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Photoinduced Rapid Multicomponent Cascade Reaction of Aryldiazonium Salts with Unactivated Alkenes and $TMSN_3$

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ABSTRACT: The first example of a photoinduced rapid multicomponent cascade reaction of aryldiazonium salts with unactivated alkenes and trimethylsilyl azide ($TMSN_3$) under oxidant-free conditions is described. This approach provides the synthetic route for a wide range of unsymmetric azo compounds. The compounds are obtained in good to excellent yields under mild reaction conditions. This transformation is applicable to various aryldiazonium salts and alkenes, providing an alternative route for the synthesis of unsymmetric azo compounds. The control experiment demonstrates that the reaction mechanism follows a radical pathway.

Aromatic azo compounds have been used for the preparation of dyes, pigments,[1] therapeutic agents,[2] food additives, and indicators (Figure 1).[3] The compounds can be obtained through the azo coupling reaction,[4] Mills reaction,[5] and Wallach reaction.[6] However, these synthetic methods for forming the azo group suffer from various disadvantages. The reactions need to be carried out at high temperatures (>100 °C) in the presence of special substrates and additives.[7] Furthermore, most of these approaches are only suitable for the synthesis of diaryl azo compounds. Therefore, it is necessary to develop a green and efficient method to synthesize aryl−alkyl azo compounds.

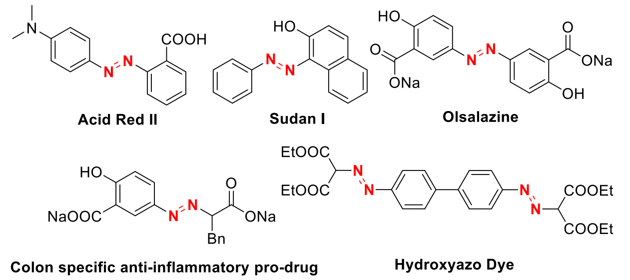
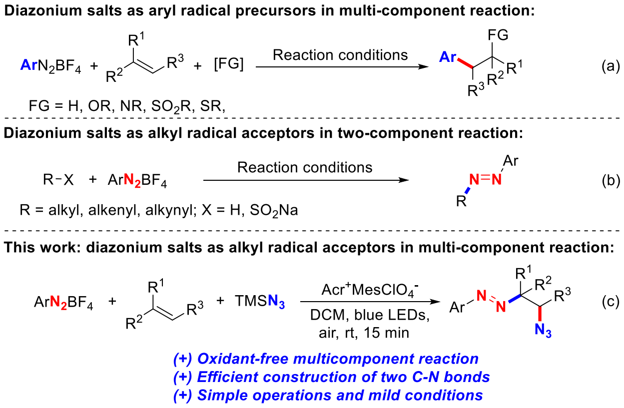


Figure 1. Representative azo compounds.

We note that diazonium salts containing the N═N group can be used as a precursor to prepare azo compounds. Aryl diazonium salts are widely available and can be easily produced from anilines that are present in abundance. In recent years, numerous attempts have been made to synthesize azo compounds via multicomponent reaction processes using diazonium salts as aryl radical precursors (Scheme 1a).[8] Studer and co-workers developed a new method for radical oxyarylation of alkenes using the readily available aryl diazonium salts as aryl radical precursors.[8a] In 2014, König and co-workers demonstrated a visible-light-induced formation of C−N bonds following an intermolecular amino-arylation process involving alkenes.[8b] In 2017, Jiang's group established the unprecedented [2 + 2] cycloaddition/S-centered radical-induced 1,4-addition cascade reactions involving benzene-linked allene−ynes. The reactions were conducted under mild and redox neutral conditions.[8c] In the same year, Wu et al. reported an efficient route to synthesize sulfonated tetrahydropyridine derivatives via a radical pathway in the presence of 1,6-enynes, sulfur dioxide, and aryl diazonium tetrafluoroborates.[8d] Reports on radical reactions where diazonium salts are

