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Visible light catalyzed aromatization of 1,3,5-triaryl-2-pyrazolines by platinum(II) polypyridyl complex under oxidant-free condition

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With visible light (λ =450 nm) irradiation of a catalytic amount of platinum(II) terpyridyl complex, 1,3,5-triaryl-2-pyrazolines can be smoothly converted to their corresponding pyrazoles and hydrogen in quantitative yields with no use of any oxidant at room temperature.

pyrazolines, pyrazoles, photoredox catalysis, visible light, platinum(II) terpyridyl complex

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1 Introduction

Pyrazoles are an important class of five-membered heterocyclic compounds that often exist in biologically active natural products and synthetic compounds of agricultural and pharmaceutical interest [1]. Furthermore they are useful ligands for the new generation of transition metal catalysts for carbon-carbon coupling reactions [2]. The synthesis of pyrazoles is by all means an attractive area in heterocyclic chemistry [3]. Among them, the oxidative aromatization of 1,3,5-trisubstituted pyrazolines provides an efficient access, because 1,3,5-trisubstituted pyrazolines can be conveniently prepared from phenylhydrazine and chalcone derivatives [4]. Actually, various oxidizing reagents have been employed to oxidize pyrazolines to the corresponding pyrazoles such as HgO [5], MnO₂[6], AgNO₃[7], Zr(NO₃)₄[8], KMnO₄ [9], Pb(OAc)₄ [10], p-chloranil [11], trichloroisocyanuric acid [12], iodobenzenediacetate [13], iodic acid/ iodine pentoxide [14], tri(4-bromophenyl)aminium (TBPA⁺)

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hexachloroanitimonate [15], Pd/C [16], activated carbon [17], FeCl₃ [18]. However, some of them suffer from drawbacks such as the use of toxic and expensive reagents, strict reaction conditions, and difficulty in purification. Photooxidation has afforded an alternative way in oxidative aromatization. Direct ultraviolet irradiation [19] and dye-sensitized photooxidation [20] of various aryl-substituted 1,3diphenyl-2-pyrazolines also led to dehydrogenation to the corresponding pyrazoles in the presence of molecular oxygen, but some of them may incorporate oxygen with ring destruction to yield carbonyl compounds. A mild and efficient photoaromatization of aryl- and hetarylpyrazolines through a photoinduced electron transfer (PET) has also been developed, with a considerable excess of carbon tetrachloride as the second reactant [21].

Recently, visible light photoredox catalysis with transition metal complexes has found broad utility in organic synthesis [22]. Among them, the square-planar platinum(II) polypyridyl complex is appealing at the forefront of photochemistry due to its unique properties of open axial coordination site, strong visible absorptions, long excited-state

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lifetime, high luminescent quantum yield, and good chemical stability [23]. With visible-light irradiation, for example, pyridine derivatives [24], 3,4-diarylpyrroles [25], and 3,4-diarylthiophenes [26] have been successfully obtained respectively, in the absence of any oxidants. To develop a photocatalytic approach for the preparation of 1,3,5-triaryl pyrazoles from synthetic point of view, we expected that platinum(II) complex may act as a catalyst to conduct such photochemical transformation. In the present work, we report that upon irradiation with visible light (λ =450 nm), a catalytic amount of platinum(II) terpyridyl complex **1** is capable of converting 1,3,5-triaryl-2-pyrrazolines (**2**) to the corresponding pyrazoles (**3**) and hydrogen (H₂) with no use of any oxidant (Scheme 1).

2 Experimental

General procedure for the photoreaction: the 1,3,5-triaryl pyrazolines **2** (0.017 mmol, 1 equiv.) and platinum(II) complex **1** (0.5 µmol, 0.03 equiv.) were dissolved in deoxygenated CH₃CN (1 mL) in a 10 mL reaction tube equipped with a rubber septum and magnetic stir bar, and then the argonpurged solution was irradiated by blue LEDs (λ_{max} =450 nm) at room temperature. The generated hydrogen was analyzed by GC using a 5 Å molecular sieve column with thermal conductivity detector. After the substrate was completely converted (monitored by TLC), the solvent was removed under reduced pressure, then the products 1,3,5-triaryl pyrazoles (**3**) were isolated by extraction with ethyl acetate or purified by column chromatography on silica gel eluting with ethyl acetate/petroleum ether and identified by ¹H NMR, ¹³C NMR and MS.

