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Efficient One-Pot Synthesis of Disubstituted 1,3,4-Oxadiazole Derivatives from the Reaction of (*N*-Isocyanimino)triphenylphosphorane, Acetaldehyde, a Secondary Amine, and an Electron-Poor (*E*)-Cinnamic Acid

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ABSTRACT: *The iminium intermediate formed by the reaction of a secondary amine with acetaldehyde was reacted by (N-isocyanimino) triphenylphosphorane in the presence of an electron-poor (E)-cinnamic acid derivative to give the corresponding iminophosphorane intermediate, whose intramolecular the aza-Wittig reaction led to disubstituted 1,3,4-oxadiazole derivatives. The reactions were completed under neutral conditions at room temperature, and the corresponding disubstituted 1,3,4-oxadiazole derivatives were produced in excellent yields.* © 2010 Wiley Periodicals, Inc. Heteroatom Chem 22:79–84, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20660

INTRODUCTION

Recently, multicomponent condensation reactions have become one of the most powerful methods for the synthesis of small molecule libraries, because products are formed in a single step by simultaneous reactions of several reagents and the molecular diversity required for such combinatorial libraries can be achieved by simply varying each component [1]. This principle, therefore, is highly efficient in terms of time as well as resources [2]. Among the known multicomponent reactions to date, the most valuable reactions are those based on isocyanides. Isocyanide-based multicomponent reactions (abbreviated as IMCRs by Ugi and Dömling) by virtue of their synthetic potential, their inherent atom efficiency, convergent nature, ease of implementation, and the generation of molecular diversity, have attracted much attention because of the advantages that they offer to the field of combinatorial chemistry [3].

Iminophosphoranes are a class of special type of zwitterions, which bear a strong nucleophilic electron-rich nitrogen. The electron distribution around the $P^+—N^-$ bond and its consequent chemical implications have been probed and assessed through theoretical, spectroscopic and crystallographic investigations [4]. The 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 [4–6].

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. The nucleophilicity at the

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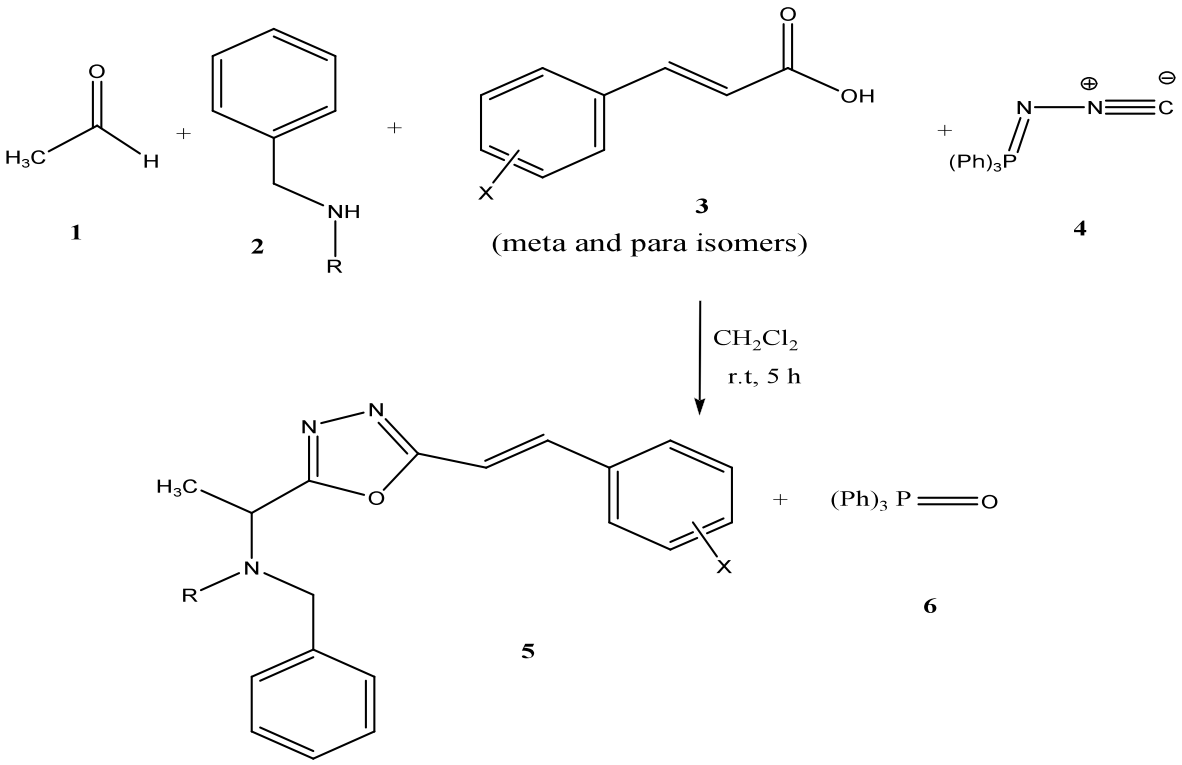
nitrogen is a factor of essential mechanistic importance in the use of these iminophosphoranes as aza-Wittig reagents. Iminophosphoranes are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products, compounds with biological and pharmacological activity [4–6]. However, the organic chemistry of (*N*-isocyanimino)triphenylphosphorane **4** remains almost unexplored. (*N*-Isocyanimino) triphenylphosphorane **4** is expected to have synthetic potential because it provides a reaction system in which the iminophosphorane group can react with a reagent having a carbonyl functionality [7,8]. In recent years, we have established a one-pot method for the synthesis of organophosphorus compounds [9–17]. As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds [18–26], we wish to report the synthesis of a disubstituted 1,3,4-oxadiazole derivatives **5** by a four-component condensation of secondary amine **2**, acetaldehyde **1**, (*N*-isocyanimino)triphenylphosphorane **4**, and an electron-poor (*E*)-cinnamic acid derivative **3** in excellent yields under neutral conditions (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, anti-inflammatory, antihypertensive, analgesic, antibacterial, hypoglycemic, antimalarial, antitubercular, and antidepressant [27]. Several methods have been reported in the literature for the synthesis of 1,3,4-oxadiazoles. These protocols are multistep in nature [28]. 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 [29].

