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Catalytic Asymmetric Dearomatization by Visible-Light-Activated [2+2] Photocycloaddition

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Abstract: A novel method for the catalytic asymmetric dearomatization by visible-light-activated [2+2] photocycloaddition with benzofurans and one example of a benzothiophene is reported, providing chiral tricyclic structures with up to four stereocenters including quaternary stereocenters. The benzofurans and the benzothiophene are functionalized at the 2-position with a chelating N-acylpyrazole moiety which permits the coordination of a visible light activatable chiral-at-rhodium Lewis acid catalyst. Computational molecular modeling revealed the origin of the unusual regioselectivity and identified the heteroatom in the heterocycle to be key for the regiocontrol.

Aromatic and heteroaromatic compounds are ubiquitous synthetic starting materials and many types of reactions have been developed for their functionalization and transformation. A recently emerging highly useful class of reactions are catalytic asymmetric dearomatizations (CADA reactions), a term coined by the You group.^[1-3] This synthetic methodology is highly appealing because it converts readily available aromatic moieties into enantio-enriched three-dimensional cyclic molecules in a catalytic fashion, which is an important objective in contemporary organic synthesis.

Asymmetric cycloadditions enable a straightforward access to complex architectures with multiple stereocenters in a single step and thereby generate structural complexity in a rapid and economical fashion.^[4] Recently, Bach,^[5] Yoon,^[6] and our groups^[6c,7] reported visible-light-activated catalytic asymmetric [2+2] photocycloadditions^[8,9] which occur directly from an electronically excited substrate/catalyst complex without the involvement of charge separation. This chemistry is attractive because it employs visible light as an abundant and mild source of energy and at the same time circumvents drawbacks resulting from radical ion intermediates which are typically generated in the course of photoredox processes.^[10]

Herein we report the first application of visible-lightactivated [2+2] photocycloadditions to catalytic asymmetric dearomatizations and we investigate the observed regioselectivity by computational modeling (Figure 1).[11,12]

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Novel method for catalytic asymmetric dearomatization Benzofuran & benzothiophene for asymmetric [2+2] photocycloaddition Visible light and mild conditions

Figure 1. Catalytic asymmetric dearomatization by visible-light-activated [2+2] photocycloaddition reported in this study and comparison with state of the art.

We started our study with benzofurans^[13] functionalized in 2-position with 2-acylimidazole (1a,b) or N-acylpyrazole (1c-h) moieties for interacting with the photoactivatable chiral-at-metal Rh catalyst Δ -RhS^[14] (Table 1).^[15,16] Initial experiments were disappointing; when 2-acyl imidazoles (1a or 1b) with styrene 2a were subjected to blue LEDs together with \triangle -RhS (2 mol%), nearly no conversion occurred (entries 1,2). Gratifyingly, when N-acylpyrazole 1c was employed instead, 68% conversion was observed for the dearomative [2+2] photocycloaddition and the main product 3c was formed with 97% ee together with diastereomer 3c' (5.4:1 dr) and regioisomer 3c" (7.3:1 rr) (entry 3). To our surprise, the main product features a head-to-tail regioselectivity in contrast to a tail-to-tail regioselectivity observed in related previous rhodium-catalyzed [2+2] photocycloadditions.^[7]

The substituents at the pyrazole have a profound effect on this reaction (entries 4-8). The best results were obtained with a Ph group in 3-position of the pyrazole (1f) which gave complete conversion after 18 hours of irradiation and afforded the main product 3f with 98% ee together with diastereomer 3f' (6.2:1 dr) and regioisomer 3f" (5.3:1 rr) (entry 6). Due to the lability of the N-acylpyrazole moiety in this structural context, a prior conversion to its methyl ester allowed to isolate the main product together with its regioisomer in an overall yield of 78%. The reaction is quite robust and performs well under air (entry 9). However, the choice of solvent is important and the best results are obtained with CH₂Cl₂ (compare entries 6, 10 and 11). The catalyst loading can be reduced to 1 mol% with almost unchanged performance (entry 12). As a control, in the absence of light or catalyst, no conversion was observed (entries 13,14). Likewise, the related Ir complex Δ -IrS^[17] was not able to catalyze this reaction (entry 15).

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Table 1. Initial experiments and optimization of conditions.



