



## Efficient solvent-free synthesis of *N*-unsubstituted ketimines from ketones and ammonia on porous solid acids



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### ABSTRACT

We quantitatively synthesized *N*-unsubstituted (*N*-H) diphenylketimine from benzophenone and ammonia without solvent under ambient conditions on some solid acids, especially the proton-exchanged Y-type zeolite (H-Y), by a simple procedure. Similarly, other *N*-unsubstituted ketimines were also obtained in high yields by the reaction of alkyl aryl ketones or a dialkyl ketone having  $\alpha$ -acidic hydrogens with a large excess of ammonia gas at 1 atm. without a solvent with the aid of the zeolite's high dehydration ability and stabilization effects.

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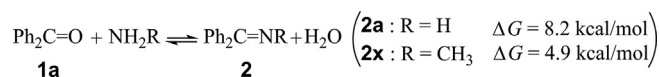
Imine compounds are valuable organic intermediates as not only C-electrophiles, but also *N*-nucleophiles for the syntheses of a variety of organic substances. Although relatively stable *N*-substituted imines are easily formed from the condensation of aldehydes or ketones with primary amines such as benzylamines [1], the *N*-substituents must be removed for the further functionalization on the nitrogen of the imines.[2] On the other hand, the condensation of aldehydes or ketones with ammonia directly produces *N*-unsubstituted imines, which can undergo the functionalization on the carbon or nitrogen. In recent years, not only the nucleophilic addition reactions to *N*-unsubstituted ketimines (*N*-H ketimines: *N*-H imines obtained from ketones) at the carbon site of the imino group,[3] but also alkylations,[4] arylations,[5] cyclizations with azides[6] and alkynes[7] of *N*-H ketimines at the nitrogen site of the imino group have been developed. The synthesis of *N*-H ketimines is, therefore, attracting much attention in terms of synthetic organic chemistry. The *N*-H ketimines are labile and easily hydrolyzed so that their successful condensation of ketones and ammonia has been limited to the use of diaryl or  $\alpha$ -perfluoroalkyl aryl ketones.[3a]

The most frequently-used methods for the formation of *N*-H ketimines are based on the additions of RMgBr or RLi to nitrile compounds.[6,8] Other methods include the denitration[9] of azide compounds catalyzed by a ruthenium complex or a strong

base, and the dehydrogenation[10] from amine compounds with an alumina-supported copper catalyst.

Scheme 1 summarizes the *N*-H ketimines synthesis from ketones, including the aza-Wittig reaction of iminophosphoranes (eq 1),[8c,11] the addition of LiN(TMS)<sub>2</sub> (eq 2),[12] and the recently-reported reactions using (TMS)<sub>2</sub>NH (eq 3,4).[13] It was also reported that a stable *N*-H ketimine was produced from the ketone having an aromatic ring with an *ortho*-hydroxy group or an isatin skeleton (eq 5,6).[14]