RESULTS AND DISCUSSION

The imine intermediate generated by the reaction of secondary amine **2** with acetaldehyde **2** is trapped by the (*N*-isocyanimino)triphenylphosphorane in the presence of an electron-poor (*E*)-cinnamic acid derivative **3** to lead to the formation of disubstituted 1,3,4-oxadiazole derivatives **5** and triphenylphosphine oxide (**6**, Scheme 1 and Table 1). The reaction



SCHEME 1 Four-component synthesis of disubstituted 1,3,4-oxadiazole derivatives **5a–g** (see Table 1 and the text).

TABLE 1 Synthesis of Disubstituted 1,3,4-Oxadiazole Derivatives **5** (see Scheme 1)

Compounds	*R*	*X*	*Yield[a] (%)*
3a	Tert-butyl	Cl (meta)	82
3b	Isopropyl	Cl (para)	85
3c	Ethyl	Cl (para)	80
3d	Tert-butyl	Cl (para)	83
3e	Ethyl	Cl (meta)	85
3f	Isopropyl	Cl (meta)	83
3g	Tert-butyl	F (para)	82

[a]Yield of isolated products.

proceeds smoothly and cleanly under mild conditions, and no side reactions were observed.

The structures of the products were deduced from their ^{1}H NMR, ^{13}C NMR, mass, and IR spectra. For example, the ^{1}H NMR spectrum of **5a** consisted of a singlet for the $3CH_3$ of amin ($\delta = 1.17$ ppm), a doublet for CH_3 ($\delta = 1.53$ ppm, $^3J_{HH} = 7.0$ Hz), and a singlet for the CH_3 ($\delta = 2.37$ ppm), a AB-quartet for CH_2 of benzyl group at $\delta = 3.98$ and 4.13 ppm ($^2J_{HH} = 17.3$ Hz), a quartet for the CH ($\delta = 4.76$ ppm, $^3J_{HH} = 7.0$ Hz), and a multiplet at $\delta = 6.94$–7.52 ppm for H-aromatic and H-vinylic. The aryl groups exhibited characteristic signals in the aromatic region of the spectrum. The ^{1}H decoupled ^{13}C NMR spectrum of **5a** showed 19 distinct resonances; partial assignment of these resonances is given in the Experimental section. The ^{1}H and ^{13}C NMR spectra of compounds **5b–g** were similar to those of **5a**, except for the aromatic and aliphatic moieties, which exhibited characteristic signals with appropriate chemical shifts.

