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The Reaction of (*N*-Isocyanimino) triphenylphosphorane with (*E*)-3-Aryl-2-propenoic Acid Derivatives: One-Pot Synthesis of 2-[(*E*)-2-Aryl-1-ethenyl]-1,3,4-oxadiazoles via Intramolecular *aza*-Wittig Reaction

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ABSTRACT: *The reaction of (E)-3-aryl-2-propenoic acid derivatives with (N-isocyanimino) triphenylphosphorane proceeds smoothly at room temperature to afford the corresponding 2-[(E)-2-aryl-1-ethenyl]-1,3,4-oxadiazole via an intramolecular aza-Wittig reaction in good yields under neutral conditions. The structures of the products were deduced from their IR, ^{1}H NMR, and ^{13}C NMR spectra and mass spectrometry.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 22:612–616, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20701

INTRODUCTION

Organophosphorus compounds [1–3] have been extensively employed in organic synthesis as useful reagents as well as ligands in a number of transition metal catalysts [1]. Iminophosphoranes are a special type of zwitterions, which bear strongly nucleophilic electron-rich nitrogen. The electron distribution around the P^{+}–N^{-} bond and its consequent chemical implications were probed and assessed through theoretical, spectroscopic, and crystallographic investigations [1–3]. They are excellent ligands and excel in their ligating functions of the unstabilized iminophosphoranes because of their ambidentate and chemically differentiating character. Proton affinity of these iminophosphoranes can be used as a molecular guide to assess their utility as synthetic reagents and their function as ligands in coordination and organometallic chemistry [1–3].

The intramolecular version of the *aza*-Wittig-type reaction has attracted considerable attention recently because of its high potential for the synthesis of a wide variety of nitrogen heterocycles, which can be attributed in good measure to the rapid progress in the preparation of functionalized iminophosphoranes, and several interesting

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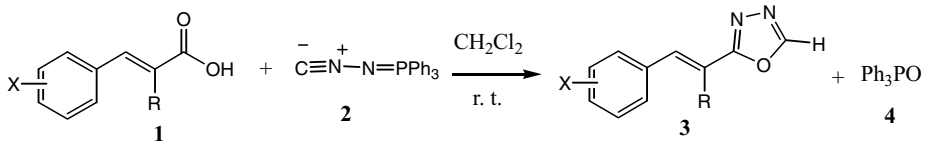


SCHEME 1 Synthesis of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazoles **3** (see Table 1).

heterocyclization reactions involving iminophosphoranes have been reviewed [3]. These compounds can be easily converted through the *aza*-Wittig reaction with isocyanates, carbon dioxide, or carbon disulfide into functionalized heterocumulenes, which exhibit a rich chemistry of unusual synthetic promise [3]. The nucleophilicity at the nitrogen is a factor of essential mechanistic importance in the use of these iminophosphoranes as *aza*-Wittig reagents. Iminophosphoranes are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products and compounds with biological and pharmacological activity [3]. There are several reports for the use of (*N*-isocyanimino)triphenylphosphorane **2** in the preparation of metal complexes [4,5] (Scheme 1). However, the role of (*N*-isocyanimino)triphenylphosphorane **2** in organic chemistry remains almost unexplored [4,5]. The (*N*-isocyanimino)triphenylphosphorane **2** is expected to have unique synthetic potential because it provides a reaction system in which the iminophosphorane group can react with a reagent having a carbonyl functionality [4,5]. In recent years, we have established a one-pot method for the synthesis of organophosphorus compounds [6–12]. As a part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds [13–28], we sought to develop a convenient preparation of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole **3** from (*E*)-3-aryl-2-propenoic acid derivatives **1** and (*N*-isocyanimino)triphenylphosphorane **2** in fairly good yield in neutral conditions. The reaction is completely stereoselective (Scheme 1).

