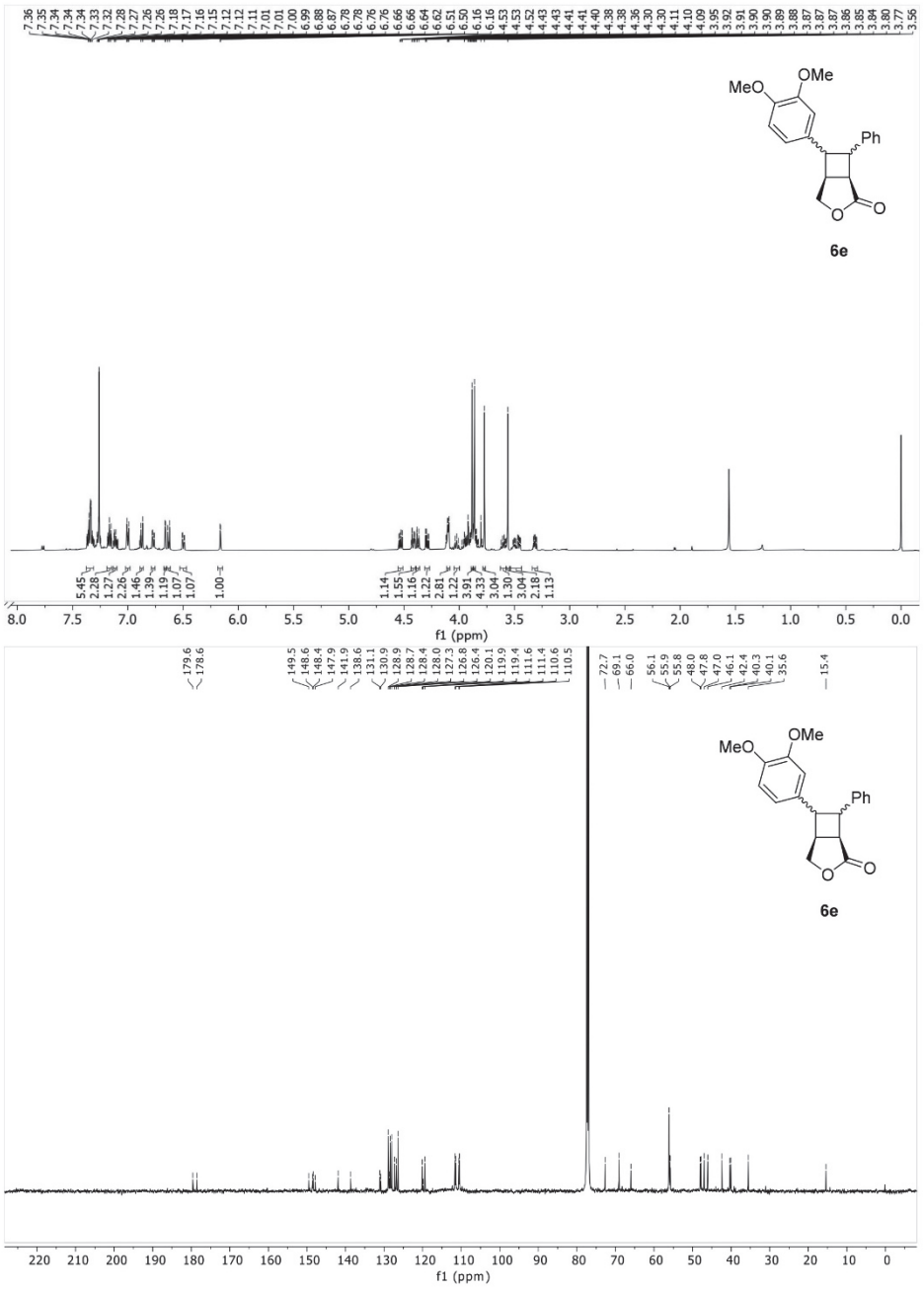


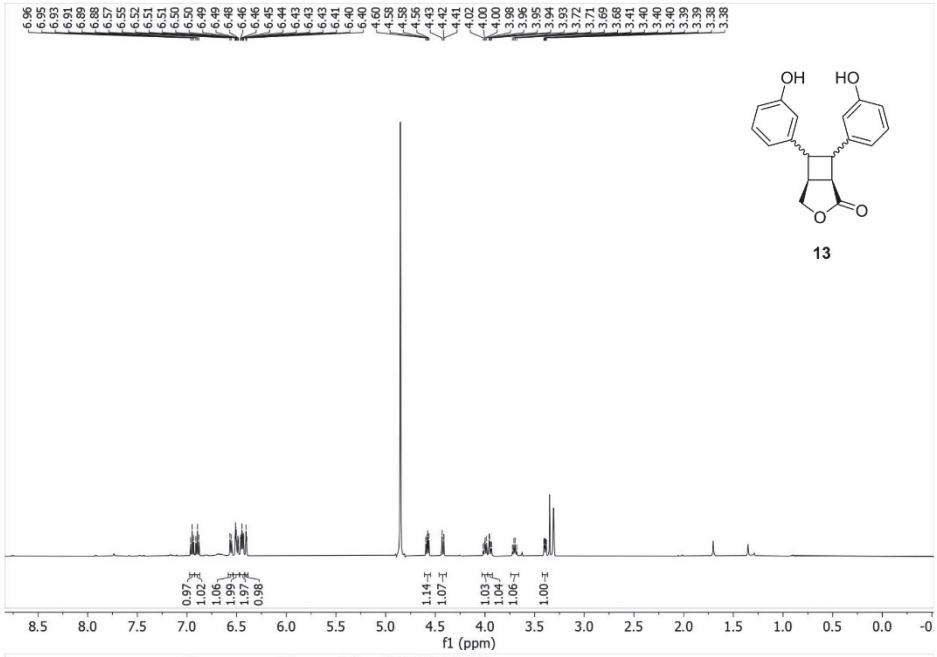


