

New and efficient synthesis of 1,4-oxazines through the reaction of acetylenic esters and nitrosonaphthols in the presence of phosphine derivatives

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Abstract

Protonation of the reactive 1:1 intermediate produced in the reaction between phosphine derivatives and dialkyl acetylenedicarboxylates with 1-nitroso-2-naphthol or 2-nitroso-1-naphthol leads to a vinylphosphonium salt which undergoes intramolecular Wittig reaction to produce 1,4-oxazine derivatives without any catalyst in good yields.

Keywords: Nitrosonaphthols, acetylenic esters, phosphine derivatives, 1,4-oxazines, intramolecular Wittig reaction

Introduction

Synthesis of compounds containing nitrogen and oxygen in a ring is of growing importance by virtue of their presence in numerous biologically important compounds.¹⁻⁴ Therefore, the development of the design and synthesis of new diverse polycyclic heterocycles with potential medicinal and biological activity from readily available starting materials in a cost and time effective manner has received significant attention in research on organic, combinatorial, and medicinal chemistry.⁵⁻¹¹

Oxazines and their derivatives are heterocyclic compounds containing one nitrogen and one oxygen.¹² Oxazine heterocycles have special interest because they constitute an important class of natural and non natural products and show useful biological activities.¹³ The 1,4-oxazine scaffold is a structural subunit of many naturally occurring and synthetic bioactive compounds and have diverse biological activities such as antiulcer,¹⁴ antihypertensive,¹⁵ antifungal,¹⁶ anticancer¹⁷ and anti-thrombotic compound.¹⁸

In recent years, the Wittig reaction¹⁹⁻²¹ has received much attention in organic synthesis because of its high reliability and its importance for the construction of carbon-carbon bonds.²²

Furthermore, the intramolecular Wittig reaction²³⁻²⁵ is one of the most powerful methods for cycloalkene and unsaturated heterocyclic compounds synthesis. While the intramolecular Wittig reaction has attracted significant attention for the syntheses of highly functionalized natural products, the intramolecular Wittig reaction between dialkyl acetylenedicarboxylates and phosphine derivatives in the presence of nitroso compounds has not been reported. In this area, the first time in 1976, McKillop and Sayer established the synthesis of naphthoxazines from dimethyl acetylenedicarboxylate and nitrosonaphthol copper complexes.²⁶

Schonberg and Brosowski²⁷ have extended the Wittig reaction by the use of nitrosobenzene instead of a carbonyl compound (Figure 1) and our attempts to effect a similar reaction between phosphine derivatives **1** and dialkyl acetylenedicarboxylates **2** with nitrosonaphthols **3**, **4** to give 1,4-oxazines **5**, **6**, were successful and proved to be an excellent method for the construction of C=N double bonds (Scheme 1).

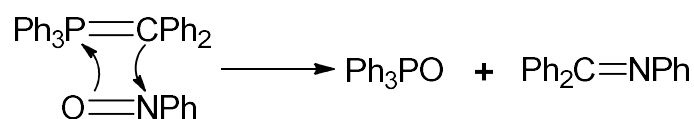
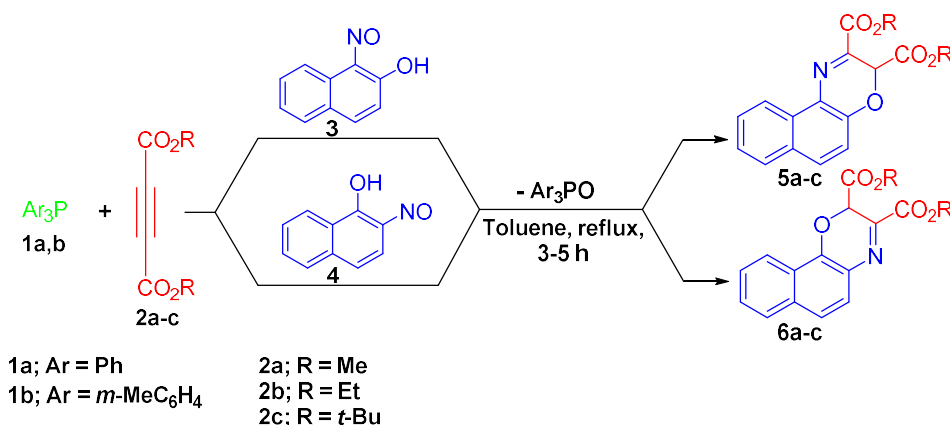


Figure 1. Wittig reaction by the use of nitrosobenzene instead of a carbonyl compound.

