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Four-Component Preparation of Disubstituted 1,3,4-Oxadiazoles from (*N*-isocyanimino)triphenylphosphorane, Phenylacetylenecarboxylic Acid, Biacetyl and Primary Amines

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A simple method has been developed for four-component synthesis of disubstituted 1,3,4-oxadiazoles using (*N*-isocyanimino)triphenylphosphorane, a primary amine, a carboxylic acid and biacetyl in CH_2Cl_2 by the Ugi-4CR/*aza*-Wittig sequence at room temperature in excellent yields.

Key Words : (*N*-Isocyanimino)triphenylphosphorane, Biacetyl, Phenylacetylenecarboxylic acid, Primary amine, 1,3,4-Oxadiazole

Introduction

Multicomponent reactions (MCR) have appeared as an efficient and powerful tool in modern synthetic organic chemistry due to their valued features such as atom economy, straightforward reaction design, and the opportunity to construct target compounds by the introduction of several diversity elements in a single chemical event. Since all the organic reagents employed are consumed and incorporated into the target compound, purification of products resulting from MCR is also simple.[1] MCR, leading to interesting heterocyclic scaffolds, are especially useful for the construction of diverse chemical libraries of ‘druglike’ molecules. The isocyanide-based MCR are very important in this area.[2-4] Among the known multicomponent reactions to date, the most valuable reactions are those based on isocyanides. Isocyanide-based multicomponent reactions (abbreviated to 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 considerable attention because of the advantages that they offer to the field of combinatorial chemistry.[5-9]

In recent years there has been considerable investigation on different classes of oxadiazoles. Particularly, compounds containing 1,3,4-oxadiazole nucleus have been shown to possess a wide range of pharmacological and therapeutic activities. Some 1,3,4-oxadiazoles have shown analgesic, anti-inflammatory, anticonvulsant, tranquilizing, myorelaxant, antidepressant, vasodilatatory, diuretic, antiulcer, antiarythmic, antiserotoninic, spasmolytic, hypotensive, antibronchocontrictive, anticholinergic, and antiemetic activities.[10-13] Several methods have been reported in the literature for the synthesis of 1,3,4-oxadiazoles. These protocols are multistep in nature.[13,14] 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.[14,15]

In the last years, several preparative procedures have been reported for the providing and synthetic applications of iminophosphoranes.[16] It is expected (*N*-isocyanimino) triphenylphosphorane (**4**) to have synthetic potential because it develops a reaction system in which the iminophosphorane group can react with a reagent having a carbonyl functionality.[16,17] In recent years, we have confirmed a one-pot method for the preparation of organophosphorus compounds.[18-24] As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,[25-38] we wish to report the preparation of a new class disubstituted 1,3,4-oxadiazole derivatives **5a-j** by a novel four-component condensation reaction of biacetyl (**1**), primary amine **2**, (*N*-isocyanimino)triphenylphosphorane (**4**) and phenylacetylenecarboxylic acid (**3**) in excellent yields under neutral conditions (Scheme 1).

Experimental

(*N*-Isocyanimino)triphenylphosphorane (**4**) was prepared based on reported procedures.[17] Other starting materials and solvents were obtained from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The methods used to follow the reactions are TLC and NMR. TLC and NMR indicated that there is no side product. Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra ($CDCl_3$) were recorded on a BRUKER DRX-250AVANCE spectrometer at 250.0 and 62.9 MHz, respectively. IR spectra were measured on a Jasco 6300 FTIR spectrometer. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 mass spectrometer operating at an ionization potential of 70 eV. Preparative layer chromatography (PLC) plates were prepared from Merck silica gel (F_{254}) powder.

