

# FUJIMURA 4029-4031

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The Synthesis of Cyclic Enol Ethers via Molybdenum  
Alkylidene-Catalyzed Ring-Closing Metathesis

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**Supplementary Material: Experimental Section**

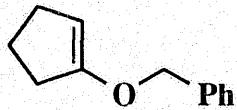
**General.**  $^1\text{H}$  spectra were recorded on a General Electric QE-300 spectrometer at ambient temperature. Data are reported as follows: chemical shift in parts per million downfield from tetramethylsilane ( $\delta$  scale) with the solvent resonance employed as internal standard ( $\text{CDCl}_3$  at  $\delta$  7.26,  $\text{C}_6\text{D}_6$  at  $\delta$  7.15,  $\text{DMSO-d}_6$  at  $\delta$  2.49), multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), integration, coupling constant (Hz), and assignment.

$^{13}\text{C}$  NMR spectra were recorded on a General Electric QE-300 spectrometer at ambient temperature.  $^{13}\text{C}$  chemical shifts are reported in parts per million downfield from tetramethylsilane ( $\delta$  scale) with the solvent resonance employed as internal standard ( $\text{CDCl}_3$  at  $\delta$  77.0,  $\text{C}_6\text{D}_6$  at  $\delta$  128.0,  $\text{DMSO-d}_6$  at  $\delta$  39.5). All  $^{13}\text{C}$  spectra were determined with complete proton decoupling.

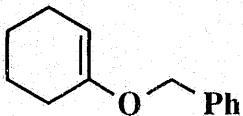
Infrared spectra were obtained on a Parkin-Elmer 1600 Series FTIR. High resolution mass spectra were provided by the Southern California Mass Spectrometry Facility (University of California, Riverside). Analytical thin layer chromatography of enol ethers was accomplished using EM Reagents 0.2mm aluminum oxide 60F<sub>254</sub> neutral (typ. E) foil. In case other than enol ethers, EM Reagents 0.25mm silica gel 60 plates was used. Flash chromatography of enol ethers was performed on Aldrich aluminum oxide basic Brockmann grade I (150 mesh), tuned to Brockmann grade III according to the literature<sup>1</sup>. In case other than enol ethers, EM reagents silica gel 60 (230-400 mesh) was used.

Argon was purified by passage through the column of BASF RS-11 (Chemalog) and Linde 4Å molecular sieves. Solvents (Benzene, n-Henane) were purified by passage through the column of La Roche A-2 Alumina and Engelhard Q-5 reactant (supported copper oxide), and degassed by Freeze-Pump-Thaw (FPT) three times and stored under argon in a flask with a Teflon valve. THF was purified by passage through two columns of La Roche A-2 Alumina and degassed by FPT three times and stored same way as above. n-Pentane was distilled from sodium benzophenone ketyl and degassed by FPT and stored under argon in a flask with a Teflon valve. **3** was prepared according to the method of Schrock.<sup>2</sup>

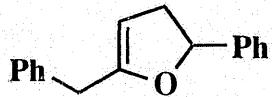
All reactions were conducted an atmosphere of argon in oven-dried glassware with magnetic stirring.



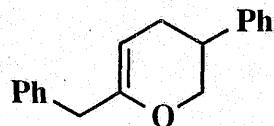
**1-Benzylloxycyclopentene:** IR (neat) 2933, 2852, 1645, 1344, 1109, 735, 698 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.65-7.05 (m, 5H, aromatic H), 4.63 (s, 2H, OCH<sub>2</sub>Ph), 4.46-4.43 (m, 1H, CH<sub>2</sub>CHC), 2.47-2.40 (m, 2H, CCH<sub>2</sub>CH<sub>2</sub>), 2.34-2.27 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>CH), 1.80-1.70 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 160.25, 137.84, 128.56, 127.87, 127.79, 94.2, 71.4, 32.4, 29.4, 21.6; Mass spectrum m/z (relative intensity): 174 (M<sup>+</sup>, 10), 91 (100), 84 (10), 65 (12); HRMS, m/z Calcd for C<sub>12</sub>H<sub>14</sub>O (M<sup>+</sup>): 174.1045 Found: 174.1042.