Considering the importance of oxazines and in continuation of our research on the development of new synthetic methods in heterocyclic chemistry,²⁸⁻³³ herein we wish to describe the preparation of functionalized 1,4-oxazine derivatives via intramolecular Wittig reaction in good yields (Scheme 1, Table 1).



Scheme 1. Synthesis of functionalized 1,4-oxazine derivatives.

Table 1. Synthesis of functionalized 1,4-oxazine derivatives in the presence of nitrosonaphthols.

Entry ^a	R	Nitrosonaphthol	Product	Yield ^{b,d} (%)	Yield ^{c,d} (%)
1	Me		5a	85	81
2	Et		5b	82	80
3	<i>t</i> -Bu		5c	78	72
4	Me		6a	87	84
5	Et		6b	82	82
6	<i>t</i> -Bu		6c	80	75

^a All reactions were carried out following the general procedure outlined in the experimental section. ^b The reaction was carried out in the presence of triphenylphosphine. ^c The reaction was carried out in the presence of tri-*m*-tolylphosphine. ^d Isolated yields.

Results and Discussion

1,4-Oxazine derivatives can be synthesized through a one-pot condensation reaction between phosphine derivatives with electron deficient acetylenic esters in the presence of nitrosonaphthols in toluene under reflux conditions. The reaction of triphenylphosphine **1a** or tri-*m*-tolylphosphine **1b** and dialkyl acetylenedicarboxylates **2** with 1-nitroso-2-naphthol **3** or 2-nitroso-1-naphthol **4** proceeded spontaneously in toluene, and was complete within a few hours. The results are summarized in Table 1. As it is clear from Table 1, the reactions were efficiently promoted in the presence of triphenylphosphine leading to increased yields compared to tri-*m*-tolylphosphine.

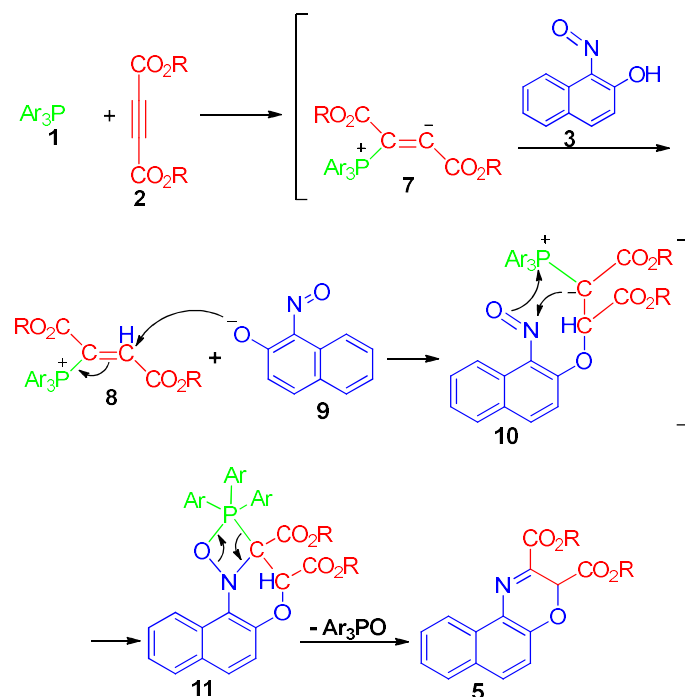
The ¹H and ¹³C NMR spectra of the crude products clearly indicated the formation of dialkyl 3*H*-naphtho[2,1-*b*][1,4]oxazine-2,3-dicarboxylates **5**. No other product could be detected by NMR spectroscopy. The compounds **5a-c** were separated from triarylphosphine oxide, and their structures were deduced from their elemental analyses and infrared (IR) and ¹H NMR and ¹³C

NMR spectroscopic data. The mass spectra of these compounds displayed molecular ion peaks at m/z 299, 327, and 383, respectively, which is consistent with the formation of a 1:1:1 adduct of phosphine derivatives, dialkyl acetylenedicarboxylate, and 1-nitroso-2-naphthol. The ^1H NMR spectrum of **5a** exhibited two single sharp lines at δ 3.96 and 3.99 ppm, characteristic for the two methyl protons of the esters; the CH proton resonates at δ 6.23 ppm. The other aromatic protons resonate as four doublets and two triplets at the range δ 7.20–7.92 ppm. The ^{13}C NMR spectrum of **5a** showed sixteen distinct resonances in agreement with the proposed structure (see Experimental section). The ^1H and ^{13}C NMR spectra of **5b–c** are similar to that of **5a**, except for the signals of the ester group. The structural assignments made on the basis of the NMR spectra of compound **5a** were supported by its IR spectrum. The CH of aliphatic, carbonyl and C=N groups exhibited strong absorption bands at 2920, 1755, 1723 and 1646 cm^{-1} .

