

Asymmetric Synthesis

Deutsche Ausgabe: DOI: 10.1002/ange.201511165
Internationale Ausgabe: DOI: 10.1002/anie.201511165Enantio- and Diastereoselective Synthesis of *exo*-Peroxyacetals: An Organocatalyzed Peroxyhemiacetalization/oxa-Michael Addition Cascade

Sanjay Maity, Biswajit Parhi, and Prasanta Ghorai*

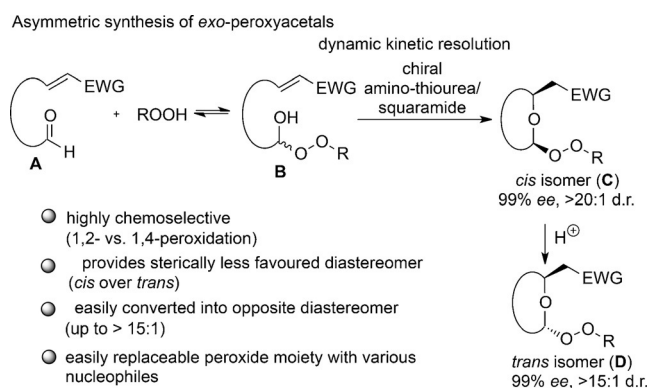
Abstract: An unprecedented enantioselective peroxyhemiacetalization/oxa-Michael addition cascade of *ortho*-formyl homochalcones has been developed using cinchona-alkaloid-based chiral bifunctional organocatalysts to provide *cis*-configured *exo*-peroxyacetals, a new class of organic peroxide, in good 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] anti-tumor, 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]

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.

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-

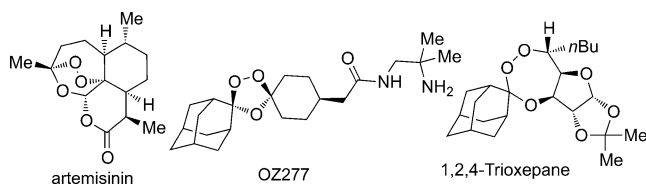


Figure 1. Natural products and bioactive molecules possessing a chiral peroxyacetal pharmacophore.

[*] 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

Supporting information for this article can be found under <http://dx.doi.org/10.1002/anie.201511165>.

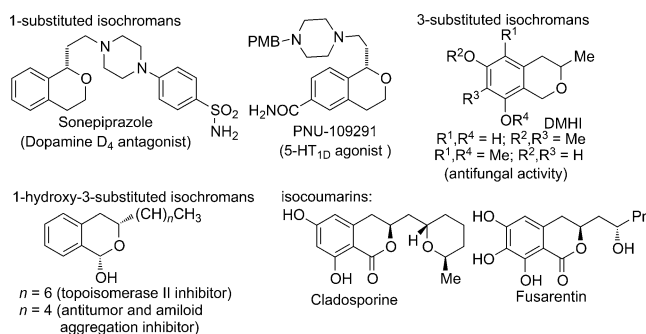
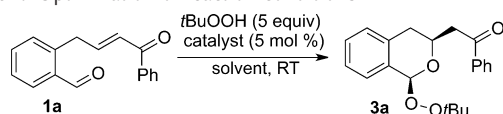
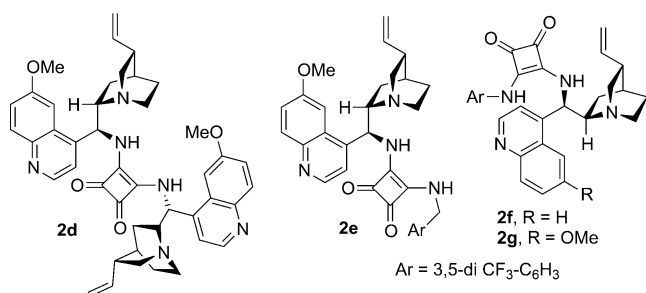


Figure 2. Natural products and bioactive molecules possessing 1- and 3-substituted isochromans, 1-hydroxy-3-substituted isochromans, and isocoumarines.

Table 1: Optimization of reaction conditions^[a]



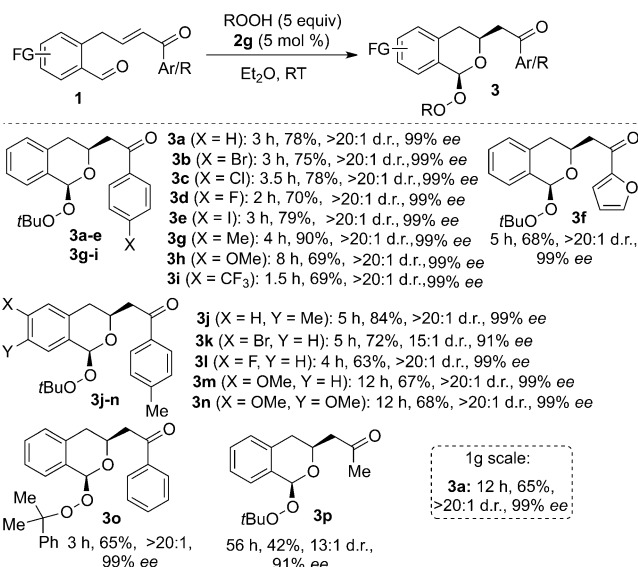
(DHQ)₂Pyr (**2a**) (DHQ)₂PHAL (**2b**) (DHQ)₂AQN (**2c**)



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 ₂ O	4	74	24:1	99

[a] Reactions were performed on a 0.02 mmol scale of aldehyde. [b] Yield was calculated based on ¹H NMR spectroscopy of the crude reaction mixture using diphenyl acetonitrile as the internal standard. [c] The diastereomeric ratio was determined by ¹H 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 Et₂O 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 Et₂O at RT. The diastereomeric ratio (d.r.) was determined by ¹H 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).

