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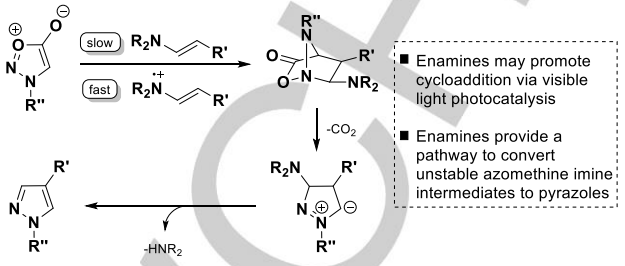
Exploiting Synergistic Catalysis for an Ambient Temperature Photocycloaddition to Pyrazoles**

Christopher P. Lakeland,[a] David W. Watson,[b] and Joseph P. A. Harrity*[a]

Abstract: Sydnone based cycloaddition reactions are a versatile platform for pyrazole synthesis, however they operate under harsh conditions (high temperature and long reaction times). Herein we report a strategy that addresses this limitation utilizing the synergistic combination of organocatalysis and visible light photocatalysis. This new approach proceeds under ambient conditions and with excellent levels of regiocontrol. Mechanistic studies suggest that photoactivation of sydnones, rather than enamines, is key to the successful implementation of this process.

Pyrazole containing molecules are prevalent within the medicinal and agrochemical industries,[1-3] and have been traditionally accessed by cycloaddition or condensation strategies.[4-5] Work within our laboratory has sought to establish sydnone cycloadditions[6] with alkynes as a versatile platform for the synthesis of pyrazole containing molecules. [7-14] While this transformation demonstrates a broad scope and is tolerant of a range of functionalities, there remain a number of associated challenges relating to reaction regiocontrol, as well as the requirement for elevated temperatures and prolonged reaction times. Recently, these problems have been addressed through the use of substrate directed cycloadditions,[14] strained alkynes[15,16] and copper promoter systems.[11,17,18] However, these methods still usually require specifically tailored substrates to proceed efficiently.

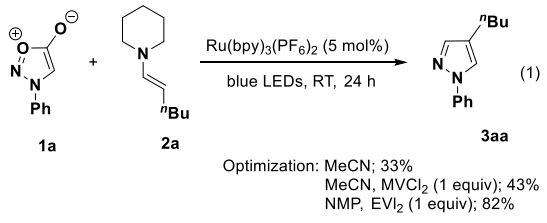
In an effort to conceive of new and alternative methods for the promotion of sydnone cycloaddition reactions, we became interested in the use of dienophiles that could be activated towards cycloaddition using visible light photocatalysis. Indeed, previous reports from Yoon and co-workers highlight the Diels-Alder reactions of electron rich dienes and dienophiles via the intermediacy of a radical cation.[19,20] An additional key consideration in the case of sydnones however, is that the initial cycloadduct undergoes rapid loss of carbon dioxide to produce a cyclic azomethine imine that can undergo several decomposition pathways. Enamines were thus identified as ideal reaction partners as: (1) they are known to undergo one electron oxidation in the presence of Ru-complexes and visible light;[21,22] (2) the amine unit could function as a leaving group that would allow the dipolar intermediate to convert to a stable pyrazole product

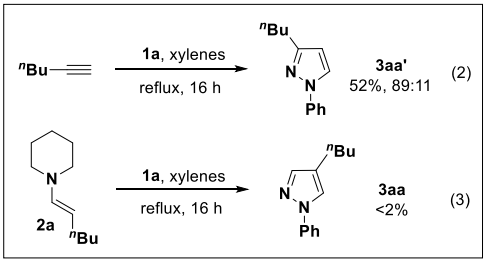


Scheme 1. Hypothesis for a photocatalytically promoted sydnone cycloaddition reaction.

(Scheme 1). Herein we report that sydnones participate in catalytic, visible-light promoted cycloaddition reactions with enamines to produce pyrazole products with excellent levels of regiocontrol.