Also, the reaction of triphenylphosphine or tri(*m*-tolyl)phosphine **1** and dialkyl acetylenedicarboxylates **2** in the presence of 2-nitroso-1-naphthol **4** led to **6** in good yields (Scheme 1). The results are given in Table 1.

Structures of compounds **6a–c** were assigned by IR, ^1H NMR, ^{13}C NMR and mass spectral data. The ^1H and ^{13}C NMR spectra of **6a–c** are similar to that of **5a–c** and partial assignments of these resonances are given in the Experimental. For example, the NMR spectrum of **6a** exhibited 13 proton resonances and 16 carbon resonances in agreement with the proposed structure.

A plausible mechanism for the formation of compound **5** is illustrated in Scheme 2. On the basis of the well-established chemistry of trivalent phosphorus nucleophiles,³³⁻³⁷ it is reasonable to assume that compound **7** results from the initial addition of triphenylphosphine or tri-*m*-tolylphosphine **1** to the acetylene diester **2** and the subsequent protonation of the 1:1 adduct by the 1-nitroso-2-naphthol **3**. Then, the positively charged ion intermediate **8** is attacked by the conjugate anion of 1-nitroso-2-naphthol **9** to form the ylide **10** and cyclization of this zwitterionic intermediate produces the oxaphosphorane **11**, which undergoes an intramolecular Wittig reaction to produce triphenylphosphine oxide and the product **5** (Scheme 2).



Scheme 2. Suggested mechanism for formation of 1,4-oxazine derivatives.

Conclusions

We have described an effective and novel intramolecular Wittig reaction for the preparation of 1,4-oxazine derivatives from nitrosonaphthols and acetylenic esters in the presence of triphenylphosphine or tri(*m*-tolyl)phosphine. The present procedure offers the advantage of carrying out the reaction under mild and neutral conditions. The starting materials and reagents can be mixed without any activation or modification which provides an efficient and economical method for the synthesis of 1,4-oxazines in good yields. Therefore, this procedure described here provides an acceptable one-pot method for the construction of C=N double bonds.

Experimental Section

General. All melting points were determined on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a Finnigan-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^1H and ^{13}C NMR spectra were recorded on Bruker DRX-400 Avance spectrometer at solution in CDCl_3 .

using TMS as internal standard. The chemicals used in this work purchased from Fluka (Buchs, Switzerland) and were used without further purification.

Synthesis of 1,4-oxazine derivatives (5,6). To a magnetically stirred solution of triphenylphosphine or tri(*m*-tolyl)phosphine (1 mmol) and 1-nitroso-2-naphthol or 2-nitroso-1-naphthol (1 mmol) in toluene (10 mL) was added dropwise a mixture of dialkyl acetylenedicarboxylate (1 mmol) in toluene (2 mL) at ambient temperature over 10 min. The reaction mixture was allowed to stir at room temperature for 1 h and then reflux for 3–5 h. After completion of the reaction (monitored by TLC), the solvent was removed under reduced pressure and the residual solid was purified by column chromatography (Merck silica gel 60, 230–400 Mesh ASTM) using *n*-hexane/EtOAc 3:1 as eluent. The solvent was removed under reduced pressure and the product was obtained.

