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3.0 Lab Techniques (Basic)

This section will cover a number of techniques that are integral to every chemist, amateur or professional. We will cover the basic techniques of refluxing, distillation, and filtration. Refluxing allows for a more complete reaction, allowing reagents to react at temperatures close to, or at, their boiling points. Distillation is useful for two reasons: it can be utilized for separating volatile reagents from their non-volatile counterparts and it can be used to drive a reaction to completion by boiling off the product. Filtering is an essential method of removing solid particulates from liquids. This is especially useful when removing solid catalysts from a reaction mixture. Filtration can also be used to remove insoluble products and effectively clean off floating pieces of gunk that come in your hardware store reagents.

After filtration, this chapter will shift into a section about titrations. This is a very good skill to have under your belt, especially if you enjoy analytical Chemistry or have the need for knowing precise concentrations of solutions. While titrating is usually used for determining the concentrations of acid or base in a sample, they can also be used to determine the oxidizing or

reducing potential of a solution and even metal concentration. Titrations are typically performed with a precisely manufactured piece of glassware called a burette, however, careful use of a graduated cylinder or pipet can give reasonable results for the home chemist.

The chapter continues with a section on heating, cooling, and maintaining reaction temperatures, followed by two methods of purification. These include removing water from gases, liquids, and solids, along with recrystallization. The last section describes how to obtain precise mass and volume measurements and their importance

All of these techniques are integral to your success as a chemist and the authors of this book highly recommend that you dedicate the time to learn each of them. After you master these, check out **Chapter 8** for advanced techniques.

3.1 Refluxing

** Some reactions are incredibly *exothermic* on their own and do not require heat to initiate. That being said, before heating a reaction, you should be certain that doing so will not cause a dangerous situation. Always research reactions before attempting them and always try to start your reactions cold or at room temperature. If heat is absolutely necessary, do so only after you mix the reagents together. **

Refluxing is a common organic synthesis technique, typically used to accelerate reaction rates, while at the same time conserving solvent. The most common method of refluxing a reaction mixture is to submerge the flask in an oil, water, or sand bath that is at a constant temperature, slightly higher than the boiling point of the solvent. A reflux condenser is then attached to the reaction vessel (see left). The condenser must be long enough and cooled with a constant flow of cold water, allowing the gaseous solvent to condense well below the top of the condenser. There will normally be a visual vapor line indicating the height of vapor in the column. Monitoring the vapor trail is especially important if long reaction times are necessary, since this will minimize the amount of solvent loss and prevent the reaction from "burning out". Always be aware of the temperature of the bath and try to keep it as constant as possible.
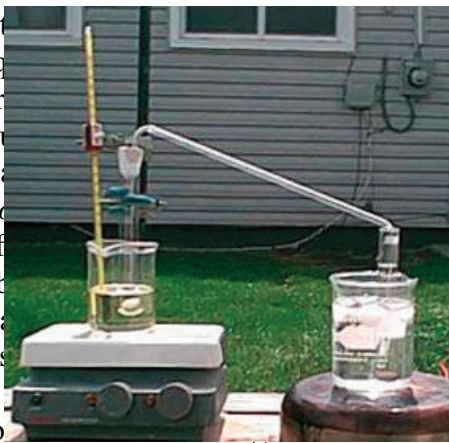
By refluxing for long lengths of time you can react systems with more than one *phase*, insoluble solids with liquids, and simply speed up a reaction. Refluxing is a very helpful and simple technique. Even though it is a relatively easy technique to master, it should not be overlooked.

Note that if you are trying to accelerate the rate of a reaction you should not go from room temperature to refluxing in one step. The safest method is to heat the reaction in increments of 10 to 20 ºC and see if the reaction occurs at a lower temperature than reflux. This is much, much safer than

immediately refluxing a reaction.

3.2a Simple Distillation Introduction

Distillation is one of t[...] all of chemistry and eq[...] Distillation is per[...] volatile product from a sol[...] simplest of distillation app[...] known as *simple distillatio[...]* it exemplifies all f[...] distillation set-up. The tub[...] flask (this is also known a[...] case a test tube. This i[...] this case a glass tube. [...] of cooling the vapors pro[...] vessel. The liquid that is condensed out is called the *distillate*. In this case, the condenser pictured is the crudest design possible. The air cooling is rather inefficient, but it is made better by allowing a fan to blow on it. All other condensers excel in efficiency by using running water to cool them. There are even condensers that accept chilling mixtures, obtaining subfreezing temperatures. A popular mixture is dry ice in an isopropyl alcohol bath, reaching temperatures of -78ºC. The condenser then leads into a receiving flask. This can be cooled, as shown in the picture, and is especially necessary if your product is substantially volatile. The last and most important part of the apparatus is the heat source. Lab hot plates with stirring capabilities are top of the line, and are usually out of the price range for the average home chemist. However, this is not absolutely necessary since anything that gets hot (heating control being preferred) will work. For more precise heat control, the use of some form of 'bath' is advised. This can be as simple as a beaker of a fluid (oil is pictured above) or sand that is heated directly. Bathing your flask provides more even and consistent heating. As a rule of thumb, the bath temperature is usually about 5-10°C higher in temperature than the liquid inside the reaction flask.

*As stated in **Chapter 2**, borosilicate glassware is always preferred for distillation.*

Heating a solution to a boil and condensing the vapour sounds easy, right? Well there are several small, but important factors that need to be kept in mind during a distillation. First of all, the purpose of distillation is to end up with the most volatile compound of your mixture in the receiving flask and the less volatile compound(s) in the distillation flask. Unfortunately, this is not always entirely possible. The other components in your distilling flask also have a vapour pressure, which rises with the temperature. As a result, the vapour you are condensing primarily consists of the most volatile compound, but it will also contain a fraction of the less volatile compound(s). This fraction will be large if the boiling points of the compounds don't differ much (less than 40°C) and small when the boiling points are substantially different.

For example, distilling wine (usually 12.5% by volume of ethanol in water) will yield a solution which is greatly enriched in ethanol content, but it will still contain a considerable (about 50-40%) amount of water. Find boiling point EtOH

The second important factor to note is that as a distillation proceeds, the concentration of the most volatile compound decreases and the boiling point of your mixture rises. As a result,

your vapour will contain more of the lower boiling compound(s) as the distillation proceeds towards completion. There are a couple ways to solve this problem, the most important being *fractional* and *azeotropical* distillation.

3.2b Fractional and Azeotropical Distillation

Fractional distillation is actually quite simple. The main idea is to redistill your distillate until it's pure. Now, if we take our previous example of wine, about three distillations would be needed to attain reasonably pure ethanol. It goes without saying that this is both time and energy consuming. However, there is a piece of glassware that provides a simple solution to this problem: the fractionating column. This simple, but efficient device allows you to separate closely boiling compounds in considerable less runs than a simple distillation would require.