3 Results and discussion

To begin this study, 1,3,5-triphenyl-2-pyrazoline (**2a**) was chosen as the standard substrate for the desired photocatalytic reaction. As shown in Figure 1(a), platinum(II) comp-



Scheme 1 Oxidant-free photocatalytic aromatization of pyrazolines to pyrazoles (color online).



Figure 1 (a) UV-Vis absorption spectra of **1**, **2a** and **3a** in CH₃CN; (b) absorption spectral change of **2a** $(1.7 \times 10^{-5} \text{ mol } \text{L}^{-1})$ and **1** $(9 \times 10^{-6} \text{ mol } \text{L}^{-1})$ in degassed CH₃CN as a function of time with irradiation at λ >450 nm. Inset: the differential absorption spectra with irradiation time of 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 16, 18, 20, 25, 30, and 40 min, respectively (color online).

lexes 1 displays broad visible light absorption assigned to a mixture of metal-to-ligand and ligand-to-ligand charge transfer transitions ranging from 400 to 500 nm, while 2a does not absorb in this region, so a glass filter was used to cut off light below 450 nm with 500 W high-pressure Hanovia mercury lamp, thus only 1 was excited. Irradiation of 1 and 2a in degassed CH₃CN solution quickly decreased the absorbance at 295–400 nm for 2a, accompanied by a growth with a maximum at 260 nm, typical absorption of 1,3,5triphenylpyrazole (3a) in CH₃CN (Figure 1(b)). The welldefined isosbestic point at 295 nm suggests that both of 2a and **3a** are present in the solution. In addition, **1** is stable without any decomposition. This process was much clearer in the differential absorption spectra (inset, Figure 1(b)). As shown in Figure 2, the pyrazoline 2a displayed intense fluorescence with λ_{max} at 460 nm in CH₃CN at room temperature which originates from an intramolecular-charge-



Figure 2 Fluorescence change of **2a** as a function of time for the photoreaction of **2a** $(1.7 \times 10^{-5} \text{ mol } \text{L}^{-1})$ and **1** $(9 \times 10^{-6} \text{ mol } \text{L}^{-1})$ in degassed CH₃CN (λ_{ex} =360 nm). The time interval is 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 16, 18, 20, 25, 30, and 40 min, respectively (color online).

transfer, where the pyrazoles **3a** are non-emissive. Upon irradiation with λ >450 nm, the typical fluorescence of **2a** disappeared when the reaction was finished, that means the reaction could be pursued up to complete conversion of **2a** to **3a**.

This observation is also in line with ¹H NMR spectral change before and after irradiation by blue LEDs (λ_{max} =450 nm). As shown in Figure 3, the three typical alkyl protons in the 4- and 5-position of the pyrazoline ring (H_a, H_b, H_c in **2a**, respectively at 3.13, 3.91, 5.4 ppm) decreased with time, while new signal appeared at 6.98 ppm, which was in accord with the typical alkene proton in the 4-positon of pyrazole ring (H₄ in **3a**). Meanwhile, all aryl protons downfield shifted. In spite of the conversion changing with irradiation time, no secondary byproduct except H₂, which was confirmed by GC analysis, was detected throughout the reaction. The final ¹H NMR spectrum coincided completely with the pure **3a** prepared by the oxidation. On the basis of the consumption of the starting material of **2a**, the conversion of the photoreaction was up to 100%.

In contrast, irradiation of **2a** in the absence of complex **1** in CH₃CN at λ =450 nm resulted in no product formation. Moreover, no products could be obtained when the reaction was carried out in the dark. Evidently, both light and **1** are essential for the dehydrogenation. In addition, only a catalytic amount of **1** (3 mol% equiv.) significantly resulted in the photochemical reaction to form **3a**, and this transformation can be of preparative interest, because it occurs with high concentration (up to 10^{-2} mol L⁻¹).

With the optimized reaction conditions in hand, a variety of 1,3,5-triaryl-2-pyrazolines (**2b–2g**) were explored, and the results obtained are summarized in Table 1. The sub-



Figure 3 1 H NMR spectra of 1 and 2a in argon-flushed CD₃CN before (a) and after (b) irradiation. For the trace amount of 1, the spectra only show the disappearance of 2a and the appearance of 3a.

