The version of SI published September 22, 2005 contained errors. The correct version was posted November 28, 2005

Supporting Information

Stereoselective Synthesis of Spirocyclic Ketones by Nazarov Reaction of 6-(1-Ethoxy-1,3-butadienyl)dihydropyran derivatives

Cristina Prandi,* Annamaria Deagostino, Paolo Venturello, and Ernesto G. Occhiato*

EXPERIMENTAL. All solvents were degassed before use in cross-coupling processes. Chromatographic separations were carried out under pressure on silica gel using flash-column techniques; Rf values refer to TLC carried out on 0.25-mm silica gel plates (Merck F254), with the same eluant indicated for the column chromatography. ^{1}H NMR spectra were recorded at 400 and 200 MHz, NOESY 1D and 2D experiments at 400 MHz, and ^{13}C NMR spectra at 100.4 and 50.33 MHz. MS spectra were recorded at an ionizing voltage of 70 eV. THF was distilled from Na/benzophenone. Compounds **1a**,[8] **2a**,[8] **2b**,[6] **3a-d**,[2,3] and **4a-d**,[3] were prepared as reported.

6-((*E*)-1-Ethoxy-4-methylpenta-1,3-dienyl)-3,4-dihydro-2*H*-pyran (4e). To a solution of crude **3a** (232 mg, 1.0 mmol) in THF (12 mL) were added, under a nitrogen atmosphere, $(Ph_3P)_2PdCl_2$ (35 mg, 0.05 mmol), boronate **2a** (252 mg, 1.0 mmol), and a 2 M aqueous K_2CO_3 solution (1 mL). The mixture was stirred for 3 h at room temperature. Water (25 mL) was then added, the mixture extracted with diethyl ether (3 x 20 mL) and dried over anhydrous sodium sulfate. Evaporation of the solvent afforded a yellow oil which was purified by chromatography (EtOAc-petroleum ether, 1:6, 1% Et_3N, R_f 0.80) to give **4e** (110 mg, 53%) as a colorless oil: ^{1}H NMR (200 MHz, $CDCl_3$): 6.20 (d, J = 9.4 Hz, 1 H), 5.65 (d, J = 9.4 Hz, 1 H), 5.15 (t, J = 2.3 Hz, 1 H), 4.14 (t, J = 6.0 Hz, 2 H), 3.85 (q, J = 6.5 Hz, 2 H), 2.20 (m, 1 H), 1.95 (m, 1 H), 1.85 (s, 3 H), 1.75 (s, 3 H), 1.26 (t, J = 6.5 Hz, 3 H), 0.95 (m, 2 H); ^{13}C NMR (50.33 MHz, $CDCl_3$): 150.0 (s), 148.7 (s), 131.3 (s), 120.4 (d), 102.7 (d, 2 C), 66.1 (t), 6^{i}3.7 (t), 26.4 (t), 22.1 (q), 20.4 (q), 18.0 (t), 14.7 (q); MS *m/z* 208 (M^+,

10[S1]0), 193 (37), 179 (42), 55 (40). Anal. Calcd for $C_{13}H_{20}O_2$: C, 74.96; H, 9.68;. Found C, 74.79; H, 9.73.

6-((E)-1-Ethoxy-4-methylpenta-1,3-dienyl)-3,4-dihydro-4-methyl-2H-pyran (4f): prepared as described for **4e** starting from 5,6-dihydro-4-methyl-4*H*-pyran-2-yl trifluoromethanesulfonate **3c** (230 mg, 1 mmol). Obtained 162 mg (73% yield) after chromatography (EtOAc-petroleum ether, 5:95). ^{1}H NMR (200 MHz, $CDCl_3$): 6.21 (dq, *J* = 10.9, 1.5 Hz, 1 H), 5.62 (d, *J* = 10.9 Hz, 1 H), 5.01 (d, *J* = 2.9 Hz, 1 H), 4.10 (m, 2 H), 3.83 (q, *J* = 7.0 Hz, 2 H), 2.12 (m, 1 H), 1.78 (s, 3 H), 1.72 (s, 3 H), 1.36 (m, 2 H), 1.33 (t, *J* = 7.0 Hz, 3 H), 1.09 (d, *J* = 6.9 Hz, 3 H); ^{13}C NMR (50.33 MHz, $CDCl_3$): 149.9 (s), 147.7 (s), 131.3 (s), 120.5 (d), 109.1 (d), 103.0 (d), 64.2 (t), 63.7 (t), 30.5 (d), 26.4 (q), 25.8 (q), 21.8 (t), 17.9 (q), 14.7 (q); MS *m/z* 222 (M^+, 48), 207 (17), 121 (32), 55 (100). Anal. Calcd for $C_{14}H_{22}O_2$: C, 75.63; H, 9.97;. Found C, 75.79; H, 9.83.

6-((E)-1-Ethoxy-4-methylpenta-1,3-dienyl)-3,4-dihydro-2-methyl-2*H*-pyran (4g): prepared as described for **4e** starting from 5,6-dihydro-6-methyl-4*H*-pyran-2-yl trifluoromethanesulfonate **3b** (230 mg, 1 mmol). Obtained 166 mg (75% yield) after chromatography (EtOAc-petroleum ether, 5:95). ^{1}H NMR (200 MHz, $CDCl_3$): 6.25 (dq, *J* = 11.0, 1.5 Hz, 1 H), 5.60 (d, *J* = 11.0 Hz, 1 H), 5.15 (t, *J* = 2.7 Hz, 1 H), 4.10 (m, 1 H), 3.80 (q, *J* = 7.0 Hz, 2 H), 2.22 (m, 2 H), 1.83 (s, 3 H), 1.70 (s, 3 H), 1.45 (d, *J* = 6.5 Hz, 3 H), 1.25 (t, *J* = 7.0 Hz, 3 H), 0.99 (m, 2 H); ^{13}C NMR (50.33 MHz, $CDCl_3$): 149.7 (s), 148.8 (s), 131.2 (s), 120.5 (d), 102.9 (d), 101.8 (d), 71.8 (d), 63.8 (t), 28.9 (t), 26.4 (q), 21.00 (q), 20.7 (t), 17.9 (q), 14.8 (q); MS *m/z* 222 (M^+, 35), 207 (27), 121 (42), 55 (100). Anal. Calcd for $C_{14}H_{22}O_2$: C, 75.63; H, 9.97;. Found C, 75.86; H, 9.78.

(1*E*, 3*E*)-6-(1-Ethoxyhexa-1,3-dienyl)-2-methyl-3,4-dihydro-2*H*-pyran (4h): prepared as described for **4e** starting from 5,6-dihydro-6-methyl-4*H*-pyran-2-yl trifluoromethanesulfonate **3b** (230 mg, 1 mmol) and **2b** (238 mg, 1 mmol). Obtained 162 mg (73% yield) after chromatography (EtOAc-petroleum ether, 1:49). ^{1}H NMR (200 MHz, $CDCl_3$): 6.44 (dd, *J* = 14.5, 11.0 Hz, 1 H), 5.65 (d, *J* = 11.0 Hz, 1 H), 5.45 (m, 1 H), 5.15 (t, *J* = 3.2, 1 H), 4.00 (m, 1 H), 3.80 (q, *J* = 7.0 Hz,

2 H), 2.20 (m, 4 H), 1.45-1.35 (m, 5 H), 1.25 (d, *J* = 6.5 Hz, 3 H), 0.95 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (50.33 MHz, $CDCl_3$): 151.7 (s), 148.5 (s), 132.5 (d), 125.3 (d), 106.1 (d), 102.4 (d), 71.8 (d), 63.6 (t), 29.9 (t), 22.9 (t), 21.9 (q), 21.6 (t), 16.5 (q), 15.7 (q); MS *m/z* 222 (M^+, 78), 193 (32), 139 (35), 123 (44), 55 (100). Anal. Calcd for $C_{14}H_{22}O_2$: C, 75.63; H, 9.97; Found C, 75.45; H, 9.65.

7-((*E*)-1-Ethoxy-4-methylpenta-1,3-dienyl)-2,3,4,5-tetrahydrooxepine (4i): prepared as described for **4e** starting from 5-trifluoro-methanesulfonic acid 4,5,6,7-tetrahydro-oxepin-2-yl ester **3e** (246 mg, 1 mmol). Obtained 180 mg (81% yield) after chromatography (EtOAc-petroleum ether, 1:9). ^{1}H NMR (200 MHz, $CDCl_3$): 6.23 (dq, *J* = 11.0, 1.5 Hz, 1 H), 5.55 (d, *J* = 11.0 Hz, 1 H), 5.44 (t, *J* = 6.1 Hz, 1 H), 4.00 (t, *J* = 5.3 Hz, 2 H), 3.80 (q, *J* = 6.9 Hz, 2 H), 2.30 (m, 2 H), 1.78 (s, 3 H), 1.72 (s, 3 H), 1.33 (t, *J* = 7.0 Hz, 3 H), 0.88-0.85 (m, 4 H); ^{13}C NMR (50.33 MHz, $CDCl_3$): 151.3 (s), 151.3 (s), 131.6 (s), 120.9 (d), 114.0 (d), 103.3 (d), 72.7 (t), 64.1 (t), 32.1 (q), 28.8 (t), 26.6 (q), 25.8 (t), 18.3 (t), 15.2 (q); MS *m/z* 222 (M^+, 100), 207 (42), 193 (35), 95 (24), 55 (48). Anal. Calcd for $C_{14}H_{22}O_2$: C, 75.63; H, 9.97; Found C, 75.44; H, 9.69.

7-((*E*)-1-Ethoxy-4-methylpenta-1,3-dienyl)-4-ethyl-2,3,4,5-tetrahydrooxepine (4l): prepared as described for **4e** starting from trifluoromethanesulfonic acid 5-ethyl-4,5,6,7-tetrahydro-oxepin-2-yl ester **3f** (274 mg, 1 mmol). Obtained 146 mg (58% yield) after chromatography (EtOAc-petroleum ether, 1:49). ^{1}H NMR (200 MHz, $CDCl_3$): 6.25 (dq, *J* = 10.5, 1.5 Hz, 1 H), 5.60 (d, *J* = 10.5 Hz, 1 H), 5.40 (t, *J* = 6.0 Hz, 1 H), 4.25 (ddd, *J* = 8.5, 5.5, 1.5 Hz, 1 H), 3.90 (m, 1 H), 3.80 (q, *J* = 7.0 Hz, 2 H), 2.20 (m, 3 H), 1.85 (s, 3 H), 1.78 (s, 3 H), 1.55 (m, 2 H), 1.45 (m, 2 H), 1.35 (t, *J* = 7.0 Hz, 3 H), 0.95 (t, *J* = 7.0 Hz, 3 H); ^{13}C NMR (50.33 MHz, CDCl3): 154.3 (s), 150.5 (s), 131.2 (s), 120.6 (d), 120.0 (d), 103.0 (d), 71.1 (t), 63.8 (t), 37.9 (q), 37.5 (t), 31.8 (q), 29.4 (d), 26.5 (t), 17.9 (t), 14.8 (q), 11.6 (q); MS *m/z* 250 (M^+, 100), 235 (43), 221 (35), 179 (24), 55 (56). Anal. Calcd for $C_{16}H_{26}O_2$: C, 76.75; H, 10.47; Found C, 76.89; H, 10.35.