Scheme 1. Radical Reaction Involving Diazonium Salts



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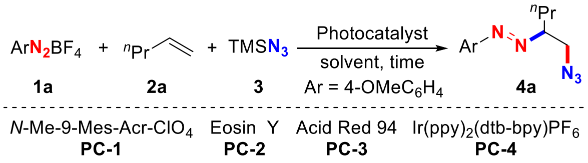
used as the radical acceptors are scarce (Scheme 1b).[9] In 2017, Hao's group developed an efficient method for the synthesis of thermally stable and pharmaceutically important unsymmetric azo compounds.[9a] Subsequently, Clayden and co-workers reported an elegant strategy for the preparation of alkyl–aryl azo compounds via a radical addition reaction involving alkyl radicals and diazonium ions.[9b] Despite these considerable advances, such transformations are still restricted to two-component reactions. The reactions employing aryl diazonium salts as intermolecular radical acceptors in multicomponent reactions have been rarely realized.[10]

Organoazides have been extensively used for structuring complex pharmaceutical molecules and preparing diversified chemical libraries.[11] The photocatalyzed three-component cascade reaction of alkenes has recently attracted attention and is an elegant example of a method that can be used for the synthesis of organoazides.[12] In 2017, Lu and co-workers reported the photocatalyzed metal-free aerobic hydroxyazidation reaction of alkenes in the presence of trimethylsilyl azide ($TMSN_3$).[12a] They were the first to report this reaction. They also reported a photocatalyzed three-component carboazidation reaction involving alkenes, $TMSN_3$, and acrylonitrile.[12b] Yang et al. developed a method for the synthesis of α-azidoketones. The photocatalyzed oxyazidation of alkenes in the presence of $TMSN_3$ afforded the desired compounds.[12c] Recently, our group developed a radical three-component cascade reaction involving quinoxalinones, $TMSN_3$, and alkenes.[13] In addition, we later disclosed a photocatalytic decarboxylative coupling strategy for the mild and efficient synthesis of 3-alkylated quinoxalinones.[14] In a continuation of our efforts, we herein demonstrate a rapid, photocatalyzed three-component cascade reaction of diazonium salts in the presence of unactivated alkenes and $TMSN_3$. This strategy provides an alternative route for the synthesis of unsymmetric azo compounds for the efficient construction of two C–N bonds (Scheme 1c).

Initially, 4-methoxybenzenediazonium tetrafluoroborate (**1a**), 1-amylene (**2a**), and $TMSN_3$ (**3**) were chosen as the starting substrates to optimize the reaction conditions (Table 1). When 4 mol % of $Acr^+MesClO_4^-$ (a visible-light photocatalyst) was used for the reactions, the target product (**4a**) was obtained in 34% yield. The reaction was carried out in MeCN (1.0 mL) at room temperature. The desired product formed within 1 h (Table 1, entry 1). Other photocatalysts, such as eosin Y, Acid Red 94, and $Ir(ppy)_2(dtb\text{-}bpy)PF_6$, afforded the products in relatively low yields under the same reaction conditions (Table 1, entries 2–5). The reaction solvent plays a very important role in this reaction. Various organic solvents, such as dichloroethane (DCE), dichloromethane (DCM), tetrahydrofuran (THF), toluene, dimethyl sulfoxide (DMSO), and *N*,*N*-dimethylformamide (DMF), were screened. It was found that the optimal product yield was obtained when DCM was used as the reaction solvent (Table 1, entries 1 and 6–11). The reaction time did not significantly effect on this transformation (Table 1, entries 12–14). To our delight, the reaction proceeded to completion within 15 min (Table 1, entries 13 and 14). This makes this method of synthesizing azo compounds highly efficient and economical. The amount of the catalyst did not influence the product yield (**4a**). Even when the catalyst loading was reduced to 2 mol %, the reaction afforded **4a** in 88% yield. When the catalyst loading was 1 mol %, the product yield could not be improved by increasing the duration of the reaction (Table 1, entries 15–17). However, the product was not formed in the absence of the photocatalyst or light (Table 1, entries 5 and 18). When the light intensity was increased from 6 to 10 W, the yield of the desired product increased to 95% (Table 1, entry 19). When the model reaction was carried out under a nitrogen atmosphere, 93% yield of the desired product was detected (Table 1, entry 20).

