Supporting information

Catalytic scalable Pauson-Khand reaction in in a plug flow reactor.

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The substrates were synthesized as described in the literature: **1a**[1], **1b**[1], **1d**[1], **1e**[1], **1f**[2], **1g**[3], **1h**[4], **3**[1], **5a**[5], **5b**[6], **5c**[7] **7**[8], **9**[9]

The substrates of the intermolecular version were commercially available.

Description of the flow microreactor system: The system is composed by a Rheodyne valve that introduces the diluted substrate in the system. The pump is continuously providing solvent to the reactor. Carbon monoxide was delivered to the mixer at a constant rate through a mass flow controller from a CO gas cylinder. The mass flow controller allows us to introduce precisely the amount of CO gas, as mLN, corresponding to 1.5, 2 or 3 equivalents of the substrate, The gas and the liquid substrate are joined in a stainless steel T-shaped mixer filled with steel turnings. Then, there is the oven that heats the tubular reactor (volume = 60.63 mL), finally a liquid-gas separator, splits the solution and the gas. The pressure of the system was controlled by a back pressure regulator located after the oven. The excess of gas goes out of the system in a controlled. Thus, once the reaction mixture pours into the gas-liquid separator, the gas is discarded to the atmosphere.

General method for sample preparation: 0.32 mmol of the compound and 0.016 mmol of $Co_2(CO)_8$ were dissolved in 0.8 mL of toluene. Then, Ar was bubbled trough the sample, the solution was filtered through 45 µm nylon filter. The injection into the system was thanks to a Rheodyne valve

General conditions: All reactions were performed at 20 bar, and a concentration of the samples of 0.4 M. Variable residence times, CO equivalents and temperatures were used as indicated in each case.

Work up general procedure: The samples were collected in test tubes, the solution was filtered through zeolite, it was washed with chloroform and the solvent was removed in *vacuo.* Then the conversion was analyzed by NMR. Then, the samples were purified by column chromatography on silica gel with mixtures of Hexane/Ethyl acetate.

Diethyl 5-oxo-3,3a,4,5-tetrahydropentalene-2,2(1H)-dicarboxylate, 2a: It was synthesized from **1a** (76 mg, 0.32 mmol), following the general procedure, with a temperature of 120 ºC, 3 or 1.5 CO equivalents, 9 minutes of residence time and a total time of 13 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 4:1), 85 (99% yield) and 83 mg (97% yield) were obtained. Spectroscopic data were in accordance with those of the literature.[10]

Scaled up reaction (entries 7 and 8): 5g of **1a** and 359 mg of $Co_2(CO)_8$ (5 mol%) were dissolved in 52.52 mL of toluene. The solution was degasified with Ar, and filtered through a nylon filter of 0.45 µm. The resultant solution was set up in the pump inlet. The conditions of the system were 120 ºC, 20 bar of pressure, and 3 or 1.5 equivalents of CO. The total time was 38 and 42 minutes respectively. The crude product was filtered through celite, which was washed with chloroform (15 mL) and concentrated *in vacuo* 5.24 and 5.18 g were obtained as crude product with >99% of conversion. After purification by silica gel column chromatography

(Hexane/Ethyl acetate 4:1), 5.02 g or 4.52 g of pure product were obtained (91% or 82% yield, respectively).

Diethyl 3a-methyl-5-oxo-3,3a,4,5-tetrahydropentalene-2,2(1H)-dicarboxylate, 2b: From **1b** (80 mg, 0.32 mmol), following the general procedure with a temperature of 120 ºC, 9 minutes of residence time and a total time of 13 minutes.3 and 1.5 CO equivalents were tested. After purification by silica gel column chromatography (Hexane/Ethyl acetate 4:1), 88 mg were obtained (98% yield) and 72 mg (81% yield respectively). Spectroscopic data were in accordance with those of the literature.[10]

(1*S,6a*S**)-Triethyl 2-oxo-1,2,6,6a-tetrahydropentalene-1,5,5(4H)-tricarboxylate, 2c (entry 14):** From **1c** (59 mg, 0.32 mmol), and 10.9 mg of $Co_2(CO)_8$ (10 mol%), following the general procedure, with a temperature of 120 ºC, 3 CO equivalents, 9 minutes of residence time and a total time of 13 minutes., 74 mg were obtained (68% yield) as yellowish oil. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.92 (s, 1H, CH=), 4.28-4.19 (m, 6H, 3xOCH_2), 3.55-3.50 (m, 1H, CH_2-C*H*), 3.42 (d, *J* = 19.1 Hz, 1H, C*H*HC=), 3.25 (d, *J* = 19.1 Hz, 1H, C*H*HC=), 3.14 (d, *J* = 3.84 Hz, 1H, CHC=O), 2.86 (dd, J_1 = 12.9 Hz, J_2 = 7.9 Hz, 1H, C*H*H-CH) 1.86 (t, *J* = 12.7 Hz, 1H, C*H*H-CH*)* 1.33-1.24 (m, 9H, 3xCH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 202.4 (CO), 184.6 (C), 170.6 (2xCO), 168.7 (CO), 123.4 (CH), 62.3 (CH_2), 62.2 (CH_2), 61.7 (CH_2), 59.4 (CH), 48.5 (CH), 37.9 (CH_2), 35.0 (CH_2), 31.0 (C), 14.2 (CH_3), 14.0 (2xCH_3). **IR** (NaCl): 1710, 1671, 1590. Anal Calcd for: $C_{17}H_{22}O_7$: C, 60.35; H, 6.55; O, 33.10. Found: C, 60.16; H, 6.41.

