

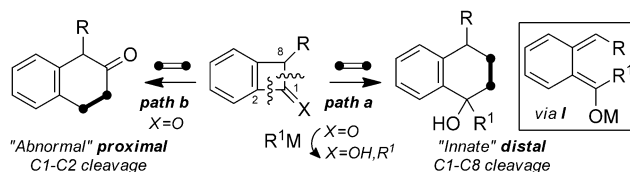
Nickel-Catalyzed Chemo-, Regio- and Diastereoselective Bond Formation through Proximal C–C Cleavage of Benzocyclobutenones**

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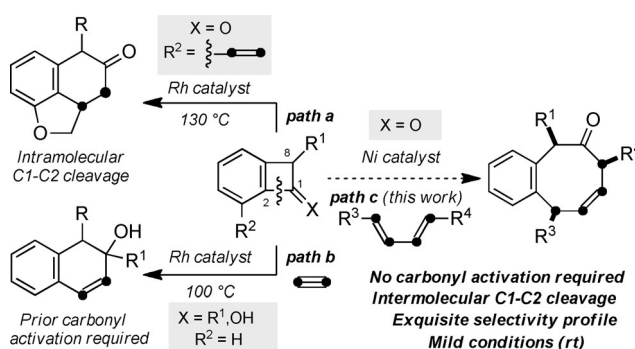
Abstract: The first catalytic intermolecular proximal C1–C2 cleavage of benzocyclobutenones (BCB) without prior carbonyl activation or employing noble metals has been developed. This protocol operates at room temperature and is characterized by an exquisite chemo-, regio- and diastereoselectivity profile, constituting a unique platform for preparing an array of elusive carbocyclic skeletons.

The development of metal-catalyzed C–C bond cleavage reactions has recently received considerable attention, holding great promise to novel syntheses of complex molecules.^[1] Among these, molecules such as cyclobutanes possessing a high ring strain have shown to be particularly effective for such purposes, providing the necessary driving force for the targeted C–C bond cleavage.^[2] Although oftentimes visualized as exotic cyclobutane analogues, benzocyclobutenones (BCB) have shown to be superb reaction intermediates in many transformations,^[3] even in the context of natural product synthesis.^[4] At present, the reactivity of BCB remains primarily confined to their proclivity for distal C1–C8 cleavage by retro-4 π cyclization (Scheme 1, via **1**).^[3] Unfortunately, such scenarios commonly require an initial pre-activation of the carbonyl group (*path a*), thus constituting a drawback from a practical and step-economical standpoint.^[3]

Despite the advances realized, the means to alter the innate distal C1–C8 cleavage of BCB has been virtually unexplored (Scheme 1, *path b*), thus representing a unique opportunity for chemists to increase our ever-expanding chemical portfolio and flexibility in synthetic design. Recently, Dong^[5] and Murakami^[6] independently reported an elegant solution to tackle the “abnormal” proximal C1–C2



Scheme 1. Innate reactivity of BCB for C1–C8 cleavage.



Scheme 2. Catalytic proximal C1–C2 cleavage strategies.

cleavage by intramolecular techniques (Scheme 2, *path a*)^[5] or by prior carbonyl activation using alkyne partners (Scheme 2, *path b*).^[6,7] While no doubt a significant step forward, these techniques unfortunately require expensive noble metals (Rh) and high temperatures, illustrating the inertness of proximal C1–C2 bonds.^[8] Ideally, this field should include unconventional intermolecular proximal C1–C2 cleavage strategies that operate under mild conditions without employing noble metals or prior carbonyl activation platforms, thus opening up new vistas in the C–C cleavage arena. Although we anticipated that such scenario would be far from trivial due to the ability of closely related unsaturated cyclobutenones to trigger catalytic distal C–C cleavage with exceptional ease,^[9] we were attracted to the challenge. As part of our interest in BCB^[10] and C–C bond-cleavage,^[11] we report herein the first intermolecular bond formation through proximal C1–C2 cleavage of BCB using Ni⁰ catalysts and without prior activation of carbonyl groups (Scheme 2, *path c*). This method is inherently modular, allowing for preparing a variety of otherwise inaccessible carbocyclic skeletons depending on the synthon utilized, including elusive benzofused eight-membered rings.^[12,13] The reaction operates at room temperature, constituting the lowest temperature achieved for catalytic C–C cleavage events of BCB reported to date. Importantly, our proximal C1–C2 cleavage event is

