

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1998 American Chemical Society

Supplementary Material

General Procedure:

2-(4'-Methoxyphenyl)-2,5-dihydrofuran (**5b**):¹

To a stirred solution of *p*-methoxyphenyllead triacetate (**1b**) (200 mg, 0.40 mmol) and NaOMe (88 mg, 1.60 mmol) in CH₃CN/benzene (1 : 1, 6 mL) was added Pd₂(dba)₃·CHCl₃ (20 mg, 5 mol %) followed by 2,3-dihydrofuran (**3**) (34 mg, 0.48 mmol) *via* syringe and the reaction mixture was stirred at room temperature for 3 h. The reaction mixture was extracted with ether (20 mL) and washed with water 3 times (20 mL x 3), and the organic layer was dried over anhydrous MgSO₄ evaporated *in vacuo*. The crude product was separated by SiO₂ column chromatography (EtOAc/hexanes 1 : 10, R_f = 0.32) to afford the coupled product **5b** (57 mg, 80%). TLC, SiO₂, EtOAc/hexanes 1 : 10, R_f = 0.32. ¹H NMR (400 MHz, CDCl₃) δ 3.80 (s, 3H), 4.74 (m, 1H), 4.83 (m, 1H), 5.75 (m, 1H), 5.87 (m, 1H), 6.04 (m, 1H), 6.89 (m, 2H), 7.24 (m, 2H). IR (neat) 3065, 2903, 1602, 1490, 1253, 793 cm⁻¹. MS (EI): m/e (relative intensity) = 176 (47), 175 (42), 147 (17), 145 (54), 135 (100), 115 (22).

Methyl *trans*-cinnamate (**4b**):

TLC, SiO₂, EtOAc/hexanes 1 : 10, R_f = 0.44. ¹H NMR (400 MHz, CDCl₃) δ 3.86 (s, 3H), 6.48 (d, 1H, *J* = 16.3 Hz), 7.41 (m, 3H), 7.55 (m, 2H), 7.73 (d, 1H, *J* = 16.3 Hz). IR (KBr) 3057, 2953, 1715, 1268 cm⁻¹. MS (EI): m/e (relative intensity) = 162 (63), 131 (100), 103 (49), 77 (34), 51 (29). Literature: *Beil.* 9, 581. Available from Aldrich Chemical Co. Inc..

(2*S*, 3*S*, 4*E*)-1-*O*-Benzyloxy-5-phenyl-4-penten-2,3-diol (**4c**):²

TLC, SiO₂, EtOAc/hexanes 1 : 1, R_f = 0.65. ¹H NMR (400 MHz, CDCl₃) δ 2.63 (m, 1H), 2.72 (m, 1H), 3.64 (m, 2H), 3.77 (m, 1H), 4.36 (m, 1H), 4.57 (m, 2H), 6.20 (dd, 2H, *J* = 16, 6.7 Hz), 6.66 (d, 1H, *J* = 16 Hz), 7.34 (m, 10H). IR (neat) 3399, 2919, 1113 cm⁻¹. MS (EI): m/e (relative intensity) = 133 (100), 115 (14), 91 (88), 77 (11).

(2*S*, 3*S*, 4*E*)-1-*O*-Benzyloxy-5-(4'-methoxyphenyl)-4-penten-2,3-diol acetonide (**4d**):

TLC, SiO₂, EtOAc/hexanes 1 : 15, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 1.48 (s, 6H), 3.64 (m, 2H), 3.74 (s, 3H), 3.98 (m, 1H), 4.39 (m, 1H), 4.59 (m, 2H), 6.03 (dd, 1H, *J* = 9.6, 4.2 Hz), 6.57 (d, 1H, *J* = 9.6 Hz), 6.84 (m, 2H), 7.30 (m, 7H). IR (neat) 3055, 2988, 1607, 1512, 1265

cm⁻¹. MS (EI): m/e (relative intensity) = 175 (25), 161 (21), 134 (72), 103 (24), 91 (100). HRMS calcd for C₂₂H₂₆O₄, 354.1763; found, 354.1831.

(2S, 3S, 4E)-1-O-Benzoyloxy-5-(4'-methoxyphenyl)-4-penten-2,3-diol cyclic carbonate (4e):

TLC, SiO₂, EtOAc/hexanes 1 : 2, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 3.66 (m, 1H), 3.80 (m, 1H), 3.82 (s, 3H), 4.50~4.65 (m, 3H), 5.13 (m, 1H), 6.05 (dd, 1H, J = 9.5, 5.1 Hz), 6.66 (d, 1H, J = 12 Hz), 6.88 (d, 1H, J = 12 Hz), 7.34 (m, 7H). IR (neat) 3065, 2986, 1802, 1607, 1264 cm⁻¹. MS (EI): m/e (relative intensity) = 249 (51), 161 (70), 147 (26), 134 (44), 121 (25), 91 (100), 65 (12). HRMS calcd for C₂₀H₂₀O₅, 340.1283; found, 340.1311.

2-Phenyl-2,5-dihydrofuran (5a):³

TLC, SiO₂, EtOAc/hexanes 1 : 30, R_f = 0.30. ¹H NMR (400 MHz, CDCl₃) δ 4.80 (m, 1H), 4.88 (m, 1H), 5.80 (m, 1H), 5.90 (m, 1H), 6.05 (m, 1H), 7.29~7.38 (m, 5H). IR (neat) 3028, 2964, 1612, 1598, 1427, 890 cm⁻¹. MS (EI): m/e (relative intensity) = 146 (50), 117 (30), 115(47), 105 (100), 91 (15), 77 (33).

2-(2'-Thienyl)-2,5-dihydrofuran (5c):⁴

TLC, SiO₂, EtOAc/hexanes 1 : 30, R_f = 0.40. ¹H NMR (400 MHz, CDCl₃) δ 4.72 (m, 1H), 4.85 (m, 1H), 5.94 (m, 1H), 6.04 (m, 1H), 6.09 (m, 1H), 6.98 (m, 2H), 7.27 (m, 1H). IR (neat) 3098, 1594, 1400, 925, 857, 830 cm⁻¹. MS (EI): m/e (relative intensity) = 152 (3), 146 (47), 117 (40), 115 (51), 105 (100), 91 (19), 77 (42).

References for Supplementary Material

1. Ozawa, F.; Kubo, A.; Hayashi, T. *J. Am. Chem. Soc.* **1991**, *113*, 1417-1419.
2. Kang, S-K.; Jung, K-Y.; Park, C-H.; Jang, S-B. *Tetrahedron Lett.* **1995**, *36*, 8047-8050.
3. Larock, R. C.; Gong, W. H. *J. Org. Chem.* **1990**, *55*, 407-408.
4. Kang, S-K.; Yamaguchi, T.; Kim, J-S.; Choi, S-C.; Ahn, C. *Synth. Commun.* **1997**, *27*, 1105-1110.