General Procedure for Compounds 5a-j. To a magnetically stirred solution of primary amine derivatives (1 mmol), biacetyl (1 mmol), and (*N*-isocyanimino)triphenylphosphorane (0.30 g, 1 mmol) in CH_2Cl_2 (5 mL) was added dropwise a solution of phenylacetylenecarboxylic acid (1 mmol) in CH_2Cl_2 (5 mL) at room temperature over 15 min. The mixture was stirred for 12 h. The solvent was removed under reduced pressure, and the viscous residue was purified by preparative layer chromatography (PLC) plates (silica gel (F_{254}) powder; petroleum ether-ethyl acetate (4:1)). The characterization data of the compounds are given below:

3-[(2-Chlorobenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5a). Yellow viscous oil, yield: 87%. IR (KBr): 3423, 2923, 2837, 2231, 1723, 1601, 1538, 1490, 1245, 1027, 760, 689 cm^{-1}; ^1H NMR δ 7.20-7.64 (m, 9H, CH_{arom}), 3.72 and 3.94 (AB quartet, J = 13.0 Hz, 2H, CH_2 of benzyl), 2.00 (s, 1H, NH), 2.29, 1.90 (s, 6H, $2CH_3$). ^{13}C NMR δ 203.82 (C=O), 166.84, 151.69 (2C=N), 136.64, 132.50, 119.60 (3C), 133.70, 132.37, 130.74, 130.11, 129.53, 128.68, 126.97 (9CH), 97.49, 72.65, 66.30 (3C), 45.20 (CH_2), 24.86, 20.41 ($2CH_3$). MS *m/z* (%) 379 (M^+), 332 (3), 290 (12), 256 (3), 184 (4), 136 (100), 119 (92), 91 (72), 43 (4). Anal. Calcd for $C_{21}H_{18}ClN_3O_2$ (379.84): C 66.40, H 4.78, N 11.06. Found: C 66.46, H 4.70, N 11.14.

3-(Benzylamino)-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5b). Yellow viscous oil, yield: 82%. IR (KBr): 3420, 3029, 2924, 2230, 1721, 1653, 1548, 1453, 1094, 750, 689 cm^{-1}; ^1H NMR δ 7.28-7.64 (m, 10H, CH_{arom}), 3.62 and 3.82 (AB quartet, J = 12.5 Hz, 2H, CH_2 of benzyl), 2.53 (s, 1H, NH), 2.30, 1.87 (s, 6H, $2CH_3$). ^{13}C NMR δ 204.25 (C=O), 167.48, 159.56 (2C=N), 132.37, 130.77, 128.70, 128.51, 128.11, 127.35 (10CH), 149.50, 119.62 (2C), 98.30, 83.23, 66.55 (3C), 47.95 (CH_2), 24.79, 20.68 ($2CH_3$). MS *m/z* (%) 345 (M^+), 329 (2), 302 (8), 275 (8), 176 (8), 135 (98), 129 (39), 106 (16), 91 (100), 77 (10), 57 (17), 43 (16). Anal. Calcd for $C_{21}H_{19}N_3O_2$ (345.39): C 73.03, H 5.54, N 12.17. Found: C 73.12, H 5.49, N 12.10.

3-[(4-Methoxybenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5c). Yellow viscous oil, yield: 85%. IR (KBr): 3425, 2931, 2230, 1722, 1611, 1512, 1442, 1247, 1032, 757, 689 cm^{-1}; ^1H NMR δ 6.83-7.63 (m, 9H, CH_{arom}), 3.77 (s, 3H, OCH_3), 3.54 and 3.75 (AB quartet, J = 12.3 Hz, 2H, CH_2 of benzyl), 2.35 (s, 1H, NH), 2.28, 1.86 (s, 6H, $2CH_3$). ^{13}C NMR δ 204.21 (C=O), 167.52, 158.91 (2C=N), 158.91, 131.35, 119.72 (3C), 132.36, 130.74, 129.33, 128.69, 113.91 (9CH), 86.45, 73.64, 66.51 (3C), 55.25 (OCH_3), 47.38 (CH_2), 24.86, 20.68 ($2CH_3$). Anal. Calcd for $C_{22}H_{21}N_3O_3$ (375.42): C 70.38, H 5.64, N 11.19. Found: C 70.46, H 5.72, N 11.11.

