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NEW, FACILE SYNTHESIS OF 3,3-DISUBSTITUTED PHTHALIDES BASED ON THE REACTION OF α-SUBSTITUTED 2-LITHIOSTYRENES WITH CARBON DIOXIDE

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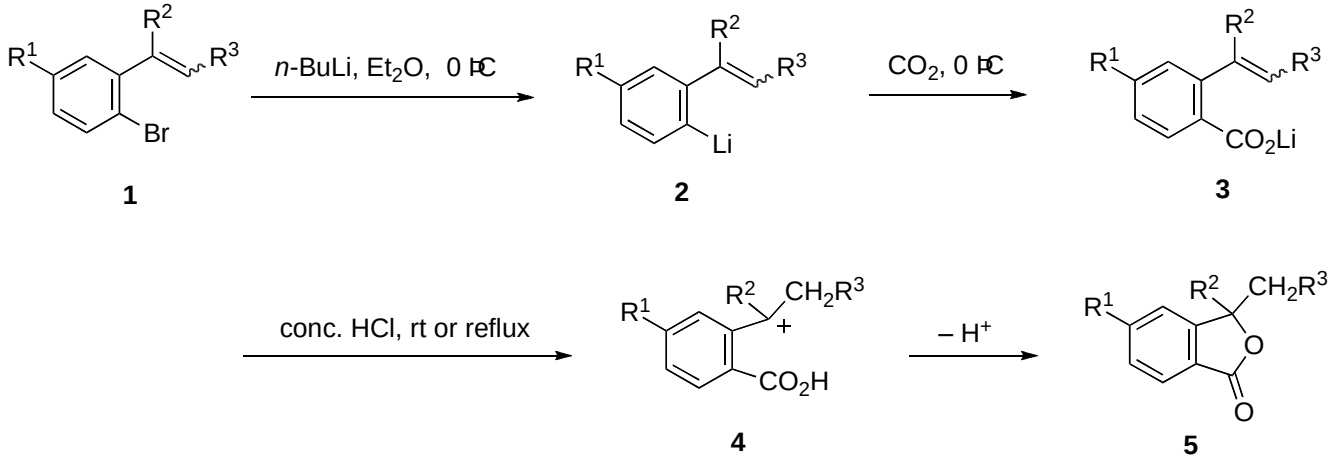
Abstract – A new method to prepare 3,3-disubstituted phthalides [isobenzofuran-1(3*H*)-ones] from α-substituted 2-bromostyrenes has been developed. The reaction of α-substituted 2-lithiostyrenes, generated in situ by bromine-lithium exchange between α-substituted 2-bromostyrenes and butyllithium, with carbon dioxide gave the corresponding lithium 2-vinylbenzoates, which upon treatment with concentrated hydrochloric acid afforded the desired products in one pot.

We have recently reported that a number of heterocyclic compounds, such as isoindolin-1-ones,[1a] 1-iminoisothiochromenes,[1b] phthalanes (1,3-dihydroisobenzofurans),[1c] 1(3*H*)-iminobenzo[*c*]thiophenes,[1d] isothiochromene-1-thiones,[1e] and isothiochroman-1-thiones,[1f] can be obtained by simply utilizing reactions of 2-lithiostyrene derivatives with various electrophiles, such as isocyanates,[1a] isothiocyanates,[1b,d] ketones,[1c] and carbon disulfide.[1e,f] As an extension of these studies, we have examined the reaction of 2-lithiostyrene derivatives with carbon dioxide, and have found that it provides an efficient method for the preparation of phthalides. Compounds having this skeleton are of particular interest since some display biological activity[2] and some have proven to occur in nature.[3] Moreover, they have been used as precursor to more complex compounds;[4] for example a method for the conversion of phthalides into 1,4-dihydro-4*H*-3,1-benzoxazin-3-ones has recently been reported.[4a] Therefore, a number of methods have been developed to prepare phthalides, particularly 3-monosubstituted derivatives including optically active compounds.[5] On the other hand, there have been quite few reports on the general synthesis of 3,3-disubstituted phthalides,[6] though Tanaka et al. have demonstrated an efficient method using rhodium-catalyzed transesterification and [2 + 2 + 2] cyclization.[6b] A 3,3-substituted phthalides has been isolated as a naturally occurring compound.[7] In this manuscript, we wish to describe a new and convenient method to prepare 3,3-disubstituted phthalides.