Entry	Х	Reaction conditions ^(a)	Conv. (%) ^[0]	dr ^[c]	rr ^{iaj}	ee (%) ^[e]
1	1a	standard	< 5	_	_	_
2	1b	standard	< 5	_	—	_
3	1c	standard	68	5.4 : 1	7.3 : 1	97
4	1d	standard	89	4.7 : 1	4.3 : 1	98
5	1e	standard	< 5	_	—	_
6	1f	standard	100 (78) ^[f]	6.2 : 1	5.3 : 1	98 ^[g]
7	1g	standard	98	6.1 : 1	5.0 : 1	98
8	1h	standard	100	5.9 : 1	4.3 : 1	82
9	1f	under air	98	5.7 : 1	4.7 : 1	97
10	1f	acetone as solvent	65	3.4 : 1	5.0 : 1	95
11	1f	THF as solvent	49	4.0 : 1	3.4 : 1	92
12 ^[h]	1f	1.0 mol% ∆-RhS	100	6.1 : 1	5.1 : 1	98
13	1f	no light	0	_	_	_
14	1f	no catalyst	0	_	—	-
15	1f	2.0 mol% ∆- IrS	0	_	_	_

[a] Deviations from standard conditions are shown. Standard conditions: Benzofuran **1a-h** (0.1 mmol), styrene **2a** (0.3 mmol) and 2.0 mol% Δ -**RhS** in CH₂Cl₂ (0.1 M, 1 mL) were stirred at r.t. under N₂ for 18 h with blue LEDs (24 W) irradiation. [b] Conversion determined by ¹H NMR of the crude product. [c] Dr values determined by ¹H NMR of the crude product. [d] Rr values determined by ¹H NMR of the crude product. [e] Ee values determined by HPLC analysis on chiral stationary phase. [f] Total isolated yield of the main product **4a** with its regioisomer **4a**" (6.7:1 rr) after the conversion to the corresponding methyl esters. [b] Reaction time was 36 h.

With optimized reaction conditions in hand, we next investigated the scope of substituted styrenes and determined isolated yields after conversion of the initial *N*-acylpyrazole products into their methyl esters (Figure 2). Overall, electron-withdrawing and electron-donating groups as well as sterically bulky groups in *para*- and *meta*-position of the phenyl moiety are well tolerated; the dearomatization products **4b-g** were obtained with 96-98% ee and 68-88% yields as single diastereomers but as regioisomeric mixtures (4.3:1 to >20:1 rr). The phenyl moiety can also be replaced with a naphthyl (**4i**) or a thiophene moiety (**4j**). However, *ortho*-substituents strongly affect the reactivity so that for 2-methylstyrene (**4h**) the catalyst loading was doubled to 4 mol% in order to obtain satisfactory results.

Generally, the reaction is expected to proceed as seen in previous Rh catalyzed [2+2] photocycloadditions as summarized in Figure 3.^[7,18] The catalytic cycle begins with the association of the *N*-acylpyrazole substrate (¹1f) with the chiral Rh catalyst Δ -**RhS**. The reactant complex ¹A absorbs blue light to reach its singlet excited state, ¹A*. After intersystem crossing (ISC) to form the triplet reactant complex ³A, it reacts with the alkene substrate to generate the 1,4-biradical intermediate ³B, which then recombines to form the desirable photocycloaddition

product.^[19] The subsequent release of product **3f** and coordination of a new equivalent of substrate complete the catalytic cycle. Our calculations confirm the location of the unpaired electrons across the ene moiety of photoexcited ³**A** as a 1,2-biradical species, as indicated with red dots in Figure 3, which reacts with the alkene substrate in a stereocontrolled fashion. During this process, the enantioselectivity^[20] is well-controlled by the chiral Rh catalyst (see SI for a structure of the transition state ³**A**-TS) and the regioselectivity is probably determined by the stability of the 1,4-biradical intermediate although other factors such as the rate of the back reaction to the ground state precursors have been determined to play an important role.^[9b,21,22]







Figure 3. Proposed catalytic cycle.

We sought to understand the origin of the unusual regioselectivity of this dearomatization using DFT calculations for the reaction with catalyst Δ -**RhS**, substrate **1f**, and styrene (Figure 4).^[23] Once the substrate-bound Rh complex ¹A is photoexcited and undergoes ISC into the triplet state to form the reactive 1,2-biradical species ³A, the styrene substrate can approach the 1,2-biradical from two possible faces. Since the

formation of the benzyl radical is much more favorable than the generation of the methyl radical, we focused on identifying the transition state that couples the terminal carbon of styrene with one of the 1,2-biradical carbons in the benzofuran fragment to afford a 1,4-biradical product. As illustrated in Figure 4a, the head-to-tail addition (solid black line) has a barrier that is 2.0 kcal/mol lower in energy than the tail-to-tail addition (dashed, blue line). The thermodynamic stability of two analogous 1,4-biradical intermediates shows the same trend, as ³B is 5.5 kcal/mol lower in free energy than ³B". To complete the cycloaddition, the 1,4-biradical species must undergo radical recombination and cross-over to the singlet surface to produce ¹C and ¹C", respectively. These processes will occur at the minimum energy crossing point (MECP), where the triplet and singlet surfaces cross.

