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A Simple Synthesis of 4*H*-1,3-Benzodioxin-2-one Derivatives by Iodocyclization of *t*-Butyl *o*-Vinylphenyl Carbonate Derivatives

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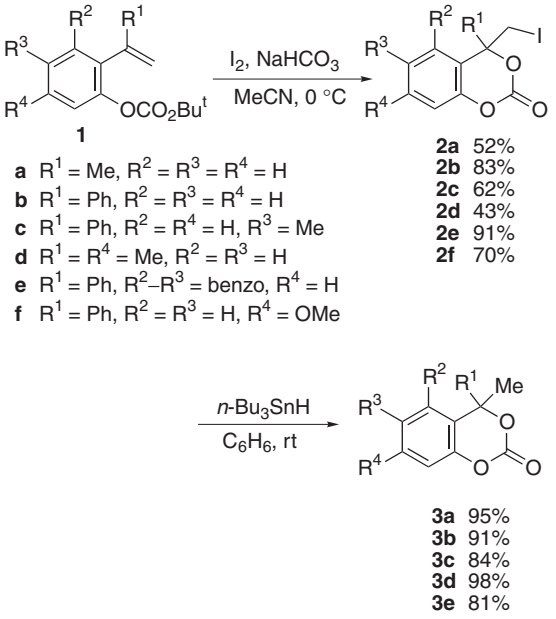
The first general method for the synthesis of 4*H*-1,3-benzodioxin-2-one derivatives has been developed. Treatment of *t*-butyl 2-vinylphenyl carbonates with iodine in the presence of sodium hydrogencarbonate gave 4-iodomethyl-4*H*-1,3-benzodioxin-2-one derivatives in moderate to good yields. These iodides were reduced with tributyltin hydride to give the corresponding 4-methyl-4*H*-1,3-benzodioxin-2-one derivatives.

Surprisingly, there have been few reports on the synthesis of 4*H*-1,3-benzodioxin-2-one derivatives. The reaction of 2-hydroxybenzyl alcohol derivatives with phosgene has been used to prepare this class of molecules.[1] This method, however, suffers from low yields and limited generality; therefore, their utility has not been examined so far. 4*H*-1,3-Benzodioxin-2-one derivatives, however, are of potential interest from a biological point of view, because their 1-nitrogen analogues, 1,4-dihydrobenz[*d*][1,3]oxazin-2-one derivatives, have been reported to exhibit a variety of biological activities.[2] We, therefore, decided to develop a simple and general method for the preparation of 4*H*-1,3-benzodioxin-2-one derivatives. In the present paper, we wish to describe the results of our work, which provide the first general access to this class of molecules. The method is based on the iodocyclization[3] of *t*-butyl *o*-vinylphenyl carbonates **1**, giving 4-iodomethyl-4*H*-1,3-benzodioxin-2-one derivatives **2**.

The method we have developed for the construction of the 4*H*-1,3-benzodioxin-2-one skeleton is outlined in Scheme 1. Thus, *t*-butyl *o*-vinylphenyl carbonates **1**, which were easily prepared by the *t*-butoxycarbonylation of readily available *o*-vinylphenols[4] with di-*t*-butyl dicarbonate using sodium hydride as a base, were treated with iodine in the presence of sodium hydrogencarbonate in acetonitrile at 0 °C to give 4-iodomethyl-4*H*-1,3-benzodioxin-2-one derivatives **2** in moderate to good yields. These products are somewhat unstable, but storable at refrigerator temperature for a few weeks.

It was found that hydrogen displaced the iodine atom of **2a**–**2e** cleanly on treatment with tributyltin hydride in benzene at room temperature and that the corresponding 4-methyl-4*H*-1,3-benzodioxin-2-one derivatives **3a**–**3e** were obtained in good to excellent yields, as shown in Scheme 1. However, when **2f** was used, this compound was unstable under the above reaction conditions and no desired product was detected; the corresponding *o*-vinylphenol derivative arising from decarboxylation was the only product, produced almost quantitatively.



Scheme 1.

The results reported above demonstrate that 4*H*-1,3-benzodioxin-2-one derivatives can be easily prepared from readily available 2-vinylphenol derivatives. Work on further applications utilizing this type of cyclization for the preparation of related heterocycles is now in progress in our laboratory.