2-Tosyl-2,3,3a,4-tetrahydrocyclopenta[c]pyrrol-5(1H)-one, 2d: From **1d** (80 mg, 0.32 mmol), following the general procedure, with a temperature of 120 ºC, 1.5 CO equivalents, 9 minutes of residence time and a total time of 13 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 9:1), 84 mg were obtained (94% yield). Spectroscopic data were in accordance with those of the literature.[10]

6-Methyl-2-tosyl-2,3,3a,4-tetrahydrocyclopenta[c]pyrrol-5(1H)-one, 2e: From **1e** (84 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 3 CO equivalents, 11 minutes of residence time and a total time of 14 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 9:1), 85 mg were obtained (91% yield). Spectroscopic data were in accordance with those of the literature.[4]

4,4-Dimethyl-2-tosyl-2,3,3a,4-tetrahydrocyclopenta[c]pyrrol-5(1H)-one, 2f: From **1f**, following the general procedure, with a temperature of 120 ºC, 3 CO equivalents, 10 minutes of residence time and a total time of 14 minutes. After purification (Hexanes/Ethyl acetate 9:1), 89 mg were obtained as colorless dense oil. (91% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.2 Hz, 2H, Ar), 7.28 (d, *J* = 8.1 Hz, 2H, Ar), 5.09 (t, *J* = 7.3 Hz, 1H, CH=), 4.06 (d, *J* = 2.4 Hz, 2H, CH_2), 3.80 (d, *J* = 7.3 Hz, 2H, CH_2), 2.42 (s, 3H, CH_3Ar), 1.97 (t, *J* = 2.4 Hz, 1H, CH), 1.71 (s, 3H, CH_3), 1.66 (s, 3H, CH_3). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 207.1 (C), 143.4 (C), 139.1 (C), 136.0 (C), 129.4 (2xCH), 127.8 (2xCH), 117.9 (CH), 73.3 (CH), 43.9 (CH_2), 35.3 (CH_2), 30.9 (C), 25.9 (CH_3), 21.5 (CH_3), 17.8 (CH_3). **IR** (NaCl): 1724, 1671, 1604 cm^{-1}. Anal Calcd for $C_{16}H_{19}NO_3S$: C 62.93; H, 6.27; N, 4.59; S, 10.50. Found: C, 62.70; H, 6.09; N, 4.71.

2,2-Dimethyl-3a',4'-dihydro-1'H-spiro[[1,3]dioxane-5,2'-pentalen]-5'(3'H)-one, 2g: From **1g** (62 mg, 0.32 mmol), following the general procedure, with a temperature of 120 ºC, 2 CO equivalents, 10 minutes of residence time and a total time of 14 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 9:1), 69 mg were obtained (96% yield) as a yellowish dense oil. ^{1}H-NMR (400 MHz, $CDCl_3$): **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.90 (s, 1H, HC=), 3.82 (d, *J* = 11.4 Hz, 1H, C*H*HO), 3.74 (d, *J* = 11.4 Hz, 1H, C*H*HO), 3.66 (d, *J* = 11.3 Hz, 1H, C*H*HO), 3.56 (d, *J* = 11.3 Hz, 2H, C*H*HO), 3.09-3.00 (m, 1H, CH), 2.68-2.57 (m, 3H, CH_2C and C*H*HC=), 2.20 (dd, J_1 = 12.8 Hz, J_2 = 8.4 Hz, 1H, C*H*HC=O), 2.07 (dd, J_1 = 17.7 Hz, J_2 = 2.6 Hz, 1H, C*H*HC=), 1.45 (s, 3H, CH_3), 1.44 (s, 3H, CH_3), 0.99 (t, *J* = 12.4 Hz, 1H,

C*H*HC=O). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 210.3 (CO), 187.7 (C), 125.4 (CH), 98.0 (C), 69.2 (CH_2), 69.0 (CH_2), 43.8 (C), 43.7 (CH_2), 42.5 (CH), 37.5 (CH_3), 35.6 (CH_3), 25.0 (CH_2), 22.4 (CH_2). **IR** (NaCl): 1704, 1633 cm^{-1}. Anal Calcd for $C_{13}H_{18}O_3$: C, 70.24; H, 8.16; O, 21.59. Found: C, 70.05; H, 8.01.

6-Phenyl-3a,4-dihydro-1H-cyclopenta[c]furan-5(3H)-one, 2h: From **1h** (56 mg, 0.32 mmol) following the general procedure, with a temperature of 170 ºC, 21 minutes of residence time, and after purification by silica gel column chromatography (Hexane/Ethyl acetate 49:1), 64 mg were obtained (99% yield). Spectroscopic data were in accordance with those of the literature.[4]

Scale up of 2h (Entry 27): 5g of **1h**, and 484 mg of $Co_2(CO)_8$ (10 mol%) were dissolved in 71.4 mL of toluene. The solution was degasified with Ar, and filtered through a 0.45 µm nylon filter.. The resultant solution was set up in the pump inlet. The conditions of the system were:

Temperature of 170 ºC, 20 bar of pressure, 2 equivalents of CO, 20 minutes of residence time, and the total time was 86 minutes. The crude product was filtered through zeolite, it was washed with chloroform and concentrated *in vacuo*. 5.88 g were obtained as crude product with a conversion of >99%. Then, it was purified by silica gel column chromatography (Hexane/Ethyl Acetate 12:1). 5.15 g were obtained (89% yield).