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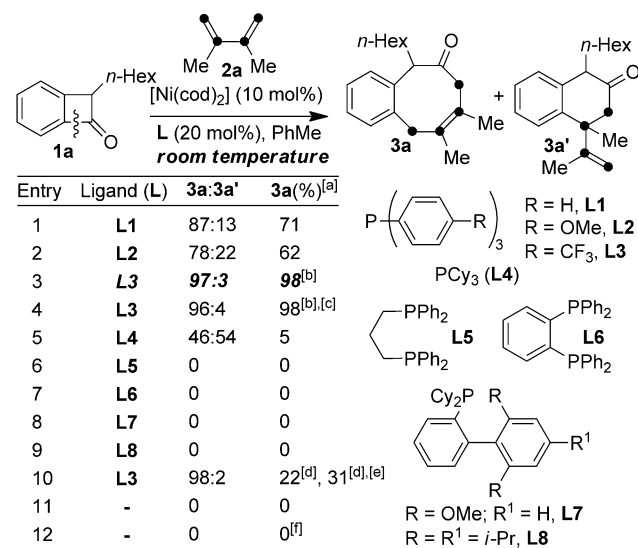
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distinguished by an exquisite chemo-, regio- and diastereoselectivity profile.

We started our investigations by evaluating the reaction of **1a**^[14] with **2a** en route to rather elusive benzofused eight-membered rings (**3a**), molecules that display significant biological properties.^[15] After systematic evaluation of all reaction parameters, we identified promising results with [Ni(cod)₂] (cod = cyclooctadiene) as precatalyst at room temperature (Scheme 3). While the utilization of **2a** as

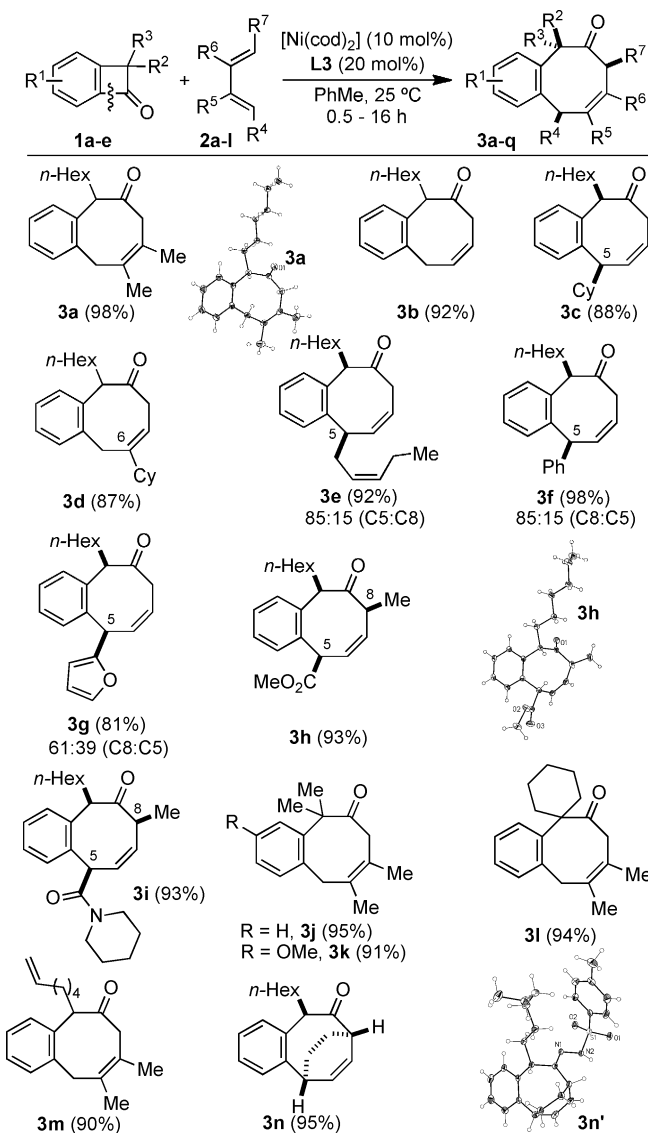


Scheme 3. Optimization of the Ni-catalyzed [4+4]-cycloaddition by proximal C1–C2 cleavage of **1a**. Reaction conditions: **1a** (0.35 mmol), **2a** (0.45 mmol), [Ni(cod)₂] (10 mol%), ligand (20 mol%), toluene (2 mL), 25 °C, 16 h. [a] GC yield using dodecane as internal standard. [b] Isolated yield, 30 min. [c] No solvent. [d] L3 (10 mol%). [e] [Ni(cod)₂] (5 mol%). [f] Conditions of Ref. [5] and [6].