A mechanistic rationalization for this reaction is provided in Scheme 2. It is conceivable that the initial event is the condensation reaction of the acetaldehyde **1**, secondary amine **2**, and electron-poor (*E*)-cinnamic acid derivative **3** that leads to an intermediate iminium ion **7**. Nucleophilic addition of the (*N*-isocyanimino)triphenylphosphorane **4** to the intermediate iminium ion **7** leads to a nitrilium intermediate **8**. This intermediate may be attacked by the conjugate base of the acid **3** to form 1:1:1 adduct **9**. This adduct may undergo an intramolecular aza-Wittig reaction of an iminophosphorane moiety with the ester carbonyl group to afford the isolated disubstituted 1,3,4-oxadiazole **5** by the removal of triphenylphosphine oxide **6** from intermediate **10**.

CONCLUSIONS

The reported method offers a mild, simple, and efficient route for the preparation of disubstituted 1,3,4-oxadiazole derivatives, by a sequence of multicomponent reactions and an intramolecular aza-Wittig closure. Because of the easy availability of the synthetic approach and the neutral ring closure conditions, this new synthetic approach discussed here has the potential in synthesis of various disubstituted 1,3,4-oxadiazoles, which are of considerable interest as potential biologically active compounds or pharmaceuticals.

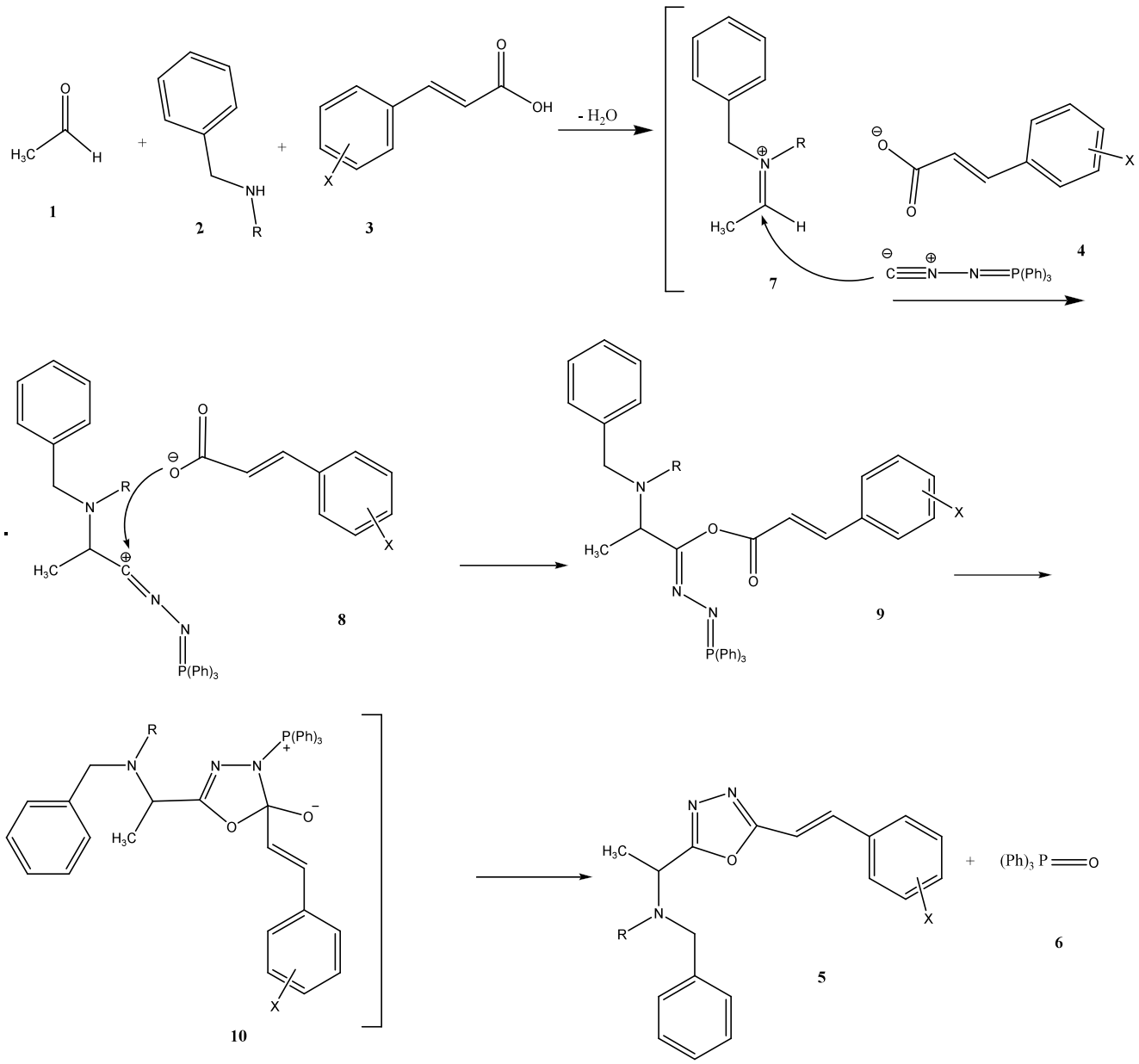
EXPERIMENTAL

Starting materials and solvents were obtained from Merck (Darmstadt, Bundesland, Germany) and Fluka (Buchs, Switzerland) and were used without further purification. The methods used to follow the reactions are thin layer chromatography (TLC) and NMR, which indicated that there is no side product. Melting points (mp) were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer. ^{1}H- and ^{13}C-NMR spectra were measured ($CDCl_3$) with a Bruker DRX-250 Avance spectrometer at 250.0 and 62.5 MHz, respectively. 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 20 eV. Flash chromatography columns were prepared with the Merck silica gel powder.

General Procedure