4,4-Dimethyl-6-oxaspiro[4.5]dec-2-en-1-one (5e): Amberlyst 15 (2.3 mequiv/g, 18 mg) was added to a solution of **4e** (104 mg, 0.5 mmol) in anhydrous DCM (5 mL) under argon atmosphere, and the resulting mixture was stirred at room temperature. The reaction was monitored by TLC:

after 8 h the resin was filtered off through a short pad of $NaHCO_3$ and the solution concentrated under vacuum. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.7) to give pure **5e** (65 mg, 72%). ^1H NMR (200 MHz, $CDCl_3$): δ 7.35 (d, *J* = 6.0 Hz, 1 H), 5.95 (d, *J* = 6.0 Hz, 1 H), 4.10 (m, 1 H), 3.71 (m, 1 H), 2.27 (m, 1 H), 1.78-1.65 (m, 4 H), 1.25 (m, 1 H), 1.23 (s, 3 H), 1.11 (s, 3 H); ^{13}C NMR ($CDCl_3$): 209.2 (s), 169.5 (d), 128.1 (d), 80.2 (s), 63.9 (t), 48.1 (s), 26.9 (t), 25.3 (t), 24.8 (t), 22.8 (q), 18.7 (q); MS *m/z* 180 (M^+, 30), 165 (100), 109 (35), 55 (23) Anal. Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95; Found C, 73.65; H, 8.81.

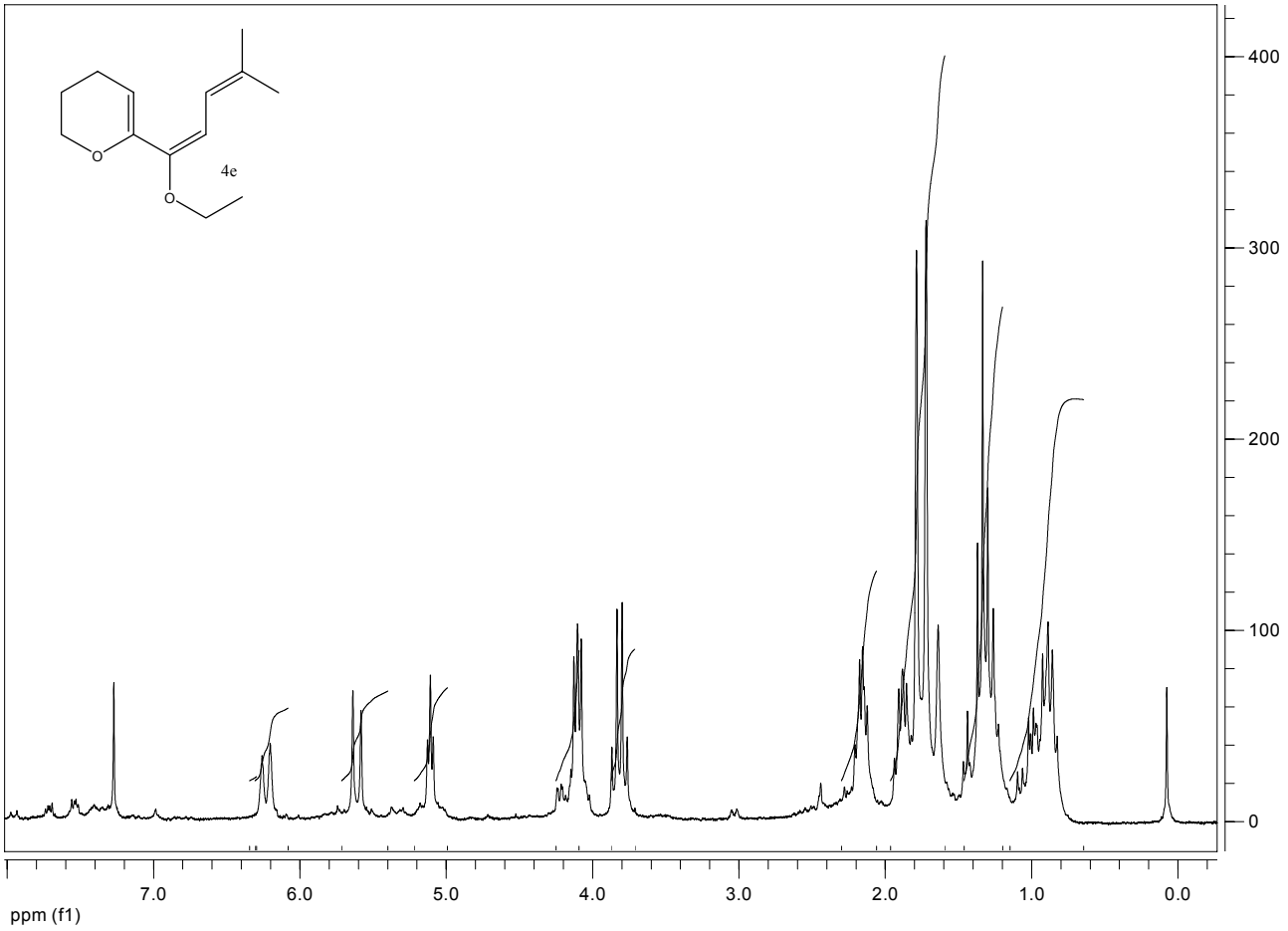
4,4,7-Trimethyl-6-oxaspiro[4.5]dec-2-en-1-one (5f+6f): prepared as described for **5e** starting from **4f** (135 mg, 0.6 mmol) after 6 h at r.t. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.76) to give **5f+6f** (86 mg, 74%). ^1H NMR (200 MHz, $CDCl_3$): δ 7.25 (d, *J* = 6.2 Hz, 2 H), 6.00 (d, *J* = 6.2 Hz, 1 H), 5.95 (d, *J* = 6.2 Hz, 1 H), 4.02 (m, 2 H), 3.71 (m, 1 H), 3.35 (m, 1 H), 2.03 (m, 2 H), 1.98-1.94 (m, 6 H) , 1.45 (s, 6 H), 1.28 (m, 2 H), 1.25 (s, 6 H), 0.94 (d, *J* = 6.4 Hz, 3 H), .0.91 (d, *J* = 6.4 Hz, 3 H); ^{13}C NMR ($CDCl_3$): 209.2 (s), 208.9 (s), 171.9 (d), 171.4 (d), 127.5 (d), 126.8 (d), 85.5 (s), 84.4 (s), 64.8 (t), 64.6 (t), 48.9 (s), 48.5 (s), 38.9 (t), 38.7 (t), 33.4 (t), 33.2.(t), 26.6 (d), 26.4 (d), 26.3 (q), 26.1 (q), 25.6 (q), 25.1 (q), 22.7 (q), 22.3 (q); MS *m/z* 194 (M^+, 26), 125 (21), 109 (41), 55 (16) .

4,4,7-Trimethyl-6-oxaspiro[4.5]dec-2-en-1-one (5g): prepared as described for **5e** starting from **4g** (222 mg, 1 mmol). The reaction was stopped after 4 h at room temperature. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.65) to give pure **5g** (112 mg, 58%) as white crystals mp 107-109 °C. ^1H NMR (200 MHz, $CDCl_3$): δ 7.25 (d, *J* = 6.2 Hz, 1 H), 6.00 (d, *J* = 6.2 Hz, 1 H), 3.69 (dqd, *J* = 11.35, 6.2, 2.2 Hz, 1 H), 1.80 (m, 1 H), 1.68-1.62 (m, 3 H), 1.43 (s, 3 H), 1.20 (d, *J* = 6.2 Hz, 3 H), 1.19 (s, 3 H); ^{13}C NMR ($CDCl_3$): 209.4 (s), 171.2 (d), 127.5 (d), 84.6 (s), 69.6 (d), 49.0 (s), 31.8 (t), 29.9 (t), 26.5 (q), 24.8 (q), 22.1 (q), 20.3 (t); MS *m/z* 194 (M^+, 16), 179 (100), 136 (65), 109 (51), 55 (20) Anal. Calcd for $C_{12}H_{18}O_2$: C, 74.19; H, 9.34; Found C, 74.85; H, 9.55

4-Ethyl-7-methyl-6-oxaspiro[4.5]dec-2-en-1-one (5h): prepared as described for **5e** starting from **4h** (280 mg, 1.26 mmol). The reaction was stopped after 8 h at room temperature. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.65) to give pure **5h** (162 mg, 66%). ^1H NMR (200 MHz, CDCl3): δ 7.78 (dd, *J* = 6.2, 4.1 Hz, 1 H), 6.21 (dd, *J* = 6.2, 1.6 Hz, 1 H), 3.70 (m, 1 H), 2.90 (m, 1 H), 2.13-2.01 (m, 1 H), 1.85-1.78 (m, 2 H), 1.63-1.59 (m, 2 H), 1.45-1.35 (m, 3 H), 1.16 (d, *J* = 5.5 Hz, 3 H), 1.12 (t, *J* = 6.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$): 207.1 (s), 165.8 (d), 128.2 (d), 80.3 (s), 69.9 (t), 52.1 (d), 32.2 (t), 25.0 (t), 24.4 (t), 22.2 (t), 20.2 (q), 11.9 (q); MS *m/z* 194 (M^+, 13), 176 (100), 133 (90), 95 (77), 55 (87). Anal. Calcd for $C_{12}H_{18}O_2$: C, 74.19; H, 9.34; Found C, 74.66; H, 9.48.

4,4-Dimethyl-6-oxaspiro[4.6]undec-2-en-1-one (5i): prepared as described for **5e** starting from **4i** (120 mg, 0.54 mmol). The reaction was stopped after 6 h at room temperature.. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.35) to give pure **5i** (86 mg, 82%). ^1H NMR (200 MHz, $CDCl_3$): δ 7.44 (d, *J* = 6.1 Hz, 1 H), 6.09 (d, *J* = 6.1 Hz, 1 H), 3.61 (t, *J* = 6.52 Hz, 2 H), 1.65 (m, 2 H), 1.58-1.51 (m, 6 H), 1.18 (s, 3 H), 1.08 (s, 3 H); ^{13}C NMR (CDCl3): 211.0 (s), 171.0 (d), 127.5 (d), 83.1 (s), 62.7 (t), 47.8 (s), 36.9 (t), 32.4 (t), 27.2 (t), 26.1 (q), 23.3 (q), 20.8 (t); MS *m/z* 194 (M^+, 44), 179 (100), 122 (60), 109 (55), 55 (71) Anal. Calcd for $C_{12}H_{18}O_2$: C, 74.18; H, 9.34; Found C, 74.25; H, 9.39.