The fractionating column is placed between the heated flask and your still head adapter. Vapours which pass through it cool down as they rise and eventually condense, the compound with the highest boiling points condense first against the walls and packing material. The substance then heats and vaporizes again, following the vapour front, eventually condensing a little bit further up the column. As a result, there are countless condensation cycles taking place in your column. The vapour that reaches the top will almost exclusively contain the most volatile component, unless you are distilling an *azeotrope*. During fractional distillation, it is said that several successive distillations are taking place simultaneously within the fractionating column.

There are several types of fractionating columns and they each have their specific uses. The most commonly used column is the Vigreux column (picture?). It has a relatively small surface area but a high flow rate. The standard 300mm Vigreux column is ideal for separating compounds that have a difference in boiling point of 20°C or more and it can be used for vacuum distillation (see **Chapter 8**). Vigreux columns come in longer sizes and can even be stacked to improve separation. For even better separation, one should consider filled columns. Filled columns come in all different sorts and sizes, but they all work on the principle of maximal surface area and therefore they are usually filled with irregularly shaped objects, such as glass beads or broken glass (see **Chapter 2**). Unfortunately, they do retain a lot of liquid, reducing yields and are not well suited for vacuum use.

When performing a fractional distillation, it is important to constantly monitor the temperature. You will notice that the temperature suddenly skyrockets when the most volatile compound is depleted from your mixture because of the good separation of your column.

Good measuring of temperature can only be achieved by correct positioning of the thermometer, which should be just below the bend towards the condenser so that it is being immersed in the vapour as it distils over. (PICTURE!) Slight deviations of a few mm can cause temperature-reading failures of several degrees Celsius. Another good technique is to insulate the column to minimize heat loss when distilling high boiling temperature compounds. This way the heat can be maintained all the way up the column.

Azeotropical distillation works via a simple trick. A third substance is added to your distillation mixture, this compound forms an azeotrope, preferably with the compound you do not need to isolate. An azeotrope is a mixture of two or more substances, which cannot be separated anymore by distillation, because both components have the same vapour pressure at a specific concentration, known as the azeotropic point. In this case, the azeotrope needs to have a boiling point that differs substantially from the desired component in your mixture. This allows the distillation to occur seamlessly due to the large boiling temperature difference. This process

is often used during esterification. Toluene is added to the mixture, which forms an azeotrope with water and boils off. After cooling down the water and toluene separate back into two layers.

Azeotropes can also complicate a good separation attempt. A good example is nitric acid. Nitric acid forms an azeotrope with water, which contains 69.2% nitric acid. To concentrate further simple distilling won't work and neither does fractional distillation. So what now? The most commonly used method is to break the azeotrope by adding another substance. This is the complete opposite of azeotropical distillation. In the case of nitric acid, the most applied method is to add sulfuric acid, as this has such a great affinity towards water it will easily "steal" the water from nitric acid. As a result the nitric acid behaves as if it were all by itself in your flask and thus obtains its' normal boiling point, which is far lower than the hydrated sulfuric acid. One can also steal the water off by adding a very *hygroscopic* salt like magnesium nitrate. The salt will become hydrated and remain in the distillation flask as nitric acid boils over.

3.2c Distillation Procedure

Chemistry lab setups for distillation use ground glassware. While these can be rather expensive, they perform incredibly well and are versatile. The joints are all tapered glass and fit together snugly, with or without the use of a sealant. There are several different sizes, as with all ground glassware, be sure to pick a size that works well for your applications.


14/20 distilling apparatus, the reaction flask being heated in an oil bath.

**Note from the Authors: The Metal Distillation Apparatus:**

Although the materials section goes to great lengths to show that glass is a great all around material in which to perform reactions, there is no reason a metal distillation apparatus

should not be considered. The most notable advantage of a metal apparatus being the high working temperature and the availability of the materials. Shown above is a copper condenser in production. Such an apparatus could be used to distil any of a number of solvents for purification purposes. It could also be used for inorganic applications such as distilling sodium, something that would be impossible in normal glassware due to the high temperature working conditions.
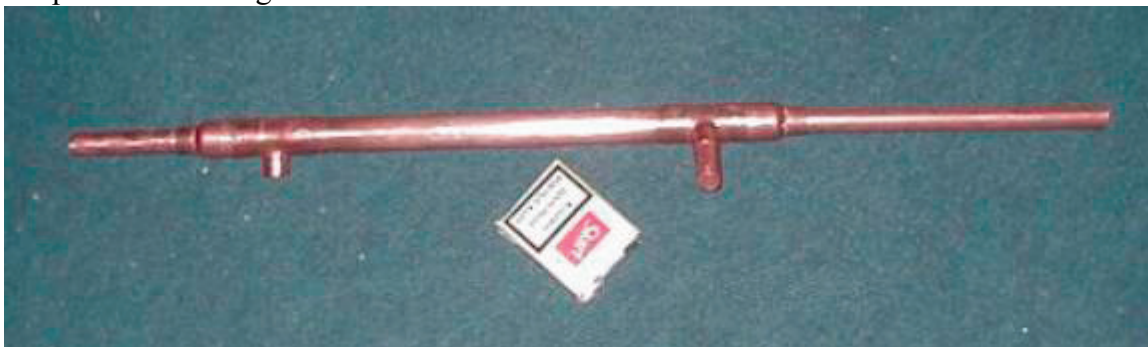
Such an apparatus can be simply put together with epoxy and then connected to glassware through rubber stoppers and tubing. Most chemists will limit themselves to the normal glassware distillation apparatus, but metals are more cost effective and much more durable. Copper is a great choice, since the metal is pliable with proper tools and it is fairly inert. However distillation apparatus can be made from other metals as well.
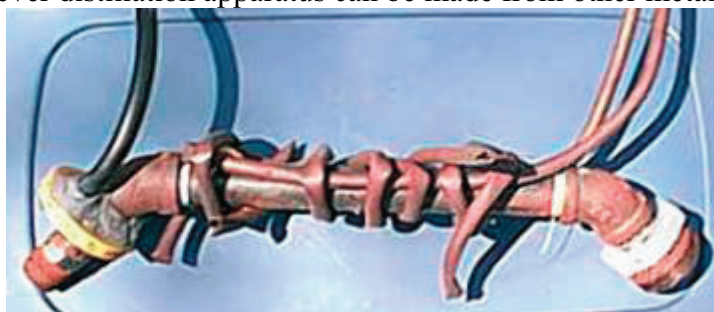
This crude apparatus above is a condenser fabricated from steel threaded pipes. The connection on the right leads from the distillation vessel and connects to the side of it. The left side actually connects to a jar with the lid attached there to distil into it. And the copper pipe around the vessel cools it, as well as additional piping added to simply hold it in place. Crude, but effective, there are many other ways to prepare distillation apparatuses from metals and over the counter items if one is willing to take the time to make one.

Here is a quick checklist to follow before performing a distillation:

1. Check if your compounds form an azeotrope. If not, or if your starting material is below or above the azeotropic concentration, proceed with a simple distillation first. If your compound does form an azeotrope, proceed to step five.

2. Check the difference in boiling points.

3. Check the vapour pressure of the higher boiling component at the boiling point of the lower boiling compound. If this is significant and purity is required, use a fractionating column.