We began our investigation of a potential photocatalytic sydnone-enamine cycloaddition (PSEC) reaction using *N*-phenylsydnone (**1a**) and *n*-butyl substituted enamine (**2a**) in acetonitrile at room temperature with $Ru(bpy)_3(PF_6)_2$ as the photocatalyst. Gratifyingly the reaction afforded 1,4-disubstituted pyrazole **3aa** in 33% yield as a single regioisomer. However, despite the low yield, analysis of the crude ^{1}H NMR spectrum showed complete consumption of sydnone **1a**. Speculating that sydnone decomposition was due to competing reduction by the photo-activated Ru-catalyst, we decided to add a competing 'electron sink'. Indeed, while the addition of methyl viologen dichloride ($MVCl_2$) resulted in only a slight improvement to the yield, it was accompanied by significantly less sydnone decomposition (43% yield at ~50% conversion of sydnone). Finally, a solvent screen identified NMP as the optimal co-solvent, and ethyl viologen diiodide (EVI_2) as a less expensive electron sink, allowing the product pyrazole to be generated in high yield (Scheme 2, see SI for full optimization). The unique reactivity offered by the combination of visible light photocatalysis and enamine dienophiles is highlighted in equations (1) and (2). Classical thermally promoted reactions of





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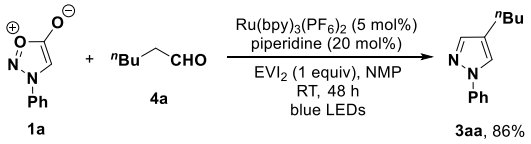
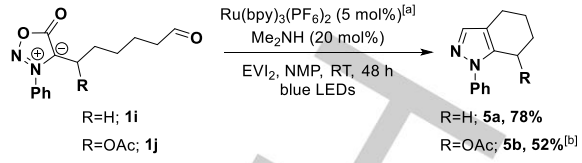
Scheme 2. PSEC reaction optimization and comparison to other methods.



Scheme 3. Amine catalyzed PSEC reaction.



Scheme 4. Intramolecular PSEC reaction. [a] Reaction conditions: sydnone (0.5 mmol), Me_2NH (0.1 mmol, 20 mol%), $Ru(bpy)_3(PF_6)_2$ (25 µmol, 5 mol%), EVI_2 (0.5 mmol), NMP (5 mL, 0.1 M), 48 h at room temperature under blue LED irradiation. [b] Reaction run at 0.025 M.

alkynes and sydnones requires long reaction times, high temperatures and provides the regioisomeric pyrazole **3aa'** as the major product. Moreover, enamines are not converted to pyrazoles under thermal conditions, even after heating over extended periods.

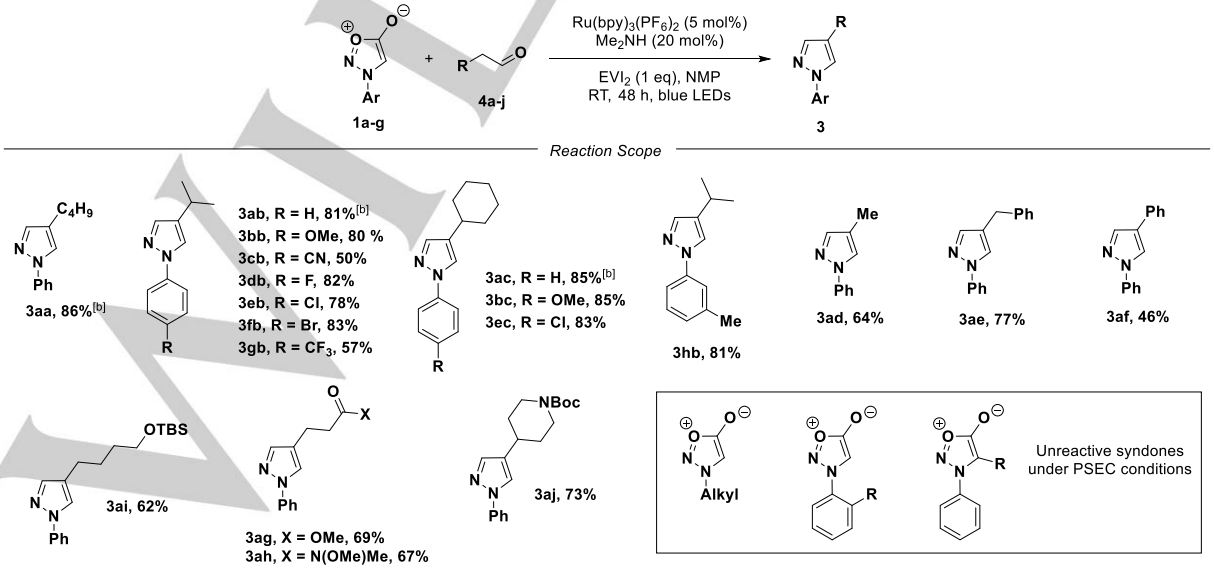
During our optimization studies, we noted that the pyrazole was generated alongside an aliphatic by-product that we assigned to the product of aldol condensation of enamine **2a**. Indeed, this by-product became significant when we undertook our PSEC reactions on larger scale, leading to lower isolated yields of pyrazole of around 50-60%. Working under the assumption that decreasing the concentration of enamine could limit this unproductive side reaction, we decided to perform the PSEC reaction on in situ generated enamine using sub-stoichiometric quantities of piperidine as an organocatalyst. To our delight, we were able to react sydnone **1a** directly with hexanal in the presence of 20 mol % piperidine to generate the desired sydnone in 86% yield (Scheme 3).