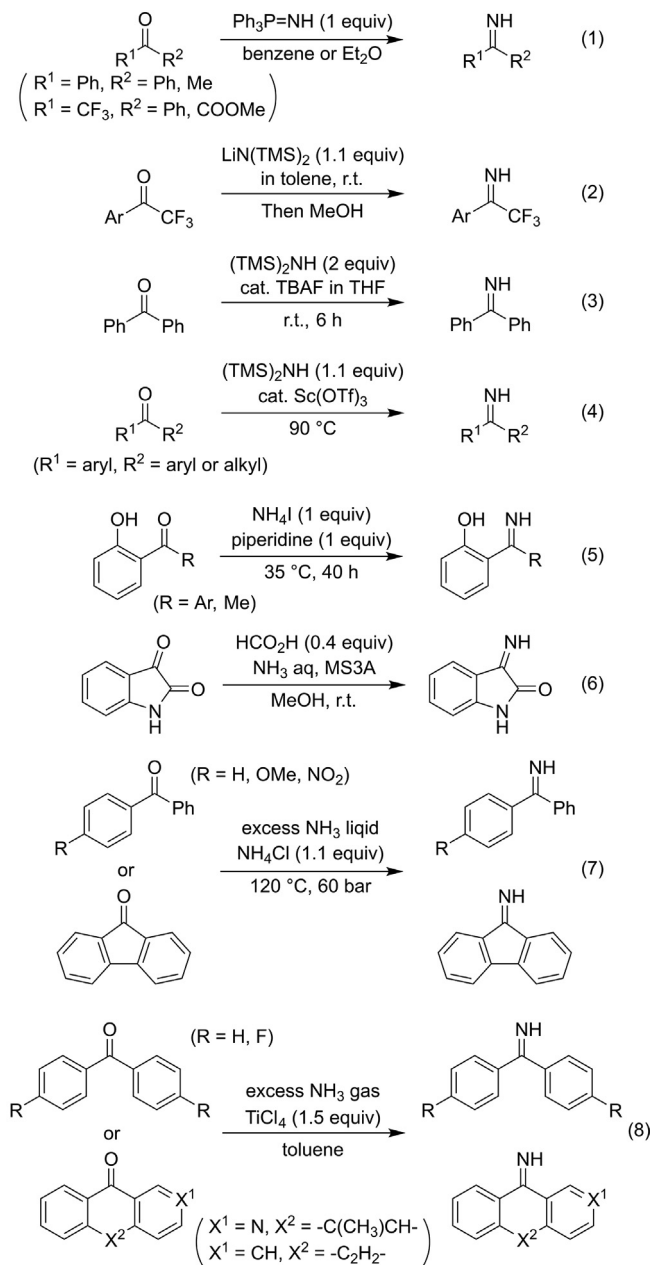
Compared to those previous methods, the simple condensation of ketones with ammonia to produce *N*-H ketimines is the most straightforward with a higher atom economy as well as a lower E-factor. However, this reaction is generally an endothermic reaction.[3a] When we calculated the condensation of benzophenone (1a) with ammonia or methylamine at the level of B3LYP/6-311 + G(2d,p)//B3LYP/6-31 + G(d,p) using Gaussian 16[15], the  $\Delta G_{\text{ammonia}}$  is + 8.2 kcal/mol and  $\Delta G_{\text{methylamine}}$  is + 4.9 kcal/mol at room temperature (RT), respectively, indicating that the formation of *N*-H ketimine (2a) is much less favorable than that of *N*-CH<sub>3</sub> ketimine (2x).



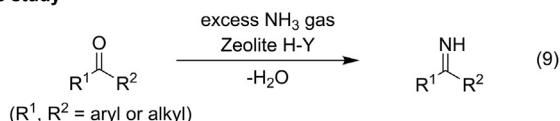
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## Previous studies



## This study



**Scheme 1.** List of previous studies and the present study for the synthesis of *N*–*H* ketimines from ketones.

This is the reason why only a limited number of studies have been reported, including the reaction under high-temperature and high-pressure conditions using ammonium chloride and an excess amount of ammonia (eq 7),<sup>[16]</sup> and the reaction with excess ammonia in the presence of titanium tetrachloride (eq 8).<sup>[17]</sup> In order to effectively synthesize *N*–*H* ketimines from ketones and ammonia, it is necessary to promote the reaction as well as to shift the chemical equilibrium to the product side. Therefore, we made the best use of various solid acids, especially the zeolite (eq 9).

We have shown that labile organic molecules are stably captured and retained in the unique pores of the zeolite. For instance, short-chain aldehydes, such as formaldehyde,<sup>[18a–c]</sup> acrolein<sup>[18d, e]</sup> and propargyl aldehyde,<sup>[18f]</sup> and cyclopentadiene<sup>[19]</sup> that were easily polymerizable, ethyl diazoacetate<sup>[20]</sup> that is easily decomposed by acids, and a diphenylmethyl cation<sup>[21]</sup> produced by dehydration from benzhydrol were preserved in the zeolite pores for a long time even at RT. At the same time, these preserved molecules were also activated to react with some nucleophiles which had been subsequently added. In a series of our research, we now report the efficient, solvent-free synthesis of *N*–*H* ketimines from ketones and ammonia gas on the surface of porous solid acids under mild conditions.

**General experimental procedure:** A solid acid, whose weight was 10 times the weight of the ketone **1**, in a 30-mL flask was activated under vacuum (<0.1 Torr) at 400 or 120 °C for 2 h, then cooled in an ice bath. The flask including the activated solid was connected to a balloon of NH<sub>3</sub> gas (2.5 L), flushed with ammonia, and a solid or liquid ketone reactant **1** (1.5 mmol) was then introduced.

