Asymmetric Synthesis

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Enantio- and Diastereoselective Synthesis of *exo*-Peroxyacetals: An Organocatalyzed Peroxyhemiacetalization/oxa-Michael Addition Cascade

*Sanjay Maity, Biswajit Parhi, and Prasanta Ghorai**

Abstract: *An unprecedented enantioselective peroxyhemiacetalization/oxa-Michael addition cascade of ortho-formyl homochalcones has been developed using cinchona-alkaloid-based chiral bifunctional organocatalysts to provide cis-configured exo-peroxyacetals, a new class of organic peroxide, in good yields with excellent enantio- and diastereoselectivities. The resulting cis-configured exo-peroxyacetals were converted into the corresponding trans-configured peroxyacetals without affecting the enantioselectivity. Furthermore, the displacement of the peroxide moiety of exo-peroxyacetals with various nucleophiles has been demonstrated to afford 1,3-disubstituted isochromans with high diastereoselectivities and excellent enantioselectivities.*

The design and synthesis of structurally distinct classes of chiral organic peroxides have always remained attractive because of their considerable appearance in natural products and bioactive molecules.[1–5] However, to date, the corresponding catalytic enantioselective strategies for the synthesis of chiral peroxides are less explored.[6–9] Moreover, as per our knowledge, there is still a scarcity of methods for the catalytic enantio- and diastereoselective synthesis of peroxides.

Inspired by the antimalarial drug candidate artemisinin (Figure 1),[10] having a peroxyacetal pharmacophore, several peroxyacetals have been evaluated for antimalarial,[11] antitumor, anti-HIV, and anti-hepatitis B activity.[12] However, enantioselective methods for the synthesis of chiral peroxyacetals are exceedingly rare. In this regard, the pioneering reports by Rovis and co-workers for the synthesis of *endo*-peroxyacetals are remarkable.[9] However, to the best of our knowledge, the synthesis of enantio- and diastereoselective *exo*-peroxyacetals has not yet been established.[13]

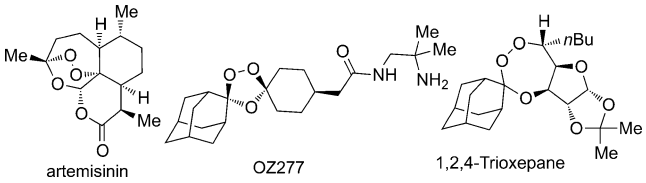
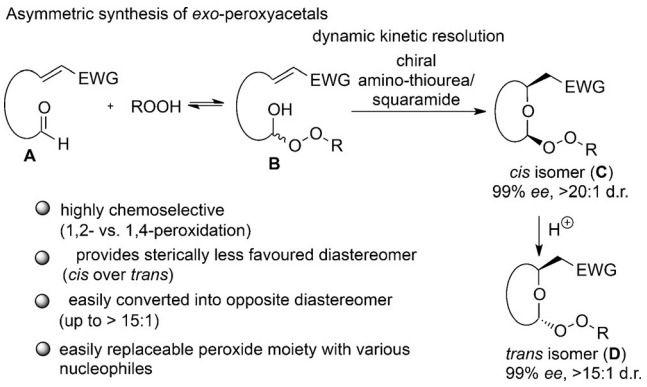


Figure 1. Natural products and bioactive molecules possessing a chiral peroxyacetal pharmacophore.

We envisioned a peroxyhemiacetalization/oxa-Michael addition cascade of the substrate **A** (Scheme 1), where the reversibly formed peroxyhemiacetal intermediate **B** could be converted into the oxa-Michael adduct **C** by a dynamic kinetic resolution process catalyzed by a chiral amino-thiourea/squaramide catalyst.[14,15] Nevertheless, overcoming the direct conjugate addition of peroxide on an α,β-unsaturated moiety remains a potential barrier to this strategy.[16] Further, both enantioselective acetalization[15,17] and oxa-Michael reactions[18–21] are challenging tasks because of their inherent reversibility. Herein we report the very first synthesis of *exo*-peroxyacetals (**C**) in excellent enantio- and diastereoselectivity, thus providing the sterically hindered *cis*-stereoisomer. Furthermore, it can also be converted into the *trans*-stereoisomer **D** by a simple acid-catalyzed process without loss in enantioselectivity.