To a magnetically stirred solution of secondary amine **2** (1 mmol), acetaldehyde **1** (1 mmol), and (*N*-isocyanimino)triphenylphosphorane **4** (1 mmol) in CH_2Cl_2 (5 mL), a solution of an electron-poor (*E*)-cinnamic acid derivative **3** (1 mmol) in CH_2Cl_2 (5 mL) at room temperature was added dropwise over 15 min. The mixture was stirred for 5 h. The solvent was removed under reduced pressure, and the viscous residue was purified by flash column chromatography (silica gel powder; petroleum ether–ethyl acetate (10:2)). The solvent was removed under reduced pressure, and the products **(5a–g)** were obtained. The characterization data of the compounds are given below.

*N-Benzyl-N-(tert-butyl)-N-(1-[5-[(E(-2-(3-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)amine (**5a**).* Yellow oil, yield: 324 mg (82%). IR (neat): 3472, 2977, 1649, 1567, 1475, 1205, 969, 784 cm^{-1}. ^{1}H NMR: 1.17 (s, 9H, CH_3 amin); 1.53 (d, 3H, $^3J_{HH}$ = 7 Hz, CH_3 aliphatic); 3.98, 4.13 (AB quartet, 2H, $^2J_{HH}$ = 17.25 Hz, CH_2 of the benzyl group); 4.76 (q, 1H, $^3J_{HH}$ = 7 Hz, CH); 6.94–7.52 (m, 11H, arom and vinylic).^{13}C NMR: 18.53, 28.64 ($4CH_3$); 56.26 (C



SCHEME 2 Proposed mechanism for the formation of disubstituted 1,3,4-oxadiazole derivatives **5a–g**.

aliphatic); 47.85 (CH_2 of the benzyl group); 48.38 (CH); 111.61, 136.83 (2CH vinylic); 135.03, 136.63, 143.61 (3C arom); 125.43, 126.09, 126.82, 127.39, 127.94, 129.70, 130.21 (9CH arom); 163.48, 168.92 (2C of oxadiazole). Anal. Calcd for $C_{23}H_{26}ClN_3O$ (395.93): C 69.77, H 6.62, N 10.61; Found: C 69.76, H 6.62, N 10.62. MS (EI): 395 (M^+, 2), 380 (8.48), 234 (38.92), 199 (11.78), 174 (12.15), 162 (72.11), 105 (32.49), 91 (100), 84 (39.08), 56 (16.32), 41 (17.52).

N-Benzyl-N-(1-[5-[(E(-2-(4-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)-N-isopropylamine (**5b**). Yellow crystals, MP: 104.3–105.6°. Yield: 324 mg (85%). IR (KBr): 3450, 2965, 2929, 1639, 1536, 1492, 1170, 1087, 817 cm^{-1}. ^1H NMR: 0.90 (d, 3H, $^3J_{HH}$ = 6.25 Hz, CH_3 amin); 1.14 (d, 3H, $^3J_{HH}$ = 6.25 Hz, CH_3 amin); 1.55 (d, 3H,$^3J_{HH}$ = 6.75 Hz, CH_3); 3.14–3.25 (m, 1H, CH amin); 3.80, 3.85 (AB quartet, 2H, $^2J_{HH}$ = 15 Hz, CH_2 of the benzyl group); 4.26 (q, 1H, $^3J_{HH}$ = 6.75 Hz, CH); 6.99–7.51 (m, 11H, arom and vinylic).^{13}C NMR: 16.89, 19.46, 21.19 ($3CH_3$); 48.32 (CH_2 of the benzyl group); 48.85, 49.61 (2CH); 110.76, 140.75 (2CH vinylic); 133.28, 135.76, 137.14 (3C arom); 126.80, 128.19, 128.22, 128.61, 129.27 (9CH arom); 163.98, 167.88