4. If you can't achieve sufficient separation with a column or you don't have a sufficient column at your disposal, check whether you can use an azeotrope to remove ONE of the components.

5. If your compounds form an azeotrope, which you want to separate, check if there's something that'll form a stronger azeotrope with it or which can break the azeotrope.

6. Last but not least, check if your components won't decompose at the necessary temperatures. If yes, carefully read section **8.6** under the advanced techniques on how to work with and distil under vacuum.

Note from the Authors: A Dangerous Experiment

Distilling HNO_3 (Nitric Acid) from a mixture of a nitrate salt and sulfuric acid is a time tested way to isolate this useful oxidizing acid. So a naïve chemist combined an unweighed quantity of $NaNO_3$ (sodium nitrate) into a flask with a large quantity of 94% H_2SO_4 (sulfuric acid) and attached a condenser though which water was run and in turn this ran to a receiving flask. Some boiling stones were also added (pieces of obsidian) to help ease the boiling process and make it smoother. However complications where run into shortly after heat was applied, the black boiling stones started to color the mixture black, and the gas running into the condenser was a dark red, not what would be expected for a clear to off yellow acid. Regardless distillation was continued and in the end the chemist ended up with 20 ml of a dark red volatile distillate. Upon addition to water it decolorized and left an acid solution. Although not obviously apparent to the chemist at the time they had distilled NO_2, a highly toxic gas that is one of the decomposition products of nitric acid. Their ambitiousness and

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inexperience resulted in them heating the reaction mixture too high and the concentration of their acid only would have allowed 95%+ HNO_3 to make it over, which would have called for a vacuum because high concentration HNO_3 decomposes somewhat more readily then the more common 70% grade. The chemist eventually realized their mistake by simply observing the physical properties of the product. Luckily the chemist made it out unscathed.

3.2d Safety Concepts

Heating:

When distilling, one should use a safe method of heating; this prevents your glass from cracking and improves its lifetime. It also protects you from a bursting apparatus, which can shower you in dangerous chemicals. Open flames are **NOT** a safe way of heating. Flames cause localized superheating and are especially a hazard when distilling flammable substances. Flames that are non-diffuse, such as flames from a torch, can cause extreme stress and failure of glassware. Either use flames to heat a bath for your reaction vessel or use a diffused flame.

e of the authors, approves of using a flame for this application, I do
y distillation. The authors of this book will not be held responsible
flame.

You do so at your own risk

Electric Heating:

Electric heating mantles or hotplates can be used, with a couple exceptions. However, one should be careful with flammable substances. If the temperature of the hotplate surface is higher than the auto ignition point of your substance, it cannot be used unless the whole apparatus is sealed to the entry of atmospheric oxygen and the output gasses are property taken care of. Same goes for electric heating mantles. Heating mantles should be used for flasks because a hotplate causes localized overheating with its flat surface! Note that heating mantles usually impair magnetic stirring, unless you buy a magnetic stirrer which fits the heating mantle, but these are usually within the $500 range.

Baths:

Baths are ideal for heating flammable substances, certainly water baths. Water has the advantage of being not flammable and its high heat capacity can be beneficial. However, water evaporates rapidly when used above 80C. Oil baths don't evaporate as quickly as water, but they have other disadvantages. Most commonly available oils smell and are flammable. They also pose a severe explosion hazard when distilling oxidizing material. Ideally, silicone baths or other non-flammable synthetic oils should be used, but these are usually hard to get and are expensive. For lower temperature applications or if you just don't care about your hot plate, a sand bath can be used. At higher temperatures the insulating effect of the sand can burn out the heating element in a hot plate, so be warned, sand baths are a risky.

Sealing Joints:

Sealing your joints is very important. Improperly sealed joints will not only cause losses, but can also pose a safety hazard. This is most dangerous when distilling flammable substances or when distilling under vacuum. The most common seal is with grease. There are several types of grease and they each have their advantages and disadvantages. For example, Vaseline is cheap and easy to use, but its chemical resistance is limited and it will contaminate organic solvents. Other options include silicon oil, but this is expensive and does not provide 100% chemical resistance either, and specialty high vacuum greases composed of fluorinated hydrocarbons, but they can cost a small fortune, typically reserved for high end Chemistry labs. A good seal can be obtained from cheap, white, Teflon tape available from any hardware store to seal pipe joints. This easily fits between the joints and has excellent chemical resistance. Wrap one layer around the ground glass male side and press into the opening opposite, giving a slight twist. Do not force or twist too much as you run the risk of cracking your glassware.

3.2e Additional Use for Distillations

Evaporating to Dryness

This is a common procedure for the inorganic chemist, less so for the organic chemist, but none the less important. Evaporating to dryness is a feasible method of recovery of a pure product providing:

1. Your intended product will not decompose at the temperatures necessary to volatize the solvent. A vacuum can decrease the necessary temperature to remove the solvent and prevent decomposition of your product.

2. The other compounds in your solution, besides your intended product, are also volatile.

3. Your intended product will not volatize to any major degree at the temperatures used.

4. Your intended product will not explode at the temperatures used. This is known as being *pyrophoric*, which is not a good thing for the amateur.

5. An extremely pure product is not required and additional purification will take place.

Examples of simple viable procedures:

- Making $AgNO_3$ by dissolving silver in HNO_3 and boiling off the HNO_3 and Water.
- Neutralizing $BaCO_3$ with HCl then boiling off the water and HCl.

Examples of procedures that will not work are:

- Boiling off the water from commercial bleach (NaOCl decomposes, NaCl/NaOH impurities)
- Dissolving Na in MeOH then boiling off the alcohol (NaOMe decomposes)
- Dissolving Al in HCl then boiling off the HCl solution ($AlCl_3 * xH_2O$ decomposes to oxychlorides)

Additional care needs to be taken when a solution has nearly all evaporated if there is a heavy precipitate on the bottom. This can cause 'bumping' which can bounce a flask off a hot plate or even crack it due to the pressure of the vapors rising though the precipitate. To avoid the worst of this you can cool the solution when a precipitate starts to form, filter it, and then resume heating. Another method would be to use magnetic stirring or heat at a lower temperature then the boiling point of the liquid. This will cause a nice and slow evaporation.

A Note from the Authors

It should be noted that if you are going to use the above technique for obtaining a salt by reacting an acid with a metal carbonate or hydroxide, be sure to use the *stoichiometric* quantity. Any excess acid will have to be boiled off, resulting in noxious clouds that kill grass, plants, eyes, and lungs. This is, of course, assuming the acid is volatile. H_2SO_4 (Sulphuric acid) will not be as easily volatile as HCl (hydrochloric acid).

3.3 Filtering

Filtration is the separation of a mixture of liquid and solid. It is simple, usually quite fast, and rather effective. The only items you really need are a funnel and a piece of filter paper, which can range from a simple coffee filter, a wad of cotton, or even high end analytical paper. There are several types of filtration; each is suited to its own particular application.