Scheme 1. Catalytic enantio- and diastereoselective synthesis of the *exo*-peroxyacetals. EWG = electron-withdrawing group.

We began our investigation of the *ortho*-formyl homochalcone **1a** as a starting substrate, aimed at the synthesis of the *exo*-peroxyacetal-containing isochroman **3a** (see Table 1). Notably, the isochroman units are frequently found in variety of natural products and bioactive molecules.[22,23] Moreover, the targeted *exo*-peroxyacetals could potentially be converted into other important bioactive cores which are shown in Figure 2.

By using *t*BuOOH as peroxide source, a variety of chiral cinchona alkaloid derived catalysts (**2a–g**) were surveyed in toluene as the solvent at room temperature (Table 1, entries 1–5). As shown in entry 5, the catalyst **2g** was found to catalyze the reaction cleanly to furnish the *exo*-peroxy-

[*] S. Maity, B. Parhi, Dr. P. Ghorai
Department of Chemistry, Indian Institute of Science Education and Research (IISER) Bhopal
Bhopal By-pass Road, Bhauri, Bhopal-462066 (India)
E-mail: pghorai@iiserb.ac.in

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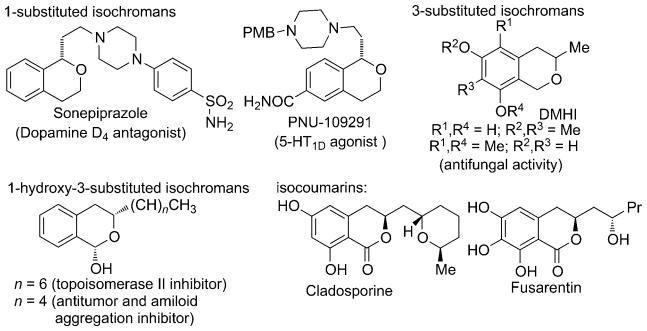
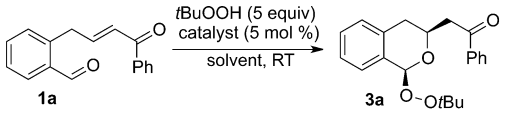
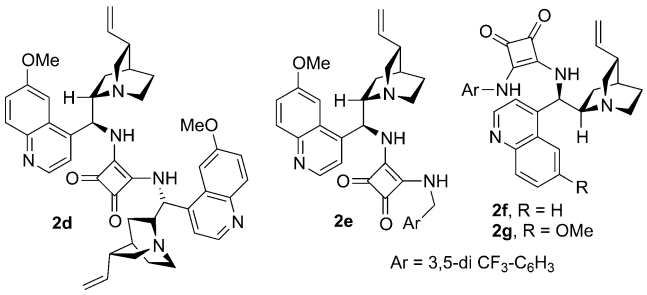


Figure 2. Natural products and bioactive molecules possessing 1- and 3-substituted isochromans, 1-hydroxy-3-substituted isochromans, and isocoumarines.

Table 1: Optimization of reaction conditions[a]



