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SYNTHESIS OF THIENOBENZOFURANQUINONE DERIVATIVES BY PHOTOINDUCED AND CAN-MEDIATED 3+2-TYPE CYCLOADDITION REACTIONS

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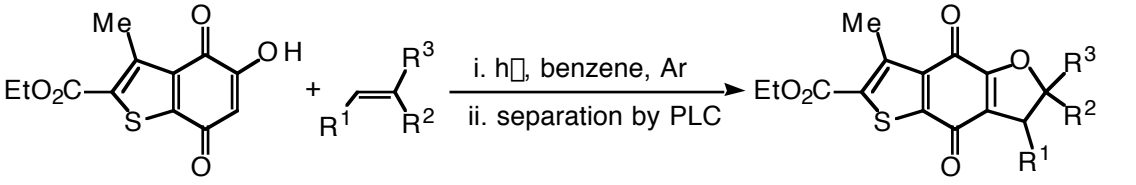
Abstract- Photoinduced cycloaddition between ethyl 5-hydroxy-3-methyl-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate (**1a**) and alkenes is reported to give ethyl 7-methyl-4,8-dioxo-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylates (**2**). Cerium(IV) ammonium nitrate (CAN)-mediated cycloaddition of ethyl 5-hydroxy-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylates (**1**) to alkenes or phenylacetylene, giving ethyl 4,8-dioxo-4,8-dihydrothieno[2,3-*f*]benzofuran-6-carboxylate (**2** or **4**) and ethyl 4,5-dioxo-4,5-dihydrothieno[3,2-*g*]benzofuran-7-carboxylate derivatives (**3** or **5**), is also reported.

The photoinduced 3+2-type cycloaddition between 2-hydroxy-1,4-naphthoquinones and alkenes has been reported by one of the present authors and shown to give 2,3-dihydronaphtho[2,3-*b*]furan-4,9-diones in generally good yields.[1] We have also reported that when 2-hydroxy-1,4-naphthoquinones were treated with alkenes or phenylacetylene in the presence of CAN,[2] naphtho[2,3-*b*]furan-4,9-dione and naphtho[2,3-*b*]furan-4,5-dione derivatives could be obtained *via* the 3+2-type cycloaddition forming a furan ring.[3] As part of our continuing study on the synthesis of heterocycle-fused quinone derivatives,[4] we have investigated the possibility of the preparation of thienobenzofuranquinone derivatives utilizing these annulation reactions. In this paper, we wish to describe the results of our investigation, which offer a simple method for preparing thieno[2,3-*f*]benzofuran-4,8-dione (**2** and **4**) and thieno[3,2-*g*]benzofuran-4,5-dione derivatives (**3** and **5**). Thieno[2,3-*f*]benzofuran-4,8-dione has been prepared by tandem directed metalation,[5] and reported to exhibit antitumor activity.[6] To the best of our knowledge, however, no other derivatives having thieno[2,3-*f*]benzofuran-4,8-dione skeleton have been prepared so far, and this is the first construction of the thieno[3,2-*g*]benzofuran-4,5-dione skeleton.

First, the photoinduced reaction of ethyl 5-hydroxy-3-methyl-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate (**1a**) with alkenes was studied. It was found that irradiation of **1a** in benzene in the presence

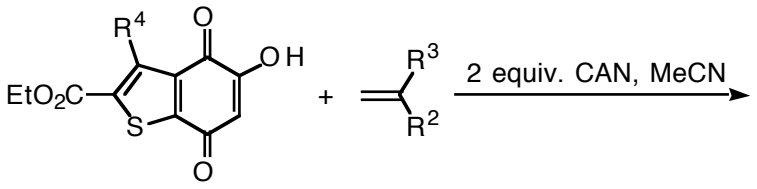
of excess alkenes through a Pyrex filter with a 500W mercury arc led to exclusive formation of 2,3-dihydrothieno[2,3-*f*]benzofuran-4,8-dione derivatives (**2**) in fair yields, as illustrated in Scheme 1. The corresponding *o*-quinone-type products were not obtained in all the present photoreactions, in analogy with the reaction of 2-hydroxy-1,4-naphthoquinone with alkenes.[1]