1,3,4-Oxadiazoles have attracted interest in medicinal chemistry as surrogates of carboxylic acids, esters, and carboxamides. They are an important class of heterocyclic compounds that have a wide range of pharmaceutical and biological activities including antimicrobial, antifungal, antiinflammatory, and antihypertensive [29–31]. Several methods have been reported in the literature for the synthesis of 1,3,4-oxadiazoles. These protocols are multistep in nature [32,33]. The most general method involves the cyclization of diacylhydrazides with a variety of reagents, such as thionyl chloride, phosphorous oxychloride, or sulfuric acid, usually under harsh reaction conditions. Few reliable and operationally simple examples have been reported for the one-step synthesis of 1,3,4-oxadiazoles, especially from readily available carboxylic acids and acid hydrazides [34,35].

RESULTS AND DISCUSSION

In the past years, several synthetic methods have been reported for the preparation of (*N*-isocyanimino)triphenylphosphorane ($CNNPPh_3$) **2** (Scheme 1) [4,5]. There are several reports for the use of (*N*-isocyanimino)triphenylphosphorane **2** in the synthesis of metal complexes [4,5]. However, application of **2** in the synthesis of organic compounds has not been reported. As a part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds [13–28], we sought to develop a convenient preparation of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole **3** from (*E*)-3-aryl-2-propenoic acid derivatives **1** and (*N*-isocyanimino)triphenylphosphorane **2** in fairly good yields in neutral conditions. The reaction is completely stereoselective (Scheme 1).

The (*E*)-3-aryl-2-propenoic acid derivatives **1** and (*N*-isocyanimino)triphenylphosphorane **2** in dry CH_2Cl_2 react together in a 1:1 ratio at room temperature to produce 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole **3** and triphenylphosphine oxide **4** (Scheme 1 and Table 1). The reaction proceeds smoothly and cleanly under mild conditions.

TABLE 1 Synthesis of 2-[(*E*)-2-Aryl-1-ethenyl]-1,3,4-oxadiazole Derivatives **3** (See Scheme 1)

Compound	*X*	*R*	*Yield (%)*[a]
3a	H	H	77
3b	4-CH_3	H	81
3c	4-Cl	H	80
3d	3-Cl	H	77
3e	3-MeO	H	76
3f	H	Me	83