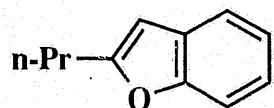
**1-Benzylloxycyclohexene:** IR (neat) 2929, 2859, 1666, 1368, 1183, 1027, 781, 734, 697 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.65-7.05 (m, 5H, aromatic H), 4.60 (t, 1H, J = 3.9, CCHCH<sub>2</sub>), 4.58 (s, 2H, OCH<sub>2</sub>Ph), 2.25-2.15 (m, 2H, CCH<sub>2</sub>CH<sub>2</sub>), 2.10-1.98 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>CH), 1.60-1.40 (m, 4H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 154.88, 138.35, 128.53, 128.18, 127.87, 94.47, 68.56, 28.32, 23.91, 23.24, 23.15; Mass spectrum m/z (relative intensity): 188 (M<sup>+</sup>, 14), 144 (6) 91 (100), 65 (6); HRMS, m/z Calcd for C<sub>13</sub>H<sub>16</sub>O (M<sup>+</sup>): 188.1201 Found: 188.1198.



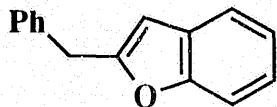
**2-Benzyl-5-phenyl-4,5-dihydrofuran:** IR (neat) 3062, 3022, 1672, 1492, 1356, 1140, 1021, 942, 754, 696 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.95-7.91 (m, 2H, aromatic H), 7.40-7.00 (m, 8H, aromatic H), 5.33 (s, 1H, CCHCH<sub>2</sub>), 5.06 (dd, 1H, J = 7.6, 6.5, OCHCH<sub>2</sub>), 2.40-2.34 (m, 2H, CCH<sub>2</sub>Ph), 1.84-1.73 (m, 1H, CHCHHCH), 1.57-1.45 (m, 1H, CHCHHCH); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 157.18, 141.86, 137.71, 128.71, 128.63, 127.89, 127.84, 125.62, 125.10, 98.16, 84.81, 33.15, 31.14; Mass spectrum m/z (relative intensity): 236 (M<sup>+</sup>, 100), 181 (17) 145 (37), 118 (98), 91 (72), 77 (17); HRMS, m/z Calcd for C<sub>17</sub>H<sub>16</sub>O (M<sup>+</sup>): 236.1201 Found: 236.1191.



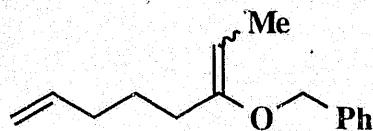
**6-Benzyl-3-phenyl-3,4-dihydro-2H-pyran:** IR (neat) 3003, 2917, 1678, 1495, 1454, 1174, 1161, 1072, 1038, 757, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.30-6.80 (m, 10H, aromatic H), 4.52-4.49 (m, 1H, CCHCH<sub>2</sub>), 3.93 (ddd, 1H, J = 10.4, 3.7, 1.7, OCHHCH), 3.60 (dd, 1H, J = 10.4, 10.4, OCHHCH), 3.31 (s, 2H, CCH<sub>2</sub>Ph), 2.79-2.69 (m, 1H, CH<sub>2</sub>CHCH<sub>2</sub>), 2.19-1.95 (m, 2H, CHCH<sub>2</sub>CH); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 153.96, 142.63, 139.14, 129.36, 128.75, 128.52, 127.60, 126.83, 126.52, 96.98, 70.66, 41.03, 38.92, 28.61; Mass spectrum m/z (relative intensity): 250 (M<sup>+</sup>, 16), 104 (100), 91 (37), 77 (10), 65 (10); HRMS, m/z Calcd for C<sub>18</sub>H<sub>18</sub>O (M<sup>+</sup>): 250.1358 Found: 250.1356.



