

# CHEM 222 LAB EXAM REVIEW:

(Compiled by MS, November 2015)

*This review covers a total of seven experiments.*

<b>Table 1:</b> Experimental Goals and Important Chemicals		
<b>Title of Experiment:</b> (# Lehman)	<b>Objective</b>	<b>Chemicals Encountered</b>
<b>Synthesis of Salicylic Acid from Wintergreen Oil (#4)</b>	To determine whether or not salicylic acid can be synthesized from synthetic methyl salicylate	<i>Salicylic acid, methyl salicylate, sulfuric acid, wintergreen oil, sodium hydroxide, sulfuric acid</i>
<b>Identification of Unknown Ketone (#11)</b>	To identify an unknown ketone	<i>DNPH reagent, ethanol, possible methyl ketones, sodium bicarbonate, ethyl acetate deuteriochloroform (CDCl<sub>3</sub>)</i>
<b>Green Synthesis of Camphor (#7)</b>	To oxidize isoborneol with bleach to see whether or not camphor is formed.	<i>Isoborneol, glacial acetic acid, camphor, sodium hypochlorite, sodium bisulfite</i>
<b>Haloform Oxidation of 4'-Methoxyacetophenone (handout)</b>	To determine whether or not 4'-Methoxyacetophenone can be converted into 4-methoxybenzoic acid with laundry bleach	<i>4'-methoxyacetophenone, aqueous sodium hypochlorite, tripropylmethylammonium chloride, acetone, ethyl ether, hydrochloric acid, aqueous ethanol, 4-methoxybenzoic acid</i>
<b>Reaction of Iodoethane with Sodium Saccharin, an Ambient Nucleophile (#20)</b>	To determine the major product of the reaction between sodium saccharin and iodoethane	<i>Sodium saccharin, iodoethane, DMF, deuteriochloroform, N-ethyl saccharin, O-ethyl saccharin,</i>
<b>Synthesis of Triphenylmethanol and the Trityl Carbocation (#30)</b>	To determine the color of the trityl carbocation	<i>Bromobenzene, magnesium, diethyl ether, benzophenone, triphenylmethanol, fluoboric acid, sodium bicarbonate, acetic anhydride, hydrochloric acid, 2:1 hexane solution,</i>
<b>Borohydride Reduction of Vanillin to Vanillyl Alcohol (#29)</b>	To devise a synthesis to convert vanillin to vanillyl alcohol	<i>Vanillin, sodium borohydride, aqueous sodium hydroxide, hydrochloric acid, vanillyl alcohol,</i>

**Table 2:** Important Operations of Experiments

<u>Title of Experiment:</u>	<u>Procedures and Operations</u>
<b>Synthesis of Salicylic Acid from Wintergreen Oil (#4)</b>	Heating under reflux, vacuum filtration, recrystallization, mixed melting point
<b>Identification of Unknown Ketone (#11)</b>	Recrystallization from mixed solvents, vacuum filtration, thin-layer chromatography, melting point, H-NMR spectroscopy
<b>Green Synthesis of Camphor (#7)</b>	Melting point, sublimation, vacuum filtration
<b>Haloform Oxidation of 4'-Methoxyacetophenone (handout)</b>	Vacuum filtration, recrystallization, melting point, infrared spectroscopy
<b>Reaction of Iodoethane with Sodium Saccharin, an Ambient Nucleophile (#20)</b>	Vacuum filtration, melting point, H-NMR spectroscopy
<b>Synthesis of Triphenylmethanol and the Trityl Carbocation (#30)</b>	Vacuum filtration, Trituration, recrystallization, melting point, washing liquids, drying liquids
<b>Borohydride Reduction of Vanillin to Vanillyl Alcohol (#29)</b>	Vacuum filtration, recrystallization, melting point,