Experimental

Starting Materials. 2-(1-Methylethenyl)phenol,[4a] 2-(1-phenylethenyl)phenol,[4b] 4-methyl-2-(1-phenylethenyl)phenol,[4c] 1-(1-phenylethenyl)naphthalen-2-ol,[4c] and 5-methoxy-2-(1-phenylethenyl)phenol[4b] were prepared by the appropriate literature methods. All other chemicals used in this study were commercially available.

***t*-Butyl 2-(1-Methylethenyl)phenyl Carbonate (1a).** To a stirred suspension of NaH (0.19 g, 60% in oil, 4.7 mmol) in THF (5 mL) at 0 °C was added dropwise a solution of 2-(1-methylethenyl)phenol[4a] (0.25 g, 1.9 mmol) in THF (2 mL). After 5 min, di-*t*-butyl dicarbonate (1.0 g, 4.7 mmol) was added and the mixture was stirred for 1 h at the same temperature. The resulting reaction mixture was treated with aqueous saturated NH_4Cl (15 mL) and the organic materials were extracted with Et_2O three times (10 mL each). The combined extracts were washed with brine and dried over anhydrous Na_2SO_4. The solvent and excess di-*t*-butyl dicarbonate were removed under reduced pressure. The residue was purified by column chromatography on silica gel to give **1a** (0.38 g, 86%); a colorless liquid; R_f 0.64 (1:3 AcOEt–hexane); IR (neat) 1759 and 1638 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.52 (9H, s), 2.08 (3H, s), 5.07 (1H, d, $J = 1.0$ Hz), 5.20 (1H, d, $J = 1.0$ Hz), 7.10 (1H, d, $J = 8.2$ Hz), 7.20 (1H, td, $J = 7.3$ and

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1.4 Hz), and 7.24–7.29 (2H, m). Found: C, 71.55; H, 8.00%. Calcd for $C_{14}H_{18}O_3$: C, 71.77; H, 7.74%.

***t*-Butyl 2-(1-Phenylethenyl)phenyl Carbonate (1b):** Prepared from 2-(1-phenylethenyl)phenol[4b] in a manner similar to that described for the preparation of **1a** in 81% yield; a colorless oil; R_f 0.47 (1:10 AcOEt–hexane); IR (neat) 1755 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.34 (9H, s), 5.36 (1H, d, $J = 0.9$ Hz), 5.70 (1H, d, $J = 0.9$ Hz), 7.16 (1H, dd, $J = 8.2$ and 1.3 Hz), 7.23–7.32 (7H, m), and 7.36 (1H, ddd, $J = 8.2$, 7.3, and 1.8 Hz). Found: C, 77.36; H, 6.87%. Calcd for $C_{19}H_{20}O_3$: C, 77.00; H, 6.80%.

***t*-Butyl 4-Methyl-2-(1-phenylethenyl)phenyl Carbonate (1c):** Prepared from 4-methyl-2-(1-phenylethenyl)phenol[4c] as described for preparation of **1a** in 89% yield; a colorless oil; R_f 0.69 (1:3 AcOEt–hexane); IR (neat) 1755 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.33 (9H, s), 2.34 (3H, s), 5.33 (1H, d, $J = 0.9$ Hz), 5.67 (1H, d, $J = 0.9$ Hz), 7.03 (1H, d, $J = 7.8$ Hz), 7.10 (1H, d, $J = 1.8$ Hz), 7.15 (1H, dd, $J = 7.8$ and 1.8 Hz), and 7.23–7.32 (5H, m). Found: C, 77.37; H, 7.36%. Calcd for $C_{20}H_{22}O_3$: C, 77.39; H, 7.14%.

2-(1-Hydroxy-1-methylethyl)-5-methylphenol: Prepared from 1-(2-hydroxy-4-methylphenyl)ethanone and MeMgBr in 92% yield; a white solid; mp 63–64 °C (hexane–Et_2O); IR (KBr disk) 3393 and 1618 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.66 (6H, s), 2.19 (1H, s), 2.28 (3H, s), 6.64 (1H, dd, $J = 7.8$ and 0.9 Hz), 6.71 (1H, s), 6.96 (1H, d, $J = 7.8$ Hz), and 8.79 (1H, s). Found: C, 72.14; H, 8.53%. Calcd for $C_{10}H_{14}O_2$: C, 72.26; H, 8.49%.