9-Ethyl-4,4-dimethyl-6-oxaspiro[4.6]undec-2-en-1-one (5l): prepared as described for **5e** starting from **4l** (146 mg, 0.58 mmol). The reaction was stopped after 3 h at room temperature. Crude products were purified by flash chromatography (Et_2O-petroleum ether, 1:1, 0.5% Et_3N, R_f 0.55) to give pure **5l** (41 mg, 32%). ^1H NMR (200 MHz, $CDCl_3$): δ 7.35 (d, *J* = 6.2 Hz, 1 H), 6.00 (d, *J* = 6.2 Hz, 1 H), 3.98 (dt, *J* = 11.5, 2.5 Hz, 1 H), 3.65 (td, *J* = 11.5, 2.5 Hz, 1 H), 1.95-1.90 (m, 1 H), 1.85-1.80 (m, 2 H), 1.35-1.25 (m, 6 H), 1.21 (s, 3 H), 1.15 (s, 3 H), 0.93 (t, *J* = 6.5 Hz, 3 H); ^{13}C NMR ($CDCl_3$): 208.3 (s), 170.1 (d), 127.4 (d), 86.2 (s), 64.8 (t), 48.2 (s), 42.82 (d), 37.6 (t), 30.6 (t), 29.6 (t), 29.4 (t), 27.1 (q), 24.0 (q), 11.9 (q); MS *m/z* 222 (M^+, 41), 207 (100), 122 (45), 109 (15), 55 (54) Anal. Calcd for $C_{14}H_{22}O_2$: C, 75.63; H, 9.97; Found C, 75.45; H, 9.85.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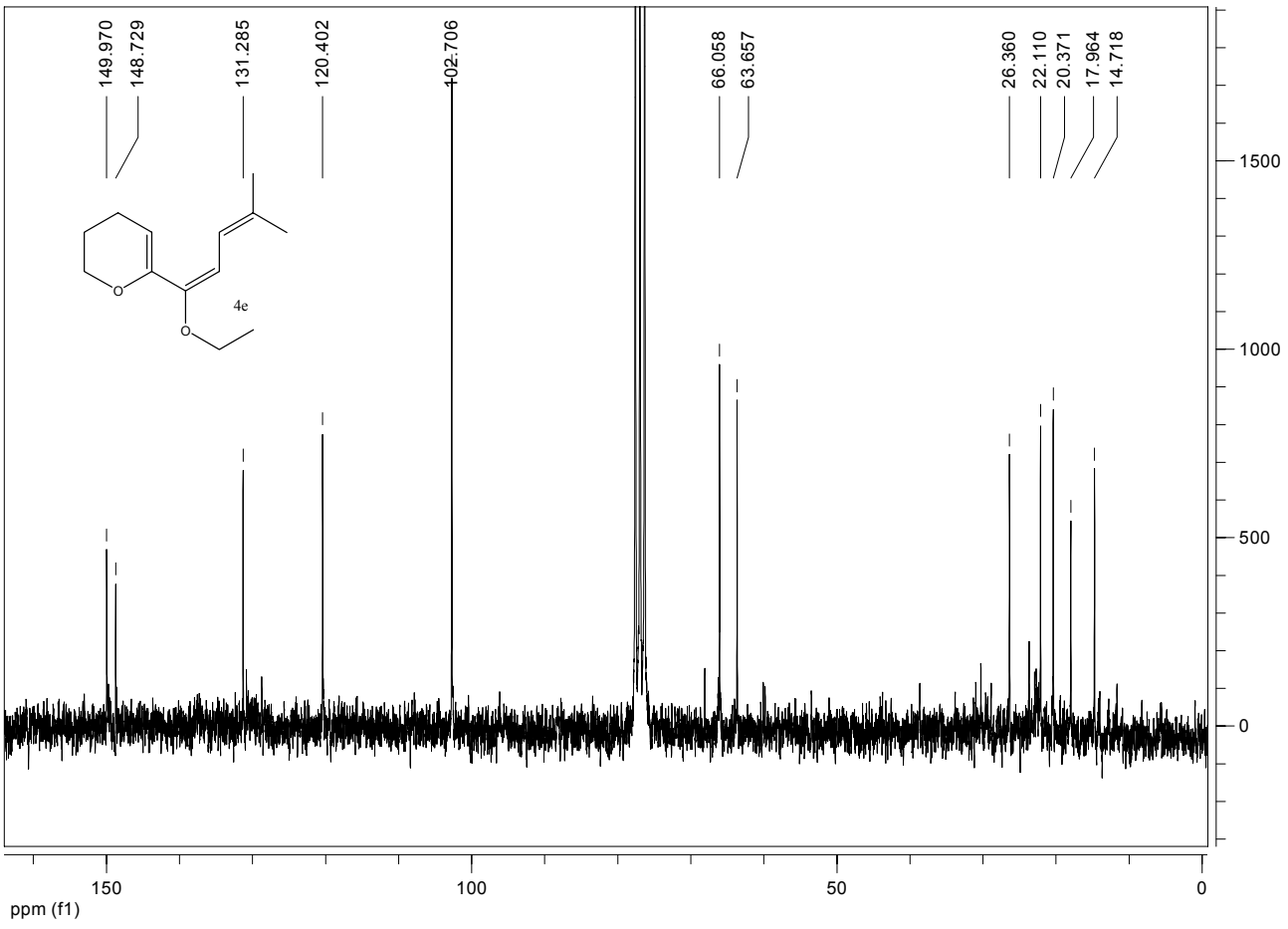