There are three common types of funnels that are useful to chemists for filtration: liquid, powder, Buchner. A liquid funnel has a long, narrow spout and is usually best for simple filtrations, using a cotton wad or a properly folded piece of filter paper or coffee filter. However because the spout and subsequent area that drains into the spout is so small it is only good for filtering a tiny amount of precipitate otherwise it may become too packed to filter, even with vacuum filtration. Powder funnels have a much wider spout and are useful for filtering things that might clog a smaller spout funnel. Buchner funnels are the best option for isolating solids, but they work best with a vacuum (see below for *vacuum filtration*).

The main goal of filtration is to separate a liquid from a solid. Now this can be done simply by *decanting* a mixture. But, there are some inherent problems associated with decanting. If the solid material left at the bottom of your flask is your desired product, decanting will leave you with an appreciable amount of reaction mixture. In addition, some of your product will inherently be lost during the actual decanting process. If the liquid is your product, a considerable amount of liquid will be trapped by your solid, which could easily be obtained by filtration. Also, small particulates of the solid may come over during the decanting, ruining the purity of your new product, since the solid may not completely settle out. It is for these reasons that filtration is almost always preferable to decanting. Before selecting a filtering method, it is important to consider the following questions:

1. What quantity of liquid will I have to filter?

    This is important since gravity filtration is not a good method to filter large quantities of liquid, since the time involved is typically quite long and there is an inherent possibility of clogging the filter paper. In this case, vacuum filtration is almost always preferred.

2. Am I trying to recover the liquid component of the reaction mixture?

    If you are trying to recover your liquid component and not your solid component, sand or diatomaceous earth (silica) could be put onto the filter paper to increase the efficiency of the filtration and speed it up. It should be noted that using diatomaceous earth should be used with a filter, since the particle size of the silica can be quite fine.

3. Am I trying to recover my solid product?

   If the solid is your goal of filtration, then special care should be taken to make sure your solid is recoverable in a high quantity. This can be accomplished in a number of different ways; these will be discussed in detail in the next section.

4. Do I think the crystals, both in volume and by their size, might clog the filter paper?

   The possibility of crystals clogging filter paper is exceptionally high in the case of fine precipitates. This can cause a complete stop to filtration. Large quantities of even larger crystals can also clog a filter or cause an overflow, ruining product yield.

5. Do I have fine particulates that might pass though filter paper?

   If there are ultra-fine particulates in the filtrate, it is possible that they could pass right through the filter paper. One solution is using high-end, very fine filters that could be used to the solid. Another solution is to try letting the solution rest for a few days. This can 'age' the precipitate resulting in a more manageable solution as the solid packs together. This can allow filtering though sand, diatomaceous earth, and fine sintered glass filters to remove many fine particles. Although quite obvious, if the fine solid is your product, separating it from the diatomaceous earth or sand could prove quite difficult.

6. Do I have to filter the solution while it is hot?

   If you have to filter a hot solution, the first addition of the solution to the filter paper can result in crystallization. This almost always leads to clogged filtration, which can be catastrophic in some situations where a very hot solution is stuck precariously in a funnel. Pre-washing the filter paper with hot solvent or heating the glass or porcelain parts of the apparatus in an oven is a viable solution.

7. Does my reaction mixture contain components that may wreck filter paper?

   Most of the time, solutions will not be greatly affected by filter paper, but some copper complexes can actually react with cellulose. In addition, strong sodium hydroxide solutions or strong oxidizing solutions, such as concentrated nitric acid, will absolutely demolish filters. This can spell doom for the reaction and, at the bare minimum, cause you to attempt filtering again. For oxidizing agents, try filtering with glass wool, as this is substantially less reactive.[1]

   The normal procedure to begin filtration, causing the least amount of problems, is to slowly decant the liquid portion into the filter paper. This should be done without adding the solid mass at the bottom. In this way, the initial filtration can proceed unhindered. As soon as the solid particles are added, the filtration itself will slow greatly. Be sure to add the initial liquid mixture in small increments. If you are taking advantage of the insolubility of a salt, yields may be increased by cooling the reaction mixture until it is near the freezing point to depress the solubility. This will cause more product to "crash" out of solution. After each addition, you should wait until it is almost entirely though the filter paper, then add more, keeping up the process till almost all of the liquid has been used. As soon as only a small amount of liquid is left in your reaction vessel, swirl around the mixture to free up any clumped solid. Then, in one smooth motion, dump it in all at once to get your solid into the filter paper. You should then rinse the vessel out with more solvent. If this is not possible, usually due to the product being

slightly soluble in the solvent, scrapping out the rest of the product is the tried and true method. If possible, the product left in the filter paper should be washed with cold solvent. This will effectively rinse a good deal of soluble contaminants out of the solid mass. Due to the theory of extraction, which will be discussed in **Chapter 8**, several small additions of solvent to rinse is the most efficient technique and greatly increases purity. After the solid is thoroughly washed, it can be removed from the filtration apparatus and spread out on filter paper or on a watch glass to dry.

The next few sections of filtering will explore the different types of filter paper and discuss the different types of filtration that will aide you in your success as a chemist. This is by far the easiest technique to master and should be learned by everyone entering the field.

(1)   In the case of filtering oxidizing solutions, you can give glass wool a try. Alternatively, fiberglass insulation can also be used. However, this should first be cleaned by stirring with hydrochloric acid and drying, if your product still looks bad, you may want to try a different brand of fiberglass.

3.3a Selection of filter paper

When it comes to filtration, size does matter. The filter paper you use **must** fit your funnel or you will encounter many problems. The filter paper should fit entirely within the sides of the funnel. In the case of coffee filters, this prevents the "creep" of solvent over the edges of the funnel. For Buchner funnels, the filter paper should cover all of the holes on the flat portion, but the edges of the filter paper should not touch the vertical sides of the funnel, otherwise it won't seal under vacuum. If you don't want to invest in several different sizes of filter paper, remember that a big piece can become a small piece, but not vice versa.

The type of filter paper you use really depends on the size of the solid material you are filtering. For most applications, coffee filters are sufficient, but coffee filters are relatively thin and can't take much abuse. If you can obtain real filter paper, do it (The filters from Whatman are the best, and not very expensive). For larger crystals a wad of cotton packed into a liquid funnel works very well. If you have a Buchner funnel, you need a flat piece of filter paper so coffee filters are no good unless you cut them up to fit your Buchner. For filtration of very fine particles, like charcoal dust, you can buy high efficiency filter paper, but the cost is rather steep. If you really need it, the best stuff is made from Teflon and can be found through chemical and chromatography suppliers. A slightly cheaper alternative is nylon filter paper, but this is still quite expensive and considered a special commodity. Using Teflon and nylon filter paper requires vacuum filtration, which is discussed below. It is possible to circumvent the need for Teflon filters by using a "filter aid", such as diatomaceous earth. It is usually sold by the trade name "Celite." Celite is essentially very, very fine dirt, since it is powdered silica. It should be noted that the diatomaceous earth sold as pest control actually contains some added pesticides and, therefore, should not be used for any Chemistry applications. As a final note, all filter paper, no matter how expensive, is really only good for one use. It's not worth ruining an experiment to save a few cents by reusing a piece of filter paper.

