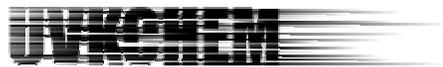


KINGS CHEMISTRY SURVIVAL GUIDE

**A guide for the hobbyist, enthusiast, or amateur for the preparation of common, and un-common
laboratory chemicals**

EDITION 1

By Jared B. Ledgard



WRITERS OF SCIENTIFIC AND TECHNOLOGY LITERATURE

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SECTION 1: Introduction to Chemistry

A quick lesson in chemistry

Part 1: Introduction to chemistry

This book has been written to teach the art of general chemistry sciences to the reader. To do this, you should take a quick, yet vital lesson in chemistry. First of all, the world of chemistry is a fascinating world filled with a huge variety of chemicals, chemical reactions, formulas, laboratory apparatus, and an arsenal of equipment. All these elements are combined and used thoroughly to bring about chemical change of matter from one form to the next. In this book, the form of change that we will deal with mostly, is the formation of compounds that are regarded as general laboratory reagents.

The world of general chemistry is absolutely huge, and in essence, deals with virtually ten's of thousands of chemical compounds. Regardless how many possible chemicals there might be, most see chemicals as something evil or something that is a troublesome or bothersome contaminant on our foods, households, and everyday possessions; however, in factuality, chemistry and the chemicals involved are responsible for our modern civilization, and without them, we would all be in big trouble. The art of chemistry is as old as life itself, and as old as our universe.

For most of you, the procedures in this book will not make sense at first, or will appear to be complicated; as a result, many of the procedures in this book may seem foreign, or unfamiliar—if this is the case, then at this exact moment, you are in the right place. By the time you have read this book, these “foreign” procedures will no longer be foreign to you, but in the meantime, lets get started on the world of chemistry.

The world of chemistry involves every single aspect, corner, and micro drop of everything that is matter. Our solar system and the entire universe all function on a chemical level—In essence, chemistry is everything. The universe and everything in it is composed of atoms and molecules, and within this massive space, there exists tens of millions of chemical compounds—either known or unknown. The compounds that are known make up only 5% of the naturally occurring compounds, leaving a massive 95% of them being synthetic (prepared in the lab)—95% of all chemicals are synthetic. Note: synthetic does not denote anything that is less superior to natural. Synthetic means creating natural in an un-natural way.

Chemistry has been divided into three fields over the last 100 years to better organize and format the system. The three major branches of chemistry include: Inorganic chemistry, Organic chemistry, and Biochemistry. In short, inorganic chemistry deals with ionic compounds, which make up the chemical compounds that do not contain active carbon. Organic chemistry is the largest branch of chemistry and it deals with covalent compounds, which make-up our everyday items like plastics, drugs, dyes, pesticides, insecticides, resins, fibers, and explosives. Organic means “carbon bearing” which means any compound that bears carbon is classified as organic. Gasoline, turpentine, and candle wax are specific examples of organic compounds. Last but not least, biochemistry studies the field of enzymes, organisms, plants, and animals and their active chemical processes. Genetics research studies the DNA and RNA of living things and is a sublevel of biochemistry. DNA and RNA is composed of organic compounds all linked and actively working together. Biochemistry deals heavily with peptides, amino acids, carbohydrates, ect., ect., all of which play a major role in natural process such as cells, metabolism, and the like.

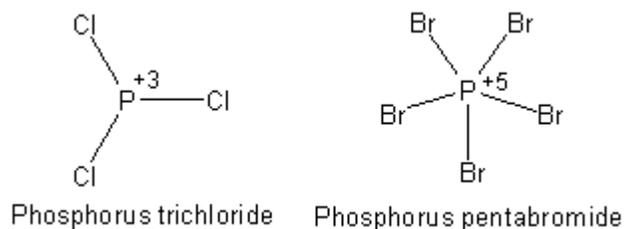
1. Chemical bonding: Oxidation states

First things first, you need to understand the nature of elements, and their oxidation states (number of bonds). Every single element is capable of forming chemical bonds with other elements (with the exception of a few “noble gases”). The oxidation states are what determines how many bonds a particular element can form, and to what other elements. When elements combine, they form chemical compounds. All of the atoms within a chemical compound show specific oxidation states. Oxidation states are not really states, but definitions of bonding, which are dictated by each individual element. Each element can form any where from either 0 to 7 bonds. These numbers represent the number of bonds the element can form (look at a modern periodic table, such that included in the “Merck Index”—the oxidations states are written in the upper left corner of each element). These numbers clearly indicate the number of bonds each element is capable of forming.

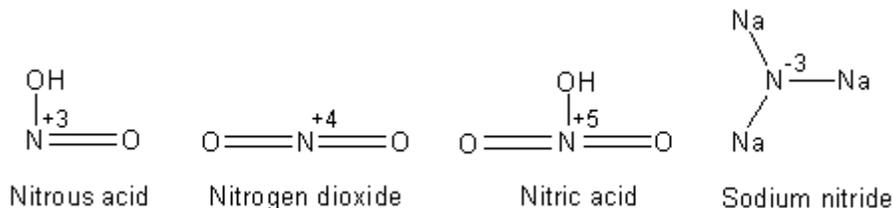
As most people are aware, periodic tables include rows and columns filled with elements. The elements within any given column have similar properties and characteristics along with similar oxidation states. For example, the elements of column 5A on the periodic table include nitrogen, phosphorus, arsenic, antimony, and bismuth. All these elements have similar oxidation states and properties. Phosphorus for example, can form compounds with three bonds or five bonds (indicated by the numbers +3, -3, and +5). Phosphorus, like arsenic and antimony have oxidation states of +3, -3, and +5. Phosphorus can form either +3 or +5 oxidation states when it bonds to elements with higher electro negativities (also listed on some periodic tables), and -3 oxidation states with elements that have lower electro negativities. Each element has different electronegative energies. Metals for example, have electro negativities ranging from

0.60 to 1.9. Non-metals have electro negativities ranging from 1.9 to 4.0. In essence, elements that are metals combine with the elements called non-metals forming positive oxidation states, with the so-called non-metals forming negative oxidation states. In a specific example, when phosphorus reacts with non-metals it forms +3 and +5 oxidation states because its electronegative energy is less than the other non-metals, but when it bonds to metals, its oxidation state is -3 because its own electro negative energy is greater than most metals.

Either way, when two elements combine for example, the element with the greater electronegative energy forms negative oxidation states, and the element with the lower electronegative energy forms positive oxidation states. In another example, chlorine and bromine both have greater electronegative energies, so when they combine with phosphorus, the phosphorus forms +3 and +5 oxidation states (see the illustration below). When elements combine they form compounds, which are called molecules.



Elements such as lithium, sodium, and potassium form only one bond, because they have only a +1 oxidation state, and because their electronegative energies are quite low (ranging from 1.0 to 0.6). A more complex array of oxidation states is demonstrated in the element nitrogen (a key element found in all amphetamines). It's capable of forming +1, +2, +3, +4, +5, -1 , -2 , and -3 oxidation states (see the illustration below). Another crucial element, carbon, is capable of forming +2, +4, and -4 oxidation states, and the all important oxygen, forms only a -2 oxidation state. Hydrogen can form +1 and -1 oxidation states. Remember the elements helium, neon, and argon (called the noble gases) form no oxidation states. Note: The oxidation states of each element (and column of elements on the periodic table) have been determined by trial and error over some 200 years of chemical research and study.



2. Ionic compounds and ionic bonds

Ionic compounds are composed of elements bonded together that have marked differences in electro negativities. Ionic compounds make up the bulk of "inorganic compounds", and are composed primarily of metals bonded to non-metals. In ionic compounds, the oxidation states of each element follows the same rules governed by the number of bonds each element can form. In the case of ionic compounds, the positive and negative numbers represented by the number of bonds each element can form, is more detailed and also represents a charge attributed to each element. For example, when phosphorus bonds to chlorine, it forms +3 or +5 oxidation states, and the chlorine forms a single -1 oxidation state; however in this example, because the electronegative difference between the phosphorus and the chlorine is not very significant, the resulting phosphorus trichloride or pentachloride is not considered fully to be ionic. However, in the case of sodium chloride, a +1 sodium ion is bonded to a -1 chlorine atom, with each positive and negative mark defined as a charge. Compounds that have their oxidation states defined as actual charges are considered to be ionic. As a reminder, remember that oxidation states (the numbers) define the number of bonds an element can form, never mind the positive or negative marks each number has. In ionic compounds the molecules are made up of positive and negatively charged atoms corresponding to their oxidation state number (the number of bonds each element can form, i.e., the oxidation state number defines the number of bonds each element can form, but not their electrical charge in all molecules—just in ionic molecules).

The electrical charge of each element within an ionic molecule is different than the element's electronegative energy. Note: Electronegative energy determines whether the element forms positive or negative oxidation states. Electrical charge is determined after the atoms combine, and is represented by the positive or negative oxidation state independently from the actual number of bonds each element can form.

As previously stated, chlorine is more electronegative than sodium, so when they combine the chlorine forms a -1 oxidation state (notice on a periodic table that chlorine has an oxidation state of $+1$, -1 , $+5$, and $+7$; and sodium has an oxidation state of $+1$). Some periodic tables give the electronegative energy of each element, and using such a periodic table, you will notice that the electro negativity of chlorine is remarkably higher than that of sodium. Because the difference between electronegative energies is so great,

the chlorine becomes negatively charged, and the sodium becomes positively charged. These charged atoms attract each other, and hence form a bond based on their electrical attractions (like two magnets)—this is the basis of “ionic” bonds.

Oxidation states also determine the number of electrons that can be captured. As previously discussed, ionic compounds like sodium chloride form their bonds based on electrical attractions. These attractions are determined by the number of electrons a particular atom captures. When chlorine combines (reacts) with sodium it forms a -1 oxidation state. Again, because the difference in electronegative energies is so great, the chlorine grabs or captures one of the sodium’s electrons. This capturing causes the chlorine to become negatively charged. As a result, the sodium atom becomes positively charged. Atoms become negatively charged when they capture electrons, and become positively charged when they lose electrons. This capturing and losing of electrons is the scientific foundation to ionic bonding and ionic compounds.

Currently there are about 200,000 ionic compounds known to man (most of them being synthetic). The most common ionic compound is table salt or sodium chloride. Some common examples of ionic compounds include potassium permanganate, sodium azide, sodium nitrate, potassium chloride, sodium fluoride, potassium chlorate, and zinc sulfate. Ionic compounds make up the majority of the earth, solar system, and the universe.

3. Covalent compounds and covalent bonds

Covalent compounds make up the bulk of chemical compounds known to man, but they only make up a small percentage of the chemical compounds found on earth and earth-like planets, and virtually most solar systems. As previously stated, there are about 200,000 ionic compounds known to man, with a potential of another 100,000 left undiscovered throughout the universe; however, covalent compounds number in the millions. For example, currently there are 16,000,000 covalent compounds known to man (as of 2003). The possible number of covalent compounds is practically endless, as the combination of these compounds is virtually infinite. Covalent compounds contain covalently bonded carbon atoms. The term “organic” means ‘carbon bearing covalent substance’. Covalent compounds all contain specific carbon atoms, which make up the foundation or infrastructure of all organic compounds. A covalent compound such as hexane for example, is composed of covalently bonded carbon atoms all bonded together to form a chain—this chain represents the backbone or infrastructure of the molecule. The carbon atoms that make up these backbones or infrastructures, are themselves bonded directly to other atoms such as hydrogen, oxygen, nitrogen, sulfur, phosphorus, arsenic, etc., etc. Such examples of covalent compounds (organic compounds) include: ethyl alcohol, isopropyl nitrate, aspirin, acetaminophen, cocaine, and octane.

Covalent bonds are much different than ionic bonds, as they share electrons rather than “capture” them. Remember that ionic bonds are formed when two or more elements with distinctive differences in electro negativities react with one another—whereby the greater electronegative element captures an electron (or more) from the less electronegative element(s). Covalent bonds, however, are formed when two or more elements combine and the electrons are shared (paired) rather than captured. In order for a covalent bond to form, the electronegative differences between the elements cannot be very significant, meaning their differences are much less than those encountered with ionic bonds.

Covalent bonds cover a whole echelon of reactions, many of which can be very complex and/or require special conditions depending on the chemicals and reaction conditions, and usually require multiple reactions and steps to achieve desired products. In other words, ionic compounds tend to be rather simplified compounds with easy formulas, whereas organic compounds can be huge molecules, which require many steps for their preparation. These multiple steps are the basis for organic chemistry, as it deals with a whole multitude of reactions and functional groups—most of these reactions and functional groups will not be discussed in this book (as it would take about 100,000+ pages), but what functional group reactions that will be discussed are the amino functional groups commonly found in amphetamines and derivatives.

In general, covalent bonds are less stable than ionic bonds. Most ionic compounds are stable solids with relatively high melting points (ranging from 200 to 2400 Celsius). Many ionic compounds can be heated to very high temperatures without any significant decomposition, such examples include: aluminum oxide, iron oxide, sodium chloride, and magnesium chloride. Most organic compounds decompose when heated to temperatures above 300 to 500 Celsius. The high melting points of ionic compounds are due primarily to crystal structure, and the result of strong electrical attractions between the elements and the molecules—these attractions can lead to super strong crystal lattices, as seen in some compounds like aluminum oxide (emeralds), and other ionic oxides (gems and sapphires). There is one mere example of an organic compound that should be demonstrated here; diamonds are composed of covalently bonded carbon atoms, with the molecules forming super strong crystal lattices.

