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### Communication

# Water and fluorinated alcohol mediated/promoted tandem insertion/ aerobic oxidation/bisindolylation under metal-free conditions: Easy access to bis(indolyl)methanes



Yong Wang<sup>a</sup>, Xinyi Cao<sup>b</sup>, Jingfei Ji<sup>b</sup>, Xiuling Cui<sup>a,\*</sup>, Chao Pi<sup>a</sup>, Leyao Zhao<sup>b</sup>, Yangjie Wu<sup>a,\*</sup>

<sup>a</sup> Henan Key Laboratory of Chemical Biology and Organic Chemistry, Key Laboratory of Applied Chemistry of Henan Universities, Green Catalysis Center, and College of Chemistry, Zhengzhou University, Zhengzhou 450052, China <sup>b</sup> International College, Zhengzhou University, Zhengzhou 450052, China

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#### ABSTRACT

A green tandem reaction, including insertion/aerobic oxidation/bisindolylation, starting from indoles and diazo compounds has been developed. The combination of water and fluorinated alcohol plays dual roles as solvent and promoter in this chemical transformation. Molecular oxygen in the air acts as an oxidant. 3,3'-Bis(indolyl)methanes with quaternary carbon were produced under metal-free conditions. No any catalyst and additive were required. N<sub>2</sub> and water were released as sole by-products. Absence of water and fluorinated alcohol resulted in Wolff rearrangement product.

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Bis(indolyl)methane (BIM) is a significant structural motif isolated from diverse natural sources [1] and exhibits broad spectrum bioactivities [2]. Traditional construction of such a skeleton bases on Friedel-Crafts reaction of indoles with carbonyl compounds (aldehydes and ketones) in the presence of Lewis acid as a catalyst [2f]. To avoid the catalyst and additives, greener procedures have been explored recently [3]. However, most products obtained are BIMs with tertiary carbon. Molecules containing quaternary carbon center are usually problematic to be prepared, but show unique bioactivities due to its special rigid three-dimensional structure [4]. Thus, a greener procedure to access BIMs with quaternary carbon is highly desired.

On the other hand, the condensation of indoles and diazo compounds *via* transition metal catalyzed carbenoid insertion to construct C—C bond has been extensively studied. Generally, all of the N, C2 and C3 atoms could work as nucleophilic site for N-H free indoles (Scheme 1a). Selective C3 attack usually was achieved with N- and C2- substituted indoles [5]. Very recently, Wang, Lu group [6] reported a copper catalyzed C3-bisindolation of 3-diazoindolin-2-imines with N substituted indoles (Scheme 1b). However, N-H free indoles were not effective. Soon after, Liu group [7] developed a gold catalyzed bisindolylation of aryldizao cyanide

\* Corresponding authors. E-mail addresses: cuixl@zzu.edu.cn (X. Cui), wyj@zzu.edu.cn (Y. Wu). and only one example of N-H free indole was used as substrate with moderate yield (Scheme 1c). Herein, we disclose a novel reaction of N-H free indoles with  $\alpha$ -diazo- $\beta$ -ketoesters under metal and additives free conditions. This procedure provides a greener protocol to afford various BIMs with quaternary carbon (Scheme 1d). Molecular oxygen from air as an oxidant, water and fluorinated alcohol as co-solvent and promoter make this procedure greener. Catalyst and additives were avoided and only N<sub>2</sub> and water were produced as by-products. This high atom economy reaction benefits sustainable development.

We initiated our study using indole (1a) and ethyl 2-diazo-3oxobutanoate (2a) as the model substrates to screen various reaction parameters (Table 1). When non-polar solvents were used, Wolff rearrangement [8] reaction occurred, affording **4a** as a main product (entries 1–4). The product **4a** was obtained in 82% yield in hexane at 120 °C under air atmosphere for 12 h (entry 4). No reaction occurred in EtOH or MeCN (entries 5 and 6). Water is an ideal solvent in organic synthesis attribute to its being green, cheap, nontoxic and non-flammable [9], moreover could accelerate reaction rate owing to its hydrophobic effect [10]. To our delight, water as solvent led to the product **3aa** in 28% yield (entry 7). Then we tried to add other polar solvent in water to improve the solubility of substrates. EtOH or MeCN as co-solvent did not give better results (entries 8 and 9). Fluorinated alcohol, 1,1,1,3,3,3hexafluoroisopropanol (HFIP) and 1,1,1-trifluoroethanol (TFE) possess unique properties, such as high hydrogen bonding donor

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Scheme 1. Divergent reactions of indole and diazo compounds.