Our one-pot procedure for the synthesis of 3,3-disubstituted phthalides (**5**) from α-substituted 2-bromostyrenes (**1**) was conducted as illustrated in Scheme 1. Thus, 2-bromostyrenes (**1**) were treated with butyllithium in diethyl ether at 0 ˚C to generate 2-lithiostyrenes (**2**), which were then exposed to carbon dioxide at the same temperature to afford lithium 2-vinylbenzoate intermediates (**3**). Conversion of these intermediates into phthalides (**5**) was successfully achieved through protonation followed by lactonization on treatment with concentrated hydrochloric acid at room temperature or reflux temperature. After usual aqueous workup and subsequent purification of the crude products by preparative TLC on silica gel, the desired products were obtained.



1 **2** **3** **4** **5**

Scheme 1

Table 1. Preparation of 3,3-Disubstituted Phthalides (**5**)

Entry	**1**	R^1	R^2	R^3	Temp	Time/h	**5** (Yield/%)[a]
1	**1a**	H	Me	H	rt	1	**5a** (68)
2	**1b**	H	Ph	H	rt	1	**5b** (62)
3	**1c**	H	4-ClC_6H_4	H	reflux	2	**5c** (36)
4	**1d**	H	4-$MeOC_6H_4$	H	rt	1	**5d** (81)
5	**1e**	H	4-$MeOC_6H_4$	Me	reflux	1	**5e** (75)
6	**1f**	OMe	Me	H	rt	1	**5f** (70)
7	**1g**	OMe	Et	H	rt	1	**5g** (56)
8	**1h**	OMe	Ph	H	rt	1	**5h** (77)
9	**1i**	OMe	4-ClC_6H_4	H	reflux	1	**5i** (37)
10	**1j**	OMe	4-$MeOC_6H_4$	H	rt	1	**5j** (78)

[a] Isolated yields.

The conditions of the reactions and the yields of the products (**5**) are summarized in Table 1. The 2-vinylbenzoic acid derivatives easily cyclized on treatment with concentrated hydrochloric acid at room temperature resulting in the formation of the desired phthalides (**5**) in fair-to-good yields, in general. However, when the substrates carrying 4-chlorophenyl group at the α-position (**1c** and **1i**) or methyl

group at the β-position (**1e**) of the styrene moiety were used, cyclization of the corresponding 2-vinylbenzoic acid derivatives proved to be very sluggish under the above conditions. Cyclization in these cases could be achieved by heating the reaction mixtures at reflux temperature to give the corresponding phthalides (**5c**, **5i**, or **5e**), respectively. Although the yield of **5e** was comparable to those of the others (Entry 5), the yields of **5c** and **5i** were rather lower compared to the other examples, as can be seen from Entries 3 and 9. The lower reactivity of these derivatives may be attributed to the instability of the benzylic cation intermediates (**4**) generated during cyclization, compared to those of the others.

In conclusion, an efficient procedure has been developed for preparing 3,3-disubstitued phthalides, which are hard to prepare by conventional methods. It may be of value, because the starting materials are readily available and manipulations are very simple.

EXPERIMENTAL

All melting points were obtained on a Laboratory Devices MEL-TEMP II melting apparatus and are uncorrected. IR spectra were determined with a Shimadzu FTIR-8300 spectrophotometer. The ^{1}H NMR spectra were determined in $CDCl_3$ using TMS as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 500 MHz. The ^{13}C NMR spectra were determined in $CDCl_3$ using TMS as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 125 MHz. Low-resolution MS spectra (EI, 70 eV) were measured by a JEOL JMS AX505 HA spectrometer. TLC was carried out on a Merck Kieselgel 60 PF_{254}. Column chromatography was performed using Merck Kieselgel 60 (0.063–0.200 mm). All of the organic solvents used in this study were dried over appropriate drying agents and distilled prior to use.