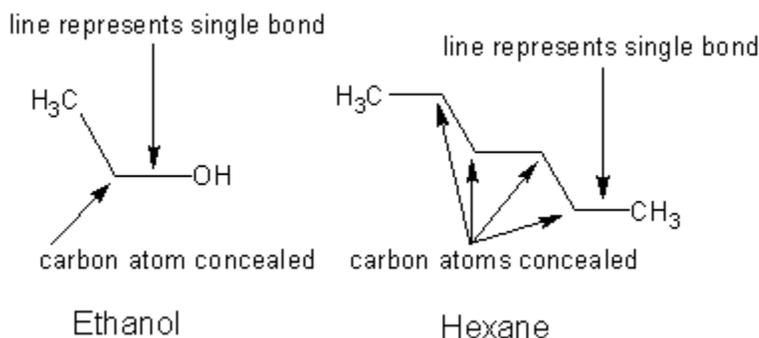
Other than this isolated example, most covalent compounds are solids or liquids with relatively low melting points and boiling points. This is the result of weaker electrical attractions between the molecules. In covalent compounds the weaker attractions exist primarily because the covalent molecules lack ionic charges, and are thereby not attracted or repelled to each other very much. Because of the lack of electrical attractions between covalent molecules, the boiling points of covalent molecules are the result of “intermolecular” forces (the melting points will be discussed shortly). Intermolecular forces are forces that exist between elements within one molecule upon different elements within another molecule. Such an example would be water, common hydrogen oxide. Water which is composed of two hydrogens bonded to a single oxygen has a significant boiling point of 100 Celsius at sea level, although it is a relatively small and light molecule. The reason water has such a high boiling point for its small size and weight, is due to intermolecular force attractions between the central oxygen atom of one molecule upon the two hydrogens of another water molecule (adjacent water molecule). The non-bonding type attractions (intermolecular forces) that water molecules have to each other is what

defines water's boiling point. In another example, methylene chloride (a common solvent you will find in this manual) has a very low boiling point for its size and weight (compared to water). The reason methylene chloride has a boiling point of about 60 degrees less than water is due to even weaker attractions between the methylene chloride molecules to each other. In essence, the weak intermolecular forces between the two chlorine atoms of one molecule upon the two hydrogen atoms of another, is what determines the low boiling point of methylene chloride.

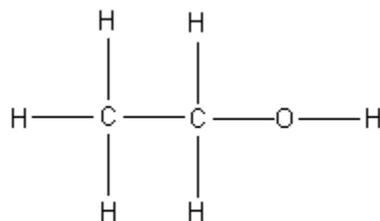
As previously stated, the melting points of ionic compounds are high because of strong electrical attractions between the elements and molecules, but a whole different scenario determines the melting points of covalent compounds. Because solid covalent compounds don't really show any significant intermolecular forces, the melting points of covalent compounds are determined by the shape, size, and bonding angles of the elements within the molecules. For example, think about blocks of wood of the same size, versus wood circular shapes of the same size—which would be easier to stack? Obviously the wood blocks would be much easier to stack than a pile of circular wood blocks. This is basically the essence behind the melting points of covalent compounds—although it gets a little bit more technical than this, but this info will be omitted because it is only of a concern to scientists. Molecules that are shaped properly, will pack together (not literally) much better than molecules that have awkward shapes. Molecules that pack together better, and more evenly, have much higher melting points than molecules that don't pack or fit together very well. Another factor that plays a role in melting point is size and weight of the molecules. Naturally, larger weight molecules tend to have higher melting points and boiling points than smaller weight molecules.

4. Understanding chemical structures and formulas

Understanding molecular structures and formulas is not necessarily needed for this manual (as all procedures are given with exact quantities) nevertheless, understanding formulas and the like can seriously help you better acknowledge what is taking place during a chemical reaction. Molecular formulas and structures are written using a variety of simple techniques. The most common of these techniques utilizes short lines, which indicate the bonds—of course the letters in the illustrations clearly indicate the elements. In short, the lines represent the chemical bonds either ionic or covalent, and the letters represent the elements (see a periodic table for each letter). In this manual, some of the letters have been omitted to reduce drawing time of the structures, and this method of omission is quite common in chemistry literature. In a common example, ethanol and hexane are both written with their central carbon atoms (and hydrogen atoms) concealed. Note: only carbon and hydrogen are commonly concealed in any given illustration. To know when a carbon has been concealed, simply look at how the lines change angles. Because carbon forms four bonds, it naturally contains two hydrogens per carbon (with the exception of alkenes, alkynes, benzenes and phenyls) within the central structure—these hydrogens are also concealed.

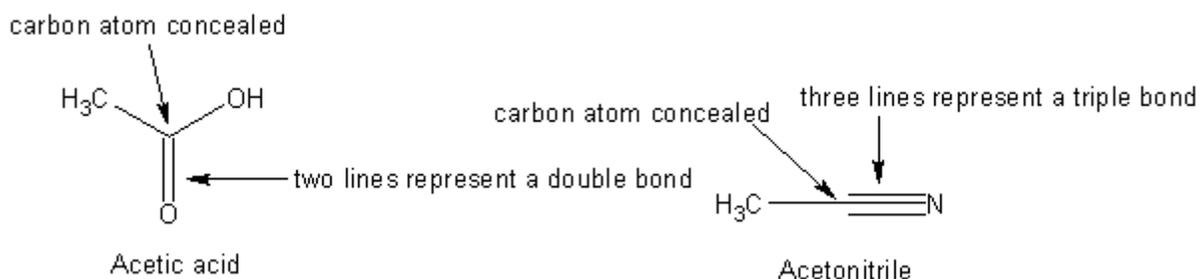


For review, the single lines represent single bonds, and the letters represent atoms. Therefore the letter C represents carbon, the letter O represents oxygen, and the letter H represents hydrogen. In the above illustration the central carbon atom in ethanol is concealed, along with two hydrogens bonded to it—this is the same scenario for hexane with a total of four carbon atoms concealed, along with eight hydrogens. Another method of writing structures and formulas is to use “expanded notation”. For example, the structure of ethanol could be written as follows:

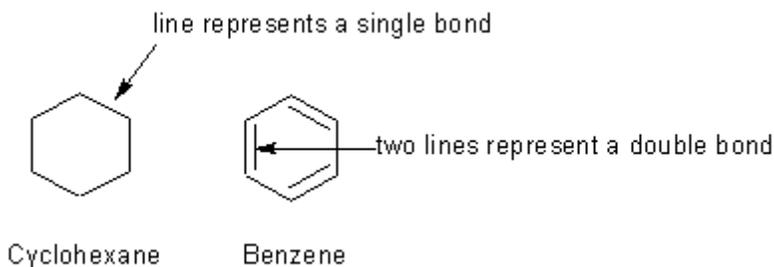


Ethanol

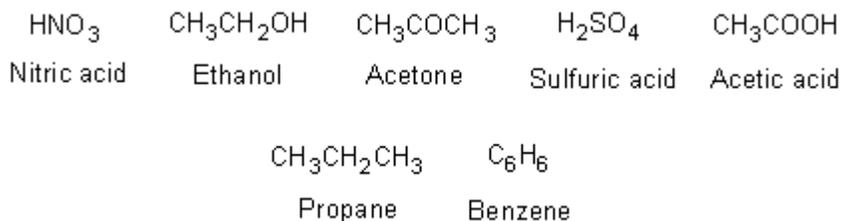
The above illustration is a common example of a molecular structure written in expanded notation. Expanded notation shows all elements within the structure. Expanded notation is seldom used in chemical literature to save writing time. In the following illustration we see a similar written structure with the central carbon atom concealed, along with the corresponding hydrogen. In this example, two lines are written to represent a double bond, in this case between the central carbon and an adjacent oxygen atom. In the right structure, a straight-line triple bond is shown, with the central carbon atom concealed as usual—as suspected, the letter N represents nitrogen.



Many covalent compounds are composed of rings. Rings are structures with a high degree of stability and belong to either a saturated group, or an unsaturated group. In the following illustration, the structure on the left is called cyclohexane, which represents a saturated ring. The right structure is the classic compound called benzene. In both structures, all carbon atoms have been concealed, along with the adjacent hydrogens—this is how most rings will be illustrated. The benzene structure represents an unsaturated ring. When discussing saturation and unsaturation, rings are not the only covalent compounds capable of these definitions. Many straight chain, and branched structures are capable of forming saturated and unsaturated structures—these are classified as alkynes, alkenes, and alkanes. An example of an unsaturated compound is the chemical acetylene, and an example of a saturated compound is the chemical propane. Another example are oils such as olive oil, which contain long chain unsaturated compounds—mainly oleic acid in this case.

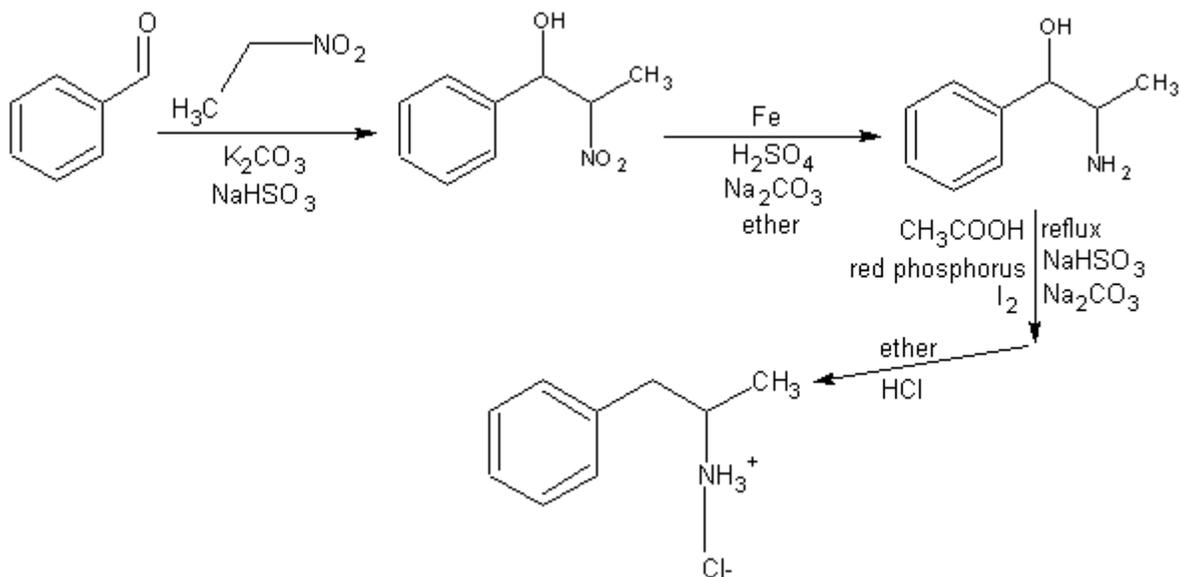


The final, and most common method of writing structures and formulas involves “condensed formula notation”. Condensed formula notation simply excludes the lines. To save time and space, many chemists use condensed formula notation. In this book, many of the reagents and solvents will be written in condensed formula notation. The following illustration gives a few examples of condensed formula notation.



5. Chemical reactions

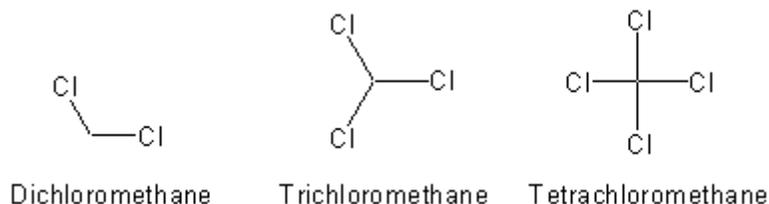
“Chemical reaction equations” are commonly used to illustrate a chemical reaction. In a chemical reaction equation the arrow represents the path the reaction takes. The items listed above and below the arrow represent the reagents, temperature, and/or conditions that exist for and during the reaction. In the following illustration we start with the “intermediate” compound called benzaldehyde (the far left structure). The intermediate compound is usually written on the left hand side, but can be written on the right hand side as long the arrow is pointing to the left. The intermediate is other wise called “the starting compound”. In the illustrated chemical reaction equation below, the arrow pointing to the right tells us that benzaldehyde is treated with a mixture of nitroethane and potassium carbonate in the presence of sodium bisulfite. The nitroethane, potassium carbonate, and sodium bisulfite are commonly called the “reagents”, and are usually written in condensed formula notation. The reagents are usually written above and/or below the arrow (basic chemistry classes often put the reagents after a + sign, but in the professional world, we don’t use + signs). Under most conditions, to shorten the illustration, we omit the by-products formed during the reaction (but sometimes it helps the reader understand better what is going on when the by-product are given; however, by-products will not be given in the illustrations of this book). Now, looking at the rest of the equation, we see the resulting product of this first reaction, is a nitro intermediate, and this new intermediate is then reacted with iron in the presence of sulfuric acid, and then so on, and so on.....Although understanding chemical reactions is not fully necessary to properly use this book, a brief understanding will better help you understand what is taking place.



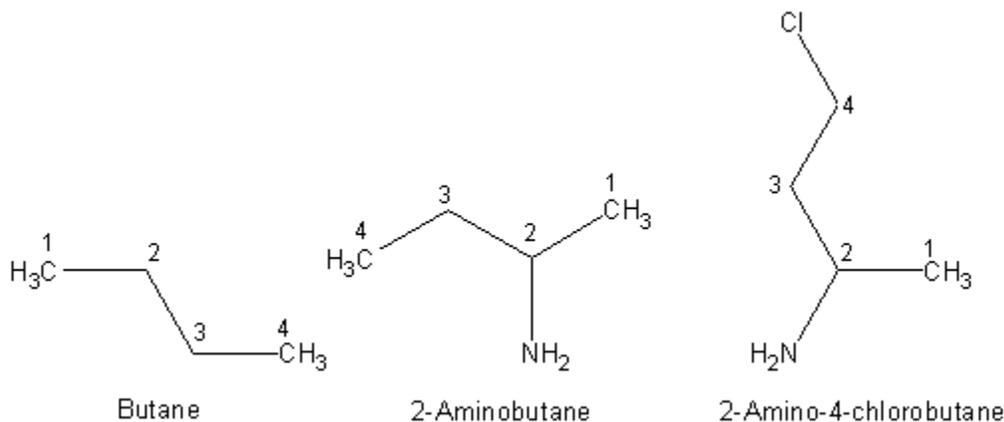
6. Language of chemistry

Chemistry has a unique language all to its own. This language is called the IUPAC language, or system. The IUPAC system of language can be quite difficult and confusing to learn, so we will not go into to much depth in this category. What we will discuss is the basic language of chemistry. For starters, you should familiarize yourself with the numbers 1 through 10. These numbers are given in the following table. After you have learned these numbers, practice them using the illustrated structures below.

| | | | | |
|---------|----------|----------|----------|----------|
| Mono: 1 | Tri: 3 | Penta: 5 | Hepta: 7 | Nona: 9 |
| Di: 2 | Tetra: 4 | Hexa: 6 | Octa: 8 | Deca: 10 |



As previously discussed, covalent compounds contain carbon chains, or infrastructures. These carbon chains are numbered so chemists are able to name them. Because the rules that govern the system of numbering can be tricky for beginners to learn, we will not go into to much depth. In the following illustration, butane is shown with correct numbering. Thereafter, another more complicated structure is shown with correct numbering, followed by an even more complicated structure. In each of these examples, the numbering demonstrates how compounds can be numbered and labeled for proper identification.