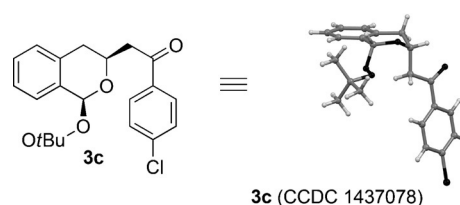
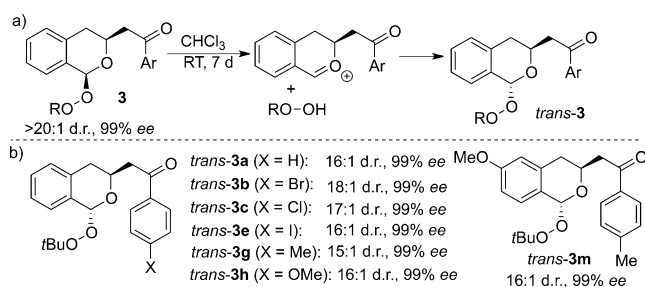


Figure 3. Crystal structure of **3c**.^[24]

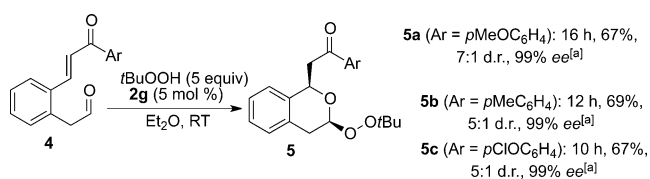
An epimerization at the peroxyacetal center was observed when the synthesized *cis* products were stored in either commercial CHCl₃, CH₂Cl₂, or CDCl₃ (Scheme 3a). Presumably, the trace acid content in the solvent is responsible for



Scheme 3. Epimerization of peroxyacetals. The diastereomeric ratio was determined by ^1H 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.

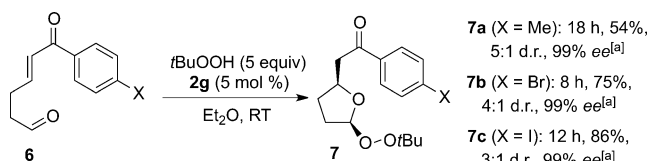
To explore the generality of the reaction conditions, *ortho*-homoformyl chalcones (**4**) were examined (Scheme 4). In general, the reactions worked equally well, but the diastereoselectivities remained moderate.



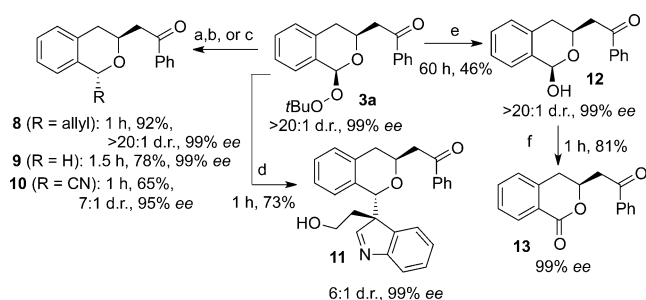
Scheme 4. Substrate scope: 1,3-Disubstituted isochromans. For reaction conditions, see: Scheme 2. [a] The ee value of the major diastereomer.

Next, we turned our attention to the corresponding cyclization of substrates without a phenyl linker (Scheme 5). 5-Peroxy 2-substituted tetrahydrofuran (**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 isocoumarins 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



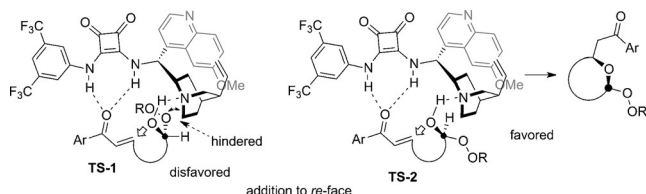
Scheme 5. Substrate scope: Aliphatic linker. For reaction conditions, see: Scheme 2. [a] The ee value of the major diastereomer.



Scheme 6. Late-stage manipulation of the products. Reaction conditions: a) In(OTf)₃ (2 mol %), allyltrimethylsilane (1.5 equiv), dry CH₂Cl₂, RT. b) Catechol borane (3 equiv), dry toluene, 0°C to RT. c) In(OTf)₃ (2 mol %), TMSCN (1.2 equiv), CH₂Cl₂, 0°C to RT. d) In(OTf)₃ (10 mol %), 3-ethanol indole, dry CH₂Cl₂, 0°C to RT. e) PPh₃ (2 equiv), CH₂Cl₂, 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 isocoumarin 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*-homochalcones 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.

Acknowledgements

This work has been funded by CSIR, New Delhi and IISER Bhopal. S.M. and B.P. thank UGC and CSIR, respectively, New Delhi, India for the fellowship. We are thankful to Mr. L. M. Jha and Ms. A. Upadhyay for the crystal analysis. The support from Mr. Barnala Ravindra (IISER-B) is acknowledged.

Keywords: asymmetric synthesis · heterocycles · Michael addition · organocatalysis · peroxides

How to cite: *Angew. Chem. Int. Ed.* **2016**, *55*, 7723–7727
Angew. Chem. **2016**, *128*, 7854–7858

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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.

Received: December 2, 2015

Revised: January 4, 2016

Published online: February 19, 2016