3-[(4-Fluorobenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5d). Yellow viscous oil, yield: 83%. IR (KBr): 3421, 2924, 2853, 2230, 1722, 1603, 1538, 1500, 1221, 757, 688 cm^{-1}; ^1H NMR δ 6.96-7.64 (m, 9H, CH_{arom}), 3.59 and 3.77 (AB quartet, J = 11.5 Hz, 2H, CH_2 of benzyl), 2.53 (s, 1H, NH), 2.29, 1.86 (s, 6H, $2CH_3$). ^{13}C NMR δ 203.92 (C=O), 168.42, 154.95 (2C=N), 160.50 (d, J = 440.3 Hz, C), 135.00, 119.55 (2C), 132.37, 130.80, 128.71 (5CH), 129.70 (d, J = 7.5 Hz, 2CH), 115.29 (d, J = 20.8 Hz, 2CH), 97.25, 88.51, 66.45 (3C), 47.19 (CH_2), 24.90, 20.71 ($2CH_3$). Anal. Calcd for $C_{21}H_{18}FN_3O_2$ (363.38): C 69.41, H 4.99, N 11.56. Found: C 69.47, H 4.93, N 11.62.

3-[5-(2-Phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-3-{[4-(trifluoromethyl)benzyl]amino}-2-butanone (5e). Yellow viscous oil, yield: 80%. IR (KBr): 3420, 2925, 2855, 2231, 1724, 1662, 1538, 1446, 1124, 757, 702 cm^{-1}; ^1H NMR δ 7.39-7.94 (m, 9H, CH_{arom}), 3.70 and 3.87 (AB quartet, J = 13.0 Hz, 2H, CH_2 of benzyl), 2.54 (s, 1H, NH), 2.30, 1.88 (s, 6H, $2CH_3$). ^{13}C NMR δ 203.75 (C=O), 166.68, 152.87 (2C=N), 140.33, 119.51 (2C), 132.38, 130.84, 128.91, 128.71, 125.50 (7CH), 130.50 (q, J = 88.0 Hz, C), 130.27 (q, J = 142.2 Hz, CF_3), 124.81, 124.18 (q, 2CH), 98.55, 72.68, 66.43 (3C), 47.42 (CH_2), 24.96, 20.79 (2CH3). Anal. Calcd for $C_{22}H_{18}F_3N_3O_2$ (413.39): C 63.92, H 4.39, N 10.16. Found: C 63.96, H 4.45, N 10.10.

3-[(2-Methoxybenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5f). Yellow viscous oil, yield: 78%. IR (KBr): 3415, 3061, 2923, 2230, 1723, 1604, 1537, 1443, 1049, 755, 688 cm^{-1}; ^1H NMR δ 6.81-7.72 (m, 9H, CH_{arom}), 3.83 (s, 3H, OCH_3), 3.62 and 3.83 (AB quartet, J = 13.0 Hz, 2H, CH_2 of benzyl), 2.35 (s, 1H, NH), 2.28, 1.89 (s, 6H, $2CH_3$). ^{13}C NMR δ 204.21 (C=O), 167.22, 156.96 (2C=N), 151.49, 127.07, 119.67 (3C), 132.31, 130.48, 129.72, 128.71, 128.47, 120.57, 110.25 (9CH), 96.42, 75.80, 66.37 (3C), 55.20 (OCH_3), 47.36 (CH_2), 24.82, 20.24 ($2CH_3$). Anal. Calcd for $C_{22}H_{21}N_3O_3$ (375.42): C 70.38, H 5.64, N 11.19. Found: C 70.32, H 5.70, N 11.25.

3-[(3,4-Dichlorobenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5g). Yellow viscous oil, yield: 75%. IR (KBr): 3418, 2925, 2853, 2230, 1723, 1655, 1596, 1470, 1030, 756, 688 cm^{-1}; ^1H NMR δ 7.12-7.89 (m, 8H, CH_{arom}), 3.59 and 3.75 (AB quartet, J = 13.2 Hz, 2H, CH_2 of benzyl), 2.41 (s, 1H, NH), 2.27, 1.84 (s, 6H, $2CH_3$). ^{13}C NMR δ 204.11 (C=O), 166.22, 160.00 (2C=N), 139.89, 138.86, 136.27, 127.20 (4C), 132.42, 130.91, 130.43, 129.90, 128.70 (8CH), 96.87, 92.20, 66,55 (3C), 47.50 (CH_2), 22.35, 24.89 ($2CH_3$). Anal. Calcd for $C_{21}H_{17}Cl_2N_3O_2$ (414.28): C 60.88, H 4.14, N 10.14. Found: C 60.97, H 4.21, N 10.23.