## Operations in Depth:

<u>Operation:</u>	<u>Type:</u>	<u>What it Does:</u>	<u>How it Works:</u>
<p><b>Heating under Reflux</b> [OP-7c]</p>	<p>Conducting Chemical Reaction</p>	<p>The term “reflux” is used here to refer to the vapors condensing into the flask. Often, the reaction solvent will have a boiling point within the desired temperature needed to speed up the reaction. In some cases, a liquid reactant can also serve as the solvent. Using a condenser, the solvent vapors will return to the reaction vessel. This process is called heating under reflux.</p>	<p>You’ll need a West condenser, heat source, round-bottom flask, magnetic stir bar, and rubber tubing. Reaction will take place in the round-bottom flask, on the heat source. Temperature is monitored constantly with a thermometer and adjusted accordingly.</p>
<p><b>Vacuum filtration</b> [OP-16]</p>	<p>Separation</p>	<p>Provides a fast method for isolating a solid (or liquid) from a liquid-solid mixture. This procedure was performed practically in every experiment.</p>	<p>The mixture is poured into a Buchner funnel with a filter paper which traps the solid, while the liquid is pulled down into the flask by a vacuum. Important thing to keep in mind is the solvent used to wash the funnel. Ideally, the solvent chosen is the same solvent as the one from which it was filtered. The solvent should also be cooled in an ice bath prior to washing. For example, if you are filtering a solid from an aqueous solution, you should use ice cold distilled water as the wash solvent. If filtering from a mixed-solvent solution, use the solvent in which the solid is least soluble in.</p>
<p><b>Thin-Layer Chromatography</b> [OP-22]</p> <p><i>(seen in Experiment #11)</i></p>	<p>Separation</p>	<p>Similar to column chromatography, though not ideal for larger quantities. Uses a solid adsorbent as the stationary phase and a liquid solvent as the mobile phase. The mobile phase travels up the adsorbent layer via capillary action. As this mobile solvent travels up the plate (coated with the adsorbent) it drags along the components of each spot. The key theory to keep in mind in this procedure is the interaction between the solvent with the adsorbent as well as the solvent with the component it is dragging. This interaction is primarily governed by the polarity or lack of it.</p>	<p>You spot the samples on one end of the plate along with a reference sample. Make sure that these spots do not submerge into the solvent in the beaker. If so, the entire procedure will be ruined. Give the spots time to travel up the plate. In this experiment, the spots were able to be visualized since the DNPH reagent made them colored. The distance of the solvent front is measured with a ruler and each spot’s distance is divided by the solvent’s distance to obtain an <math>R_f</math> value. You can compare these values to the reference sample in order to determine the identity of the unknown sample. Some people add a paper wick inside the chamber in order to saturate the</p>

			environment with the solvent vapors.
<b>Washing Liquids</b> [OP-24]	Washing & Drying	Similar to extraction, though with a different purpose. One would perform extraction to isolate a desired substance from an impure mixture. Washing, however, removes impurities from the substance of interest. The key is to make sure that this substance of interest is not washed away with the impurities so it should therefore not dissolve appreciably in the washing solvent	You'll usually use a separatory funnel for this operation. If a gas is produced during the washing (e.g. after adding sodium bicarbonate), be sure to open the funnel in between shakes to allow the gas to escape. Two layers should be observed, with the denser one on the bottom. It is up to you to decide which one is to be kept and which is to be discarded.
<b>Drying Liquids</b> [OP-25] <i>(seen in Experiment #30)</i>	Washing & Drying	The purpose of this operation is to get rid of any water which the substance-of-interest may have come into contact with. For instance, if an aqueous wash liquid was used during the experiment, there may still be traces of water by the end of the reaction which will have to be removed. Organic liquids can be dried with drying agents, also known as desiccants. Examples of drying agents include anhydrous calcium chloride, sodium sulfate, and others. These anhydrous salts become hydrated and the "dried" liquid can now be separate, usually with filtration.	In Experiment #30, anhydrous sodium sulfate was used as the drying agent. The agent is added with the liquid and the contents are mixed together vigorously. The liquid is then separated usually with filtration. (vacuum or gravity)
<b>Recrystallization</b> (single solvent) [OP-28a]	Purification	A common technique to purify chemicals by dissolving the compound with its impurities into a suitable solvent at a high temperature. This technique takes advantage of the idea that solids generally dissolved better in hot solutions than in cold ones. When the solution cools, only the solid of interest (i.e. with no impurities) should crystallize out of the solution. Choosing the correct solvent is crucial for this operation to work. Should be somewhat less/more polar than solid. Solid should be very soluble in hot solvent, and relatively insoluble in cold solvent.	Knowing the solubility of the solid in the boiling solvent, you can estimate how much solvent to use. You will heat the solvent until it boils. Add it slowly to the solid while stirring vigorously. Keep the solid on the hot plate too. Once all the solid dissolves, you can let it cool first in room temperature, and then on an ice bath.
<b>Recrystallization</b> (mixed solvents) [OP-28b]	Purification	Same theory as above. However in this case, a single solvent is not possible. A mixture of two solvents, though, can do the job. Examples include ethanol-water, ether-methanol, ethanol-petroleum ether, etc. The two solvents must be miscible. The solid compound should be quite soluble in one solvent while relatively insoluble in the other.	Heat compound in solvent in the solvent in which it is most soluble until it dissolves, then add enough of second solvent to bring solution to saturation point.
<b>Sublimation</b> [OP-29] <i>(seen in Experiment #7)</i>	Purification	Sublimation is a phase change in which a solid passes directly into a vapor phase, skipping the liquid phase. This method works best if impurities do not sublime appreciably. This operation is not as selective as recrystallization.	A heating source as well as a cooling source is necessary for this operation. The solid which is being heated is called the " <u>sublimand</u> ". The solid which forms afterwards, after making contact with the cold source, is called the " <u>sublimate</u> ". Recap: the impure sublimand is

