Organic Chemistry I – CH03-400112

General Information

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Chapter 1

General Lab Instructions

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Welcome to the Organic Chemistry I Lab. Please pay close attention to instructions given here. They will help you complete your work faster and will result in a better grade.

1.1 Safety Instructions

Safety rules are provided to ensure that the risk to individuals in laboratory work is minimized. The department has legal obligations to ensure that these rules are kept at all times.

1. Safety glasses MUST be worn at all times when in a chemistry laboratory, whether or not experimental work is being performed.
2. Safety requires appropriate dress. No open shoes are to be worn in the laboratory. No short pants are allowed either. Long hair must be tied. Avoid very expensive clothing to the lab as it might get damaged easily. Always wear a lab coat when in the lab.
3. It is prohibited to eat, drink, smoke or mouth pipette in the laboratory.
4. Familiarize yourself with the locations of fire extinguishers, first aid boxes, emergency showers and other safety equipment in or near your laboratory, and how to leave the laboratory in case of an emergency.
5. DO NOT ingest, inhale, or have skin contact with chemicals in the laboratory: if an accidental ingestion, inhalation or contact occurs take

remedial action IMMEDIATELY. The exhaust system is very efficient and circulates the air several times per hour. By handling vaporizing liquids in the hood, you can further help to improve the atmosphere in the lab and reduce smells.

6. DO NOT remove any chemical from the lab without the explicit approval of a supervisor.
7. Keep benches and fume cupboards clean and tidy
8. Handle balances (clean the balance after use), pumps, and other special apparatus in the laboratory with greatest care.
9. Always read the Material Safety Data Sheets (MSDS) of the chemicals you work with. Check these at `http://www.merck.de` or at `http://www.sigma.com`.

1.2 Cleaning

<u>CLEANING GLASSWARE AFTER USE</u>

- rinse the remaining **organic** material with acetone using the bottle you will find in your hood. Rinse directly into the waste bottle in the hood.

- in case of aqueous solutions rinsing with tab water (plus soap if necessary) and deionized water will be sufficient.

- wash the glassware with water and soap using a brush if necessary with normal tab water. Rinse with deionized water afterwards.

- In case you need to use the glassware again please dry with one of the heating guns you will find in the lab or put the glassware in the big drying oven in the rear left corner of the lab.

1.3 Lab Report Format

Laboratory reports are a good way to evaluate how well the student understands the theoretical and practical aspects of the experiments that they are conducting. All lab reports must be written in the lab on the same day the experiment is conducted. Try and utilize the free time during experiments to write up the apparatus, theory and procedures. Fill in the observations and results once the experiment is completed.

Be concise. Do not blindly copy procedures and theory from the handout. Write these up in your own words as this will help develop your understanding of the subject matter.

Please note: It is strictly forbidden to write lab reports with gloves on your hand and to handle with chemicals on your working bench.

The laboratory reports are restricted to 2 sheets of A4 paper which are part of your laboratory manual (after each experiment). All should have, at least, the following sections:

Aim: The aim should outline the general objective of the experiment.

Apparatus and Materials List all the apparatus, glassware, chemicals etc. that were used during the experiment.

Theory and Mechanism: The theory is one of the most important parts of the lab report. Explain the mechanism of the reactions in consideration and the techniques you used to complete the experiment successfully.

Procedure and Observations: Write down the procedure of the experiment briefly in your own words along with what you observed during the course of the reaction. Try to elaborate on the purpose of each step of the experiment.

Results and Yield Calculations: Outline the results of your measurements and calculations. Assert whether the experiment was successful (as expected) or whether something went wrong. In the latter case, explain possible reasons for an unsuccessful experiment.

