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(N-Isocyanimino)triphenylphosphorane-Mediated, One-Pot, Efficient Synthesis of Sterically Congested 1,1,1-Trifluoro-2-(5-aryl-1,3,4-oxadiazol-2-yl)-2-propanol Derivatives via Intramolecular Aza-Wittig Reaction

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(N-ISOCYANIMINO)TRIPHENYLPHOSPHORANE-MEDIATED, ONE-POT, EFFICIENT SYNTHESIS OF STERICALLY CONGESTED 1,1,1-TRIFLUORO-2-(5-ARYL-1,3,4-OXADIAZOL-2-YL)-2-PROPANOL DERIVATIVES VIA INTRAMOLECULAR AZA-WITTIG REACTION

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Reactions of (N-isocyanimino)triphenylphosphorane with 1,1,1-trifluoroacetone in the presence of aromatic (or heteroaromatic) carboxylic acids (3-methylbenzoic acid, 1-naphthalenecarboxylic acid, 2-naphthalenecarboxylic acid, 2-furancarboxylic acid, and 2-thiophenecarboxylic acid) proceed smoothly at room temperature and in neutral conditions to afford sterically congested 1,1,1-trifluoro-2-(5-aryl-1,3,4-oxadiazol-2-yl)-2-propanol derivatives in high yields. The reaction proceeds smoothly and cleanly under mild conditions, and no side reactions were observed.

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Keywords Aromatic carboxylic acid; aza-Wittig reaction; heteroaromatic carboxylic acid; (N-isocyanimino)triphenylphosphorane; 1,3,4-oxadiazole; 1,1,1-trifluoroacetone

INTRODUCTION

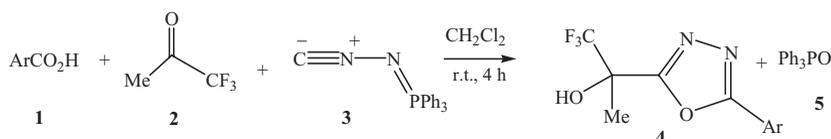
Organophosphorus compounds^{1–5} have been extensively employed in organic synthesis as useful reagents, as well as ligands, in a number of transition metal catalysts.³ Iminophosphoranes are a class of special type of zwitterions that bear a strongly nucleophilic, electron-rich nitrogen. The electron distribution around the P⁺–N[–] bond and its consequent chemical implications have been probed and assessed through theoretical, spectroscopic, and crystallographic investigations.⁶ 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.^{6–14}

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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. Several interesting heterocyclization reactions involving iminophosphoranes have been reviewed.^{6–12} These compounds can easily be 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.^{6–12} 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.^{6–12} However, the organic chemistry of (*N*-isocyanimino)triphenylphosphorane **3** remains almost unexplored.^{13,14} (*N*-Isocyanimino)triphenylphosphorane **3** 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.^{13,14} In recent years, we have established a one-pot method for the synthesis of organophosphorus compounds.^{15–24} As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,^{25–32} we sought to develop a convenient preparation of 1,1,1-trifluoro-2-(5-aryl-1,3,4-oxadiazol-2-yl)-2-propanols **4a–f** from (*N*-isocyanimino)triphenylphosphorane **3**, 1,1,1-trifluoroacetone **2**, and aromatic (or heteroaromatic) carboxylic acids **1** (3-methylbenzoic acid, 1-naphthalenecarboxylic acid, 2-naphthalenecarboxylic acid, 2-furancarboxylic acid, and 2-thiophenecarboxylic acid) in excellent yields under neutral conditions (Scheme 1).