Table 1. Optimization of Reaction Conditions[a,b]



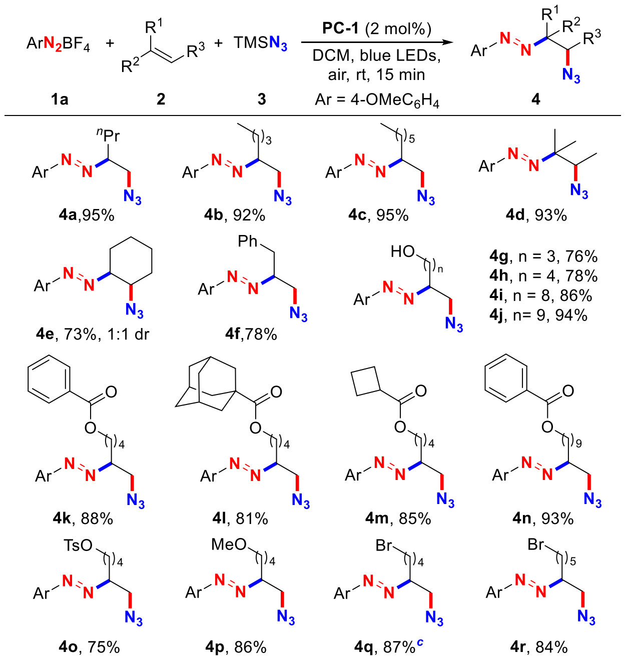
entry	photocatalyst	solvent	time	yield[b] (%)
1	**PC-1**	MeCN	1 h	34
2	**PC-2**	MeCN	1 h	12
3	**PC-3**	MeCN	1 h	21
4	**PC-4**	MeCN	1 h	0
5		MeCN	1 h	0
6	**PC-1**	DCE	1 h	74
7	**PC-1**	DCM	1 h	87
8	**PC-1**	THF	1 h	17
9	**PC-1**	toluene	1 h	25
10	**PC-1**	DMSO	1 h	32
11	**PC-1**	DMF	1 h	18
12	**PC-1**	DCM	2 h	81
13	**PC-1**	DCM	15 min	86
14	**PC-1**	DCM	10 min	63
15[c]	**PC-1**	DCM	15 min	89
16[d]	**PC-1**	DCM	15 min	88
17[e]	**PC-1**	DCM	2h	68
18[d]	**PC-1,dark**	DCM	15 min	0
19[d,f]	**PC-1**	DCM	15 min	95
20[d,f,g]	**PC-1**	DCM	15 min	93

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (1.3 equiv), **3** (1.3 equiv), photocatalyst (4 mol %), solvent (1.0 mL), room temperature, irradiation with 6 W blue LEDs under air. [b] Isolated yields. [c] Photocatalyst (5 mol %). [d] Photocatalyst (2 mol %). [e] Photocatalyst (1 mol %). [f] 10 W blue LEDs were used as the light source. [g] N_2 atmosphere.

With the optimized reaction conditions in hand, the substrate scope of the cascade reaction was explored by reacting various alkenes (**2**) with 4-methoxybenzenediazonium tetrafluoroborate (**1a**) and $TMSN_3$ (**3**) (Scheme 2). Non-functionalized linear and cyclic olefins afforded the desired products in excellent yields (**4a–e**). However, linear alkenes bearing the sensitive hydroxyl groups and multiple substituted aromatic alkenes yielded the corresponding products (**4f–j**) in relatively low yields (76–94%). It should be mentioned that the olefins bearing various functional groups such as ester, ether, sulfonyl, and halo substituents were well tolerated under the optimized reaction conditions (**4k–r**), affording the corresponding products in 75–93% yields.