3.3b Gravity Filtration

Gravity filtration is the process of adding a mixture to a filter and allowing gravity to pull the liquid through. Gravity filtration is substantially slower than vacuum filtration, but requires less equipment and is generally more effective. It is better for isolating the liquid phase than the solid phase, but if you don't have a vacuum, it will still work reasonably well for isolating solids.

To perform a gravity filtration, you need a funnel (liquid funnel is best, but a powder funnel is okay, so long as you use filter paper and not cotton), filter paper, and a flask to collect the liquid. There are many ways to fold a flat piece of filter paper in to a cone shape. The best way is called "fluting" and consists of folds in opposite directions along the diameter of the filter paper (FIGURE). A simpler, although somewhat less effective method, is to fold the circle in half and then fold the semi-circle in half again. This wedge can be opened to form a cone, such that half of the cone has a one-ply layer of paper and the other half has a 3-ply layer. Figure. Fluting the filter paper will give a significantly faster flow rate. After folding the filter paper, place it inside the funnel and clamp your receiving flask in place (this is very important!) with the funnel set on top. Follow the basic procedure given in the first section of filtration and you will have performed a flawless separation.

Gravity filtration is relatively slow, so just add enough of your mixture to get close to the top and then let all of the liquid flow through before adding more. This may seem unnecessarily slow, but if you slip and add too much of the mixture and some gets through unfiltered, you will have to start over again. Once you've poured in all of your mixture, rinse your reaction vessel with the solvent and then rinse the solids thoroughly.

Gravity filtration cannot be used reliably for solids that precipitate as 'gels' such as iron or aluminum hydroxide, these are very difficult to remove water from and easily clog filter papers if not assisted by vacuum. Also note that filtering organic liquids of low density through filter paper can prove difficult without vacuum as their low density gives little incentive to pull them through the filter paper. Plus, filter paper contains water since they are hydrophilic and can 'intimidate' organic liquids from readily passing through them.

3.3c Vacuum Filtration

Filtration using a hand vacuum pump and a Buchner Funnel in a Erlenmeyer Flask with Side arm.

Vacuum filtration is the fastest way to filter a mixture, but can be finicky. This is the most efficient method for isolating solids, since the liquid is pulled almost entirely through the filter paper. To run a vacuum filtration you will need a side arm Erlenmeyer flask, a suitable

funnel (Buchner and Hirsch work best), a vacuum, and a rubber sleeve that fits as an adapter between the funnel and the flask. To begin, put the rubber sleeve around the spout of the Buchner funnel. The sleeve should fit snuggly and be larger than the opening at the top of the Erlenmeyer to ensure an airtight fit. Be sure to clamp your Erlenmeyer in place, sometimes the vacuum can cause the tubing to whip the flask, not a good thing for your yield or flask! Then set the funnel on top of the Erlenmeyer. Next, position the filter paper such that it covers all of the holes in the funnel. Wet the filter paper with a little bit of the solvent from your reaction as this will help the vacuum seal. Then, connect the hose from your vacuum to the side arm on the Erlenmeyer. Finally, turn on the vacuum and slowly pour the filtrate onto the filter paper. The solvent should be sucked through quickly, if it isn't, try pressing down on the funnel since the rubber may not be sealing. Try to keep the filter paper moist during the filtration, since it will not seal when it is dry and the solid can be sucked through around the edges of the paper. Once all your mixture has been poured into the filter, rinse your reaction vessel with solvent a couple of times, scrapping any sticky solids as needed. Then rinse the solid in the filter paper a few times. Remember, if the solid is slightly soluble in the solvent, be sure to use only small amounts of ice cold solvent. Pull air through the solid for a few minutes to dry the solid and then turn off the vacuum. Vacuum filtration provides a simple and effective separation in very little time.

For filtration with Celite (diatomaceous earth), set up the filtration apparatus as described above and then add an even layer of Celite, 2-3 cm thick over the top of the filter paper. Turn on the vacuum for a few seconds. The layer of Celite should compress a little. Wet the Celite with solvent and then turn the vacuum on again briefly. Repeat this process, adding little solvent slowly packing it down with the vacuum. This process is essential to separating very fine dust from your mixture. When you are satisfied with the packing of the Celite, turn on the vacuum and slowly pour your mixture onto the filter. Try not to disturb the Celite too much, you want it to stay as even as possible. Next rinse the reaction vessel and the Celite. Rinse the Celite multiple times to ensure that all of the product has been pulled through. There is much more to rinse than in a regular filtration.

An important note, if you are using Celite as a filter aid, it is almost impossible to recover your solid material, you have been warned!

3.4 Titration
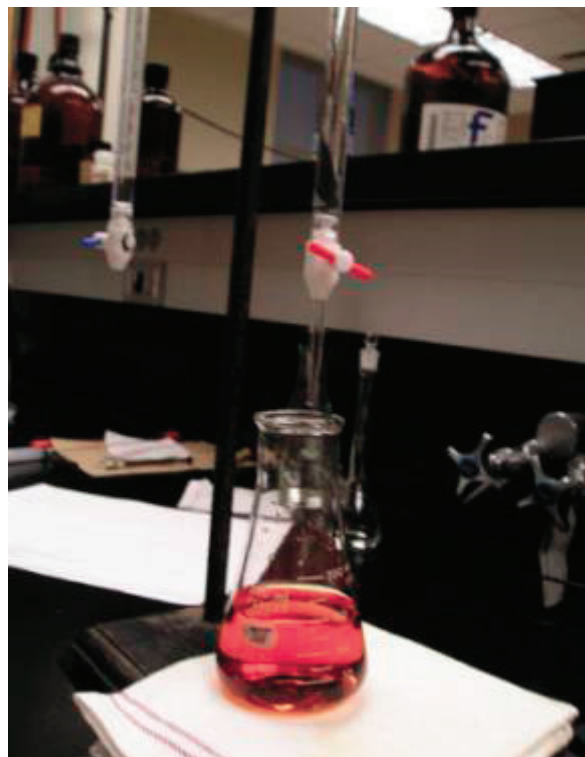
A titration is perhaps the cheapest and most useful quantitative test that can be done in home chemistry. Titrations are useful in a variety of circumstances for analyzing a plethora of products. There are many different types of titrations ranging from acid-base, to redox, to iodometric, and many more. It is important to select the right method to get the fastest and most accurate results. It is most useful when you want to know the concentration and purity of a given reagent, for instance, hydrochloric acid. However, before you could do this, you still need to know the basics and have the necessary materials.

Materials:
To do an accurate titration, you will need:

A glass buret (50mL with 0.1mL graduations and PTFE stopcock is recommended)

A small glass funnel to facilitate filling the buret
A standardized titrant
An indicator suitable for the pH range
An accurate balance for weighing dry materials
Buret Clamp
Ring stand

A stirrer plate with PTFE magnetic stir bar can also be used in titration

3.4a The Terminology

It is important to understand the jargon associated with doing a titration so that this section is easier to follow:

Concentration is usually expressed with molarity. Molarity (M) is moles per liter of solution.