Dimethyl 3*H*-naphtho[2,1-*b*][1,4]oxazine-2,3-dicarboxylate (5a). Yellow crystals; yield in the presence of Ph₃P: 0.254 g (85%), in the presence of (*m*-MeC₆H₄)₃P: 0.242 g (81%); mp 220–222 °C; IR (KBr) (ν_{\max} cm⁻¹): 2920 (CH of aliphatic), 1755, 1723 (2C=O), 1646 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 3.96, 3.99 (6H, s, 2OCH₃), 6.23 (1H, s, CH), 7.19 (1H, d, *J* 8.8 Hz, Ar-H), 7.47 (1H, t, *J* 8.0 Hz, Ar-H), 7.53 (1H, d, *J* 8.4 Hz, Ar-H), 7.64 (1H, t, *J* 8.0 Hz, Ar-H), 7.81 (1H, d, *J* 8.4 Hz, Ar-H), 7.89 (1H, d, *J* 8.8 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 52.1, 53.4 (2OCH₃), 84.1 (CH), 112.6, 120.0, 122.3, 123.9, 126.2, 127.0, 127.5, 129.6, 130.9, 137.6 (C-Ar), 150.6 (C=N), 165.5, 168.3 (2C=O) ppm; MS (*m/z*, %): 299 (M⁺, 7); Anal. Calcd. for C₁₆H₁₃NO₅: C, 64.21; H, 4.38; N, 4.68 %. Found: C, 64.38; H, 4.46; N, 4.51%.

Diethyl 3*H*-naphtho[2,1-*b*][1,4]oxazine-2,3-dicarboxylate (5b). Yellow crystals; yield in the presence of Ph₃P: 0.268 g (82%), in the presence of (*m*-MeC₆H₄)₃P: 0.262 g (80%); mp 236–238 °C; IR (KBr) (ν_{\max} cm⁻¹): 2917 (CH of aliphatic), 1746, 1720 (2C=O), 1642 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 1.20, 1.25 (6H, t, *J* 7.2 Hz, 2CH₃), 4.18, 4.28 (4H, q, *J* 7.2 Hz, 2OCH₂), 6.30 (1H, s, CH), 7.20 (1H, d, *J* 8.8 Hz, Ar-H), 7.46 (1H, t, *J* 8.0 Hz, Ar-H), 7.57 (1H, d, *J* 8.4 Hz, Ar-H), 7.68 (1H, t, *J* 8.4 Hz, Ar-H), 7.79 (1H, d, *J* 8.0 Hz, Ar-H), 7.92 (1H, d, *J* 8.8 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 14.7, 15.6 (2CH₃), 60.1, 61.5 (2OCH₂), 86.3 (CH), 113.5, 121.1, 123.0, 125.2, 126.7, 127.6, 128.9, 130.6, 131.9, 138.8 (C-Ar), 153.4 (C=N), 166.4, 170.6 (2C=O) ppm; MS (*m/z*, %): 327 (M⁺, 12); Anal. Calcd. for C₁₈H₁₇NO₅: C, 66.05; H, 5.23; N, 4.28%. Found: C, 66.21; H, 5.40; N, 4.12%.

Di-*tert*-butyl 3*H*-naphtho[2,1-*b*][1,4]oxazine-2,3-dicarboxylate (5c). Yellow crystals; yield in the presence of Ph₃P: 0.299 g (78%), in the presence of (*m*-MeC₆H₄)₃P: 0.276 g (72%); mp 241–243 °C; IR (KBr) (ν_{\max} cm⁻¹): 2932 (CH of aliphatic), 1744, 1728 (2C=O), 1648 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 1.31, 1.42 (18H, s, 2C(CH₃)₃), 6.32 (1H, s, CH), 7.21 (1H, d, *J* 8.4 Hz, Ar-H), 7.45 (1H, t, *J* 8.0 Hz, Ar-H), 7.55 (1H, d, *J* 8.4 Hz, Ar-H), 7.67 (1H, t, *J* 8.0 Hz, Ar-H), 7.80 (1H, d, *J* 8.4 Hz, Ar-H), 7.93 (1H, d, *J* 8.4 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 27.6, 28.2 (2OC(CH₃)₃), 80.2, 82.6 (2OC(CH₃)₃), 85.3 (CH), 112.8, 119.7, 120.1, 121.2, 126.6, 126.7, 129.7, 131.2, 133.2, 147.6 (C-Ar), 150.2 (C=N), 166.7, 168.6 (2C=O) ppm; MS (*m/z*, %): 383

(M⁺, 10); Anal. Calcd. for C₂₂H₂₅NO₅: C, 68.91; H, 6.57; N, 3.65 %. Found: C, 69.04; H, 6.46; N, 3.78%.