Another important tool for being able to name chemical compounds, is knowing the correct functional group. Functional groups are bits and pieces of molecules that have distinctive properties to them. Functional groups play a major role in determining the correct identification for any given compound. Functional groups can be tricky for many beginners to memorize, so we will not go into to much depth here as well. However, we will discuss a few common functional groups that you will encounter in this book. Take a look now at the following table. Notice each unique functional group, and the corresponding chemical compound it is attached to—notice any patterns? The primary functional groups that we will deal with in this book are amine groups.

| Some common functional groups | | | | | |
|-------------------------------|--|--------------|---------------------------|--|--------------|
| -NO ₂ (nitro) | | Nitromethane | -NH ₂ (amine) | | Ethyl amine |
| -COOH (carboxylic acid) | | Acetic acid | -CN (Nitrile) | | Acetonitrile |
| -OH (alcohol) | | Ethanol | -CH ₃ (Methyl) | | Methane |

As far as the IUPAC system and functional group are concerned, most chemical compounds are identified and named in these manners; although, in some cases, common names have been attributed to many chemical compounds to simply make it easier to identify them. For example, the names of the three chemical formulas illustrated at the top of the page are written in IUPAC nomenclature, but experienced chemists will simply name these compounds methylene chloride (dichloromethane), chloroform (trichloromethane), and carbon tetrachloride (tetrachloromethane). Even though common names are quite common for identifying chemicals, the correct IUPAC name should be given in special cases to correctly identify the compound. For example, 2-amino-4-chlorobutane would not make sense if we simply called it aminochlorobutane. Saying aminochlorobutane does not depict where on the carbon chain the amino functional group is, or the chlorine atom.

7. Conversion factors

For some readers (especially Americans), the metric system (other wise known as the SI system) is vague, or somewhat unfamiliar. 99% of all the units of weight and measurement in this book are given using the SI system; therefore, a translation from one unit to equipment is automatically calibrated in SI units, so even inexperienced persons will not have to worry too much about knowing the SI system. Regardless, try a few conversions of your own just for practice. Example: Convert 150 Celsius into Fahrenheit—Solution: multiply 150 by 1.8 and then add 32. The answer would be 302 Fahrenheit. Example 2: Convert 1.2 gallons into milliliters—Solution: multiply 1.2 by 3,785. The answer would be 4542 milliliters.

| To convert | Into | Multiply By | To convert | Into | Multiply By |
|------------------------|---------------|----------------|-----------------|----------------|-------------------------|
| Atmospheres | Cm of mercury | 76 | Liters | Gallons | 0.2642 |
| Atmospheres | Mm of mercury | 760 | Liters | Ounces (fluid) | 33.814 |
| Atmospheres | Torrs | 760 | Meters | Feet | 3.281 |
| Atmospheres | In of mercury | 29.92 | Meters | Inches | 39.37 |
| Atmospheres | psi | 14.7 | Milligrams | Ounces | 3.527×10^{-5} |
| Celsius | Fahrenheit | $1.8 + 32$ | Milligrams | Pounds | 2.2046×10^{-6} |
| Centimeters | Inches | 0.3937 | Milliliters | Gallons | 2.642×10^{-4} |
| Centimeters | Meters | 0.01 | Milliliters | Ounces (fluid) | 0.0338 |
| Centimeters of mercury | Atmospheres | 0.01316 | Millimeters | Feet | 3.281×10^{-3} |
| Centimeters of mercury | psi | 0.1934 | Millimeters | Inches | 0.03937 |
| Fahrenheit | Celsius | $0.556 - 17.8$ | Ounces | Grams | 28.349527 |
| Feet | Meters | 0.3048 | Ounces | Kilograms | 0.0283 |
| Feet | Millimeters | 304.8 | Ounces | Milligrams | 28,349.5 |
| Gallons | Liters | 3.785 | Pints (liquid) | Liters | 0.4732 |
| Gallons | Milliliters | 3,785 | Pints (liquid) | Milliliters | 473.2 |
| Grams | Ounces | 0.03527 | Pounds | Grams | 453.5924 |
| Inches | Centimeters | 2.540 | Pounds | Kilograms | 0.4536 |
| Inches | Millimeters | 25.40 | psi | Atmospheres | 0.06804 |
| Inches of mercury | Atmospheres | 0.03342 | Quarts (liquid) | Liters | 0.9464 |
| Inches of mercury | psi | 0.4912 | Quarts (liquid) | Milliliters | 946.4 |
| Kilograms | Ounces | 35.274 | Torr | Mm of mercury | 1.0 |
| Kilograms | Pounds | 2.205 | Torr | Atmospheres | 1.316×10^{-3} |

SECTION 2: Laboratory Techniques

Part 2: General Laboratory Procedures

A. Methods of heating

For heating purposes in the lab, garage, home, or office, a variety of heating methods can be used. Several factors are involved in determining what method of heating should be used. These factors include the shape and size of the reaction vessel, the desired reaction temperature, and whether the reaction mixture must be stirred at the same time it is heated. The most common methods of heating used in labs are listed below.

1) Free flame

Bunsen burners refer to the term free flame. The Bunsen burner is a commonly used heating device in general chemistry labs, but its use in modern labs is limited. It is very inexpensive to purchase and operate, and permits mixtures to be heated rapidly. Bunsen burners are also commonly used to heat solids. Their use in heating liquids is limited due to potential hazards. Heating liquids with Bunsen burners can lead to violent bumping and foaming. This bumping and foaming can lead to flashovers. In general, never heat flammable liquids with Bunsen burners. When using Bunsen burners, be certain there are no flammable solids, liquids, or vapors in the vicinity. Bunsen burners can be used to heat high boiling liquids such as in the distillation of benzyl chloride, which has a high boiling point—however, never heat volatile chemicals with free flames. Bunsen burners are commonly used in roasting solids and mixtures, such as dehydrating solids as seen when heating Epsom salt to remove its water of hydration, and to form anhydrous magnesium sulfate.

Standard propane tanks obtainable from any hardware store can be used as a fuel source, and the lower portion of a torch nozzle (the angled metal nozzle commonly screwed into the propane tank for use in soldering or heating pipes), can be unscrewed from the upper angled nozzle portion, and then screwed into the propane tank, and then a piece of tubing, say latex tubing, is then connected from there, to the Bunsen burner. Bunsen burners can be purchased on online auction sites and similar places. Propane camping stoves, or kerosene camping stoves can be used as heating sources in place of Bunsen burners.

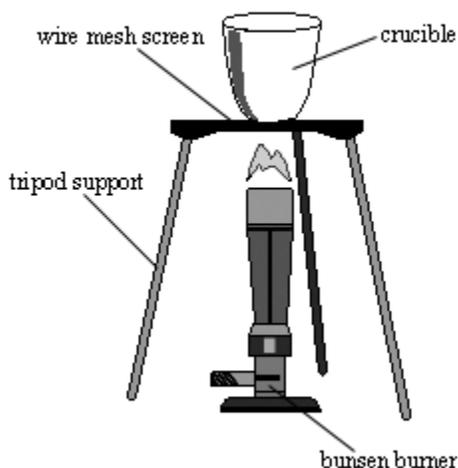


Figure 001. Common laboratory Bunsen burner with support stand.

2) Steam bath

Steam baths are an inexpensive and useful way for heating mixtures up to 100 Celsius. Steam baths can also be used to heat mixtures from 50 to 90 Celsius. Steam baths are very easy to use and operate, and they heat mixtures without blind spots. Blind spots occur

when heating is not even. A steam bath is much more useful for heating low-boiling liquids than a free flame, and any vapors which may escape from the distillation apparatus simply dissipate with the steam.

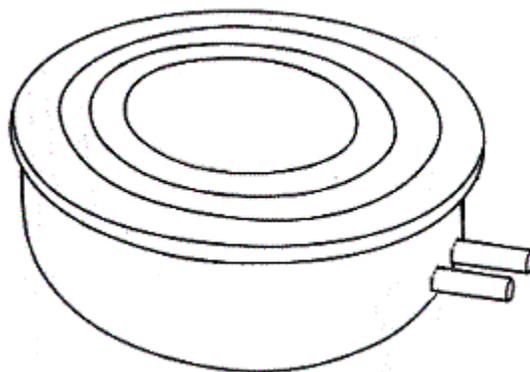


Figure 002. A common steam bath. To use a steam bath, remove enough rings so that a round-bottom flask will rest on a ring enough so to expose it to the steam without falling through.

3) Oil bath

Oil baths are useful for heating mixtures. The contact of the flask with the hot oil heats the flask perfectly because the hot oil completely surrounds the sides of the flask. This results in even heating and effective temperature control. Oil baths are relatively inexpensive and are safe to operate because they lack an open flame. Oil baths are slow to heat, and they cool slowly after use. These are some of the drawbacks associated with oil baths. In addition, the flask retains an oily residue, which is slippery and must be cleaned off.

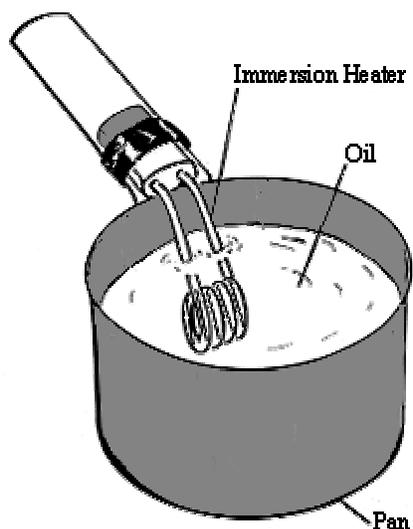


Figure 003. A typical immersion heater used with an oil bath. The flask is immersed about half way into the oil.

4) Electric Heating Mantles

Heating mantles are the most common method of heating round bottom glassware, and they come in a wide variety of shapes and sizes. Sizes ranging from 10 milliliters to a whopping 12 liters are available. The most common sizes are the 250 milliliter, 500 milliliter, and 1000 milliliter models. These models range in price from 80 to 200 dollars. A voltage regulator is usually used to control the heating, and is sold separately. Exercise care in setting the voltage of a heating mantle because too much voltage can lead to undesired temperature. Test the voltage regulator on an empty flask equipped with a thermometer to familiarize you with the temperature settings. Some voltage regulators will clearly indicate the temperature. A label is usually attached to the heating mantle, which indicates the maximum safe voltage. Note: A heating mantle designed to tolerate a maximum of 20 volts quickly burns out if 120 volts is applied. Read the maximum tolerances aloud for your heating mantle before using it.

Most 100 to 500 milliliter heating mantles tolerate a full 120-volt input, and some large mantles even require two voltage regulators. On a final note, be certain the heating mantles size is appropriate for the flask being used.

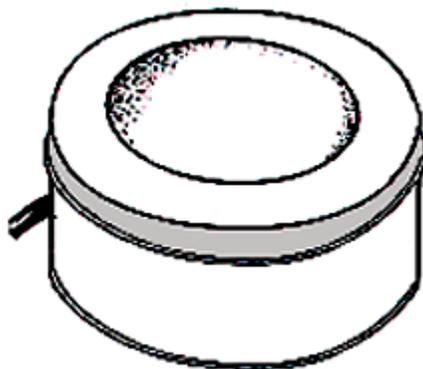


Figure 004. A classic heating mantle.

5) Hot Plates

Hot plates are by far the most common method of heating flat bottom laboratory glassware. Hotplates are exclusively used in heating Erlenmeyer flasks and beakers. Many hot plates come doubled with a magnetic stirrer and are usually called hot plate/stirrers. These hot plate/stirrers are very useful in the heating and the simultaneous mixing of liquids. Some hot plates come without magnetic stirrers. Laboratory hotplates heat relatively slow and they cool slowly, but their energy efficient and they maintain the desired temperatures for indefinite time.

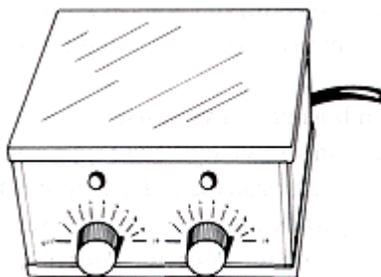


Figure 005. A common hot plate with a magnetic stirrer. Most hot plates double as magnetic stirrers.

B. Methods of Cooling

Cooling is often required during a chemical reaction in order to maintain proper reaction temperatures. Not properly cooling reaction mixtures can lead to conditions including evolution of poisonous gases, decomposition of products, and unwanted side reactions. Cooling baths are cheap and readily available. Dry ice is readily available and is used to make excellent cooling baths. Cooling is not as easy as it may appear. In some ice baths the ice will melt rapidly during the chemical reaction. Ice that rapidly melts must be continuously refilled in order to maintain proper reaction temperature.

Cooling baths should be at least three times the volume of the reaction flask. For example, if using a 1-liter flask to contain the reaction mixture, a 3-liter container should be used to house the 1-liter flask. Before adding the cooling agent (ice water, ice, or dry ice) to the bath, make sure the 1-liter flask is seated in the bath container. Then fill the container with the cooling agent. The 1-liter flask should be submerged as far as possible into the ice bath. In other words, 80% of the total height of the 1-liter flask should be submerged in the cooling bath. In some cases the flask being cooled will displace the cooling agent (cold water, or ice water) causing it to float and possible tip over. Lead rings, or even heavy set solder, which are cheap and commercially available, make useful weights to keep the reaction flask seated in the cooling bath.



Figure 006. Setup for cooling bath. The container can be used to house any cooling medium.

1) Cold water bath

Simple cooling utilizes a cold-water bath. Cold-water baths are used to keep the reaction temperature from 15 to 50 Celsius. In some cases the water bath will have to be quickly drained, and then refilled with cold water in order to maintain the desired reaction temperature. In most cases cold-water baths are used for general long-term temperature control.

2) Ice water bath

Ice water baths are commonly used to keep reaction temperatures around 5 to 30 Celsius. Ice water baths are used in place of cold-water baths where long term cooling, but a slight colder temperature is needed.

3) Standard ice bath

The standard ice bath is the most common method of cooling reaction mixtures. This method of cooling can produce temperatures of 0 to 20 Celsius. Ice baths are composed of chopped up pieces of ice, and the ice should be finely crushed so that it adheres to the wall of the reaction flask as much as possible. Remember to place the reaction flask into the empty bath container before adding the ice. As the cooling proceeds the ice may melt rapidly, moderately, or slow. If the ice is melted, drain off the water and then add more finely crushed ice. Continue the process as many times as needed. Depending on the time and conditions, the ice may not have to be replaced.