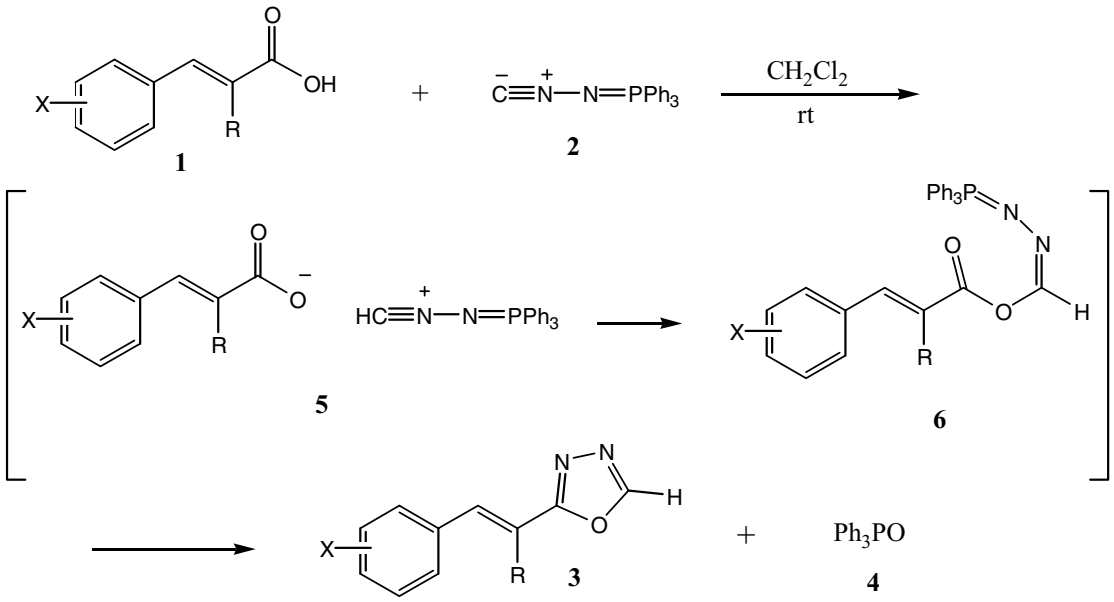
[a]Isolated yields

The structures of the products were deduced from their IR, ^{1}H NMR and ^{13}C NMR spectra, and mass spectrometry (see the Experimental section). The mass spectra of compound **3** displayed molecular ion peak at the *m/z* 172. The ^{1}H NMR spectrum of **3** compound exhibited five signals readily recognized as arising from two vinylic groups (δ = 7.09, d, 1 H, $^3J_{HH}$ = 16.5 Hz and δ = 7.62, d, 1 H, $^3J_{HH}$ = 16.5 Hz), aromatic moiety (δ = 7.41–7.45 ppm, m and δ = 7.58, d, 2 H, $^3J_{HH}$ = 7.0 Hz, arom) and a CH of oxadiazole ring (δ = 8.38, s, 1 H). Coupling constant ($^3J_{HH}$) between the two vinylic groups is ca. 16.5 Hz that is completely in agreement with trans geometry around the carbon–carbon double bond (C=C) of the molecule **3** [3]. The ^{1}H decoupled ^{13}C NMR spectrum of **3** showed eight distinct resonances (δ = 109.53 (CH, vinylic); δ = 127.57, 129.03, and 130.14 (CH, arom); δ = 134.53 (C, arom); δ = 139.85 (CH, vinylic); δ = 152.00 (CH, oxadiazole) and δ = 164.28 (C, oxadiazole)) in agreement with the **3** formula. A partial assignment of these resonances is given in the spectral analysis section (see the Experimental section). The ^{1}H and ^{13}C NMR spectra of compounds **3b–3f** were similar to those of **3a**, except for the aromatic or heteroaromatic moieties, which exhibited characteristic signals with appropriate chemical shifts. We have also used ((*E*)-3-(3-hydroxyphenyl)-2-propenoic acid, (*E*)-3-(3,4-dihydroxyphenyl)-2-propenoic acid, (*E*)-3-(4-hydroxyl-3-methoxyphenyl)-2-propenoic acid, acetic acid, phthalic acid, maleic acid, chloroacetic acid, trichloroacetic acid, benzylic acid, 3,5-dinitrosalicylic acid, (2-hydroxyphenyl)acetic acid, 4-hydroxybenzoic acid, 3-hydroxybenzoic acid, salicylic acid, 4-aminobenzenesulfonic acid, 5-sulfosalicylic acid, nicotinic acid, 3-methoxyphenylacetic acid, 4-methoxyphenylacetic acid, diphenylacetic acid, 4-fluorobenzeneacetic acid, 3-phenylpropanoic acid, 4-chlorobenzeneacetic acid, benzenesulfonic acid, acetylenedicarboxylic acid, and methyl red) in this reaction, but no products were observed after 24 h. TLC indicated that the solution contained starting materials and some minor colored products.

The mechanism of the reaction between the (*E*)-3-aryl-2-propenoic acid derivatives **1** and (*N*-isocyanimino)triphenylphosphorane **2** has not been established experimentally. However, a possible explanation is proposed in Scheme 2. On the basis of the well-established chemistry of isocyanides [36], it is reasonable to assume that protonation of **2** by (*E*)-3- aryl-2-propenoic acid **1** followed by quenching of the cationic center by the conjugate base of carboxylic acid (**5**) can generate the iminophosphorane **6** [3]. The intramolecular *aza*-Wittig [3] reaction of the iminophosphorane **6** would lead to formation of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole**3** and triphenylphosphine oxide **4** (Scheme 2).

CONCLUSION

In summary, we have found a new method for the stereoselective preparation of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole **3** from



SCHEME 2 Proposed mechanism for the formation of 2-[(*E*)-2-aryl-1-ethenyl]-1,3,4-oxadiazole derivatives **3**.

(*E*)-3-aryl-2-propenoic acid derivatives **1** and (*N*-isocyanimino)triphenylphosphorane **2** in fairly moderate yield in neutral conditions. The reaction is completely stereoselective (Scheme 1). We believe that the reported method offers a mild and simple route for the preparation of 2-substituted 1,3,4-oxadiazole derivatives. Its ease of work-up and fairly mild reaction conditions makes it a useful addition to modern synthetic methodologies. Other aspects of this process are under investigation.

