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# Visible light catalyzed Mannich reaction between *tert*-amines and silyl diazoenolates†

Mukund M. D. Pramanik,<sup>a,b</sup> Savita B. Nagode,<sup>a,b</sup> Ruchir Kant<sup>c</sup> and Namrata Rastogi <sup>(b)</sup> \*<sup>a,b</sup>

The present work documents the  $\alpha$ -C–H functionalization of tertiary amines *via* the visible light catalyzed Mannich reaction with silvl diazoenolates. The reaction takes place at room temperature with an organic dye, Rose Bengal, as a photocatalyst and oxygen as the oxidant. The resulting multifunctional products bearing an  $\alpha$ -diazo- $\beta$ -keto group undergo Rh-carbenoid mediated cyclization, affording stable ammonium ylides in high yields.

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# Introduction

The  $\alpha$ -functionalization of tertiary amines by activation of sp<sup>3</sup> C-H bonds adjacent to nitrogen is a powerful strategy for the synthesis of functionalized amines.1 Out of various methods known for the α-C-H activation of tertiary amines, the most widely used one is the oxidation of the amine into a reactive iminium intermediate which is then trapped by a suitable carbon or heteroatom nucleophile. Although the oxidative activation of tertiary amines is predominantly carried out under transition metal mediated conditions,<sup>2</sup> several elegant metal-free protocols have been reported in recent times.<sup>3</sup> Lately, the application of visible light photoredox catalysis (VLPC) has gained popularity over traditional metal-catalysis as a greener and more sustainable alternative.<sup>4</sup> In 2010, Stephenson and co-workers reported the oxidative coupling of tertiary amines with nitroalkanes as nucleophiles under visible light catalysis.<sup>5</sup> Since then, visible light photoredox catalysis has been regularly utilized for the oxidative functionalization of tertiary amines with various nucleophiles employing organic dyes,<sup>6</sup> Ru/Ir complexes,<sup>7</sup> or inorganic semiconductor photocatalysts.8 Usually, these reactions proceed via iminium ions with oxygen as an oxidant; but, in the absence of oxygen, the α-amino radical gives addition products with suitable radical trapping agents.9 However, there are examples of reactions following a radical mechanism under aerobic

Diazo compounds have been extensively utilized as nucleophiles owing to the nucleophilicity of their  $\alpha$ -carbon.<sup>15</sup> Despite this, there are only two reports for  $\alpha$ -CH functionalization of tertiary amines with  $\alpha$ -diazocarbonyl compounds.<sup>16</sup> Notably, the nucleophilicity values "*N*" for the diazocarbonyl compounds employed in these reactions were well within the predicted range (for example, *N* = 4.91 for ethyl diazoacetate)<sup>15a</sup> for the reaction with iminium ions (electrophilicity value E = -5 to -10)<sup>17</sup> according to Mayr's rule of thumb for suitable nucleophile–electrophile combinations.<sup>18</sup> Also, there are plenty of reports employing silyl enol derivatives for the oxidative functionalization of tertiary amines.<sup>13a,19</sup> However, to the best of our knowledge, diazoenolates have not been used as nucleophiles for oxidative coupling with tertiary amines.

We here report, for the first time, the use of silyl diazoenolates as nucleophiles for the  $\alpha$ -CH functionalization of tertiary amines under VLPC conditions (Scheme 1).

#### **Results and discussion**

We started our investigation with *N*-phenyltetrahydroisoquinoline **1a**, ethyl 3-((*tert*-butyldimethylsilyl)oxy)-2-diazobut-3enolate **2** and Rose Bengal (2 mol%) as the photocatalyst in DMF (entry 1; Table 1).



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conditions<sup>10</sup> and iminium ion formation under anaerobic conditions.<sup>11</sup> Recently, visible light mediated  $\alpha$ -CH functionalization of tertiary amines without any photocatalyst<sup>12</sup> or under sequence/dual catalysis has helped in the development of novel transformations of tertiary amines.<sup>13</sup> Furthermore, successful adoption of VLPC reactions in continuous flow microreactors has introduced an automated approach to the method.<sup>14</sup> With such progress in terms of catalysts, oxidants and reaction conditions, the search for hitherto unknown nucleophiles has become even more vigorous.