Diethyl 2-oxo-4,6,7,7a-tetrahydro-1H-indene-5,5(2H)-dicarboxylate, 4: From **3**, (80 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 3 CO equivalents, 10 minutes of residence time and a total time of 15 min. After purification by silica gel column chromatography (Hexane/Ethyl acetate 6:1), 80 mg were obtained (89% yield). Spectroscopic data were in accordance with those of the literature.[10]

3a,4-Dihydro-3H-cyclopenta[c]chromen-2-one, 6a: From **5a** (50 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 3 CO equivalents, 15 minutes of residence time and a total time of 19 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 4:1), 47 mg were obtained (79% yield). Spectroscopic data were in accordance with those of the literature.[5]

5-Hydroxy-3,3a,4,5-tetrahydro-2H-cyclopenta[a]naphthalen-2-one, 6b: From **5b** (54 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 15 minutes of residence time and a total time of 19 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 2:1), 62 mg were obtained (96% yield). Spectroscopic data were in accordance with those of the literature.[5]

7-Methoxy-3,3a,4,5-tetrahydro-2H-cyclopenta[a]naphthalen-2-one, 6c: From **5c** (60 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 3 CO equivalents, 15 minutes of residence time and total time of 19 minutes. After purification by silica gel column chromatography (Hexane/Ethyl acetate 2:1), 62 mg were obtained as a yellowish oil (92% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (d, J = 8.7 Hz, 1H, Ar), 6.83 (dd, J_1 = 8.7 Hz, J_2 = 2.6 Hz, 1H, Ar), 6.73 (d, J = 2.3 Hz, 1H, Ar), 6.28 (d, J = 1.8 Hz, 1H, CH=), 3.85 (s, 3H, CH_3O), 3.06-2.96 (m, 3H, CH_2-C*H*-CH_2 + CH_2-C*H*H-CH + C*H*H-Ar), 2.76 (dd, J_1 = 18.1 Hz, J_2 = 6.5 Hz, 1H, C*H*H-C=O), 2.30-2.24 (m, 1H, C*H*H-Ar), 2.20 (dd, J_1 = 18.1 Hz, J_2 = 3.7 Hz, 1H, C*H*H-C=O), 1.71-1.63 (m, 1H, CH_2-C*H*H-CH). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 208.0 (C), 175.3 (C), 161.8 (C), 141.6 (C), 129.0 (CH), 122.9 (C), 121.6 (CH), 113.7 (CH), 113.3 (CH), 55.4 (CH_3), 42.6 (CH_2), 40.1 (CH), 30.4 (CH_2), 29.6 (CH_2). **IR** (NaCl): 1690, 1671, 1599. Anal Calcd for $C_{14}H_{14}O_2$: C 62.93; H, 6.27; N, 4.59; S, 10.50. Found: C, 62.78; H, 6.10; N, 4.39.

Scale up of **6c:** 5 g of **6c** and 459 mg of $Co_{2(}CO)_8$ were dissolved in 79 mL of toluene. The solution was degasified with Ar, and filtered through a 0.45 µm nylon filter. The resultant solution was set up in the pump inlet. The conditions of the system were:

Temperature of 150 ºC, 20 bar of pressure, 3 equivalents of CO, 15 minutes of residence time, and the total time was 81 minutes. The crude product was filtered through

zeolite, it was washed with chloroform and concentrated *in vacuo*. 5.61 g were obtained as crude product with a conversion of >99%. Then, it was purified by silica gel column chromatography (Hexane/Ethyl Acetate 2:1). 5.08 g were obtained (89% yield).

11-((tert-Butyldimethylsilyl)oxy)-1,11,12,12a-tetrahydrocyclopenta[5,6]azepino[1,2-a]indol-2(4H)-one, **8:** A solution of the starting material **7** (81 mg, 0.32 mmol) and 10.9 mg of $Co_2(CO)_8$ (10 mol%) was heated before injected into the system in order to reach total solubility. Then, following the general procedure, with a temperature of 170 ºC, 3 CO equivalents, 20 minutes of residence time and a total time of 25 minutes. After purification by silica gel column chromatography (Hexane), 83 mg were obtained (71% yield). Spectroscopic data were in accordance with those of the literature.[8]

N,N',N''-(Benzene-1,2,4-triyltris(methylene))tris(4-methyl-N-(pent-4-en-1-yl)benzenesulfonamide), 10: From **9** (89 mg, 0.32 mmol), following the general procedure, with a temperature of 150 ºC, 3 CO equivalents, 20 minutes of residence time, and a total time of 25 minutes. After purification (Hexane/Ethyl acetate 5:1), 28 mg were obtained as a yellowish oil (30% yield). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75-7.70 (m, 6H, Ar),7.36-7.26 (m, 9H, Ar), 5.60-5.51 (m, 3H, 3xCH=), 4.88-4.84 (m, 6H, 3xCH_2=), 4.45 (s, 2H, CH_2N), 4.38 (s, 2H, CH_2N), 4.27 (s, 2H, CH_2N), 3.06-2.94 (m, 6H, 3xCH_2), 2.43 (s, 6H, 2xCH_3), 2.42 (s, 3H, CH_3) 1.88-1.78 (m, 6H, 3xCH_2) 1.37-1.25 (m, 6H, 3xCH_2). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.6 (C), 143.5 (C), 143.4 (C), 137.20 (2xCH), 137.19 (CH), 136.7 (C), 136.6 (C), 135.9 (C), 135.7 (C), 135.0 (C), 134.8 (C), 129.89 (2xCH), 129.86 (2xCH), 129.78 (2xCH), 129.6 (CH), 129.5 (CH), 129.2 (CH), 127.29 (2xCH), 127.27 (2xCH), 127.1 (2xCH), 115.25 (2xCH_2), 115.22 (CH_2), 51.7 (CH_2), 50.7 (CH_2), 50.2 (CH_2), 48,5 (CH_2), 48.3 (CH_2), 47.9 (CH_2), 30.8 (CH_2), 30.7 (CH_2), 30.6 (CH_2), 27.4 (CH_2), 27.3 (CH_2), 27.2 (CH_2), 21.5 (2xCH_3), 21.4 (CH_3). **IR** (NaCl): 1643, 1599 cm^{-1}. Anal Calcd for $C_{45}H_{57}N_3O_6S_3$: C, 64.95; H, 6.90; N, 5.05; S, 11.56. Found: C, 64.72; H, 7.01; N, 5.18.