EXPERIMENTAL

Starting materials and solvents were obtained from Merck (Darmstadt, Buchs, Germany) and Fluka (Switzerland) and were used without further purification. The methods used to follow the reactions were TLC and NMR. TLC and NMR indicated that there is no side product. Melting points were measured on an Electrothermal 9100 apparatus (LABEQUIP LTD., Markham, Ontario, Canada) and are uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer (JASCO Ltd., Easton, MD). ^{1}H and ^{13}C NMR spectra were measured ($CDCl_3$ solution) with a Bruker DRX-250 Avance spectrometer (Bruker Ltd., Ettlingen, Germany) at 250.0 and 62.5 MHz, respectively. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer (Heraeus Ltd., Vienna, Austria). Mass spectra were recorded on a Finnigan-Matt 8430 mass spectrometer operating at an ionization potential of 20 eV (Thermo Scientific Ltd., Waltham, MA). Flash chromatography columns were prepared from Merck silica gel powder.

General Procedure for the Preparation of Compounds **3** *and* **6**

To a magnetically stirred solution of (*N*-isocyanimino)triphenylphosphorane [7,8] **2** (0.302 g, 1 mmol) in dry CH_2Cl_2 (4 mL), a solution of (*E*)-3-aryl-2-propenoic acid **1** (1 mmol) was added dropwise in dry CH_2Cl_2(4 ml) over 15 min. The mixture was stirred for 3 h at room temperature. The solvent was removed under reduced pressure, and the viscous residue was purified by flash column chromatography (silica gel; petroleum ether-ethyl acetate (10:1)). The solvent was removed under reduced pressure, and the product **3** was obtained. The characterization data of the compound are given below:

*2-[(E)-2-Phenyl-1-ethenyl]-1,3,4-oxadiazole (***3a***).* Light yellow oil, yield: 77%; IR (neat): (υ_{max}, cm^{-1}): 2930 (m), 1639 (s), 1523 (m), 1100 (s), and 977 (m); ^{1}H NMR (250 MHz, $CDCl_3$): δ_H 7.09 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 7.45–7.41 (m, 3H, arom); 7.58 (d, 2H, $^3J_{HH}$ = 7.0 Hz, arom); 7.62 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 8.38 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 109.53 (CH, vinylic); 127.57, 129.03 and 130.14 (CH, arom); 134.53 (C, arom); 139.85 (CH, vinylic); 152.00 (CH, oxadiazole); 164.28 (C, oxadiazole); $C_{10}H_8N_2O$ (172.06) MS (EI) *m/z,%*: 172 (M^+, 100); 115 (22); 89 (9); 77 (7); 63 (8) and 51(8).

*2-[(E)-2-(4-Methylphenyl)-1-ethenyl]-1,3,4-oxadiazole (***3b***).* White crystals; mp: 83.2–84.1°C; yield: 81%; IR (KBr): (υ_{max}, cm^{-1}): 2926 (m), 1643 (s), 1533 (m), 1100 (s), and 972 (m); ^{1}H NMR (250 MHz, $CDCl_3$): δ_H 2.39 (s, 3H, CH_3); 7.02 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 7.22 (d, 2H, $^3J_{HH}$ = 7.75 Hz, arom); 7.46 (d, 2H, $^3J_{HH}$ = 7.75 Hz, arom); 7.57 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 8.36 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 21.45 (CH_3); 108.46 (CH, vinylic); 127.5 and 129.75 (CH, arom); 134.53 and 140.57 (C, arom); 139.82 (CH, vinylic); 151.91 (CH, oxadiazole); 164.47 (C, oxadiazole); Anal Calcd for $C_{11}H_{10}N_2O$ (186.21): C, 70.95; H, 5.41; N, 15.04%; Found: C, 70.99; H, 5.44; N, 15.01.