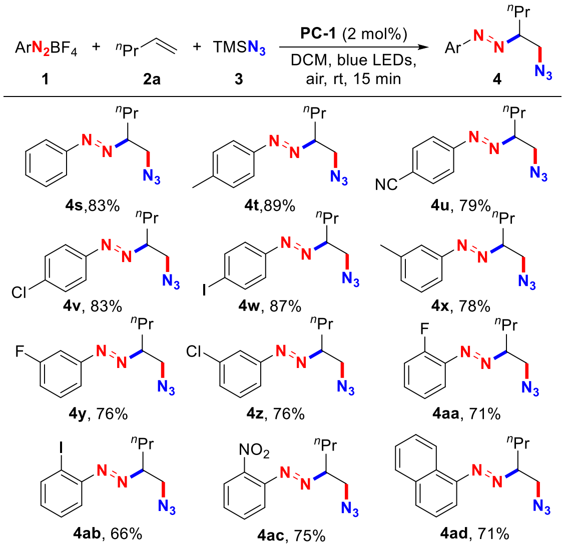
Following this, the substrate scope of aryl diazonium salt (**1**) in the presence of 1-amylene (**2a**) and $TMSN_3$ (**3**) was evaluated (Scheme 3). It was observed that aryldiazonium salts bearing electron-withdrawing or electron-donating groups, at the *para*-, *meta*-, or ortho-positions afforded the corresponding products (**4s–4ac**) in good yields. Moreover, the reactions with 1-naphthyldiazonium salt proceeded smoothly to afford the target product (**4ad**) in 71% yield.

Scheme 2. Substrate Scope of Alkenes[a,b]



[a]Reaction conditions: **1a** (0.2 mmol), **2** (1.3 equiv), **3** (1.3 equiv), PC-1 (2.0 mol %), DCM (1.0 mL), 10 W blue LEDs, 15 min, under air. [b]Isolated yields. [c]Reaction was performed on a 1 mmol scale.

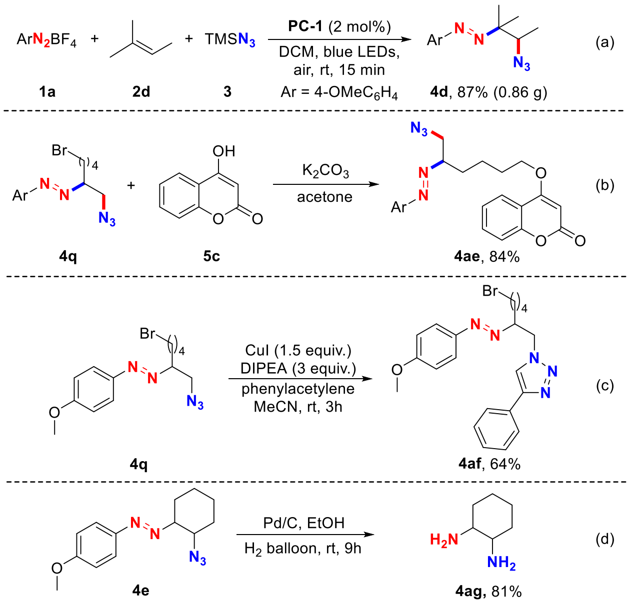
Scheme 3. Substrate Scope of Aryl Diazonium[a,b]



[a]Reaction conditions: **1** (0.2 mmol), **2a** (1.3 equiv), **3** (1.3 equiv), PC-1 (2.0 mol %), DCM (1.0 mL), 10 W blue LEDs, 15 min, under air. [b]Isolated yields.

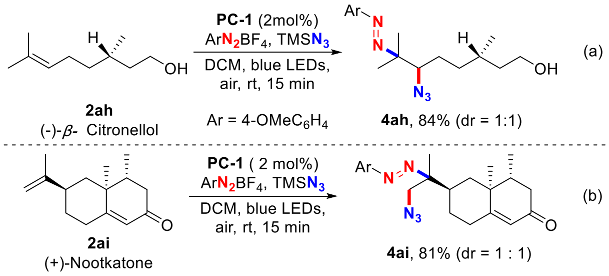
The practical synthetic application of the procedure was also investigated. First, a gram-scale synthesis was performed. The product (**4d**) was obtained in 87% yield (Scheme 4a). Following this, a potentially bioactive molecule (**4ae**) was easily synthesized in 84% yield via a dehalogenation pathway. The molecule **4q** was reacted with 4-hydroxycoumarin[15] to afford the said bioactive molecule (Scheme 4b). Additionally, triazole **4af** could be obtained through "click" reaction with phenylacetylene (Scheme 4c). 1,2-diaminocyclohexane **4ag** was also obtained through hydrogenation (Scheme 4d).

Scheme 4. Gram-Scale Synthesis and Further Chemistry



Finally, natural products (−)-ß-citronellol and nootkatone were structurally modified to yield the products **4ah** and **4ai** in good yields (Scheme 5).