Table 1 Photocatalytic reaction of 1,3,5-triarylpyrrazolines by platinumnum(II) terpyridyl complex 1^{a_i}

ң _о н		H		
H Ar ₁	3 mol% 1	Ar_2 Ar_1	+	н.
N-N	CH ₃ CN, Ar	N-N	1	112
Ph´ 2	blue LED	Pn 3		

2			3			
Entry	Substrate	Ar ₁	Ar ₂	Time (h)	Conv. (%) ^{b)}	H ₂ (%) ^{c)}
1	2a	Ph	Ph	6	36	35
2	2a	Ph	Ph	12	100	98
3	2b	Ph	p-CH ₃ C ₆ H ₄	12	68	65
4	2b	Ph	p-CH ₃ C ₆ H ₄	24	100	-
5	2c	Ph	p-OCH ₃ C ₆ H ₄	12	60	60
6	2c	Ph	p-OCH ₃ C ₆ H ₄	24	100	-
7	2d	Ph	o-ClC ₆ H ₄	12	48	42
8	2d	Ph	o-ClC ₆ H ₄	24	85	-
9	2e	p-CH ₃ C ₆ H ₄	p-NO ₂ C ₆ H ₄	12	$55^{\text{ d}}$	0
10	2e	p-CH ₃ C ₆ H ₄	$p-NO_2C_6H_4$	24	90 ^{d)}	0
11	2f	p-CH ₃ C ₆ H ₄	2-furyl	12	26	15
12	2f	p-CH ₃ C ₆ H ₄	2-furyl	24	66	-
13	2g	p-CH ₃ C ₆ H ₄	2-thienyl	12	36	30
14	2g	p-CH ₃ C ₆ H ₄	2-thienyl	24	100	-

a) Conditions: **2** (0.017 mmol), 3 mol% **1** (0.5 μ mol), in CH₃CN (1 mL) under an argon atmosphere, irradiation with 3 W blue LEDs, r.t.; b) conversion of **3** were determined by ¹H NMR; c) GC yields using pure methane as an internal standard; d) trace amount of corresponding amino derivative **3i** were observed, Ar₂=*p*-NH₂C₆H₄.

stituents on pyrazolines caused the rate of dehydrogenation to decrease. With prolonged reaction time, electronic effects of the substituents on phenyl seemed not to affect the yield of the reaction very much, corresponding pyrazoles (**3b–3g**) was formed in good to excellent yield. In all cases the reaction was rather clean, except **2e** (Table 1, entries 9, 10), no H₂ obtained, and **3e** formed accompanied by a minor amount of the reduced amino derivative **3i**. The reaction time listed in Table 1 roughly indicated that the substituent of aryl groups Ar₂ in 5-position of 2-pyrazolines seemed to have a higher influence on the reactivity compared to Ar₁ in 4-position of 2-pyrazolines.

To understand the primary process of the photocatalytic reaction, we examined the interaction between 2 and complex 1. Complex 1 is emissive with λ_{max} at 628 nm in degassed CH₃CN at room temperature (Figure 4), which is readily quenched by 1,3,5-triaryl-2-pyrazoline of 2. The quenching process follows Stern-Volmer kinetics (Figure 4) and the quenching constants (k_q) were calculated from emission intensity measurements to be on the order of 10⁹ L mol⁻¹ s⁻¹ (Table 2). The emission quenching is due to electron transfer from the 1,3,5-triaryl pyrazoline substrate to the excited 1 since the singlet energy transfer from 2a-2g to the excited **1** is thermodynamically impossible with the energy of the singlet excited state of 1 much lower than that of 2a-2g. Calculation of the free energy change (ΔG) by the Rehm-Weller equation reveals that the electron transfer process from 2a–2g to the triplet 1 is exergonic (Table 2). As only



Figure 4 Emission spectra of complex 1 in degassed CH_3CN as a function of concentration of 2a. The inset shows the Stern-Volmer plot for luminescence intensity quenching of 1 by 2a.

Table 2 Data of k_q and ΔG for the photocatalytic conversion

	2a	2b	2c	2d	2e	2f	2g
$k_{\rm q} (\times 10^9 {\rm L} {\rm mol}^{-1} {\rm S}^{-1})$	7.3	8.9	11.6	9.9	4.8	10.1	8.0
$\Delta E_{\rm ox}$ (V) ^{a)}	1.11	1.16	1.12	1.16	1.17	1.10	1.16
$\Delta G (\mathbf{V})^{\mathbf{a}}$	-0.52	-0.47	-0.51	-0.47	-0.46	-0.53	-0.47

a) ΔG was calculated by equation: $\Delta G = \Delta E_{\text{ox}} - \Delta E_{\text{red}} - \Delta E_{0.0} - e^2/\epsilon a$, where ΔE_{ox} and ΔE_{red} versus NHE are oxidative and reductive potentials of **2** and **1**, respectively, which were measured by cyclic voltammetry in degassed CH₃CN solution with 0.1 mol L⁻¹ nBu₄NPF₆ as supporting electrolyte. ΔE_{red} is -0.53 V for **1** and $e^2/\epsilon a$ is 0.05 V in CH₃CN. $\Delta E_{0.0}$ refers to the lowest excited energy of **1** in CH₃CN (2.11 V).

the complex 1 was excited, it is therefore reasonable to consider that the photoinduced electron transfer from 2 to the excited 1 is responsible for luminescence quenching and the products generation.