*2-[(E)-2-(4-Chlorophenyl)-1-ethenyl]-1,3,4-oxadiazole (***3c***).* White crystals; mp: 95.4–96.3°C; yield: 80%; IR (KBr): (υ_{max}, cm^{-1}): 2933 (m), 1646 (s), 1510 (m), 1088 (s), and 980 (m); ^{1}H NMR (250 MHz, $CDCl_3$): δ_H 7.04 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 7.39 (d, 2H, $^3J_{HH}$ = 8.50 Hz, arom); 7.49 (d, 2H, $^3J_{HH}$ = 8.50 Hz, arom); 7.55 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 8.38 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 110.07 (CH, vinylic); 128.72 and 129.31 (CH, arom); 134.62 and 136.57 (C, arom); 138.41 (CH, vinylic); 152.11 (CH, oxadiazole); 164.51 (C, oxadiazole); Anal Calcd for $C_{10}H_7ClN_2O$ (206.63): C, 58.13; H, 3.41; N, 13.56%; Found: C, 58.15; H, 3.44; N, 13.52.

*2-[(E)-2-(3-Chlorophenyl)-1-ethenyl]-1,3,4-oxadiazole (***3d***).* White crystals; mp: 81.4–82.2°C; yield: 77%. IR (KBr) (υ_{max}, cm^{-1}): 2933 (m), 1650 (s), 1509 (m), 1101 (s), and 971 (m); ^{1}H NMR (250 MHz, $CDCl_3$): δ_H 7.08 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 7.28–7.57 (m, 4H, arom); 7.54 (d, 1H, $^3J_{HH}$ = 16.5 Hz, vinylic); 8.39 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 110.94 (CH, vinylic); 125.64, 127.45, 130.03, and 130.27 (CH, arom); 134.64 and 136.60 (C, arom); 138.25 (CH, vinylic); 152.19 (CH, oxadiazole); 164.53 (C, oxadiazole); Anal Calcd for $C_{10}H_7ClN_2O$ (206.63): C, 58.13; H, 3.41; N, 13.56%; Found: C, 58.14; H, 3.42; N, 13.54.

*2-[(E)-2-(3-Methoxyphenyl)-1-ethenyl]-1,3,4-oxadiazole (**3e**).* Light yellow oil, Yield: 76%; IR (neat): (υ_{max}, cm^{-1}): 2932 (m), 1649 (s), 1599 (m), 1509 (m), 1240 (s), 1173 (s), 1099 (s), and 970 (m); ^1H NMR (250 MHz, $CDCl_3$): δ_H 3.85 (s, 3H, OCH_3), 6.93–7.36 (m, 4H, arom); 7.00 (d, 1H, $^3J_{HH} = 16.5$ Hz, vinylic); 7.57 (d, 1H, $^3J_{HH} = 16.5$ Hz, vinylic); 8.37 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 55.33 (OCH_3), 109.81 (CH, vinylic); 112.58, 115.90, 120.25, and 130.03 (CH, arom); 134.59 and 160.59 (C, arom); 139.75 (CH, vinylic); 152.03 (CH, oxadiazole); 163.82 (C, oxadiazole); Anal Calcd for $C_{11}H_{10}N_2O_2$ (202.21): C, 65.34; H, 4.98; N, 13.85%. Found: C, 65.36; H, 4.99; N, 13.83.

*2-[(E)-1-Methyl-2-phenyl-1-ethenyl]-1,3,4-oxadiazole (**3f**).* Light yellow oil, yield: 83%; IR (neat): (υ_{max}, cm^{-1}): 2994 (m), 1630 (s), 1529 (m), 1096 (s), and 980 (m); ^1H NMR (250 MHz, $CDCl_3$): δ_H 2.42 (s, 3H, CH_3); 7.34–7.57 (m, 6H, arom and vinylic); 8.38 (s, 1H, oxadiazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_C 14.68 (CH_3); 121.12 (C, vinylic); 128.44, 128.52, and 129.56 (CH, arom); 135.04 (CH, vinylic); 135.35 (C, arom); 152.35 (CH, oxadiazole); 166.82 (C, oxadiazole); Anal Calcd for $C_{11}H_{10}N_2O$ (186.21): C, 70.95; H, 5.41; N, 15.04%; Found: C, 70.97; H, 5.43; N, 15.06.

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