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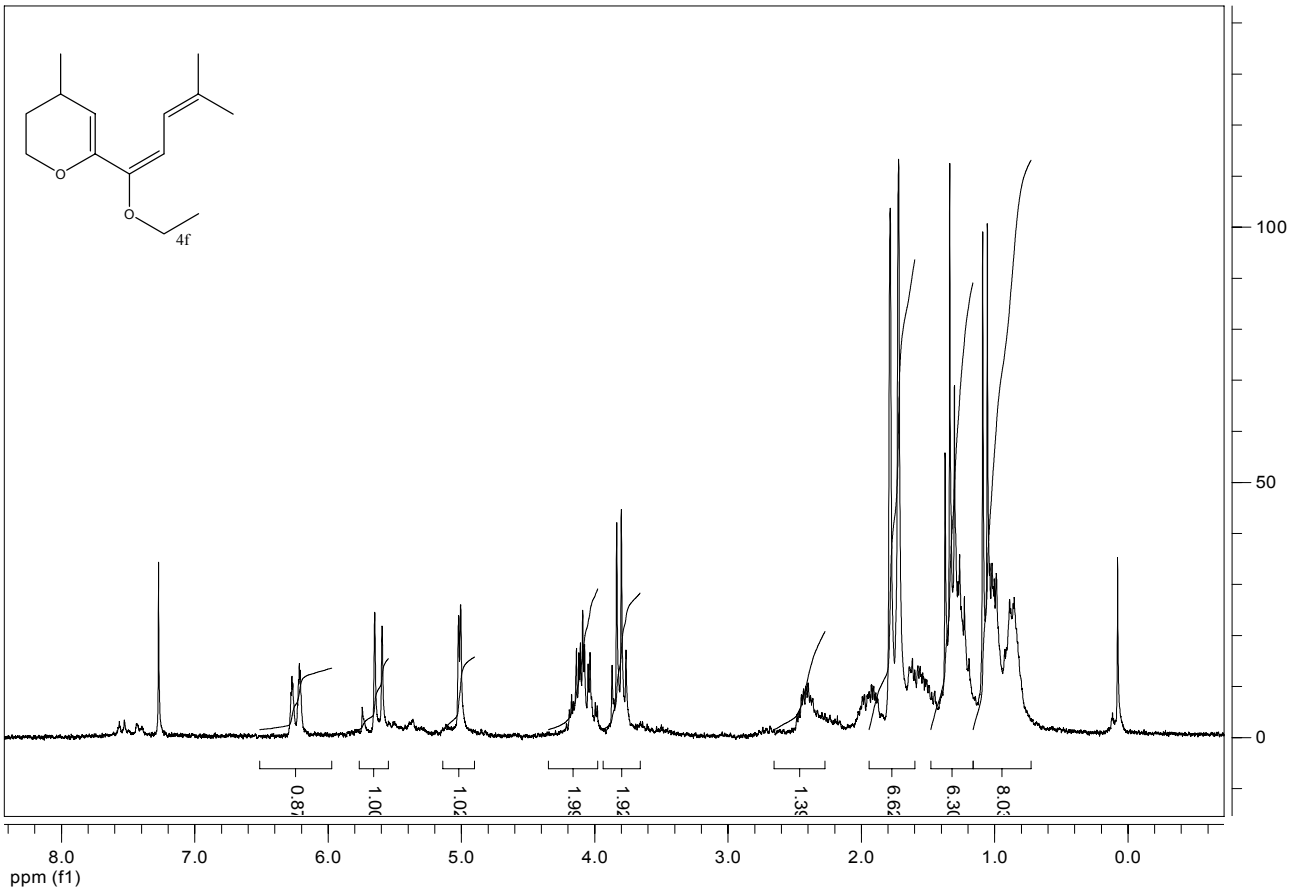
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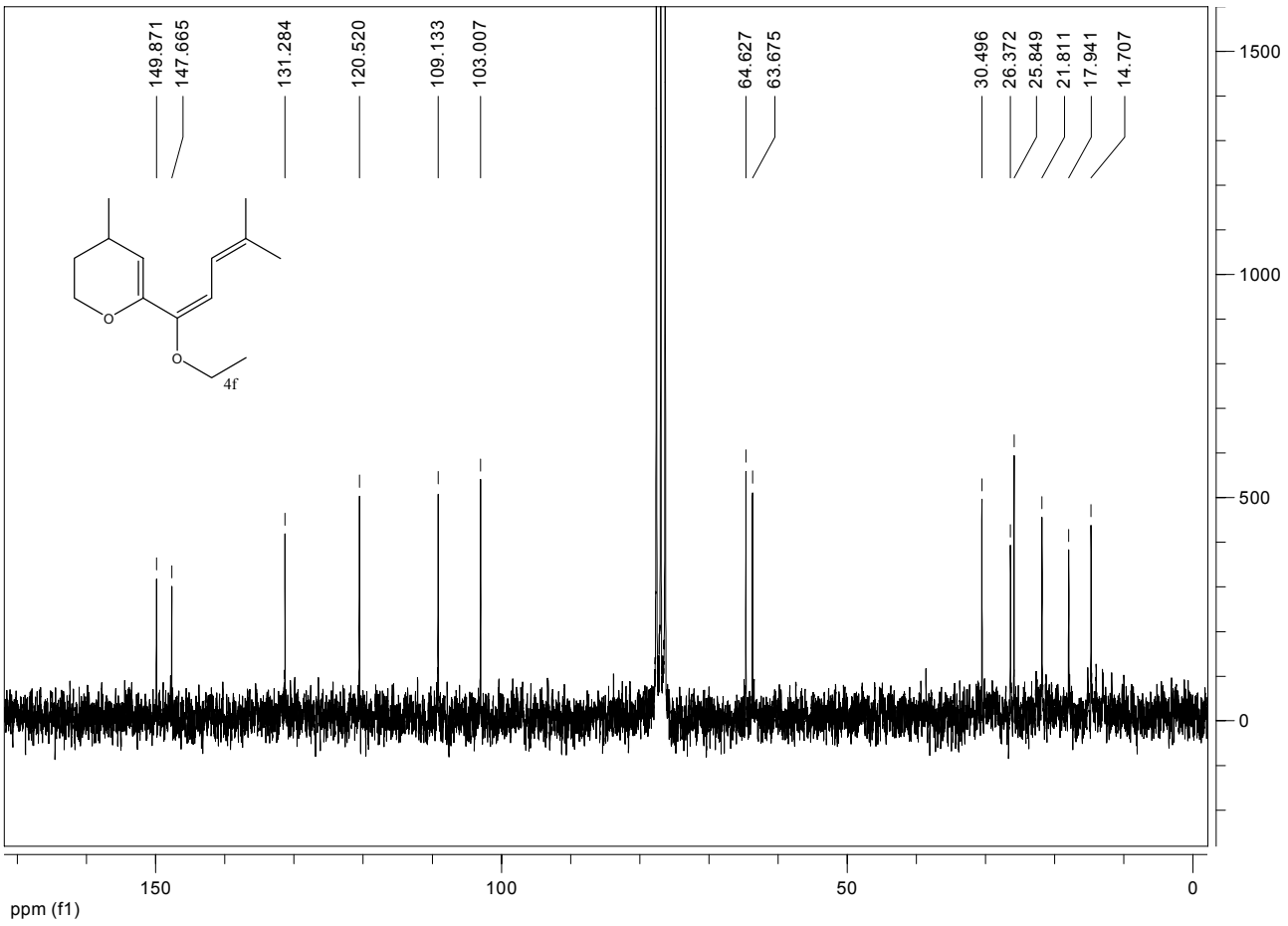
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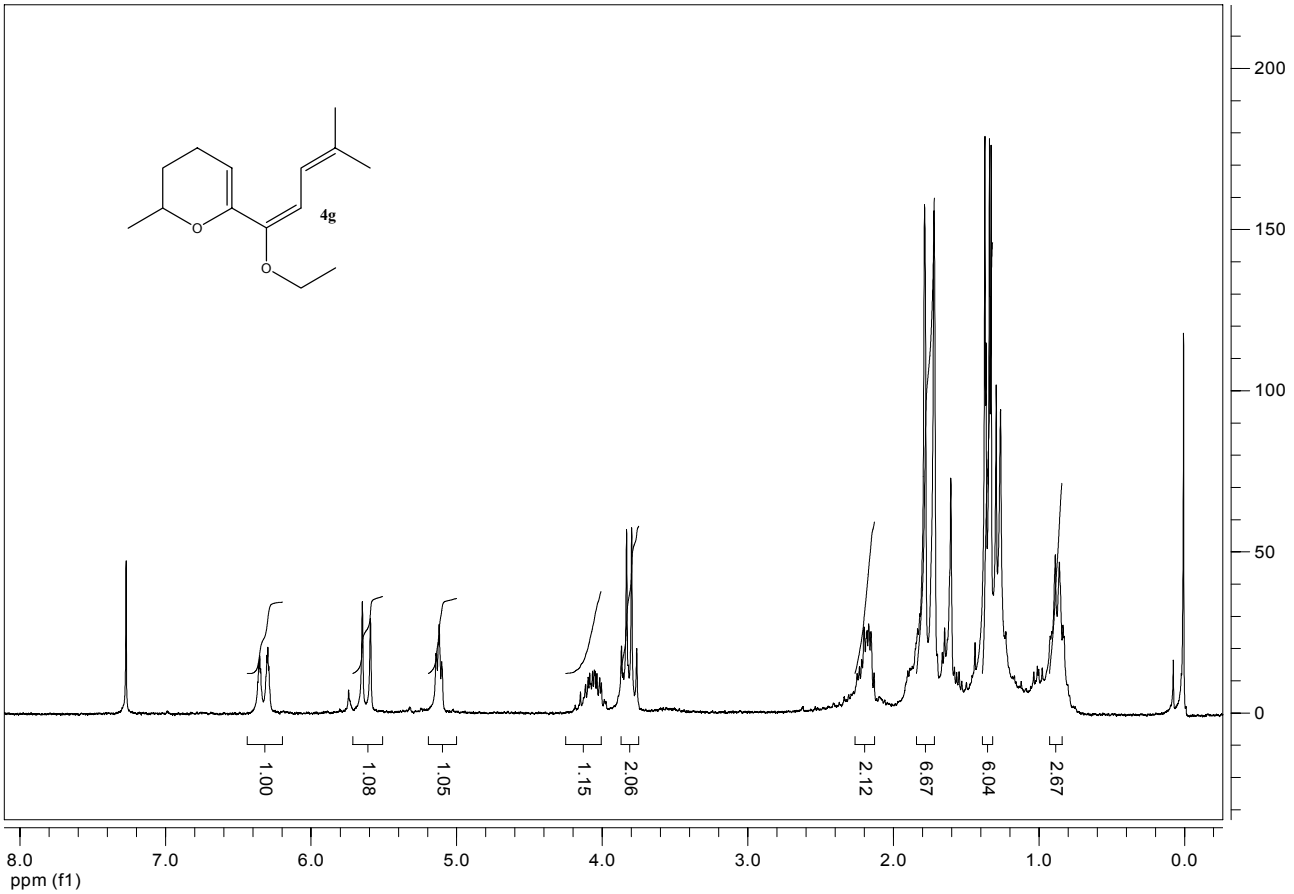
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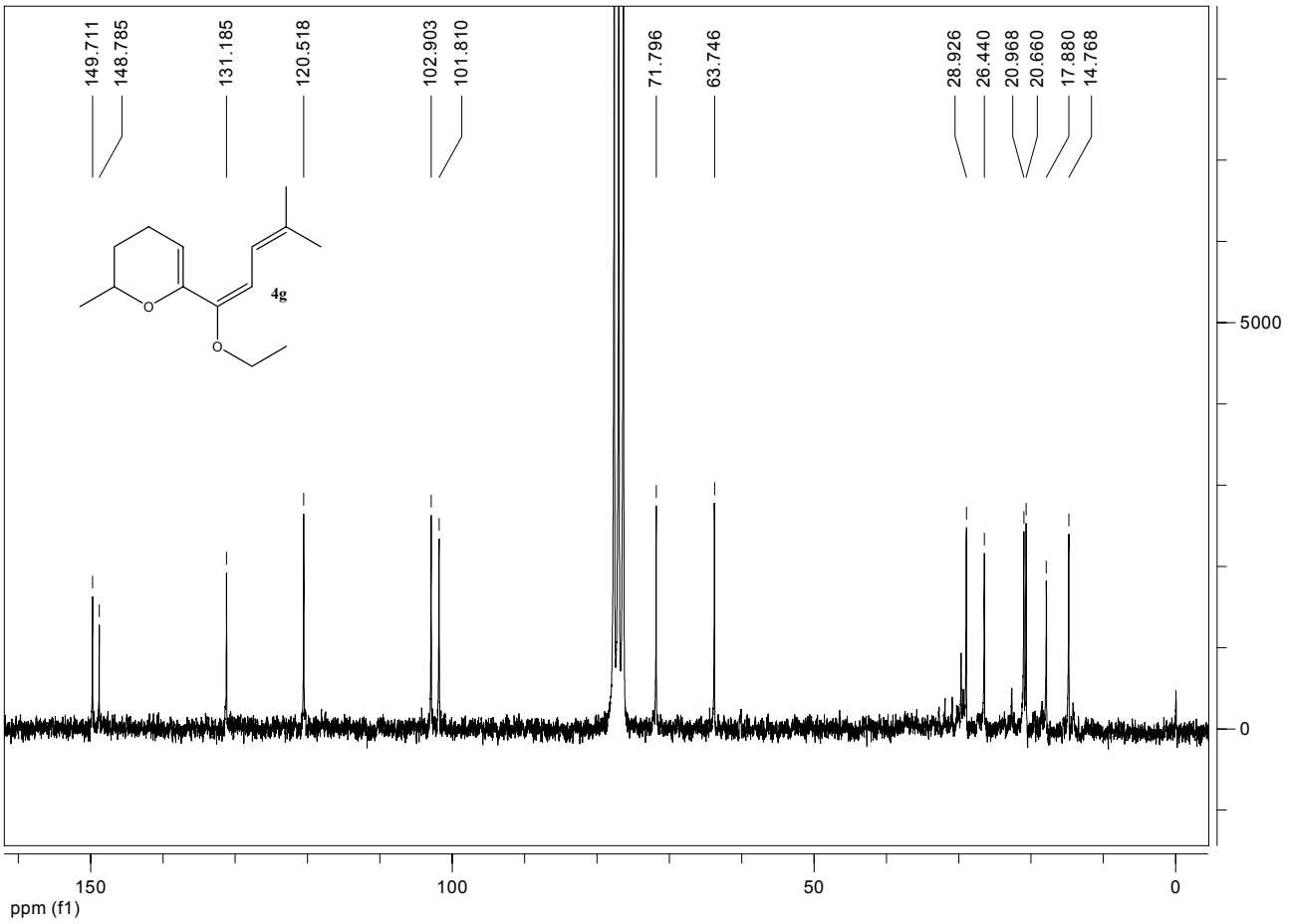
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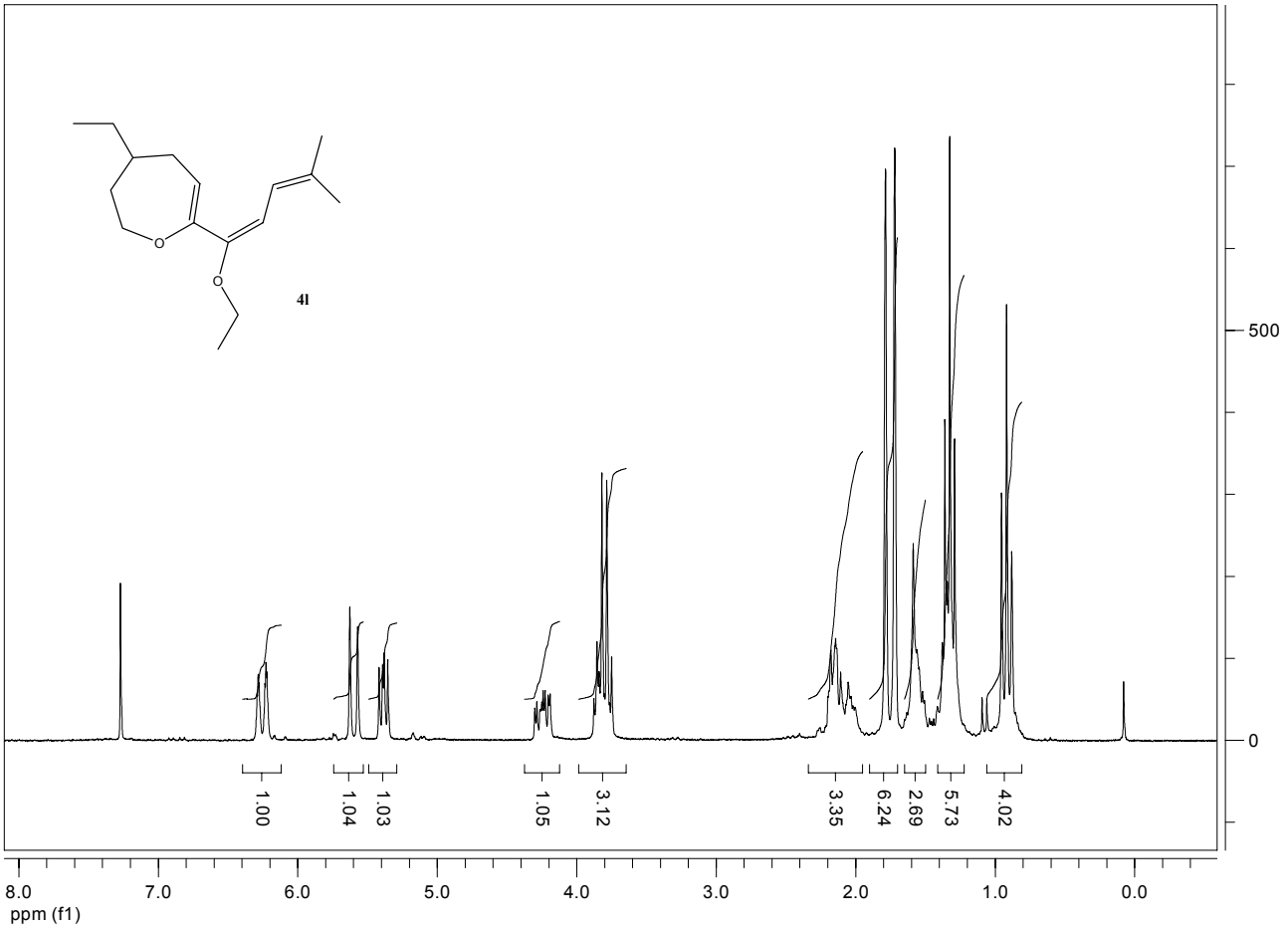
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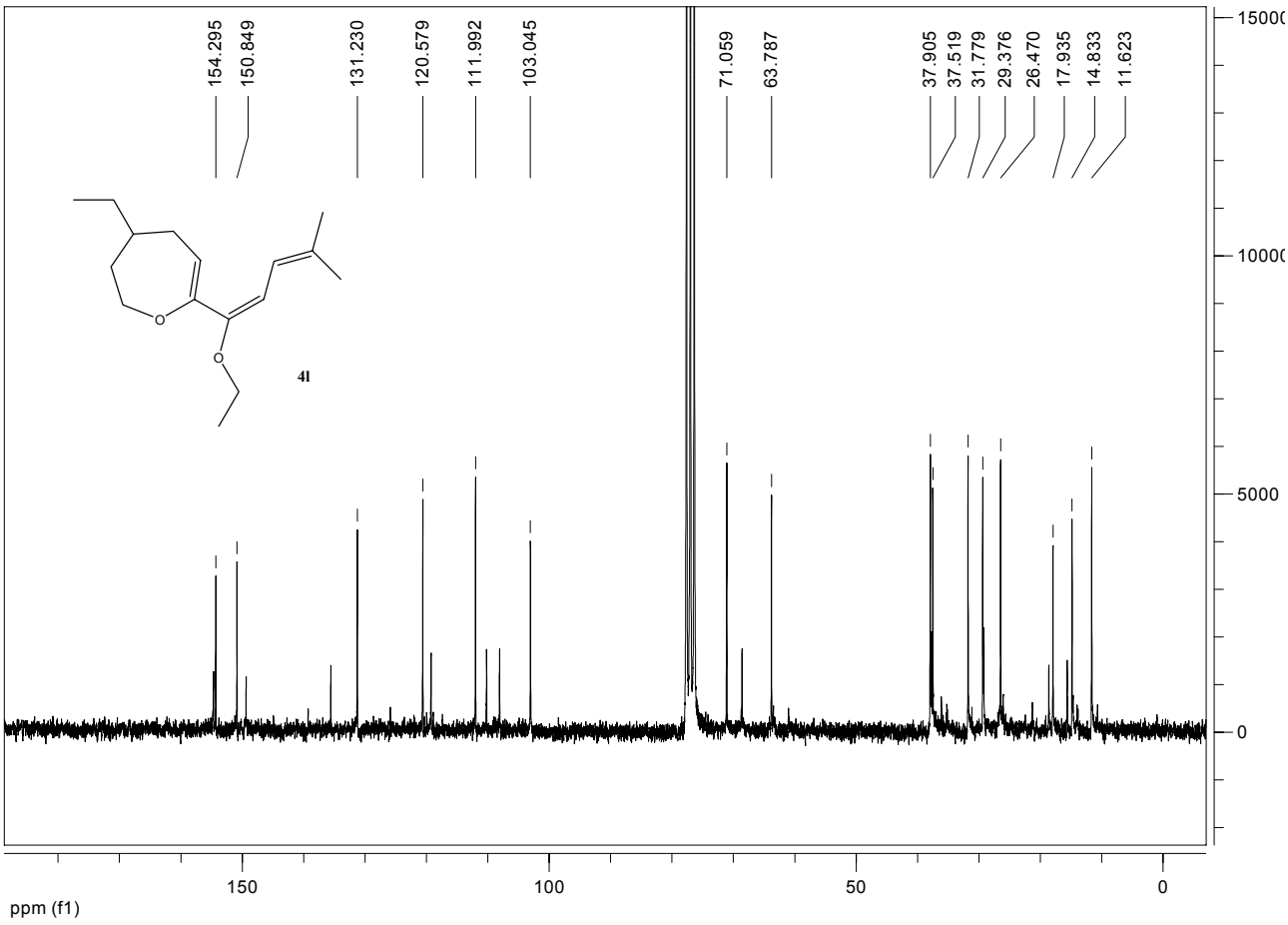
