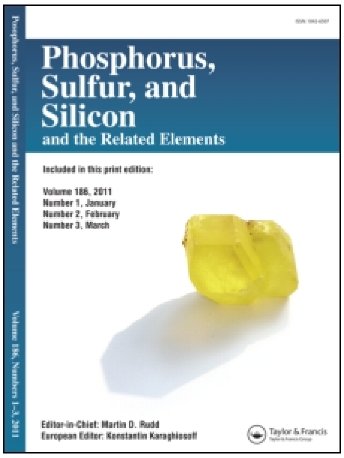

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STEREOSELECTIVE VINYLATION OF METHYL-2-HYDROXY-3-NAPHTHOATE MEDIATED BY VINYL TRIPHENYLPHOSPHONIUM SALTS

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STEREOSELECTIVE VINYLATION OF METHYL-2-HYDROXY-3-NAPHTHOATE MEDIATED BY VINYL TRIPHENYLPHOSPHONIUM SALTS

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*Protonation of the highly reactive intermediate produced in the reaction between triphenylphosphine and dialkyl acetylenedicarboxylate **1a–c** by methyl-2-hydroxy-3-naphthoate lead to vinyl triphenylphosphonium salts which undergo aromatic electrophilic substitution reaction with phenolate-conjugated base to produce **2a–c**.*

Keywords: Methyl-2-hydroxy-3-naphthoate; triphenylphosphine; vinyltriphenyl phosphonium salts

INTRODUCTION

Vinylation of aromatic compounds is an important reaction in organic synthesis, and much attention has been paid to the synthesis of vinyl compounds and their functionalized derivatives. However, there are few reports on direct vinylation of aromatic compounds.[1] In 1908, pure o-vinyl phenol was synthesized for the first time by decarboxylation of o-hydroxycinnamic acid.[2] Since than, a number of methods were devoted to the synthesis of vinylphenols. Electrophilic acylation of phenol followed by reduction and dehydration was employed in the commercial production of *p*-vinylphenol by Maruzen Petrochemicals Co.[3] Another method that utilized benzylic oxidation of ethylphenol was reported.[4] Halophenol derivatives could be vinylated by the Heck reaction.[5] Pyrolysis of hetrocyclic compounds was reported.[6] The palladium-catalyzed arylation of olefins with aryl halides is a well-established method of carbon–carbon bond formation. Because this versatile reaction,

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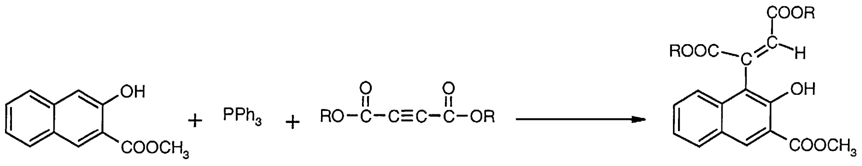
discovered by Heck[7] and Mizoroki,[8] is tolerant of a wide variety of functional groups on either coupling partner, it has gained much interest for the preparation of elaborate arenes from relatively simple starting materials.[9] *Ortho*-vinylation reaction of anilines was reported.[10]

In recent years there has been increasing interest in the synthesis of organophosphorus compounds, particularly those bearing a carbon atom bound directly to a phosphorus atom. This interest has resulted from the recognition of the value of such compounds for a variety of industrial and chemical synthetic uses. Organophosphorus compounds also have been extensively used in organic synthesis as useful reagents as well as ligands of a number of trasition metal catalysts.[11–17] However, there are few reactions in which organophosphorus (III) species work as catalysts.[17–19] A facile one-pot synthesis of vinyl compounds **2a–c** is reported here (Scheme 1).



1a-c **2a-c**

1,2	R
a	$-CH_3$
b	$-CH_2-CH_3$
c	$-CH(CH_3)_2$

SCHEME 1

Compounds **2a–c** apparently result from initial addition of triphenylphosphine to acetylenic ester with concomitant protonation of the reactive 1:1 adduct, followed by electrophilic attack of the vinyl triphenyl phosphonium cation to the aromatic ring at *ortho* position relative to the strong activating group.[20]

Structures **2a–c** were assigned to the isolated products on the bases of their elemental analyses and IR, ^{1}H, and ^{13}C NMR spectra. The mass spectra of the products confirm their molecular weights.

We anticipate that the reaction described herein represents a simple entry into the synthesis of polyfunctional benzene systems. We are currently using this methodology in the preparation of novel functionalized aromatic compounds.