(2C of oxadiazole). Anal. Calcd for $C_{22}H_{24}ClN_3O$ (381.90): C 69.19, H 6.33, N 11.0; Found: C 69.15, H 6.35, N 11.02. MS (EI): 381 (M^+, 1.93), 249 (5.66), 233 (16.58), 213 (26.53), 148 (69.35), 105 (21.69), 91 (100), 83 (36.98), 69 (33.01), 56 (61.04), 41 (65.77).

N-Benzyl-N-(1-[5-[(E(-2-(4-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)-N-ethylamine (**5c**). Yellow oil, yield: 294 mg (80%). IR (neat): 3459, 2979, 2937, 1645, 1530, 1491, 1089, 970, 815 cm^{-1}. ^1H NMR: 1.09 (t, 3H, $^3J_{HH}$ = 7 Hz, CH_3 of Et); 1.57 (d, 3H, $^3J_{HH}$ = 7 Hz, CH_3); 2.42–2.52 and 2.67–2.78 (2m, CH_2 of Et); 3.54, 3.87 (AB quartet, 2H, $^2J_{HH}$ = 14.25 Hz, CH_2 of the benzyl group); 4.26 (q, 1H, $^3J_{HH}$ = 7 Hz, CH); 6.97–7.51 (m, 11H, arom and vinylic).^{13}C NMR: 13.75, 15.42 ($2CH_3$); 44.46 (CH_2 of ethyl); 50.77 (CH_2 of the benzyl group); 54.42 (CH aliphatic); 109.14, 137.35 (2CH vinylic); 133.26, 139.78 and 140.38 (3C arom); 126.97, 128.27, 128.49, 128.61, 129.28 (9CH arom); 164.54, 166.65 (2C of oxadiazole). Anal. Calcd for $C_{21}H_{22}ClN_3O$: C 68.56; H 6.03; N 11.42; Found: C 68.58; H 6.01; N 11.44. MS (EI): 367 (M^+, 2.1), 277 (5.23), 247 (20.72), 233 (44.99), 205 (16.25), 162 (24.36), 149 (41.56), 134 (100), 105 (22.24), 91 (65.12), 69 (26.47), 56 (42.43), 41 (16.98).

N-Benzyl-N-(tert-butyl)-N-(1-[5-[(E(-2-(4-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)amine (**5d**). Yellow crystals, mp: 90.2–91.8°. Yield: 328 mg (83%). IR (KBr): 3472, 2972, 2929, 1637, 1536, 1491, 1085, 997, 817 cm^{-1}. ^1H NMR: 1.17 (s, 9H, CH_3 amin); 1.53 (d, 3H, $^3J_{HH}$ = 7 Hz, CH_3 aliphatic); 3.98, 4.13 (AB quartet, 2H, $^2J_{HH}$ = 17.25 Hz, CH_2 of the benzyl group); 4.76 (q, 1H, $^3J_{HH}$ = 7 Hz, CH); 6.95–7.50 (m, 11H, arom and vinylic).^{13}C NMR: 18.60, 28.63 ($4CH_3$); 56.25 (C aliphatic); 47.86 (CH_2 of the benzyl group); 48.38 (CH); 110.76, 137.01 (2CH vinylic); 133.31, 135.70, 143.67 (3C arom); 126.07, 126.81, 127.94, 128.59, 129.25 (9CH arom); 163.64, 168.84 (2C of oxadiazole). Anal. calcd for $C_{23}H_{26}ClN_3O$ (395.93): C 69.77, H 6.62, N 10.61; Found: C 69.78, H 6.60, N 10.63.