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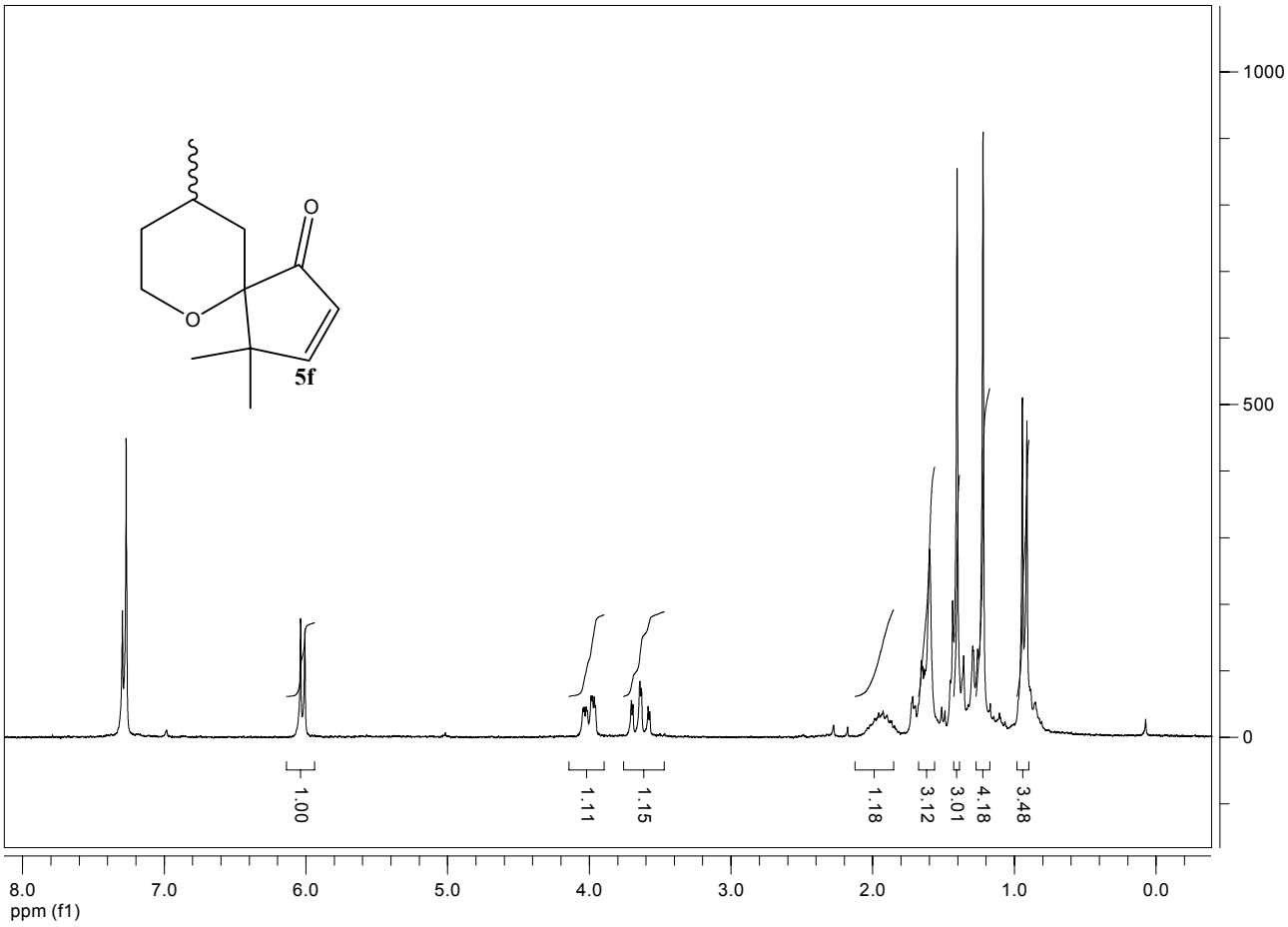
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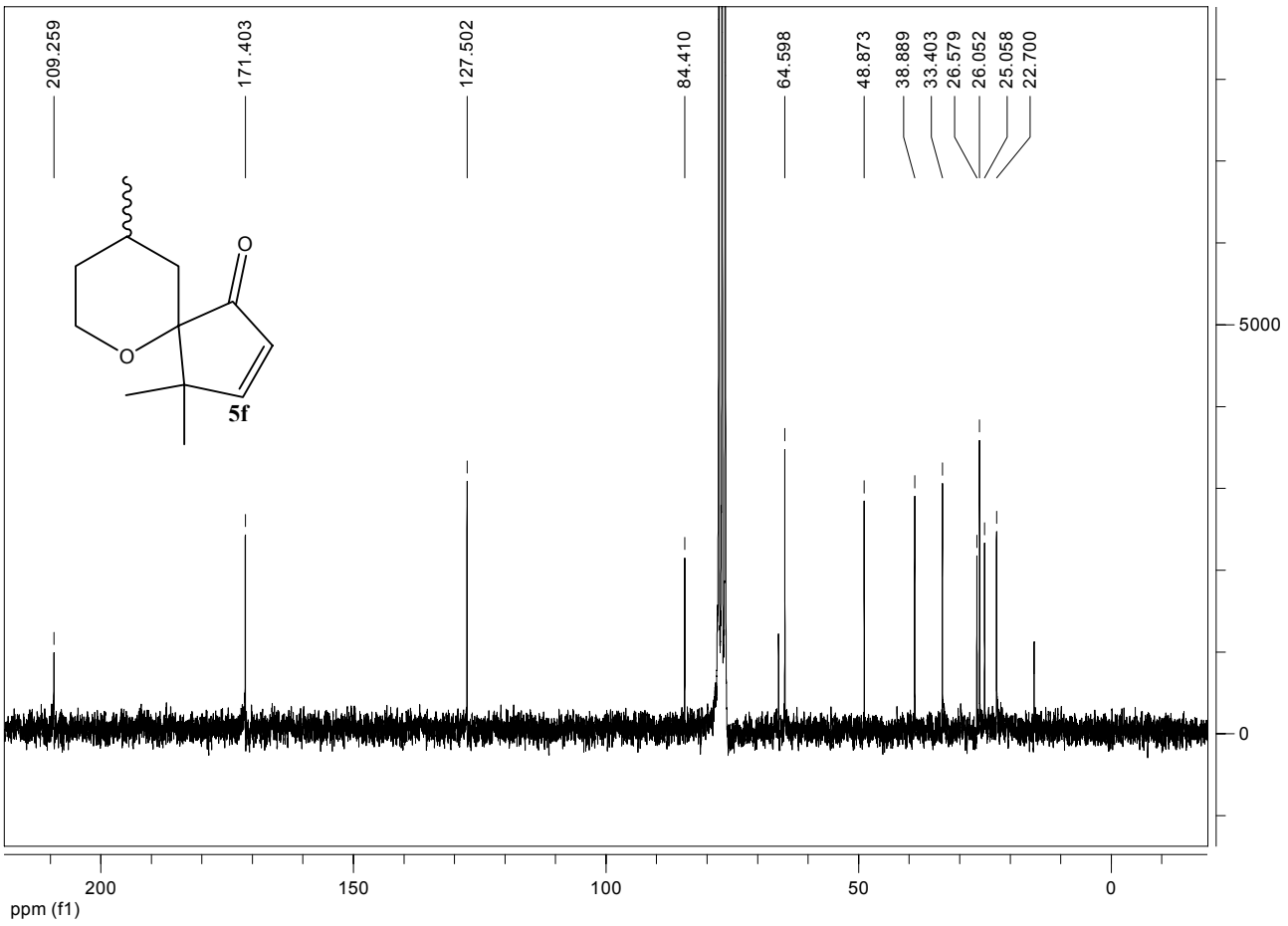
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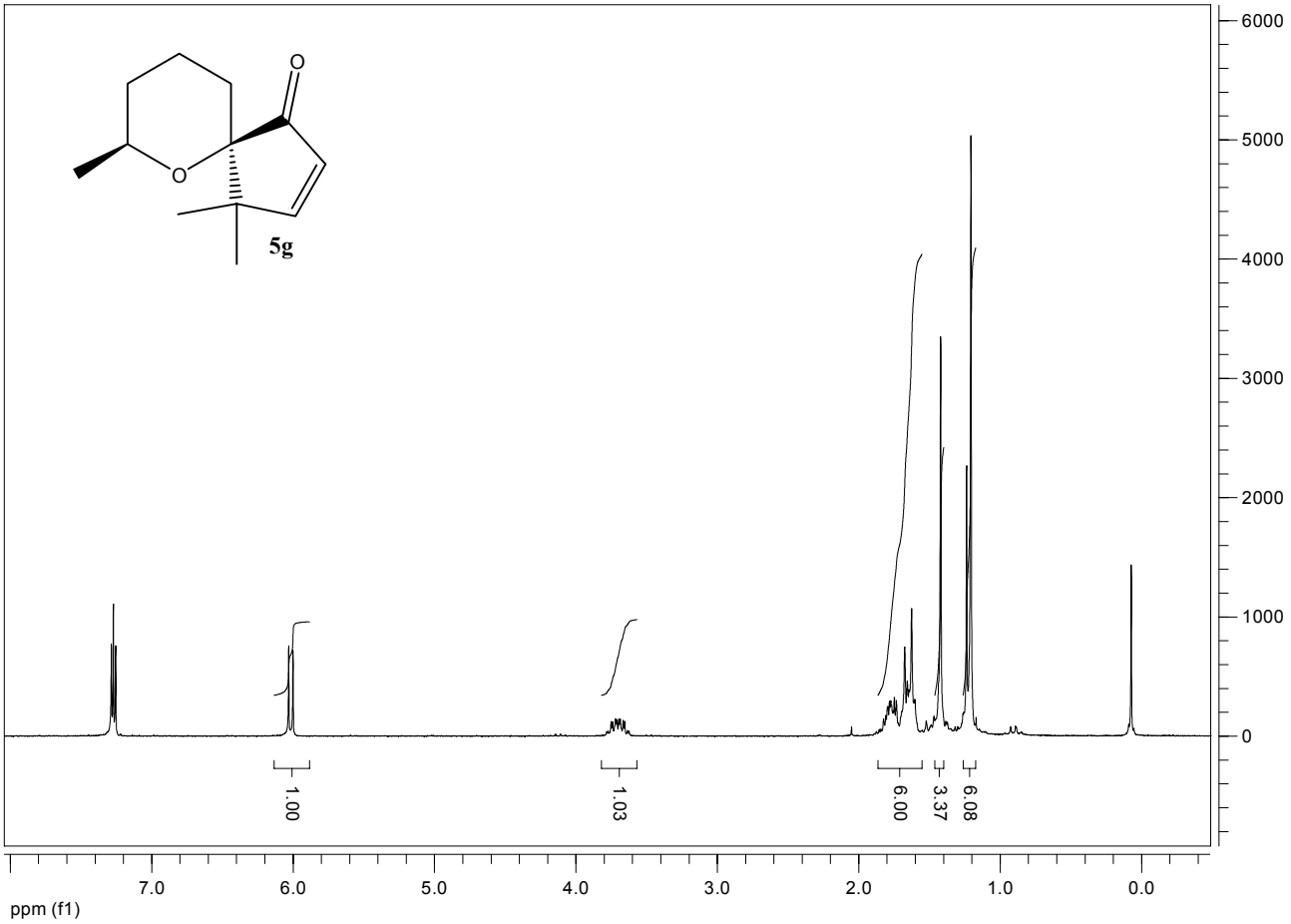
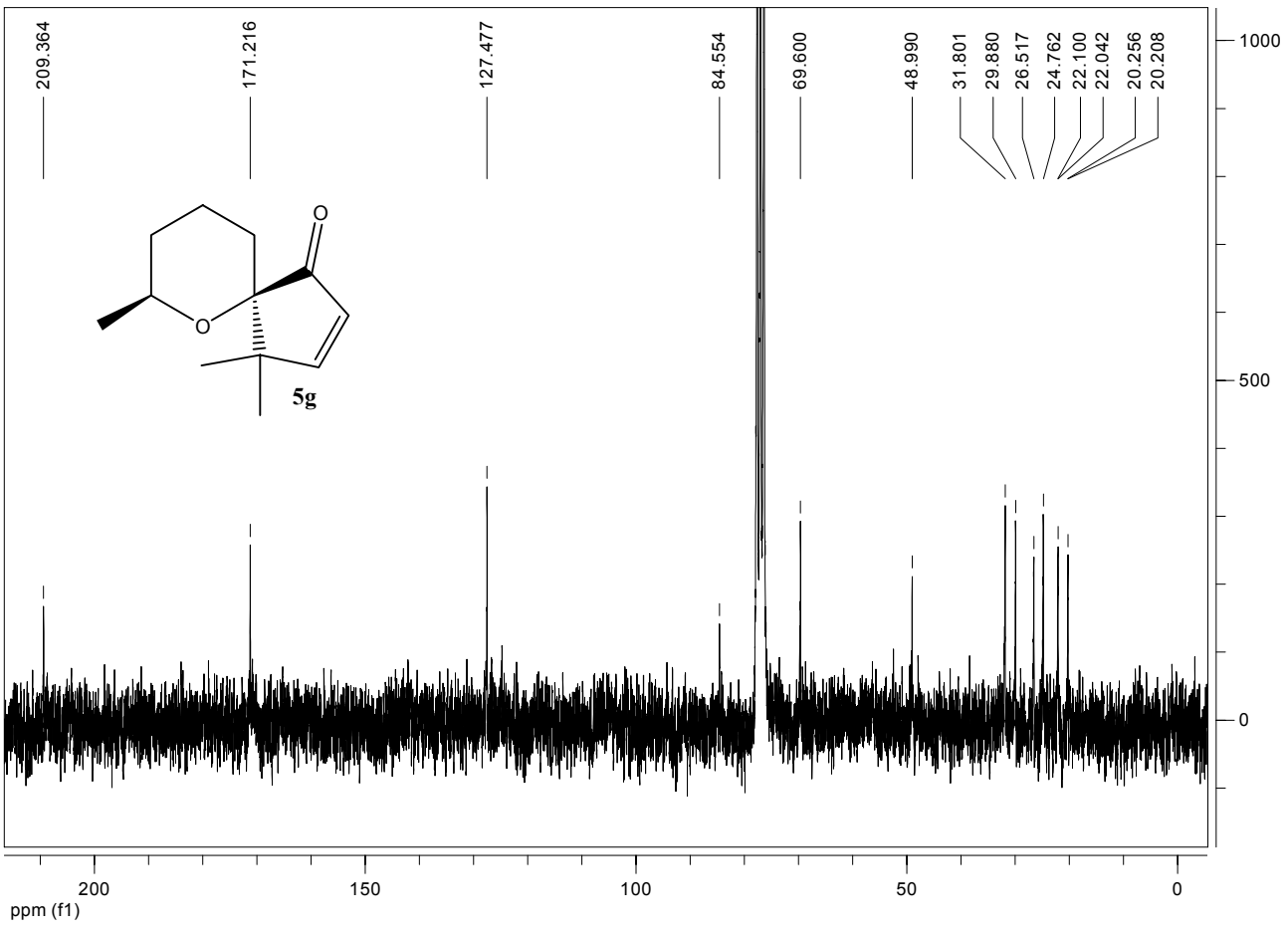
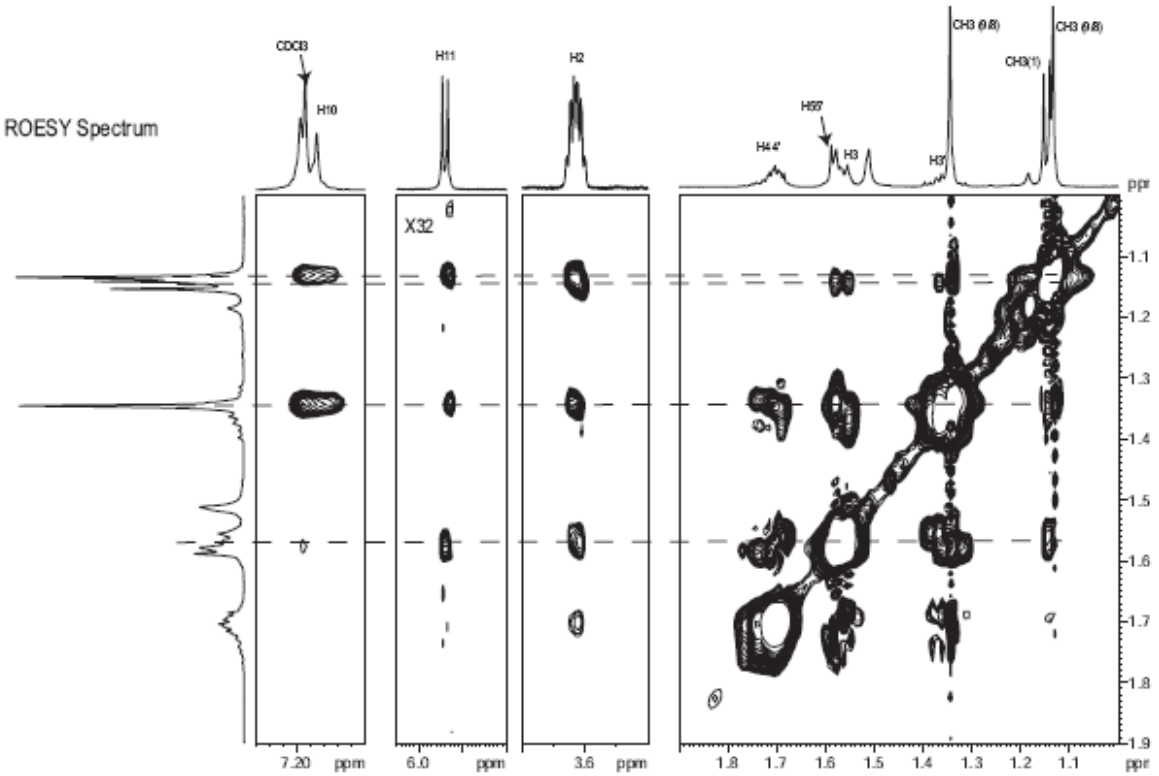
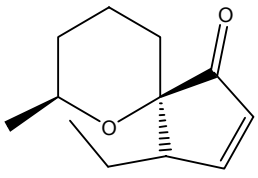
171.403
127.502
84.410
64.598
48.873
38.889
33.403
26.579
26.052
25.058
22.700
O
O
5f
5000
0
200
150
100
50
0
ppm (f1)