Nanosecond transient absorption spectroscopy further confirms that the emission quenching is due to the electron transfer from 2 to the excited 1. Figure 5 shows the time-resolved transient absorption spectra observed following 355 nm excitation of 1, 1 with 2a, 2a, respectively. The spectrum exhibits strong transient absorption of the lowest ³MLCT state for 1 throughout all wavelengths examined (370-800 nm) and decays with a lifetime of 270 ns, matching that of the emission. When 2a was introduced into 1, the lifetime of **1** decreased and the ³MLCT absorption of **1** was replaced by a new bleaching in the region of 400-600 nm (Figure 5(b)). It is apparent that a strong similar bleaching for 2a emerged immediately under the same condition (Figure 5(c)). However, the pyrazoline-quenched sample does exhibit a maximum at 520 nm in the differential absorption spectra (Figure 5(d)) that is clearly not present in the transient absorption spectra of 2a. With reference to the detailed spectroscopic work by our group [24-26] and Schmehl et al. [27] on platinum(II) complexes, the observed intense peak around 520 nm was possibly resulted from the intermediate species formed by electron transfer. As in this case only **1** was excited, the PET from **2a** to **1** indeed takes place. According to the lifetimes of **1** with and without **2a** in CH₃CN, the rate constant for the PET process can be consequently obtained as $k_{\rm ET}=10.5\times10^9$ L mol⁻¹ s⁻¹, close to the diffusion limit. Affected by the strong fluorescence bleaching of **2a**, the lifetime of the newly formed intermediate species by photoinduced electron transfer could not be determined, nor the rate ($k_{\rm CR}$) for the back electron transfer process. Prolonged irradiation of **1** and **2** in CH₃CN led to no permanent change, indicating the reduced platinum species is stable.

Careful re-examination of this reaction for the hydrogen production revealed that the molar ratio of 3 to H_2 is approximate to 1:1 (Table 1), and the material balance is greater than 99%. On the basis of the above results, we may deduce that when 2 is used as a quencher, electron transfer pathway followed by proton-coupling pathway leading to five coordinated platinum(III)-H species are possible. In fact, the formation of 3 and H_2 by 1 supports an effective intramolecular H abstraction. On the basis of the above results, we proposed that the photocatalytic reaction is initiated by photoinduced electron transfer followed by proton-coupling process (Scheme 2). Under the irradiation condition, 2 not only serves as the electron donor to the excited complex 1 but also acts as proton donors to the photoreduced platinum center of 1. The generated platinum(III)-H species would consecutively react with the pyrazoline radical to form 3 and H₂ and simultaneously to regenerate



Figure 5 (a) Transient absorption spectra of **1** in CH₃CN at room temperature; (b) transient absorption spectra of **1** (5.0×10^{-5} mol L⁻¹) with **2a** (7.7×10^{-4} mol L⁻¹); (c) transient absorption spectra of **2a** in CH₃CN; (d) transient absorption spectra of **1** and **2a** (triangles, blue) in CH₃CN after subtraction of the **2a** spectrum. λ_{ex} =355 nm (color online).



Scheme 2 Proposed mechanism.

complex 1 and completes the photocatalytic cycle.

Conclusions 4

In conclusion, we have presented a new and environmentally benign methodology to prepare 1,3,5-triaryl pyrazoles. With visible light irradiation of the platinum(II) complex 1, the efficient photocatalytic conversion from readily available 1,3,5-triaryl pyrazolines (2a-2g) to the corresponding pyrazoles (3a-3g) is achieved. Spectroscopic study and product analysis demonstrate that the reaction is initiated by photoinduced electron transfer followed by proton-coupling process, as a result, leading to H_2 production and 1,3,5triaryl pyrazoles formation. In contrast to previous dehydrogen aromatization systems, the reaction was carried out in organic solvent and H₂ is generated directly from the substrates, and that makes this photocatalytic protocol particularly attractive, in particular, for the oxidation of pyrazolines that is unstable when traditional metal oxidants are used.

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Conflict of interest The authors declare that they have no conflict of interest.

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