GENERAL LABORATORY INFORMATION

Each student will be permitted to do laboratory work only after she/he has:

1. Read the Departmental safety rules and certified so in writing.

2. Furnished her/his own acceptable safety goggles.

By enrolling in this class, students agree to perform all experimental and written work according to California State University, Northridge policies of academic honesty and integrity. Students violating these standards will receive a zero for the work in question and will have their case referred to the Student Affairs Office for appropriate disciplinary action. See the California State University, Northridge catalog for details of the University policies.

Students are required to attend all laboratory periods of the section in which they are enrolled. Excused absences, substantiated by an appropriate written confirmation received within two weeks, will result in no penalty. Unexcused absences will result in a zero for the experiment(s) in question. A maximum of two excused absences will be allowed.

Before coming to laboratory sessions, read the procedures for the assigned experiment in the Chemistry 333L laboratory manual. Watch the current experiment's video(s) that are listed in the Schedule of Experiments handout. Also read any suggested material in the lecture textbook. Complete the appropriate pre-lab preparation (viz., items 1–7 on the next page) in your laboratory notebook.

Bring the following items to laboratory sessions: a pair of safety goggles, the California State University, Northridge, Chemistry 333L laboratory manual and a **bound** (not a loose-leaf or spiral-bound) laboratory notebook (e.g., National #43-461) with every page numbered in ink.

Laboratory sessions of Chemistry 333L begin with a pre-laboratory conference lasting approximately fifteen to twenty minutes, during which various aspects of the experiment to be performed will be discussed. A laboratory quiz may be given during this time, also. Experiments are designed to be carried out in approximately 2 1/2 hours.

All work in the laboratory notebook must be written in ink (not pencil). Never use white-out. Use the left-hand pages for initial data collection and rough calculations. Reserve the right-hand pages for the experiment write-ups. Do **not** use loose scratch paper to record data!

Each laboratory notebook write-up should be concise, but it should also be accurate, neat, well organized, and complete. The following items normally are required in a complete write-up.

1. Date.

2. Title and/or statement describing the experiment to be performed.

3. Balanced equation(s) for any reaction(s) involved, using structural formulas for organic reactants and products. Include any potentially important side-reactions.

4. Brief but specific reference to the source of directions.

5. Table of physical properties for all reactants, solvents, and products involved in preparative experiments. Use the following column headings:

   compound MW mg or mL mmol ratio m.p. b.p. density

   For each reactant, the weight in milligrams (or volume in milliliters) and the number of millimoles should be those actually used in the experiment. For products, the figures in these two columns should be the amounts expected on the basis of theory if the reaction went 100% to completion as indicated in the balanced equation. Include solvents in this table, but do not calculate the number of millimoles used.

6. Summary of safety precautions and hazardous chemicals.

7. If appropriate, a flowchart of the workup, isolation, and purification sequences.
   Short phrases should indicate the methods employed.

8. Changes, if any, in the experimental directions made by the instructor.

9. Record of any qualitative observations, numerical data, calculations, and results.
   Detailed description of any unexpected experimental behavior.

10. Conclusions, with comparisons to data from the chemical literature (preferably in the form of a concise table.) Discussion of any problems, and how the experiment could be conducted more successfully.

Note: The reference room in Oviatt library contains some very useful books for obtaining information for items 5, 6, and 10. Consult these references for pre-lab preparation and notebook write-ups.

Section QD (Chemistry)

1. The Aldrich Catalog and Handbook of Fine Chemicals
2. The Dictionary of Organic Compounds
3. The CRC Handbook of Chemistry and Physics
4. Lange's Handbook of Chemistry

Section T (Engineering/Technology)

1. Dangerous Properties of Industrial Materials, 7th edition

Additional reference books are available in the Chemistry section of Oviatt library.

Safety Data Sheets (SDSs) contain an abundance of safety information for most common chemicals and are available online at the Sigma-Aldrich Web site.

See pages 4–9 of this manual for representative laboratory notebook write-ups.

A Representative Laboratory Notebook Write-up for a Preparative Experiment

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5/1/95

WILLIAMSON SYNTHESIS OF A BENZYL ETHER

1 2

References:

1. California State University, Northridge, Department of Chemistry, Spring 1995 Chemistry 411L laboratory manual, pages 17-21.