4) Salt/ice bath

The salt/ice bath is a modified version of the ice bath. Depending on the type of salt used, salt/ice baths are very useful for producing temperatures ranging from -55 to 0 Celsius. To prepare a salt/ice bath, simply mix the finely crushed ice with 20% of its weight in salt. Salt/ice baths can maintain their temperatures for varying amounts of time depending on the heat evolved during a particular chemical reaction, time, and/or other conditions. In some procedures the salt/ice bath will have to be replaced with a fresh batch. When the salt used is potassium chloride the temperature achieved will be around -10 to 0 Celsius. When the salt used is sodium chloride the temperature achieved will be -20 to 0 Celsius. When the salt used is anhydrous magnesium chloride the temperature achieved will be -30 to 0 Celsius, and when the salt used is calcium chloride hexahydrate the temperature achieved will be -55 to 0 Celsius.

5) Dry ice/acetone bath

Dry ice baths are very common in the modern laboratory. Dry ice is readily available and can achieve temperatures of -70 to -30 Celsius. Dry ice is seldom used along for cooling purposes due to its volatility. It is usually used in combination with a solvent. The solvent is normally acetone, but ethanol, ethyl acetate, or ether can be used. To use a dry ice/acetone bath, add the dry ice to its same weight in acetone (50/50) and then place this mixture into the bath container. Then place this dry ice/acetone filled bath container into a second yet larger container and then fill this second larger container with ice/salt. The second container bath acts like an insulator to the inner bath container giving longer life to the dry ice/acetone bath. The dry ice bath may rapidly deplete if you withhold the second cooling bath. For short-term cooling and use, the second cooling bath will not be needed. For long term cooling, withhold the second cooling bath and place the dry ice/acetone bath into a refrigerator freezer.

6) Cooling tricks of the trade

One method of cooling is to place the reaction apparatus, flask, or beaker into a refrigerator or freezer (as long as it fits). This allows for complete cooling without refilling containers with ice or cold water. A major draw back to doing this is a lack of ventilation. In some procedures highly poisonous and corrosive gases are evolved and hence must be properly vented. If a procedure is relatively free

from toxic or corrosive emissions, the apparatus can be placed into a freezer or refrigerator if it fits. Refrigerators and freezers are also very handy when having to store reaction mixtures for several hours or several days. Simply place the reaction flask into the refrigerator or freezer and then cool for the amount of time needed. This eliminates the need for ice baths and the like.

C. Extraction

Extraction is a major part of many chemical procedures, and is usually conducted before the recrystallization process. Extraction is used to “separate” a product from a reaction mixture. The reaction mixture (the chemical mixture to be extracted), or another source of chemicals, such as a food product, is merely shaken with a certain solvent multiple times. During this shaking, the desired product in the reaction mixture or food product, plant, ect., is dissolved into the solvent. The solvent is then removed from the extracted mixture, and the product recrystallized there from.

The volume of solvent used is dependent on the desired products solubility in it. When the volume of the solvent has been determined, it is broken into small portions, and then each portion is shaken with the reaction mixture independently. After all the portions have been shaken with the reaction mixture, they are combined and then the product is recrystallized. For the chemical procedures in this manual, the solvent, quantity, and volume size of each portion is given in detail.

1) Funnel Size

The size of the seperatory funnel is of practical consideration when carrying out the extraction process. A seperatory funnel is the piece of glass traditionally used in extraction. In order to leave room for shaking the solution the funnel should be 30 to 50% larger than the total combined volume of liquid. For example, use a 250-milliliter seperatory funnel when extracting 100 milliliters of reaction mixture with 50 milliliters of solvent. If you are extracting large volumes of liquid, and you don’t have a proper sized seperatory funnel, simply divide the reaction mixture into smaller portions and do the same for the solvent portions.



Figure 007. A standard laboratory seperatory funnel.

2) Performing the Extraction

The first step in extraction is to pour the reaction mixture, or place the food product, plant, ect., to be extracted, and the solvent into the seperatory funnel or appropriate container. If extracting a chemical reaction mixture a two-layer mixture will result. Which layer is what depends on the densities of the chemicals in the reaction mixture verses the density of the solvent. If the density of the solvent is greater then the chemicals in the reaction mixture, the solvent will be the bottom layer. If the opposite is true, the solvent will be the upper layer. For example, when a water solution is to be extracted with two portions of methylene chloride, the water solution and the first portion of methylene chloride are placed into the seperatory funnel (make sure the stopcock is closed). A two-layer mixture results. The methylene chloride will be the bottom layer because methylene chloride is denser then water. If extracting a food product, plant, seed, ect., the solvent and the material to be extracted are placed into an extraction apparatus, or suitable container or blender and then heated and/or blended for a specified amount of time.

When extracting a water mixture with methylene chloride, for example, the next simple step is to shake the mixture for several minutes. Afterwards, drain-off the bottom methylene chloride layer only, leaving the water solution in the separatory funnel. After the bottom methylene chloride layer is removed, pour the second methylene chloride portion into the separatory funnel and then begin shaking. Then once again, drain-off the bottom methylene chloride layer. At this point the water solution has been successfully extracted. Both drained-off methylene chloride portions can then be combined (if not already done so), and the product recrystallized. Note: If sulfuric acid is present in the reaction mixture, the methylene chloride will always be the upper layer. Sulfuric acid is denser than methylene chloride. Which layer is what will be described for each extraction process in this book. Certain solvent combinations (a water solution of sodium hydroxide and chloroform) lead to emulsions when shaken together. Emulsification cannot always be anticipated, so choose the solvent wisely, or wait along time after shaking for the emulsion to dissipate.

I. For extracting a chemical reaction mixture:

1. Place the reaction mixture to be extracted into a separatory funnel (make sure the bottom stopcock is closed).
2. Add the solvent portion slowly to the separatory funnel.
3. Stopper the separatory funnel, and then begin shaking the funnel for a few minutes.
4. After shaking for a few minutes, allow the two layers to completely settle, and then properly vent the funnel as shown in the following illustration. Then slightly open the bottom stopcock and slowly drain-off the bottom layer. If the upper layer is the solvent, the bottom reaction mixture layer will have to be drained off first, and then poured back into the same separatory funnel after the upper solvent layer has been drained off. If the bottom layer is the solvent, simply drain it off only, and leave the upper reaction mixture layer.
5. After the appropriate layer or layers have been drained off, and the reaction mixture is the only liquid in the separatory funnel, add the second portion of the solvent and repeat steps 1 through 5.
6. Repeat steps 1 through 5 as many times indicated in the procedure. For example, if an extraction calls for three portions of methylene chloride, conduct steps 1 through 5 three times.
7. After the number of extractions has been completed, combine all drained-off solvent portions (if not already done so).

Note: In some cases the reaction mixture will be very dark in appearance, and when extracted, forms another dark appearance with the solvent making the phase boundary between upper and bottom layers hard to see. If this happens, hold the separatory funnel up to a light, or use a flashlight.

Note: While shaking the funnel, vapors from the reaction mixture and/or solvent can increase pressure inside the separatory funnel. Proper venting of the separatory funnel is necessary in order to relieve this pressure. To properly vent a separatory funnel, rest the funnel in one hand while grasping the glass stopper. Then tilt the funnel so that the stopcock end is pointed up and away from anyone including yourself. After which rotate the stopcock to the open position. Be certain that the level of the liquid is below the stopcock opening so that none is forced out when the stopcock is opened.

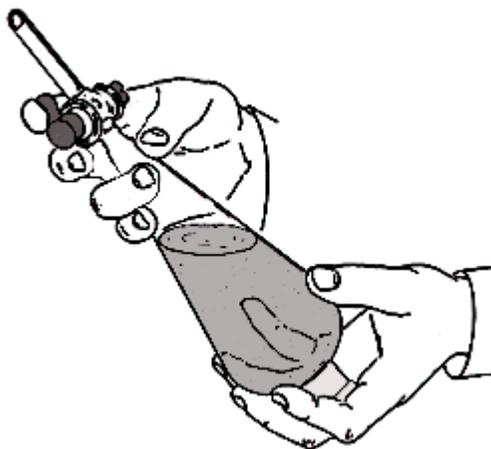


Figure 008. Correct way of venting a separatory funnel.

3) Draining the funnel

After shaking the funnel, the layer or layers must be drained off. To do this, simply place the separatory funnel into a ring stand supported by a base support. The stopper must be off in order to drain the funnel, and before opening the stopcock remove the stopper. Attempting to drain the funnel before removing the stopper can result in a vacuum making it difficult to remove the stopper.

When draining the bottom layer, the speed should be adequate as to not over drain. Over draining means to accidentally drain-off some the upper layer. The opening of the stopcock (either fully or partially open) is determined as the phase boundary of the upper

liquid approaches the stopcock. When the phase boundary is far away, draining can be done rapidly. When the phase boundary approaches the stopcock, the drain speed should be reduced to a drip.

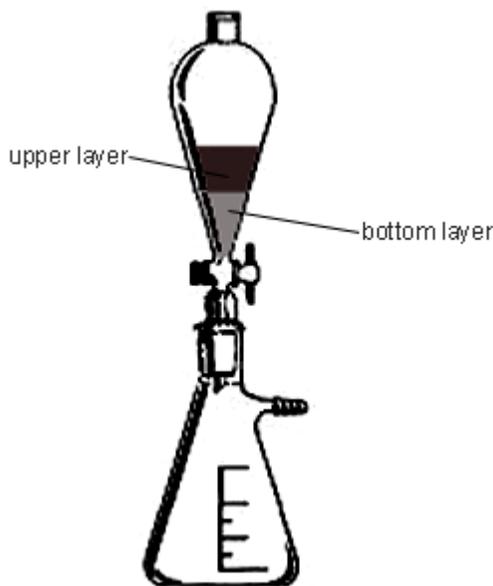


Figure 009. Separatory funnel positioned for draining.

Separatory funnel can be purchased from on-line auction sites, and other places for reasonable prices.

II. If extracting a food product, plant, seed, ect:

1. Place into an extraction apparatus (more detail of this will be given when applicable), flask, or appropriate container, the material to be extracted, such as a plant, seed, root, ect., followed by the appropriate solvent. Note: in most cases, the material to be extracted should be ground-up or pulverized thoroughly before placing into the extraction apparatus, flask, or container.
2. Reflux, and/or blend the mixture for the specified amount of time (conditions and time will be specified by each procedure). The specified amount of time can range from 30 minutes to 18 hours.
3. After the refluxing/blending operation, the resulting mixture is then filtered to remove it from the insoluble organic matter.
4. Now, depending on the extraction process, and what is being extracted, the filtered solvent mixture is either treated with chemical reagents, filtered, and then evaporated, or simply evaporated to remove the solvent and leave behind the extracted substance. In some cases, this step can be quite complex, as some extraction processes require treatment with multiple reagents, titrations, filtrations, ect., in order to facilitate proper extraction. It should be noted that exact instructions will be given for each extraction process where applicable.

4) Salting Out

In some cases, an organic compound (usually a liquid) dissolved in water can be precipitated by the addition of sodium chloride, sodium sulfate, or magnesium sulfate. These salts have a much higher affinity for water than most organic compounds, so they tend to dissolve in the water leaving the dissolved organic compound with no room to remain dissolved. The lack of space causes the organic compound to precipitate (organic liquids form a second layer). Water solutions of isopropyl alcohol (rubbing alcohol) for example, can be salted out by the addition of sodium chloride to the mixture followed by rapid shaking of the mixture. The quantity of sodium chloride used is determined by the alcohol concentration. The weaker the concentration is, the more salt is needed. After shaking, a two-layer mixture results. The isopropyl alcohol will be the top layer, and the brine solution the bottom. Try it out for yourself, i.e., salt out a sample of rubbing alcohol using a separatory funnel and salt.

Now that you are familiar with the extraction process, let's practice this skill by familiarizing yourself with some common extraction processes. To do this, it is good to practice on extracting chemicals from food products as they are readily available, and can be quite interesting to do so. Some extraction processes are very simple and straightforward, such as simple extraction of a reaction mixture, but some extraction processes can be quite complex or more difficult. Some extractions require refluxing operations, and some require steam distillation. Whatever is required for the extraction, extraction is a necessary knowledge to have and the following extraction processes will help you better understand the nature of extraction as a whole.

- Methods of Extraction -

Extraction process 1: Extraction of Piperine from black pepper

Piperine

Piperine forms monoclinic crystals or prisms when recrystallized from alcohol. The crystals have a melting point of 130 Celsius. The crystals are at first tasteless, but then rapidly impart a burning taste when ingested. Piperine is insoluble in water, slightly soluble in alcohol, and soluble in chloroform, benzene, and acetic acid. Piperine is readily extracted from black pepper, and is one of the chief compounds responsible for the characteristic taste of black pepper.

Method 1: Extraction of piperine from black pepper

Materials:

| | |
|--|---|
| 1. 75 grams (2.6 oz.) of powdered or finely ground black pepper | 4. 65 milliliters (2.2 fluid oz.) of warm water |
| 2. 750 milliliters (25.3 fluid oz.) of 95% ethyl alcohol | 5. 65 milliliters (2.2 fluid oz.) of more water |
| 3. 50 milliliters (1.7 fluid oz.) of a 10% potassium hydroxide solution in 95% ethyl alcohol | 6. 100 milliliters (3.4 fluid oz.) of acetone |

Hazards: Wear gloves when handling potassium hydroxide, which is very corrosive. Extinguish all flames before using ethyl alcohol, and acetone, both of which are flammable.