			being changed from a solid to gas by heat. It then condenses on a cold surface and is called the sublimate.
<b>Melting Point [OP-33]</b>	Physical Constant Measurement	This is a crucial physical property of a substance. This is the temperature at which a solid changes its physical state into a liquid. At the exact melting point, the solid and liquid form are said to be in equilibrium, at 1 atmosphere. Presence of impurities in a substance lowers the melting point, and broadens the melting point range.	A small portion of the sample is packed into a capillary tube, closed at one end, and then placed into a heating device. The melting point range is recorded. The presence of impurities likely will yield a lower melting point than what is written in the literature.
<b>Infrared Spectrometry [OP-39]</b>	Instrumental Analysis	Theory covered in lecture.	
<b>H-NMR Spectroscopy [OP-40]</b>	Instrumental Analysis	Theory covered in lecture.	

### Chemicals in Depth:

(Note, I did not talk about each substance. What is important, is the role the substance played in the experiment)

<b>Table 4</b>		
<b>Chemical Name:</b>	<b>About</b>	<b>Role in the Experiment</b>
<b>Expt #4: Salicylic Acid from Wintergreen Oil</b>		
Methyl salicylate	An ester, known as the oil from Wintergreen.	The starting material and limiting reactant of the reaction.
Sodium hydroxide	Aqueous base	Used in excess to ensure fast reaction. Reaction is hydrolysis.
[di]sodium salicylate	A salt of salicylic acid	Intermediate of synthesis reaction. When H <sub>2</sub> SO <sub>4</sub> is added, the salicylic acid precipitates, via the Common-Ion Effect
Methanol		A by-product formed at the same time as salicylic acid.
Aqueous sulfuric acid (3M)		This precipitates salicylic acid from disodium salicylate
Salicylic acid	A type of phenolic acid; important active metabolite of aspirin	This is the compound which we want to synthesize
<b>Expt #11: Unknown Ketone Identification</b>		
2-propanone (acetone)	These are all homologues methyl ketones	One of these compounds were given at the start of the experiment. The objective was to identify which one you had. Three pieces of evidence were melting point, TLC, and H-NMR spectrum. For TLC, all these ketones will be spotted on the plate.
2-butanone (MEK)		
2-pentanone		
2-hexanone		
2-heptanone		
2-octanone		
95% ethanol	Part of the DNPH reagent	
DNPH reagent	Contains sulfuric acid and 2,4-dinitrophenylhydrazine, and aqueous ethanol	This reagent creates a ketone derivative for the ketone to be studied under TLC, since the color spots can easily be located. (Otherwise, a visualizing reagent would have to be used to see the spots on the plate)

Sodium bicarbonate (aqueous, 5%)		Removes any excess sulfuric acid (from the DNPH reagent)
Ethyl acetate		Used in TLC to dissolve the ketone derivative
3:1 toluene/petroleum ether		Used in TLC to develop the plate in chamber.
Silica gel		The adsorbent on the TLC plate.
Deuteriochloroform (CDCl <sub>3</sub> )		Used in H-NMR. Dissolved with the unknown ketone
TMS		Used as a reference standard in H-NMR spectrum

### Expt #7: Green Synthesis of Camphor

Isoborneol	A secondary alcohol. (Note, also sublimes along with camphor during sublimation. Though we can calculate how much using the melting-point depression equation)	Will be <u>oxidized</u> to a ketone i.e. camphor
Glacial acetic acid		Facilitates in the oxidation by converting bleach to HOCl
Sodium hypochlorite solution (bleach, NaOCl)		This plays the role as the oxidizing agent and will oxidize isoborneol into a ketone, i.e. camphor. This will be kept in excess (if H <sup>+</sup> is present), though must be added a little at a time, since reaction is exothermic. Otherwise, camphoric acid may form.
Hypochlorous acid (HOCl)		Formed from NaOCl and acetic acid. Likely is the active oxidizing agent.
Sodium bisulfite solution		This will reduce/get rid of any excess bleach remaining after the reaction is done.
Camphor	A ketone. Very compact molecular structure which makes it an ideal candidate for sublimation since it changes directly from a solid to a vapor.	The product
Camphoric acid		Unwanted by-product.