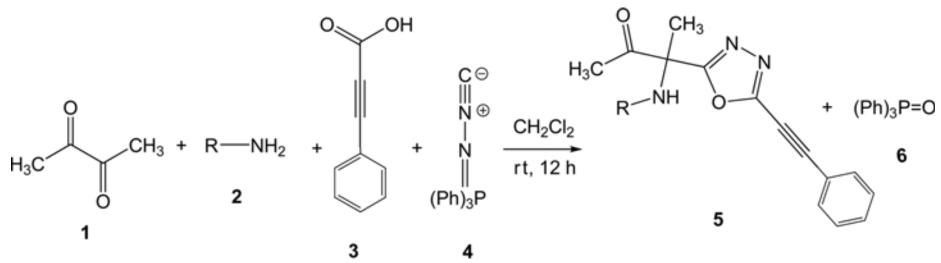
3-[(2-Furylmethyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5h). Yellow viscous oil, yield: 72%. IR (KBr): 3446, 2924, 2853, 2217, 1723, 1648, 1491, 1384, 1287, 741, 688 cm^{-1}; ^1H NMR δ 9.22 (s, 1H, CH_{furan}), 7.33-7.61 (m, 5H, CH_{arom}), 6.26 (s, 1H, CH_{furan}), 6.11 (s, 1H, CH_{furan}), 3.69 and 3.86 (AB quartet, J = 10.5 Hz, 2H, CH_2 of benzyl), 2.52, 2.24 (s, 6H, $2CH_3$), 2.34 (s, 1H, NH). ^{13}C NMR δ 204.15 (C=O), 168.00, 167.65 (2C=N), 142.16, 128.70 (2C), 132.95, 132.35, 130.85, 128.70, 110.09, 107.60 (8CH), 92.23, 86.45, 67.65 (3C), 40.95 (CH_2), 24.80, 20.15 ($2CH_3$). Anal. Calcd for $C_{19}H_{17}N_3O_3$ (335.36): C 68.05, H 5.11, N 12.53. Found: C 68.13, H 5.03, N 12.44.

3-(Allylamino)-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5i). Yellow viscous oil, yield: 75%. IR (KBr): 3425, 3079, 2925, 2853, 2230, 1723, 1643, 1536, 1443, 1090, 764, 688 cm^{-1}; ^{1}H NMR δ 7.37-7.63 (5H, m, CH$_{arom}$), 5.86 (m, 1H, =CH), 5.20 (d, 1H, =CH, J = 17.0 Hz), 5.09 (d, 1H, =CH, J = 10.0 Hz), 3.06 and 3.26 (AB quartet, J = 13.5 Hz, 2H, CH$_2$ of benzyl), 2.52 (s, 1H, NH), 2.26, 1.81 (s, 6H, 2CH$_3$). ^{13}C NMR δ 204.55 (C=O), 168.12, 157.32 (2C=N), 135.71, 132.36, 130.77, 128.69, 116.54 (8CH), 119.95 (C), 90.00, 73.45, 66.24 (3C), 46.32 (CH$_2$), 24.84, 20.60 (2CH$_3$). Anal. Calcd for $C_{17}H_{17}N_3O_2$ (295.34): C 69.14, H 5.80, N 14.23. Found: C 69.07, H 5.87, N 14.30.