1.4 Grading

The grading scheme of the lab is as follows:

- 50% for laboratory reports. The quality, conciseness and accuracy of the student's lab reports shall determine their grade in this evaluation.
- 25 % for general laboratory practice and laboratory evaluation. The lab instructor and teaching assistants will evaluate the performance of the student on criteria such as cleanliness, care for glassware and equipment, careful handling of chemical (according to MSDSs) etc.
- 25 % Yield from the experiment.
  After each experiment you will obtain something to fill in a bottle or a viol you will be provided with in the laboratory.
  Depending on the type of experiment it will be a solid or a liquid. Please never discard it but store it somewhere in the back of your working bench.

Chapter 2

Laboratory Apparatus And Techniques?

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The organic chemistry lab has a variety of tools and glassware to ensure a high quality and quantity of synthesis. The apparatus and the way they are used as described in this chapter.

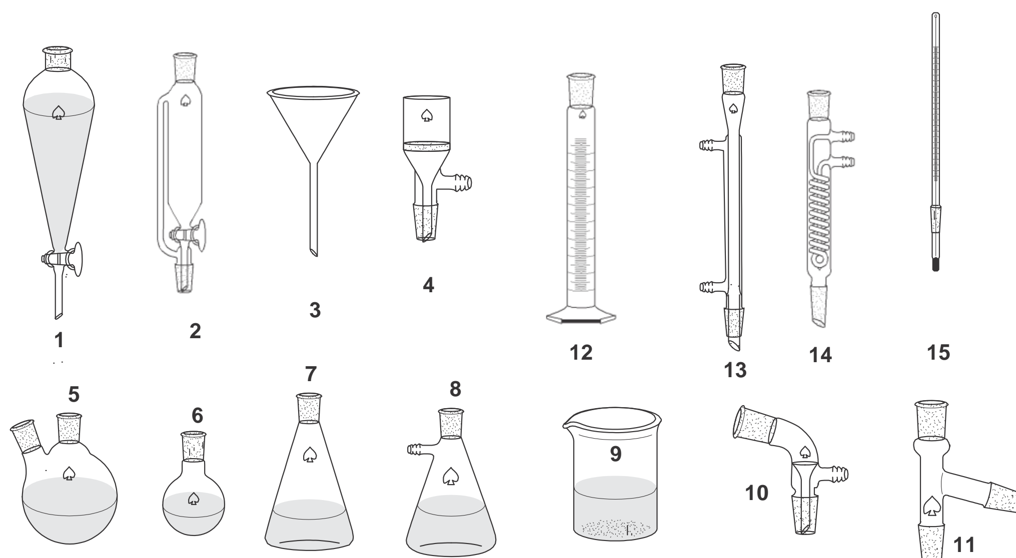

Figure 2.1: *Different kinds of glassware in the lab*

2.1 Glassware

You will mostly be using the following glassware in this lab course (see Figure 2.1).

1. Separatory funnel
2. Dropping funnel
3. Simple glass funnel
4. Vacuum filtration funnel
5. Two-necked round bottom flask
6. Round bottom flask
7. Erlenmeyer flask
8. Vacuum filtration flask
9. Glass beaker
10. Distillation adaptor (used also for vacuum distillation)
11. Three-way adaptor
12. Measuring cylinder
13. Liebig condenser
14. Reflux condenser
15. Thermometer

2.2 Solvent Extraction

Apparatus: *Separatory funnel, plastic stopper, beaker or Erlenmeyer flask.*

Theory: Organic and aqueous phases are immiscible (e.g. oil and water) The resulting Gibb's free energy (ΔG) of mixing is positive and hence they do not mix. Also, organic molecules dis- solve in organic solvents and ionic molecule dissolve in water (like dissolves like). Hence, we can *extract* organic molecules present in the aqueous phase by choosing an appropriate organic solvent and *washing* the water aqueous phase with it.

Method: Pour the aqueous phase and the organic solvent into the separatory funnel. Make sure the stop-cork at the bottom of the funnel is closed. Close the funnel with the plastic stopper. Holding the stopper firmly against the funnel, invert the funnel and shake gently. Release the pressure built up inside the funnel by opening the stop-cock at the bottom of funnel. Close the stop-cork. Repeat this process until no more pressure is created. Shake vigorously for a short time. Clamp the funnel from the top and let the two phases separate. Identify the organic phase and isolate it in a flask or beaker. It is a good idea to store the aqueous phase until you have your final product in hand.