2-Phenyl-3a,4,5,6,7,7a-hexahydro-1H-4,7-methanoinden-1-one, 11a: From 30 mg (0.32 mmol) of 2-norbornene, and 0.037 mL (0.32 mmol) of phenylacetylene, following the general procedure, with a temperature of 120 ºC, 1.5 CO equivalents, 10 minutes of residence time and 14 minutes of total time. After purification by silica gel column chromatography (Hexane/Ethyl Acetate 19:1), 57 mg were obtained. Spectroscopic data were in accordance with those of the literature.[11]

Scale up reaction of **11a:** 2.5 g of 2-norbornene (26.56 mmol) and 2.92 mL of phenylacetylene (26.56 mmol) were dissolved in 66.4 mL of toluene. Then, 453 mg of $Co_2(CO)_8$ were added. The solution was degasified with Ar, and filtered through a nylon filter of 0.45 µm. The resultant solution was set up in the pump inlet.

The conditions of the system were 120 ºC, 20 bar of pressure, and 1.5 equivalents of CO. The residence time was 10 minutes and the total time of the process was 42 min. The crude product was filtered through zeolite, it was washed with chloroform and concentrated in vacuo. 5.76 g were obtained as crude product with >99% of conversion. Then, it was purified by silica gel column chromatography (Hexane/Ethyl Acetate 19:1). 4.82 grams were obtained (81% yield). Additionally, 440 mg of the [2+2+2] product of phenylacetylene **(4'-phenyl-1,1':2',1''-terphenyl)** were obtained. (16 % yield).

3-Methyl-2-phenyl-3a,4,5,6,7,7a-hexahydro-1H-4,7-methanoinden-1-one, 11b: From 30 mg (0.32 mmol) of 2-norbornene and 0.040 mL (0.32 mmol) of 1-phenyl-1-propyne, following the general procedure, with a temperature of 170 ºC, 3 CO equivalents 20 minutes of residence, and a total time of 25 minutes. After purification by silica gel column chromatography (Hexane/Ethyl Acetate 9:1), 32 mg were obtained (42% yield). Additionally, 15 mg of **3',4',6'-trimethyl-5'-phenyl-1,1':2',1''-terphenyl, 12** were obtained (40% yield). Spectroscopic data were in accordance with those of the literature.[12, 13]

2-Butyl-3a,4,5,6,7,7a-hexahydro-1H-4,7-methanoinden-1-one, 11c: From 30 mg (0.32 mmol) of 2-norbornene and 0.037 mL (0.32 mmol) of 1-hexyne, following the general procedure, with a temperature of 120 ºC, 1.5 CO equivalents, 10 minutes of residence time and a total time of 14 minutes. After purification by silica gel column chromatography (Hexane/Ethyl Acetate 49:1), 56 mg were obtained (85% yield). Spectroscopic data were in accordance with those of the literature.[11]

2,3-bis(Methoxymethyl)-3a,4,5,6,7,7a-hexahydro-1H-4,7-methanoinden-1-one, 11d: From 30 mg (0.32 mmol) of 2-norbornene and 0.038 mL (0.32 mmol) of 1,4-dimethoxy-2-butyne, following the general procedure, with a temperature of 170 ºC, 3 CO equivalents, 15 minutes of residence time and a total time of 19 minutes. After purification by silica gel column chromatography (Hexane/Ethyl Acetate 12:1), 51 mg were obtained (60% yield). Spectroscopic data were in accordance with those of the literature.[14]

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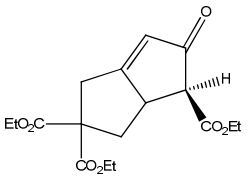

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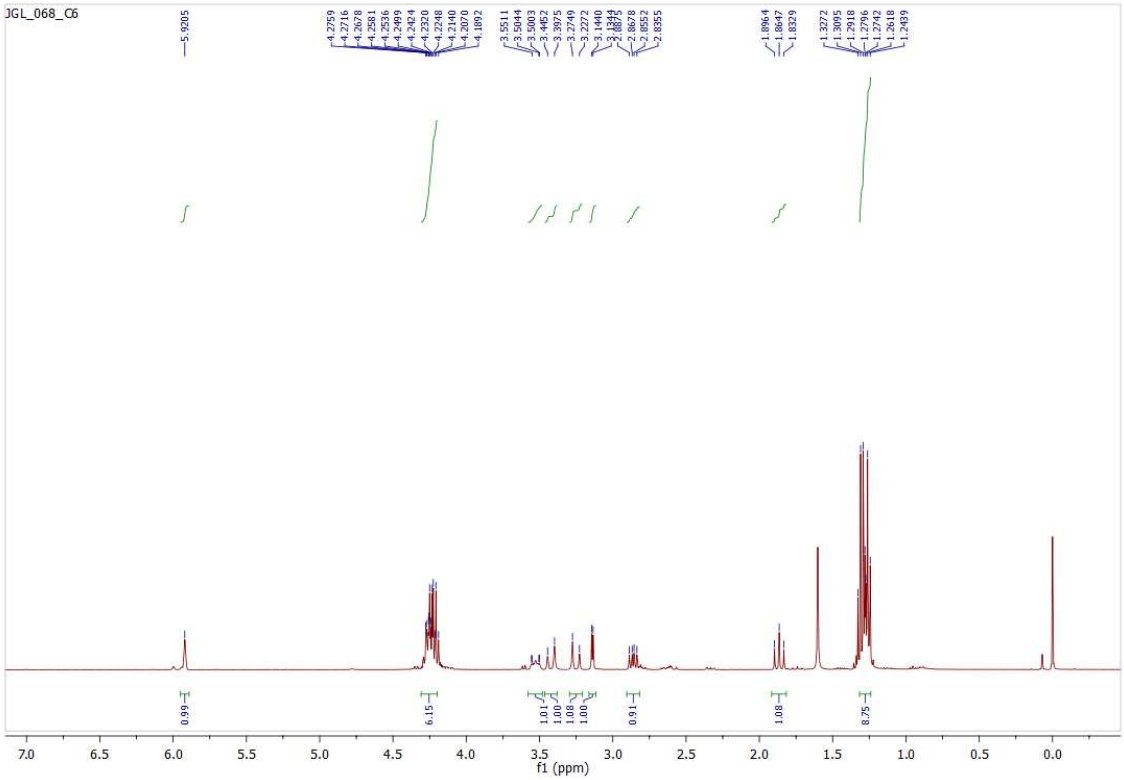

