

# A Modular Approach to Catalytic Stereoselective Synthesis of Chiral 1,2-Diols and 1,3-Diols

Corresponding Author: Professor Wangqing Kong

This manuscript has been previously reviewed at another journal. This document only contains information relating to versions considered at Nature Communications.

**This file contains all reviewer reports in order by version, followed by all author rebuttals in order by version.**

Version 0:

Reviewer comments:

Reviewer #1

(Remarks to the Author)

Based on their point-by-point response letter, the authors have conducted additional experiments to enhance the quality of the work. Their responses to the reviewers' questions are highly informative, providing valuable insights and context. Notably, their response to Reviewer 1's question, "Aside from these concerns, it came to my attention that the same group has already reported..." effectively highlights the novelty and challenges addressed in this study. This discussion should be incorporated into the main text to better inform readers. Overall, I strongly encourage the authors to integrate all additional discussions, including tables and figures from the response letter, into the main text or supporting information to further enrich the content. In this revised form, the manuscript appears well-suited for publication in Nature Communications after the incorporation of above suggestion.

Reviewer #2

(Remarks to the Author)

The authors have thoroughly addressed all the concerns raised in the initial review, and the revisions significantly enhance the clarity and robustness of the manuscript.

1. Sequential Diastereoselective C-H Functionalization:

The addition of new data and examples, particularly for the improved 1,3-dia stereoselective synthesis, provides strong support for the practicality of their approach. The revised manuscript now clearly demonstrates improved diastereoselectivities (>15:1 for 1,3-diaryl-1,3-diols, >12:1 for alkyl-substituted 1,3-diols), aligning with the revised claims on the efficiency of sequential diastereoselective C-H functionalization.

2. Inclusion of Prior Work:

The authors have appropriately acknowledged the relevant contributions from Krische et al., contextualizing their work within the broader field of asymmetric C-H functionalization. The revised introduction clarifies the complementarity of their method with Krische's strategy, particularly the use of an inexpensive nickel catalyst and the broader substrate scope enabled by their approach.

3. One-Pot Approach:

The development of a one-pot protocol for converting unprotected diols to optically pure 1,2- and 1,3-diols is a significant practical improvement. Although the authors explain that integrating enantioselective and diastereoselective C-H functionalization in one pot remains challenging, they have demonstrated a feasible workaround and clarified the limitations.

4. HPLC Data:

The authors have addressed the concerns related to HPLC data. The revised Supporting Information includes fully resolved chromatograms, ensuring the accuracy of the ee values. The issues with peak area ratios and baseline irregularities have been corrected.

In conclusion, the authors have effectively addressed all raised issues, and the manuscript now meets the high standards required for publication in Nature Communications. Thus, this reviewer supports its acceptance.

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## REVIEWERS' COMMENTS

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[Answer: All the questions raised by Reviewer 1 have been added to the revised manuscript.](#)

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[Answer: No additional revision was needed.](#)