Equivalence Point—The point at which stoichiometrically equivalent amounts of reactants have reacted. This can be shown as the general equation of acid reacting entirely with base. In this case, one part acid reacts with one part base and, at the equivalence point, are completely reacted:

$$H^+ + OH^- \text{-----}> H_2O$$
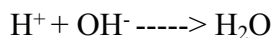
End Point—The point at which an indicator changes its color or the reaction causes a color change on its own. This signals the completion of the titration, but is not necessarily the same as the equivalence point. The final volume is recorded at this point.

S*tarting Point*—The point at which the titration begins. This is where the initial volume is recorded.

Midpoint (Half Titration)—The point at which one of the reactants is half-reacted. In some cases, as with a weak acid or base, the pH at this point correlates to the pK_a or pK_b.

Titrant—The titrating reagent which is typically of a known concentration and in the buret.

Standardization—The process of making a solution of known concentration.

3.4b Titration Overview

In case you are completely new to the concept of a titration, here is a complete overview of the process.

Let's say that you have bought some battery acid from your local auto supply store and you haven't a clue its concentration. Reading the MSDS, you realize the ingredients are sulfuric acid and water, no additional reagents. The most effective way to find the concentration is with an acid/base titration. You would take your acid of unknown concentration and react it with a base of known concentration until the acid has been completely reacted. There are two key points to consider in any titration: your standard and indicator.

The standard solution

The base or acid that you are reacting against the unknown must be of a known concentration. This solution is an absolute necessity, since the concentration calculations will be based off of it. A standard solution must either be purchased from a chemical supplier or prepared. It is not recommended to use over the counter (OTC) liquids as standards as they can be quite different in concentration from their labels. For example, the amount of ammonia in OTC ammonium hydroxide can vary by several percent; it can evaporate, there was a slight variation with the production batch, or something may have contaminated it. OTC chemicals are meant for cleaning, not for acid-base titrations. Therefore, the most effective reagent to start with is usually a solid that will dissolve easily in water and yield a solution with a known concentration. The solid should be something non-hydroscopic or at least have known water content (i.e. known water of hydration). Even better, it should be dried in an oven shortly before preparing the solution, especially if you are unsure how much water it may contain. The most effective temperature range for drying a small amount of solid is around 100-150°C for two hours, assuming the compound will not decompose at that temperature. It is important to spread out the solid in an evaporating dish while it is drying since pockets of solid may trap water.

It is important to use a titrant that is classified as strong. Weak acids and bases provide an added complexity to titrations and are thus not recommended as a standard. Keep this in mind as you are choosing a standard for the titration.

Now, to prepare your standard you are going to need a scale and a graduated cylinder, volumetric flask, or another container that can measure liquids fairly accurately. It is important to never use beakers for this, since these have highly inaccurate graduations. If your scale does not measure accurately, for example, no decimal places, you might want to make a large amount of the solution since this will minimize the relative errors. If you are desperate, you can find the solubility of a substance at room temperature and saturate a solution with it at 25°C, then filter off excess solid. This is a simple trick for standardization without a scale, however it may deposit solid on cooling and therefore can induce error. A standard prepared by this method should be used immediately to standardize another, more stable solution.

Properly standardizing a titrant is perhaps the most important step in every titration. No matter how precise your hands are when actually performing the titration, an inaccurate standard will always provide an inaccurate titration. Do not overlook this integral procedure in Chemistry.

Sample Standard Preparation of a Sodium Carbonate Solution

So, you need a standardized sodium carbonate solution for your titration. Say that you weigh out 25.0 grams of anhydrous sodium carbonate and you dissolve this in a 250 mL volumetric flask.

25.0 g Na_2CO_3 / 106 g/mol = .236 mol

> Then, from moles and the volume of solution, you can calculate the molarity, mol/L.
>
> .236 mols Na_2CO_3 / .250L = 0.943 M Na_2CO_3
>
> Now you have a standardized sodium carbonate solution which you can then titrate with.

Indicators

The best method for determining an endpoint is to add a chemical indicator to the flask containing your unknown. There are many acid-base indicators, but for going from an acidic to a basic solution, the key indicator is phenolphthalein, which must be purchased from a chemical supplier. In the presence of acid or in neutral conditions, solutions containing this organic molecule are clear, but around a pH of 8 the solution sharply turns pink. Be aware that only a small amount of indicator in dilute solution are needed to observe the color change.

There are also naturally occurring indicators; a number of natural extracts will change color depending on the pH of the solution and could therefore potentially be useful for home preparations of acid-base indicators. The most famous of all of these natural indicators is the Red Cabbage Indicator[1], its preparation is easily found online.

When searching for the perfect indicator, it is important to have a rough estimate as to where the equivalence point lies. Performing a few calculations, or even just looking at the pKa of a weak acid or base, can provide hints as to which indicator should be used. In the appendix of this book, a chart detailing the common indicators and their color ranges has been included.

(1) The Red Cabbage Indicator has a wide range or colors that it changes between from a pH of 0 – 14, making it useful for quite a few operations, but it is sensitive to oxidizing conditions as well as keeping it at high or low pH, also solutions of it tend to break down over time but the reagents for its preparation are cheap and available, one site detailing the preparation of the indicator along with it color ranges can be found at http://chemistry.about.com/library/weekly/aa012803a.htm as can more information on acid-base chemistry.

3.4c Performing the Titration

The integral piece of glassware used in titrations is called a buret. It is a tall, thin glass tube with a stopcock on the bottom. The side of the buret has highly accurate graduations. It is filled to a specific level with the titrant and a flask containing a precise volume of your unknown solution, with a small amount of indicator, is placed just below it. The stopcock is turned and the flask is slowly stirred by hand or a stir plate. Once the color starts to linger for longer periods of time, the flow of the titrating liquid should be lessened to short bursts and even drops. As soon as the solution's color changes entirely, the endpoint has been reached. Read the volume on the buret and record this for your calculations. Knowing the initial and final volume in the buret, you can determine the amount of standard it took to react with your unknown.

> **Continued Example: Before titrating, try the math in this example!**
>
> Using the .943 M Na_2CO_3 solution mentioned above, let's assume you want to perform a titration with it. The initial volume of the .943 M Na_2CO_3 in your buret was 0.00 (Burets start at 0 and increase as they dispense liquid) and the initial volume of sulfuric acid in a beaker beneath it is 20 ml. On your first attempt 43.10 ml of Na_2CO_3 was needed to turn the indicator pink. On your second attempt 41.2 ml was needed to do the same, and on your third attempt 41.27 ml was needed. First, what is the reaction?

$$Na_2CO_{3(aq)} + H_2SO_{4(aq)} \Rightarrow Na_2SO_{4(aq)} + H_2O_{(l)} + CO_{2(g)}$$
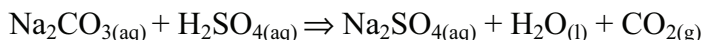
Although the process is complicated by H_2SO_4 being diprotic, having two acidic protons, the stoichiometry is simply 1:1. Therefore, the number of moles Na_2CO_3 used equals the number of moles H_2SO_4 in solution. Continuing with the calculation:

(Molarity of titrating solution) x (Volume of titrating solution in ml) x 1L/1000mL = mols titrating solution

It should be noted that sodium carbonate is a weak base, so then why should it be used as a titrant? Well, this is a great choice for the at home chemist for a few reasons:

1. It is readily available from the decomposition of sodium bicarbonate (baking soda).
2. It is usually a somewhat pure reagent, especially if heated first to drive off excess moisture and decompose remaining bicarbonate.
3. It is easily weighed and handled.

