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Microwave-Promoted Rapid Synthesis of 1-Aryl-1, 2, 3-Triazoles

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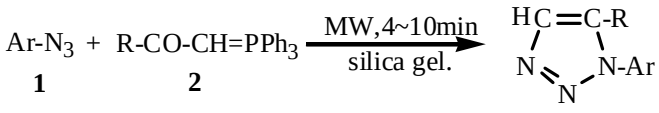
Abstract: Aryl azides and α-keto phosphorus ylides were reacted within 4~10 minutes with silica gel support, under microwave irridiation to afford corresponding 1-aryl-1, 2, 3-triazoles in moderate to good yields.

Keywords: Microwave, triazole.

1, 2, 4- and 1, 2, 3-triazole derivatives have found wide applications as plant growth regulators[1], bactericides and medical fungicides[2], insecticides[3] and in dyeing and color development[4].

One of the general routes of synthesis of 1-aryl-1, 2, 3-triazoles was to treat aryl azides with α-keto phosphorus ylides in dry refluxing benzene for 0.5 to 2 days[5]. Herein we report, for the first time, a very quick and simple, microwave-promoted synthesis of 1-aryl-1, 2, 3-triazoles **3** from aryl azides **1** and α-keto phosphorus ylides **2** on the silica gel support.

The results showed that, using microwave irridiation for 4 to 10 minutes, the reaction completed in moderate to good yields.



1 **2** **3**

	R	Ar
a.	CH_3	C_6H_5
b.	C_6H_5	C_6H_5
c.	*p*-NO_2-C_6H_4	C_6H_5
d.	CH_3	*p*-NO_2-C_6H_4
e.	C_6H_5	*p*-NO_2-C_6H_4
f.	*p*-NO_2-C_6H_4	*p*-NO_2-C_6H_4

Experimental

Typical procedure: A mixture of phenyl azide (1 mmol), triphenylacetomethylene-phosphorane (1 mmol), and silica gel (200 mesh, 2 g) was introduced into the domestic microwave oven in an open container, and microwave irridiation was carried out at an

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output of about 400 W for 4 minutes. Then methylene dichloride (30 mL) was added into the cooled mixture, the extractive was evaporated in *vacuo* to remove the solvent and the residue was purified by a silica gel column chromatography eluting with benzene/ethyl acetate (8/1,V/V) to give **3a**.

3a, 5-methyl-1-phenyl-1, 2, 3-triazole, pale yellow solid, 82%, mp 59-61°C (lit.[5] 60-62°C), IR (KBr, cm^{-1}): 1600,1500,1415 (aromaticC=C); 1225 (triazole), ^{1}HNMR ($CDCl_3$, δ ppm): 2.39 (s, 3H), 7.47-7.57 (m, 5H), 7.6695 (s, 1H), MS (EI, *m/z*): 159 (M^+, 21), 130 (100), 103 (22), 89 (4), 77 (81).

3b, 1,5-diphenyl-1, 2, 3-triazole, pale yellow solid, 75% (1it.[6] 80%), mp 113-115°C (lit.[6] 116-117°C), IR (KBr, cm^{-1}): 1600,1500,1450 (aromaticC=C); 1230 (triazole), ^{1}HNMR ($CDCl_3$, δ ppm): 7.22-7.47 (m, 10H), 7.94 (s, 1H), MS (EI, *m/z*): 221 (M^+, 19), 193 (100), 165 (53), 116 (44), 89 (53), 77 (69).

3c, 1-phenyl-5-(4-nitrophenyl)-1, 2, 3-triazole, yellow solid, 68% (1it.[6] 67%), mp 149.5-150.5°C (lit.[6] 150-151°C), IR (KBr, cm^{-1}): 1600,1520,1460 (aromaticC=C); 1345 (NO_2); 1245 (triazole), ^{1}HNMR ($CDCl_3$, δppm): 7.27-7.52 (m, 7H), 8.00 (s,1H), 8.21-8.23 (d, 2H, J=8.0Hz), MS (EI, *m/z*): 266 (M^+, 20), 238 (70), 191 (60), 89 (50), 77 (100).

3d, 5-methyl-1-(4-nitrophenyl)-1, 2, 3-triazole, yellow solid, 78% (1it.[6] 73%), mp 139-141°C (1it.[6] 139-140.5°C), IR (KBr, cm^{-1}): 1600, 1525, 1400 (aromaticC=C); 1345 (NO_2); 1245 (triazole), ^{1}HNMR ($CDCl_3$, δ ppm): 2.48 (s, 3H), 7.70 (s, 1H), 7.76-7.78 (d, 2H, J=8.0Hz), 8.45-8.47 (d, 2H, J=8.0Hz), MS (EI, *m/z*): 204 (M^+, 32), 175 (100), 129 (72), 103 (38), 90 (10), 76 (66).

3e, 5-phenyl-1-(4-nitrophenyl)-1,2,3-triazole, yellow solid, 79% (1it.[6] 77%), mp 159.5-161.5°C (1it.[6] 162.5-164℃), IR (KBr, cm^{-1}): 1600,1520,1450 (aromaticC=C); 1350 (NO_2); 1250 (triazole), ^{1}HNMR ($CDCl_3$, δ ppm): 7.24-7.61 (m, 7H), 7.91 (s, 1H), 8.30-8.32 (d, 2H, J=8.0Hz), MS (EI, *m/z*): 266 (M^+, 22), 238 (100), 192 (87), 116 (47), 89 (46), 76 (44).

3f, 1,5-bis(4-nitrophenyl)-1, 2, 3-triazole, yellow solid, 90% (1it.[6] 98%), mp 201-202°C (1it.[6] 200.5-202°C), IR (KBr, cm^{-1}): 1600,1510,1450 (aromaticC=C); 1345 (NO_2); 1240 (triazole), ^{1}HNMR ($CDCl_3$, δ ppm): 7.45-7.47 (d, 2H, J=8.0Hz), 7.58-7.60 (d, 2H, J=8.0Hz), 8.02 (s,1H), 8.28-8.30 (d, 2H, J=8.0 Hz), 8.36-8.38 (d, 2H, J= 8.0Hz), MS (EI, *m/z*): 311 (M^+, 19), 283 (100), 237 (42), 190 (71), 89 (53), 76 (49).

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