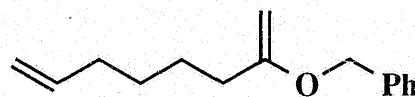
**2-Propylbenzofuran:** IR (neat) 2966, 1596, 1578, 1455, 1249, 1167, 944, 791, 750, 732 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.41-7.35 (m, 2H, **4-H,7-H**), 7.13-7.02 (m, 2H, **5-H,6-H**), 6.11 (m, 1H, **3-H**), 2.45 (td, 2H, J = 7.4, 0.9, CH<sub>2</sub>CH<sub>2</sub>C), 1.54 (tq, 2H, J = 7.4, 7.4, CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>), 0.77 (t, 3H, J = 7.4, CH<sub>3</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 159.54, 155.31, 129.50, 123.53, 122.78, 120.52, 111.09, 102.31, 30.50, 21.19, 13.70; Mass spectrum m/z (relative intensity): 160 (M<sup>+</sup>, 26), 131 (100), 115 (5), 103 (5), 77 (14); HRMS, m/z Calcd for C<sub>11</sub>H<sub>12</sub>O (M<sup>+</sup>): 160.0888 Found: 160.0889.



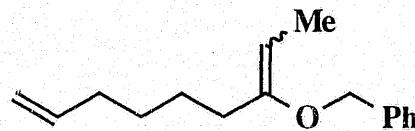
**2-Benzylbenzofuran:** IR (neat) 3013, 1600, 1454, 1252, 750, 704 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.34-7.28 (m, 2H, **4-H,7-H**), 7.15-6.99 (m, 7H, aromatic H, **5-H,6-H**), 6.06 -6.05(m, 1H, **3-H**), 3.75 (s, 2H, PhCH<sub>2</sub>C); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 158.16, 155.55, 137.54, 129.29, 129.21, 128.78, 126.89, 123.82, 122.87, 120.71, 111.22, 103.69, 35.05; Mass spectrum m/z (relative intensity): 208 (M<sup>+</sup>, 88), 207 (100), 178 (23), 131 (67), 84 (31), 77 (17); HRMS, m/z Calcd for C<sub>15</sub>H<sub>12</sub>O (M<sup>+</sup>): 208.0888 Found: 208.0892.



**3-Benzyl-2-methyl-7-octadiene:** IR (neat) 2932, 2861, 1681, 1643, 1454, 1324, 1056, 1027, 911, 734, 696 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) (Z) δ 7.45-7.05 (m, 5H, aromatic H), 5.79-5.65 (m, 1H, H<sub>2</sub>CCHCH<sub>2</sub>), 5.05-4.94 (m, 2H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.56 (s, 2H, OCH<sub>2</sub>Ph), 4.56 (q, 1H, J = 6.7, CH<sub>3</sub>CHC), 2.10-1.93 (m, 4H, CHCH<sub>2</sub>CH<sub>2</sub>, CH<sub>2</sub>CH<sub>2</sub>C), 1.68 (d, 3H, J = 6.7, CH<sub>3</sub>CH), 1.63-1.47 (m, 2H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 154.77, 138.91, 129.59, 128.62, 128.52, 127.82, 127.44, 114.93, 105.18, 70.30, 33.48, 31.67, 26.75; Mass spectrum m/z (relative intensity): 217 (MH<sup>+</sup>, 100), 199 (45), 173 (10), 127 (20), 91 (84); HRMS, m/z Calcd for C<sub>15</sub>H<sub>21</sub>O (MH<sup>+</sup>): 217.1592 Found: 217.1588.