2. L.G. Wade, Jr., Organic Chemistry, 3rd edition, pages 604-605.

Procedure:

See 411L laboratory manual. No changes were made to this procedure.

Physical Properties of Reactants, Solvents and Products:

compound	MW	g or mL	mole	ratio	mp	bp	density
3-phenyl-1-propanol*	136.19	0.20g	.0015	1.0	-18°	235°	1.008
sodium hydride	24.00	0.04g	.0017	1.1			
benzyl bromide*	171.04	0.18 mL	.0015	1.0	-3°	198°	1.438
tetrahydrofuran (THF)	72.11	10 mL			-108°	65°	0.889
diethyl ether	74.12	60mL total			-116°	35°	0.708
benzyl ether 2	226.32	0.33g (theoretical yield)					

* limiting reagents.

Safety:

3-phenyl-1-propanol is an irritant.
Sodium hydride is a flammable solid and is moisture-sensitive.
Benzyl bromide is corrosive and is a lachrymator.
Tetrahydrofuran is a flammable liquid and is an irritant.
Diethyl ether is a flammable liquid and is toxic.

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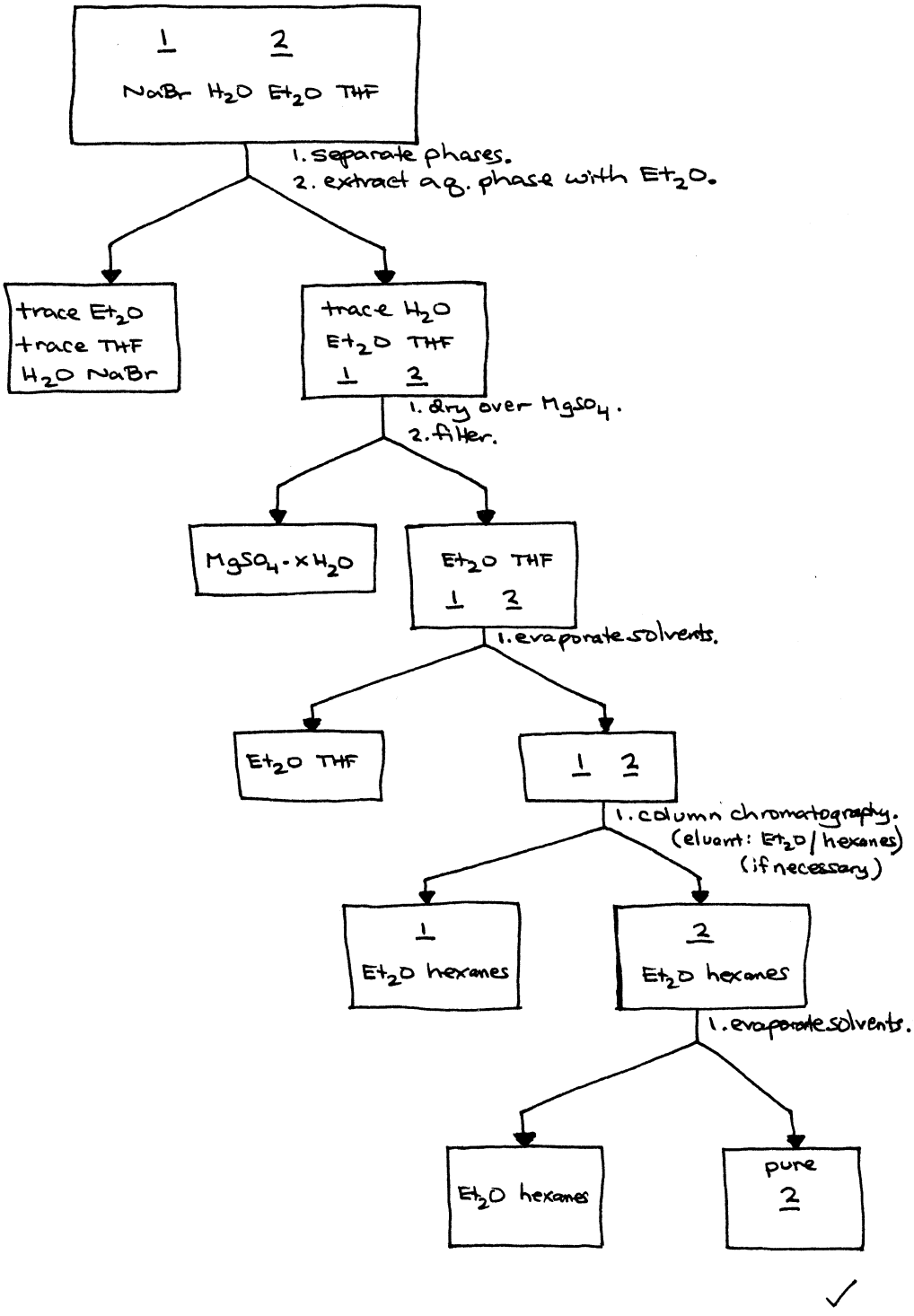
Flowchart:



✓

Inorganics are removed by extraction into water. The ether 2 is separated from any residual starting material 1 by column chromatography.

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Observations:

starting material 1	sodium hydride	benzyl bromide
		180 μL (by syringe)
0.607	0.423	
0.407	0.383	tetrahydrofuran
0.200 g	0.040 g	10 mL (by pipette)

2:30 When NaH was added to the solution of 1 and benzyl bromide in THF, vigorous gas evolution occurred. (H_2 gas) Gas evolution lasted ~5 minutes. All the gray NaH solid disappeared by that time. A clear, pale-yellow solution was obtained.

The reaction was refluxed for 30 minutes before cooling to room temperature. Extractions with Et_2O: used 3× 20mL Et_2O. No problems. Got a cloudy organic phase, which cleared up after drying with $MgSO_4$.

Crude product after evaporation of THF and Et_2O:

0.700
0.417
0.283g

$\frac{0.28}{0.33} \times 100 = 85\%$ yield

IR spectroscopy showed no 3300 cm^{-1} absorption. ∴ NO starting material 1 remains in product! Absorptions at 3050 cm^{-1} (aromatic C-H stretch), 2950 cm^{-1} (alkyl C-H stretch), 1600 cm^{-1} and 1500 cm^{-1} (aromatic C=C stretch) and 1150 cm^{-1} (C-O stretch) all consistent with desired product 2.

Column chromatography not done because crude product is pure 2.

Conclusions:

Successful preparation of benzyl ether 2 in 85 % yield.

A Representative Laboratory Notebook Write-up for a Non-Preparative Experiment

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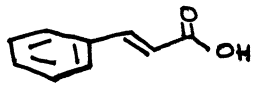
3/1/05

PURIFICATION OF CINNAMIC ACID BY RECRYSTALLIZATION



Reference:

California State University, Northridge, Department of Chemistry and Biochemistry, Spring 2006 Chemistry 333L laboratory manual, pages 29 - 39.

Procedure:

See reference above.

Modifications:

1. Use cinnamic acid instead of acetanilide.
2. Recrystallize cinnamic acid from a mixture of diethyl ether (Et_2O) and petroleum ether.
3. Perform a hot filtration, if necessary.

Physical Properties:

compound	MW	mp	bp	density
cinnamic acid	148.16	133 - 134 °C	300 °C	
diethyl ether	74.12	-116 °C	35 °C	0.708
petroleum ether			35 - 60 °C	0.640

Safety:

Cinnamic acid is an irritant.
Diethyl ether is a flammable liquid and is toxic.
Petroleum ether is a flammable liquid and is toxic.
Sand baths can be very hot when in use.