Having established successful conditions for an organocatalytic PSEC reaction, we next turned our attention to the reaction scope. Electronically rich and neutral *N*-aryl sydnones reacted smoothly to afford pyrazoles **3ab/3bb** in high yield and with complete regioselectivity. Furthermore, sydnones bearing electron withdrawing groups such as nitrile (**3cb**) and halides (**3db-3gb**) are tolerated, opening up the possibility for further functionalization of the pyrazole products. In addition to butyl and isopropyl moieties, various other alkyl groups including methyl (**3ad**), cyclohexyl (**3ac-3ec**) and benzyl (**3ae**) could be installed in high yield. The use of phenylacetaldehyde as a substrate allowed access to 1,4-diphenylpyrazole in moderate yield. Protected amines (**3aj**) and alcohols (**3ai**) were also tolerated and carbonyl derivatives including ester (**3ag**) and amide (**3ah**) had no adverse impact on the reaction.

Limitations to the method were also uncovered; while meta-substituted *N*-aryl sydnones were found to react smoothly (**3hb**), ortho-substituted analogues failed to react under the optimized conditions. Moreover, *N*-alkyl sydnones and 4-substituted sydnones were also found to be inert to intermolecular cycloaddition under PSEC conditions. However, we were able to apply the PSEC reaction to the synthesis of bicyclic pyrazole scaffolds by employing sydnone substrates bearing a tethered aldehyde moiety (Scheme 4). As such, pyrazoles **5a** and **5b** were synthesized in 78% and 52% yield, respectively.

We next investigated the mechanism of the PSEC reaction using various methods. Firstly, control and on/off experiments demonstrated the light promoted nature of the PSEC reaction (see supporting information). The existence of radical intermediates was then probed using several radical traps (Scheme 5). Interestingly, the yield of the PSEC reaction remained essentially unchanged in all cases and none of the expected radical combination adducts were observed.

Table 1. Scope of the organocatalytic PSEC reaction.[a]



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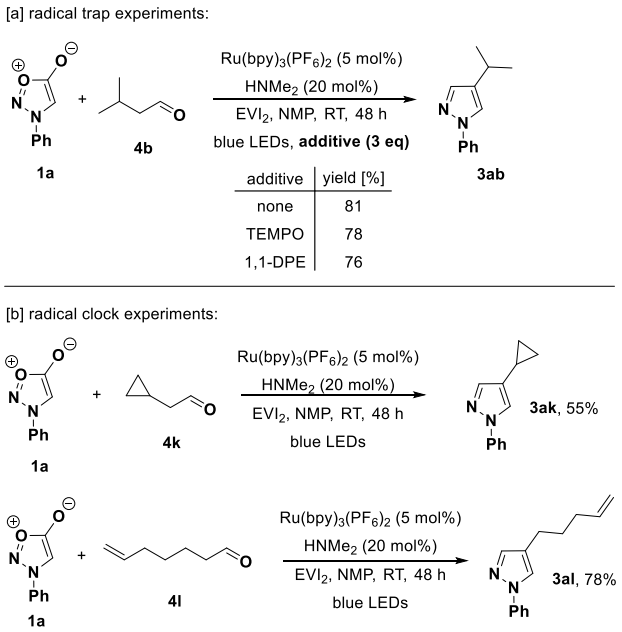
[a] Reaction conditions: sydnone (0.5 mmol), aldehyde (1.5 mmol), Me_2NH (0.1 mmol, 20 mol%), $Ru(bpy)_3(PF_6)_2$ (25 µmol, 5 mol%), EVI_2 (0.5 mmol), NMP (5 mL, 0.1 M), 48 h at room temperature under blue LED irradiation. [b] Piperidine (0.1 mmol, 20 mol%).



