

## Appendix P.1 : Recrystallization conditions and melting point data

### 2-Chlorobenzoic acid: (m.p. 138-140 °C)

Recrystallize in pure water (Completely insoluble in cold water)  
20 mL was needed for 0.52 g (more solvent would have been OK)  
A yellow mother liquor was obtained (from the pure compound).  
Nice big flaky crystals were obtained  
Yield: 98 %

**Solvent: H<sub>2</sub>O**  
**20-25 mL for 0.52 g**  
**Dry in oven (about 30 minutes) and determine the melting point.**

### 2-Methoxybenzoic acid or o-anisic acid (m.p. 98-100 °C)

Recrystallize in pure water  
Most seemed to dissolve in 5 mL of hot water (completely and easily soluble in 8 mL) for 0.50 g  
Separated as an oil, crystallized after scratching/stirring and increased volume to 10 mL  
Small crystals were obtained  
Yield: 92 %  
Do not put in the oven to dry

**Solvent: H<sub>2</sub>O**  
**10 mL for 0.50 g**  
**Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.**

### 4-Methoxybenzoic acid or p-anisic acid (m.p. 182-185 °C)

Recrystallize in ethanol  
0.50 g all dissolved in 6 mL of ethanol. (Very soluble in hot solvent)  
Small needles were obtained.  
Yield: 80 %

**Solvent: 55% ethanol/45% H<sub>2</sub>O**  
**20-22 mL for 0.49 g**  
**The hot solution must be cooled slowly.**  
**Dry in oven (about 30 minutes) and determine the melting point.**

Recrystallize in a mixture 50% ethanol - 50 % water (a little more of ethanol)  
0.49 g dissolved in total of 22 mL of hot solvent (maybe a bit less would have been OK)  
Slow to crystallize, be patient. Once crystals start forming, put in the ice bath.  
Nice crystals (small needles) were obtained  
Yield: 92 %

#### 4-Ethoxybenzoic acid (m.p. 197-199 °C)

Recrystallize in water

Not very soluble in hot solvent... Stays on the surface.  
30 mL of hot water and still not all dissolved

Recrystallize in Ethanol

0.50 g were dissolved in 10 mL of hot ethanol  
Crystallize on counter and in the ice bath  
Small crystals were obtained  
Yield: 86 %

Recrystallize in a mixture 50% ethanol - 50% water (a little more ethanol)

0.50 g dissolved in a total of 26 mL  
Wait for it to cool on the counter than put in the ice bath  
Small crystals were obtained  
Yield: 78 %

**Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 30 minutes) and determine the melting point.**

#### 3,4- Dimethoxybenzoic acid (m.p. 179-182°C)

Not very soluble in hot water

0.49 g dissolved in 15 mL of 20% ethanol/80% water  
Crystallized in ice bath, small crystals obtained  
Yield:

**Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 30 minutes) and determine the melting point.**

Unknown 1: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 2: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 3: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

- Unknown 4: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 5: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 6: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 7: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 8: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 9: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 10: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 11: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 12: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 13: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 14: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 15: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 16: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 17: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 18: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 19: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 20: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

Unknown 21: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.

Unknown 22: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.

Unknown 23: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.

Unknown 24: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.

Unknown 25: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.