3-[(4-Methylbenzyl)amino]-3-[5-(2-phenyl-1-ethynyl)-1,3,4-oxadiazol-2-yl]-2-butanone (5j). Yellow viscous oil, yield: 72%. IR (KBr): 3426, 2917, 2849, 2230, 1722, 1621, 1537, 1443, 1094, 741, 688 cm^{-1}; ^{1}H NMR δ 7.11-7.64 (9H, m, CH$_{arom}$), 3.57 and 3.78 (AB quartet, J = 12.5 Hz, 2H, CH$_2$ of benzyl), 2.32 (s, 1H, NH), 2.32, 2.29, 1.87 (s, 9H, 3CH$_3$). ^{13}C NMR δ 204.73 (C=O), 160.00, 153.48 (2C=N), 142.25, 137.42, 120.00 (3C), 97.00, 92.15, 67.24 (3C), 134.50, 132.36, 129.17, 128.70, 128.07 (9CH), 47.32 (CH$_2$), 24.50, 22.10, 20.03 (3CH$_3$). MS m/z (%) 359 (M$^+$), 357 (3), 329 (8), 316 (60), 198 (8), 146 (12), 129 (40), 120 (64), 105 (100), 91 (28), 77 (48), 43 (20). Anal. Calcd for $C_{22}H_{21}N_3O_2$ (359.42): C 73.52, H 5.89, N 11.69. Found: C 73.60, H 5.81, N 11.61.

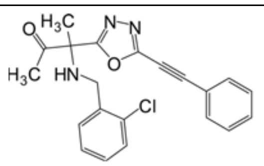
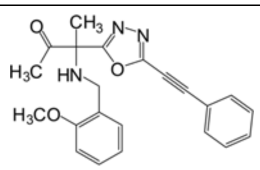
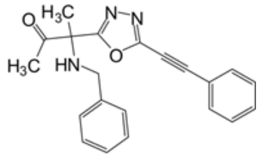
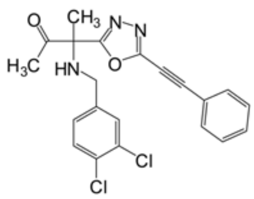
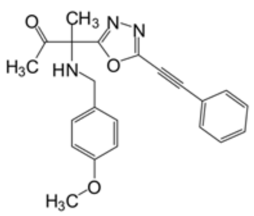
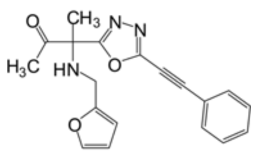
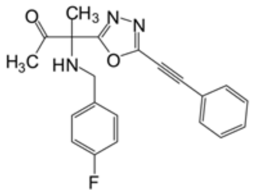
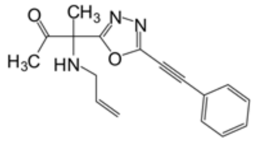
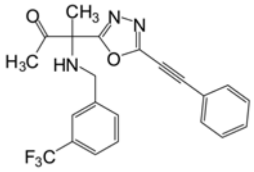
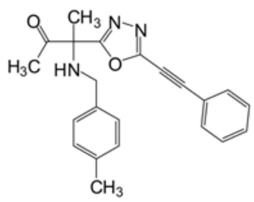
Results and Discussion

The 1:1 imine intermediate generated by the condensation

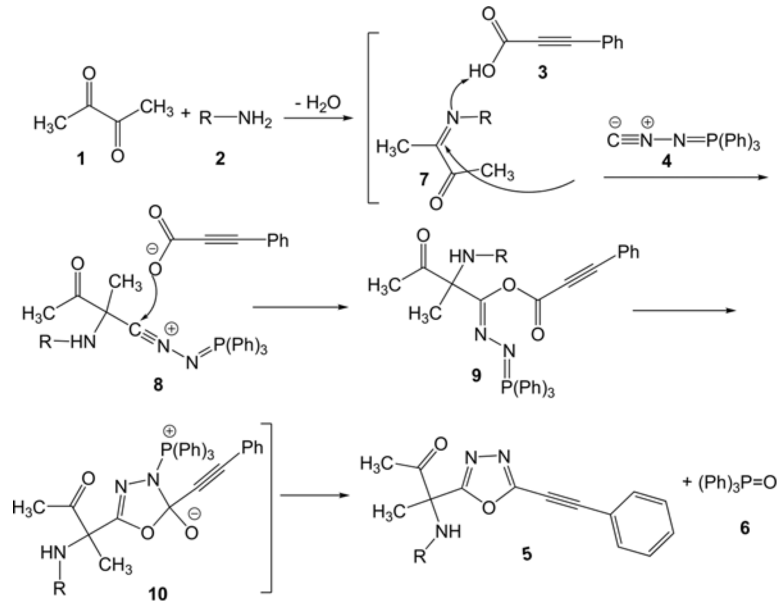


Scheme 1. Four-component synthesis of sterically congested 2,5-disubstituted 1,3,4-oxadiazoles **5** (see Table 1).