5-Methyl-2-(1-methylethenyl)phenol:[4d] Prepared by thermal dehydration of 2-(1-hydroxy-1-methylethyl)-5-methylphenol (160 °C, neat) in 73% yield; a colorless liquid; R_f 0.61 (3:1 hexane–AcOEt); IR (neat) 3512 and 1631 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.10 (3H, t, $J = 0.9$ Hz), 2.30 (3H, s), 5.12 (1H, quint, $J = 0.9$ Hz), 5.37 (1H, quint, $J = 0.9$ Hz), 5.65 (1H, s), 6.71 (1H, dd, $J = 7.8$ and 0.9 Hz), 6.76 (1H, s), and 7.02 (1H, d, $J = 7.8$ Hz).

***t*-Butyl 5-Methyl-2-(1-methylethenyl)phenyl Carbonate (1d):** Prepared from 5-methyl-2-(1-methylethenyl)phenol as described for the preparation of **1a** in 89% yield; R_f 0.71 (3:1 hexane–AcOEt); IR (neat) 1759, 1638, and 1620 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.52 (9H, s), 2.06 (3H, s), 2.34 (3H, s), 5.05 (1H, d, $J = 0.9$ Hz), 5.16 (1H, d, $J = 0.9$ Hz), 6.92 (1H, s), 7.00 (1H, d, $J = 7.8$ Hz), and 7.16 (1H, d, $J = 7.8$ Hz). Found: C, 72.88; H, 8.19%. Calcd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12%.

***t*-Butyl 1-(1-Phenylethenyl)naphthalen-2-yl Carbonate (1e):** Prepared from 1-(1-phenylethenyl)naphthalen-2-ol[4c] as described for the preparation of **1a** in 84% yield; a white solid; mp 69–70 °C (hexane–Et_2O); IR (KBr disk) 1753 and 1618 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.39 (9H, s), 5.36 (1H, d, $J = 1.4$ Hz), 6.15 (1H, d, $J = 1.4$ Hz), 7.20–7.27 (3H, m), 7.30–7.36 (3H, m), 7.41 (1H, ddd, $J = 7.8$, 7.3, and 1.4 Hz), 7.46 (1H, ddd, $J = 7.8$, 7.3, and 1.4 Hz), and 7.85–7.89 (3H, m). Found: C, 79.70; H, 6.50%. Calcd for $C_{23}H_{22}O_3$: C, 79.74; H, 6.40%.

***t*-Butyl 2-(1-Phenylethenyl)-5-methoxyphenyl Carbonate (1f):** Prepared from 5-methoxy-2-(1-phenylethenyl)phenol[4b] as described for the preparation of **1a** in 87% yield; a colorless oil; R_f 0.76 (1:3 AcOEt–hexane); IR (neat) 1759 and 1618 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.35 (9H, s), 3.82 (3H, s), 5.32 (1H, d, $J = 1.4$ Hz), 5.63 (1H, d, $J = 1.4$ Hz), 6.71 (1H, d, $J = 2.3$ Hz), 6.79 (1H, dd, $J = 8.2$ and 2.3 Hz), 7.19 (1H, d, $J = 8.2$ Hz), and 7.24–7.32 (5H, m). Found: C, 73.54; H, 6.84%. Calcd for $C_{20}H_{22}O_4$: C, 73.60; H, 6.79%.

4-Iodomethyl-4-methyl-4*H*-1,3-benzodioxin-2-one (2a). To a stirred solution of **1a** (0.21 g, 0.90 mmol) in MeCN (9 mL) containing $NaHCO_3$ (0.23 g, 2.7 mmol) at 0 °C was added iodine (0.69 g, 2.7 mmol) portionwise. After stirring for 30 min, 10% aqueous $Na_2S_2O_3$ was added until the color of iodine disappeared, and the mixture was extracted with CH_2Cl_2 three times (10 mL each). The combined extracts were washed with aqueous saturated $NaHCO_3$ and brine, dried over anhydrous K_2CO_3, and evaporated. The residue was purified by column chromatography on silica gel (6:1 hexane–AcOEt) to give **2a** (0.14 g, 52%); a pale-yellow solid; mp 96–97 °C (hexane–CH_2Cl_2); IR (KBr disk) 1761 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.95 (3H, s), 3.61 (1H, d, $J = 11.5$ Hz), 3.64 (1H, d, $J = 11.5$ Hz), 7.13 (1H, dd, $J = 7.8$ and 1.4 Hz), 7.17 (1H, dd, $J = 7.8$ and 1.4 Hz), 7.26 (1H, td, $J = 7.8$ and 1.4 Hz), and 7.41 (1H, dt, $J = 7.8$ and 1.4 Hz); MS m/z 304 (M^+, 0.28) and 260 (100). Found: C, 39.48; H, 2.99%. Calcd for $C_{10}H_9IO_3$: C, 39.50; H, 2.98%.