ability, low nucleophilicity, high ionizing power, extreme polarity, enhanced acidity, ability to stabilize cationic species and solvate water [11]. This kind of alcohol has been employed to promote diverse reactions including oxidation [11a-c] and Friedel-Crafts reactions [3a,12]. Hence, we hypothesized that fluorinated alcohol might facilitate the desired reaction. As expected, 3aa was obtained in elevated yield (39%) when water and TFE were used as co-solvent in 1:1 ratio of volume (entry 10). None of **3aa** was afforded under nitrogen atmosphere (entry 11). Oxygen atmosphere gave the similar result to air (entry 12). These results suggested that oxygen from air might play the key role in the reaction. Increasing the solvent to 3 mL, 79% isolated yield of 3aa was obtained (entry 13). 75% Yield of **3aa** was attained when water and HFIP were employed as co-solvents (entry 15). Not any transformation was detected when TFE or HFIP as sole solvent (entry 16). The results obtained above indicated that water and fluorinated alcohol played synergic role to accelerate the reaction.

Under the optimal conditions in hand, scope of indoles was surveyed (Scheme 2, detailed experimental procedure see Supporting information). In general, electron-rich indoles worked well when either TFE or HFIP as co-solvent with water (3ba-3ga). In particularly, methoxy at different position of indoles all gave the corresponding products in good to excellent yields (3da-3fa, 81%–92%). For the indoles with halogen and electron-withdrawing groups, reactions proceeded smoothly in HFIP than TFE as cosolvent with water (3ha-3pa). F, Cl, Br and I were tolerated well and the desired products were obtained in good yields (3ha-3ma, 68%-86%). Ester or carboxyl group in the indole afforded the desired products in moderate yields (3na-3pa, 43%-55%) as well as tertiary alcohols (5na-5pa) as by-products. While, tertiary alcohols were given as the main products (5qa and 5ra) for indoles with strong electron-withdrawing groups (nitrile and nitro group). Subsequently, N-substituted indoles were investigated. Both N-alkyl and phenyl indoles led to bisindolylated products in good yields (3sa-3wa and 3ya, 67%–89%). While, N-tosyl indole did not work (3xa).

#### Table 1

Optimization of the reaction conditions.<sup>a</sup>

$ \begin{array}{c} \begin{array}{c} \begin{array}{c} \\ \end{array} \end{array} + \begin{array}{c} N_{2} \\ Ac \end{array} + \begin{array}{c} \begin{array}{c} \text{solvent} \\ 120 \text{ °C, air, 12 h} \end{array} + \begin{array}{c} \begin{array}{c} Ac \\ HN \end{array} + \begin{array}{c} COOEt \\ HN \end{array} + \begin{array}{c} \begin{array}{c} \\ \\ \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \\ \\ \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \\ \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \\ \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \end{array} + \begin{array}{c} \end{array} + \begin{array}{c} \end{array} + \end{array} + \end{array} + \begin{array}{c} \end{array} + \end{array} $						
Id	Za	3aa H	4a			
Entry	Solvent	Recovery of <b>1a</b> (%) <sup>b</sup>	Yield (%) <sup>b</sup>	Yield (%) <sup>b</sup>		
			3aa	4a		
1	Toluene	24	_	66		
2	1,4-Dioxane	54	-	33		
3	PhCl	-	-	81		
4	Hexane	-	-	85(82) <sup>c</sup>		
5	EtOH	67	-	-		
6	MeCN	99	-	-		
7	H <sub>2</sub> O	29	28	-		
8	$H_2O/MeCN (1:1)^d$	78	-	-		
9	H <sub>2</sub> O/EtOH (1:1) <sup>d</sup>	40	4	-		
10	$H_2O/TFE (1:1)^d$	9	39	-		
11	H <sub>2</sub> O/TFE (1:1) <sup>d,e</sup>	99	trace	-		
12	H <sub>2</sub> O/TFE (1:1) <sup>d,f</sup>	-	39	-		
13	H <sub>2</sub> O/TFE (1:1) <sup>g</sup>	-	70(79) <sup>c</sup>	-		
14	H <sub>2</sub> O/TFE (2:1) <sup>g</sup>	-	67(76) <sup>c</sup>	-		
15	H <sub>2</sub> O/HFIP (1:1) <sup>g</sup>	-	68(75) <sup>c</sup>			
16	HFIP or TFE <sup>d</sup>	99	-	-		

 $^a\,$  Reaction conditions:  $1a\,(0.1$  mmol),  $2a\,(0.1$  mmol) and solvent (0.5 mL) at 120  $^\circ\text{C}$  under air atmosphere for 12 h.