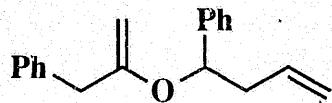


**2-Benzyl-1-methyl-7-octadiene:** IR (neat) 2929, 1653, 1604, 1455, 1274, 911, 799, 737, 696 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.35-7.05 (m, 5H, aromatic H), 5.89-5.75 (m, 1H, H<sub>2</sub>CCHCH<sub>2</sub>), 5.04-4.92 (m, 2H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.75 (s, 2H, OCH<sub>2</sub>Ph), 3.97 (d, 1H, J = 11.5, CCHH), 3.96 (d, 1H, J = 11.5, CCHH), 2.15 (t, 2H, J = 7.3, CCH<sub>2</sub>CH<sub>2</sub>), 1.95 (dt, 2H, J = 6.7, 6.7, H<sub>2</sub>CCHCH<sub>2</sub>), 1.61-1.51 (m, 2H, CCH<sub>2</sub>CH<sub>2</sub>), 1.38-1.30 (m, 2H, CHCH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 163.43, 138.98, 128.53, 127.75, 127.67, 127.64, 114.53, 81.91, 69.46, 35.35, 33.87, 28.73, 27.23; Mass spectrum m/z (relative intensity): 217 (MH<sup>+</sup>, 43), 199 (37), 173 (10), 157 (7), 133 (17), 117 (19), 91 (100); HRMS, m/z Calcd for C<sub>15</sub>H<sub>21</sub>O (MH<sup>+</sup>): 217.1592 Found: 217.1595.

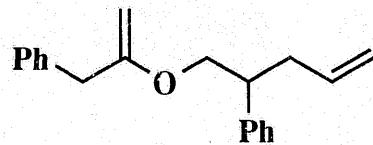


**3-Benzyl-2-methyl-8-nonadiene:** IR (neat) 2918, 2848, 1672, 1448, 1319, 1184, 1048, 908, 732, 691 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) (Z) δ 7.40-7.05 (m, 5H, aromatic H), 5.79-5.65 (m, 1H, H<sub>2</sub>CCHCH<sub>2</sub>), 5.03-4.92 (m, 2H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.56 (s, 2H, OCH<sub>2</sub>Ph), 4.56 (q, 1H, J = 6.8, CHCH<sub>3</sub>), 2.40-1.90 (m, 4H, CCH<sub>2</sub>CH<sub>2</sub>, H<sub>2</sub>CCHCH<sub>2</sub>), 1.67 (d, 3H, J = 6.8, CHCH<sub>3</sub>), 1.62-1.24 (m, 4H, CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) (E/Z mixture) δ 155.15, 138.97, 128.52, 127.21, 114.58, 105.01, 91.73, 81.91, 70.42, 69.46, 68.73, 33.90, 32.24, 30.12, 28.85, 28.76, 27.24, 27.05, 10.65; Mass spectrum m/z (relative intensity): 231 (MH<sup>+</sup>, 91), 213 (25), 188 (19),

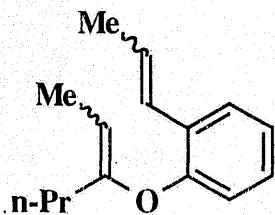
147 (12), 139 (21), 91 (100); HRMS, m/z Calcd for C<sub>16</sub>H<sub>23</sub>O (M<sup>+</sup>): 231.1749 Found: 231.1738.



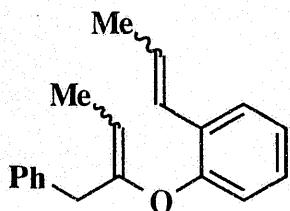
**2-Benzyl-3-oxa-4-phenyl-1,6-heptadiene:** IR (neat) 3013, 2908, 1649, 1490, 1267, 914.2, 803, 744, 691 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.30-7.00 (m, 10H, aromatic H), 5.74-5.60 (m, 1H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.94-4.87 (m, 2H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.79 (dd, 1H, J = 7.4, 5.5, OCHPh), 3.89 (d, 1H, J = 16.9, CCHH), 3.88 (d, 1H, J = 16.9, CCHH), 3.35 (s, 2H, PhCH<sub>2</sub>C), 2.59-2.52 (m, 1H, CHCHHCH), 2.36-2.27 (m, 1H, CHCHHCH); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 160.54, 141.85, 138.89, 129.54, 129.43, 128.50, 128.46, 128.41, 126.51, 126.05, 117.26, 85.68, 78.98, 42.85, 42.27; Mass spectrum m/z (relative intensity): 264 (M<sup>+</sup>, 1), 131 (100), 115 (15), 104 (22), 91 (65); HRMS, m/z Calcd for C<sub>19</sub>H<sub>20</sub>O (M<sup>+</sup>): 264.1514 Found: 264.1520.