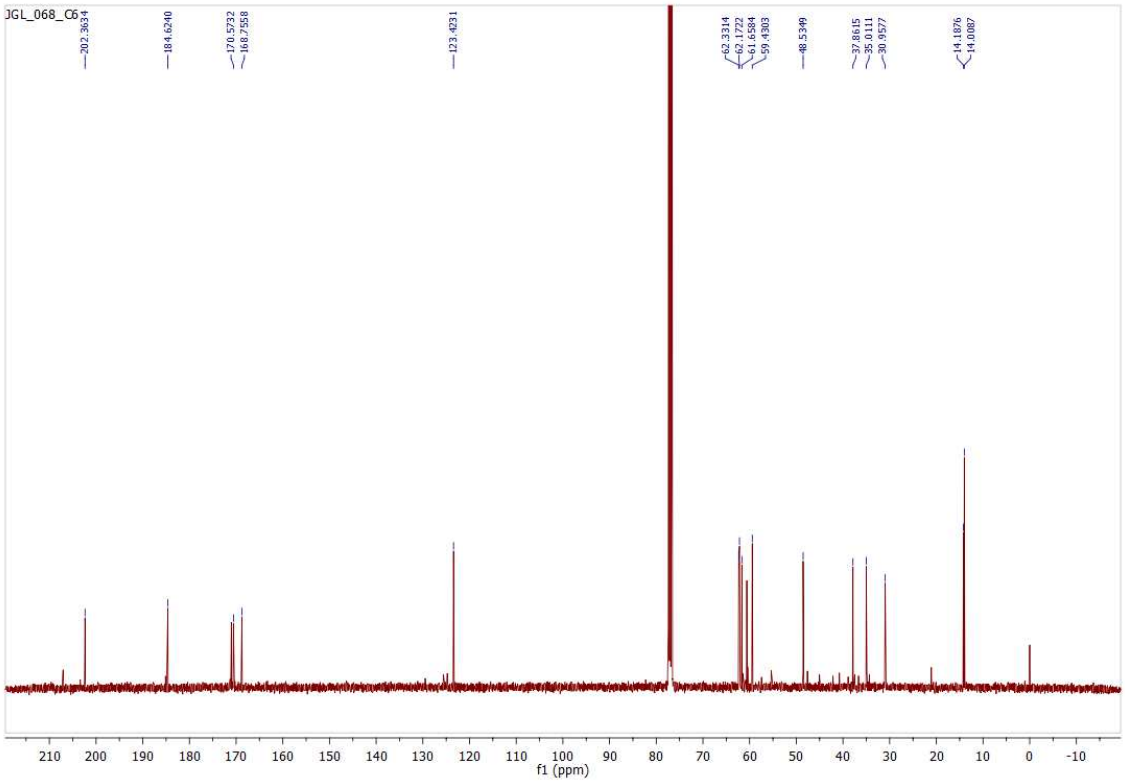

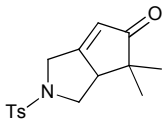
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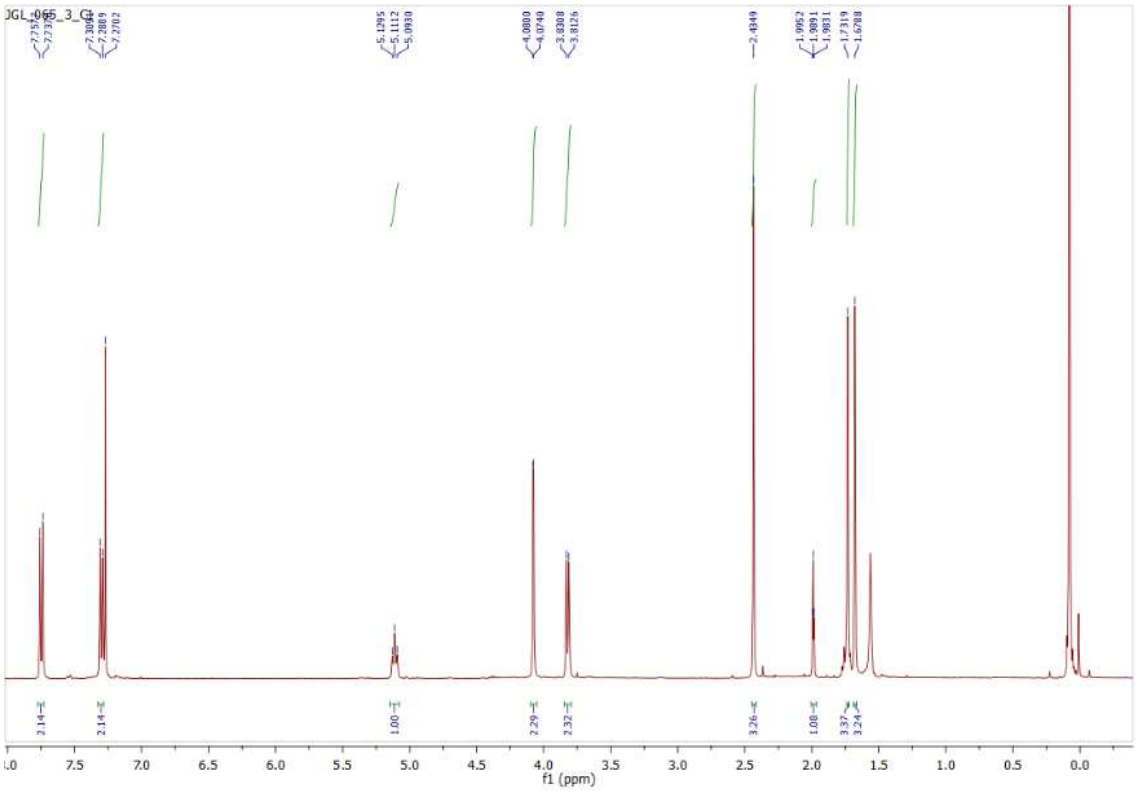