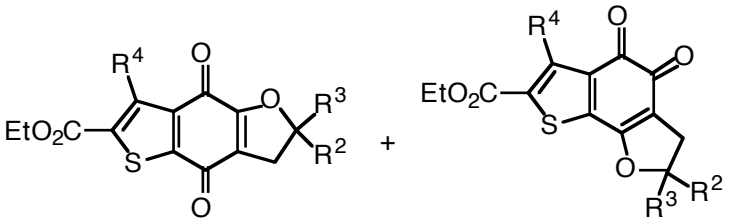
2a R^1 = H, R^2 = R^3 = Et 56%
2b R^1 = H, R^2 = Me, R^3 = Ph 55%
2c R^1 = R^2 = R^3 = Me 55%

Scheme 1.

The CAN-mediated cycloaddition of ethyl 5-hydroxy-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylates (**1a** and **1b**) with alkenes was then carried out. Treatment of **1** with excess alkenes in acetonitrile in the presence of 2 molar amounts of CAN at 0 ˚C afforded the expected products, 2,3-dihydrothieno[2,3-*f*]benzofuran-4,8-dione (**2**) and 2,3-dihydrothieno[3,2-*g*]benzofuran-4,5-dione derivatives (**3**), in reasonable combined yields, as illustrated in Scheme 2. Separation of these regioisomers was readily achieved by preparative TLC on silica gel. While each of the *p*-quinone-type products (**2**) was more mobile on silica gel chromatography and was isolated as a yellow solid, each of the *o*-quinone-type products (**3**) was less mobile and a red solid, as shown by us previously.[3]



1a R^4 = Me
1b R^4 = Ph



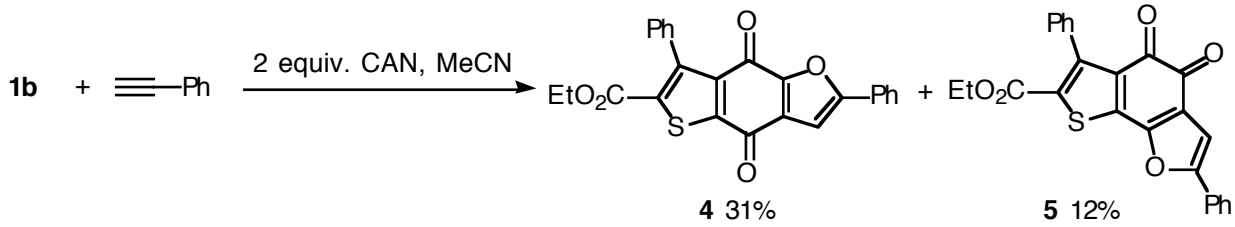
2a R^2 = R^3 = Et, R^4 = Me 44%
2b R^2 = R^4 = Me, R^3 = Ph 35%
2d R^2 = Me, R^3 = R^4 = Ph 44%

3a R^2 = R^3 = Et, R^4 = Me 12%
3b R^2 = R^4 = Me, R^3 = Ph 35%
3d R^2 = Me, R^3 = R^4 = Ph 39%

Scheme 2.

Finally, phenylacetylene proved to be usable in this CAN-mediated cycloaddition. Thus, reaction of **1b**

with an excess phenylacetylene under similar conditions applied to the reactions using alkenes afforded the corresponding thieno[2,3-*f*]benzofuran-4,8-dione (**4**) and thieno[3,2-*g*]benzofuran-4,5-dione derivatives (**5**) in the yields indicated in Scheme 3.



Scheme 3.

In summary, we have demonstrated that the present photoinduced and CAN-mediated 3+2-type cycloaddition reactions provide methodologies for the convenient preparation of thienobenzofuranquinone derivatives, which are hard to be obtained by previous synthetic methods. The ease of operation makes the present procedures attractive.