Procedure: Into a standard reflux apparatus, place 75 grams (2.6 oz.) of powdered or finely ground black pepper (if using fresh black pepper corns or granules, the corns or granules should be finely ground before using). Note: 75 grams of black pepper is about 2/3 of a normal bottle sold in the grocery store. After adding the black pepper to the reflux apparatus, add in 750 milliliters (25.3 fluid oz.) of 95% ethyl alcohol. Thereafter, reflux the mixture at 78 Celsius for about 4 or 5 hours. After the reflux extraction process, remove the heat source, and allow the alcohol mixture to cool to room temperature. Thereafter, filter the alcohol extract to remove insoluble materials, and then place this filtered alcohol extract into a distillation apparatus, and distill-off the ethyl alcohol at 78 Celsius until the total remaining volume is about 75 milliliters (2.5 fluid oz.). When most of the ethyl alcohol has been removed, and the left over remaining alcohol concentrate is around 75 milliliters (2.5 fluid oz.) in volume, stop the distillation process, and collect the left over remaining alcohol concentrate (after it has cooled), and place it into a clean beaker. Then, into a second clean beaker, add in 50 milliliters (1.7 fluid oz.) of a 10% potassium hydroxide solution in 95% ethyl alcohol. Thereafter, to the potassium hydroxide/alcohol solution, add in the concentrated alcohol extract, and thereafter, heat the total mixture at about 60 to 70 Celsius. When the temperature of this mixture reaches 60 to 70 Celsius, slowly add drop wise, 65 milliliters (2.2 fluid oz.) of warm water. Note: during the addition of the water, the desired piperine compound will gradually precipitate. When precipitation begins, remove the heat source, and allow the alcohol mixture to cool to room temperature, and during this cooling period continue to add the water, slowly and drop-wise. When the mixture has cooled to room temperature, add in 65 milliliters (2.2 fluid oz.) of more water (cold water this time), and then stir the entire mixture for about 30 minutes at room temperature, and then allow the entire mixture to stand (no stirring) for several hours at room temperature. Afterwards, filter-off the precipitated solid, and then vacuum dry or air-dry it. Finally, recrystallize this dry solid from 100 milliliters (3.4 fluid oz.) of acetone, and after the recrystallization process, vacuum dry or air-dry the filtered-off crystals. The result will be about 3 grams (0.1 oz.) of the desired piperine compound with a melting point of 128 Celsius.

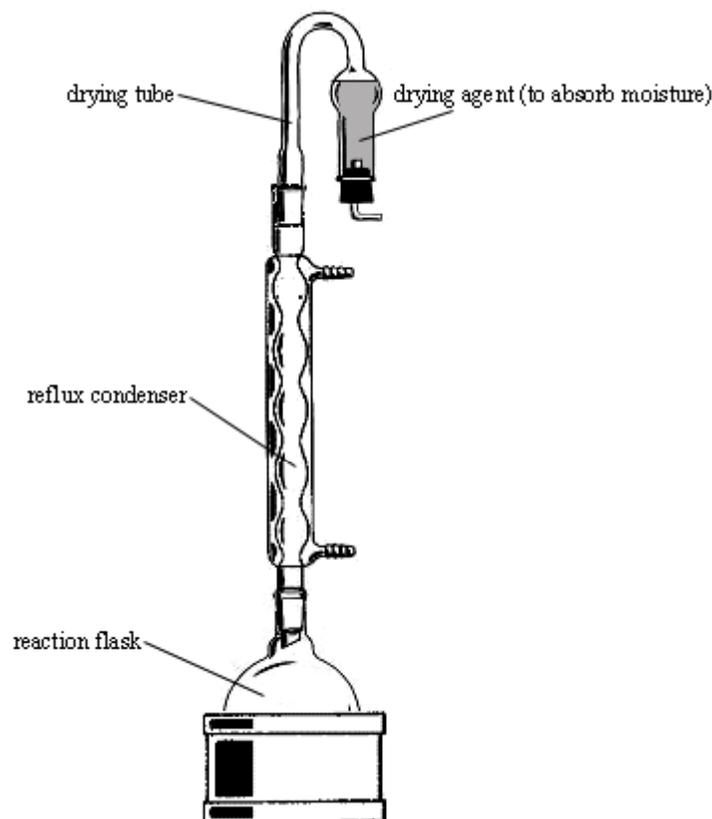


Figure 010. Reflux apparatus equipped with drying tube for the extraction of piperine from black pepper. Cold water should be circulated through the reflux condenser jacket.

Extraction process 2: Extraction of vanillin from vanilla extract

Vanillin (4-hydroxy-3-methoxybenzaldehyde)

Vanillin forms white to slightly yellow needle like crystals, which have a very pleasant taste and odor. The crystals are slowly oxidized on exposure to air and light, and should be stored in airtight amber glass bottles. The crystals have a melting point of 80 to 81 Celsius, and a boiling point of 285 Celsius with some possible decomposition. The crystals are not very soluble in water, but are freely soluble in alcohol, chloroform, and most common solvents. Vanillin is one of the major compounds responsible for the characteristic taste of vanilla.

Method 1: Extraction of vanillin from store bought vanilla extract

Materials:

| | |
|---|---|
| 1. 75 milliliters to 118 milliliters (2.5 to 4 fluid oz.), of grocery store brand vanilla extract | 3. Three 50-milliliter portions (three 1.6 fluid oz. portions) of diethyl ether |
| 2. 50 milliliters (1.6 fluid oz.) of warm water | 4. 10 grams (0.35 oz.) of anhydrous magnesium sulfate |

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable and capable of forming explosive mixtures with air.

Procedure: Pour a large bottle (75 milliliters to 118 milliliters, 2.5 to 4 fluid oz.) of grocery store brand vanilla extract into a suitable beaker, and then add in 50 milliliters (1.6 fluid oz.) of warm water. Then extract this entire mixture with three 50-milliliter portions (three 1.6 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions (if not already done), and then

dry this combined ether portion by adding to it, 10 grams (0.35 oz.) of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the heating process, and recover the left over remaining residue (after it has cooled to room temperature), and then vacuum dry or air-dry this collected residue. Thereafter, set this dry residue aside just for a moment. Now, depending on how much residue you have (based on what quantity of grocery store vanilla extract you purchased), add your collected left over residue into heated water contained in suitable sized beaker. In other words, place 20 milliliters (0.67 fluid oz.) of water per 1 gram (0.04 oz.) of your residue into a breaker, and heat to 80 Celsius—thereby, add in your residue. After you add in the residue, continue to heat the water mixture at 80 Celsius with moderate stirring for about 15 minutes, and then quickly filter this water mixture (before it cools), and then place the filtered water mixture into a clean beaker, and allow it to cool to room temperature—whereby crystals of vanillin will form. After the water mixture has cooled to room temperature, place it into an ice bath (or use a freezer), and allow the mixture to stand at 0 Celsius for 1 hour. Then filter-off the precipitated crystals of vanillin, and then vacuum dry or air-dry the crystals. Note: the crystals should be stored in airtight bottles in a cool place to prevent oxidation. Note: there are numerous modifications to this extraction process.

Extraction process 3: Extraction of Eugenol from cloves

Eugenol (*4-allyl-2-methoxyphenol*)

Eugenol forms a colorless to pale yellowish liquid with a boiling point of 255 Celsius. Eugenol slowly turns dark on exposure to air, so it should be stored in airtight bottles in a cool place. Eugenol has a powerful odor of cloves, from which it is readily extracted from ordinary spice cloves. Eugenol has a melting point of -9 Celsius, so the oil may crystallize on standing under cold temperatures. Eugenol is miscible with alcohol, methylene chloride, and ether, but insoluble in water. Eugenol is a major starting point for the preparation of psychedelic amphetamines.

Method 1: Extraction of eugenol from store bought cloves

Materials:

| | |
|---|--|
| 1. 100 grams (3.5 oz.) of cloves (regular store bought cloves) | 7. 250 to 300 milliliters (8.5 to 10.1 fluid oz.) of a 5% hydrochloric solution |
| 2. 500 milliliters (17 fluid oz.) of water | 8. Four 40-milliliter portions (four 1.4 fluid oz. portions) of methylene chloride |
| 3. 250 milliliters (8.4 fluid oz.) of water | 9. 50-milliliter portion (1.7 fluid oz.) of water |
| 4. three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride | 10. 50 milliliter portion (1.7 fluid oz.) of a 23% sodium chloride solution |
| 5. six 50-milliliter portions (six 1.7 fluid oz. portions) of a 5% potassium hydroxide solution | 11. 15 grams (0.52 oz.) of anhydrous sodium sulfate |
| 6. 50 milliliters (1.7 fluid oz.) of methylene chloride | |

Hazards: Wear gloves when handling potassium hydroxide and hydrochloric acid, both of which are capable of causing skin burns.

Procedure: Into a suitable steam distillation apparatus (fitted with a 250 milliliter addition funnel, or better), place 100 grams (3.5 oz.) of cloves (regular store bought cloves). Thereafter, add in 500 milliliters (17 fluid oz.) of water, and then add 250 milliliters (8.4 fluid oz.) of water to the addition funnel. This 250-milliliter addition funnel should contain about 200 milliliters of water at all times, and the water therein should be added to the cloves and water mixture periodically to keep the flasks water volume at around 500 milliliters all throughout the steam distillation process. Then heat the cloves and water mixture to 105 to 110 Celsius, and allow the mixture to be steam distilled. The process should take about 150 minutes, and thereafter, stop the steam distillation process, and then recover the oily distillate in the receiver flask. Then extract this oily distillate with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride, and after the extraction, combine both methylene chloride portions (if not already done so). Note: after each extraction, the methylene chloride will be the bottom layer each time. After the extraction, the upper water layer can be discarded. Now, extract the combined methylene chloride portion with six 50-milliliter portions (six 1.7 fluid oz. portions) of a 5% potassium hydroxide solution. After the extraction, combine all aqueous alkaline portions (if not already done so), and then briefly wash this combined aqueous alkaline portion with one portion of 50 milliliters (1.7 fluid oz.) of methylene chloride. Note: after the extraction and washing, the aqueous alkaline portion will be the upper layer each time. After the extraction and washing, the methylene chloride can be recycled if desired. Then place this combined aqueous alkaline portion into a large beaker, and then carefully add in, slowly, 250 to 300 milliliters (8.5 to 10.1 fluid oz.) of a 5% hydrochloric solution. Note: more or less acid may or

may not be needed, and the acid is added solely to bring the pH of the aqueous mixture (in the beaker) to about 1—add as much acid as needed to reach a pH of about 1. After adding the acid, moderately stir the entire acidic mixture for about 30 minutes. Then, extract this entire acidic mixture with four 40-milliliter portions (four 1.4 fluid oz. portions) of methylene chloride. After the extraction process, combine all methylene chloride portions (if not already done so), and then wash this combined methylene chloride portion with one 50-milliliter portion (1.7 fluid oz.) of water, followed by one 50 milliliter portion (1.7 fluid oz.) of a 23% sodium chloride solution. Note: after the extraction and washings, the methylene chloride will be the lower layer each time. After the extraction and washing portions, dry the washed methylene chloride portion by adding to it, 15 grams (0.52 oz.) of anhydrous sodium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the sodium sulfate. Finally, place this filtered dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, remove the left over remaining pale yellow oil (after it has cooled), and then store it in an amber glass bottle in a refrigerator until use. Note: the eugenol at this point will have a purity of about 98%.

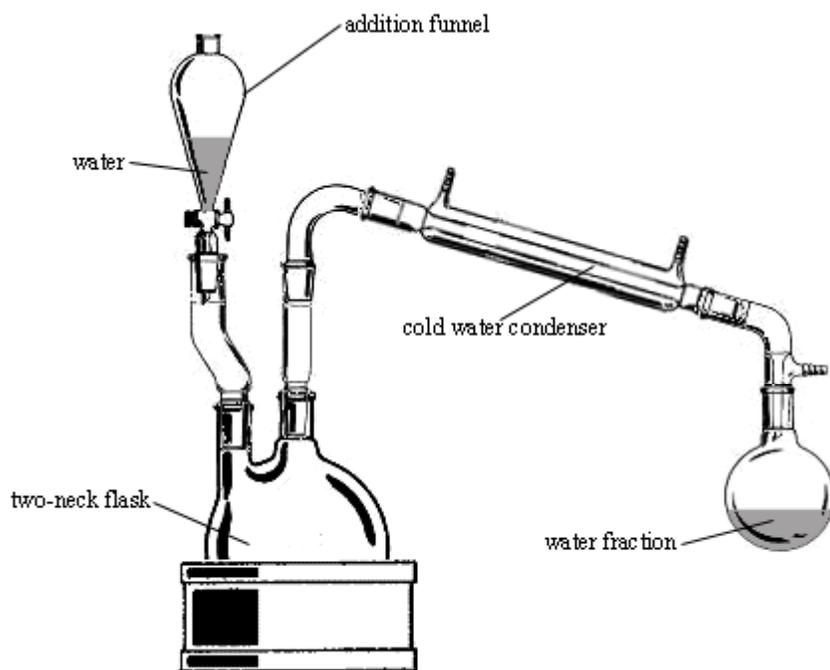


Figure 011. Standard steam distillation apparatus. The addition funnel should be filled with water at all times.

Extraction process 4: Extraction of Myristicin from nutmeg or nutmeg butter

Myristicin (6-allyl-4-methoxy-1,3-benzodioxole)

Myristicin forms a colorless to yellowish oil (depending on purity), with a boiling point (at 40 milliliters of mercury) of 173 Celsius. Myristicin exists naturally in nutmeg, carrots, and parsley, from which it can be extracted—especially from the corresponding oils.

Method 1: Extraction of myristicin from store bought nutmeg

Materials:

| | |
|---|---|
| 1. 100 grams (3.5 oz.) of powdered nutmeg (regular store bought nutmeg) | 4. 10 grams (0.35 oz.) of anhydrous magnesium sulfate |
| 2. 750 milliliters (25.3 fluid oz.) of water | 5. 200 milliliters (6.8 fluid oz.) of boiling 95% ethyl alcohol |
| 3. Three 75-milliliter portions (three 2.5 fluid oz. portions) of pre-heated methylene chloride | 6. 5 grams (0.17 oz.) of anhydrous sodium sulfate |

Hazards: Ethyl alcohol is flammable, so extinguish all flames before using.