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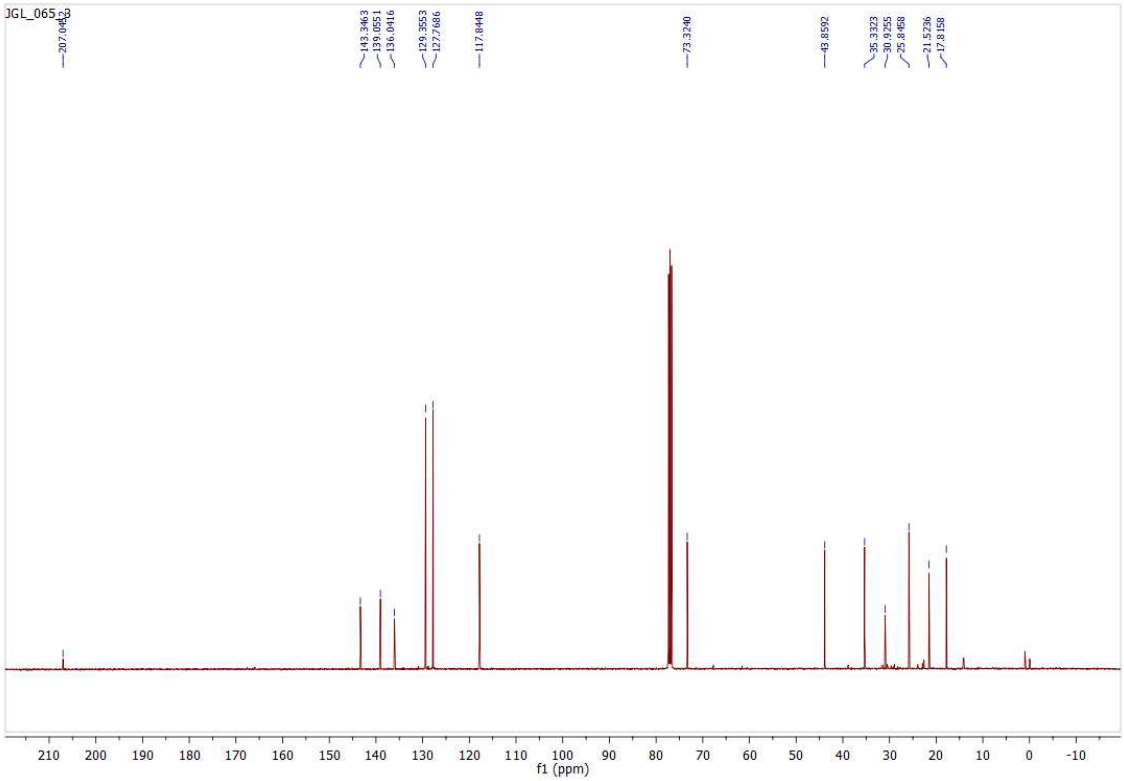
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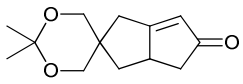