Dimethyl 2*H*-naphtho[1,2-*b*][1,4]oxazine-2,3-dicarboxylate (6a). Yellow crystals; yield in the presence of Ph₃P: 0.260 g (87%), in the presence of (*m*-MeC₆H₄)₃P: 0.251 g (84%); mp 225-227 °C; IR (KBr) (ν_{\max} cm⁻¹): 2918 (CH of aliphatic), 1745, 1716 (2C=O), 1638 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 3.87, 3.91 (6H, s, 2OCH₃), 6.28 (1H, s, CH), 7.57 (1H, d, *J* 8.4 Hz, Ar-H), 7.66 (1H, d, *J* 8.4 Hz, Ar-H), 7.89 (1H, t, *J* 8.0 Hz, Ar-H), 7.94 (1H, d, *J* 8.4 Hz, Ar-H), 8.03 (1H, t, *J* 8.0 Hz, Ar-H), 8.21 (1H, d, *J* 8.4 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 52.0, 54.1 (2OCH₃), 84.3 (CH), 112.2, 121.5, 122.8, 124.1, 127.5, 127.9, 128.3, 129.8, 131.6, 137.8 (C-Ar), 153.1 (C=N), 167.5, 170.4 (2C=O) ppm; MS (*m/z*, %): 299 (M⁺, 10). Anal. Calcd. for C₁₆H₁₃NO₅: C, 64.21; H, 4.38; N, 4.68 %. Found: C, 64.11; H, 4.50; N, 4.54%.

Diethyl 2*H*-naphtho[1,2-*b*][1,4]oxazine-2,3-dicarboxylate (6b). Yellow crystals; yield in the presence of Ph₃P: 0.268 g (82%), in the presence of (*m*-MeC₆H₄)₃P: 0.268 g (82%); mp 231-233 °C; IR (KBr) (ν_{\max} cm⁻¹): 2920 (CH of aliphatic), 1744, 1713 (2C=O), 1640 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 1.22, 1.26 (6H, t, *J* 7.2 Hz, 2CH₃), 4.14, 4.22 (4H, q, *J* 7.2 Hz, 2OCH₂), 6.25 (1H, s, CH), 7.42 (1H, d, *J* 8.8 Hz, Ar-H), 7.50 (1H, d, *J* 8.4 Hz, Ar-H), 7.81 (1H, t, *J* 8.0 Hz, Ar-H), 7.89 (1H, d, *J* 8.0 Hz, Ar-H), 8.02 (1H, t, *J* 8.4 Hz, Ar-H), 8.22 (1H, d, *J* 8.8 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 14.3, 15.7 (2CH₃), 60.3, 62.7 (2OCH₂), 84.2 (CH), 112.7, 121.8, 123.5, 124.8, 126.3, 127.1, 128.4, 129.9, 132.7, 136.8 (C-Ar), 156.2 (C=N), 169.1, 172.5 (2C=O) ppm; MS (*m/z*, %): 327 (M⁺, 12). Anal. Calcd. for C₁₈H₁₇NO₅: C, 66.05; H, 5.23; N, 4.28 %. Found: C, 65.92; H, 5.11; N, 4.40 %.

Di-tert-butyl 2*H*-naphtho[1,2-*b*][1,4]oxazine-2,3-dicarboxylate (6c). Yellow crystals; yield in the presence of Ph₃P: 0.306 g (80%), in the presence of (*m*-MeC₆H₄)₃P: 0.287 g (75%); mp 238-240 °C; IR (KBr) (ν_{\max} cm⁻¹): 2937 (CH of aliphatic), 1741, 1710 (C=O), 1635 (C=N); ¹H NMR (400 MHz, CDCl₃): δ 1.37, 1.46 (18H, s, 2C(CH₃)₃), 6.39 (1H, s, CH), 7.41 (1H, d, *J* 8.4 Hz, Ar-H), 7.54 (1H, d, *J* 8.0 Hz, Ar-H), 7.83 (1H, t, *J* 8.0 Hz, Ar-H), 7.88 (1H, d, *J* 8.4 Hz, Ar-H), 8.01 (1H, t, *J* 8.0 Hz, Ar-H), 8.13 (1H, d, *J* 8.4 Hz, Ar-H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 27.5, 29.0 (2OC(CH₃)₃), 81.1, 82.8 (2OC(CH₃)₃), 85.8 (CH), 111.9, 117.1, 121.3, 123.2, 126.5, 127.0, 131.1, 132.5, 133.8, 148.6 (C-Ar), 153.2 (C=N), 165.7, 169.3 (2C=O); MS (*m/z*, %): 383 (M⁺, 8); Anal. Calcd. for C₂₂H₂₅NO₅: C, 68.91; H, 6.57; N, 3.65%. Found: C, 68.80; H, 6.69; N, 3.48%.

Acknowledgments

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