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Synthesis of 2,3-Dihydro-4-pyridones Using $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2 as an Efficient Heterogeneous Catalyst

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A simple and efficient synthesis of 2,3-dihydro-4-pyridones has been accomplished by three-component condensation reactions of Danishefsky's diene with aldehydes and amines in the presence of $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2.

Key Words: 2,3-Dihydro-4-pyridones, $HClO_4$-SiO_2, $NaHSO_4$-SiO_2, Danishefsky's diene.

INTRODUCTION

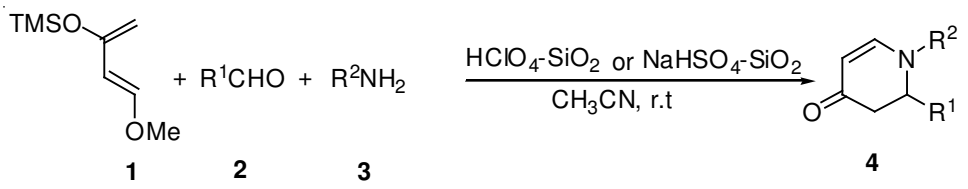
2,3-Dihydro-4-pyridones have become important intermediates for the synthesis of various alkaloids and bioactive compounds[1]. Aza-Diels-Alder reaction is one of the most straightforward approach for the synthesis of 2,3-dihydro-4-pyridones. This method involves Lewis acids[2] or protonic acid[3] catalyzed three-component condensation reactions of Danishefsky's diene with aldehydes and amines or two-component condensation reactions of Danishefsky's diene with imine. Recently, heterogeneous solid acid catalysts[4] have been shown to be effective for the synthesis of 2,3-dihydro-4-pyridones. However, most of these procedures have signicant drawbacks such as long reaction times, low yields, harsh reaction conditions, difficult work-up and use of environmentally toxic reagents or media. Thus, there is still need of a simple and general procedure for synthesis of 2,3-dihydro-4-pyridones under mild conditions.

In recent years, the use of heterogeneous catalysts has received considerable interest in various disciplines including organic synthesis. They are advantageous over their homogeneous counterparts due to the prime advantage that in most of the cases the catalyst can be recovered easily and reused[5]. Silica-supported perchloric acid ($HClO_4$-SiO_2) and silica-supported sodium hydrogen sulfate ($NaHSO_4$-SiO_2) have been used as efficient heterogeneous catalysts for many organic transformations because of their low cost, ease of preparation, catalyst recycling and ease of handling[6,7]. We now report a simple and efficient route for synthesis of 2,3-dihydro-4-pyridones using $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2 as an efficient and environmentally benign catalyst (**Scheme-I**).

EXPERIMENTAL

NMR Spectra were determined on Bruker AV-300 spectrometer at room temperature using TMS as internal standard, coupling constants (*J*) were measured in

Hz; Elemental analysis were performed by a Vario-III elemental analyzer, melting points were determined on a XT-4 binocular microscope and were uncorrected. The commercially available reagents were used throughout without further purification unless otherwise stated.



Scheme-I

General procedure for the preparation of 4: The aldehyde (1 mmol), amine (1 mmol) and Danishefsky's diene (2 mmol) was added to 10 mL of CH_3CN sequentially. After $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2 (0.1 mmol, 10 mol %) was introduced. The mixture was stirred vigorously at room temperature for an appropriate time (Table-1). After completion of the reaction (TLC), 20 mL diethyl ether was added to the reaction mixture and the catalyst was recovered by filtered. The organic layer was washed with 1 M HCl, brine, dried over $MgSO_4$. The solvent was evaporated and puried by column chromatography on silica gel using EtOAc-petroleum ether (1:1) to afford pure 2,3-dihydro-4-pyridones in 68-91 % yields.