The flask was once evacuated, and then filled with the ammonia gas at 0 °C with stirring for 30 min. The solid mixture was stirred for a certain time at a specified temperature. As a work-up, dry methanol (100 mL) was added to extract the products from the porous solid followed by suction filtration. The methanol extract was concentrated, and dry ether was added. The resulting suspension was passed through a celite pad with dry ether (30 mL). The filtrate was condensed, and a reference substance was added, and the yields of **1** and **2** were quantified by <sup>1</sup>H NMR in CDCl<sub>3</sub> or THF-*d*<sup>8</sup>.

First, the formation of diphenylketimine **2a** from benzophenone **1a** with ammonia was examined using various solid acids such as zeolite, silica and alumina (Table 1). Among the solid acids, the

**Table 1**  
 Synthesis of diphenylketimine using various solid acids<sup>a</sup>

Entry	Solid acid	Yield of <b>2a</b> (%) <sup>b</sup>	Recovery of <b>1a</b> (%) <sup>b</sup>
1	MS3A	n.d.	quant.
2	Na-X	9	10
3	Na-Y	50	49
4	NaY + MS3A	71	26
5	Ag-Y	59	32
6	H-Y (1) <sup>c</sup>	quant.	n.d.
7	H-Y (2) <sup>c</sup>	89	10
8	H-Y (4) <sup>c</sup>	67	32
9	H-Y (40) <sup>c</sup>	12	87
10	H-Beta	87	12
11	H-Mor	39	35
12	H-ZSM-5	56	28
13	SiO <sub>2</sub> , neutral	80	19
14	SiO <sub>2</sub> , acidic	73	26
15 <sup>d</sup>	CARIACT Q-3	86	12
16	Al <sub>2</sub> O <sub>3</sub> , neutral	86	10
17	Al <sub>2</sub> O <sub>3</sub> , acidic	77	21
18 <sup>e</sup>	K10	40	59
19 <sup>f</sup>	NH <sub>4</sub> Cl	n.d.	quant.

<sup>a</sup> 8 ~ 12 wt% of **1a** (0.8 ~ 1.8 mmol) for the solid acid and 90 mmol of NH<sub>3</sub> gas were employed.

<sup>b</sup> <sup>1</sup>H NMR yields based on the ratios of **1a** to **2a**.

<sup>c</sup> The figures in parentheses indicate the number of adsorbed molecules per superpage of H-Y.

<sup>d</sup> CARIACT Q-3 is porous SiO<sub>2</sub>.

<sup>e</sup> Activated at 120 °C.

<sup>f</sup> More than 100 eq of NH<sub>4</sub>Cl were used.

proton-exchanged Y-type zeolite (Si/Al = 2.75; abbreviated as H-Y) gave the best result.

For the results of the H-Y zeolite, the number  $n$  in H-Y( $n$ ) indicates that  $n$  molecules of **1a** per supercage of H-Y were used. As the  $n$  increased, the yield of **2a** clearly decreased (entries 6–9): H-Y (1) was the H-Y zeolite of choice. Hereafter, H-Y stands for H-Y (1) in this paper. H-Beta zeolite also gave a high yield, but H-Mor and H-ZSM-5 zeolite were not suitable for the reaction (entries 10–12). Among a series of the H-zeolite, this trend in the yields does not simply correlate with the solid acidity: H-Mor > H-ZSM > H-Beta > H-Y,[22] indicating that the effective formation of **2a** is not directly dependent on the solid acidity.

Among the metal-ion exchanged zeolite, such as potassium A-type zeolite (MS3A), sodium X- and Y-type zeolite (Na-X, Na-Y) and silver Y-type zeolite (Ag-Y), Na-Y and Ag-Y[23] gave 50 and 59% yields of **2a**, respectively (entries 3, 5), while the yield dropped to 9% together with a low recovery of **1a** (10%) on Na-X which has the same zeolite framework as Na-Y (entry 2). MS3A has very small cavities of 0.3 nm, which are too small for **1a** to be allowed to react with NH<sub>3</sub>, and **1a** was completely recovered (entry 1). Interestingly, co-adding the same amount of MS3A as Na-Y caused an increase in the yield of **2a** by ca. 20% (entry 4). This is probably due to the powerful dehydration effect of MS3A on the condensation.

By using the neutral and acidic silica, **2a** was obtained in 80% and 73% yields, respectively, with some **1a** remaining (entries 13,14). In addition, CARICT Q-3, which is a commercially- available mesoporous silica with a large surface area, showed a better yield of 86% than the normal silica (entry 15). Neutral as well as acidic alumina also promoted the reaction in good yields of 86% and 77%, respectively (entries 16, 17). The reaction using K10, which is a commercially-available acid-treated clay mineral, proceeded in only 40% yield (entry 18). With an excess amount of ammonium chloride, no reaction took place (entry 19).