### Expt #42 (handout) Haloform Oxidation of 4'-Methoxyacetophenone

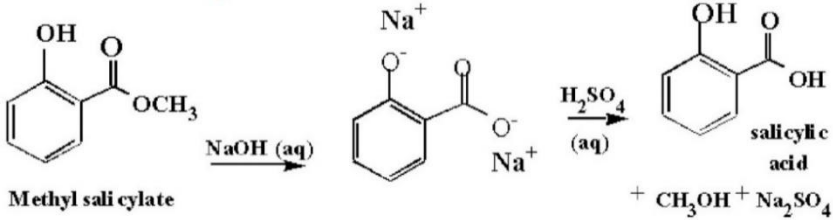
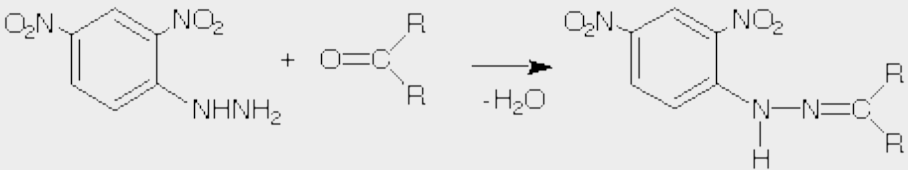
4'-methoxyacetophenone		Starting material. Limiting reagent. Reacted with bleach in the haloform oxidation reaction
Tricaprylmethylammonium chloride		A phase transfer catalyst. Helps ionic species involved in reaction cross the phase boundary. Similar to "detergent" in that it bridges molecules between two phases, the same way detergents bridge polar and nonpolar compounds
Bleach (NaOCl)		Reacts with starting material to convert to the carboxylic salt, sodium hydroxide and chloroform. Reagent in excess
Acetone		Consumes any excess bleach
Chloroform	A haloform. Toxic	A by-product in the reaction
Ethyl ether solvent		Used to wash the reaction mixture in a separatory funnel. It is the aqueous layer which is saved.

Hydrochloric acid		Precipitates product, mixture tested to ensure pH is below 2
Cold water		Washing solvent in vacuum filtration
4-methoxybenzoic acid		Final product
60% aqueous ethanol		Recrystallization [mixed] solvent
<b>Expt #20: Reaction of Iodoethane with Sodium Saccharin, an Ambient Nucleophile</b>		
Sodium saccharin		This is the ambient nucleophile i.e. contains two atoms which can potentially be the nucleophile (nitrogen and oxygen)
Iodoethane		The iodide in this compound is the leaving group
N-N-dimethylformamide (DMF)		The solvent in this reaction. It is polar and aprotic which is ideal for our S <sub>N</sub> 2 reaction since it will not weaken our nucleophile by solvating it [h-bonding]
N-ethylsaccharin		Possible product. More stable. <u>Thermodynamic product</u>
O-ethylsaccharin	Has a higher melting point than N-ethylsaccharin, and further upfield on H-NMR spectrum	Possible product. Forms faster as oxygen is more electronegative. <u>Kinetic product.</u>
<b>Expt #30: Synthesis of Triphenylmethanol and the Trityl Carbocation</b>		
Bromobenzene		Starting materials; together they will form a Grignard reagent, phenylmagnesium bromide.
Magnesium		
Phenylmagnesium bromide	Grignard reagent.	
Diethyl ether		Solvent for reaction
Biphenyl		An undesirable by-product when too much bromobenzene is used. Formed via a side reaction with the metal's surface. Removed via trituration. Constant dilution with diethyl ether limits the formation of biphenyl.
Benzophenone	A phenyl ketone	Added to the Grignard reagent (i.e. PhMgBr) to form the magnesium salt of triphenylmethanol (pink), a "building block" for our synthesis
Water		Added after the benzophenone to form the triphenylmethanol
Hydrochloric acid (1.4M)		Dissolves the basic magnesium salts which have formed along with product.
Triphenylmethanol		Precipitates once Grignard reagent is added with benzophenone followed by addition of water and HCl. This is the precursor for the carbocation
Aqueous (5%) Sodium bicarbonate		Used to wash the ether layer when washing in separatory funnel (note, the aqueous layer is discarded)
Saturated aqueous sodium chloride		Also used to wash the ether layer when washing in separatory funnel
Hexanes		Triturates the triphenylmethanol
2:1 mixture of hexanes		Recrystallization solvent

