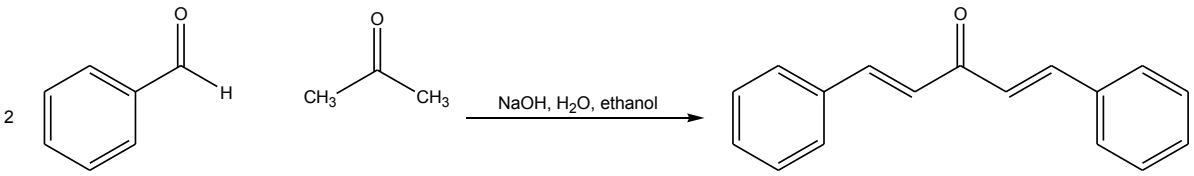
Lecture Notes

Chemistry 337
Unit 5 Synthesis of E,E-dibenzalacetone (E,E-DBA)

Introduction to Unit 5

The centerpiece of this unit is the synthesis of E,E-dibenzalacetone (E,E-DBA) for which we will employ a base-catalyzed aldol condensation. The crude product will be purified by recrystallization. The crude and purified products will be characterized by melting point determination.



Benzaldehyde Acetone E,E-dibenzalacetone

Purpose of Unit 5

To employ a based-catalyzed aldol condensation in the synthesize of E,E-dibenzalacetone

To characterize the crude and purified samples of E,E-dibenzalacetone by melting point determination

Overview of these lecture notes

These lecture notes provide guidelines for the preparation of your Unit 5 formal report. Please note the aldol condensation will be covered in Unit 11. The chemistry of Unit 5 will be discussed in the Unit 11 lecture notes. Recrystallization will be discussed in the Unit 2 lecture notes.

Lecture notes

General comments about the formal lab report

One of the most difficult problems a student faces in this course is estimating the appropriate length of the formal report. The following guidelines are designed to help you with your report preparation.

As a general point of style, reports should be written impersonally in the passive voice. While this style is somewhat clumsy, it is widely accepted in scientific writing. Although we are not grading your reports for prose style and literary merit, we suggest that, to save your time and ours, you write in the simplest and most direct English that you can command.

In order to provide adequate space for the grader's comments we ask that reports be set in double-spaced format and with a 2" right margin.

Components of the formal lab report (portions of a sample formal report are given on the next two pages)
Title, Name, Date (~3%)

Abstract (~5%)
State briefly the purpose of the experiment, the principal results, the mode(s) of isolation, separation and purification (as appropriate) and the major conclusions. You may refer to schemes and tables found in the introduction, results and discussion sections by number.

Introduction (~5%)
State clearly the synthetic goal of the experiment. Do not begin your report with a lengthy description of background theory. The time to demonstrate your knowledge of background theory is in explaining the results you have obtained. One or two paragraphs should be adequate. Include the appropriate balanced chemical equation(s) (including structural drawings for all organic reactants and products, inorganic reagents above the arrows, and solvents and conditions below the arrows) labeled as Scheme 1. See Scheme 1 of the sample formal report.

Results (~35%)
Tabulation of experimental results is encouraged whenever it leads to a more effective presentation. Tables must be in the same format as Table 1 of the sample formal report. Graders will not spend time rooting through pages of text searching for experimental data. They will simply assign a grade of zero for this portion of the report.

Discussion (~50%)
A separate paragraph should be included for each one of the following items:
-a written account of how the product was prepared (i.e. a description of the chemistry involved) along with appropriate references to any balanced reaction equations given elsewhere (e.g. in the introduction)

-a written account of how the crude product was isolated (include chemistry if appropriate)

-a written account of how the crude product was identified, wherein you cite and interpret relevant experimental data making reference to appropriate literature data

-a written account of how the crude product was purified and an evaluation of the efficiency of the purification, wherein you cite and interpret relevant experimental data making reference to appropriate literature data

-a written account of how the purified product was identified, wherein you cite and interpret relevant experimental data making reference to appropriate literature data

-a discussion of yields

Please remember, we are looking for an account of the results you obtained and the conclusions that you drew from them.

Experimental Procedure (~2%)
Do not copy out the complete experimental procedure from the manual. “The preparation was carried out according to the prescribed method” will be sufficient in many instances.