But there is one aspect sodium carbonate fails in: since sodium bicarbonate is not a strong base and therefore cannot react completely with an acid. Sodium carbonate decomposes to carbon dioxide when it reacts with an acid. This drives the reaction foreword. In order to ensure that every last bit of CO2 has been driven out of solution, sodium carbonate should only be used when titrating strong acids. It also helps to heat the solution being tested to drive off dissolved carbon dioxide which can cause a slightly acidic solution.

Useful things to remember that can lead to more accurate results:

Keep your buret clean—Always rinse twice with distilled water and then three times with your titrant before running a titration. This ensures little to no contamination ruins the accuracy of your experiment.

Place a white piece of paper underneath the flask—Titrations are able to give highly accurate results. To obtain such accuracy, one must look for the first indication that the reaction is finished. This is usually when an indicator changes colors as the pH changes its propertes. For instance, phenolphthalein is deep pink when in a highly basic solution. Therefore, it is imperative to stop when the slightest pink color is observed, any darker means that the end point was "overshot" and the results will be quite poor. Using a white piece of paper underneath the reaction greatly aids in distinguishing the contrast of colors.

Fill the tip—Before you take your initial volume reading, fill the tip of the buret with the titrant solution by opening the stopcock. It is also important to allow any large bubbles caught near the stopcock to flow through at this time. They can, in fact, drastically influence your results.

Repetition—The first titration is usually performed as an indication of what volume is expected to be used. Regardless, proceed with caution to ensure you obtain a reliable estimate. If you have followed all of the above pointers, the second, third, and fourth titrations should be within +/- 0.1 ml. More titrations are integral for a more accurately determined concentration. It is standard practice to exclude the first titration and take the mean of the others as your concentration; calculating the standard deviation is also useful in evaluating your performance.

Pick the right reaction—It is important to select the right type of titration for the job. Determining the concentration of a $KMnO_4$ solution requires a redox reaction and is different from determining the concentration of HBr in an acid-base reaction. Even determining the concentration of halide ions (I.e. an iodometric titration with a starch indicator.) can be performed with the right reaction.

3.5 Temperature Control and Measuring

Temperature control varies in its importance from reaction to reaction. Two extreme examples would be a nitration reaction where you are controlling the temperature of the reaction between 10ºC to 30ºC and the total opposite might be trying to make phosphorus where the temperature might be 1200ºC and you are trying for as high a temperature as possible. Both of these reactions pose their own difficulties for both measuring the temperature and controlling it.

Basic reactions are easily controlled with water. Water has a high specific heat, meaning that it can absorb a lot of heat before rising in temperature, or conversely, it can hold enough heat to warm another mass significantly. Water is a safe and effective way to heat and cool reaction mixtures. There are, however, other mixtures that can achieve even more extreme temperatures. KOH or HNO_3 mixed with ice can achieve extremely low temperatures. As can a dry ice/acetone slurry. Even cooler mixtures can be obtained with hydrocarbon baths with liquid nitrogen added periodically. Liquid nitrogen can be obtained from welding suppliers. Obtaining temperatures any lower than this is hard to accomplish in a home lab. It should be noted that the most common super-coolant is liquid helium, but I doubt you'll find this in any local super market.

A Note from the Authors: The Cooling Effect of Winter

Winter is a good time to perform those exothermic reactions that would usually require external cooling or precooling of the reagents. Simply place them outside before and during the reaction. Many chemicals are also less volatile under these conditions; bromine is friendlier the colder you get. The preparation of chloroform from acetone and sodium hypochlorite is aided by allowing the reagents to come to temperature outside then mixing and cooling in a snow bank; it's cheap and it's effective.

Snow is a great insulator as well. This is a bad thing since air pockets may form as the snow melts preventing your reaction from cooling. For this reason snow is best used as a slush around the reaction vessel with occasional stirring. In this way, heat is conducted further away faster and there is no insulating effect.

Heating is a much different process than cooling. Most heating can easily be accomplished with electric heating, like a hot plate, or chemical heating with combustion. Commonly, electric heating devices can get to 250°C. Lab grade hot plates can obtain even higher temperatures. High temperatures can also be achieved with suitable furnaces, but most amateurs do not have access to these. Therefore, the next best thing is combustion. There are many different types of torches and burners: Meker burners, Bunsen burners, propane torches, and butane torches, but each has their own limitations. Common butane torches can reach 700-800°C. Methane is also a good carrier of potential energy, so hooking directly into your home gas line has its advantages. To be clear, improper rerouting of your gas line is dangerous; *the authors of this book are not responsible for your actions.* **You have been warned.**

Measuring these temperatures though pose their own difficulties. Household thermometers are fine for common temperatures, since -30-300°C can be easily found on many candy and espresso thermometers. Beyond this it becomes necessary to obtain more sophisticated equipment. Bimetal thermometers, which hook into electrician tools, can reach 1000°C, but you will have to invest in a good probe and the thermometer itself is quite sensitive to chemical attack. There are three different types of probes, the most common is the K type, these can cost quite a bit, and you need to also purchase the reader to plug it into. Not all K type probes are the same since they have different ranges. Always stay within the manufacturer's

temperature recommendations to prevent destroying your expensive probe. Another type of thermometer useful for high temperature is the infrared thermometer. Just point and click and you get a non-invasive temperature of up to 1000°C. These can be quite expensive for high quality ones or the lesser quality ones can be found on eBay for much less.

The cheapest and most easily accessible tool for high temperature measurement available to the amateur chemist is the melting point of other compounds. Molten metal baths can give an approximation of the temperature being used to keep the bath molten. Different substances can be found to give a wide variation of temperature baths, just be wary of decomposition problems at these temperatures. Simply these can be used to get an estimate of the lower end of the temperature present.

3.6 Removing water from gasses, solids, and liquids with drying agents

Referring to the section on dehydrating agents and desiccants, section 4.6, you can find a list of compounds that are good for removing water. Both dehydrating agents and desiccants (which are also known as drying agents) can be used to remove water from a system, however the action of dehydrating agents is strong enough that it can attack chemical bonds and this reactivity should be considered. The basic premise for drying chemicals is to find a compound that really likes water, more so then the chemical you want to dry, and add that compound to remove the water present in your product. The desired compound then needs to be separated from the desiccant. Some chemicals hold water very strongly and their dehydration proves to be very difficult, but generally procedures for removing water are simple. This section will focus on the basics of water removal from each state of matter.