' NMR yield.

<sup>c</sup> Isolated yield in parentheses.

<sup>d</sup> 1 mL.

<sup>e</sup> Under N<sub>2</sub>. <sup>f</sup> Under O<sub>2</sub>.

<sup>g</sup> 3 mL

In addition, **3aa** was obtained in 78% for *N*-Boc indole. Fused indole also could provide the desired product in good yield (**3ya**).

Then scope of diazo compounds **2** was also investigated (Table 2). Apart from acetyl,  $\alpha$ -diazo alkyl ketoesters with cyclopropyl, propyl and isopropyl also led to the corresponding products in moderate to good yields (**3sb-3sd**). Various *para*-and *meta*-substituted benzoyl diazos gave the products in good yields (**3sf-3sk**, 60%–82%). Yield of product **3sl** (49%) was generated for  $\alpha$ -diazo *ortho*-F aryl ketoesters.  $\alpha$ -Diazo 2-furyl and 2-thienyl ketoesters provided the corresponding products in 70% (**3sm**) and 77% (**3sn**) yields, respectively. Monosubstituted  $\alpha$ -diazo aryl ketone (**2o**) and ethyl diazoacetate (**2p**) produced **3so** and **3sp** with 1-methylindole in moderate yields. While,  $\alpha$ -diazo- $\beta$ -diketo (**2r** and **2u**),  $\alpha$ -diazo- $\beta$ -diester (**2s**) and ethyl 2-diazo-2-phenylacetate (**2t**) failed to give the desired products, perhaps due to the reason that the O-H insertion intermediates could not be transformed to vicinal tricarbonyl compounds under air.

This protocol was also applied to the synthesis of unsymmetric BIMs. The results are depicted in Scheme 3. In view of the fact that electron-rich indoles react faster than deficient ones, the loading of the comparative electron-deficient indole (1a, 1k or 1e) is slightly excessive than 1-methylindole (1s). As a result, unsymmetric BIMs **3saa, 3ska, 3sea** and **3vea** were produced in moderate yields (41%–57%). By utilizing the discrepant nucleophilicity of indoles with different substituents, **3zsa, 3zaa** and **3zka** were obtained in moderate to good yields (47%–68%). And **3eaa** was provided in a 31% yield from similar nucleophilicity of 1e and 1a.

To clarify the reaction mechanism, a series of control reactions were conducted. First, the reactions of 1-methylindole **1s** and **6e** were carried out in different solvents (Table 3). Mixed solvent of water and TFE led to the quantitative bisindolylated product **3se** (entry 1). **6e** might be a key intermediate from diazo compound **2e**. Water or TFE as sole solvent resulted in lower yield of **3se** (entries 2 and 3). These results demonstrate that combination of water and



**Scheme 2.** Scope of indole derivatives. Reaction conditions: **1** (0.1 mmol), **2a** (0.1 mmol) and solvent A or B at 120 °C under air atmosphere for 12 h. Isolated yields. Solvent A:  $H_2O$  (2 mL) and TFE (1 mL); B:  $H_2O$  (1 mL) and HFIP (0.5 mL).



Scope of diazo compounds.<sup>a</sup>



Entry	R <sup>1</sup>	R <sup>2</sup>	3	Yield (%)
1	c-C <sub>3</sub> H <sub>5</sub>	COOEt	3sb	86
2	<sup>n</sup> Pr	COOEt	3sc	62
3	<sup>i</sup> Pr	COOEt	3sd	56
4	Ph	COOEt	3se	69
5	4-OMe-C <sub>6</sub> H <sub>4</sub>	COOEt	3sf	60
6	$4-F-C_6H_4$	COOEt	3sg	79
7	4-Cl-C <sub>6</sub> H <sub>4</sub>	COOEt	3sh	82
8	4-Br-C <sub>6</sub> H <sub>4</sub>	COOEt	3si	74
9	$4-CF_3-C_6H_4$	COOMe	3sj	71
10	3-F-C <sub>6</sub> H <sub>4</sub>	COOEt	3sk	77
11	2-F-C <sub>6</sub> H <sub>4</sub>	COOEt	3sl	49
12	2-furyl	COOEt	3sm	70
13	2-thienyl	COOEt	3sn	77
14	OEt	Н	3so	47
15	Ph	Н	3sp	52



fluorinated alcohol paly the key role to accelerate reaction. Second, treatment of  $\alpha$ -diazo- $\beta$ -ketoester **2f** in the mixed solvent of water and TFE under air produced the product **6f** in 16% as well as Wolff rearrangement/decarboxylation product **7f** in 69% yield (Scheme 4, entry 1). However, **6f** was not detected in the water or TFE as sole solvent and only Wolff rearrangement products (**7f** or **8**) were isolated (entries 2 and 3).