Note: It is a good idea to wash the aqueous phase in 2-3 aliquots of the solvent as this increases the amount of organic molecules extracted and will increase the yield of your reaction. Also, before isolating anything from the funnel, make sure you uncap the plastic stop-cork.

2.3 Drying

Apparatus: *Filter paper, simple glass funnel, Erlenmeyer flask, hygroscopic salt.*

Theory: After solvent extractions there is however a slight amount of water still present in the organic phase. We remove this water by mixing the organic phase with a strongly hygroscopic salt (e.g. Na_2SO_4, $MgSO_4$ or $CaCl_2$) and then carrying out a simple gravity filtration (since the salt is insoluble in the organic phase).

Method: To the organic phase in the Erlenmeyer flask, add 5-6 spatula tips of the hygroscopic salt and gently swirl for 2 minutes. Gravity filtrate the mixture into the desired flask. Always check if there is any water still present in the filtrate. As a rule, the organic solution should turn from opaque to clear during drying and the salt should float like snowflakes when you shake the solution.

Note: There are other ways of drying. This is by washing the organic phase with a saturated solution of a highly ionic salt (like NaCl). Based on the principle of *osmosis*, the saturated salt solution will try to dilute itself by pulling the water in the organic phase into the aqueous phase. This method is

however rarely used. You may also add salts to accelerate phase separation.

2.4 Filtration

Apparatus: *Vacuum filtration flask and funnel, rubber ring, vacuum connection, filter paper, simple glass funnel*

Theory: We use filtration to separate any solid substrate from a solution by passing the solution through a filter paper of a particular pore size. It is important to judge however, when to use vacuum filtration and simple gravity filtration for separation. When we are interested in isolating the solid substrate in the solution, we use vacuum filtration and when we are interested in isolating the liquid phase, we generally use gravity filtration.

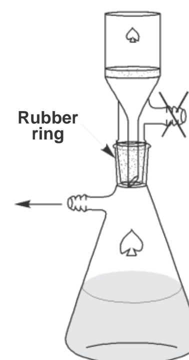

Figure 2.2: *Vacuum Filtration Apparatus*

Method: The setup for vacuum filtration is shown in Figure 2.2. Do not forget to put the rubber ring on the Filtration flask as this maintains the vacuum in the flask. Place the filter paper in the funnel. Connect the vacuum pipe (in our case, the vacuum shall be created from running water with a water aspirator) to the flask and turn it on. Put some drops of the solution you want to filtrate. You will know if you apparatus is correctly setup if you see the paper being sucked by the funnel. If this does not happen, press down on the funnel firmly into the flask. Pour your solution into the funnel. Sometimes it is necessary to *wash* the product in the funnel with some other solution. Pour out the entire amount of this solution into the funnel in one go without spilling anything. Do not pour it in small amounts. It is a good idea to leave the vacuum on after the filtration so that the product in the funnel gets dried.

Note: *Always remove the vacuum pipe from the filtration flask BEFORE turning off the vacuum.* If you do not do this, the vacuum already present in the flask will suck the water from the tap providing the vacuum, into the filtration flask.

2.5 Distillation

Apparatus: *Round bottom flasks, Liebig condenser, three-way adaptor, distillation adaptor, thermometer, magnetic stirrer with stirbar, water/oil bath, water pipes and water supply, vacuum connection*

Theory: Distillation is the process by which we separate components of a mixture based on the fact that they have different boiling points. During distillation, the thermometer reading will stabilize to a particular value, which is the boiling point of the component being distilled. The component

(now in vapour phase) is condensed into a liquid using a condenser. The distillation process is complete when there is a noticeable drop 10-20°C in the temperature reading of the thermometer. Vacuum distillation is a technique by which we can distill components with a very high boiling point. *The boiling point of a substance is defined as the temperature at which its vapour pressure is equal to the external pressure*. Hence, if we reduce the external pressure by applying a vacuum, the boiling point of the component will also be decreased. In general, the boiling point can be easily lowered by ca. 50°C in this way (ca. 100°C if you apply better pumps).