EXPERIMENTAL

All melting points were obtained on a Laboratory Devices MEL-TEMP II melting apparatus and are uncorrected. IR spectra were determined with a Perkin-Elmer 1600 Series FT IR spectrophotometer as KBr disk. The ^{1}H NMR spectra were determined in $CDCl_3$ using TMS as an internal reference with a JEOL JNM-GX270 FT NMR spectrometer operating at 270 MHz. *J* values are given in Hz. The ^{13}C NMR spectra were determined using TMS as an internal reference with a JEOL ECP500 FT NMR spectrometer operating at 125 MHz in $CDCl_3$. Low-resolution MS spectral measurements were performed on a JEOL AUTOMASS 20 spectrometer (Center for Joint Research and Development, this University). TLC was carried out on a Merck Kieselgel 60 PF_{254}. All of the organic solvents used in this study were dried over appropriate drying agents and distilled prior to use.

Starting Materials. Ethyl 3-methyl(or phenyl)-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate were prepared by the method reported previously by us.[7] All of the other chemicals used in this study were commercially available.

Ethyl 5-Hydroxy-3-methyl-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylates (1a). This compound was prepared according to the procedure for the conversion of methyl 4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate into methyl 5-hydroxy-4,7-dioxo-4,7-dihydrobenzo-[*b*]thiophene-2-carboxylate reported by Ruiz *et al.*[8] Thus, ethyl 3-methyl-4,7-dioxo-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate[7] was treated with acetic anhydride in the presence of boron trifluoride diethyl etherate to give ethyl 4,5,7-triacetoxy-3-methylbenzo[*b*]thiophene-2-carboxylate (73%): a pale-yellow solid; mp 153–156 °C (EtOH); ν_{max}/cm^{-1} 1777, 1715; δ_H 1.40 (3H, t, *J* 7.3), 2.30 (3H,

s), 2.38 (6H, s), 2.83 (3H, s), 4.37 (2H, q, *J* 7.3), 7.29 (1H, s). Anal. Calcd for $C_{18}H_{18}O_8S$: C, 54.82; H, 4.60; S, 8.13. Found: C, 54.80; H, 4.49; S, 8.23.

Hydrolysis of this triacetate with hydrochloric acid in EtOH, followed by oxidation with $FeCl_3$ gave **1a** (58%): a yellow solid; mp 160–162 °C (decomp) (Et_2O–hexane); ν_{max}/cm^{-1} 3231, 1724, 1674, 1639; δ_H 1.40 (3H, t, *J* 7.3), 2.86 (3H, s), 4.37 (2H, q, *J* 7.3), 6.23 (1H, s), 7.41 (1H, br s). Anal. Calcd for $C_{12}H_{10}O_5S$: C, 54.13; H, 3.79; S, 12.04. Found: C, 54.15; H, 3.76; S, 12.03.

Ethyl 5-Hydroxy-4,7-dioxo-3-phenyl-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate (1b). This compound was prepared from ethyl 4,7-dioxo-3-phenyl-4,7-dihydrobenzo[*b*]thiophene-2-carboxylate[7] in a manner similar to that described for the preparation of **1a**. Ethyl 4,5,7-triacetoxy-3-phenylbenzo[*b*]thiophene-2-carboxylate (94%): a pale-yellow solid; mp 174–178 °C (EtOH); ν_{max}/cm^{-1} 1774, 1721; δ_H 1.11 (3H, t, *J* 7.3), 1.44 (3H, s), 2.23 (3H, s), 2.43 (3H, s), 4.14 (2H, q, *J* 7.3), 7.25–7.35 (3H, m), 7.4–7.45 (3H, m). Anal. Calcd for $C_{23}H_{20}O_8S$: C, 60.52; H, 4.42; S, 7.02. Found: C, 60.29; H, 4.63; S, 6.99. **1b** (59%): a yellow solid; 170–175 °C (hexane–Et_2O); ν_{max}/cm^{-1} 3226, 1703, 1680, 1636, 1622; δ_H 1.16 (3H, t, *J* 7.3), 4.19 (2H, q, *J* 7.3), 6.26 (1H, s), 7.22 (1H, s), 7.25–7.3 (2H, m), 7.4–7.55 (3H, m). Anal. Calcd for $C_{17}H_{12}O_5S$: C, 62.19; H, 3.68; S, 9.77. Found: C, 62.17; H, 3.61; S, 10.00.