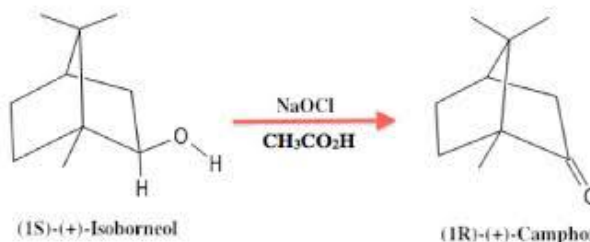
Fluoboric acid (48%)	Strong acid, weak nucleophile anion.	Added in final step to triphenylmethanol to form the carbocation
Acetic anhydride		Added to consume the water from HBF <sub>4</sub> and water that may have been formed during C <sup>+</sup> formation. Forms acetic acid. Reaction can be seen in Table 5
Trityl Fluoborate		The final product. Contains the carbocation which is of interest in this experiment. Color is to be determined.
<b>Expt #29: Borohydride Reduction of Vanillin to Vanillyl Alcohol</b>		
Vanillin		Starting material
Sodium borohydride (NaBH <sub>4</sub> )		Reducing agent.
Aqueous sodium Hydroxide		Reaction solvent.
Hydrochloric acid (3M)		Decomposes excess sodium borohydride
Vanillyl alcohol		Product
Distilled water		Recrystallization solvent. (vanillyl alcohol is soluble in hot water, relatively insoluble in cold water) (ethanol could have also worked)
Hydrogen gas		A by-product; generated when HCl is added to acidify excess NaBH <sub>4</sub>
Diborane (B <sub>2</sub> H <sub>6</sub> )		Another by-product which can generate when acid is added.

### Key Reactions of Each Experiment:

**Table 5**

<u>Title of Experiment:</u>	<u>Reaction</u>
<b>Synthesis of Salicylic Acid from Wintergreen Oil (#4)</b>	 <p style="text-align: center;">This is an organic synthesis. Hydrolysis reaction. Note methanol, water and Na<sub>2</sub>SO<sub>4</sub> are by-products</p>
<b>Identification of Unknown Ketone (#11)</b>	 <p style="text-align: center;">The big molecule on the left is the DNPH reagent, reacting with the methyl ketone. The product is 2,4-dinitrophenylhydrazone. This product is prepared solely for the analysis in TLC. The spots will be colored and be easy to view on the plate. (No visualizing reagent will be necessary)</p>

**Green Synthesis of Camphor (#7)**



We are oxidizing a secondary alcohol, i.e. isoborneol to a ketone, i.e. camphor.

The oxidizing agent used here is bleach, i.e. NaOCl.

A little bit of acetic acid is added in order to facilitate the oxidation by converting NaOCl to HOCl.

Reaction is exothermic. Therefore NaOCl is added a little bit at a time, while monitoring the temperature to remain below 50°C

Excess bleach is reduced by sodium bisulfite to form HCl and HSO<sub>4</sub><sup>-</sup>

After sublimation, it is assumed only camphor and isoborneol remain. By measuring the melting point of camphor, you can calculate how much impurity (i.e. isoborneol) is present using the following

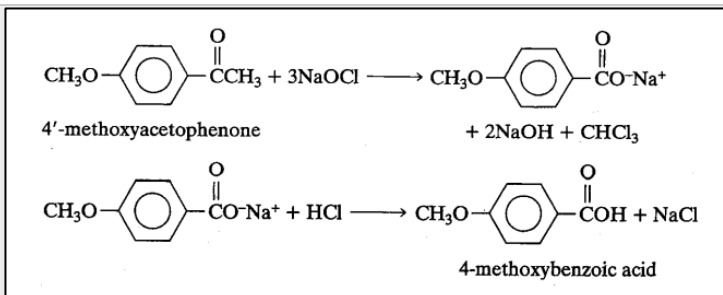
equation:  $\Delta T = K_f \times m$

$\Delta T$  = melting point depression (literature m.p. - observed m.p.)