## Appendix P.2 : List of possible starting aldehydes

<b>4-methoxybenzaldehyde</b> $n_D^{20}$ 1.5730 Cannizzaro Products:	<b>(<i>p</i>-Anisaldehyde)</b> 4-methoxybenzoic acid 4-Methoxybenzyl alcohol	(unknown # 1-5) m.p. 182-185 <sup>0</sup> C $n_D^{20}$ 1.5440
<b>2-Chlorobenzaldehyde</b> $n_D^{20}$ 1.5660 Cannizzaro Products:	2 -Chlorobenzoic acid 2 -Chlorobenzyl alcohol	(unknown # 6-10) m.p. 138-140 <sup>0</sup> C m.p. 69-71 <sup>0</sup> C
<b>4-Ethoxybenzaldehyde</b> $n_D^{20}$ 1.5590 Cannizzaro Products:	4 -Ethoxybenzoic acid 4 -Ethoxybenzyl alcohol	(unknown # 11-15) m.p. 197-199 <sup>0</sup> C $n_D^{20}$ 1.5350
<b>3,4-Dimethoxybenzaldehyde</b> mp 42-45 <sup>0</sup> C Cannizzaro Products:	3,4 -Dimethoxybenzoic acid m.p. 3,4 -Dimethoxybenzyl alcohol	(unknown # 16-20) 179-182 <sup>0</sup> C $n_D^{20}$ 1.5520
<b>2-methoxybenzaldehyde (<i>o</i>-Anisaldehyde)</b> mp 34-40 <sup>0</sup> C Cannizzaro Products:	2-Methoxybenzoic acid 2-Methoxybenzyl alcohol	(unknown # 21-25) m.p. 98 - 100 <sup>0</sup> C $n_D^{20}$ 1.5470

## Appendix P.3 : Evaluation Grid

<b>Prelab Report</b>	
Cannizzaro reaction (1)	/2
Mechanism (2)	/4
Explanation of $\alpha$ -H (3)	/3
Flow chart of extraction (4)	/5
How to identify the products (5)	/3
Hydride transfer (6)	/3
<b>Lab notebook:</b>	
Reaction Equation	/4
Mass of aldehyde	/4
Protocol	/8
Observations	/9
Mass of alcohol	/15
Mass of acid	/15
IR spectrum	/15
Melting Point	/10
<b>Lab report:</b>	
Objectives	/3
Protocol	/5
Results	/6
Discussion	/8
Conclusion	/4
References	/1
Questions	/13
<b>Evaluation by team member</b>	/10
<b>Total</b>	<b>/150</b>

## Appendix P.4 : List of materials

### Cannizzaro: Week one

- Microscale glass kit
- Small stir bar +stir bar retriever
- Stirrer/hot plate
- Grease
- Ice
- Small separatory funnel 30 mL
- three finger clamps
- Tubing for condenser
- Dessicooler
- Filter paper 4.25 cm + 11.0 cm
- pH paper 0-14

### Cannizzaro: Week two

#### **\* Turn on oven: 80 ° C**

- Ethanol 95 %
- Hot Plates
- Filter paper 4.25 cm
- Watch glasses
- Beaker tongs
- Three finger clamps
- Waste for organic acids +Alcohols
- I.R.
- Melting point apparatus

Compounds	Instruction	Amount per student	Location
Methanol	Automatic dispenser	4 mL	M-13
HCl 12 M	Dropping bottle	4 mL	
KOH 12 M	336 g/500 mL Dropping bottle	4 mL	P-22
Saturated sodium chloride	317 g /L	A few mL	S-23
5 % aqueous sodium bicarbonate	58g Na <sub>2</sub> CO <sub>3</sub> · H <sub>2</sub> O / L	20 mL	S-21
Anhydrous magnesium sulfate	-----	2 g	M-4
Methylene Chloride	-----	20 mL	M-25

## Appendix E.1 : Project description and requirements

### ORGANIC CHEMISTRY I. INTEGRATION OF KNOWLEDGE PROJECT

#### Introduction:

The Integration of Knowledge component (Objective 00UU) of the Revised Science Program is “to apply acquired knowledge to one or more subjects in the sciences.” (i.e. to be able to apply knowledge and skills gained in one discipline to a problem in another). Objective 00UU is fully achieved by successful completion of at least three courses containing a 10-hour module of integrative activities each (as described in the Champlain-Lennoxville Revised Science Program). These projects contribute to the Comprehensive Assessment, which verifies that you have achieved the attributes of the graduate profile. In summary the project described below should be completed in such a way so as to demonstrate that you have achieved as many of the objectives and attributes of the Program as possible. The most important evaluation criteria will be:

- The ability to apply acquired knowledge and skills to a new situation in the same or another discipline
- Scientific and problem-solving skills
- Communication skills: written
- Computer technology literacy
- Autonomous learning
- Team work

#### The Project:

This is a combined written and laboratory project and you will each work as part of a team of two. Each team will be presented with an unknown aldehyde (one of several possibilities). Over two laboratory periods you will perform a Cannizzaro reaction on the aldehyde, isolate, purify and identify the products. You will also be required to research the reaction before the first laboratory period, to write a laboratory report including some research questions. Guidelines on the times to complete each part of the project along with more details follow.