1,2-Diphenyl-2,3-dihydropyridin-4(1*H*)-one (4a): White soild, m.p. 48-49 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.70 (dd, *J* = 1.0, 7.6 Hz, 1H), 7.40-6.95 (m, 10H), 5.36-5.25 (m, 2H), 3.32 (dd, *J* = 7.3, 16.6 Hz, 1H), 2.76 (ddd, *J* = 1.1, 2.8, 16.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.6, 147.2, 145.1, 137.2, 130.4, 128.0, 125.6, 123.9, 118.6, 104.2, 62.2, 43.4; anal. calcd. (%) for $C_{17}H_{15}NO$: C 81.90, H 6.06, N 5.62; found (%): C 81.78, H 6.14, N 5.59.

1-(4-Methoxyphenyl)-2-phenyl-2,3-dihydropyridin-4(1*H*)-one (4b): White soild, m.p. 105-106 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.68 (d, *J* = 7.9 Hz, 1H), 7.36-6.98 (m, 8H), 6.88-6.82 (m, 1H), 5.32-5.25 (m, 2H), 3.80 (s, 3H), 3.29 (dd, *J* = 7.0, 16.4 Hz, 1H), 2.74 (dd, *J* = 3.0, 16.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 191.0, 150.1, 146.8, 144.8, 136.2, 129.5, 126.0, 125.8, 117.8, 115.2, 102.6, 63.8, 57.8, 43.2; anal. calcd. (%) for $C_{18}H_{17}NO_2$: C 77.40, H 6.13, N 5.01; found (%): C 77.56, H 6.18, N 4.92.

1-(2-Methoxyphenyl)-2-phenyl-2,3-dihydropyridin-4(1*H*)-one (4c): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.23-7.05 (m, 7H), 6.92 (dd, *J* = 1.4, 5.0 Hz, 2H), 6.79-6.65 (m, 1H), 5.22-5.11 (m, 2H), 3.78 (s, 3H), 3.02 (dd, *J* = 6.5, 16.2 Hz, 1H), 2.72 (dd, J = 8.2, 16.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 191.0, 155.4, 153.9, 141.2, 133.6, 128.8, 128.5, 128.2, 127.6, 127.2, 120.8, 111.2, 102.4, 64.1, 56.8, 44.0; anal. calcd. (%) for $C_{18}H_{17}NO_2$: C 77.40, H 6.13, N 5.01; found (%): C 77.48, H 6.10, N 4.98.

TABLE-1
PREPARATION OF 2,3-DIHYDRO-4-PYRIDONES
CATALYZED BY $HClO_4$-SiO_2 AND $NaHSO_4$-SiO_2[a]

Entry	R^1	R^2	Catalyst[b]	Time (h)	Yield (%)
a	Ph	Ph	i	1.5	89 (86, 87, 85)[c]
			ii	1.5	86 (85, 83, 81)[c]
b	Ph	4-(MeO)C_6H_4	i	1.5	85
			ii	1.5	82
c	Ph	2-(MeO)C_6H_4	i	2.0	72
			ii	2.0	70
d	Ph	4-(Cl)C_6H_4	i	1.5	78
			ii	1.5	79
e	Ph	3-(Br)C_6H_4	i	2.0	70
			ii	2.0	68
f	4-(MeO)C_6H_4	Ph	i	1.5	89
			ii	1.5	91
g	4-(Cl)C_6H_4	Ph	i	1.5	81
			ii	1.5	79
h	4-(F)C_6H_4	Ph	i	1.5	83
			ii	1.5	80
i	4-(NO_2)C_6H_4	Ph	i	2.0	88
			ii	2.0	86
j	2-pyridyl	Ph	i	2.5	74
			ii	2.5	71
k	4-(Cl)C_6H_4	4-(MeO)C_6H_4	i	2.5	74
			ii	2.5	72
l	PhCH=CH	Ph	i	2.0	88
			ii	2.0	84
m	$PhCH_2CH_2$	Ph	i	2.0	89
			ii	2.0	86
n	$(CH_3)_2CH$	Ph	i	2.5	73
			ii	2.5	72

[a]All of the isolated products except **4e**, **4h** are known compounds and their spectra and physical data have been reported in the literature 8. [b]Catalyst (i) $HClO_4$-SiO_2 (10 mol %), (ii) $NaHSO_4$-SiO_2 (10 mol %). [c]Isolated yields after recycling of catalyst.