Scheme 5. Investigations into radical intermediate formation. [a] Radical trap experiments: **1a** (0.5 mmol), **4b** (1.5 mmol), Me_2NH (0.1 mmol), $Ru(bpy)_3(PF_6)_2$ (25 µmol), EVI_2 (0.5 mmol), additive (1.5 mmol) NMP (5 mL) under blue LED irradiation for 48 h. [b] Radical clock experiments: **1a** (0.5 mmol), aldehyde (1.5 mmol), Me_2NH (0.1 mmol), $Ru(bpy)_3(PF_6)_2$ (25 µmol), EVI_2 (0.5 mmol), NMP (5 mL) under blue LED irradiation for 48 h.

Additionally, radical clock experiments using cyclopropyl substituted aldehyde **4k** and tethered alkene containing aldehyde **4l** afforded the corresponding pyrazoles **3ak** and **3al** in good yield without any evidence of rearrangement. Overall these results suggest that formation of any radical species on the enamine is unlikely.[23]

Stern-Volmer luminescence quenching was then employed to determine the catalyst quenching species within the PSEC reaction. As expected, enamine **A** exhibited strong quenching of the photoexcited catalyst (K_{SV} = 9.08 x 10^1 M^{-1}) while the corresponding aldehyde **B** provided no observable decrease in luminescence. Surprisingly, *N*-phenylsydnone **C** also gave a prominent quenching of the photocatalyst (K_{SV} = 2.01 x 10^2 M^{-1}) while *N*-benzylsydnone **D** (unreactive under PSEC conditions) gave, by comparison, negligible quenching (Figure 1). In an attempt to understand the role of sydnone based luminescence quenching we performed photocatalytic cycloaddition reactions with other dipolarophiles; specifically, those which cannot quench the excited state photocatalyst. The reaction of *N*-phenylsydnone and 1-hexyne[24] under photocatalytic conditions failed to afford any of the desired pyrazole **3aa**, but 2,3-dihydrofuran (E_{red} ~ +1.5 V vs SCE)[22] reacted with *N*-phenylsydnone to afford pyrazole **3am** in 38% yield (Scheme 6). This result demonstrates that photocatalytic activation of the sydnone partner alone is sufficient for a successful cycloaddition reaction. Taken together with the improved yield under organocatalytic conditions and superior rate of quenching for *N*-phenylsydnone over enamine **A**, these results indicate that the sydnone substrate is likely the major photocatalyst quenching species under the PSEC reaction conditions.[25]

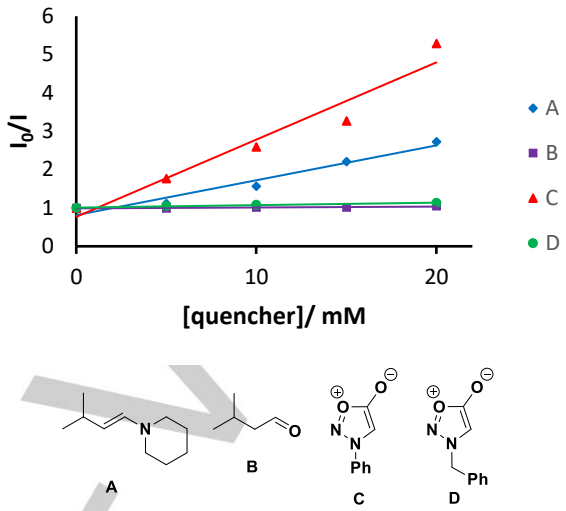


Figure 1. Luminescence quenching of $Ru(bpy)_2(PF_6)_2$.