4-Iodomethyl-4-phenyl-4*H*-1,3-benzodioxin-2-one (2b). The preparation of this compound was conducted as described for the preparation of **1a** (reaction time: 3 h). After treatment of the reaction mixture with 10% aqueous $Na_2S_2O_3$, MeCN was evaporated. The resulting precipitate was collected by filtration and recrystallized from hexane–CH_2Cl_2 to give **2b**: a white solid; mp 124–127 °C (hexane–CH_2Cl_2); IR (KBr disk) 1771 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 3.89 (1H, d, $J = 11.9$ Hz), 3.98 (1H, d, $J = 11.9$ Hz), 7.15 (1H, d, $J = 8.2$ Hz), 7.31–7.38 (7H, m), and 7.47 (1H, ddd, $J = 9.2$, 8.2, and 2.8 Hz); MS m/z 366 (M^+, 2.3), 322 (11), and 195 (100). Found: C, 48.90; H, 3.11%. Calcd for $C_{15}H_{11}IO_3$: C, 49.20; H, 3.03%.

4-Iodomethyl-6-methyl-4-phenyl-4*H*-1,3-benzodioxin-2-one (2c): Prepared from **1c** in a manner similar to that described for the preparation of **2b**; a white solid; mp 127–130 °C (hexane–CH_2Cl_2); IR (KBr disk) 1773 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.45 (3H, s), 3.87 (1H, d, $J = 11.9$ Hz), 3.97 (1H, d, $J = 11.9$ Hz), 7.02 (1H, d, $J = 8.2$ Hz), 7.10 (1H, d, $J = 1.4$ Hz), 7.25 (1H, dd, $J = 8.2$ and 1.4 Hz), and 7.32–7.38 (5H, m); MS m/z 336 [$(M - CO_2)^+$, 5.9] and 209 (100). Found: C, 50.39; H, 3.51%. Calcd for $C_{16}H_{13}IO_3$: C, 50.55; H, 3.45%.

4-Iodomethyl-4,7-dimethyl-4*H*-1,3-benzodioxin-2-one (2d): Prepared from **1d** as described for the preparation of **2b** (reaction time: 30 min); a white solid; mp 121–123 °C (hexane–CH_2Cl_2); IR (KBr disk) 1755 and 1634 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 1.92 (3H, s), 2.39 (3H, s), 3.58 (1H, d, $J = 11.0$ Hz), 3.62 (1H, d, $J = 11.0$ Hz), 6.93 (1H, s), and 7.02–7.07 (2H, s); MS m/z 318 (M^+, 0.50), 274 (6.2), and 147 (100). Found: C, 41.34; H, 3.50%. Calcd for $C_{11}H_{11}IO_3$: C, 41.53; H, 3.49%.

1-Iodomethyl-1-phenyl-1*H*-2,4-naphtho[2,1-*d*]dioxin-3-one (2e): Prepared from **1e** as described for the preparation of **2b** (reaction time: 1 day); a white solid; mp 103–105 °C (hexane–CH_2Cl_2); IR (KBr disk) 1776 and 1630 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 4.53 (1H, d, $J = 11.5$ Hz), 4.65 (1H, d, $J = 11.5$ Hz), 7.30–7.40 (6H, m), 7.42–7.48 (3H, m), 7.89 (1H, d, $J = 8.2$ Hz), and 7.96 (1H, d, $J = 8.7$ Hz); MS m/z 372 [$(M - CO_2)^+$, 20] and 245 (100). Found: C, 55.10; H, 3.25%. Calcd for $C_{19}H_{13}IO_3$: C, 54.83; H, 3.15%.