$K_f$  = f.p. depression constant for camphor = 40°C kg mol<sup>-1</sup>

$M$  = molal concentration of isoborneol

**Haloform Oxidation of 4'-Methoxyacetophenone (handout)**

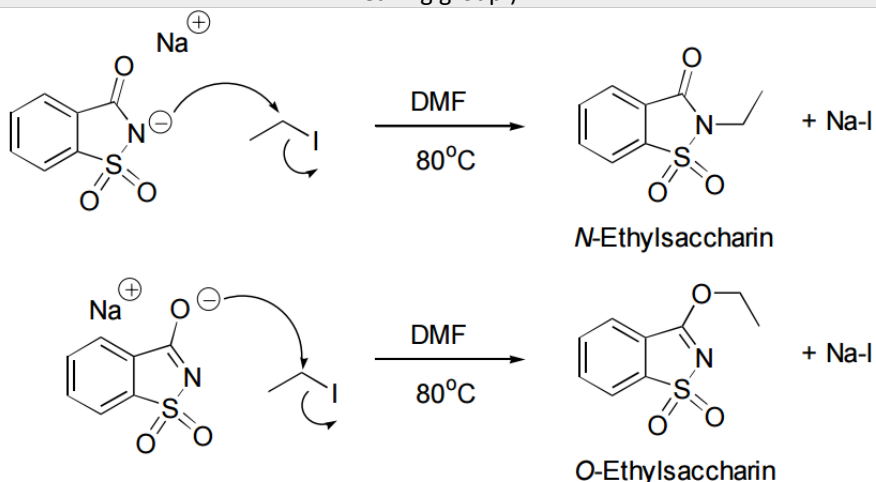


Top reaction is essentially two separate stages:

First, base-catalyzed halogenation reaction

Second, nucleophilic acyl substitution. (Hydroxide ion is the nucleophile and CCl<sub>3</sub><sup>-</sup> is the leaving group.)

**Reaction of Iodoethane with Sodium Saccharin, an Ambient Nucleophile (#20)**



**Figure 2. Mechanism of Alkylation of Sodium Saccharin**

This is a nucleophilic substitution reaction.

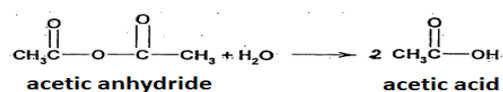
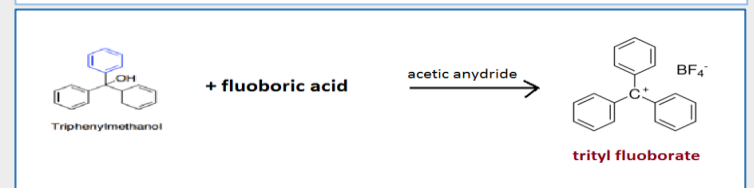
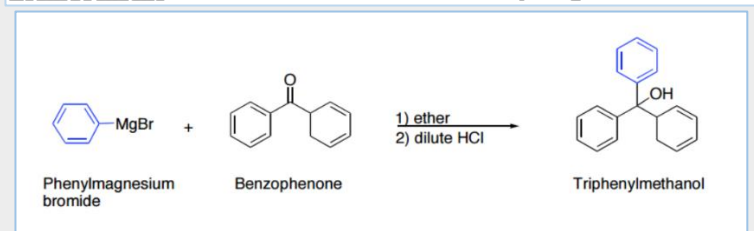
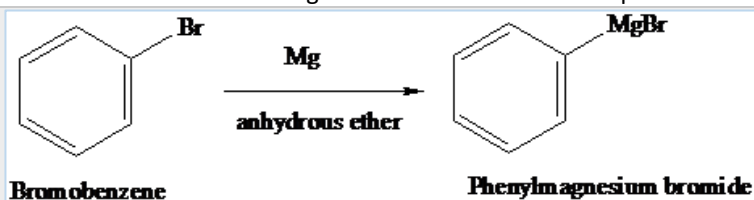
The nucleophile here can be either oxygen or nitrogen. Hence, the term “ambient nucleophile”. Each nucleophile will obviously yield a slightly different product. The objective here is to see which product is major, i.e. which nucleophile dominated the reaction.

The I<sup>-</sup> from iodoethane acts as the leaving group.

The solvent used, DMF, is polar aprotic. Aprotic solvents are essential in nucleophilic reactions, as we do not want to hinder the strength of our nucleophile by solvating it. In other words, water would be a terrible choice for a solvent.

The H-NMR spectrum showed a peak at about 4.7ppm for the O-product and a peak at around 3.9ppm for the N-product. Most students obtained a larger peak at the nitrogen peak, and it was concluded the nitrogen dominated as the nucleophile in this reaction.