Ethyl 2,2-Diethyl-7-methyl-4,8-dioxo-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2a). A solution of hydroxybenzothiophenequinone (**1a**) (0.37 mmol, 0.10 g) and 2-ethyl-1-butene (3.7 mmol, 0.31 g) in benzene (19 mL) under argon was irradiated through a Pyrex filter with 500W mercury arc for 38 h. After evaporation of the solvent and excess alkene, the residue was purified by preparative TLC on silica gel (hexane–EtOAc 3:1) to give **2a** (72 mg, 56%): a yellow solid; mp 77–80 °C (Et_2O–hexane); ν_{max}/cm^{-1} 1721, 1677, 1640, 1615; δ_H 0.97 (6H, t, *J* 7.3), 1.39 (3H, t, *J* 7.3), 1.7–1.9 (4H, m), 2.85 (3H, s), 2.91 (2H, s), 4.37 (2H, q, *J* 7.3); MS *m*/*z* 348 (M^+, 100). Anal. Calcd for $C_{18}H_{20}O_5S$: C, 62.05; H, 5.79; S, 9.20. Found: C, 62.04; H, 6.00; S, 9.21.

Ethyl 2,7-Dimethyl-4,8-dioxo-2-phenyl-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2b). Similar photolysis of quinone (**1a**) (0.37 mmol, 0.10 g) and α-methylstyrene (1.5 mmol, 0.18 g) in benzene (20 mL) as described above for 32 h gave **2b** (78 mg, 55%): a yellow solid; mp 133–135°C (Et_2O–hexane); ν_{max}/cm^{-1} 1721, 1678, 1643, 1618; δ_H 1.39 (3H, t, *J* 7.3), 1.86 (3H, s), 2.87 (3H, s), 3.33 (1H, d, *J* 16.8), 3.39 (1H, d, *J* 16.8), 4.38 (2H, q, *J* 7.3), 7.25–7.5 (5H, m); MS *m*/*z* 382 (M^+, 100). Anal. Calcd for $C_{21}H_{18}O_5S$: C, 65.95; H, 4.74; S, 8.38. Found: C, 65.85; H, 4.93; S, 8.45.

Ethyl 2,2,3,7-Tetramethyl-4,8-dioxo-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2c). Similar photolysis of quinone (**1a**) (0.37 mmol, 0.10 g) and 2-methyl-2-butene (7.4 mmol, 0.52 g) in benzene (20 mL) as described above for 32 h gave **2c** (70 mg, 55%): a yellow solid; mp 138–140 °C (Et_2O–hexane); ν_{max}/cm^{-1} 1721, 1676, 1637, 1606; δ_H 1.28 (3H, d, *J* 7.3), 1.40 (3H, t, *J* 7.3), 1.47 (3H, s), 1.50 (3H, s), 2.84 (3H, s), 3.21 (1H, q, *J* 7.3), 4.38 (2H, q, *J* 7.3); MS *m*/*z* 334 (M^+, 100). Anal. Calcd for $C_{17}H_{18}O_5S$: C, 61.06; H, 5.43; S, 9.59. Found: C, 61.11; H, 5.46; S, 9.58.