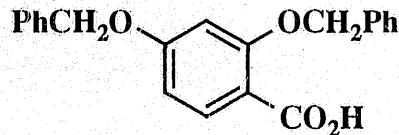
**2-Benzyl-3-oxa-5-phenyl-1,7-octadiene:** IR (neat) 3028, 2919, 1653, 1603, 1495, 1454, 1277, 1053, 915, 802, 746, 698 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) δ 7.22-6.90 (m, 10H, aromatic H), 5.62-5.49 (m, 1H, H<sub>2</sub>CCHCH<sub>2</sub>), 4.92-4.83 (m, 2H, H<sub>2</sub>CCHCH<sub>2</sub>), 3.90 (d, 1H, J = 10.8, CCHH), 3.89 (d, 1H, J = 10.8, CCHH), 3.68-3.58 (m, 2H, OCH<sub>2</sub>CH), 3.29 (s, 2H, PhCH<sub>2</sub>C), 2.89-2.80 (m, 1H, CH<sub>2</sub>CHPh), 2.47-2.37 (m, 1H, CHCHHCH), 2.27-2.17 (m, 1H, CHCHHCH); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) δ 162.56, 142.52, 139.00, 136.50, 129.32, 128.56, 128.44, 128.29, 126.73, 126.46, 116.36, 82.69, 71.07, 45.36, 41.99, 37.14; Mass spectrum m/z (relative intensity): 279 (M<sup>+</sup>, 18), 250 (4), 145 (100), 135 (52), 91 (18); HRMS, m/z Calcd for C<sub>20</sub>H<sub>22</sub>O (M<sup>+</sup>): 278.1671 Found: 278.1668.



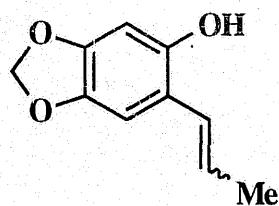
**3-(2-Propenylphenoxy)-2-hexene:** IR (neat) 2960, 2932, 2872, 1687, 1675, 1482, 1452, 1227, 1180, 1111, 990, 969, 750  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ) (4 isomers)  $\delta$  7.50-7.35 (m, 1H, aromatic H), 7.25-6.75 (m, 4H, aromatic H,  $\text{CH}_3\text{CHCH}$ ), 6.26-6.08 (m, 0.96H,  $\text{CH}_3\text{CHCH}$ ), 5.82-5.60 (m, 0.04H,  $\text{CH}_3\text{CHCH}$ ), 4.88 (q, 0.57H,  $J = 6.7$ ,  $\text{CH}_3\text{CHC}$ ), 4.69 (q, 0.43H,  $J = 7.0$ ,  $\text{CH}_3\text{CHC}$ ), 2.45 (t, 0.28H,  $J = 7.4$ ,  $\text{CH}_2\text{CH}_2\text{C}$ ), 2.22 (t, 0.79H,  $J = 7.3$ ,  $\text{CH}_2\text{CH}_2\text{C}$ ), 1.99 (t, 0.93H,  $J = 7.4$ ,  $\text{CH}_2\text{CH}_2\text{C}$ ), 1.75-1.35 (m, 8H,  $\text{CH}_3\text{CH}_2\text{CH}_2$ ,  $\text{CH}_3\text{CHC}$ ,  $\text{CH}_3\text{CHCH}$ ), 0.91 (t, 1.29H,  $J = 7.4$ ,  $\text{CH}_3\text{CH}_2$ ), 0.77 (t, 1.71H,  $J = 7.4$ ,  $\text{CH}_3\text{CH}_2$ ); Mass spectrum m/z (relative intensity): 216 ( $\text{M}^+$ , 3), 187 (14), 160 (100), 131 (62), 119 (33), 105 (10), 91 (23); HRMS, m/z Calcd for  $\text{C}_{15}\text{H}_{20}\text{O}$  ( $\text{M}^+$ ): 216.1514 Found: 216.1514.