Method: The distillation apparatus setup is shown in Figure 2.3. Make sure all the apparatus is dry before use. Do not forget to clamp all joints (adaptors with the Liebig condenser) with the plastic clamps provided. Use a water OR oil bath for heating the solution. Always stir while distilling. If the distillation gets too violent (bubbling into the condenser) due to exceeding temperature or high vacuum, lower the water OR oil bath immediately, then turn down the heat. *Never turn the heater to above 250°C*. This will damage the magnetic stirrer. Note the input and output for the water passing through the condenser. In order to save water, do not turn the water on too high through the condenser. When vacuum distilling, *always remove the vacuum pipe from the apparatus before turning off the vacuum*.

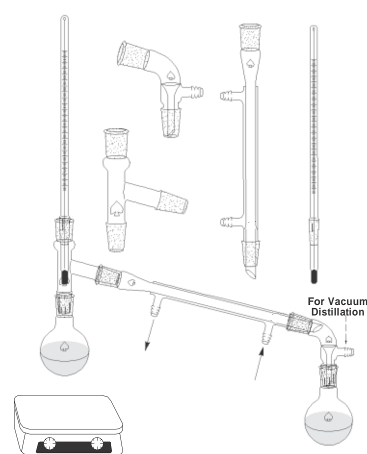

Figure 2.3: *Distillation Apparatus*

2.6 Refluxing

Apparatus: *Round-bottom flask, Reflux condenser,Magnetic stirrer with stirbar, water OR oil bath, water pipes, and water supply*

Theory: Refluxing is a method by which a reaction or recrystallization can be carried out at higher temperatures while not allowing the reactants or solvent to evaporate. A reflux condenser is attached to the top of a round bottom flask and cold water is passed through it. All vapour *condense* on the interior surface of the condenser and are dropped back into the reaction mixture.

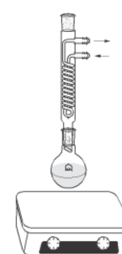
Figure 2.4: *Refluxing Apparatus*

Method: The refluxing apparatus arrangement is shown in Figure 2.4. Always stir while refluxing. In order to save water, do not turn the water on too high through the condenser. *Do not cap the reflux condenser*.

2.7 Recrystallization

Apparatus: *Round-bottom flask, water OR oil bath, refluxing apparatus* (when instructed)

Theory: Recrystallization is a techniques used to purify an organic product. The principle behind recrystallization is that the amount of solute that can be dissolved by a solvent increases with temperature. At the cooler temperature, the solution is saturated at a much lower concentration of solute. The solute that can no longer be held in solution forms purified crystals of solute, which can later be collected. Recrystallization works only when the proper solvent is used. The solute must be relatively insoluble in the sol- vent at room temperature but much more soluble in the solvent at higher temperature. At the same time, impurities that are present must either be soluble in the solvent at room temperature or become soluble in the solvent at a high temperature but do not crystallize out when cooled down again.

Method: Take the crude product in a round bottom flask and add enough solvent to cover the entire product. Put in a magnetic stirrer and start stirring. Using a water bath or oil bath, heat the mixture while stirring to the temperature given in the procedure. Use a thermometer to keep check of the temperature of the water or oil bath. When there is no more solid substrate left to be dissolved in the flask, stop the heating and stirring. If required, a little more solvent can be added through the reflux condenser. Remove the magnetic stirrer and set the flask aside for cooling. In some cases, ice will be needed for further cooling. When no more crystals form on further cooling, the recrystallization is complete.Vacuum filtrate the mixture to obtain the pure solid product.