The observation that styrene engages the 1,2-biradical with the terminal methylene moiety to generate a benzyl radical is plausible. However, the energy difference of 5.5 kcal/mol between the 1.4-biradical intermediates ³B and ³B" is not as easy to understand, but it is a key feature leading to the regioselectivity. To separate the impact of the chiral Rh-fragment from the intrinsic energy differences of the two possible intermediate isomers, we deleted the Rh-fragment and evaluates the relative energies of the two biradicals, labeled as ³b and ³b". Interestingly, our calculations indicate that ³b is 6.2 kcal/mol lower in energy than ³b", as illustrated in Figure 4b. An inspection of the underlying electronic structure reveals a simple reason for this energy difference, outlined in Figure 4c. When the unpaired electron is placed at the benzylic C3-position, it is resonance-stabilized by the phenyl group. At the tertiary C2position, however, the unpaired electron is stabilized by the neighboring carbonyl group and, more importantly, by the oxygen lone-pair orbital, shown in blue and red in Figure 4c, respectively.^[24] A more detailed energy decomposition is provided in the SI. Thus, the calculations suggest that the regiocontrol is based on an intrinsic electronic preference of placing an unpaired electron of the 1,4-biradical intermediate at the tertiary carbon next to an oxo functionality.

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Figure 4. (a) Gibbs free energy profile. (b) Energy differences of two analogous 1,4-biradical intermediates. (c) Schematic illustrations of the radical stabilizations.

Finally, we investigated reactions with internal alkenes which provide an additional stereocenter. To our delight, when (E)- β -methylstyrene 5a was tested with benzofuran 1f and subsequently converted to its methyl ester, the dearomatization product 6a was obtained in 90% yield and 99% ee as a single diastereomer (> 20:1 dr) and as a single regioisomer (> 20:1 rr) (Figure 5). With the geometrical isomer (Z)- β -methylstyrene 5a' the dearomatization product 6a' was formed as the main product instead, albeit with a low dr of 1.8:1. Gratifyingly, when performed at -30 °C, only a single diastereomer was obtained (>20:1 dr). This strong temperature dependence of the diastereoselectivity supports a mechanism through an intermediate but very short-lived 1,4-biradical intermediate in which the temperature affects the rotation around the single C-C bond before the radical recombination occurs and thus conserves the memory of the radical conformation.[25]

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Figure 5. Asymmetric dearomatization with (*E*)- and (*Z*)- β -methylstyrene. For the reaction at -30 °C, 5 W blue LEDs were employed (see SI for setup) and reaction time was 60 h. The absolute configuration of a derivative of **6a** was determined by X-ray crystallography. The relative configuration of its diastereoisomer **6a**' was determined by NMR studies.

Encouraged by these results, the substrate scope with internal (*E*)-alkenes was investigated (Figure 6). A variety of substituents at the benzene moiety of the benzofurans were well tolerated (**6b-g**) and the methyl group of (*E*)- β -methylstyrene could also be replaced by long-chain and functionalized alkyl groups without affecting the outcome (**6h-i**). All these dearomatization products were formed with high yields (80-93%), excellent enantioselectivities (98-99% ee) and virtually with complete diastereo- and regioselectivities, except for **6g** (16.7:1 dr). This catalytic asymmetric dearomatization can also be applied to benzothiophene^[26] to give the dearomatization product **6j** in 87% yield with >20:1 dr and >20:1 rr, albeit with a diminished enantioselectivity (73% ee).



Figure 6. Substrate scope with internal alkenes. The absolute configuration of a derivative of 6j was determined by X-ray crystallography and all other compounds assigned by analogy.

In summary, we developed the first example of catalytic asymmetric dearomatizations by visible-light-activated [2+2] photocycloadditions and investigated the observed regioselectivity with computational molecular modeling. With internal alkene substrates, almost perfect head-to-tail regioselectivity was observed with the formation of a single diastereomer and a very high enantioselectivity for benzofuran substrates of 98-99% ee to provide. The *N*-acylpyrazole moiety of the tricyclic structures with up to four stereocenters including one quaternary stereocenter can be easily further functionalized (see SI for examples), thus indicating the potential synthetic value of this new methodology.^[27]

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Keywords: photocycloaddition • dearomatization • asymmetric catalysis • chiral-at-metal

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- [27] The reaction can be scaled up to 1 mmol scale without a significant change in performance as demonstrated for the conversion $1f+5a\rightarrow 6a$ (see SI for details).

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