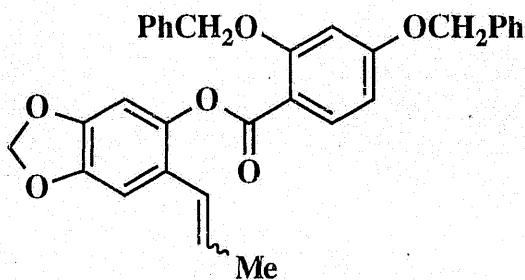
**1-Phenyl-2-(2-propenylphenoxy)-2-butene:** IR (neat) 3028, 2915, 1685, 1666, 1599, 1482, 1452, 1224, 1156, 1084, 1002, 753, 700  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ) (4 isomers)  $\delta$  7.50-6.50 (m, 10H, aromatic H,  $\text{CH}_3\text{CHCH}$ ), 6.12-5.98 (m, 1H,  $\text{CH}_3\text{CHCH}$ ), 4.85 (q, 0.44H,  $J = 6.8$ ,  $\text{CH}_3\text{CHC}$ ), 4.62 (q, 0.56H,  $J = 6.8$ ,  $\text{CH}_3\text{CHC}$ ), 3.76 (s, 0.33H,  $\text{PhCH}_2\text{C}$ ), 3.50 (s, 0.90H,  $\text{PhCH}_2\text{C}$ ), 3.26 (s, 0.77H,  $\text{PhCH}_2\text{C}$ ), 1.63 (dd, 1.68H,  $J = 6.6, 1.7$ ,  $\text{CH}_3\text{CHCH}$ ), 1.58 (dd, 1.32H,  $J = 6.6, 1.7$ ,  $\text{CH}_3\text{CHCH}$ ), 1.49 (d, 1.32H,  $J = 6.8$ ,  $\text{CH}_3\text{CHC}$ ), 1.43 (d, 1.68H,  $J = 6.8$ ,  $\text{CH}_3\text{CHC}$ ); Mass spectrum m/z (relative intensity): 264 ( $\text{M}^+$ , 4), 235 (12), 208 (100), 131 (27), 91 (71); HRMS, m/z Calcd for  $\text{C}_{19}\text{H}_{20}\text{O}$  ( $\text{M}^+$ ): 264.1514 Found: 264.1526.



**2,4-Dibenzylbenzoic acid:**  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  10.80-10.40 (br s, 1H,  $\text{CO}_2\text{H}$ ), 8.15 (d, 1H,  $J = 8.8$ , **6-H**), 7.48-7.42 (m, 10H, aromatic H), 6.73 (dd, 1H,  $J = 8.8, 2.2$ , **5-H**), 6.69 (d, 1H,  $J = 2.2$ , **3-H**), 5.22 (s, 2H,  $\text{PhCH}_2\text{O}$ ), 5.11 (s, 2H,  $\text{PhCH}_2\text{O}$ ). Identical to the literature data.<sup>3a</sup>