Procedure: Into the steam distillation apparatus as illustrated in the following drawing, place 100 grams (3.5 oz.) of powdered nutmeg (regular store bought nutmeg), followed by 750 milliliters (25.3 fluid oz.) of water. Thereafter, steam distill this mixture at 100 Celsius for about 4 to 6 hours. Note: the exact steam distillation process may vary, and should be continued until no more oily resinous material is seen collecting in the receiver flask. When no more oily resinous material is seen collecting in the receiver flask, stop the steam distillation process, and then recover the entire oily resinous aqueous mixture from the receiver flask, and then place this mixture into a beaker, and then gently heat to about 50 Celsius for about 10 minutes. Then, before the oily water mixture cools to below 50 Celsius, place it into a separatory funnel, and then collect the upper oil layer. In some cases, the oil layer will be the bottom layer. Thereafter, extract this collected oil layer (before it cools to below 40 Celsius), with three 75-milliliter portions (three 2.5 fluid oz. portions) of pre-heated methylene chloride (pre-heated to about 40 Celsius), and after the extraction process, combine all warm methylene chloride portions (if not already done so), and then dry this combined warm methylene chloride portion by adding to it, 10 grams (0.35 oz.) of anhydrous magnesium sulfate. Note: after each extraction, the warm methylene chloride portion can be simply decanted-off rather than recovered by using a separatory funnel. After adding in the anhydrous magnesium sulfate, stir the entire combined warm methylene chloride portion for about 10 minutes, and then filter-off the magnesium sulfate. Note: if during the stirring process (with the magnesium sulfate), the combined methylene chloride portion cools to below 30 Celsius, gently warm the entire mixture to 40 Celsius. Then place this warm methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius. When no more methylene chloride passes over or is collected, stop the distillation process, and then recover the left over remaining oil (before it cools to below 40 Celsius). Immediately thereafter, dissolve this recovered warm oil into 200 milliliters (6.8 fluid oz.) of boiling 95% ethyl alcohol (pre-heated to about 78 Celsius), and then quickly stir the entire alcohol mixture for about 5 minutes, and then filter-off any insoluble impurities (if any). Note: filter the alcohol mixture while its still boiling hot. After the filtration process, allow the alcohol mixture to slightly cool to about 60 Celsius, and then place it into an ice bath, and chill it to about 0 Celsius for about 2 hours. Note: a freezer can be used by itself or in combination with the ice bath. After chilling the alcohol mixture for about 2 hours, filter-off the crystallized myristicin, and then quickly vacuum dry this myristicin product (before it warms to above 5 Celsius). Note: air drying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the myristicin to induce liquification, and then adding in 5 grams (0.17 oz.) of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.

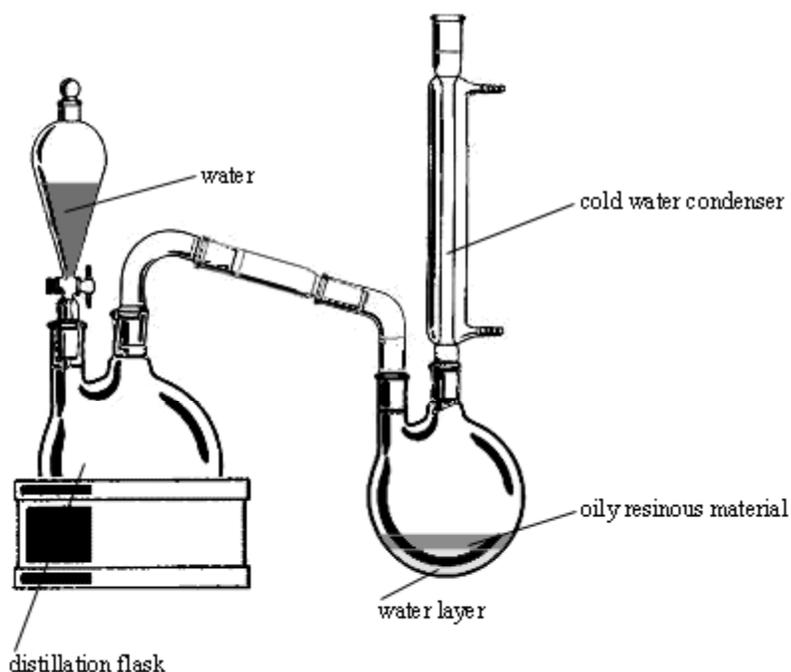


Figure 012. Setup for the steam distillation of nutmeg.

Method 2: Extraction of myristicin from nutmeg butter

Materials:

| | |
|--|--|
| 1. 50 grams (1.8 oz.) of commercially available nutmeg butter | 3. 100 milliliters (3.4 fluid oz.) of pre-heated diethyl ether |
| 2. 500 milliliters (17 fluid oz.) of boiling 95% ethyl alcohol | 4. 5 grams (0.17 oz.) of anhydrous sodium sulfate |

Hazards: Use care when handling diethyl ether, which is highly flammable, and capable of forming explosives mixtures with air—use proper ventilation and extinguish all flames before using.

Procedure: Nutmeg butter is a product that is obtained by pressing nutmeg between heated plates in the presence of a small amount of steam. Nutmeg butter is composed primarily of myristicin, glycerides of myristic acid and other fats, and residue. The myristicin portion can be obtained by treating the nutmeg butter with ether or alcohol. To isolate myristicin from nutmeg butter, thoroughly mix 50 grams (1.8 oz.) of commercially available nutmeg butter with 500 milliliters (17 fluid oz.) of boiling 95% ethyl alcohol. Note: make sure the 95% ethyl is boiling at 78 to 79 Celsius before adding in the nutmeg butter. While adding in the nutmeg butter, rapidly stir the boiling alcohol mixture, and after the addition of the nutmeg butter, place the entire alcohol mixture (including any and all insoluble solids) into a reflux apparatus (before the alcohol cools), and then reflux the entire mixture at about 79 Celsius for 2 hours. After 2 hours, quickly remove the reflux condenser, and replace it with a conventional condenser fitted with a receiver flask, and then distil-off the 95% ethyl alcohol until about 50% of the total volume remains (distill-off about 250 milliliters of the ethyl alcohol). When the alcohol mixture has been reduced to a total volume of about 50%, allow the alcohol concentrate to cool to about 60 Celsius, and then filter the entire alcohol mixture to remove any insoluble impurities. Note: this filtration process should be carried out before the alcohol mixture cools to below 60 Celsius. After the filtration process, place the entire filtered alcohol concentrate (even if two or more layers exist) into an ice bath, and chill it to about 0 Celsius. Note: a freezer can be used by itself or in combination with the ice bath. Then allow the alcohol concentrate to chill at 0 Celsius for about 2 hours. After 2 hours, filter-off the precipitated crystals of the myristicin (before the alcohol concentrate warms to above 5 Celsius), and then place these filtered-off crystals (before they have a chance to warm to above 10 Celsius) into a suitable beaker, and then add in 100 milliliters (3.4 fluid oz.) of pre-heated diethyl ether (pre-heated to about 40 Celsius). Thereafter, stir the entire warm ether mixture for about 30 minutes, and then filter-off any insoluble impurities (if any). Then, place this warm ether mixture into a distillation apparatus, and distil-off the ether only until about 25% of the total volume has been reduced (distill-off only about 25 milliliters of ether). When 75% of the total ether volume remains, stop the distillation process, and then place the ether concentrate into an ice bath (before it cools), and then chill it to about 0 Celsius for about 1 hour. Note: a freezer can be used instead of an ice bath or in combination with. After chilling this ether concentrate to about 0 Celsius for 1 hour, filter the ether mixture to recover the crystallized myristicin (before it warms to above 5 Celsius), and then vacuum dry these filtered-off crystals of the myristicin (before they warm to above 5 Celsius). Note: air drying will not work, and if desired, the myristicin can be dried by gently heating the crystals of the myristicin to induce liquefaction, and then adding in 5 grams (0.17 oz.) of anhydrous sodium sulfate (to absorb any moisture). After adding in the sodium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the sodium sulfate. The oil should then be stored in an amber glass bottle until use. Note: there are numerous modifications to this process, and those with experience should attempt any modifications they see fit.

Extraction process 5: Extraction of Caffeine from tealeaves

Caffeine

Caffeine forms white hexagonal crystals by sublimation. Caffeine has a melting point of 238 Celsius, but the crystals begin to sublime when heated to 178 Celsius. Caffeine is only moderately soluble in water, but more soluble in hot water. The crystals are also moderately soluble in alcohol, acetone, but are much more soluble in methylene chloride, chloroform, and practically insoluble in ether. Caffeine is capable of forming a hydrate, which loses its water of hydration when heated to 80 Celsius. Caffeine is a widely used stimulant, ingested by millions in the form of coffee, tea, etc.,

Method 1: Extraction of caffeine from tea leaves

Materials:

| | |
|---|--|
| 1. 825 milliliters (28 fluid oz.) of water | 5. Four 90-milliliter portions (four 3 fluid oz. portions) of methylene chloride |
| 2. 60 grams (2.1 oz.) of sodium carbonate | 6. 15 grams (0.52 oz.) of anhydrous sodium sulfate |
| 3. 30 to 40 tea bags (any brand of tea can be used) | 7. 21 milliliters (0.71 fluid oz.) of toluene |
| 4. 90 milliliters (3 fluid oz.) of methylene chloride | 8. 30 milliliters (1 fluid oz.) of hexane |

Hazards: Use proper ventilation when using toluene and hexane, and avoid inhalation of the fumes.

Procedure: Into a suitable beaker or flask, place 825 milliliters (28 fluid oz.) of water, and then add and dissolve 60 grams (2.1 oz.) of sodium carbonate. Thereafter, boil the mixture, and once the water begins to boil, add in 30 to 40 tea bags (any brand of tea can be

used). Thereafter, boil the mixture and allow the tea bags to soak for 15 minutes in the usual manner. After 15 minutes, remove the heat source, and allow the tea mixture to cool to about 50 Celsius. Thereafter, remove the tea bags, and then allow the tea mixture to cool to room temperature. Thereafter, add in 90 milliliters (3 fluid oz.) of methylene chloride, and then stir the mixture gently for about 30 to 40 minutes. Note: do not shake the mixture vigorously as an emulsion will form. After stirring the mixture for about 30 to 40 minutes, gently pour the mixture into a separatory funnel, and then remove the lower organic solvent layer. Thereafter, place this lower organic layer aside temporarily, and then repeat the extraction process with four 90-milliliter portions (four 3 fluid oz. portions) of methylene chloride upon the upper water layer. After extracting the upper water layer four more times, combine all lower methylene chloride portions, if not already done so, and then dry the combined methylene chloride portions by adding in 15 grams (0.52 oz.) of anhydrous sodium sulfate. Then stir the mixture briefly, and then filter-off the sodium sulfate. Now, place the dried methylene chloride portion into a distillation apparatus, and distill-off the methylene chloride until a dry residue remains. When this point is achieved, remove the heat source, and then collect the dry residue. Finally, recrystallize this dry residue from a toluene/hexane solvent mixture prepared by adding and dissolving 21 milliliters (0.71 fluid oz.) of toluene to 30 milliliters (1 fluid oz.) of hexane, and after the recrystallization process, vacuum dry or air dry the collected caffeine crystals. These crystals can be sublimed using a standard sublimation setup (see iodine) to afford highly pure crystals of 99% purity.

Extraction process 6: Extraction of Apiole from parsley (advanced process)

Apiole

Apiole forms crystals with a melting point of 30 Celsius. Fresh apiole may be a semi-solid liquid. The compound can be distilled at 294 Celsius. Apiole is soluble in alcohol, benzene and chloroform, but insoluble in water. Apiole is a major constitute of parsley, and is responsible for the aroma and taste of parsley.

Method 1: Extraction of Apiole from parsley seeds

Materials:

| | |
|--|--|
| 1. 1 kilogram (2.2 pounds) of parsley seeds | 5. 50 milliliters (2 fluid oz.) of warm water |
| 2. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol | 6. 10 grams (0.35 oz.) of lead-II-oxide |
| 3. with three 50-milliliter portions (three 2 fluid oz. portions) of diethyl ether | 7. 50 milliliters (2 fluid oz.) of additional warm water |
| 4. 10 grams (0.35 oz.) of anhydrous sodium sulfate | 8. 10 grams (0.35 oz.) of anhydrous sodium sulfate |

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air—use caution.

Procedure: Grind up 1 kilogram (2.2 pounds) of parsley seeds until the seeds are of a finely ground nature. Then place the finely ground seeds into a large reflux apparatus (equipped with motorized stirrer or other stirring means), and then add in 500 milliliters (17 fluid oz.) of 95% ethyl alcohol. Thereafter, reflux the entire mixture at 78 Celsius for about 6 to 8 hours while moderately stirring the ethyl alcohol mixture. After refluxing for about 6 to 8 hours, remove the heat source, and then allow the entire alcohol mixture to cool to room temperature. Thereafter, filter the entire alcohol mixture to remove any insoluble materials, and then place this ethyl alcohol mixture into a distillation apparatus, and distill-off the ethyl alcohol until about 50% of its total volume has been reduced (about 250 milliliters of ethyl alcohol removed). Note: the recovered ethyl alcohol can be recycled if desired. When about 50% of the ethyl alcohol mixture has been removed, stop the distillation process, and then place the ethyl alcohol mixture into a suitable sized beaker (before it cools), and then allow it to cool to room temperature. Then, quickly filter this alcohol concentrated mixture to remove any potential insoluble impurities (if any). Now, extract this entire alcohol mixture with three 50-milliliter portions (three 2 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion by adding to it, 10 grams (0.35 oz.) of anhydrous sodium sulfate. Note: after each extraction, the ether will be the upper layer each time. After adding in the sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Then, place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (when it cools to about 40 Celsius). Then place this warm collected left over oily residue into a clean beaker, and then add in 50 milliliters (2 fluid oz.) of warm water, followed by 50 grams (1.8 oz.) of sodium carbonate, and then followed by 10 grams (0.35 oz.) of lead-II-oxide. Thereafter, rapidly blend this entire mixture for about 1 hour at a temperature of about 40 Celsius—a hot plate will be needed in order to keep the temperature of the mixture at about 4 Celsius. After rapidly stirring for about 1 hour, add in 50 milliliters (2 fluid oz.) of additional warm water, and then continue to stir the entire mixture at about 40 Celsius for an additional hour. Thereafter, filter the

entire mixture through a layer of charcoal (place a bed of charcoal over the filter paper), before the mixture's temperature drops below 40 Celsius. After the filtration process, place the entire filtered mixture into a clean beaker. Now, extract this entire mixture with three 50-milliliter portions of diethyl ether, and after the extraction process, combine all ether portions (if not already done so), and then dry this combined ether portion, by adding to it, 10 grams (0.35 oz.) of anhydrous sodium sulfate. Note: after each ether extraction, the ether will be the upper layer each time. After adding in the anhydrous sodium sulfate, stir the entire ether mixture for about 10 minutes, and then filter-off the sodium sulfate. Finally, place this entire ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining oily residue (before it cools to below 40 Celsius). Then place this warm collected left over residue (composed primarily of the desired apiole) into an amber glass bottle, and then store it in a refrigerator until use. Note: There are numerous medications to this process, and those who are willing, should carry out any modifications that would seem fit.

Method 2: Extraction of Apiole from oil of parsley

Materials:

| | |
|---|---|
| 1. 150 grams (5.3 oz.) of commercially available "Oil of Parsley" | 2. 150 milliliters (5.1 fluid oz.) of ether |
|---|---|

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air—use caution.

Procedure:

This procedure is an advance procedure for the extraction of apiole from oil of parsley. This process utilizes vacuum distillation.