Starting Materials. (2-Bromophenyl)(4-methoxyphenyl)methanone,[8] 1-(2-bromo-5-methoxyphenyl)-1-propanone,[9] 1-bromo-2-(1-methlethenyl)benzene (**1a**),[9] 1-bromo-2-(1-phenylethenyl)benzene (**1b**),[10] 1-bromo-2-[1-(4-chlorophenyl)ethenyl]benzene (**1c**),[1b] 1-bromo-4-methoxy-2-(1-methylethenyl)benzene (**1f**),[11] 1-bromo-4-methoxy-2-(1-phenylethenyl)benzene (**1h**),[1b] 1-bromo-2-[1-(4-chlorophenyl)ethenyl]-4-methoxybenzene (**1i**),[1e] and 1-bromo-4-methoxy-2-[1-(4-methoxyphenyl)ethenyl]benzene (**1j**)[1e] were prepared by the appropriate previously reported procedures. All other chemical used in this study were commercially available.

1-Bromo-2-[1-(4-methoxyphenyl)ethenyl]benzene (1d). This compound was prepared by treating (2-bromophenyl)(4-methoxyphenyl)methanone[8] with methylenetriphenylphosphorane in THF at 0 ˚C in 82% yield; a pale-yellow oil; R_f 0.44 (1:10 THF–hexane); IR (neat) 1605 cm^{-1}; ^{1}H NMR δ 3.80 (3H, s), 5.15 (1H, s), 5.74 (1H, d, J = 0.9 Hz), 6.83 (2H, d, J = 9.2 Hz), 7.18–7.22 (3H, m), 7.30 (1H, dd, J = 7.8, 1.8 Hz), 7.34 (1H, dd, J = 8.2, 7.3 Hz), 7.59 (1H, dd, J = 8.2 Hz). Anal. Calcd for $C_{15}H_{13}BrO$: C, 62.30; H, 4.53; Found: C, 62.37; H, 4.48.

1-Bromo-2-[1-(4-methoxyphenyl)prop-1-enyl]benzene (1e). This compound was prepared by treating

(2-bromophenyl)(4-methoxyphenyl)methanone[8] with ethylenetriphenylphosphorane in THF at 0 ˚C in 61% yield; a mixture of stereoisomers (*E*:*Z* = 3:7); R_f 0.51 (1:10 THF–hexane); IR (neat) 1607 cm^{-1}; ^1H NMR δ 1.59 (2.1H, d, *J* = 6.9 Hz), 1.92 (0.9H, d, *J* = 6.9 Hz), 3.78 (2.1H, s), 3.80 (0.9H, s), 5.77 (0.3H, q, *J* = 6.9 Hz), 6.22 (0.7H, q, *J* = 6.9 Hz), 6.80 (1.4H, d, *J* = 9.1 Hz), 6.85 (0.6H, d, *J* = 8.7 Hz), 7.10–7.14 (2.3H, m), 7.16–7.21 (m, 1H), 7.27 (0.7H, d, *J* = 7.3 Hz), 7.33–7.36 (1H, m), 7.52 (0.3H, d, *J* = 7.8 Hz), 7.64 (0.7H, dd, *J* = 7.8, 0.9 Hz). Anal. Calcd for $C_{16}H_{15}BrO$: C, 63.38; H, 4.99. Found: C, 63.45; H, 5.18.

1-Bromo-2-(1-ethylethenyl)-4-methoxybenzene (1g). This compound was prepared by treating 1-(2-bromo-5-methoxyphenyl)-1-propanone[9] with methylenetriphenylphosphorane in THF at 0 ˚C in 58% yield; a pale-yellow liquid; R_f 0.63 (1:10 THF–hexane); IR (neat) 1639 cm^{-1}; ^1H NMR δ 1.05 (3H, t, *J* = 7.3 Hz), 2.41 (2H, q, *J* = 7.3 Hz), 3.79 (3H, s), 4.94 (1H, s), 5.20 (1H, d, *J* = 0.9 Hz), 6.68 (1H, d, *J* = 3.2 Hz), 6.70 (1H, dd, *J* = 8.7, 3.2 Hz), 7.42 (1H, d, *J* = 8.7 Hz). Anal. Calcd for $C_{11}H_{13}BrO$: C, 54.79; H, 5.43. Found: C, 54.93; H, 5.49.