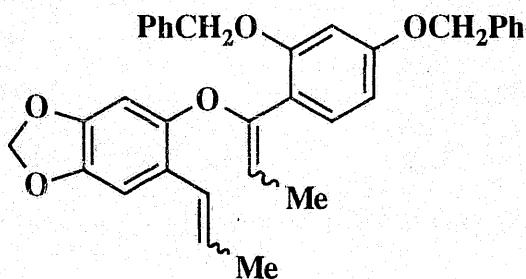


**6-Propenylsesamol:** IR (neat) 3309 (br), 2884, 1625, 1495, 1443, 1165, 1026, 920  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ) (E)  $\delta$  6.86 (s, 1H, **5-H**), 6.02 (s, 1H, **2-H**), 6.60 (dq, 1H,  $J = 15.8, 1.7$ ,  $\text{CHCHCH}_3$ ), 5.79 (qd, 1H,  $J = 6.6, 15.8$ ,  $\text{CHCHCH}_3$ ), 5.27 (s, 2H,  $\text{OCH}_2\text{O}$ ), 4.00 (br s, 1H, OH), 1.61 (dd, 3H,  $J = 6.6, 1.7$ ,  $\text{CHCHCH}_3$ ); (Z) 6.61 (s, 1H, **5-H**), 6.44 (s, 1H, **2-H**), 6.13 (dq, 1H,  $J = 11.3, 1.9$ ,  $\text{CHCHCH}_3$ ), 5.53 (qd, 1H,  $J = 7.1, 11.3$ ,  $\text{CHCHCH}_3$ ), 5.29 (s, 2H,  $\text{OCH}_2\text{O}$ ), 4.62 (br s, 1H, OH), 1.46 (dd, 3H,  $J = 7.1, 1.9$ ,  $\text{CHCHCH}_3$ );  $^{13}\text{C}$  NMR (75 MHz,  $\text{C}_6\text{D}_6$ ) (E/Z mixture)  $\delta$  147.87, 142.28, 129.55, 125.48, 124.81, 124.40, 108.82, 105.88, 100.97, 98.37, 98.02, 18.66, 14.39; Mass spectrum m/z (relative intensity): 178 ( $\text{M}^+$ , 100), 147 (23), 133 (15), 120 (11), 91 (27); HRMS, m/z Calcd for  $\text{C}_{10}\text{H}_{10}\text{O}_3$  ( $\text{M}^+$ ): 178.0630 Found: 178.0632.

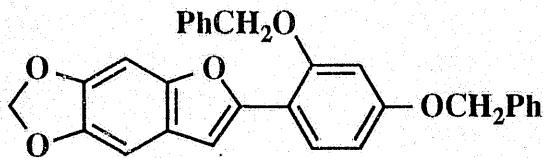


**6-Propenyl-3,4-methylenedioxyphenyl 2',4'-dibenzylbenzoate:** IR (neat) 3025, 2908, 1731, 1708, 1602, 1572, 1496, 1373, 1150, 1020, 932, 873, 732, 685  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ) (E/Z mixture)  $\delta$  8.22-8.19 (m, 1H, **6'-H**), 7.45-6.32 (m, 15H, aromatic H, **2-H**, **5-H**, **3'-H**, **5'-H**,  $\text{CHCHCH}_3$ ), 5.88-5.77 (m, 0.68H,  $\text{CHCHCH}_3$ ), 5.56-5.50 (m, 0.32H,  $\text{CHCHCH}_3$ ), 5.26 (s, 0.64H,  $\text{OCH}_2\text{O}$ ), 5.24 (s, 1.36H,  $\text{OCH}_2\text{O}$ ), 4.73 (s, 1.36H,  $\text{OCH}_2\text{Ph}$ ), 4.72 (s, 0.64H,

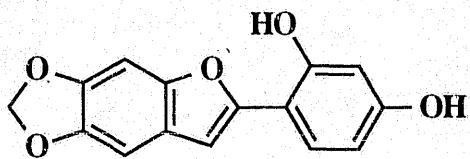
OCH<sub>2</sub>Ph), 4.61 (s, 0.64H, OCH<sub>2</sub>Ph), 4.59 (s, 1.36H, OCH<sub>2</sub>Ph), 1.60 (dd, 0.96H, J = 7.1, 1.6, CH<sub>3</sub>CHCH), 1.49 (dd, 2.04H, J = 6.6, 1.4, CH<sub>3</sub>CHCH); <sup>13</sup>C NMR (75 MHz, C<sub>6</sub>D<sub>6</sub>) (E/Z mixture) δ 164.13, 161.32, 147.43, 147.20, 136.95, 136.70, 134.91, 134.82, 128.76, 128.64, 127.77, 127.39, 127.35, 127.15, 127.05, 127.00, 125.70, 125.10, 109.25, 106.11, 106.03, 105.00, 104.89, 101.79, 101.46, 70.29, 70.16, 18.58, 14.54; Mass spectrum m/z (relative intensity) 494 (M<sup>+</sup>, 8), 318 (24), 317 (100), 227 (4), 181 (8), 147 (12), 91 (67); HRMS, m/z Calcd for C<sub>31</sub>H<sub>26</sub>O<sub>6</sub> (M<sup>+</sup>): 494.1729 Found: 494.1750.