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

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, and antihypertensive.^{33–39} Several methods have been reported in the literature for the synthesis of 1,3,4-oxadiazoles. These protocols are multistep in nature.^{40–45} 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.^{46–50}

RESULTS AND DISCUSSION

In the last years, several synthetic methods have been reported for the preparation of (*N*-isocyanimino)triphenylphosphorane (CNNPPh_3) **3** (Scheme 1).^{13,14} There are

Table I Synthesis of sterically congested 2,5-disubstituted 1,3,4-oxadiazoles derivatives **4** (see Scheme 1)

4	Ar	Yield% ^{a,b}
a	3-MeC ₆ H ₄	88
b	1-Naphthyl	86
c	2-Naphthyl	83
d	2-Furyl	87
e	2-Thienyl	80
f	2-BrC ₆ H ₄	82

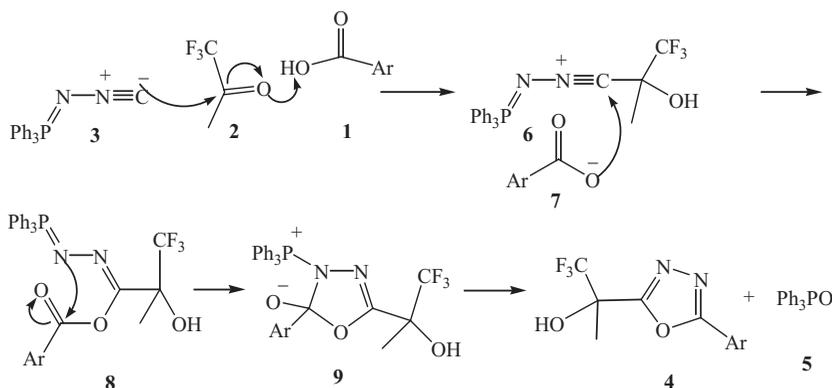
^aWe also used acetone and acetophenone instead of 1,1,1-trifluoroacetone **2** in this reaction, but no corresponding products **4** were observed.

^bIsolated yields.

several reports for the use of (*N*-isocyanimino)triphenylphosphorane **3** in the synthesis of metal complexes.^{13,14} However, the application of **3** in the synthesis of organic compounds is rare.^{25–31} As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds,^{25–32} we sought to develop a convenient preparation of sterically congested 2,5-disubstituted 1,3,4-oxadiazoles **4** from (*N*-isocyanimino)triphenylphosphorane **3**, 1,1,1-trifluoroacetone **2**, and aromatic (or heteroaromatic) carboxylic acids **1** (3-methylbenzoic acid, 1-naphthalenecarboxylic acid, 2-naphthalenecarboxylic acid, 2-furancarboxylic acid, and 2-thiophenecarboxylic acid) in excellent yields under neutral conditions (Scheme 1). The carboxylic acid derivative **1** with 1,1,1-trifluoroacetone **2** and (*N*-isocyanimino)triphenylphosphorane **3** in CH₂Cl₂ react together in a 1:1:1 ratio at room temperature to produce sterically congested 2,5-disubstituted 1,3,4-oxadiazoles **4** and triphenylphosphine oxide **5** (Scheme 1 and Table I). The reaction proceeds smoothly and cleanly under mild conditions, and no side reactions were observed. We also used acetone and acetophenone instead of 1,1,1-trifluoroacetone **2** in this reaction, but no corresponding products **4** were observed. In the both cases, 2-aryl-1,3,4-oxadiazoles and triphenylphosphine oxide **5** were observed, as has been previously reported,²⁶ and the acetone and acetophenone were recovered unreacted at the end of reaction. As indicated in Table I, the reactions proceeded efficiently with an electron-poor ketone (1,1,1-trifluoroacetone) **2**, and electron-rich ketones such as acetone and acetophenone are not suitable starting materials in these reactions.