Typical Procedure for the Preparation of Isobenzofuran-1(3*H*)-ones (5). 3,3-Dimethylisobenzofuran-1(3*H*)-one (5a). To a stirred solution of **1a** (0.20 g, 1.0 mmol) in Et_2O (3 mL) at 0 ˚C was added butyllithium (1.6 M in hexane, 1.0 mmol) dropwise. After 1 h, CO_2 was slowly bubbled via a glass capillary that was drawn from a Pasteur pipette for 30 min. Two mL of concentrated HCl was added, and the resulting mixture was vigorously stirred for an additional 1 h. Five mL each of Et_2O and water were added, and the layers were separated. The aqueous layer was extracted with Et_2O twice (5 mL each), and the combined extracts were washed with brine, dried over anhydrous Na_2SO_4, and evaporated. The residue was purified by column chromatography on silica gel (1:2 Et_2O–hexane) to afford **5a** (0.11 g, 68%); a white solid; mp 63–65 ˚C (hexane–Et_2O). The IR and ^1H NMR data for this product were identical to those reported previously.[12]

3-Methyl-3-phenylisobenzofuran-1(3*H*)-one (5b):[13] mp 76–77 ˚C (hexane–Et_2O) (lit.,[13a] mp 77–78 ˚C); IR (KBr) 1771 m^{-1}; ^1H NMR δ 2.05 (3H, s), 7.31 (1H, tt, *J* = 7.3, 1.4 Hz), 7.36 (2H, dd, *J* = 7.8, 7.3 Hz), 7.45 (2H, d, *J* = 7.8 Hz), 7.47 (1H, d, *J* = 7.3 Hz), 7.52 (1H, ddd, *J* = 7.8, 7.3, 0.9 Hz), 7.66 (1H, td, *J* = 7.3, 0.9 Hz), 7.91 (1H, d, *J* = 7.8 Hz).

3-(4-Chlorophenyl)-3-methylisobenzofuran-1(3*H*)-one (5c): a pale-yellow oil; R_f 0.35 (1:5 THF–hexane); IR (neat) 1767 cm^{-1}; ^1H NMR δ 2.02 (3H, s), 7.32 (2H, d, *J* = 8.7 Hz), 7.38 (2H, d, *J* = 8.7), 7.44 (1H, d, *J* = 7.8 Hz), 7.54 (1H, dd, *J* = 7.8, 7.3 Hz), 7.67 (1H, dd, *J* = 7.8, 7.3 Hz), 7.92 (1H, d, *J* = 7.8 Hz); MS *m/z* 258 (M^+, 15), 243 (100). Anal. Calcd for $C_{15}H_{11}ClO_2$: C, 69.64; H, 4.29. Found: C, 69.61; H, 4.20.

3-(4-Methoxyphenyl)-3-methylisobenzofuran-1(3*H*)-one (5d): a pale-yellow solid; mp 140–141 ˚C (lit.,[14] 140 ˚C); IR (KBr) 1771, 1614 cm^{-1}; ^1H NMR δ 2.03 (3H, s), 3.79 (3H, s), 6.87 (2H, d, *J* = 9.2 Hz), 7.34 (2H, d, *J* = 9.2 Hz), 7.42 (1H, d, *J* = 7.8 Hz), 7.52 (1H, dd, *J* = 7.8, 7.3 Hz), 7.66 (1H, td, *J* = 7.3, 1.4 Hz), 7.91 (1H, d, *J* = 7.3 Hz).

3-Ethyl-3-(4-methoxyphenyl)isobenzofuran-1(3*H*)-one (5e): a pale-yellow oil; R_f 0.23 (1:5 THF–hexane); IR (neat) 1771, 1614 cm^{-1}; ^1H NMR δ 0.80 (3H, t, J = 7.3 Hz), 2.25 (1H, dq, J = 14.6, 7.3 Hz), 2.48 (1H, dq, J = 14.6, 7.3 Hz), 3.79 (3H, s), 6.88 (2H, d, J = 9.2 Hz), 7.40 (2H, d, J = 9.2 Hz), 7.48 (1H, d, J = 7.8 Hz), 7.51 (1H, ddd, J = 7.8, 7.3, 1.4 Hz), 7.66 (1H, ddd, J = 7.8, 7.3, 1.4 Hz), 7.89 (1H, d, J = 7.8 Hz); ^{13}C NMR δ 8.05, 33.07, 55.26, 90.44, 113.96, 122.12, 125.76, 125.81, 126.48, 128.97, 132.27, 134.13, 152.83, 159.33, 170.21; MS *m/z* 268 (M^+, 12), 239 (100). Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found: C, 75.85; H, 6.03.