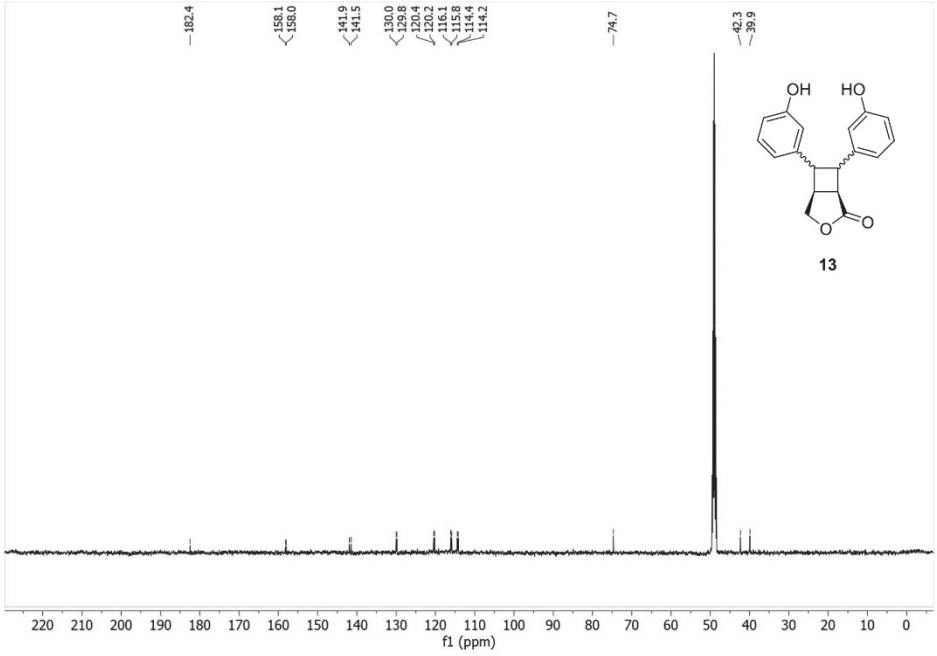


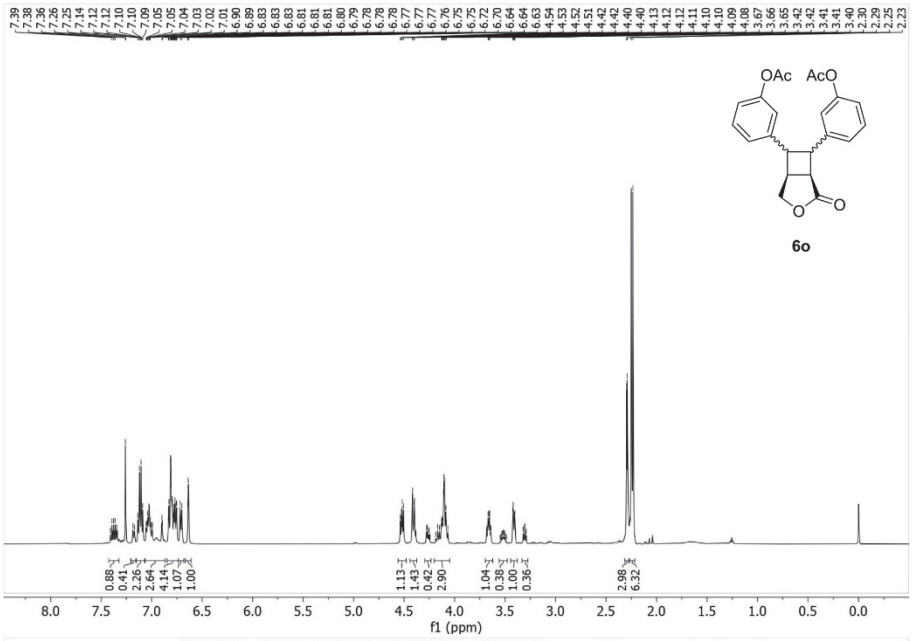