2.8 Rotavaporization

Apparatus: *Round-bottom flask, Rotatory evaporator*

Theory: Rotavaporization or *rotatory evaporation* is used to remove the solvent from our reaction mixture or from the liquid phase obtained after drying and filtration. It is based on the principle of distillation. This is usually aprecursor to recrystallization. We normally use this technique when we know that the product of our reaction is solid. Frequently, it has been used to recycle organic solvents.

2.9 Characterization

2.9.1 Chromatography

Apparatus: *Silica-gel TLC Plate, Ruler, Pencil, capillary tube, polar and non polar solvents, beaker.*

Theory: Chromatography is an extremely versatile method in analytics as well as in preparation. It is commonly used for the following purposes:

- To control the reaction progress.
- To check the identity and purity of educts and products.
- To separate mixtures of compounds.

There are 2 kinds if chromatography

Liquid Chromatography: Column or Layer chromatography is useful in the separation of solids and oils with a high vapor-pressure but not useful to separate low-boiling point liquids.

Gas Chromatography: Gas chromatography is very good for separation of volatile compounds.

The most common method is the analytical layer chromatography, (Thin Layer Chromatography, TLC). Normally, silica-gel with a thickness of 0.2mm as supporting material is standard.

Method:

Preparing a TLC: A diluted solution of the test substance (or reaction mixture) is applied to a start line on the TLC plate together with solutions of the educt (to compare the reaction progress) with the aid of a capillary tube. Then the chromatogram is developed in a special chromatography vessel. After drying the chromatogram, the substances on the TLC can be made visible.

Making substance on a TLC visible:

- The easiest way is to use a lamp that radiates UV light (254nm) is layers with a fluorescent indicator were used.
- The vapor of iodine tints the spot on the TLC.
- Spraying methods: A solution that contains 1% $KMnO_4$ for -OH, -SH, -NH, C=C and C=O group containing substances; 2,4-dinitrophenylhydrazine solution for C=O group containing substances and sugars; Conc. H_2SO_4 and heating to 120^oC for almost all organic substances.

The development of a TLC is done either with a pure solvent or with a mixture of different solvents. It is useful to check several solvent mixtures to get and optimal separation.

By using pure solvents (eluents), the so-called eluotropic row, where the solvents are ordered by increasing polarity:

Pentane - Cyclohexane - Tetrachloromethane - Benzene (or Toluene) - Dichloromethane - Diethyl ether - Ethylacetate - Acetone - Methanol - Water - Acetic Acid - Pyridine

Well proved eluent systems are:

- For neutral compounds: $CHCl_3$/MeOH (100:1, 10:1 or 2:1), Diethyl ether/*n*-Hexane (1:1), Diethyl ether/Acetone (1:1), Ethylacetate/*n*-Hexane (1:1), Ethylacetate/Isopropanol (3:1).
- For acidic compounds: $CHCl_3$/MeOH/AcOH (100:10:1).
- For basic compounds: $CHCl_3$/MeOH/conc. NH_3 (100:10:1)

Determining the R_f value: For documentation, the TLCs are drawn and the relative position of the center of a spot to the start line, divided by the distance traveled by the eluent from the start line is calculated. This quotient gives the R_f value of the substance.

Column Chromatography: A preparative separation of compound mixtures is possible by using column chromatography. The choice of the best eluent for a preparative column separation is determined by TLC. The substance should form a round spot and should have a R_f-value between 0.2 and 0.3 to have a good result.

The quantity of the adsorbent should be at least hundred-fold, by weight, larger than that of the mixture to be separated. The adsorbent is combined with the eluent in a laboratory cup and stirred with a glass rod slowly to get rid of bubbles. This mixture is then carefully transferred into the column. The formation of bubbles must be avoided. It is therefore helpful to turn around the column between the hands. For an ambitious separation, the column should stand for 12 hours.

The eluent is then dropped off until a layer of 1mm rests over the adsorbent surface and the substance, dissolved in the eluent, is carefully introduced into the column. The substance is then infiltrated and covered again with a small quantity of eluent, and infiltrated again. This procedure is repeated several times. Then, the surface of the adsorbent is covered with a swab and the column filled with eluent. The column is now ready to run. (For a 40cm column, the dropping speed should be around 3-5ml/min)

Fractions are collected in test tubes and analyzed by TLC. Uniform fractions are put together and the solvent is removed by rotavaporation. Do not forget to make a mass balance of all fractions and to correlate it with the substance quantity you started with.