Ethyl 2,2-Diethyl-7-methyl-4,8-dioxo-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2a) and Ethyl 2,2-Diethyl-6-methyl-4,5-dioxo-2,3,4,5-dihydrothieno[3,2-*g*]benzofuran-7-carboxylate (3a). To a stirred solution of hydroxyquinone (**1a**) (0.37 mmol, 0.10 g) and 2-ethyl-1-butene (3.7 mmol, 0.31 g) in MeCN (17 mL) at 0 ˚C was added CAN (0.74 mmol, 0.41 g) portionwise. After stirring for 30 min, aqueous saturated NH_4Cl was added. Most of acetonitrile was evaporated and the mixture was extracted with Et_2O twice (15 mL each). The extracts were washed with aqueous saturated $NaHCO_3$ and then brine, and dried over anhydrous Na_2SO_4. After evaporation of the solvent the residue was purified by preparative TLC on silica gel (AcOEt–hexane 1:3) to give **2a** (57 mg, 44%) and **3a** (16 mg, 12%). **3a:** a red solid; mp 175–178 ˚C (Et_2O–hexane); ν_{max}/cm^{-1} 1717, 1690, 1647; δ_H 0.97 (6H, t, *J* 7.3), 1.39 (3H, t, *J* 7.3), 1.75–1.95 (4H, m), 2.84 (3H, s), 2.86 (2H, s), 4.38 (2H, q, *J* 7.3); MS *m*/*z* 348 (M^+, 100). Anal. Calcd for $C_{18}H_{20}O_5S$: C, 62.05; H, 5.79; S, 9.20. Found: C, 62.24; H, 5.75; S, 9.22.

Ethyl 2,7-Dimethyl-4,8-dioxo-2-phenyl-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2b) and Ethyl 2,6-Dimethyl-4,5-dioxo-2-phenyl-2,3,4,5-tetrahydrothieno[3,2-*g*]benzofuran-7-carboxylate (3b). Similar reaction of hydroxyquinone (**1a**) (0.37 mmol, 0.10 g) with α-methylstyrene (1.1 mmol, 0.13 g) in MeCN (17 mL) in the presence of CAN (0.74 mmol, 0.41 g) for 25 min gave **2b** (50 mg, 35%) and **3b** (50 mg, 35%). **3b:** a red solid; mp 175–178˚C (Et_2O–hexane); ν_{max}/cm^{-1} 1720, 1688, 1651; δ_H 1.41 (3H, t, *J* 7.3), 1.89 (3H, s), 2.86 (3H, s), 3.29 (1H, d, *J* 15.5), 3.35 (1H, d, *J* 15.5), 4.39 (2H, q, *J* 7.3), 7.3–7.45 (5H, m); δ_C 14.22, 14.36, 29.58, 41.06, 61.74, 96.95, 113.61, 124.08, 128.13, 128.76, 130.87, 135.61, 139.79, 143.92, 148.72, 161.65, 162.89, 175.67, 176.52; MS *m*/*z* 382 (M^+, 100). Anal. Calcd for $C_{21}H_{18}O_5S$: C, 65.95; H, 4.74; S, 8.38. Found: C, 65.66; H, 5.04; S, 8.18.