#### **Before the first lab (Suggested time required: 2 hours) (2 copies)**

1. Write an equation to illustrate the Cannizzaro reaction.
2. Write equations to show the steps in the mechanism for the reaction. Describe each step in your own words.
3. Explain how the presence or the absence of a H  $\alpha$ - to the carbonyl will control the reaction of an aldehyde with base.
4. After the reaction is completed, you will separate the products by liquid-liquid extraction (Draw a flow chart to show how it could be done). Explain how the two components are separated into the two layers.
5. Explain how you might identify the products of the reaction. Give specific details and numbers where possible.

6. The hydride ion does not act as a leaving group under normal circumstances. Why and how is it transferred in the Cannizzaro reaction?

**The laboratory (time required: 5 hours):**

You will be provided with an experimental procedure but only after submission of the preliminary research.

**During the laboratory** you must keep a notebook and record relevant observations, results and any deviations from the published procedures (see the following page). Think very carefully about what results are important in an Organic Chemistry experiment.

**The laboratory report (3 hours) (2 copies):**

Write a detailed computer generated lab report, including:

- The objective of the experiment
- Summary of the reaction and procedure (keep it brief, a couple of paragraphs)
- Laboratory notebook (as written in the lab)
- Results, including calculation of percent yield of products
- Discussion
  - Justification of the main experimental steps and how you arrived at the identification of the unknown
  - Discussion of the questions below
- Conclusion

**Questions to be discussed:** Your answers should be documented, i.e. give references.

1. Oxidation and reduction of organic compounds is important both in the laboratory and in biological systems. Why are the conditions different in each case? Illustrate your answer by writing equations for the following:
  - a) A laboratory oxidation of ethanol
  - b) The reduction of a ketone in the presence of NADH: In what way is this similar to the Cannizzaro reaction?
2. Ethylene glycol, a constituent of automobile antifreeze, is highly toxic and each year deaths result from its ingestion. Explain the toxicity. Describe an effective treatment and how it works.
3. What is a MSDS and what is its function?
4. Refer to the MSDS for a substance used in this experiment and describe three or four of the most important items of information that it supplies.
5. Describe the meaning of the term LD<sub>50</sub>. What are some limitations of this information?

## **Evaluation:**

This activity counts for 15% of the course grade as described on p. 4 of the course outline. The distribution of these marks will be approximately as follows:

Pre-laboratory report	2 marks
The laboratory and lab notebook	8 marks
The laboratory report	4 marks
Evaluation by other team member	1 mark

## **The Laboratory Notebook:**

The laboratory notebook provides a permanent and legal record of what was done and of the results obtained in the laboratory. For these reasons, each page must be dated and no pages are to be removed. The pages on the left side of the book may be used for rough note work but work on the right must be done in ink and nothing is to be covered up or erased (i.e. no whiteout). A neat line can be drawn through any errors or work that is changed.

Your notebook should contain:

- The information necessary for the identification of the unknown. This could include the state and any color.
- A brief summary of the experimental procedure, including amounts used. example: 1.52 g of aldehyde was dissolved in ----- and the mixture refluxed ---for 1 hour.
- Significant observations, such as a color change.
- Amounts (yields) obtained.
- Necessary data on the possible unknown

### Cannizzaro Experiment: Evaluation Grid

<b>Prelab Report</b>	/20
Cannizzaro reaction (1)	/2
Mechanism (2)	/4
Explanation of $\alpha$ -H (3)	/3
Flow chart of extraction (4)	/5
How to identify the products (5)	/3
Hydride transfer (6)	/3
<b>Lab notebook:</b>	
Reaction Equation	/4
Mass of aldehyde	/4
Protocol	/8
Observations	/9
Mass of alcohol	/15
Mass of acid	/15
IR spectrum	/15
Melting Point	/10
<b>Lab report:</b>	
Objectives	/3
Protocol	/5
Results	/6
Discussion	/8
Conclusion	/4
References	/1
Questions	/13
<b>Evaluation by team member</b>	/10
<b>Total</b>	/150

## Annexe E.2 : Laboratory Protocol

### WEEK 1:

#### Caution:

Be sure that there is no acetone present on any of the glassware. Acetone will interfere with the desired reaction.