The structures of the products were deduced from their IR, mass, H NMR, and ¹³C NMR spectra. The mass spectra of these compounds displayed molecular ion peaks at the appropriate *m/z* values. The H NMR spectrum of **4a** consisted of two singlets for the 2 CH₃ (δ = 1.93 and 2.43), a broad singlet for a OH (δ = 4.95–5.50, exchangeable by D₂O), and two multiplets for the aromatic protons [δ = 7.18–7.61 (m, 2H, arom) and δ = 7.63–8.02 (m, 2H, arom)]. The ¹H decoupled ¹³C NMR spectrum of **4a** showed 12 distinct resonances, partial assignment of these resonances is given in the Experimental section. The ¹H and ¹³C NMR spectra of compounds **4b–f** were similar to those of **4a**, except for the aromatic or heteroaromatic moieties, which exhibited characteristic signals with appropriate chemical shifts.

A mechanistic rationalization for this reaction is provided in Scheme 2. On the basis of the chemistry of isocyanides,^{19–25} it is reasonable to assume that the first step may involve nucleophilic addition of the (*N*-isocyanimino)triphenylphosphorane **3** to the 1,1,1-trifluoroacetone **2**, which facilitates by its protonation with the acid **1**, leading to nitrilium intermediate **6**. This intermediate may be attacked by conjugate base of the acid **7** to form 1:1:1 adduct **8**. This adduct may undergo intramolecular aza-Wittig^{25–29} reaction



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

of the iminophosphorane moiety with the ester carbonyl to afford the isolated sterically congested 2,5-disubstituted 1,3,4-oxadiazoles **4** by removal of triphenylphosphine oxide **5** from intermediate **9**.

We believe that the reported method offers a mild, simple, and efficient route for the preparation of sterically congested 2,5-disubstituted 1,3,4-oxadiazole derivatives. Its ease of workup, high yields, and fairly mild reaction conditions make 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 (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 and are uncorrected. IR spectra were measured on a Jasco 6300 FTIR spectrometer. ^1H and ^{13}C NMR spectra were measured (CDCl_3 solution) 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-MATT 8430 mass spectrometer operating at an ionization potential of 20 eV. Flash chromatography columns were prepared from Merck silica gel powder.

General Procedure for the Preparation of Compounds 4a–f (Exemplified for 4a)

To a magnetically stirred solution of (N-isocyanimino)triphenylphosphorane **3** (1 mmol) and 1,1,1-trifluoroacetone **2** (1 mmol) in CH_2Cl_2 (7 mL), a solution of 3-methylbenzoic acid **1a** (1 mmol) in CH_2Cl_2 (5 mL) was added dropwise at room temperature over 15 min. The mixture was stirred for 4 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 (2:1)]. The solvent was removed under reduced pressure, and the products were obtained. The characterization data of the compounds are given below:

1,1,1-Trifluoro-2-[5-(3-methylphenyl)-1,3,4-oxadiazol-2-yl]-2-propanol (4a)

Yellow crystals; mp: 60.1–61.6°C; Yield: 88%. IR (KBr) (ν_{\max} , cm^{-1}): 3292 (br), 1692, 1560, 1162, 1088, 724. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.93 and 2.42 (2 s, 6 H, 2 CH_3), 4.95–5.50 (br. s, 1 H, OH; exchanged by D_2O addition), 7.18–7.61 (m, 2 H, arom), 7.63–8.02 (m, 2 H, arom). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 21.19 and 22.27 (2 CH_3), 72.93 (q, $^2J_{\text{CF}} = 32.1$ Hz, C- CF_3), 123.65 and 139.23 (2 C, arom), 124.64 (q, $^1J_{\text{CF}} = 286.2$ Hz, CF_3), 125.34, 128.63, 130.07 and 134.32 (4 CH, arom), 164.99 and 167.54 (2 C=N). Anal. Calcd for $\text{C}_{12}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_2$ (272.2): C, 52.94; H, 4.07; N, 10.29%. Found: C, 52.99; H, 4.04; N, 10.32. MS (EI): m/z (%) = 272 (M^+ , 48), 203 (48), 161 (48), 159 (50), 136 (32), 119 (50), 91 (100), 77 (11), 65 (21) and 43 (28).