Scheme 3. Synthesis of unsymmetric bis(indolyl)methanes. Reaction conditions: 1 (electron-rich indole, 0.1 mmol), 1 (electron-deficient indole, 0.15 mmol), 2a (0.2 mmol), H<sub>2</sub>O (2 mL) and TFE (1 mL) at 120 °C under air atmosphere for 12 h. Isolated yields.

#### Table 3

2

3

Reaction of 1-methylindole and **6e** in different solvents.<sup>a</sup>

 $\frac{1}{1} + \frac{1}{120 \circ C, 12 h, air} + \frac{1}{3se} + \frac{1}{120 \circ C, 12 h, air} + \frac{1}{3se} +$ 

<sup>a</sup> Reaction conditions: **1s** (0.1 mmol), **6e** (0.1 mmol) and solvent at 120 °C under air atmosphere for 12 h. Isolated yields.

54%

15%

H<sub>2</sub>O (2 mL)

TFE (2 mL)

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\begin{array}{c}
\begin{array}{c}
\end{array}\\
\end{array}\\
\end{array} \\ 1a \\ 2a \\ \end{array} \\ \begin{array}{c}
\end{array} \\ 2a \\ \end{array} \\ \begin{array}{c}
\end{array} \\ solvent \\ HN \\ 3aa \\ \end{array} \\ \begin{array}{c}
\end{array} \\ \begin{array}{c}
\end{array} \\ Ac \\ COOEt \\ HN \\ 3aa \\ \end{array} \\ \begin{array}{c}
\end{array} \\ \end{array} \\ \begin{array}{c}
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\end{array} \\ \end{array} \\ \begin{array}{c}
\end{array} \end{array}$$
 (1) \\ \end{array}

In addition, the reaction of **1s** and ethyl 2-hydroxy-3-oxo-3phenylpropanoate **9e** was conducted under the standard reaction conditions. The desired product **3se** was obtained in 89% yield (Eq. 1). The above results obtained imply that the diazo was oxidized by oxygen in the air, which was promoted by water and fluorinated alcohol. Generally, stoichiometric oxidant, such as *tert*butyl hypochlorite [13], dimethyl dioxirane [14] or 2,6-dichloropyridine *N*-oxides [15], is required for the oxidation of  $\alpha$ -diazo- $\beta$ ketoester.

Based on the results above, a plausible reaction mechanism was proposed and depicted in Scheme 5.  $\alpha$ -Diazo- $\beta$ -ketoester **2a** was protonated by fluorinated alcohol as Brønsted acid to afford the diazonium I [16]. Then nucleophilic substituent of diazonium I with water produced  $\alpha$ -hydroxy- $\beta$ -ketoester II as well as N<sub>2</sub> [16,17].



**Scheme 4.** Reaction of  $\alpha$ -diazo- $\beta$ -ketoesters in different solvents. Reaction conditions: 2f (0.1 mmol) and solvent at 120 °C under air atmosphere for 12 h. Isolated yields.



Scheme 5. Plausible mechanism.

Subsequently, II was oxidized into 6a by oxygen molecule from air atmosphere [18]. Through this umpolung transformation, the nucleophile 2a was transformed as an electrophile 6a [19]. Subsequently, the *in situ* generated carbonyl group was activated by water or fluorinated alcohol with H-bond. Following by the nucleophilic addition of this carbonyl group with the indole gave III [20]. Intermediate IV was generated through elimination of one molecular H<sub>2</sub>O. Then the second nucleophilic attack of indole produced 3aa.

In conclusion, we have developed a green, efficient and novel protocol to access BIMs with quaternary carbon starting from indoles and  $\alpha$ -diazo- $\beta$ -ketoesters. This approach is promoted by water and fluorinated alcohol as co-solvent under metal catalyst and additive free conditions. Only N<sub>2</sub> and water were released as by-products. This high atom economy reaction benefits environmental protection and sustainable development. Moreover, a tandem reaction of O-H insertion, aerobic oxidation and bisindolylation were involved in this transformation and also applied in the synthesis of unsymmetric BIMs.

#### **Declaration of competing interest**

The authors report no declarations of interest.

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#### Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2020.12.026.

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