Next, for the reaction of **1a** with ammonia using the best promoter, H-Y, the reaction temperature and time were optimized (Table 2). When the reaction time was set at 16 h, and the temperature was changed from RT to 60 °C, it was found that the reaction was completed even at RT (entries 1–3). When the reaction temperature was fixed at 40 °C, **2a** was quantitatively obtained in ca. 3 h (entries 4, 5).

Note that **1a** is a solid material at RT. In order to confirm that **1a** was completely incorporated into the zeolite pores simply by mechanically mixing the solid materials of H-Y and **1a** at RT for 30 min in the air, we compared the solution NMR spectrum of **1a** in CDCl<sub>3</sub> (Fig. 1(a)) with the <sup>13</sup>C DD/MAS NMR spectrum (Fig. 1(b)) of the solid mixture, where one molecule of **1a** was added to one supercage of H-Y, which is expressed here as **1a@H-Y**.

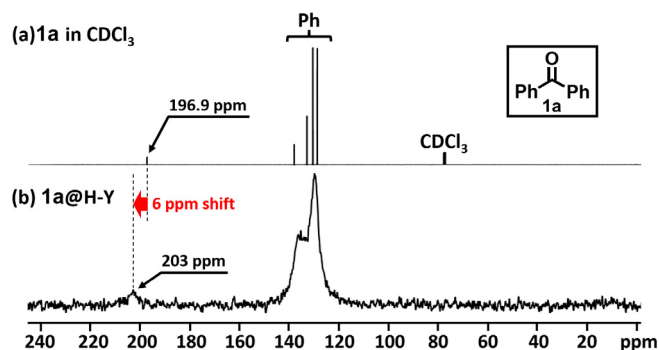


Fig. 1. <sup>13</sup>C NMR spectrum of **1a** in CDCl<sub>3</sub> (a), and <sup>13</sup>C DD/MAS NMR spectrum of **1a@H-Y** (b).

In the solution NMR, the carbonyl carbon of **1a** appeared at 196.9 ppm. On the other hand, in the solid-state NMR, that of **1a@H-Y** was observed at 203 ppm, which was shifted by 6 ppm lower than that in CDCl<sub>3</sub> together with no peak of **1a**. From this, it can be said that the solid ketone material is completely occluded into the H-Y pores only by simple mechanical mixing for such a short period of time.

We previously observed that the carbonyl carbons of formaldehyde, acrolein, propargyl aldehyde and diphenylketene adsorbed on the sodium ion sites in Na-Y showed the lower magnetic field shifts of <sup>13</sup>C DD/MAS NMR by 5 to 6 ppm than those in solution. [18,23] It can be assessed that **1a** is also coordinated to the proton in the supercage of H-Y, demonstrating the same down-field shift of the carbonyl carbon as shown in Fig. 1. It is apparent that the protonated **1a** in the H-Y was so activated that the addition of ammonia and the successive dehydration to the N–H ketimine **2a** were efficiently promoted. It should be also considered that the zeolite's high dehydration ability[24] and stabilization effects [18] on the labile ketimines caused the chemical equilibrium shift to the ketimine formation even though it is intrinsically unfavored.

When the intact **1a** remains together with **2a**, for example, **2a/1a** = 8/2, in the reaction mixture, it is necessary to separate **2a** from **1a**. As the boiling points of **1a** and **2a** are close to each other, it was difficult to purify **2a** by simple distillation. Our trial to purify the mixture on an open column using neutral silica gel and an eluent system of hexane/AcOEt/Et<sub>3</sub>N (40/1/1) failed because part of **2a** underwent hydrolysis during passing through the column. Fortunately, **2a** was reported to be purified by using flash column chromatography.[13] In the case of a solid ketimine product like **2c**, it is able to easily obtain the pure **2c** by recrystallization.

Table 2  
Optimization of reaction conditions<sup>a</sup>

Entry	Temp. (°C)	Time (h)	Yield of <b>2a</b> (%) <sup>b</sup>	Recovery of <b>1a</b> (%) <sup>b</sup>
1	60	16	98	n.d.
2	40	16	quant.	n.d.
3	r.t.	16	98	1
4	40	3	quant.	n.d.
5	40	1	96	2

<sup>a</sup> 1.7 mmol of **1a**, 90 mmol of NH<sub>3</sub> gas and 2.5 g of HY were employed.