1-(4-Chlorophenyl)-2-phenyl-2,3-dihydropyridin-4(1*H*)-one (4d): White soild, m.p. 144-145 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.68 (d, *J* = 7.6 Hz, 1H), 7.28-7.15 (m, 7H), 6.90-6.84 (m, 2H), 5.25-5.22 (m, 2H), 3.18 (dd, *J* = 6.7, 16.7 Hz, 1H), 2.88-2.79 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.6, 148.2, 145.1, 136.9, 130.6, 129.8, 129.6, 128.8, 125.8, 120.16, 102.8, 63.6, 44.2; anal. calcd. (%) for $C_{17}H_{14}NOCl$: C 71.96, H 4.97, N 4.94; found (%): C 72.11, H 4.89, N 4.98.

1-(3-Bromophenyl)-2-phenyl-2,3-dihydropyridin-4(1*H*)-one (4e): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.68 (dd, *J* = 1.1, 7.8 Hz, 1H), 7.37-7.22 (m, 6H), 7.02-6.81 (m, 3H), 5.31 (dd, *J* = 1.0, 7.4 Hz, 1H), 5.25 (dd, *J* = 3.0, 7.4 Hz, 1H), 3.32 (dd, *J* = 7.1, 16.7 Hz, 1H), 2.77 (ddd, *J* = 1.2, 2.8, 16.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 191.2, 160.4, 146.5, 146.1, 136.0, 131.2, 128.7, 127.6, 126.3, 118.6, 116.9, 106.3, 103.2, 62.0, 42.2; anal. calcd. (%) for $C_{17}H_{14}NOBr$: C 62.21, H 4.30, N 4.27; found (%): C 62.45, H 4.41, N 4.19.

2-(4-Methoxyphenyl)-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4f): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.60 (d, *J* = 7.1 Hz, 1H), 7.36-7.05 (m, 7H), 6.90 (d, *J* = 6.6 Hz, 2H), 5.35-5.28 (m, 2H), 3.80 (s, 3H), 3.25 (dd, *J* = 7.0, 16.5 Hz, 1H), 2.78 (dd, *J* = 3.3, 16.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.8, 158.6, 147.9, 144.8, 130.1, 129.8, 126.8, 125.0, 117.9, 115.5, 102.2, 62.4, 44.2; anal. calcd for $C_{18}H_{17}NO_2$: C 77.40, H 6.13, N 5.01; found: C 77.48, H 6.09, N 5.02.

2-(4-Chlorophenyl)-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4g): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.66 (d, *J* = 7.4 Hz, 1H), 7.38-7.12 (m, 7H), 6.98-6.91 (m, 2H), 5.33-5.25 (m, 2H), 3.30 (dd, *J* = 7.0, 16.2 Hz, 1H), 2.76 (dd, *J* = 3.0, 16.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 191.0, 159.8, 146.1, 143.2, 130.5, 129.6, 128.5, 126.6, 125.9, 118.9, 115.8, 112.4, 107.2, 62.8, 44.0; anal. calcd. (%) for $C_{17}H_{14}NOCl$: C 71.96, H 4.97, N 4.94; found (%): C 72.15, H 4.98, N 4.90.

2-(4-Fluorophenyl)-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4h): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ:7.73 (dd, *J* = 1.2, 7.7 Hz, 1H), 7.33-7.14 (m, 7H), 7.06. (d, *J* = 7.8 Hz, 2H), 5.33 (dd, *J* = 1.3, 8.8 Hz, 1H), 5.28 (dd, *J* = 3.6, 7.8 Hz, 1H), 3.38 (dd, *J* = 7.3, 16.7 Hz, 1H), 2.77 (ddd, *J* = 1.2, 3.6, 16.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 192.4, 160.7, 145.2, 142.5, 130.8, 130.0, 128.3, 127.9, 126.8, 119.8, 116.4, 112.7, 108.7, 62.4, 41.9; anal. calcd. (%) for $C_{17}H_{14}NOF$: C 76.39, H 5.28, N 5.24; found (%): C 76.30, H 5.39, N 5.12.