a two-carbon synthon might result in a formal [4+2]-cycloaddition (**3a'**), we anticipated that the nature of the ligand might exert a profound influence on both reactivity and selectivity. As shown in Scheme 3, this turned out to be the case. Among all the ligands examined, we found that **L3** was particularly suited for our purposes, giving rise to **3a** in 98% yield in 30 min with traces of **3a'** in the crude mixtures (entry 3).^[16] Notably, the reaction could even be performed in the absence of solvent in identical yield (entry 4). Interestingly, the inclusion of electron-rich triarylphosphines significantly eroded the selectivity profile (entry 2).^[17] Likewise, little reactivity, if any, was observed when operating with bulkier ligands or bidentate phosphines (entries 5–9), thus suggesting an intimate interplay of stereoelectronic effects on the ligand backbone with both reactivity and selectivity. As expected, control experiments revealed that all reaction parameters were critical for success (entries 10 and 11). In order to put these results into perspective, we found no reaction under previously reported Rh-catalyzed conditions for proximal C1–C2 cleavage with π -systems (entry 12).^[5,6] We believe these results show the unique features of our novel Ni-catalyzed intermolecular protocol compared to other

distal bond-cleavage events,^[9] intramolecular reactions^[5] or by prior carbonyl activation (Scheme 2, *paths a* and *b*).^[6]

We next turned our attention to examine the scope of our Ni-catalyzed intermolecular [4+4]-cycloaddition through proximal C1–C2 bond cleavage. As shown in Scheme 4,

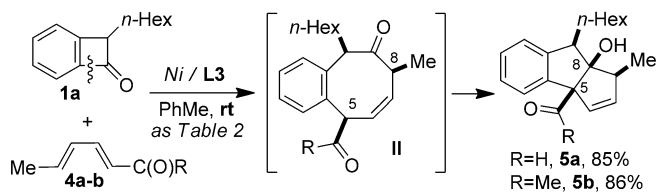


Scheme 4. [4+4]-cycloaddition by Ni-catalyzed proximal C–C cleavage at room temperature. Reaction conditions: as for Scheme 3, entry 3; Isolated yields, average of at least two independent runs. In all cases analyzed, traces (< 5%) of [4+2]cycloadducts were observed in the crude mixtures.

substrates with aromatic as well as aliphatic backbones on the 1,3-diene motif reacted equally well under our optimized protocol. Importantly, the reaction showed an excellent chemoselectivity profile, as heterocycles (**3g**), amides (**3i**), esters (**3h**) and alkenes (**3e**, **3m**) were perfectly accommodated. Although we anticipated selectivity issues with unsymmetrical 1,3-dienes,^[11] we found an unexpected exquisite regio- and diastereoselectivity profile. Specifically, a single diastereoisomer with *syn* configuration was obtained in all

cases analyzed (**3c–3g**). Notably, the electronic nature of the substituents on the 1,3-diene core did not exert a profound influence on regioselectivity, invariably locating the larger residues at either C5 or C6 (**3c–3i**). While tentative, these results suggest that steric effects might dictate the regioselectivity pattern.^[18] Such argument goes in line with the observation that **3e–3g** were obtained as mixtures of regioisomers with a slight preference for C5 selectivity.^[19] Notably, the presence of a pending alkene on both BCB (**2m**) and 1,3-diene core (**2e**) did not result in a competitive intramolecular [4+2]-cycloaddition. Of particular interest was the observation that unsymmetrically disubstituted 1,3-dienes could be coupled with excellent yields and as single regio- and diastereoisomers (**3h** and **3i**), with **3h** unambiguously characterized by X-ray analysis.^[20,21] Gratifyingly, cyclic 1,3-dienes could also participate in the ring-expansion event (**3n**); in sharp contrast with acyclic 1,3-dienes, however, we observed an opposite *anti*-diastereoselectivity pattern.^[22] Although such observation was evident by extensive NMR spectroscopical studies, we univocally assigned the structure of **3n** by comparison with the corresponding tosyl hydrazone **3n'** by X-ray crystallography.^[23]