1,1,1-Trifluoro-2-[5-(1-naphthyl)-1,3,4-oxadiazol-2-yl]-2-propanol (4b)

White crystals; mp: 149.2–151.0°C; Yield: 86%. IR (KBr) (ν_{\max} , cm^{-1}): 3472 (br), 1540, 1165, 1103, 772. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.99 (s, 3 H, CH_3), 4.17 (s, 1 H, OH), 7.52–7.78 (m, 3 H, arom), 7.95 (d, $^3J_{\text{hh}} = 8.0$ Hz, 1 H, arom), 8.08 (d, $^3J_{\text{hh}} = 8.0$ Hz, 1 H, arom), 8.18 (d, $^3J_{\text{hh}} = 7.2$ Hz, 1 H, arom), 9.13 (d, $^3J_{\text{hh}} = 8.5$ Hz, 1 H, arom). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 20.46 (CH_3), 72.38 (q, $^2J_{\text{CF}} = 32.1$ Hz, C- CF_3), 119.89, 130.32 and 134.16 (3 C, arom), 124.01 (q, $^1J_{\text{CF}} = 286.2$ Hz, CF_3), 125.16, 126.15, 127.26, 128.83, 129.14, 129.27 and 133.69 (7 CH, arom), 163.63 and 166.87 (2 C=N). Anal. Calcd for $\text{C}_{15}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_2$ (308.2): C, 58.45; H, 3.60; N, 9.09%. Found: C, 58.41; H, 3.62; N, 9.03. MS (EI): m/z (%) = 308 (M^+ , 22), 239 (4), 197 (14), 155 (50), 153 (100), 126 (75), 76 (16), 63 (18) and 43 (62).

1,1,1-Trifluoro-2-[5-(2-naphthyl)-1,3,4-oxadiazol-2-yl]-2-propanol (4c)

Yellow crystals; mp: 138.5–140.2°C; Yield: 83%. IR (KBr) (ν_{\max} , cm^{-1}): 3274 (br), 1556, 1155, 1096, 763. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.99 (s, 3 H, CH_3), 4.94–5.18 (br. s, 1 H, OH; exchanged by D_2O addition), 7.47–7.67 (m, 2 H, arom), 7.74–7.97 (m, 3 H, arom), 8.07 (d, $^3J_{\text{hh}} = 8.7$ Hz, 1 H, arom), 8.52 (s, 1 H, arom). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 21.27 (CH_3), 72.99 (q, $^2J_{\text{CF}} = 32.1$ Hz, C- CF_3), 120.99, 133.63 and 135.91 (3 C, arom), 123.96, 128.26, 128.96, 129.37, 129.90 and 130.19 (7 CH, arom), 124.69 (q, $^1J_{\text{CF}} = 286.2$ Hz, CF_3), 165.04 and 167.61 (2 C=N). Anal. Calcd for $\text{C}_{15}\text{H}_{11}\text{F}_3\text{N}_2\text{O}_2$ (308.2): C, 58.45; H, 3.60; N, 9.09%. Found: C, 58.51; H, 3.62; N, 9.05. MS (EI): m/z (%) = 308 (M^+ , 55), 239 (12), 197 (29), 155 (100), 153 (84), 127 (82), 100 (11), 77 (11), 63 (10) and 43 (39).

1,1,1-Trifluoro-2-[5-(2-furyl)-1,3,4-oxadiazol-2-yl]-2-propanol (4d)