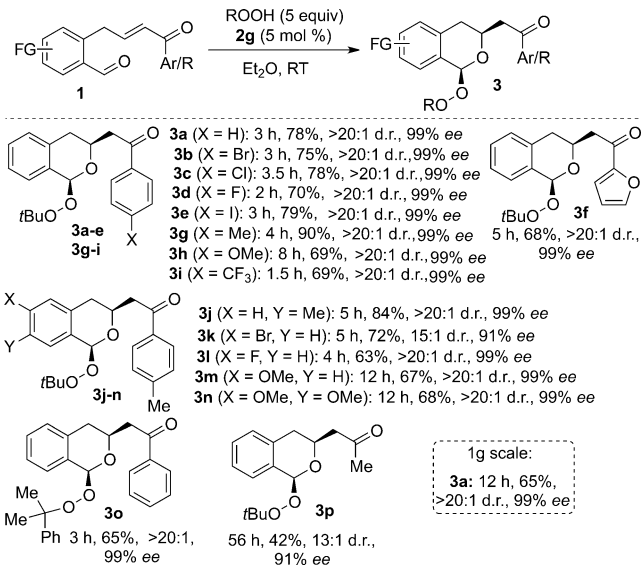
(DHQ)$_2$Pyr (**2a**) (DHQ)$_2$PHAL (**2b**) (DHQ)$_2$AQN (**2c**)



Entry	2	Solvent	*t* [h]	Yield [%][b]	d.r.[c]	*ee* [%][d]
1	**2a–c**	toluene	12	–	–	–
2	**2d**	toluene	12	35	19:1	40
3	**2e**	toluene	12	55	19:1	93
4	**2f**	toluene	12	55	13:1	62
5	**2g**	toluene	7	70	21:1	99
6	**2g**	Et_2O	4	74	24:1	99

[a] Reactions were performed on a 0.02 mmol scale of aldehyde. [b] Yield was calculated based on ^{1}NMR spectroscopy of the crude reaction mixture using diphenyl acetonitrile as the internal standard. [c] The diastereomeric ratio was determined by ^{1}H NMR analysis of the crude reaction mixture. [d] Enantiomeric excess was determined by HPLC analysis using a chiral stationary phase.

acetal **3a** (70% yield; NMR) with excellent diastereo- and enantioselectivity (>20:1 d.r. and 99% *ee*). Further, screening of various organic solvents (see the Supporting Information) using **2g** revealed that Et_2O is the best solvent as it led to an improved yield (74%), as well as diastereoselectivity (>20:1 d.r.; entry 6). However, the corresponding thiourea catalysts were not suitable here (see the Supporting Information). Notably, replacement of hydroperoxide with an alcohol such as MeOH provided the corresponding *exo*-acetal, which was found to be unstable (see the Supporting Information).



Scheme 2. Substrate scope. Reaction conditions: **1** (0.1 mmol), *t*BuOOH (0.5 mmol, 5 equiv), **2g** (0.005 mmol, 5 mol%) in Et_2O at RT. The diastereomeric ratio (d.r.) was determined by ^{1}H NMR analysis of the unpurified reaction mixtures. Unless stated otherwise, yields are of pure isolated major diastereomers. The *ee* value of the major diastereomer was determined by chiral-phase HPLC analysis. FG = functional group.

Using these optimized reaction conditions, we explored the substrate scope, which proved to be quite general (Scheme 2). Excellent yields, diastereoselectivities (>20:1 in all cases), and enantioselectivities (almost all cases 99% *ee*) were obtained with substrates containing substituted aryls (**3a–e** and **3g–i**) and heteroaryl (**3f**) groups. Furthermore, substitutions on the central aryl moiety (**3j–n**) were well tolerated. Replacement of *t*BuOOH with cumene hydroperoxide provided the corresponding peroxide **3o** in excellent stereoselectivity. Not only aryl moieties on the α,β-unsaturated ketone, but also an alkyl group was also tolerated (**3p**). However, a decrease in yield and stereoselectivity was observed. Even upon scaling up (1 g, 4 mmol) the reaction of **1a**, the corresponding product **3a** was obtained without any loss of stereoselectivity. The *cis* stereochemistry of the product was confirmed based on the crystal structure of **3c** (Figure 3).