2-(4-Nitrophenyl)-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4i): White soild, mp 152-153 °C; 1H NMR (CDCl3, 300 MHz) d: 8.22 (d, J = 8.6 Hz, 2H), 7.70 (d, J = 7.6 Hz, 2H), 7.42. (d, J = 7.8 Hz, 2H), 7.40-7.30 (m, 2H), 7.22 (t, J = 7.2 Hz, 1H), 7.02 (d, J = 7.6 Hz, 2H), 5.38-5.30 (m, 2H), 3.36 (dd, J = 6.8, 16.2 Hz, 1H), 2.76 (dd, J = 3.2, 16.7 Hz, 1H); 13C NMR (CDCl3, 75 MHz) d: 190.1, 148.7, 146.9, 145.5, 143.8, 130.2, 127.8, 125.1, 124.3, 117.6, 113.6, 104.8, 62.0, 43.1. anal. calcd. (%) for $C_{17}H_{15}N_2O_3$: C 69.38, H 4.79, N 9.52; found (%): C 69.52, H 4. 89, N 9.50.

1-Phenyl-2-(pyridin-2-yl)-2,3-dihydropyridin-4(1*H*)-one (4j): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ:8.60-8.55 (m, 1H), 7.75-7.60 (m, 2H), 7.36-6.95 (m, 7H), 5.38-5.30 (m, 2H), 3.32 (dd, *J* = 7.2, 16.5 Hz, 1H), 3.15-3.08 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 192.2, 153.8, 148.0, 145.1, 143.8, 135.6, 128.6, 120.5, 117.0, 113.2, 102.5, 58.8, 43.2. Anal. calcd. (%) for $C_{16}H_{14}N_2O$: C 76.78, H 5.64, N 11.19; found (%): C 76.58, H 5. 70, N 11.22.

2-(4-Chlorophenyl)-1-(4-methoxyphenyl)-2,3-dihydropyridin-4(1*H*)-one (4k): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.60 (d, *J* = 7.6 Hz, 1H), 7.33-7.20 (m, 4H), 6.98-6.94 (m, 2H), 6.85-6.79 (m, 2H), 5.30-5.18 (m, 2H), 3.78 (s, 3H), 3.25 (dd, *J* = 7.2, 16.4 Hz, 1H), 2.76 (dd, *J* = 3.6, 16.7 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.2, 156.2, 148.9, 137.2, 135.9, 133.2, 129.25, 126.8, 120.7, 115.2, 102.0, 62.2, 54.8, 43.0; anal. calcd. (%) for $C_{18}H_{16}NO_2Cl$: C 68.90, H 5.14, N 4.46; found (%): C 68.96, H 6.18, N 4.50.

1-Phenyl-2-styryl-2,3-dihydropyridin-4(1*H*)-one (4l): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.58 (d, *J* = 7.6 Hz, 1H), 7.38-7.20 (m, 10H), 6.60 (d, *J* = 16.0 Hz, 1H), 6.42 (dd, *J* = 6.0, 16.2 Hz, 1H), 5.25 (d, *J* = 9.2, 1H), 4.85-4.80 (m, 1H), 3.32 (dd, *J* = 9.2, 16.2 Hz, 1H), 2.70 (d, *J* = 16.5 Hz, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ:

190.8, 145.2, 144.8, 135.5, 130.0, 129.6, 128.9, 128.7, 128.2, 125.8, 116.9, 113.2, 105.2, 54.4, 43.2; anal. calcd. (%) for $C_{19}H_{17}NO$: C 82.88, H 6.22, N 5.09; found: C 82.90, H 6.30, N 5.01.

2-Phenethyl-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4m): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.45-7.02 (m, 11H), 5.25 (dd, *J* = 1.0, 7.2 Hz, 1H), 4.25-4.20 (m, 1H), 3.02 (dd, *J* = 7.0, 16.4 Hz, 1H), 2.77-2.52 (m, 2H), 2.42-2.36 (m, 1H), 2.10-2.02 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.2, 144.9, 144.2, 137.9, 130.0, 128.6, 128.2, 125.68, 117.4, 113.0, 101.3, 51.2, 41.4, 33.2, 30.8; Anal. calcd. (%) for $C_{19}H_{19}NO$: C 82.28, H 6.90, N 5.05; found (%): C 82.31, H 6.88, N 5.02.