Sample formal lab report (except for the discussion) for you to use

Preparation Of E-α-Phenylcinnamic Acid And Z-α-Phenylcinnamic Acid

Your Name
Submission Date

Title (11 pt, bold)
Name (11 pt)
Date (11 pt, italics)

Abstract title (10 pt, bold)

Abstract

The preparation of Z-α-phenylcinnamic acid (**1**) and E-α-phenylcinnamic acid (**2**) (Scheme 1), from benzaldehyde and phenylacetic acid in the presence of triethylamine and acetic anhydride, is described. Both acids were purified by recrystallization; Z-α-phenylcinnamic acid was recrystallized from methanol/water and E-α-phenylcinnamic acid was recrystallized from diethyl ether/petroleum ether. Z-α-Phenylcinnamic acid was obtained in 13.0% yield. E-α-Phenylcinnamic acid was obtained in 26.7% yield. The acids were characterized by melting point, infrared spectroscopy and proton NMR spectroscopy.

Abstract (10 pt, double spaced) -should state briefly the purpose of the experiment, the principal results and major conclusions. Reference to structural formulas or tables in the text, by number, may be made in the abstract.

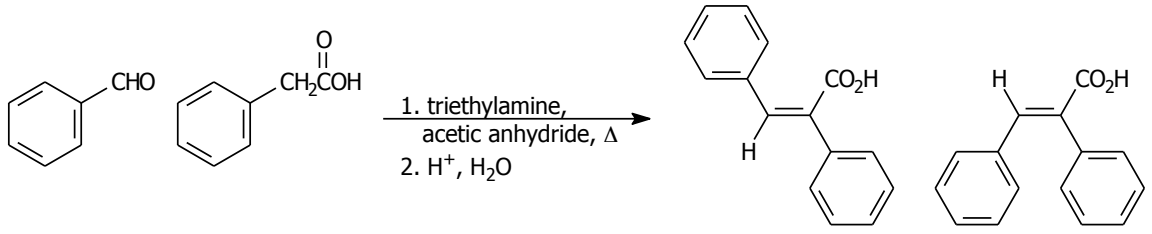
Introduction title (10 pt, bold)

Introduction

The purpose of this experiment was to employ the Perkin Reaction in the preparation of Z-α-phenylcinnamic acid (**1**) and E-α-phenylcinnamic acid (**2**). As outlined in Scheme 1 this involved allowing benzaldehyde to react with phenylacetic acid at reflux in the presence of triethylamine and acetic anhydride.

Introduction (10 pt, double spaced) -should clearly state the purpose and objectives of the experiment. In general, the introduction should be no more than one or two paragraphs.

Scheme 1



1 **2**

Results

Results title (10 pt, bold)

Results (10 pt, double spaced) -tabulation of experimental results is encouraged whenever it leads to a more effective presentation.

The results of the synthesis of Z-α-phenylcinnamic acid and E-α-phenylcinnamic acid are summarized in Table 1.

Table 1. Synthesis of Z-α-Phenylcinnamic Acid And E-α-Phenylcinnamic Acid

Compound	**molar mass (g/mol)**	**volume/mass moles**	**yield**	**mp (°C)**
phenylacetic acid (limiting reagent)	136.15	5.00 g 0.0368 moles	N/A	77°C
acetic anhydride (density 1.08 g/mL)	102.09	4.00 mL 4.32 g 0.0423 moles	N/A	N/A
benzaldehyde (density 1.046 g/mL)	106.12	6.00mL 6.28 g 0.0592 moles	N/A	N/A
triethylamine (density 0.729 g/mL)	101.19	4.00 mL 2.92 g 0.0289 moles	N/A	N/A
α-phenyl-cinnamic acids	224.26		8.23 g 0.0368 moles (theoretical yield)	174 (E) 138 – 139 (Z) (literature values)
Z-α-phenyl-cinnamic acid	224.26		2.38* g; 28.9% (crude) 1.07* g; 13.0% (purified)	125.0 – 129.1 (crude) 134.1 – 135.9 (purified)
E-α-phenyl-cinnamic acid	224.26		3.84* g; 46.7% (crude) 2.20* g; 26.7% (purified)	167.0 – 171.0 (crude) 173.0 – 173.5 (purified)

* white crystalline material