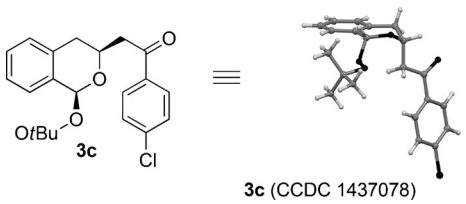
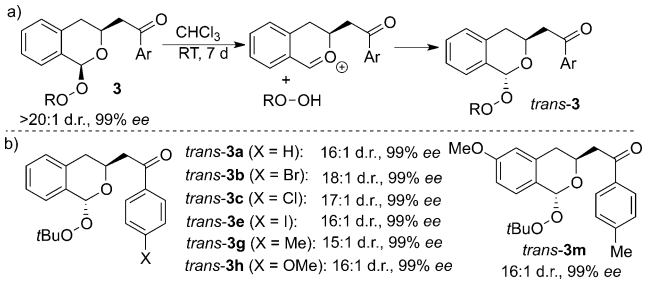


Figure 3. Crystal structure of **3c**.[24]

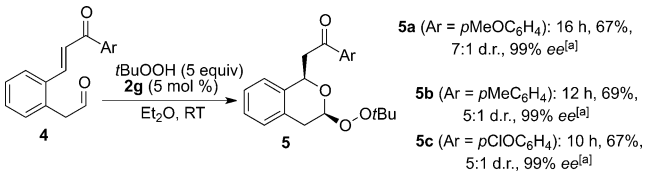
An epimerization at the peroxyacetal center was observed when the synthesized *cis* products were stored in either commercial $CHCl_3$, CH_2Cl_2, or $CDCl_3$ (Scheme 3a). Presumably, the trace acid content in the solvent is responsible for



Scheme 3. Epimerization of peroxyacetals. The diastereomeric ratio was determined by ^{1}H NMR analysis. The *ee* value was determined by HPLC analysis using a chiral stationary phase.

such a phenomenon, thus leading to the corresponding sterically stable *trans* isomers via the formation of an oxocarbenium ion. However, such epimerization was suppressed by using acid-free solvents (passed through a plug of basic alumina). As shown in Scheme 3b, various *cis* stereoisomers were converted into the corresponding *trans* isomers with excellent d.r. values (>15:1), without affecting the enantioselectivity.

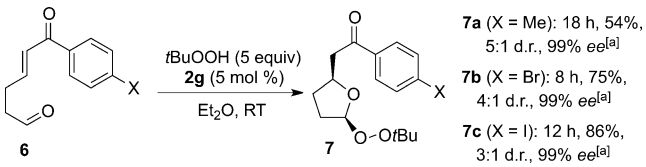
To explore the generality of the reaction conditions, *ortho*-homoformyl chalcones (**4**) were examined (Scheme 4). In general, the reactions worked equally well, but the diastereoselectivities remained moderate.



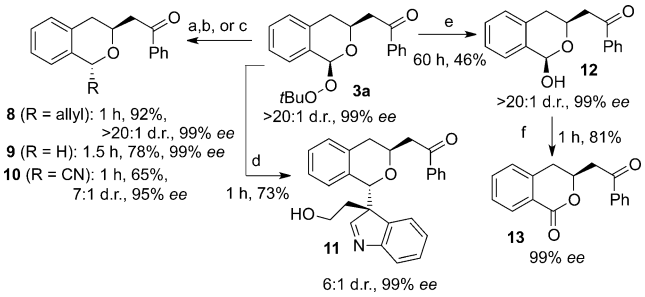
Scheme 4. Substrate scope: 1,3-Disubstituted isochromans. For reaction conditions, see: Scheme 2. [a] The *ee* value of the major diastereomer.

Next, we turned our attention to the corresponding cyclization of substrates without a phenyl linker (Scheme 5). 5-Peroxy 2-substituted tetrahydrohydrofurans (**7a–c**) can be synthesized with good yields and excellent enantioselectivities. However, moderate diastereoselectivities were observed.