Methylene chloride has a higher density than water. It is your responsibility to determine which layer is which in the extractions and to choose appropriate glassware for each step.

#### Procedure:

Add 1.5 g of your unknown and 4.0 mL of methanol to a 10-mL round-bottom flask. Add a stir bar to the flask. With gentle swirling, carefully add 4.0 mL of 12 M aqueous potassium hydroxide solution with a Pasteur pipet. **Avoid getting potassium hydroxide solution on your skin or on the ground-glass joint.** Lightly grease the joint and attach a water-cooled condenser to the flask. Using a hot water bath, reflux (i.e. boil gently) the reaction mixture for one hour. Cool the mixture to room temperature. Pour the mixture into a small separatory funnel containing 5 mL of water, and use another 5 mL of water to aid the transfer. Extract the reaction mixture with 10 mL methylene chloride (twice) and combine the organic layers. The organic layer contains Compound 1, and the aqueous layer contains Compound 2.

#### Organic Layer:

Wash the organic layer twice with 10 mL portions of 5% aqueous sodium bicarbonate solution. Then wash the organic layer with an equal volume of water. If an emulsion forms, add a little saturated sodium chloride solution to break it. Dry the organic layer over anhydrous magnesium sulfate. After filtering the magnesium sulfate, the organic layers should contain mainly Compound 1 and methylene chloride. Let the methylene chloride evaporate to recover Compound 1. Clearly label your container and store it in the designated fume hood.

#### Aqueous Layer:

To precipitate Compound 2, add 10 mL of cold water and acidify with 12 M HCl while stirring the mixture and keeping the solution in an ice bath. Add just enough acid to ensure complete precipitation of the compound (do not overacidify the solution). Filter the solid and let it dry in a petri dish (clearly labelled in your locker).

### WEEK 2:

- Compound 1 will probably be a liquid. Obtain an IR spectrum and interpret all the important bands.
- Recrystallize Compound 2 (see recrystallization conditions) and dry the crystals in the oven. Determine the melting point of the purified product. Submit your crystals to the lab instructor.

**Possible starting aldehydes:**

Benzaldehyde  
o-methoxybenzaldehyde  
p-methoxybenzaldehyde  
m-nitrobenzaldehyde  
o-chlorobenzaldehyde

m-chlorobenzaldehyde  
p-chlorobenzaldehyde  
2,3-dimethoxybenzaldehyde  
3,4-dimethoxybenzaldehyde  
p-ethoxybenzaldehyde

### Appendix E.3 : Recrystallization conditions

- Unknown 1: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 2: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 3: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 4: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 5: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
20-22 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 6: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 7: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 8: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 9: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 10: Recrystallization Solvent: H<sub>2</sub>O  
20-25 mL for 0.52 g  
Dry in oven (about 45 minutes) and determine the melting point.

- Unknown 11: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 12: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 13: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 14: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 15: Recrystallization Solvent: 55% ethanol/45% H<sub>2</sub>O  
26 mL for 0.50 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 16: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 17: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 18: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 19: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.

- Unknown 20: Recrystallization Solvent: 20% ethanol/80% H<sub>2</sub>O  
15 mL for 0.49 g  
The hot solution must be cooled slowly.  
Dry in oven (about 45 minutes) and determine the melting point.
- Unknown 21: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.
- Unknown 22: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.
- Unknown 23: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.
- Unknown 24: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.
- Unknown 25: Recrystallization Solvent: H<sub>2</sub>O  
10 mL for 0.50 g  
Do not put in oven; put in dessicator for a minimum of 24 hours. Come back to do the melting point.