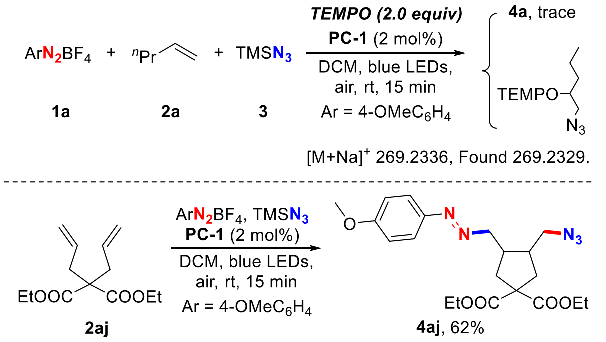
Scheme 5. Modification of Natural Products



We conducted a preliminary mechanistic investigation to gain insight into the reaction mechanism.[16] The reaction did not proceed when two equivalents of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) were added as the radical inhibitor. The corresponding adducts formed were detected by the high-resolution mass spectrometry technique, revealing that a free-radical process can be potentially operative. The radical clock reaction of diethyl 2,2-diallylmalonate **2aj** was performed to afford the radical ring-closure product **4aj** in 62% yield, suggesting that this protocol advances via a radical pathway (Scheme 6). This was further proved by the fluorescence quenching study, as shown in Figure 2. The fluorescence of the photocatalyst could be quenched in the presence of TMSN$_3$. This observation was consistent with our hypothesis.[17]

Based on the above results and previous reports,[16,18] we proposed the possible reaction mechanism (Scheme 7). We proposed that, initially, the photocatalyst **PC-1** was photo-promoted to the excited state producing the ***PC-1** species (E_{red}* = 2.06 V vs SCE).[19] Following this, a single electron transfer (SET) process occurred between TMSN$_3$ (**3**) and ***PC-1** to produce the azido radical and **PC-1**$^{\cdot-}$ radical anion. The oxidation potential of the free azide anion was found to be +1.32 V (NHE).[20] Subsequently, the azido radical attacked olefin **2a** rapidly to generate an alkyl radical (**A**), which was

Scheme 6. Control Experiments



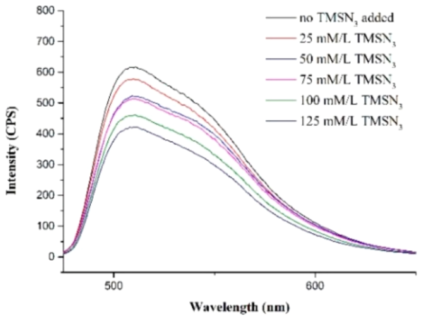
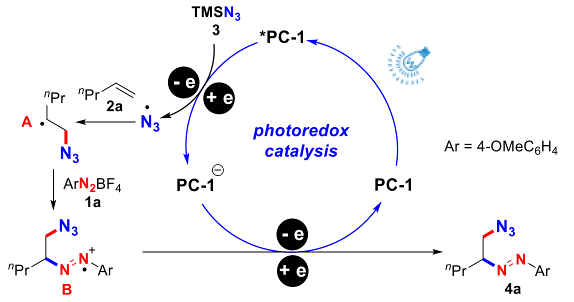


Figure 2. Quenching of Acr$^+$MesClO$_4^-$ fluorescence emission in the presence of TMSN$_3$.

Scheme 7. Plausible Mechanism



trapped by the aryldiazonium salt **1a** to produce the radical cation (**B**).[21] Finally, another SET process between the radical cation (**B**) and the **PC-1**$^{\cdot-}$ radical anion afforded the target product **4a** with the simultaneous regeneration of the photocatalyst **PC-1**.

In conclusion, we have reported a photocatalyzed, rapid three-component cascade reaction involving aryl diazonium, unactivated alkenes, and TMSN$_3$. The substrates bearing various functional groups were well tolerated under the mild catalytic conditions, affording the corresponding products in good to excellent yields. This reaction provides a simple and efficient method for the synthesis of various useful azo compounds. It was hypothesized that this three-component transformation proceeded via a radical mechanism.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acs.orglett.0c04148.

Experimental procedures and characterization data for all the compounds (PDF)

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Notes

The authors declare no competing financial interest.

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