### Synthesis of Triphenylmethanol and the Trityl Carbocation (#30)

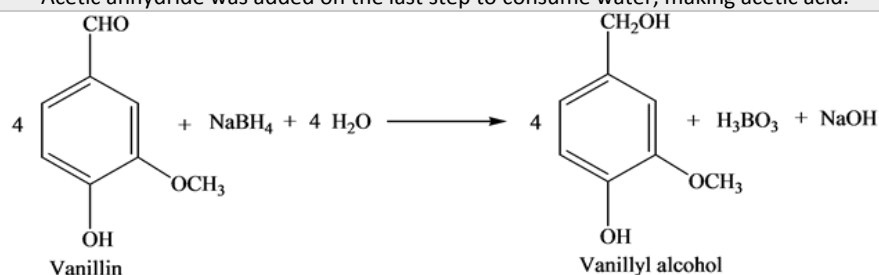


Conditions must be extremely dry in order for Grignard reaction to proceed properly. The Grignard reagent, phenylmagnesium bromide reacts with benzophenone, and then water, to form triphenyl methanol, a magnesium salt.

The final step of the reaction involves adding strong fluoboric acid to prepare the carbocation.

Acetic anhydride was added on the last step to consume water, making acetic acid.

### Borohydride Reduction of Vanillin to Vanillyl Alcohol (#29)



This week was a synthesis reaction.

Students had to create their own plan on how to convert vanillin to vanillyl alcohol.

The reaction would be a reduction of vanillin, as there is a loss of the carbonyl group.

The absence of the carbonyl group should be a apparent in the IR spectrum obtained at the end of the experiment.

In order for vanillin to be reduced, we will need a reducing agent. Sodium borohydride (NaBH<sub>4</sub>) was then reducing agent.





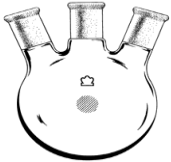
(Lithium aluminum hydride, another reducing agent we learnt in the lecture, is too powerful and hazardous to use in this lab)

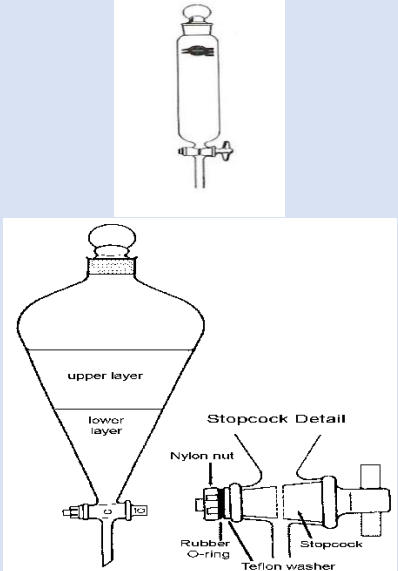

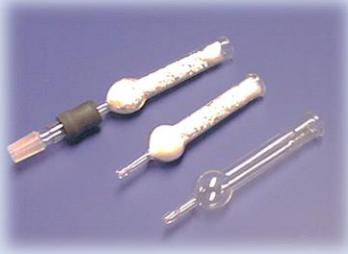
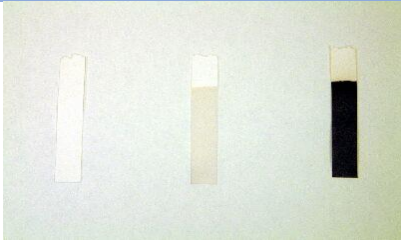


Dilute aqueous NaOH was the solvent in this reaction.

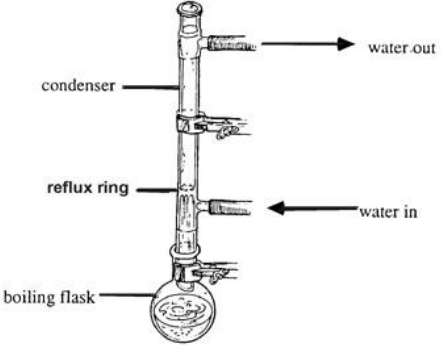
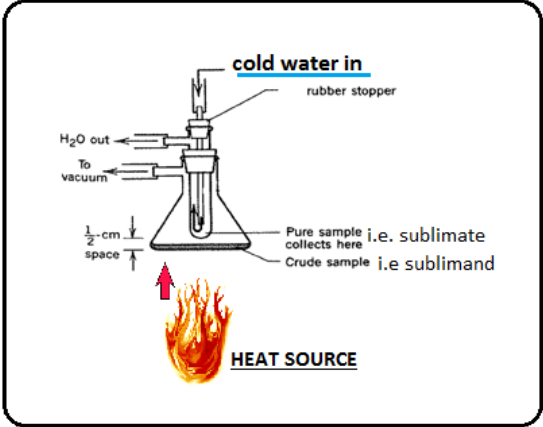
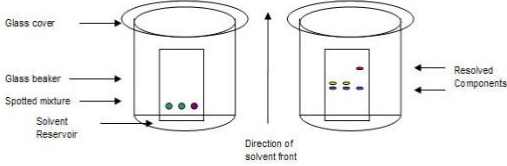
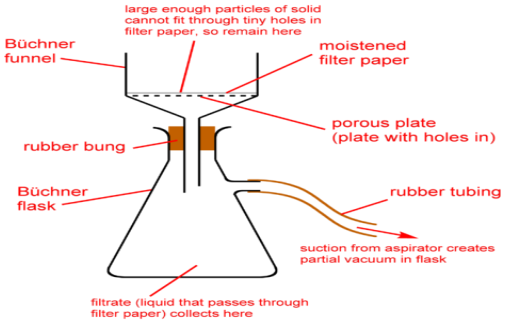
Reaction kept below 25°C