Removing water from a solid:

The most common method to remove water from a solid is to heat it to drive off the water or to use heat in combination with a vacuum. However, some compounds are subject to decomposition when heated and drying agents are the only viable option. A desiccator is the simplest way to achieve dryness. Desiccators are air tight containers with a desiccant (drying agent) in the bottom with an area to place a dish or beaker. As the sample sits above the drying agent, it readily loses water to the environment. In a desiccator, almost no water is present, therefore any water lost to the environment is automatically absorbed by the desiccant. This process can occasionally take weeks, depending on the material, so patience is the key to a dry sample. Besides a desiccator, a method for drying organic compounds involves dissolving it in a tiny amount of hot organic solvent, in which water is insoluble, it will then separate. The organic layer can then be dried down to yield a relatively dry product. If at all possible, using non-aqueous environments and anhydrous reagents during the reaction can ensure water does not get into the reaction environment at all. This, however, is typically impossible for most reactions.

Removing water from a liquid:

Once a majority of water has been removed from a liquid usually through careful distillation or washing a fairly insoluble liquid with a saturated salt solution, drying agents can be used to remove the last small percentage. The usual procedure is to take the liquid and add a bit of insoluble drying agent to the liquid. It is then stirred and observed. If the drying agent at the bottom seems clumpy, more must be added. Only when the drying agent is free flowing is the liquid considered dry. In some exceptionally hydroscopic substances, this process can take quite some time; for example, the drying of ethanol with anhydrous copper sulfate can take days or weeks for example. The drying agent does not always need to be a solid, sulfuric acid can

be used to dry some solvents that do not react with it and are insoluble, such as bromine and acetonitrile.

Removing water from a gas:

Gas drying tubes are available for plugging on the end of distillation apparatuses. What they are is a short length of wide glass tube, followed by a bulge that tapers down to a thinner glass tube. The thinner part is usually attached to the apparatus via a small length of rubber tubing. A piece of cotton is crammed in the larger part and the bulbous part filled with a desiccant, such as anhydrous calcium sulfate, and another piece of cotton pressed on top of that. Gasses entering the apparatus are thus treated to remove some of the water. In gas generation removing water from the gas produced is sometimes referred to as scrubbing (see the section on gasses). Picture

3.7 Recrystallization

Recrystallization is a powerful and effective means of purifying a solid. The basic principle is to pick out a solid that you want purified, for example, ammonium nitrate. The next step is to find a suitable solvent for it. The solvent should possess the following properties:

1. Be able to dissolve a large quantity of the desired product when hot and have only a low solubility when cold.

2. Not affect the product by either causing decomposition or reacting with it.

3. Be useful under atmospheric conditions and does not possess properties that are affected by heating and cooling.

Finding such a solvent is usually difficult. Many books and sites do not list the solubility of a substance in anything but water. Therefore, some trial and error may be involved when you are getting started. It is also important to do some research to find similar substances and their recrystallization solvents.

Once your solvent is picked out, in our case, water can be used for the ammonium nitrate, the first step is to heat the solvent to a high temperature, almost boiling. Using this solvent and a pipet, saturate a solution with as much of the solvent that it takes to dissolve the solid. Depending on the purity of your substance, after complete dissolving and while still hot, a quick filtration can be used to remove any insoluble materials. Exercise caution since a hot, saturated solution can crystallize in cool filter paper rather quickly and can plug filter paper. It is for this reason that you will want to have something to scrape the filter paper handy and always pre-treat the filter paper with some extra hot solvent.

After your hot solution has been quickly filtered you must let it cool. Once a certain temperature has been reached, crystals will typically start to "crash" or "fall" out of solution. This can take a long amount of time, so be patient. Sometimes, super-saturated solutions, one that is not depositing crystals at all, will need a little kickstart. In this case one of two things can be done, a crystal of pure compound can be added, this is called a seed crystal, or you can scrape the side of your container. Either of these processes will allow for a crystal growing surface.

It is important to let the solution continue cooling on its own. Mechanically cooling the solution may cause the impurities to also crystallize out. This also ruins the natural crystalline shape to form, causing crystals to just look nasty.

If crystals still fail to form after an hour or two, it may be due to too much solvent present. The only things left to do is heat the solution to boiling and evaporate off some solvent, repeating the cooling step. If this too fails, then a new solvent should be tried, as this one is much too soluble for your solid.

After a good crop of crystals forms, the next step is to filter off the solvent and dry the crystals. Vacuum filtration is preferred since you may be filtering off a large quantity of solid. Sometimes gravity filtration works depending on the size of the crystals and the amount of solvent to filter. Regardless, the crystals should be washed, while still in the filter, with a few quantities of cold solvent to remove any adhering particulates. The crystals, depending on how sensitive they are to the environment, can be left sitting to dry. Some crystals may need more sensitive procedures, such as drying in a desiccator or under high vacuum; this will be decided by the research of your compound beforehand. Usually with salt products, they are hydrated and need to be heated to remove the attached water molecules. More hygroscopic products will need their own procedures to achieve dryness.
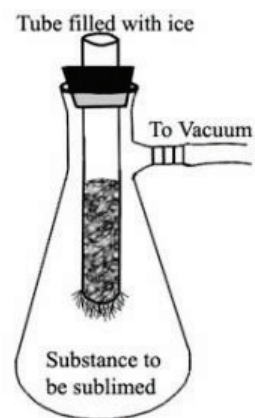
3.8 Sublimation

Sublimation is defined as the process by which a solid passes into the gas phase without passing through the normal intermediate liquid stage. A number of substances are known to sublime, common substances include naphthalene, paradichlorobenzene, and, the most famous of all, iodine. However many substances can and do sublime even if a liquid phase does exist. The main thing to consider is vapor pressure. All substances have a vapor pressure, even high melting solids. Normally, a substance has a higher vapor pressure the closer it is to the melting point and from there the closer it is to the boiling point. But many substances, especially organics, have vapor pressures that are appreciable at room temperature. If the vapor pressure of a substance can be increased, either by applying vacuum or by heating, then there is a better chance of subliming the product.

Sublimation can be considered a form of short path distillation and can be done providing two criteria are met:

Tube filled with ice

To Vacuum

Substance to be sublimed

1. The substance to be sublimed must have a high vapor pressure.
2. The substance from which the product is to be sublimed must have a relatively low vapor pressure.

Sublimation at room temperature is usually slow, but as stated above, applying a vacuum and heating increase the vapor pressure of substances and allows more of the molecules to escape in the gas phase. This then allows the molecules to condense on the cooler areas of a vessel. The picture shown here is just one way to setup a sublimation apparatus using a sidearm filter flask. The crystals will form on the cold test tube full of ice. When the sublimation is complete, the stopper is removed and the crystals are scraped off.

Sublimation is great for substances with high freezing points which could otherwise allow the substance to solidify in a distillation apparatus and possibly clog it during operation. It is also great for substances that might decompose at higher temperature or have prohibitively high boiling points (some substances with high boiling points can sublime at considerably lower temperatures). Usually not a procedure for the large scale, sublimation does offer yet another tool

with which a chemist can retrieve a product from a reaction mixture or a reagent from an over the counter product.