Set-up the vacuum fractional distillation apparatus as illustrated below, and then fractionally distil 150 grams (5.3 oz.) of commercially available "Oil of Parsley", at 167 Celsius under a vacuum of 27 millimeters of mercury. After the vacuum distillation process, remove the ice trap from the receiver flask, and then allow the receiver flask to warm to room temperature. Thereafter, gently warm the receiver flask using a small Bunsen flame or other means, and allow the crystallized apiole to liquefy into an oil. Then pour this oil into an amber glass bottle, and then store it in a refrigerator until use. Note: in some cases, the apiole can be obtained by allowing the commercial oil of parsley to stand in an ice bath for several hours (a freezer can be used as well). During the chilling process, crystals of apiole will slowly form. These crystals can then be filtered-off, and then dissolved into 150 milliliters (5.1 fluid oz.) of ether. The ether mixture should then be briefly stirred for about 30 minutes, and then filtered to remove any potential insoluble impurities (if any). Then place this filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When all the ether has been removed, place the left over remaining oily residue of the apiole (after it has cooled) into a amber glass bottle and store in a refrigerator until use. Note: There are numerous modifications to this process, and those who are willing, should carry out any modifications that would seem fit.

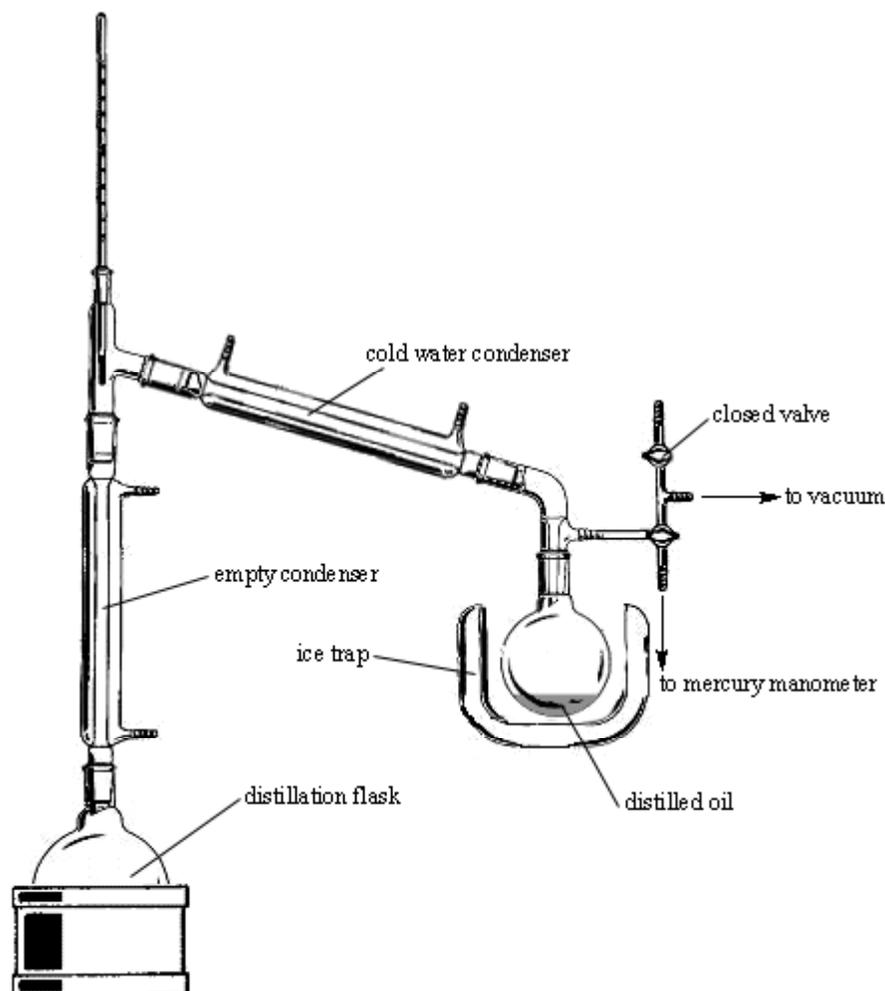


Figure 013. Advanced setup for vacuum fractional distillation for the isolation of apiole from commercial oil of parsley.

Extraction process 7: Extraction of Safrole (advanced process 2)

Safrole

Safrole forms a colorless to slightly yellow liquid with the odor of sassafras. The oil is insoluble in water, but very soluble in alcohol, and miscible with chloroform and ether. The oil has a boiling point of 232 Celsius, but can be distilled under high vacuum at 100 Celsius under 11 millimeters of mercury. Safrole is the main component of sassafras oil, from which it makes up 70 to 75% of the oil by weight. Safrole also exists in *Ocotea cymbarum* oil (Brazilian oil of sassafras), which it exists up to 90% by weight. The oil of massoria bark and *Cinnamomum massoia* contains about 14% safrole. Safrole can be extracted from sassafras oil by the means described later, and it can be extracted from Massoria bark oil and *Cinnamomum massoia* by washing the corresponding oil with sodium hydroxide solution to remove the phenols, and then vacuum distilling to obtain the safrole boiling at about 100 Celsius under a vacuum of 11 millimeters of mercury, or by carefully fractionally distilling (two path distillation) the phenol free oil at 228 to 235 Celsius. Safrole can also be made synthetically from rather inexpensive reagents (see safrole). Sassafras oil can be obtained by steam distilling the ground up roots of the sassafras tree, which grows in the mid western United States. Other sassafras species of trees elsewhere in the world can also be used to obtain the safrole by steam distillation from the root. To identify a sassafras tree, consult a book that discusses the various types of trees and plants. The dried root bark of the sassafras tree contains about 10% safrole by weight, and the remainder of the root contains only about 1%. The dried root bark can be obtained from numerous sources, including herb stores, health food suppliers, and botanical suppliers. Sassafras oil can also be obtained from these aforementioned sources; if however your local suppliers do not offer the sale of sassafras oil, request them to order some for you, which they should have no problem doing. Safrole is also used in perfumes, so check out the types of perfumes, and their ingredients. Note: checkout your local

aromatherapy suppliers, as they are major consumers of oils, one of which may be sassafras oil. Sassafras oil may be used in adulterants in massage oils for use in aromatherapy. Ocotea cymbarum oil is obtained by steam distillation of the wood of the Ocotea pretiosa tree, which grows in South America. The wood contains about 1% oil by weight, which is easily collected by steam distillation of the wood chips, and the resulting steam distilled product contains about 90% safrole by weight. Distributors of perfume and flavoring compounds may contain this Ocotea cymbarum oil. Check the OPD directory for essential oils and botanical companies; also checkout small herb shops nationwide.

Method 1: Extraction of Safrole from sassafras oil

Materials:

| | |
|---|--|
| 1. Sassafras oil or any safrole containing oil, plant, root, ect. | |
|---|--|

Hazards: None.

Procedure: Note: as previously mentioned, sassafras oil can be obtained from the root bark of the sassafras tree. To do this, setup a standard steam distillation apparatus, and then steam distill the root bark (grind the root bark into pieces before use). The oil and water collect in the receiver flask, where upon the oil can be seen as droplets. The oil is denser then water so it will form droplets below the water. After the steam distillation process, the oil can be collected by placing the water/oil mixture into a seperatory funnel, and then recovering the lower oil layer. The collected oil layer should then be dried by mixing with it, a small amount of anhydrous calcium chloride. After filtering-off the calcium chloride, place the oil into a vacuum distillation apparatus, and vacuum distill at 100 Celsius under a vacuum of 11 millimeters of mercury (see figure below). Note: other oils containing safrole can be vacuum distilled in a similar manner. Note: because of the expense involved in purchasing vacuum distillation apparatus, try freezing the sassafras oil, or other oils that contain the safrole. Safrole has a melting point of 11 Celsius, and it may be possible to crystallize the safrole out of any oil solution by using ice baths, cold-water baths, or even a freezer. Experiment with various techniques; solvent extractions may also work.

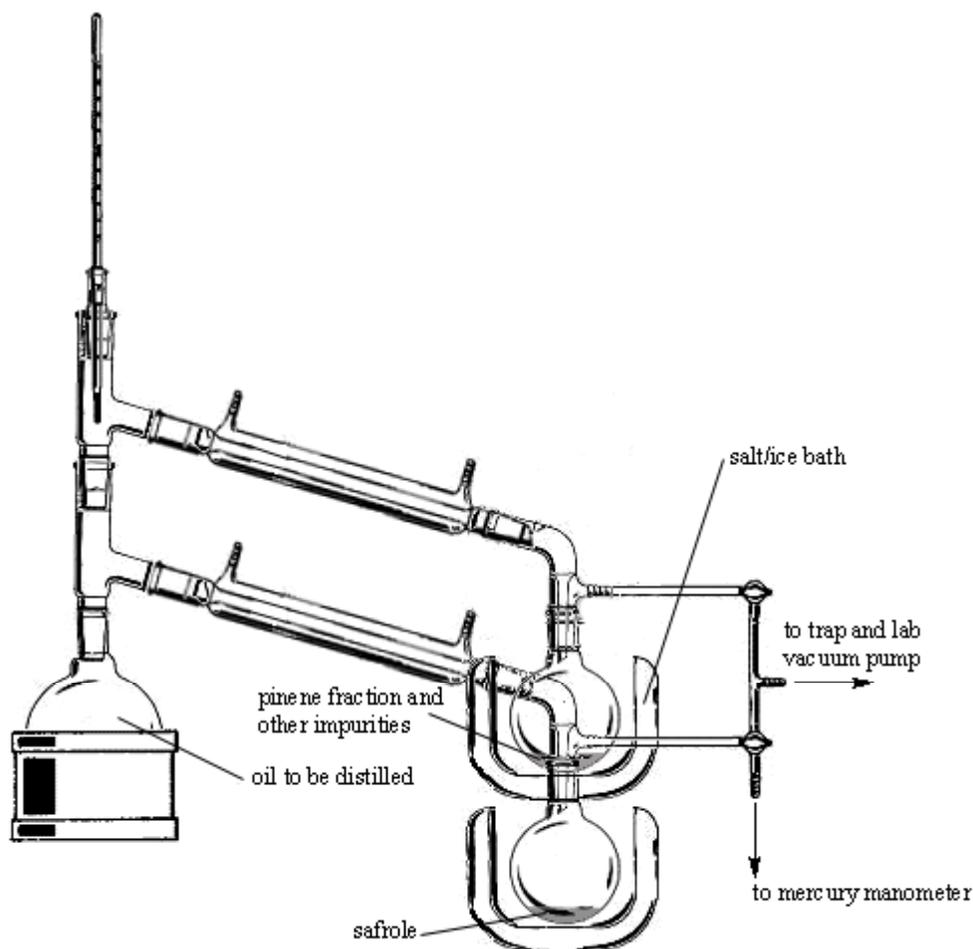


Figure 014. Advanced setup, two-path vacuum distillation apparatus for collecting safrole Note: in some cases, the safrole may be the upper fraction, depending on density, and impurities.

D. Recrystallization, product recovery, and filtration

Recrystallization is a very important tool for purifying solids. In recrystallization, solubility differences allow solids to be separated from each other and recovered from the solvent. In the recrystallization process, molecules slowly deposit from solution and attach to each other to form crystals. As the aggregates of crystals grow large enough, they precipitate. After precipitation, the solids can be recovered by filtering them off.

Choosing the appropriate solvent is the most crucial aspect of the recrystallization process. The best solvent for recrystallization is one in which the material is less soluble at room temperature but more soluble when hot. At higher temperatures, solvents that form super saturated solutions with certain solids meet this requirement.

Solvent choice is also governed by another important factor, the ease of solvent removal. Solvents with low boiling points are preferred because their removal is easy. A third consideration in selecting a solvent is the temperature at which the solvent solidifies. Benzene was once widely used in recrystallization, but when placed in an ice bath, crystals of benzene would also precipitate (benzene crystallizes at 6 Celsius). A final consideration in choosing a solvent is reactivity. Obviously a solvent that reacts with a solid cannot be used for recrystallization.

Recrystallization depends on super saturation. Super saturated solutions are formed when mixtures containing the dissolved solid and the solvent (or solvent mixture) are heated, or evaporated. When the mixture is heated, the solvent begins to evaporate, as the evaporation proceeds, the concentration of the dissolved solid(s) begins to increase. During this evaporation, the solids become *over dissolved* leading to the super saturated solution. When tiny crystals start forming on the surface of the mixture during heating, super saturation has been reached. When the supersaturated solution is cooled, recrystallization begins and some of the dissolved solid precipitates as crystals. Not all the solid will precipitate out on cooling. After the supersaturated solution has cooled for some time, equilibrium sets in restoring the original solubility of the mixture. The only difference is that some of the solvent and solid have been removed. The precipitated crystals are then collected by filtering the mixture. To recover more solid, the mixture must be re-heated and allowed to evaporate to the point of super saturation again.

I. The recrystallization process

The recrystallization process is simple. Boil the mixture that contains the dissolved product and the solvent (or solvent mixture). The mixture can be placed into a distillation apparatus and distilled at the boiling point of the solvent to collect the solvent. Using a distillation apparatus is preferred rather than just boiling-off the solvent, which would be a waste of solvent. Boil off the solvent until super saturation is achieved. When tiny crystals begin to form on the surface of the mixture, super saturation has been achieved. Then remove the heat source (turn off the heat source) and allow the mixture to cool to room temperature. Afterwards, place the mixture into a cold-water bath or ice bath for thirty minutes. After which, remove the cooling bath, and then filter-off any precipitated product. Then place the filtered mixture back into the same distillation apparatus, and re-distill again until a super saturated solution is achieved. When super saturation is achieved, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, place the mixture into a cold-water bath or ice bath for thirty minutes. Then remove the cooling bath, and then filter-off any precipitated product. Then place the filtered mixture back into the same distillation apparatus, and distill until a super saturated solution is achieved. When it is achieved, remove the heat source, and allow the mixture to cool to room temperature. After which, place the mixture into a cold-water bath or ice bath for thirty minutes. Then filter-off any precipitated product. At this point much of the solvent has been removed by distillation, and much of the product has been recovered. The remaining mixture is called the mother liquor, and can be recycled to a future recrystallization of the same product (using the same solvent or solvent mixture). This process of boiling, cooling, and filtering should be repeated as many times as necessary. When recrystallizing a product from a solvent or solvent mixture, continue the process until 90% of the solvent has been removed. Depending on the solubility of the product, continue the recrystallization process until 75 to 98% of the solvent has been removed. After most of the product has been collected, it can then be washed. To wash a solid product, simply leave it in the filtering funnel, and then pass an inert solvent over it many times. Choose a solvent that does not dissolve the product. Water is usually used to wash organic solids.