<sup>&</sup>lt;sup>a</sup>Medicinal and Process Chemistry Division, CSIR-Central Drug Research Institute, Lucknow 226031, India. E-mail: namrata.rastogi@cdri.res.in

<sup>&</sup>lt;sup>b</sup>Academy of Scientific and Innovative Research, New Delhi, India

<sup>&</sup>lt;sup>c</sup>Molecular & Structural Biology Division, CSIR-Central Drug Research Institute, Lucknow 226031, India

<sup>&</sup>lt;sup>†</sup>Electronic supplementary information (ESI) available: <sup>1</sup>H and <sup>13</sup>C NMR spectra of all new compounds. CCDC 1539489. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/c7ob01756a



Scheme 1  $\alpha$ -CH functionalization of tertiary amines with  $\alpha$ -diazocarbonyl compounds.

Table 1 Optimization of reaction conditions<sup>a</sup>

	N <sub>Ph</sub> +	OTBDMS CO2Et N2 2 photocatalyst visible light solvent, air rt, 3-4 h		CO <sub>2</sub> Et 3a	N.Ph
Entry	2 (eq.)	Photocatalyst (mol%)	Solvent (2 mL)	<b>3a</b> <sup>b</sup> (%)	3a' <sup>b</sup> (%)
1	1.5	Rose Bengal (2)	DMF	48	$0^c$
2	1.5	Eosin Y (2)	DMF	45	10
$3^d$	1.5	$[Ru(bpy)_3Cl_2](2)$	DMF	40	16
4	1.5	Rose Bengal (2)	$CH_2Cl_2$	40	0
5	1.5	Rose Bengal (2)	MeCN	52	0
6	1.5	Rose Bengal (2)	MeOH	60	0
7	1.5	Rose Bengal (1)	MeOH	69	0
8	1.5	Rose Bengal (0.5)	MeOH	70	0
9	1.5	Rose Bengal (0.3)	MeOH	65	0
10	1.0	Rose Bengal (0.5)	MeOH	55	0
11	2.0	Rose Bengal (0.5)	MeOH	72	0
12	2.5	Rose Bengal (0.5)	MeOH	78	0
13	3.0	Rose Bengal (0.5)	MeOH	78	0
14	2.5	_	MeOH	0	<10
$15^e$	2.5	Rose Bengal (0.5)	MeOH	0	<10
$16^{f}$	2.5	Rose Bengal (0.5)	MeOH	0	0

<sup>*a*</sup> Unless otherwise noted, all reactions were performed at 0.5 mmol of **1a** scale under green light irradiation for 4 h except for entries 11–13 (3 h). <sup>*b*</sup> Isolated yields. <sup>*c*</sup> 12% when reaction was carried out with oxygen balloon. <sup>*d*</sup> Blue light irradiation. <sup>*e*</sup> Reaction without visible light. <sup>*f*</sup> Under nitrogen.

Upon irradiation of the reaction mixture with green light (530 nm) for 4 h at room temperature, the ethyl 2-diazo-3-oxo-4-(2 phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)butanoate 3a was isolated in 48% yield (entry 1). Upon changing the photocatalyst to Eosin Y or [Ru(bpy)<sub>3</sub>Cl<sub>2</sub>], a slight reduction in yield was noted and the oxidized product 3a' was isolated as a sideproduct (entries 2 and 3). Therefore, Rose Bengal was selected as the preferred photocatalyst for further optimization. Solvent optimization led to the selection of MeOH<sup>20b</sup> (60%) over other solvents tested, for e.g., CH<sub>2</sub>Cl<sub>2</sub> (40%) and MeCN (52%) (entries 4-6). Further changing the catalyst loading (entries 6-9) and the amount of diazoenolate 2 (entries 8, 10-13) revealed that 0.5 mol% of Rose Bengal and 2.5 equivalents of 2 were the best conditions in terms of reaction yield and economy (entry 12). Additionally, control experiments conducted in the absence of photocatalyst or visible light or under inert atmosphere (entries 14-16) substantiated the fact that visible light, photocatalyst as well as oxygen were necessary for the reaction.