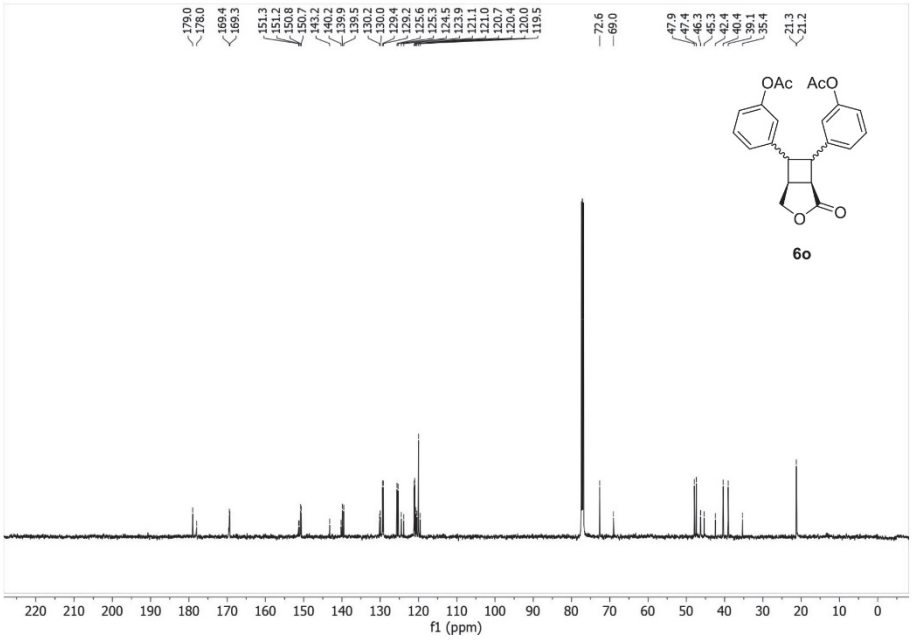












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