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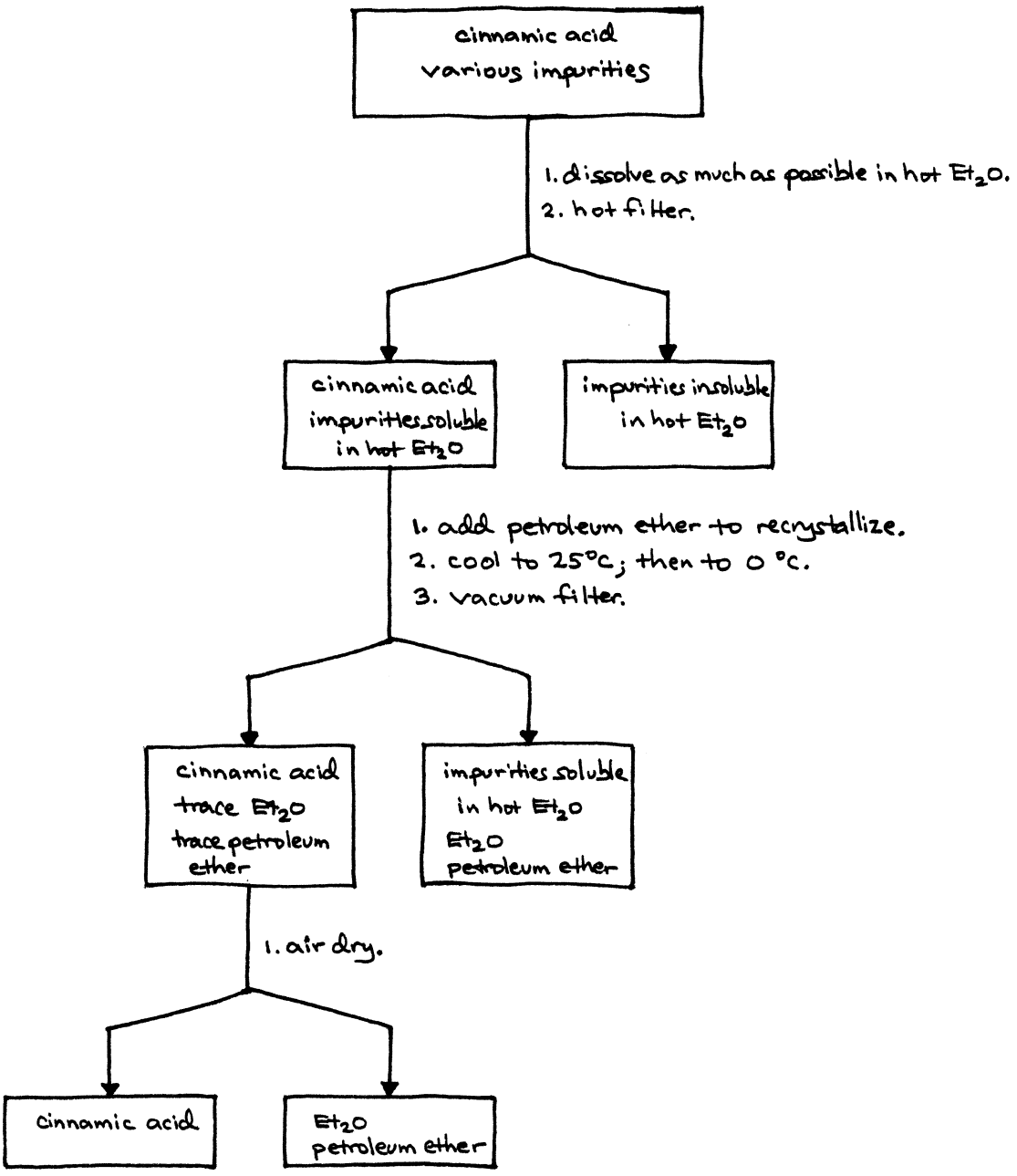
Flowchart:



Observations:

original mixture:

1.65
0.31
1.34 g

Color of crude mixture: mostly off-white, with dark specks interspersed throughout.

Tried to dissolve the sample in 10 mL of hot (boiling) Et_2O. Most of the sample dissolved, but the dark, granular material did not. Hot filtration through fluted filter paper removed the dark, granular material. Washed the filter paper with an additional 4 mL of hot Et_2O. Then concentrated solution back to about 10mL.

Added petroleum ether dropwise, while still boiling the solution. After approximately 7 mL of petroleum ether was added, the solution became cloudy. Added about $\frac{1}{2}$ mL hot Et_2O to get a clear solution, again. Then removed flask from sand bath.

Beautiful crystals formed upon cooling. Even more when cooled to 0 °C. Collected crystals by vacuum filtration. Washed with cold petroleum ether. Then air-dried the crystals.

recrystallized sample:

1.36
0.29
1.07 g

$\frac{1.07}{1.34} \times 100 = 80\%$ mass recovery

melting-point data:

original mixture: 118 – 126 °C

recrystallized sample: 132-133 °C literature value (Aldrich): 133-134 °C

Conclusions:

Successful recrystallization.
Reasonable mass recovery.
Good purity, based upon comparison of observed mp to literature value.