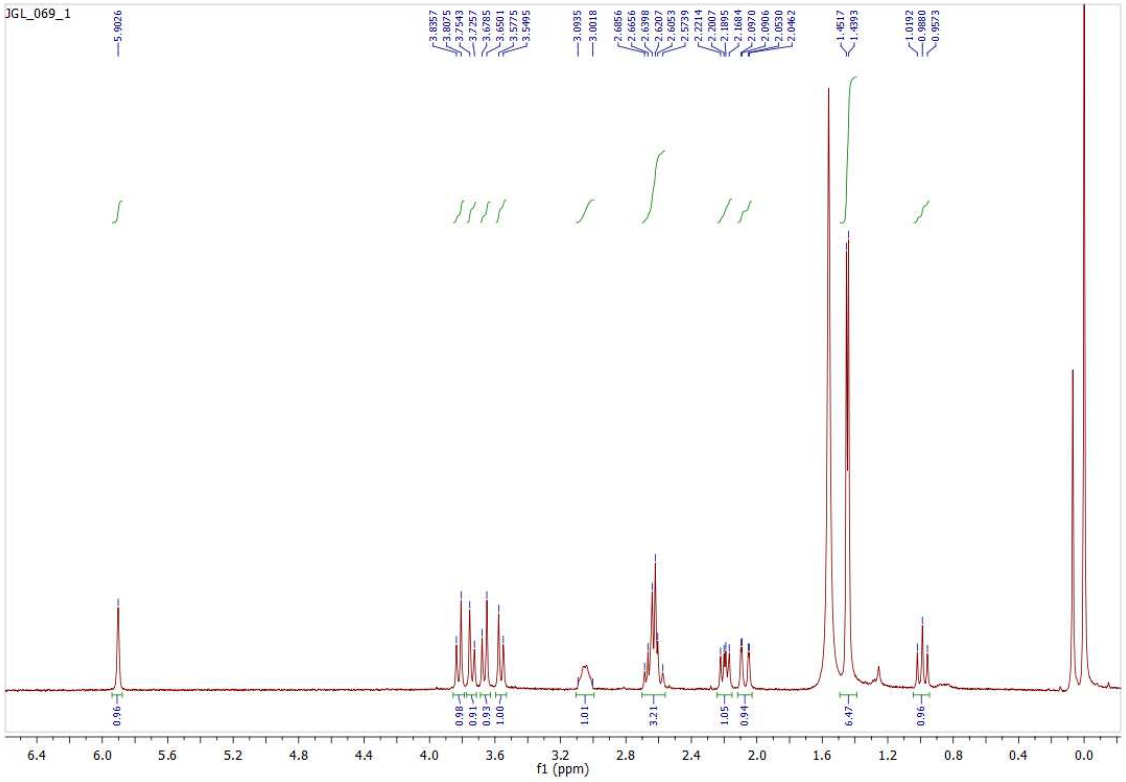
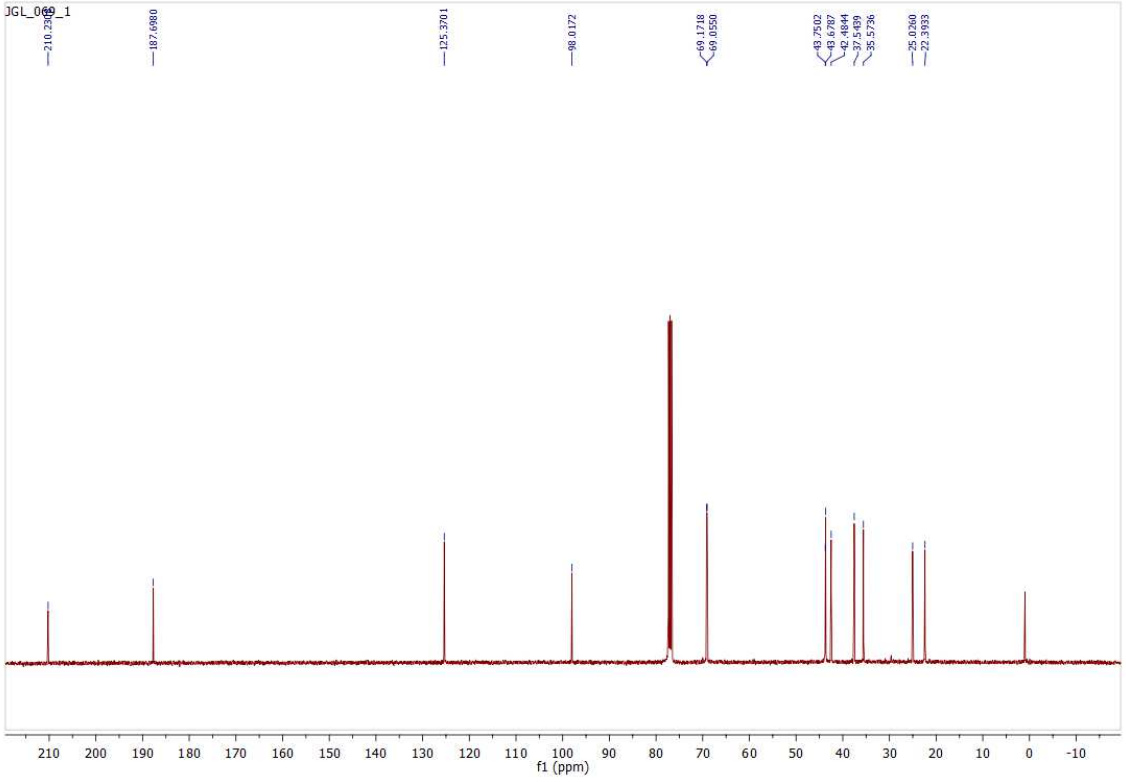
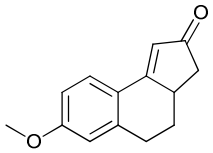
40
30
20
10
0
-10
f1 (ppm)