Ethyl 2-Methyl-4,8-dioxo-2,7-diphenyl-2,3,4,8-tetrahydrothieno[2,3-*f*]benzofuran-6-carboxylate (2d) and Ethyl 2-Methyl-4,5-dioxo-2,6-diphenyl-2,3,4,5-tetrahydrothieno[3,2-*g*]benzofuran-7-carboxylate (3d). Similar reaction of hydroxyquinone (**1b**) (0.30 mmol, 0.10 g) with α-methylstyrene (0.91 mmol, 0.11 g) in MeCN (14 mL) in the presence of CAN (0.61 mmol, 0.35 g) for 25 min gave **2d** (59 mg, 44%) and **3d** (52 mg, 39%). **2d**: a yellow solid; mp 74–83 ˚C (Et_2O–hexane); ν_{max}/cm^{-1} 1731, 1682, 1643, 1619; δ_H 1.14 (3H, t, *J* 7.3), 1.82 (3H, s), 3.34 (1H, d, *J* 16.8), 3.41 (1H, d, *J* 16.8), 4.17 (2H, q, *J* = 7.3), 7.2–7.5 (10H, m); δ_C 13.76, 29.88, 41.31, 61.77, 94.62, 121.08, 124.08, 127.69, 127.87, 128.27, 128.61, 128.64, 133.28, 134.75, 135.74, 144.41, 147.16, 148.61, 159.66, 160.91, 173.02, 176.92; MS *m*/*z* 444 (M^+, 100). Anal. Calcd for $C_{26}H_{20}O_5S$: C, 70.25; H, 4.54; S, 7.21. Found: C, 70.44; H, 4.56; S, 6.96. **3d**: a red solid; mp 144–146 ˚C (Et_2O–hexane); ν_{max}/cm^{-1} 1727, 1698, 1658, 1608 ; δ_H 1.14 (3H, t, *J* 7.3), 1.92 (3H, s), 3.28 (1H , *J* 15.8), 3.37 (1H, d, *J* 15.8), 4.19 (2H, q, *J* 7.3), 7.3–7.45 (10H, m); δ_C 13.76, 29.59, 41.09, 61.78, 97.04, 113.89, 124.10, 127.66, 128.16, 128.39, 128.65, 128.78, 132.67, 132.72, 135.35, 139.90, 143.89, 149.23, 160.87, 162.66, 175.02, 175.54; MS *m*/*z* 444 (M^+, 100). Anal. Calcd for $C_{26}H_{20}O_5S$: C, 70.25; H, 4.54; S, 7.21. Found: C, 70.23; H, 4.64; S, 6.97.

Ethyl 4,8-dioxo-2,7-diphenyl-4,8-dihydrothieno[2,3-*f*]benzofuran-6-carboxylate (4) and Ethyl 4,5-

dioxo-2,6-diphenyl-4,5-dihydrothieno[3,2-*g*]benzofuran-7-carboxylate (5). Similar reaction of hydroxyquinone (**1b**) (0.30 mmol, 0.10 g) with ethynylbenzene (0.91 mmol, 93 mg) in MeCN (14 mL) in the presence of CAN (0.61 mmol, 0.35 g) for 25 min gave **4** (40 mg, 31%) and **5** (15 mg, 12%). **4**: an orange solid; mp 208–210 °C (Et_2O–pentane); ν_{max}/cm^{-1} 1731, 1697, 1666; δ_H 1.15 (3H, t, *J* 7.3), 4.19 (2H, q, *J* 7.3), 7.12 (1H, s), 7.25–7.35 (2H, m), 7.4–7.5 (6H, m), 7.75–7.8 (2H, m); δ_C 13.79, 61.90, 102.50, 125.28, 127.72, 128.17, 128.30, 128.64, 129.12, 130.23, 131.06, 133.50, 136.49, 137.46, 147.06, 147.72, 151.76, 159.97, 160.81, 169.01, 175.46; MS *m/z* 428 (M^+, 43), 399 (100). Anal. Calcd for $C_{25}H_{16}O_5S$: C, 70.08; H, 3.76; S, 7.48. Found: C, 70.00; H, 3.76; S, 7.35. **5**: a purple solid; mp 235–237 °C (Et_2O–pentane); ν_{max}/cm^{-1} 1736, 1678; δ_H 1.14 (3H, t, *J* 7.3), 4.19 (2H, q, *J* 7.3), 7.06 (1H, s), 7.2–7.3 (2H, m), 7.4–7.55 (6H, m), 7.7–7.8 (2H, m); δ_C 13.81, 61.74, 103.62, 123.31, 124.60, 127.76, 128.21, 128.41, 128.53, 129,12, 12.66, 130.30, 132.21, 132.96, 140.88, 149.84, 154.61, 157.92, 160.95, 174.31, and 174.44; MS *m/z* 428 (M^+, 100). Anal. Calcd for $C_{25}H_{16}O_5S$: C, 70.08; H, 3.76; S, 7.48. Found: C, 70.01; H, 4.01; S, 7.26.

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