Yellow crystals; mp: 120.7 (dec) °C; Yield: 87%. IR (KBr) (ν_{\max} , cm^{-1}): 3245 (br), 1642, 1526, 1155, 1096, 751. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.92 (s, 3 H, CH_3), 4.51 (s, 1 H, OH; exchanged by D_2O addition), 6.62 (s, 1 H, furan), 7.25 (s, 1 H, furan), 7.66 (s, 1 H, furan). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 20.39 (CH_3), 72.27 (q, $^2J_{\text{CF}} = 32.1$ Hz, C- CF_3), 112.71, 115.76 and 146.73 (3 CH, furan), 123.86 (q, $^1J_{\text{CF}} = 286.2$ Hz, CF_3), 138.78 (C, furan), 159.42 and 163.38 (2 C=N). Anal. Calcd for $\text{C}_9\text{H}_7\text{F}_3\text{N}_2\text{O}_3$ (248.2): C, 43.56; H, 2.84; N, 11.29%. Found: C, 43.62; H, 2.86; N, 11.24. MS (EI): m/z (%) = 248 (M^+ , 69),

194 (32), 179 (91), 137 (100), 108 (39), 95 (99), 52 (31) and 43 (69). Sample NMR, IR, and mass spectra for **4d** are shown in Figures S1–S6 (Supplemental Materials, available online).

1,1,1-Trifluoro-2-[5-(2-thienyl)-1,3,4-oxadiazol-2-yl]-2-propanol (**4e**)

Yellow crystals; mp: 94.2–96.1 °C; Yield: 80%. IR (KBr) (ν_{\max} , cm^{-1}): 3237 (br), 1603, 1163, 1108, 727. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.92 (s, 3 H, CH_3), 4.41–4.61 (br s, 1 H, OH; exchanged by D_2O addition), 7.14–7.24 (m, 1 H, thiophene), 7.60 (d, $^3J_{\text{hh}} = 4.2$ Hz, 1 H, thiophene), 7.81 (s, 1 H, thiophene). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 21.11 (CH_3), 72.89 (q, $^2J_{\text{cf}} = 32.1$ Hz, C- CF_3), 124.54 (q, $^1J_{\text{cf}} = 286.2$ Hz, CF_3), 124.92 (C, thiophene), 129.30, 131.88 and 132.20 (3 CH, thiophene), 163.61 and 164.17 (2 C=N). Anal. Calcd for $\text{C}_9\text{H}_7\text{F}_3\text{N}_2\text{O}_2\text{S}$ (264.2): C, 40.91; H, 2.67; N, 10.60%. Found: C, 40.99; H, 2.72; N, 10.56. MS (EI): m/z (%) = 264 (M^+ , 4), 195 (4), 153 (7), 124 (8), 111 (100), 96 (7), 82 (19), 69 (24) and 42 (32).

2-[5-(2-Bromophenyl)-1,3,4-oxadiazol-2-yl]-1,1,1-trifluoro-2-propanol (**4f**)

Yellow crystals; mp: 83.5–85.1 °C; Yield: 82%. IR (KBr) (ν_{\max} , cm^{-1}): 3431 (br), 1456, 1167, 1089, 728. ^1H NMR (CDCl_3 , 250 MHz): δ_{H} 1.93 (s, 3 H, CH_3), 4.08–4.32 (br s, 1 H, OH; exchanged by D_2O addition), 7.35–8.02 (m, 4 H, arom). ^{13}C NMR (CDCl_3 , 62.5 MHz): δ_{C} 20.36 (CH_3), 72.36 (q, $^2J_{\text{cf}} = 32.1$ Hz, C- CF_3), 122.15 and 124.76 (2 C, arom), 123.90 (q, $^1J_{\text{cf}} = 286.2$ Hz, CF_3), 128.06, 132.23, 133.48 and 134.97 (4 CH, arom), 164.62 and 165.78 (2 C=N). Anal. Calcd for $\text{C}_{11}\text{H}_8\text{BrF}_3\text{N}_2\text{O}_2$ (337.1): C, 39.19; H, 2.39; N, 8.31%. Found: C, 39.22; H, 2.44; N, 8.22. MS (EI): m/z (%) = 338 ($[\text{M}^+ + 2]$, 8), 336 (M^+ , 7), 304 (6), 269 (10), 225 (31), 183 (58), 155 (29), 102 (22), 89 (36), 75 (100), 62 (35) and 43 (100).

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