The column should never run dry and a started separation should not be interrupted. The room temperature should be low to avoid decomposition of substances and the formation of glass bubbles (Diethyl ether, Pentane and Dichloromethane as eluents).

Most separations in organic chemistry are carried out on silica-gel. If this is not successful, however, Aluminium oxide, cellulose, or polyamide could be used.

Criteria for purity and control of purity: To check the purity of a known synthesized compound, boiling point, melting point and eventually, refractive index measurements as well as spectroscopic data (UV-Vis, IR, NMR, MS) are determined and compared with literature data.

To identify small quantities of impurities, gas-chromatography, TLC and high performance liquid chromatography (HPLC) are useful methods, especially to determine the purity of newly synthesized compounds. An elementary analysis that fits with theoretical values is also a good criteria for purity.

2.9.2 Melting Point

Apparatus: *Glass tubules, Melting point machine*

Theory: Most *solid* compounds have distinct melting points, i.e. the temperature at which a phase change from solid to liquid takes place. On this basis, we can identify certain molecules based on their known melting points.

Method: The solid analyte is loaded into a glass tubule and put in the melting point machine. The approximate melting point of the suspected substance is entered into the machine. The melting point program is started and three points are noted (on the machine) based on the observations of the experimenter. One, when the melting starts, the second, when the solid completely liquifies, and third, when the liquid starts to vaporize. The average of these three values give the melting point of the solid analyte.

Please ask the TAs to assist you while taking these measurements.

Note: It is common to see higher melting points than expected. This is indicative of the higher purity of the analyte.

2.9.3 Refractive Index

Apparatus: *Refractometer, pasture pipette.*

Theory: In order to characterize *liquid* compounds, the property of their refractive index is exploited. The refractive index of a material is defined as the ratio of the speed of light in vacuum to that of the speed of light in that

particular liquid compound

$$\mu = \frac{c_o}{c}$$

Method: A few drops of solution are added to the glass slide of the refractometer. It is adjusted such that a distinct division is made (half bright and half dark) right through the center of the objective. The refractive index is then observed on the green scale which is observable through the objective of the refractometer.

Please ask the TAs to assist you while taking these measurements.

2.9.4 Spectroscopy

NMR

Nuclear Magnetic Resonance Spectroscopy is the name given to the technique which exploits the magnetic properties of nuclei.

Many areas of information can be obtained from this single phenomenon. In its simplest form NMR allows identification of individual atoms in a pure molecule. Much like using infrared spectroscopy to identify functional groups, analysis of a 1D NMR spectrum tells the scientist what atom environments (like a methyl proton), and in some cases how many atoms of each type, exist within the sample. NMR is based in quantum mechanical properties of nuclei, and as such is very reliable, predictable and reproducible.

NMR Spectroscopy is much more powerful than this everyday usage. It can be used to study mixtures of analytes; to understand dynamic effects such as change in temperature and reaction mechanisms; it can be used in the solution and solid state; and critically it is an invaluable tool in understanding protein and nucleic acid structure and function.[1]

For a more detailed treatment of NMR spectroscopy, see http://www.cis.rit.edu/htbooks/nmr/inside.htm

IR

An invaluable tool in organic structure determination and verification involves the class of electromagnetic (EM) radiation with frequencies between 4000 and 400 cm^{-1} (wavenumbers). The category of EM radiation is termed infrared (IR) radiation, and its application to organic chemistry known as IR spectroscopy. Radiation in this region can be utilized in organic structure determination by making use of the fact that it is absorbed by interatomic bonds in organic compounds. Chemical bonds in different environments will absorb varying intensities and at varying frequencies. Thus IR spectroscopy involves collecting absorption information and analyzing it in the form of

[1] http://en.wikipedia.org/wiki/NMR_Spectroscopy

a spectrum - The frequencies at which there are absorptions of IR radiation ("peaks" or "signals") can be correlated directly to bonds within the compound in question.[2]

UV-Vis

Many molecules absorb ultraviolet or visible light. The absorbance of a solution increases as attenuation of the beam increases. Absorbance is directly proportional to the path length, b, and the concentration, c, of the absorbing species. *Beer's Law* states that

$$A = e \cdot b \cdot c$$

where e is a constant of proportionality, called the absorbtivity.