Excess  $\text{NaBH}_4$  was reduced by hydrochloric acid.

### Important Instruments and Apparatus Setups:

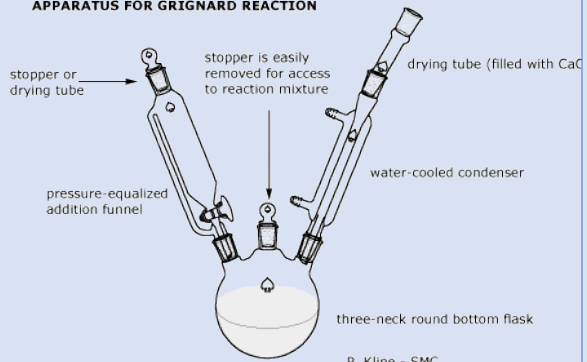
<u>Name:</u>	<u>Diagram:</u>	<u>About:</u>	<u>When it's Used:</u>
<b>Flat-bladed micro spatula</b>			
<b>Scoopula</b>			
<b>Boiling chips</b>		A tiny, unevenly shaped piece of substance added to liquids to make them boil more calmly. They are usually made from porous pieces of alumina, carbon, etc	Used when heating chemical reactions to prevent "bumping" or violent bubbles. Usually just a few are added to reaction vessel.
<b>Buchner funnel</b>		Traditionally made of porcelain. (Hirsch funnel has a similar design; it is used similarly, but for smaller quantities of material.)	Commonly used in vacuum filtration operation. Filter paper is usually added on top to collect solid.
<b>Three-neck round-bottom flask</b>			In this course, this was used in experiment #30 during the Grignard reaction. One opening contained the separatory funnel. One had a stopper. The third opening has a condenser with a drying tube on top. The apparatus is included below in this table under "Grignard Reaction"

<p><b>Separatory funnel</b></p>			<p>Usually used in liquid-liquid extractions and also for washing solvents. Two layers can be separated by allowing one to flow out into a flask or beaker.</p>
<p><b>[West] Condenser</b></p>		<p>Used to condense (change the physical state of) a substance from its gaseous to its liquid state</p>	
<p><b>Drying Tube</b></p>		<p>Essentially a glass tube packed with a drying agent. Its function is to keep its hos vessel dry and free from any moisture,</p>	<p>Encountered in Expt #30. This ensured that any moisture would not enter into the Grignard reaction, which was meant to be as dry as possible.</p>
<p><b>Iodide-starch test</b></p>		<p>Used to test the presence of starch, producing purple-black color. This indicator paper is impregnated with starch and potassium iodide what twill will oxidize ions to iodine, making the dark color.</p>	<p>Encountered in experiment #7, to test the presence of excess bleach, the oxidant.</p>
<p><b>Filter flask</b></p>		<p>Thick-walled with a side arm on its neck</p>	<p>Used primarily in vacuum filtration. Sidearm attached to inlet of water aspirator or to a vacuum line.</p>
<p><b>Magnetic stirrer/ stir bar</b></p>		<p>Employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly, thus stirring it. Most common</p>	<p>Often used in conjunction with heating mantles. There lies a bar magnet underneath the metal platform which rotates. As the bar magnet spins, the</p>

<p><b>Heating Under Reflux</b></p>		<p>one used is a Teflon-coated magnet.</p>	<p>stir magnet in the vessel spins along with it.</p>
<p><b>Sublimation setup</b></p>			
<p><b>TLC</b></p>		<p>Encountered in Experiment #11 to identify the unknown ketone</p>	
<p><b>Vacuum Filtration</b></p>			

**Grignard  
Reaction from  
Expt # 30**

**APPARATUS FOR GRIGNARD REACTION**



P. Kline - SMC

*Good Luck*



*All The Best*