Intrigued by the recurrent existence of 1-/3-substituted isochromans and isocoumarines in natural products and bioactive molecules (Figure 2),[22] the versatility and potential application of the stereoenriched *exo*-peroxyacetals were illustrated for the site-selective transformations to construct



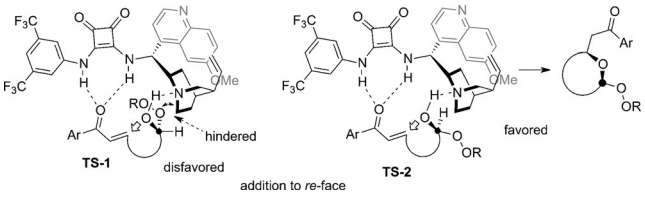
Scheme 5. Substrate scope: Aliphatic linker. For reaction conditions, see: Scheme 2. [a] The *ee* value of the major diastereomer.



Scheme 6. Late-stage manipulation of the products. Reaction conditions: a) $In(OTf)_3$ (2 mol%), allyltrimethylsilane (1.5 equiv), dry CH_2Cl_2, RT. b) Catechol borane (3 equiv), dry toluene, 0°C to RT. c) $In(OTf)_3$ (2 mol%), TMSCN (1.2 equiv), CH_2Cl_2, 0°C to RT. d) $In(OTf)_3$ (10 mol%), 3-ethanol indole, dry CH_2Cl_2, 0°C to RT. e) PPh_3 (2 equiv), CH_2Cl_2, RT. f) IBX (1.2 equiv), EtOAc/DMSO (9:1), reflux. DMSO = dimethylsulfoxide, IBX = *o*-iodoxybenzoic acid, Tf = trifluoromethanesulfonyl, TMS = trimethylsilyl.

such chiral building blocks (Scheme 6). For instance, Lewis acid catalyzed allylation, hydride addition, cyanation, and indole addition enabled the selective conversion of a C–OO bond into either a C–C bond or C–H bond without loss of enantiopurity, thus, 1,3-disubstituted isochromans (**8**, **10**–**11**) were obtained with a high level of optical purity. Notably, the current protocol could be superior compared to our previous report in terms of enantioselectivity for the synthesis of the 3-substituted isochroman **9**.[21,23] It is noteworthy that such substitution of a peroxide moiety is not usual in literature. Furthermore, the decomposition of a peroxy linkage can give the corresponding 1-hydroxy-3-substituted isochromans (**12**). Subsequently, oxidation of a hydroxy into a carbonyl functionality provides the chiral isocoumarine core **13**. And the corresponding aliphatic peroxy-tetrahydrofurans **7a–c** could be a potential substrate for enantiopure γ-lactones. The absolute stereochemistry of **9** was confirmed by the correlation with the literature value.[21b]

To explain the observed absolute stereochemical outcome, a bifunctional mechanism similar to those previously proposed for the squaramide/thiourea-catalyzed oxa/aza-Michael reaction of enones[15,19–21] may be invoked (Scheme 7). The *re*-face of the enone in either **TS-1** or **TS-2** is in perfect alignment to drive the formation of the product with the desired stereochemistry. As shown in **TS-1**, the OOR group at the β-position imposes a steric hindrance with the bicyclic skeleton of the catalyst, thus prohibiting the interaction of the catalyst with the substrate. Whereas, in **TS-2** the OOR group is situated away from the bicyclic skeleton, and thus does not hinder the catalyst–substrate association.



Scheme 7. Proposed model for enantioselectivity-determining step.

In summary, a sequential peroxyhemiacetalization followed by an enantioselective intramolecular oxa-Michael reaction of *ortho*-formyl homochalcones and *ortho*-homoformyl chalcones have been developed using a chiral bifunctional organocatalyst. This process provides the very first, promising approach for the synthesis of *exo*-peroxyacetals with excellent enantio- and diastereoselectivities. The methodology contributes to the development of new catalytic asymmetric protocols for the synthesis of chiral peroxides. Based upon this oxa-Michael reaction of peroxyhemiacetals, the synthesis of chiral lactones is under active consideration in our group.

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[24] CCDC 1437078 (**3c**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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