2-Isopropyl-1-phenyl-2,3-dihydropyridin-4(1*H*)-one (4n): Oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.45-7.36 (m, 3H), 7.26-7.20 (m, 3H), 5.20 (dd, *J* = 1.0, 7.6 Hz, 1H), 4.18-4.10 (m, 1H), 2.98 (dd, *J* = 7.0, 16.5 Hz, 1H), 2.66-2.60 (m, 1H), 2.30-2.24 (m, 1H), 1.02 (d, *J* = 7.0 Hz, 3H), 0.93 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ: 190.4, 144.8, 144.8, 130.2, 118.0, 113.2, 101.6, 62.4, 38.2, 31.0, 20.2; anal. calcd. (%) for $C_{14}H_{17}NO$: C 78.10, H 7.96, N 6.51; found: C 78.12, H 8.02, N 6.55.

RESULTS AND DISCUSSION

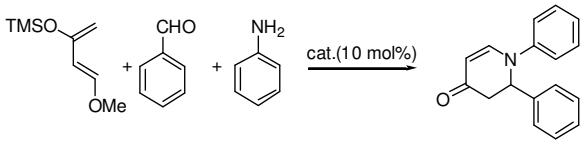
To study this reaction by examining the amount of catalyst for the reaction involving Danishefsky's diene (2 mmol) with benzaldehyde (1 mmol) and aniline (1 mmol) to afford the product 1,2-diphenyl-2,3-dihydropyridin-4(1*H*)-one in CH_3CN at room temperature. The best result was obtained with 10 mol % $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2. Higher amounts of catalyst did not improve the result to any greater extent.

A range of 2,3-dihydro-4-pyridones was synthesized by the reaction of Danishefsky's diene (**1**, 2 mmol) with aldehydes (**2**, 1 mmol) and amines (**3**, 1 mmol). The reaction proceeded at room temperature within 150 min in excellent yields after the addition of the acid catalyst $HClO_4$-SiO_2 or $NaHSO_4$-SiO_2 (Table-1). In these experiments the product was isolated by filtration and the catalyst could be reloaded with fresh reagents for further runs. Thus, recyclization of catalyst is possible without significant loss of activity (Table-1, entry 1). In addition, we noticed also that the yields of the products were almost similar with both of these two catalysts.

In order to show the merit of the presented protocol, we have compared some of the results obtained by the other catalysts such as HBF_4, silica sulfuric acid (SSA), $Yb(OTf)_3$, $Ag(OTf)_3$, which have been reported recently for the reaction of Danishefsky's diene with with benzaldehyde and aniline (Table-2). It revealed that $HClO_4$-SiO_2 and $NaHSO_4$-SiO_2 are equally efficient, cost-effective and environmentally benign catalysts useful in the synthesis of 2,3-dihydro-4-pyridones.

In conclusion, we have developed a simple and highly efficient practical method for synthesis of 2,3-dihydro-4-pyridones using $HClO_4$-SiO_2 and $NaHSO_4$-SiO_2 in CH_3CN. The notable features of this procedure are mild reaction conditions, simple experimental procdure and excellent yields (68-91 %), which make it a useful and

TABLE-2
COMPARISON OF THE EFFECT OF CATALYSTS
IN SYNTHESIS OF 2,3-DIHYDRO-4-PYRIDONES



Entry	Catalysis	Solvent	Temp. (ºC)	Time (min)	Yield (%)	Reference
1	HBF_4	MeOH-H_2O	-40	30	85	**3a**
2	SSA	CH_3CN	r.t.	120	85	**4**
3	Yb(OTf)$_3$	CH_3CN	r.t.	1200	72	**2g**
4	Ag(OTf)$_3$	H_2O	r.t.	180	74	**2e**
5	$HClO_4$-SiO_2	CH_3CN	r.t.	75	89	–
6	$NaHSO_4$-SiO_2	CH_3CN	r.t.	75	86	–

attractive process for the synthesis of 2,3-dihydro-4-pyridones. It is believed that this methodology will be a valuable addition to the existing methods in the field of synthesis of 2,3-dihydro-4-pyridones.

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