Table 1. synthesis of 2,5-disubstituted 1,3,4-oxadiazole derivatives **5a-j** from biacetyl (**1**), primary Amine **2**, and phenylacetylenecarboxylic acid (**3**) in the presence of (N-Isocyanimino)triphenylphosphorane (**4**) (see Scheme 1)

4	R	Product	Yield^a (%)	4	R	Product	Yield^a (%)
a	2-chlorobenzyl		87	**f**	2-methoxybenzyl		78
b	benzyl		82	**g**	3,4-dichlorobenzyl		75
c	4-methoxybenzyl		85	**h**	Furan-2-ylmethyl		72
d	4-fluorobenzyl		83	**i**	allyl		75
e	3-trifluoromethylbenzyl		80	**j**	4-methylbenzyl		72

^{a}Yield of isolated **5**.



Scheme 2. Proposed mechanism for the formation of sterically congested 2,5-disubstituted 1,3,4-oxadiazole derivatives **5**.

reaction of primary amine **2** with biacetyl (**1**) is trapped by the (*N*-isocyanimino)triphenylphosphorane (**4**) in the presence of phenylacetylenecarboxylic acid (**3**) to lead the formation of 1,3,4-oxadiazole derivatives **5** and triphenylphosphine oxide (**6**) (Scheme 1 and Table 1). The reaction proceeds smoothly and clearly under mild and neutral conditions and no side reactions were observed.

The structures of the products were deduced from their IR, ^{1}H NMR, ^{13}C NMR, and Mass spectra. For example the ^{1}H NMR spectrum of **5a** consisted of two singlet for the $2CH_3$ (δ 1.90 and 2.29), a singlet for the NH (δ 2.00), a AB-quartet for CH_2 benzyl group at (δ 3.72 and 3.94, J = 13.0 Hz), 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-j** were similar to those of **5a**, except for the aromatic and aliphatic moieties, which exhibited characteristic signals with appropriate chemical shifts.

A mechanistic pathway for the reaction is provided in Scheme 2. On the basis of the chemistry of isocyanides, it is reasonable to assume that the first step may involve the formation of imine **7** by the condensation reaction of primary amine **2** with biacetyl (**1**), the next step may involve nucleophilic addition of the (*N*-isocyanimino)triphenylphosphorane (**4**) to the imine intermediate **7**, which is facilitated by its protonation with the phenylacetylenecarboxylic acid (**3**), leading to nitrilium intermediate **8**. This intermediate may be attacked by conjugate base of the carboxylic acid to form 1:1:1 adduct **9**. The intermediate **9** may undergo intramolecular *aza*-Wittig reaction[25-39] of iminophosphorane moiety with the ester carbonyl to afford the isolated sterically congested 1,3,4-oxadiazole derivatives **5** by removal of triphenylphosphine oxide (**6**) from intermediate **10**.

We also used (*E*)-cinnamic acid derivatives (4-chloro cinnamic acid, α-methyl cinnamic acid and 3-methoxy cinnamic acid), aromatic carboxylic acids (benzoic acid, 4-bromo benzoic acid, 4-methyl benzoic acid, 4-methoxy benzoic acid and 2-thiophene carboxylic acid) and aliphatic carboxylic acids (acetic acid, cyclohexane carboxylic acid and cyclopropane carboxylic acid) instead of phenylacetylenecarboxylic acid in this reaction, but no corresponding products **5** were observed. Starting materials were recovered without any reaction at the end of reaction and in all the cases, several colored products were detected by TLC monitoring.

Conclusions

We believe that the reported method offers a mild, simple, and efficient route for the preparation of fully substituted 1,3,4-oxadiazol derivatives of type **5**. Due to the easy availability of the synthetic method and the neutral ring closure conditions, this new discussed synthetic method has the potential in synthesis of various disubstituted 1,3,4-oxadiazoles, which are of considerable interest as potential biologically active compounds or pharmaceuticals. Other aspects of this synthetic process are under investigation.

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