4-Iodomethyl-7-methoxy-4-phenyl-4*H*-1,3-benzodioxin-2-one (2f): Prepared from **1f** as described for the preparation of **2b**; a white solid; mp 85–88 °C; IR (KBr disk) 1784 and 1626 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 3.846 (3H, s), 3.848 (1H, d, $J = 11.9$ Hz), 3.97 (1H, d, $J = 11.9$ Hz), 6.66 (1H, d, $J = 2.3$ Hz), 6.88 (1H, dd, $J = 8.2$ and 2.3 Hz), 7.21 (1H, d, $J = 8.2$ Hz), and 7.28–7.37 (5H, m); MS m/z 396 (M^+, 0.21), 352 (20), and 225 (100). Found: C, 48.47; H, 3.37%. Calcd for $C_{16}H_{13}IO_4$: C, 48.51; H, 3.31%.

4,4-Dimethyl-4*H*-1,3-benzodioxin-2-one (3a). After a mixture of **2a** (0.18 g, 0.59 mmol) and *n*-Bu_3SnH (0.34 g, 1.2 mmol) in benzene (4 mL) was stirred at room temperature for 2 h, the solvent was evaporated and the residue was purified by column chromatography on silica gel to give **3a** (0.10 g, 95%); a colorless oil; R_f 0.40 (1:4 AcOEt–hexane). IR and ^{1}H NMR data of this product were identical to those reported previously.[1c]

4-Methyl-4-phenyl-4*H*-1,3-benzodioxin-2-one (3b). Compound **2b** was treated with *n*-Bu_3SnH in benzene in a manner similar to that described above for the preparation of **3a**. After evaporation of the solvent, the residual solid was recrystallized from hexane–CH_2Cl_2 to give **3b**; a white solid; mp 113–114 °C; IR (KBr disk) 1771 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.08 (3H, s), 7.13 (1H, d, $J = 8.7$ Hz), 7.26–7.37 (7H, m), and 7.42 (1H, ddd, $J = 7.8$, 6.4, and 2.3 Hz); MS m/z 196 [$(M - CO_2)^+$, 100]. Found: C, 74.79; H, 5.12%. Calcd for $C_{15}H_{12}O_3$: C, 74.99; H, 5.03%.

4,6-Dimethyl-4-phenyl-4*H*-1,3-benzodioxin-2-one (3c): Prepared as described for the preparation of **3b**; a white solid; mp 116–118 °C (hexane–CH_2Cl_2); IR (KBr disk) 1771 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.06 (3H, s), 2.40 (3H, s), 7.01 (1H, d, $J = 8.2$ Hz), 7.07 (1H, d, $J = 1.4$ Hz), 7.20 (1H, dd, $J =$ 8.2 and 1.4 Hz), and 7.28–7.37 (5H, m); MS m/z 210 [$(M - CO_2)^+$, 58] and 209 (100). Found: C, 75.43; H, 5.60%. Calcd for $C_{16}H_{14}O_3$: C, 75.57; H, 5.55%.

4,4,7-Trimethyl-4*H*-1,3-benzodioxin-2-one (3d): Prepared as described for the preparation of **3b**; a white solid; mp 62 °C (hexane–CH_2Cl_2); IR (KBr disk) 1774 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.73 (6H, s), 2.37 (3H, s), 6.91 (1H, s), 7.01 (1H, d, $J = 7.8$ Hz), and 7.06 (1H, d, $J = 7.8$ Hz); MS m/z 192 (M^+, 0.42) and 148 (100). Found: C, 68.50; H, 6.31%. Calcd for $C_{11}H_{12}O_3$: C, 68.74; H, 6.29%.

1-Methyl-1-phenyl-1*H*-2,4-naphtho[2,1-*d*]dioxin-3-one (3e): Prepared as described for the preparation of **3b** (reaction time: 2 days); a white solid; mp 106–108 °C (hexane–CH_2Cl_2); IR (KBr disk) 1774 and 1630 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.41 (3H, s), 7.24–7.29 (2H, m), 7.31 (1H, d, $J = 9.2$ Hz), 7.36–7.41 (4H, m), 7.45–7.48 (2H, m), 7.84 (1H, d, $J = 8.2$ Hz), and 7.90 (1H, d, $J = 8.7$ Hz); MS m/z 246 [$(M - CO_2)^+$, 100]. Found: C, 78.54; H, 5.00%. Calcd for $C_{19}H_{14}O_3$: C, 78.61; H, 4.86%.

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