O
O
5g
6000
5000
4000
3000
2000
1000
0
1.00
1.03
6.00
3.37
6.08
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)

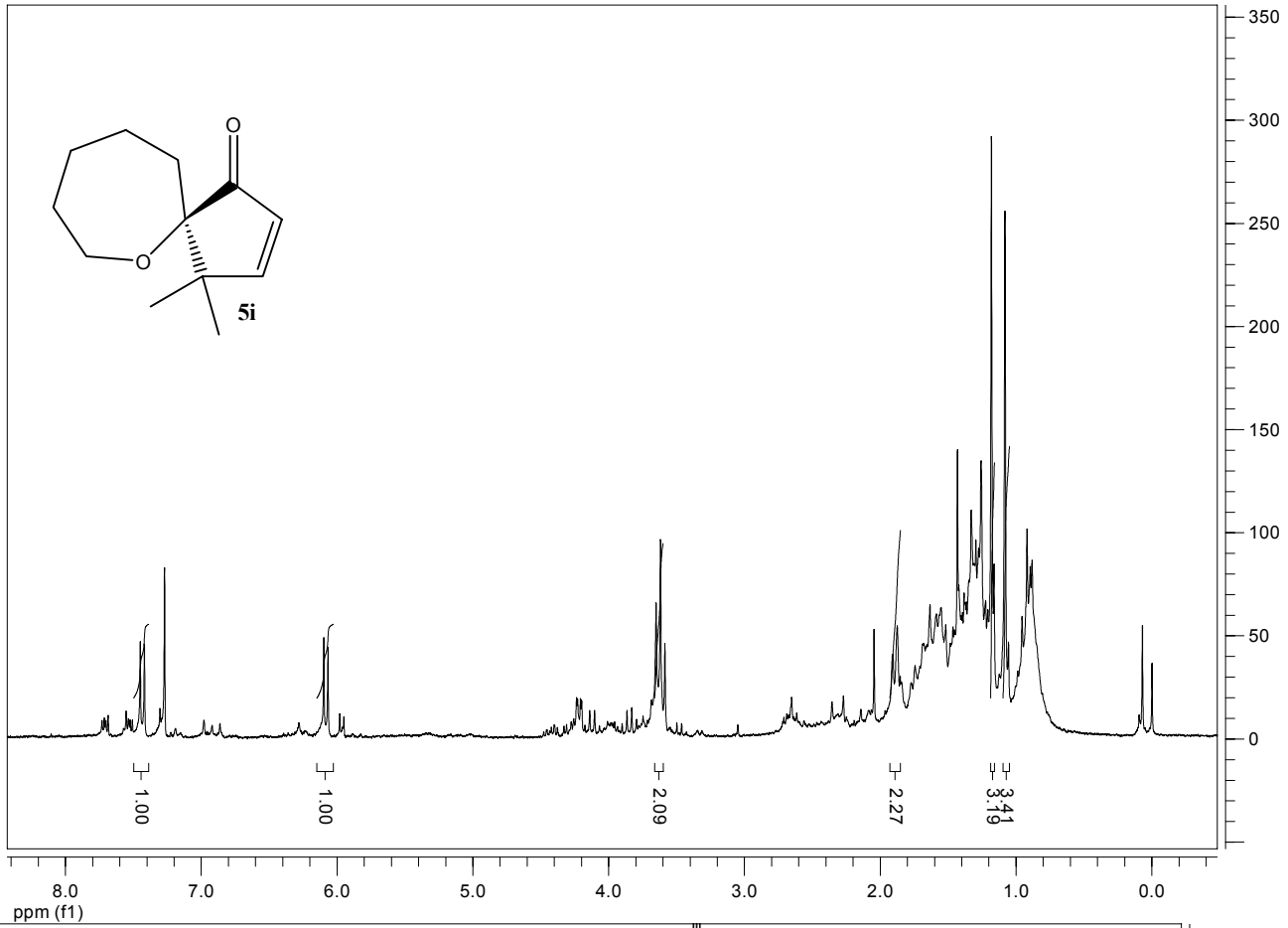

209.364
171.216
127.477
84.554
69.600
48.990
31.801
29.880
26.517
24.762
22.100
22.042
20.256
20.208
O
O
5g
1000
500
0
200
150
100
50
0
ppm (f1)

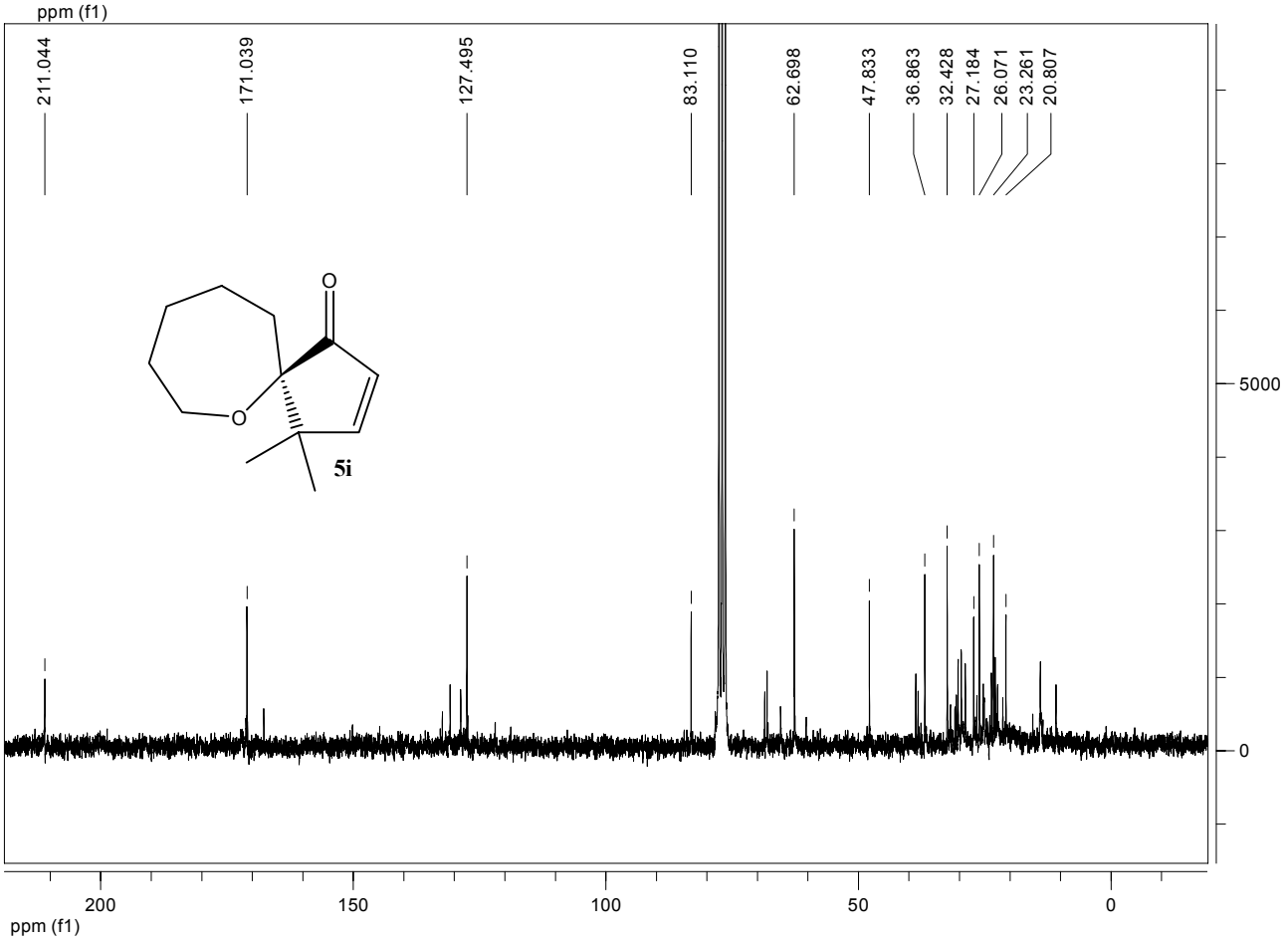

ROESY Spectrum
CDCl3
H10
H11
H2
H4'
H6'
H3
H7'
CH3 (6,8)
CH3 (6,8)
CH3(1)
X32
7.20 ppm
6.0 ppm
3.6 ppm
1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 ppm


O
O

5h

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


O
O
5i
1.00
1.00
2.09
2.27
3.41
3.19
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (f1)
350
300
250
200
150
100
50
0


211.044
171.039
127.495
83.110
62.698
47.833
36.863
32.428
27.184
26.071
23.261
20.807
O
O
5i
5000
0
200
150
100
50
0
ppm (f1)

Crystal structure determination - Colorless crystals of 4,4,7-trimethyl-6-oxaspiro[4.5]dec-2-en-1-one by slow evaporation from methanol. Crystal dimensions : 0.5 x 0.5 x 0.1 mm^3.