In addition, cyclic voltammetry was used to determine the mode of sydnone based luminescence quenching. Voltammetric measurements of *N*-phenylsydnone discovered an irreversible oxidation peak ($E_{p/2}$ = + 1.48 vs Fc/Fc^+) and irreversible reduction peak ($E_{p/2}$ = - 1.87 vs Fc/Fc^+), indicating that an electron transfer event between *N*-phenylsydnone and $Ru(bpy)_3^{2+}$ would be significantly endergonic (see supporting information). Therefore, it seems likely that the fluorescence quenching of $Ru(bpy)_3^{2+}$ by *N*-phenylsydnone operates via a photosensitisation mechanism.[26] Finally, we sought to determine the fate of the photoexcited sydnone species generated during the course of the reaction. Previous reports on UV promoted sydnone cycloadditions indicate that product formation occurs via the reaction of an in-situ formed nitrilimine dipole following a decarboxylative rearrangement of the sydnone.[27] In the case of C4-unsubstituted sydnone this would lead to the sydnone hydrogen atom ending up on either the C3 or C5 position of the pyrazole product, depending on whether the photoexcited sydnone undergoes decarboxylative rearrangement or a direct cycloaddition reaction, respectively (Scheme 7[a]). In the event, deuterated sydnone **3k** underwent a successful PSEC reaction to produce pyrazole **3kb** in 87% yield with no evidence of a decarboxylative rearrangement (Scheme 7[b]).

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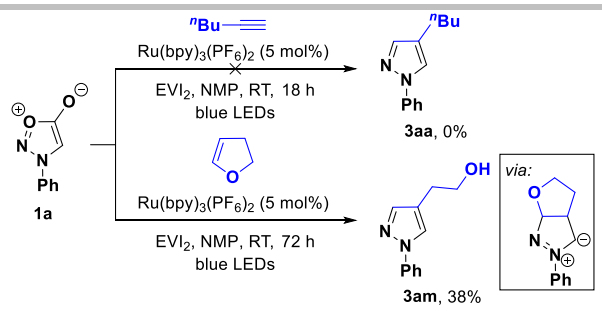
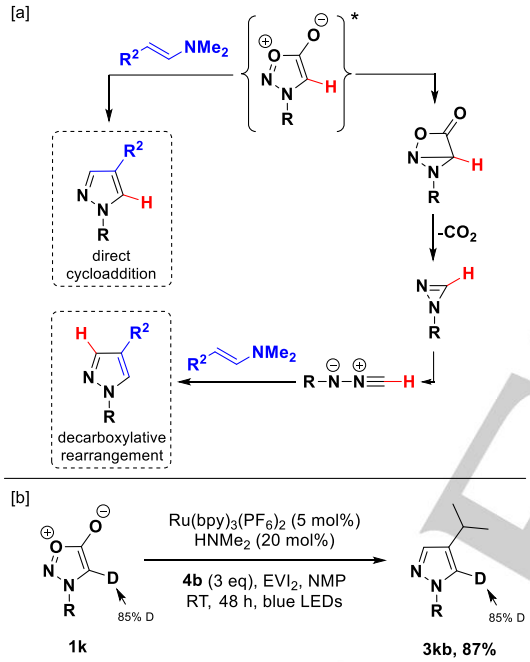


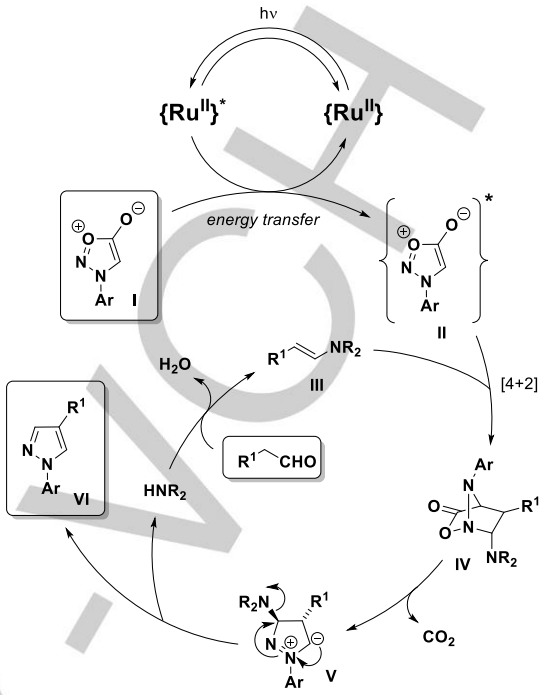
Scheme 6. Photocatalytic cycloaddition with other dipolarophiles. Reaction conditions: **1a** (0.5 mmol), dipolarophile (1.5 mmol), $Ru(bpy)_3(PF_6)_2$ (25 µmol), EVI_2 (0.5 mmol), NMP (5 mL, 0.1 M) at room temperature under blue LED irradiation.



Scheme 7. Investigating the fate of photoexcited sydnone.