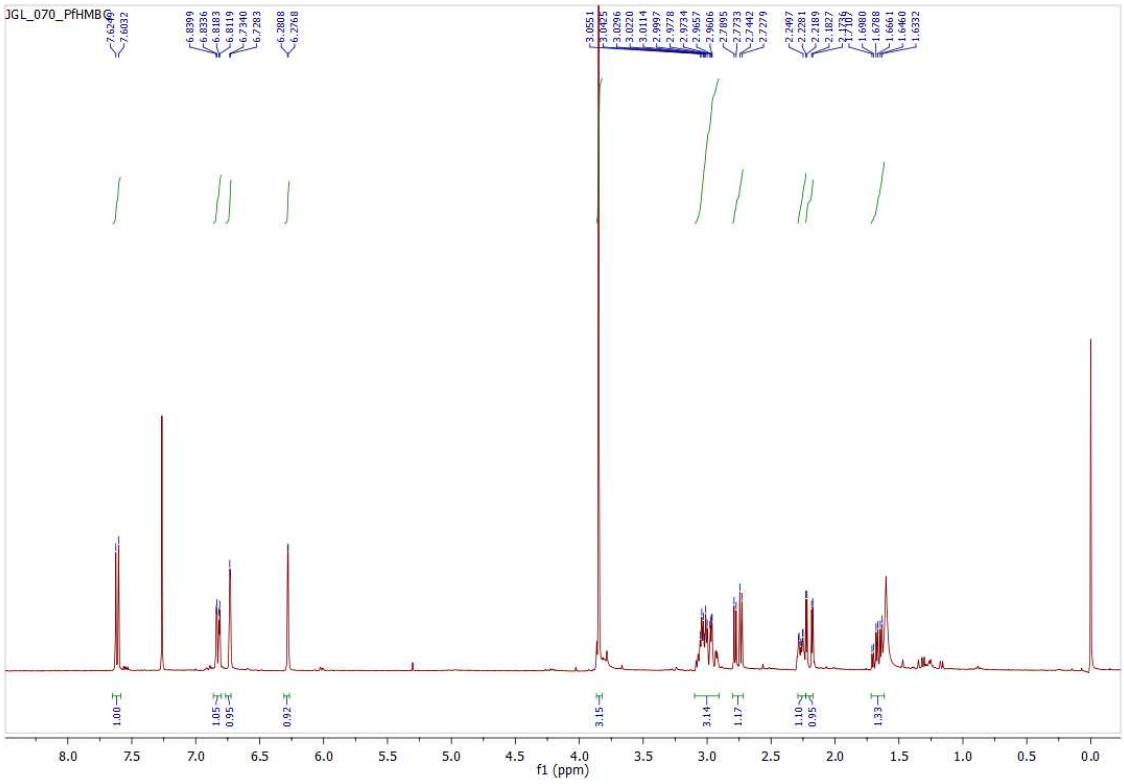
2g







6c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6.8399
6.8336
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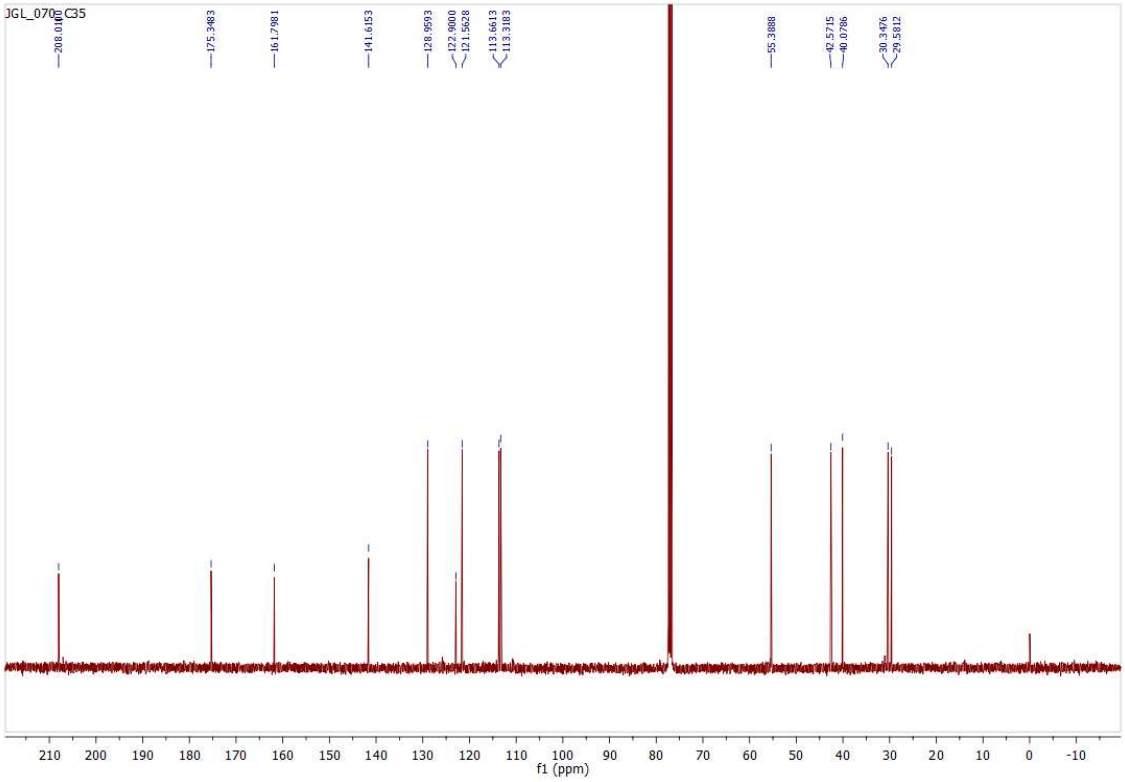
6.7340
6.7283
6.2808
6.2768
3.0551
3.0425
3.0296
3.0220
3.0114
2.9997
2.9778
2.9734
2.9657
2.9606
2.7895
2.7733
2.7442
2.7279
2.2497
2.2281
2.2189
2.1827
2.1610
1.6980
1.6788
1.6661
1.6460
1.6332
1.00
1.05
0.95
0.92
3.15
3.14
1.17
1.10
0.95
1.33
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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141.6153
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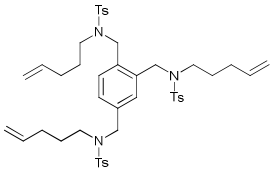
122.9000
121.5628
113.6613
113.3183
55.3888
42.5715
40.0786
30.3476
29.5812
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



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