Single crystal diffraction data were collected at 100 K, using a Oxford Cryostream low temperature device, on a Oxford Xcalibur CCD area detector diffractometer, using graphite monochromatised Mo Kα (λ = 0.71069Å) radiation. Data reduction and absorption correction were performed using CrysAlis RED 1.171.26 (Oxford Diffraction). The structure was solved by direct methods using SIR2004[ii] and refined by full-matrix least squares using SHELX-97.[iii] All hydrogen atoms were located in the electron density map and then refined using isotropic displacement parameters.

The Crystallographic Information File (CIF) has been deposited by the Cambridge Crystallographic Data Centre, with deposition number CCDC 274489.

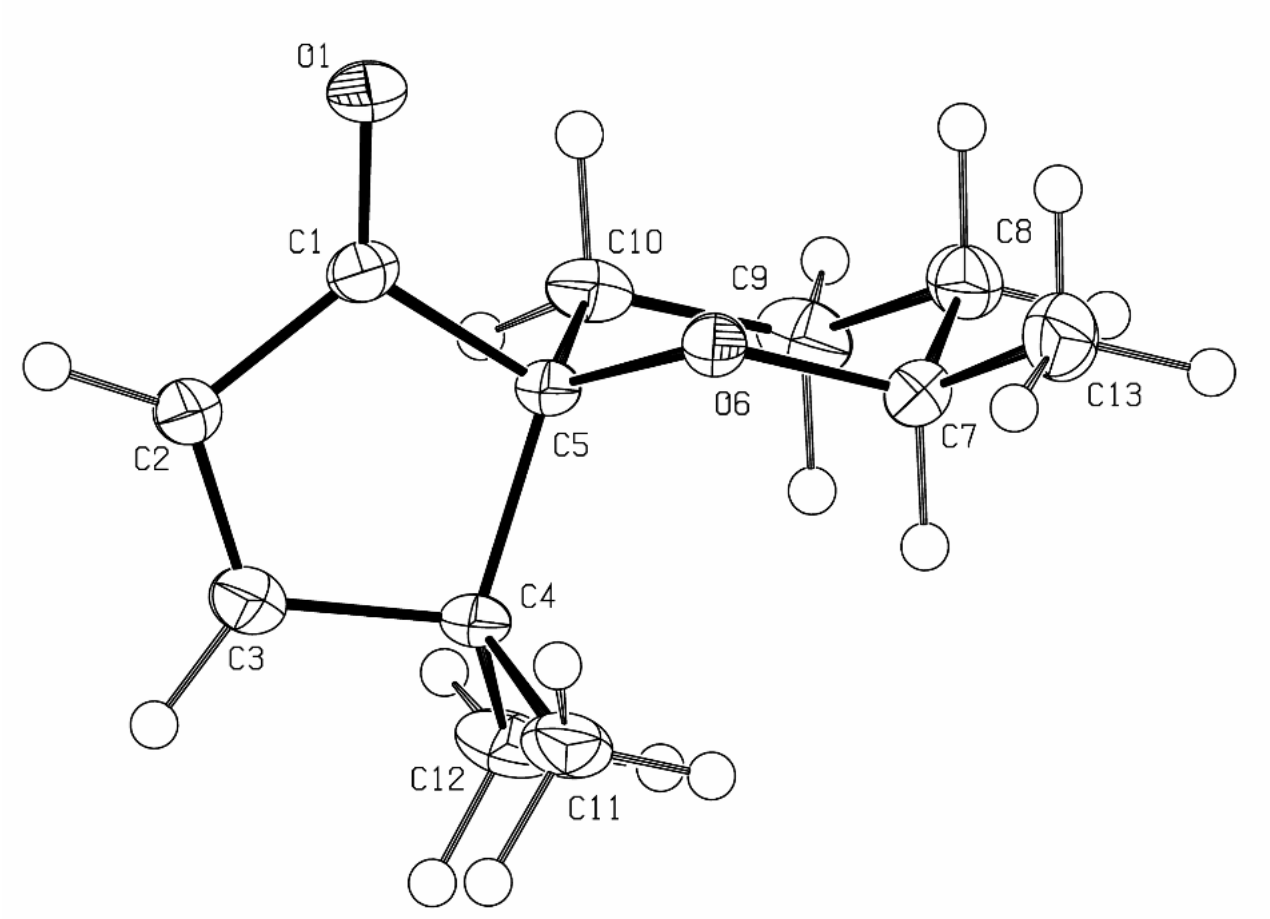


Figure 1. Drawing of the asymmetric unit of 4,4,7-trimethyl-6-oxaspiro[4.5]dec-2-en-1-one, showing the adopted labelling scheme, with atomic displacement ellipsoids drawn at the 50% probability level.

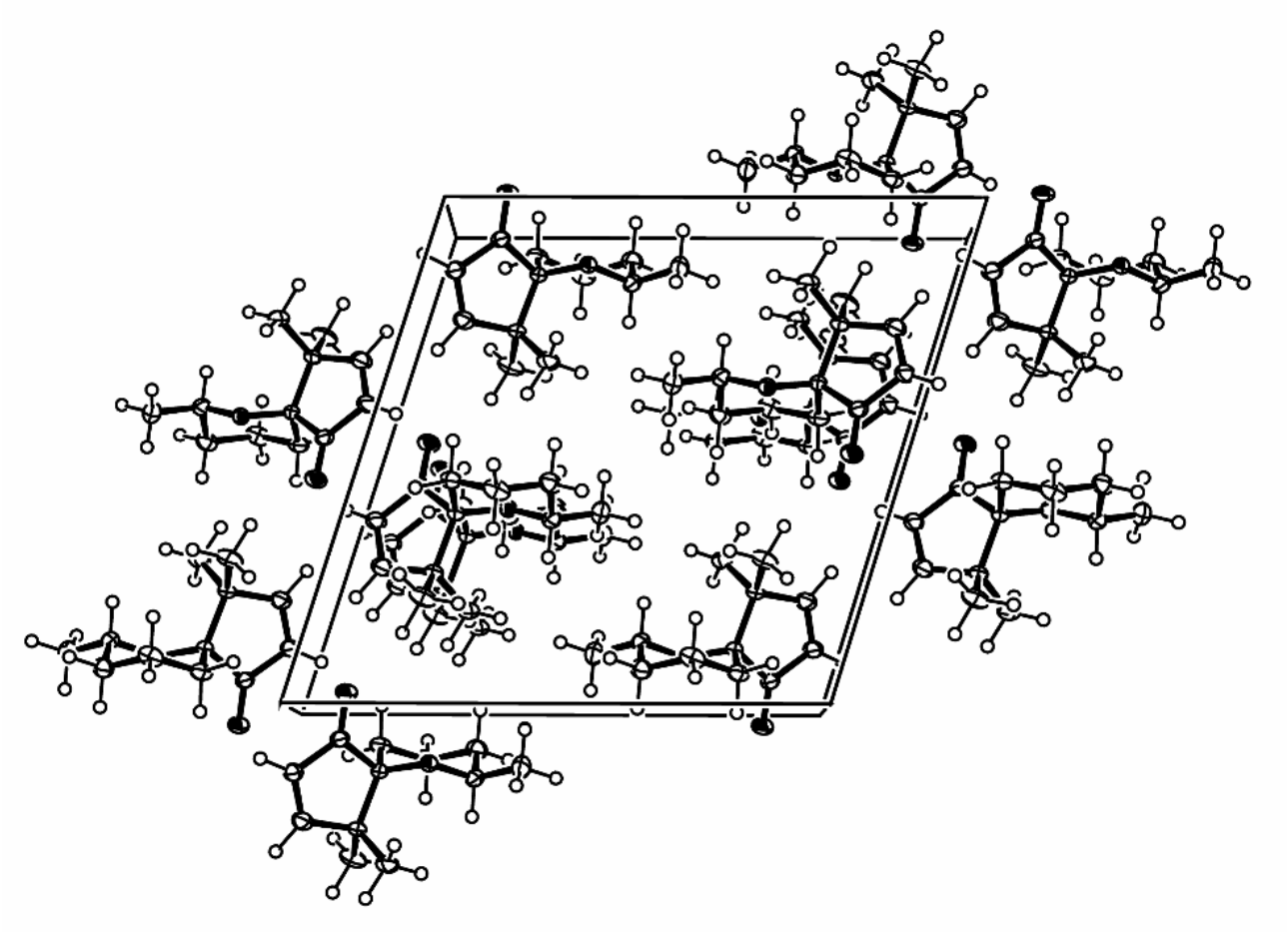


Figure 2. Crystal packing of 4,4,7-trimethyl-6-oxaspiro[4.5]dec-2-en-1-one.

Table 1. Crystal data and structure refinement for 4,4,7-trimethyl-6-oxaspiro[4.5]dec-2-en-1-one

Identification code	CCDC 274489
Empirical formula	C13 H18 O2
Formula weight	206
Temperature	100 K
Wavelength	0.71069 Å
Crystal system	*P21/a*
Space group	Monoclinic
Unit cell dimensions (Å and °)	a = 13.38(5) b = 6.27(9) c = 13.70 β= 107.3(5)
Volume (A^3)	1099
Z	4
Density (calculated)	0.799Mg/m^3
Absorption coefficient	0.057 mm^{-1}
F(000)	296

Crystal size	0.5 x 0.5 x 0.1 mm^3
Theta range for data collection	4.45 to 29.43°.
Index ranges	-18<=h<=15 -8<=k<=8 -17<=l<=18
Reflections collected	7405
Independent reflections	2856 [R(int) = 0.0405]
Completeness to theta = 29.43°	93.8 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2856 / 0 / 200
Goodness-of-fit on F^2	1.072
Final R indices [I>2sigma(I)]	R1 = 0.0638, wR2 = 0.1319
R indices (all data)	R1 = 0.0965, wR2 = 0.1517
Largest diff. peak and hole	0.309 and -0.247 $e.Å^{-3}$

1

[ii] M.C. Burla, R. Caliandro, M. Camalli, B. Carrozzini, G.L. Cascarano, L. De Caro, C. Giacovazzo, G. Polidori, R. Spagna Sir2004: an improved tool for crystal structure determination and refinement. J. Appl. Cryst. (2005). **38**, 381-388. Web site: http://www.ic.cnr.it/

[iii] Programs for Crystal Structure Analysis (Release 97-2). Sheldrick, G.M., Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, 1998.; Web site: http://shelx.uni-ac.gwdg.de/SHELX/.