Different molecules absorb radiation of different wavelengths. An absorption spectrum will show a number of absorption bands corresponding to structural groups within the molecule. For example, the absorption that is observed in the UV region for the carbonyl group in acetone is of the same wavelength as the absorption from the carbonyl group in diethyl ketone[3].

For a more detailed treatment of UV-Vis absorption spectroscopy, see http://www.cem.msu.edu/~reusch/VirtualText/Spectrpy/UV-Vis/spectrum.htm

2.9.5 Mass Spectrometry (MS)

Mass spectrometry has grown into one of the most powerful tools a chemist has for the identification of unknown compounds. Though not practical for many substances, the technique is useful for a large majority of organic compounds. Coupled with a gas chromatograph, it is possible in many cases to collect mass spectra of pure compounds from submitted samples (of unknown purity) with very little manual sample preparation.

For analytical purposes, the mass spectrum generally contains data corresponding to the masses of decomposition products of a molecular ion. The nature of the decomposition reactions relates quite specifically to the structure of the molecular ion. In a sense, by measuring the mass spectrum, a set of reactions of the molecular ion is inferred. We then use this chemistry to deduce the structure of the unreacted molecular ion.

In brief summation, measurement of the mass spectrum requires ionization of a sample with sufficient energy to cause decomposition reactions, filtering the reactant and product ions based on mass, detection of the ions after filtering and storing the data. Very few mass spectrometry systems are

[2] http://www.chem.ucla.edu/~webspectra/irintro.html

[3] http://www.shu.ac.uk/schools/sci/chem/tutorials/molspec/uvvisab1.htm

operated without computer data systems, and the data system used in this laboratory will be briefly covered in a subsequent chapter.[4]

2.10 Yield Calculations

The theoretical yield of a reaction is the amount of product that would be formed if the reaction went to completion. It is based on the stoichiometry of the reaction and ideal conditions in which starting material is completely consumed, undesired side reactions do not occur, the reverse reaction does not occur, and there no losses in the work-up procedure.

In order to calculate the theoretical yield, you must first balance the reaction. Then, look closely to determine which reagents are being used in excess and which is the *limiting reagent*. The overall yield of product depends on the limiting reagent. Remember that catalysts, solvents, or any compounds that are not part of the actual chemical reaction cannot be the limiting reagent. Theoretical yield calculations are carried out in the same way as they were in general chemistry: the moles of limiting reactant determines the moles of product.

After your laboratory reaction is complete, you will isolate and measure the amount of product, then compare the actual yield to the theoretical yield to determine the percent yield:

$$\%\ Yield = \frac{Actual\ Yield}{Theoretical\ Yield} \times 100$$

In the laboratory, the percent yield has the practical aspect of telling you how successful was your synthesis scheme. A low percentage yield means that the conditions were not optimal and could be improved. Perhaps there are competing reactions occurring or some of the product is being lost in the purification steps. [5]

To calculate theoretical yield:

1. Balance the reaction and determine the stoichiometry or ratios of reactants to products.
2. Find the number of moles of each starting material used.
3. Determine which reagent is limiting.
4. Calculate the moles of product expected if the yield were 100% based on the limiting reagent.
5. Calculate the grams of product corresponding to the number of moles expected.

[4] http://www.dsbscience.com/freepubs/forensic_intern/node39.html
[5] http://orgchem.colorado.edu/hndbksupport/labnb/theoryield.html