**1-(6-Propenyl-3,4-methylenedioxophenoxy)-1-(2',4'-dibenzoyloxyphenyl)-1-propene:** IR (neat) 3025, 2919, 1666, 1643, 1608, 1449, 1149, 1073, 1020, 938, 832, 732, 691 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, C<sub>6</sub>D<sub>6</sub>) (4 isomers) δ 8.14 (d, 1H, J = 8.6, **6'-H**), 7.48-6.50 (m, 14H, aromatic H, **2-H**, **5-H**, **3'-H**, **5'-H**), 6.36-6.22 (m, 1H, CHCHCH<sub>3</sub>), 6.12-5.88 (m, 1H, CHCHCH<sub>3</sub>), 5.40-5.17 (m, 3H, OCH<sub>2</sub>O, CCHCH<sub>3</sub>), 4.73-4.54 (m, 4H, OCH<sub>2</sub>Ph), 1.79 (d, 1.50H, J = 6.9, CCHCH<sub>3</sub>), 1.72 (dd, 1.50H, J = 6.7, 1.0, CHCHCH<sub>3</sub>), 1.67 (dd, 1.50H, J = 6.5, 0.8, CHCHCH<sub>3</sub>), 1.56 (d, 1.50H, J = 7.0, CCHCH<sub>3</sub>); Mass spectrum m/z (relative intensity) 506 (M<sup>+</sup>, 4), 450 (8), 329 (20), 178 (4), 147 (5), 91 (100); HRMS, m/z Calcd for C<sub>33</sub>H<sub>30</sub>O<sub>5</sub> (M<sup>+</sup>): 506.2093 Found: 506.2081.



**2-(2',4'-Dibenzoyloxyphenyl)-5,6-methylenedioxobenzofuran:** <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.94 (d, 1H, J = 9.3, **6'-H**), 7.60-7.40 (m, 10H, aromatic H), 7.09 (s, 1H, **7-H**), 7.02 (s, 1H, **4-H**), 6.89 (s, 1H, **3-H**), 6.72-6.69 (m, 2H, **3'-H**, **5'-H**), 5.97 (s, 2H, OCH<sub>2</sub>O), 5.20 (s, 2H, OCH<sub>2</sub>Ph), 5.09 (s, 2H, OCH<sub>2</sub>Ph). Identical to the literature data.<sup>3a</sup>; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 159.38, 156.09, 151.95, 148.79, 145.54, 144.15, 136.60, 136.41, 128.65, 128.59, 128.13, 128.07, 127.61, 127.54, 127.31, 123.08, 113.45, 106.07, 105.03, 101.03, 100.72, 99.28, 93.08, 70.46, 70.13.



**2-(2',4'-Dihydroxyphenyl)-5,6-methylenedioxobenzofuran** (*Sophora* compound I): <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>) δ 10.18 (br s, 1H, OH), 9.60 (br s, 1H, OH), 7.55 (d, 1H, J = 8.6, 6'-H), 7.20 (s, 1H, 3-H), 7.07 (s, 1H, 7-H), 7.05 (s, 1H, 4-H), 6.44 (d, 1H, J = 1.9, 3'-H), 6.34 (dd, J = 8.6, 1.9, 5'-H), 6.01 (s, 2H, OCH<sub>2</sub>O). Identical to the literature data.<sup>3a</sup>; <sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>) δ 158.35, 155.47, 152.72, 147.82, 144.96, 143.91, 126.50, 122.85, 108.91, 107.10, 103.31, 102.92, 101.01, 99.19, 93.19. Identical to the literature data.<sup>3b</sup>

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