After establishing the optimized conditions for the reaction, we set out to examine the scope of tertiary amines 1 in the reaction (Table 2). It is evident from Table 2 that substrates bearing electron-withdrawing groups such as chloro, bromo, nitro as well as electron-releasing groups such as methyl, butyl, and methoxy in the N-aryl ring react smoothly with diazoenolate 2 and provide the corresponding products 3a-h in good yields (54%-82%). However, it is to be noted that the reaction in the case of substrates possessing electronwithdrawing substituents on the N-aryl ring 1f-h was sluggish, taking up to 12 h for completion. Furthermore, the introduction of electron-releasing methoxy groups into the phenyl ring of THIQ did not cause much difference in terms of reaction yields (52%-63%) but the reaction time significantly increased in the case of substrates 1i-m. On the other hand, introducing an electron-withdrawing bromo group into the phenyl ring of THIQ resulted in low product yield (35%) in 3 h whereas a complex mixture formed upon continuing the reaction for a longer time. The reaction worked smoothly with N-aryl pyrrolidine 10 too albeit in low yield but it was disappointing that the desired product could not be isolated with aliphatic tertiary amine 1p even with high catalyst loading.

Mechanistically, the reaction can be depicted as a Mannich reaction of *in situ* generated iminium ions with a diazoenolate nucleophile (Scheme 2).<sup>20</sup>

Table 2 Scope of tert-amines 1 in the coupling reaction<sup>a</sup>



<sup>*a*</sup> Reaction conditions: **1** (0.5 mmol), **2** (1.25 mmol), Rose Bengal (0.5 mol%), MeOH (2 mL), green light at rt in open air. <sup>*b*</sup> With 5 mol% Rose Bengal.



Initially, the photocatalyst excited by visible light RB<sup>\*</sup> undergoes reductive quenching by single electron transfer (SET) from tertiary amine **1**. This process generates tertiary amine radical cation **A** and the reduced photocatalyst. The oxygen present in the air oxidizes back the reduced photocatalyst and forms the superoxide radical anion. This radical anion removes a hydrogen atom from the radical cation **A**, leading to the formation of iminium ion **B** and the peroxide anion. The peroxide anion undergoes protonation in the solvent to form hydrogen peroxide and nucleophilic attack of diazoenolate on the iminium ion **B** followed by the elimination of the <sup>*t*</sup>BuMe<sub>2</sub>Si<sup>+</sup> yields the desired Mannich type product **3**.

Furthermore, in an attempt to establish the synthetic potential of  $\beta$ -oxo- $\alpha$ -diazocarboxylate products **3**, we decided to investigate their behaviour in metal carbenoid-mediated reactions.<sup>15e,21</sup> Upon treating compound **3b** with 1 mol% of Rh<sub>2</sub>(OAc)<sub>4</sub> in dichloromethane at room temperature, a gummy solid was obtained which upon recrystallization provided a colorless solid. The structure of this solid on the basis of spectroscopic and single crystal X-ray analysis was established to be the ammonium ylide **4a** (Scheme 3).<sup>22</sup>

It is well known in the literature that metal carbenoids derived from diazocarbonyl compounds form transient ammonium ylides with amines which undergo intramolecular rearrangement(s) or trapping with suitable dipolarophiles to afford stable scaffolds. However, to the best of our knowledge, there are only handful of reports documenting the isolation of stable ammonium ylides.<sup>23</sup> The reaction works smoothly with other substrates as well affording the ylide products in high yields (Table 3).

Rh<sub>2</sub>(OAc),

(1 mol%) CH<sub>2</sub>Cl<sub>2</sub>, N



Table 3 Scope of substituted THIQ's 3 in the ylide formation<sup>a</sup>

1 mol% Rh<sub>2</sub>(OAc)<sub>4</sub>



Scheme 4 Mechanism of ammonium ylide 4 formation.

The reaction proceeds *via* the rhodium carbenoid **A** which interacts with the lone pair of electrons on nitrogen. The resulting rhodium-ylide **B** dissociates to provide the resonance stabilized ammonium ylide **4** (Scheme 4). It is noteworthy that the aryl group on the nitrogen did not undergo 1,2-migration in the reaction.<sup>23b</sup>

# Conclusions

In summary, we developed the first Mannich reaction between tertiary amines and diazoenolates under visible light photoredox catalysis. The resulting  $\alpha$ -diazo- $\beta$ -oxo-carboxylate products undergo rhodium carbenoid mediated intramolecular ylide formation with amine. Further exploration of the method to expand the scope of tertiary amines as well as silyl diazoenolates is currently underway in our laboratory.

# Conflicts of interest

There are no conflicts to declare.

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Scheme 3 Rh-Carbenoid mediated ammonium ylide 4a formation.

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