5-Methoxy-3,3-dimethylisobenzofuran-1(3*H*)-one (5f): a white solid; mp 66–67 ˚C (hexane–Et_2O); IR (KBr) 1747, 1607 cm^{-1}; ^1H NMR δ 1.64 (6H, s), 3.91 (3H, s), 6.80 (1H, d, J = 2.3 Hz), 7.01 (1H, dd, J = 8.3, 2.3 Hz), 7.77 (1H, d, J = 8.3 Hz); MS *m/z* 192 (M^+, 20), 177 (100). Anal. Calcd for $C_{11}H_{12}O_3$: C, 68.74; H, 6.29. Found: C, 68.49; H, 6.30.

3-Ethyl-5-methoxy-3-methylisobenzofuran-1(3*H*)-one (5g): a yellow liquid; R_f 0.31 (1:1 Et_2O–hexane); IR (neat) 1755, 1607 cm^{-1}; ^1H NMR δ 0.77 (3H, t, J = 7.3 Hz), 1.62 (3H, s), 1.88 (1H, dq, J = 14.6, 7.3 Hz), 2.05 (1H, dq, J = 14.6, 7.3 Hz), 3.91 (3H, s), 6.76 (1H, d, J = 2.3 Hz), 7.00 (1H, dd, J = 8.7, 2.3 Hz), 7.78 (1H, d, J = 8.7 Hz); ^{13}C NMR δ 7.76, 25.69, 32.81, 55.79, 87.09, 105.16, 115.76, 118.53, 127.20, 156.32, 164.67, 169.83; MS *m/z* 206 (M^+, 90), 191 (100). Anal. Calcd for $C_{12}H_{14}O_3$: C, 69.88; H, 6.84. Found: C, 69.82; H, 7.02.

5-Methoxy-3-methyl-3-phenylisobenzofuran-1(3*H*)-one (5h): colorless needles; mp 77–80 ˚C (hexane–Et_2O); IR (KBr) 1757, 1607 cm^{-1}; ^1H NMR δ 2.03 (3H, s), 3.87 (3H, s), 6.83 (1H, d, J = 2.3 Hz), 7.01 (1H, dd, J = 8.3, 2.3 Hz), 7.31 (1H, tt, J = 7.3, 1.4 Hz), 7.36 (2H, dd, J = 7.8, 7.3 Hz), 7.44 (2H, d, J = 7.8 Hz), 7.82 (1H, d, J = 8.3 Hz); MS *m/z* 254 (M^+, 89), 239 (100). Anal. Calcd for $C_{16}H_{14}O_3$: C, 75.57; H, 5.55. Found: C, 75.28; H, 5.68.

3-(4-Chlorophenyl)-5-methoxy-3-methylisobenzofuran-1(3*H*)-one (5i): a pale-yellow oil; R_f 0.25 (benzene); IR (neat) 1755, 1607 cm^{-1}; ^1H NMR δ 2.00 (3H, s), 3.88 (3H, s), 6.80 (1H, d, J = 2.3 Hz), 7.03 (1H, dd, J = 8.3, 2.3 Hz), 7.33 (2H, d, J = 8.7 Hz), 7.37 (2H, d, J = 8.7 Hz), 7.82 (1H, d, J = 8.3 Hz); MS *m/z* 288 (M^+, 22), 273 (100). Anal. Calcd for $C_{16}H_{13}ClO_3$: C, 66.56; H, 4.54. Found: C, 66.32; H, 4.81.

5-Methoxy-3-(4-methoxyphenyl)-3-methylisobenzofuran-1(3*H*)-one (5j): a pale-yellow oil; R_f 0.24 (1:1 hexane–Et_2O); IR (neat) 1755, 1607 cm^{-1}; ^1H NMR δ 2.00 (3H, s), 3.79 (3H, s), 3.87 (3H, s), 6.79 (1H, d, J = 2.3 Hz), 6.87 (2H, d, J = 8.7 Hz), 7.01 (1H, dd, J = 8.3, 2.3 Hz), 7.33 (2H, d, J = 8.7 Hz), 7.81 (1H, d, J = 8.3 Hz); ^{13}C NMR δ 26.87, 55.26, 55.81, 86.64, 106.22, 113.91, 116.07, 117.45, 126.66, 127.32, 132.57, 157.15, 159.49, 164.78, 169.68; MS *m/z* 284 (M^+, 27), 269 (100). Anal. Calcd for $C_{17}H_{16}O_4$: C, 71.82; H, 5.67. Found: C, 71.99; H, 5.80.

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