N-Benzyl -N-(1-[5-[(E(-2-(3-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)-N-ethylamine (**5e**). Yellow oil, yield: 287 mg (85%). IR (neat): 3473, 2933, 1650, 1563, 1456, 1080, 969, 783 cm^{-1}. ^1H NMR: 1.08 (t, 3H, CH_3 of Et); 1.59 (d, 3H, CH_3); 2.42–2.55 and 2.70–2.79 (2m, CH_2 of Et); 3.64, 3.78 (AB quartet, 2H, $^2J_{HH}$ = 13.75 Hz, CH_2 of the benzyl group); 4.65 (q, 1H, CH); 6.99–7.53 (m, 11H, arom and vinylic).^{13}C NMR: 13.80, 18.12 ($2CH_3$); 44.43 (CH_2 of ethyl); 52.23 (CH_2 of the benzyl group); 54.48 (CH aliphatic); 111.45, 136.35 (2CH vinylic); 135.04, 136.62, 143.60 (3C arom); 125.49, 127.01, 127.40, 128.28, 128.54, 129.79,130.23 (9CH arom); 164.54, 166.65 (2C of oxadiazole). Anal. Calcd for $C_{21}H_{22}ClN_3O$ (367.87): C 68.56, H 6.03, N 11.42, Found: C 68.59, H 6.05, N 11.43.

N-Benzyl-N-(1-[5-[(E(-2-(3-chlorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)-N-isopropylamine (**5f**). Yellow oil, yield: 317 mg (83%). IR (neat): 3458, 2971, 2935, 1648, 1566, 1455, 1173, 968, 783 cm^{-1}. ^1H NMR: 0.90 (d, 3H, $^3J_{HH}$ = 6.25 Hz, CH_3 amin); 1.14 (d, 3H, $^3J_{HH}$ = 6.25 Hz, CH_3 amin); 1.55 (d, 3H,$^3J_{HH}$ = 6.5 Hz, CH_3); 3.17–3.22 (m, 1H, CH amin); 3.77, 3.88 (AB quartet, 2H, $^2J_{HH}$ = 15 Hz, CH_2 of the benzyl group); 4.26 (q, 1H, $^3J_{HH}$ = 6.5 Hz, CH); 6.98–7.54 (m, 11H, arom and vinylic). ^{13}C NMR: 16.87, 19.47, 21.18 ($3CH_3$), 48.34 (CH_2 of the benzyl group); 48.87 and 49.61 (2CH); 111.62, 136.97 (2CH vinylic); 135.05, 136.60, 143.65 (3C arom); 125.47, 126.80, 127.40, 128.11, 128.23, 129.75, 130.22 (9CH arom); 163.81, 168.75 (2C of oxadiazole). Anal. Calcd for $C_{22}H_{24}ClN_3O$ (381.90): C 69.19, H 6.33, N 11; Found: C 69.21, H 6.30, N 11.02. MS (EI): 381 (M^+, 1.6), 247 (10.52), 233 (33.61), 213 (18.96), 205 (28.15), 163 (28.37), 148 (100), 105 (78.74), 91 (75.68), 63 (42.29), 56 (99.02), 41 (41.37).

N-Benzyl-N-(tert-butyl)-N-(1-[5-[(E(-2-(4-fluorophenyl)-1-ethenyl]-1,3,4-oxadiazol-2-yl]ethyl)amine (**5g**). Yellow oil, yield: 311 mg (82%). IR (neat): 3456, 2970, 2934, 1646, 1532, 1236, 979, 835 cm^{-1}. ^1H NMR: 1.17 (s, 9H, CH_3 amin); 1.53 (d, 3H, $^3J_{HH}$ = 6.75 Hz, CH_3 aliphatic); 3.99, 4.13 (AB quartet, 2H, $^2J_{HH}$ = 17.25 Hz, CH_2 of the benzyl group); 4.76 (q, 1H, $^3J_{HH}$ = 6.75 Hz, CH); 6.87–7.56 (m, 11H, arom and vinylic).^{13}C NMR: 18.65, 28.61 ($4CH_3$); 56.24 (C aliphatic), 47.88 (CH_2 of the benzyl group); 48.39 (CH); 109.96, 137.16 (2CH vinylic); 133.31, 143.70, 163.76 (3C arom); 115.96, 116.31, 126.08, 126.81, 127.95, 129.71, 129.30 (9CH arom); 161.60, 165.76 (2C of oxadiazole). Anal. Calcd for $C_{23}H_{26}FN_3O$ (379.47): C 72.80, H 6.91, N 11.07; Found: C 72.78, H 6.89, N 11.10.

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