EXPERIMENTAL

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-rapid analyzer. Infrared (IR) spectra were recorded on a Philips PU 9800 Fourier transform infrared (FTIR) spectrometer. ^{1}H and ^{13}C NMR spectra were measured with JEOL EX-90A spectrometer at 90 and 22.6 MHz, respectively. Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 70 eV. Methyl-2-hydroxy-3-naphthoate, triphenylphosphine dialkyl acetylenedicarboxylate were obtained from Fluka (Buchs, Switzerland) and were used without further purification.

GENERAL PROCEDURE

To a magnetically stirred solution of triphenylphosphine (0.261 g, 1 mmol) and methy12-hydroxy-3-naphthoate (0.202 g, 1 mmol) in dichloromethane (5 ml) was added dropwise a mixture of methyl acetylene dicarboxylate (0.14 g, 1 mmol) in dichloromethane (5 ml) at −5°C over 10 min.

The reaction mixture was then allowed to warm up to room temperature, after which it was refluxed for 100 h. Then silica gel (0.5 g) was added and the solvent was evaporated. The residue on silica gel was placed over a column of silica gel (10 g) and elution first made with n-hexane, and the polarity of the eluent was gradually increasing by using 80:20, 60:40, and 50:50 compositions of hexane and diethyl ether. The solvent was removed under reduced pressure and products were obtained.

Dimethyl (Z)-2-[2-Hydroxy-3-(methoxycarbonyl)-1-naphthyl]-2-butendioate (2a)

Yield 85%; m.p., 180°C; ^{1}H NMR (90 MHz, $CDCl_3$): 3.5 (s, 6H, OCH_3), 3.75 (s, 3H, OCH_3), 4 (1H, C=C—H), 7.1–8.5 (5H, H_{ar}), 10.75 (s, 1H, OH) ppm; ^{13}C NMR (22.6 MHz, $CDCl_3$): 51.6 (OCH_3), 52.7 (OCH_3), 52.7 (OCH_3), 113.5 (C), 116.3 (C), 123.1 (C), 123.7 (C), 126.5 (C), 129.5 (C), 129.8 (CH), 131.4 (CH), 132.9 (CH), 135.5 (CH), 139.8 (CH), 152.8 (C—OH), 164.6 (CO_{ester}), 166.5 (CO_{ester}), 170.1 (CO_{ester}) ppm; ms (70 eV) m/z = 343.93.

Diethyl (Z)-2-[2-Hydroxy-3-(methoxycarbonyl)-1-naphthyl]-2-butendioate (2b)

Yield 82%; m.p., 65°C; ^{1}H NMR (90 MHz, δ, $CDCl_3$): 1.01 (t, 3H, CH_3), 1.22 (t, 3H, CH_3), 3.8 (q, 2H, CH_2), 4 (s, 1H, C=C–H), 4.2 (q, 2H, CH_2), 7.4–8.5 (5H, H_{ar}), 10.3 (s, 1H, OH) ppm; ^{13}C NMR (22.6 MHz, δ, $CDCl_3$): 13.5 (CH_3), 14.0 (CH_3), 52.6 (OCH_3), 60.5 (OCH_2), 61.8 (OCH_2), 113.1 (C), 114 (C), 123.4 (C), 127.3 (C), 127.6 (C), 129.5 (C), 131.81 (CH), 132.8 (CH), 135.8 (CH), 142.7 (CH), 143.1 (CH), 155.4 (C–OH), 164.5 (CO_{ester}), 166.09 (CO_{ester}), 167.5 (CO_{ester}) ppm; ms (70 eV) m/z = 372.19.

Diisopropyl (Z)-2-[2-Hydroxy-3-(methoxycarbonyl)-1-naphthyl]-2-butendioate (2c)

Yield 87%; m.p., 85°C; ^{1}H NMR (90 MHz, δ, $CDCl_3$): 1 (d, 3H, CH_3), 1.01 (d, 3H, CH_3), 1.23 (d, 3H, CH_3), 1.3 (d, 3H, CH_3), 4 (1H, C=C–H), 4.6 (m, CH), 5.15 (m, CH), 7–8.5 (5H, H_{ar}), 10.61 (s, 1H, OH) ppm; ^{13}C NMR (22.6 MHz, δ, $CDCl_3$): 21.0 (CH_3), 21.3 (CH_3), 21.5 (CH_3), 21.6 (CH_3), 52.6 (OCH_3), 68.05 (OCH), 69.5 (OCH), 113.4 (C), 114.2 (C), 123.7 (C), 126.6 (C), 127.2 (C), 128.4 (C), 129.7 (CH), 132.05 (CH), 132.8 (CH), 135.9 (CH), 139.5 (CH), 152.9 (C–OH), 164.3 (CO_{ester}), 165.6 (CO_{ester}), 170.2 (CO_{ester}) ppm; ms (70 eV) m/z = 400.22.

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