<sup>b</sup> <sup>1</sup>H NMR yields based on the ratios of **1a** to **2a**.

**Table 3**  
Synthesis of *N*-H ketimines using various ketones<sup>a</sup>

Reaction scheme: Ketone **1** (R<sup>1</sup>-C(=O)-R<sup>2</sup>) reacts with excess NH<sub>3</sub> and H-Y at 40 °C for 3 h to form ketimine **2** (R<sup>1</sup>-C(=NH)-R<sup>2</sup>).

Entry	Ketimine	Yield of <b>2</b> (%) <sup>b</sup>	Recovery of <b>1</b> (%) <sup>b</sup>
1		59 (71) <sup>c</sup> ( <i>E/Z</i> = 3/1)	25 (11) <sup>c</sup>
2		80 (37) <sup>d</sup> ( <i>E/Z</i> = 3/1)	17 (30) <sup>d</sup>
3		86	11

<sup>a</sup> 1.6 mmol of **1**, 90 mmol of NH<sub>3</sub> gas and 2.5 g of H-Y were employed, where one molecule of **1** per supercage of H-Y was added.

<sup>b</sup> <sup>1</sup>H NMR yield using mesitylene or 1,1,2,2-tetrachloroethane as the internal standard.

<sup>c</sup> Reaction time was 16 h.

<sup>d</sup> Instead of H-Y, SiO<sub>2</sub> neutral was used.

Finally, it was investigated if this simple method can be applied to ketones such as alkyl aryl ketones and dialkyl ketones with  $\alpha$ -acidic hydrogens (Table 3). When acetophenone **1b** and 2'-acetonaphthone **1c** were employed, the desired ketimines **2b** and **2c** were obtained in good yields of 71% and 80%, respectively. The *E/Z* ratios of **2b** and **2c** were 3/1 based on the <sup>1</sup>H NMR analysis. On the other hand, the use of SiO<sub>2</sub> neutral only stood at a low yield (37%) of ketimine **2c** as well as poor recovery (30%) of **1**. Also, the dicyclohexyl ketone **1d** yielded the corresponding ketimine **2d** in 86% yield. As far as we surveyed, there are no studies regarding the synthesis of *N*-H ketimines with  $\alpha$ -acidic hydrogens by the condensation of the ketones and ammonia. It should be noted that such *N*-H ketimines were found to be more easily hydrolyzed than those of the *N*-H diaryl ketimines.

We now consider that why the H-Y zeolite is so efficient for the formation of *N*-H ketimines from ketones and ammonia is ascribed to the following reasons: 1) Under the ammonia-rich microenvironment inside the H-Y pores, some protonated ammonia molecules could interact with the ketones to promote and the formed ketimines to stabilize, and 2) the condensation equilibrium is shifted to the ketimine products by isolating the produced water which is hydrogen-bonded with excess ammonia inside the cavities of the zeolite.

In summary, we found that *N*-H ketimines were effectively synthesized from ketones and ammonia by using solid acids, especially zeolite H-Y. The remarkable features of this method are (1) *N*-H ketimines can be obtained in high yields by using ketones and a large excess of ammonia gas at 1 atm. without a solvent in the presence of H-Y; (2) not only a diaryl ketone, but also alkyl aryl ketones or a dialkyl ketone with  $\alpha$ -acidic hydrogens can be converted to the *N*-H ketimines; (3) even solid ketone substances are incorporated in the pores by mechanical mixing with the zeolite for a short time in advance; (4) the work-up procedure is simple by adding methanol and extracting the ketimines from

the zeolite pores, and does not require any treatment with acid or alkali; (5) under the ammonia-rich microenvironment inside the H-Y pores, some protonated ammonia molecules could interact with the ketones to promote the nucleophilic addition of ammonia and have the formed ketimines stabilized, and 6) the condensation equilibrium is shifted to the ketimine formations by isolating the produced water that is hydrogen-bonded with excess ammonia inside the cavities of the zeolite.

This method is expected to be applied to a wide range of ketones and the resulting *N*-H ketimines can be transformed into a variety of amine compounds.

### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.tetlet.2021.152840>.

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- [24] We observed that 0.26 g of water was adsorbed into one gram of H-Y zeolite.