Based on the combination of these results we propose the mechanism shown in Scheme 8. The cycle begins with an energy transfer from the excited state photocatalyst to sydnone I.[28] Excited sydnone II can then undergo a [4+2] cycloaddition reaction with enamine III to generate the bridged intermediate IV. Rapid loss of CO_2 from IV leads to the dipolar intermediate V which can then undergo an elimination event to deliver the pyrazole product VI and regenerate the amine organocatalyst.

In conclusion, a visible light promoted synthesis of pyrazoles has been developed. The reaction takes advantage of a synergistic combination of photo- and organocatalysis to promote a cycloaddition between sydnones and enamines under mild conditions and with excellent regioselectivity. This process represents the first photocatalytic cycloaddition reaction involving sydnones. Preliminary mechanistic investigations indicate that an energy transfer between the excited state photocatalyst and sydnone substrate is crucial for the observed reactivity.



Scheme 8. Proposed mechanism of the PSEC reaction.

Experimental Section

Typical photocatalytic cycloaddition procedure as exemplified by the formation of 3aa: A flame-dried screw cap vial was charged with *N*-phenylsydnone (81 mg, 0.5 mmol), $Ru(bpy)_3(PF_6)_2$ (21 mg, 25 µM) and EVI_2 (230 mg, 0.5 mmol) under nitrogen. Anhydrous, degassed NMP (5.0 mL) was then added via syringe followed by hexenal (150 mg, 1.5 mmol) and dimethylamine (0.05 mL, 2.0 M in THF). The vial was then subjected to three vacuum/nitrogen cycles, sealed and irradiated using a kessil A160WE tuna blue aquarium light for 48 hours. The reaction was then diluted with water (40 mL) and extracted with diethyl ether (4 x 25 mL). The combined organic layers were then washed with brine (25 mL), dried over anhydrous magnesium sulfate and concentrated under vacuum. Purification by flash silica column chromatography (gradient from 0-5% Et_2O in 40-60 petroleum ether) afforded **3aa** as a yellow oil (86 mg, 86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 0.5 Hz, 1H), 7.69 – 7.64 (m, 2H), 7.55 (s, 1H), 7.46 – 7.39 (m, 2H), 7.28 – 7.21 (m, 1H), 2.53 (t, *J* = 7.5 Hz, 2H), 1.65 – 1.56 (m, 2H), 1.45 – 1.35 (m, 2H), 0.95 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.3, 140.6, 129.6, 126.2, 125.0, 124.3, 119.0, 33.2, 24.2, 22.6, 14.2; FTIR ν_{max} 2927, 2855, 1599, 1502, 1464, 1395, 1041, 1015, 952, 753 cm^{-1}.

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[24] Cyclic voltammetry of aliphatic alkynes showed no oxidation/reduction peaks within the electrochemical window of acetonitrile.

[25] Under our optimized conditions, an excess of sydnone should be present relative to in situ generated enamine until high reaction conversions. Given the superior rate of quenching by sydnone relative to enamine, we believe that the mesoionic reagent should dominate the excited catalyst quenching step. Furthermore, if photoredox mediated oxidation of the enamine is responsible for formation of the aldol dimerization product, then decreasing enamine concentration under organocatalytic conditions could be responsible for minimizing the formation of this side-product.

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[28] Recording a UV-vis spectrum of **1a** allowed us to estimate its singlet excited state energy through a λ_{tail} analysis, which was found to be 3.42 eV, a significantly larger value than the triplet excited state energy of 2.12 eV for the excited Ru catalyst. This experiment effectively rules out energy transfer to the singlet excited state of the sydnone and so our current working hypothesis is that energy transfer to the sydnone triplet excited state is the most likely mode of activation (see Supporting Information). We would like to thank one reviewer for helpful discussions on the possible mechanism of sydnone photoactivation.

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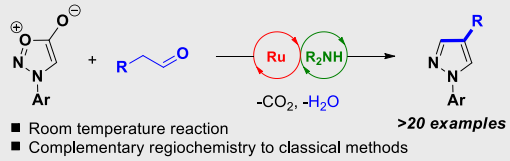

A photo finish! The synergistic combination of organocatalysis and visible light photocatalysis offers a mild route to pyrazoles that proceeds under ambient conditions and with excellent levels of regiocontrol.

*Christopher P. Lakeland, David W. Watson, Joseph P. A. Harrity**

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Exploiting Synergistic Catalysis for an Ambient Temperature Photocycloaddition of Sydnones