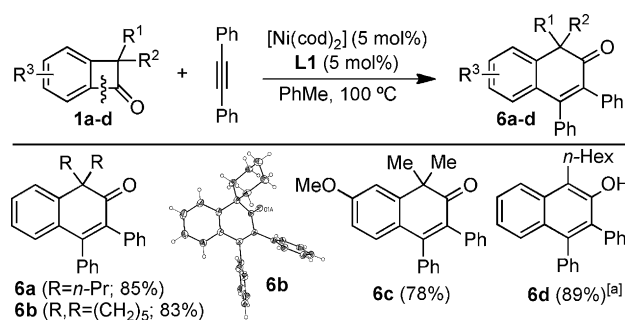
In light of these results, we wondered whether our Ni-catalyzed intermolecular proximal C1–C2 bond cleavage of BCB could be utilized for the synthesis of other polycyclic skeletons. Specifically, we hypothesized that intramolecular transannular aldol reactions^[24] from in situ generated benzo-fused eight-membered rings **II** (Scheme 5) would be within



Scheme 5. Tricyclic skeletons by C1–C2 bond cleavage of **1a**.

reach when utilizing 1,3-dienes substituted with carbonyl groups with relatively acidic α -hydrogens. As shown in Scheme 5, this was indeed the case, obtaining exclusively tricyclic **5a** and **5b** as single regioisomers and diastereoisomers under otherwise identical reaction conditions to that shown in Scheme 4.^[25,26]

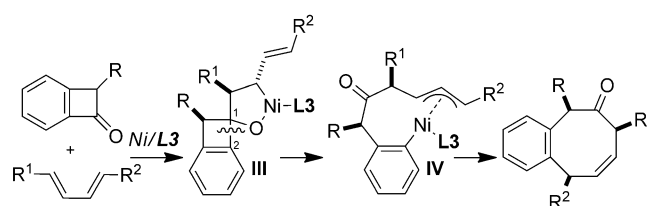
As becomes evident from the results compiled in Scheme 6, [4+2]-cycloadducts could easily be obtained upon exposure of **1a–1d** with alkyne counterparts^[27] to a protocol based on a $[\text{Ni}(\text{cod})_2]/\text{L1}$ couple. By definition, such transformation constitutes an straightforward alternative to the elegant [4+2]-cycloaddition technique recently reported by Murakami using BCB and noble Rh catalysts, but without requiring prior activation of the carbonyl group (Scheme 2, *path b*).^[6,28] In all cases analyzed, **6a–6d** were obtained in high yields as single regioisomers resulting from a proximal C1–C2 bond cleavage event, as illustrated by X-ray crystallographical analysis of **6b**.^[29] In all cases analyzed, not even traces of the corresponding distal C–C cleavage were detected in the crude reaction mixtures.^[9] Interestingly, spontaneous aromatization



Scheme 6. [4+2]-Cycloaddition by Ni-catalyzed proximal C1–C2 cleavage. Conditions: **1a–d** (0.35 mmol), diphenylacetylene (0.525 mmol), $[\text{Ni}(\text{cod})_2]$ (5 mol%), PPh_3 (5 mol%), in toluene at 100 °C. Isolated yields, average of at least two independent runs.^[c] $[\text{Ni}(\text{cod})_2]$ (10 mol%), PPh_3 (10 mol%).

occurred with α -monosubstituted BCB, hence leading the exclusive formation of naphthol derivatives (**6d**). Overall, we believe the results in Scheme 4–6 tacitly suggest that non-precious Ni catalysts might pave the way for preparing a priori inaccessible polycyclic structures by unconventional C–C bond cleavage disconnections.

Although a full mechanistic picture should await further studies, we propose a mechanistic scenario consisting of an initial coordination of the 1,3-diene and BCB to a low-valent Ni complex that precedes oxidative cyclization (**III**, Scheme 7).^[30,31] A subsequent β -carbon elimination event



Scheme 7. Mechanistic rationale.

from either **III** or in situ generated seven-membered metal-lacycle^[32,33] might lead to the formation of putative π -allyl Ni^{II} metallacycle (**IV**) that ultimately undergoes reductive elimination to form the corresponding benzofused eight-membered ring.^[34] An otherwise similar rationale might account for [4+2]-cycloadducts utilizing alkyne counterparts (Scheme 6).^[35]

In conclusion, we have described the first intermolecular catalytic bond formation by proximal C1–C2 cleavage of benzocyclobutenones without prior carbonyl activation or employing noble metals. This procedure is characterized by its exceptional mild conditions and exquisite chemo-, regio- and diastereoselectivity profile using non-precious Ni catalysts, allowing for the preparation of a variety of otherwise inaccessible carbocyclic skeletons in a straightforward manner. The asymmetric version and further investigations of other related transformations are currently underway in our laboratories.

Keywords: benzocyclobutenones · C–C activation · nickel catalysis · ring strain

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