Lab 16: Reduction of Vanillin to Vanillyl Alcohol

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Objectives

By the end of this laboratory, you should have developed the skills to do the following:

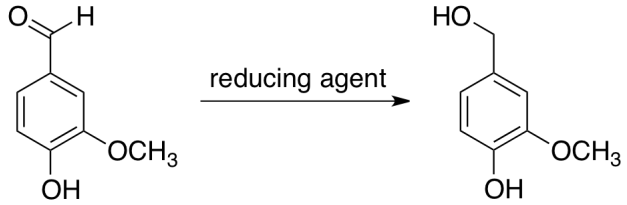
- Use SciFinder to search the chemical literature.
- Reproduce a procedure in the chemical literature.
- Compare and contrast different synthetic approaches.

Recommended Resources

- Website ~ Create a SciFinder Account (works only on the CSUB campus): https://scifinder.cas.org/registration/index.html?corpKey=61B9B7D2X86F350ABX6C8654CB4D67E6F318
- Website ~ Login to SciFinder (works only on the CSUB campus): https://scifinder.cas.org/
- Website ~ SciFinder Training Materials: https://www.cas.org/training/scifinder

Background

In this experiment you will research the literature to find and develop a procedure for the reduction of vanillin to vanillyl alcohol. Sodium borohydride is a common reducing reagent that can be used for this reaction, although other reducing agents are possible.



Your team will use SciFinder to search the literature and find possible procedures. As a team, you must attempt at least two procedures in duplicate (twice each to confirm results). You may attempt more procedures if time and resources allow. Note that these procedures must have literature precedence from a recognized chemistry journal (other procedures found online will not be approved). If you prefer, you may use one procedure and propose one modification to that procedure (i.e., a different solvent, temperature, concentration, etc.). Pay attention to the scale used in the literature, and modify it to a more manageable amount if necessary. Be sure to include a detailed list of items you will need for your procedure (include solvents, TLC plates, specialty glassware, etc.).

Once your proposal has been approved, you will need to calculate the grams/volume needed for your particular reagents. You may have to monitor your reaction by TLC to determine when it has finished. After the reaction is complete, you may need to purify your product using recrystallization or column chromatography (if you think your product is pure enough that it does not require further purification, you will need to support this with melting point, IR, and NMR data). You can then confirm the identity of your product and determine purity using melting point, IR, and NMR.

Please note: Project proposals that include the use of highly toxic or highly air/water reactive chemicals (e.g., hydrazine, super-hydride, or lithium aluminum hydride) will not be approved.

Project Proposal Guidelines

In lieu of an online prelab, you will be submitting a formal proposal for your project. Include the following sections:

1. Proposal Title
2. List of Investigators (i.e., all group members)
3. Abstract: Summarize your proposal plans. (Two to four sentences.)
4. Proposed Budget: List all required chemicals and supplies not available in your drawer (including those available in the stockroom). You will also need to provide an estimated cost for any specialized chemicals/supplies. You do not need to estimate costs for common lab solvents and solutions. Your estimated total cost for your project should not exceed $100.
5. Statement of the Problem and Significance of the Proposed Research: Provide a summary of your proposed project. State succinctly the problem that is to be addressed. Clearly outline the significance of the problem, the impact it may have if successful, and the hypothesis or primary idea that underpins the proposed work. (Two or more paragraphs.)
6. Plan of Procedure: Outline your plan of attack and its feasibility, providing details sufficient to support feasibility. Explain in detail the chemical principles involved and support your explanation and feasibility by citing pertinent references. Indicate how this plan will contribute to the solution of the problem being addressed. Be sure to include a detailed description of your proposed experimental procedures. (Two or more paragraphs.)
7. List of References: Use appropriate ACS formatting. (Don't forget to use in-text citations for your references in the body of your proposal.)

Lab Notebook Preparation

Before coming to lab, the following items must be in your lab notebook:

1. Title of experiment
2. Date the experiment is to be performed
3. The chemical reaction you are attempting (with skeletal structures)
4. A table with information about your starting materials. Include molecular weight, molar equivalents, and mmoles to be used. For solids include grams. For liquids, include grams, density, and volume. For solutions, include the concentration and volume.
5. The molecular weight and theoretical yield of any possible products

6. Any relevant physical properties
7. Hazards of and appropriate precautions for the safe handling of any new chemicals you will be working with
8. References

Safety Notes

- Be sure to review the MSDS of any chemicals you are working with to determine their hazards (if any).

Directions

1. Use this link to create a SciFinder account (this link and SciFinder will only work on campus as access is limited to campus IP addresses):

   https://scifinder.cas.org/registration/index.html?corpKey=61B9B7D2X86F350ABX6C8654CB4D67E6F318

2. Once you have an account, use this link to login: https://scifinder.cas.org/

   a. You may occasionally get this message when trying to login: "Your allocated system resources are currently unavailable to access SciFinder. Please try again later." This is because there is a limited number of people that can access SciFinder at the same time. Wait a few minutes and try again later.

   b. You can search SciFinder by keyword, reactant/reagent, or reaction. For this particular project, it is recommended that you conduct a reaction search. A video tutorial on how to search how to search for specific reactions or reaction type is available here: http://www.cas.org/training/scifinder/need-to-know-reaction-searching?seek=0

3. Use the results you find on SciFinder to submit a project proposal (guidelines above).
4. Once your project has been approved and you have obtained all necessary supplies, follow the procedures you proposed in your project proposal.
5. If you decide to make any changes to your proposed procedures, check with your instructor before proceeding.
6. Ask your instructor if you get stuck, or if your procedure does not work as expected.

Reporting Your Results

Write your report according to the guidelines described in "Topic 4: Writing an Organic Chemistry Lab Report". Work with your team on this report.

References & Additional Resources

1. Lehman, J. W. *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*, 3rd ed.; Prentice Hall: Upper Saddle River, NJ, 1999; pp 223-231.