Seed Crystals

In some cases recrystallization of super saturated solutions can be initiated with a seed crystal. A seed crystal is simply a small crystal of the product. It is added to the super saturated solution, and the dissolved product begins to grow on the seed crystal. The seed crystal induces recrystallization by giving the dissolved product a surface from which to grow on. The recrystallization of the product stops when equilibrium of the solution is reached.

Recovering the product through low heat or no heat evaporation

In most modern labs, the recrystallization process is passed over by a rotary evaporator. A rotary evaporator, as pictured earlier in this section, is the most common method of recovering dissolved product. To use, the reaction mixture is placed there into, and then a vacuum is applied. The flask containing the reaction mixture is partly submerged in a water bath, and the necessary amount of heat is applied. Because liquids have decreased boiling points with decreasing pressure, solvents can be removed at much lower temperatures thanks to the vacuum. This process is similar to vacuum distillation. The great thing about rotary evaporators is their ability to run for hours on end without having to interact, monitor, or take part in the process. Simply insert the reaction mixture, apply the necessary heat, attach the vacuum, and let the machine do the rest of the work. Rotary evaporators sell for about \$3,000 to \$10,000 a piece. The oldest method of product recovery is placing the reaction mixture into a crystallizing dish, or shallow pan, and then allowing the solvent(s) to air evaporate. This method is a good idea for crystallizing stable, light insensitive products, where good crystal size is desired; for example, allowing a solution containing sodium chlorate to air-evaporate, large brilliant crystals of the chlorate are obtained. If this same solution was recrystallized, or evaporated under vacuum, usually small crystals of the chlorate are obtained. The problem with air-evaporation is the amount of time required, especially if the product is hygroscopic. In some examples, air-evaporation is impossible. Examples include zinc chloride, lithium perchlorate, and calcium chloride. These substances are so hygroscopic that placing the dry crystals into a beaker will produce a self-induced aqueous solution on standing after several days or weeks due to moisture absorption from the air. In warm dry climates, such as dessert climates, air-evaporation has its advantages. Good crystal size can be rapidly achieved by allowing reaction mixtures to air-evaporate in the sunlight.

II. Filtration

Filtration and recrystallization run hand in hand. When a product precipitates, it must be collected. Filtration is the most common method of collecting precipitated products. The two methods of filtration include gravity, and vacuum. Vacuum filtration is the most common method of filtering in the lab, and it is also the fastest.

1) Gravity filtration

Gravity filtration is the oldest and slowest method of filtering. In most regards gravity filtration should be avoided due to the slow nature. In many examples gravity filtration can take hours, and even days. Even so, gravity filtration is useful for removing charcoal, which is difficult to remove from mixtures when using vacuum filtration. Gravity filtration is also the cheapest method of filtration, and plastic funnels and coffee filters are cheap and readily available from many stores.

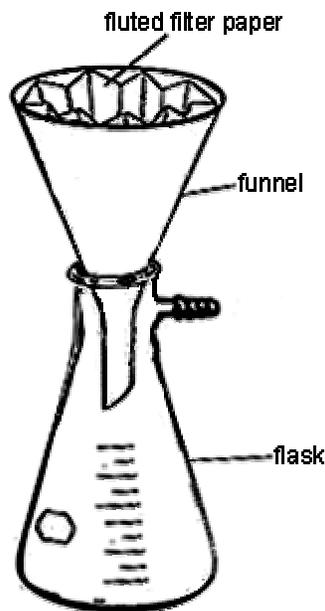


Figure 015. Apparatus for gravity filtration.

Gravity filtration is sometimes used to remove impurities rather than to collect a precipitated product. In this case the filtration takes place with the use of a filter aid. A filter aid is an insoluble substance used to absorb impurities. Some examples of filter aids include Celite, silicon dioxide, sand, zeolites, and even pebbles. Celite is a diatomaceous earth material that is most commonly used. Although filter aids can be used to speed up the filtration of finely divided precipitates, which otherwise get stuck in the tiny holes of the filter paper.

2) Fluting Filter Paper

Laboratory filter paper, and store bought coffee filters are usually round or cup shaped, so fluting the paper is necessary. Fluted filter paper is superior to flat filter paper because fluted filter paper allows for better airflow between the funnel wall and the fluted filter paper.

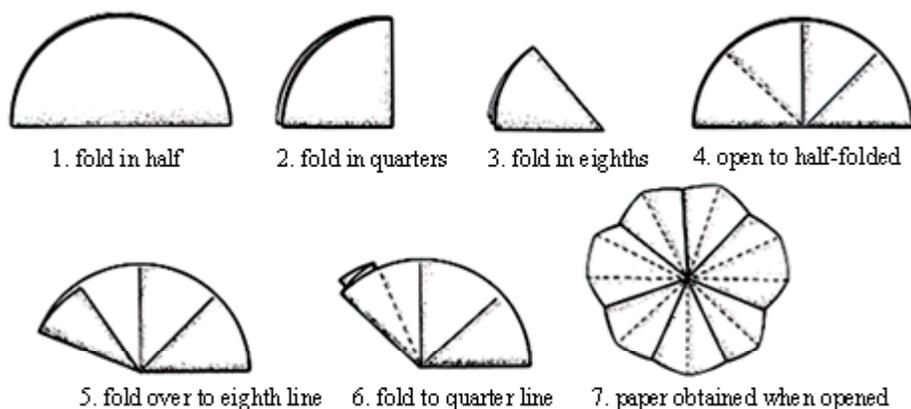


Figure 016. Fluting filter paper.

3) Vacuum Filtration

Vacuum filtration is definitely the method of choice for filtration, and it is the most common method. Vacuum filtration is superior in that suction is used to force the liquid through the filter paper allowing for rapid filtration. Precipitates can be recovered quickly and easily. After the precipitate has been recovered, it can then be vacuum dried. Vacuum drying is simply allowing suction to continue after the liquid has been removed. The suction creates an excellent airflow, which dries the collected precipitate as it flows. Ten to twenty minutes is adequate time to dry any product.

When first starting the filtration process, the vacuum will suck some of the product into the flask. The contents of the flask should then be re-filtered to ensure no product loss. The suction force is generated by a vacuum pump, which is commercially available in many styles and sizes; hand driven pumps can be used as well. Note: The suction force should not be too great. Placing your hand completely over the funnel until the suction grips your hand moderately indicates the proper suction. Never underestimate the power of a vacuum.

A Buchner funnel is used in vacuum filtration, and is a glass or plastic funnel. Plastic Buchner funnels are composed of two pieces. The funnel cup makes up the top piece, and the stem makes up the bottom piece. Glass Buchner funnels are composed of one or two pieces, and some come with glass joints. To use the funnel simply attach it to the filtering flask (see illustration below), and then place a piece of round filter paper into the bottom of the funnel. The filter paper is simply held in place by gravity and the suction force. Before filtration begins, lightly moisten the filter paper with water or fresh solvent (the solvent used should be the same as in the mixture being filtered, or an inert solvent that does not dissolve the precipitate). Once the precipitate has been filtered and dried, simply remove the suction source and then casually remove the filter paper from the Buchner funnel. Then gently scrape off the product from the filter paper. Vacuum pumps can be either hand pumps (like a ball pump), or a fancy lab pump. In any case, vacuum pumps are readily available from online auction sites and many online stores for reasonable amounts of money.

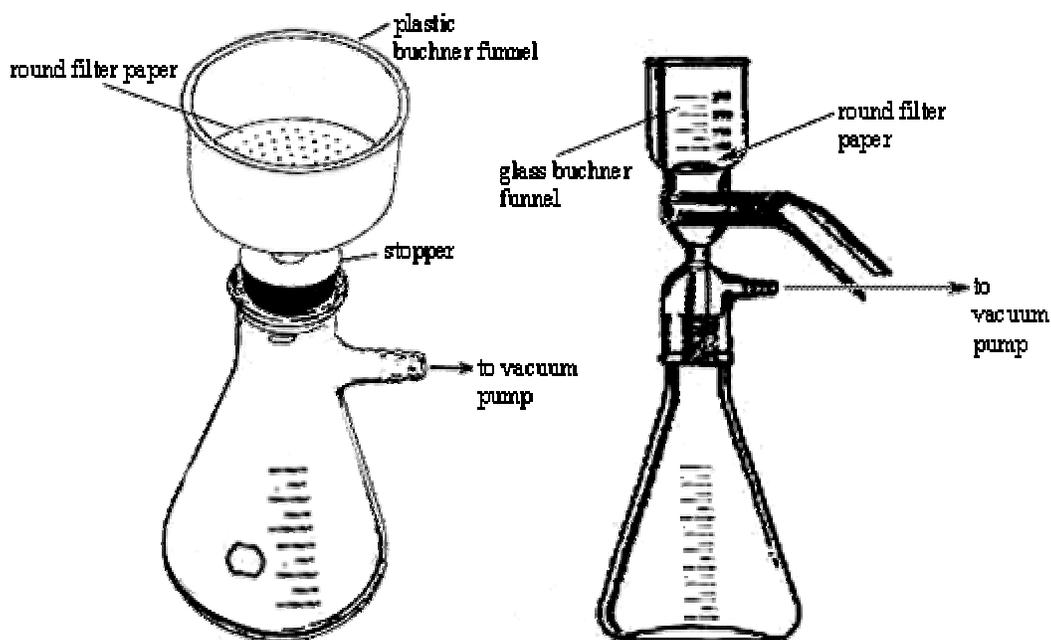


Figure 017. Left: Plastic Buchner funnel. Right: All glass set-up.

E. Washing and drying liquids and solids

I. Washing liquids and solids

Solids are easily washed by passing water, or the desired solvent over the solid product, which is contained in the filter funnel. For washing solids in this way, vacuum filtration should be used. Washing solids in this way using gravity filtration is a long and time consuming process. Obviously, do not wash the filtered-off solid with any liquid that reacts with, or dissolves the solid product.

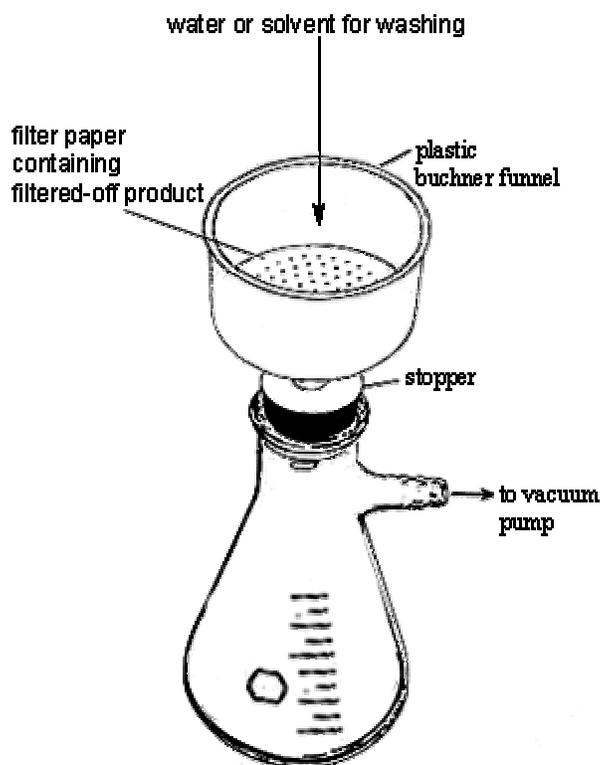


Figure 018. Washing a solid product. If using gravity filtration, this style of washing can take much longer.

Another method of washing a solid product is to place it into a beaker or other suitable container, and then add an excess of water or solvent, stirring the mixture for several minutes, and then allowing the mixture to stand long enough for the solid product to settle. After the solid product settles, much of the water above the settled solid product can be removed by carefully tilting the beaker, and pouring it off. This method of washing is useful for washing large amounts of water-insoluble product.

Washing liquids is done in a similar manner as just described. For washing a liquid, usually with water, place the liquid into a beaker, and then add the desired amount of water. Thereafter, stir the mixture for several minutes, and then allow the mixture to stand. After the two-phase mixture has settled, remove the water layer either by using a separatory funnel, or by pouring it off.

II. Drying agents and drying liquids

Water is called the universal solvent, but in many cases its considered to be an impurity. After the extraction process, the combined solvent portions sometimes contain a small amount of water. This water is removed by treating the combined solvent portions with an inert drying agent. The drying agent simply absorbs the water. The most commonly used drying agents are listed below.

To dry liquids solvents, or reaction mixtures, simply add to it, a small amount of the specified drying agent. And then stir the entire mixture for about 10 minutes (or much longer if drying a reaction mixture)—thereafter, filter-off the insoluble drying agent. It should be noted that drying agents are insoluble in solvent in which they are to dry, and this is what is desired. Drying agents cannot be used to dry liquids in which they are soluble in.

1) Anhydrous sodium sulfate

Anhydrous sodium sulfate is the most common general-purpose drying agent. It is inexpensive and has a very large capacity of absorption because it can form a decahydrate. Anhydrous sodium sulfate is relatively inert, and it does not react with most organic compounds. Anhydrous sodium sulfate can be regenerated from used sodium sulfate by heating to 200 Celsius for 1 hour.

2) Anhydrous magnesium sulfate

Anhydrous magnesium sulfate is the second most commonly used drying agent. Similar to anhydrous sodium sulfate, it to has a high capacity for absorption, and low cost. Although unlike anhydrous sodium sulfate, it has a faster drying rate, but is more reactive. It can be regenerated in the same manner as anhydrous sodium sulfate.

3) Calcium chloride

Calcium chloride is very inexpensive, and is an excellent drying agent. Its very high capacity and rapid drying ability makes it the reagent of choice for drying hydrocarbons, chlorinated solvents, halogens, and ethers. Unfortunately, calcium chloride is much more reactive than either sodium or magnesium sulfate and thus cannot be used to dry amines, alcohols, acid gases, or ammonia. It can be regenerated in a similar manner as anhydrous sodium sulfate.

F. Distillation

Distillation is a very common method for purifying liquids. Atmospheric distillation (general distillation), vacuum distillation, and steam distillation are the three common methods of distillation. Atmospheric distillation takes place at atmospheric pressure, which means the distillation apparatus is open to the air. Vacuum distillation utilizes reduced pressure to distill a liquid at lower temperature. Vacuum distillation is commonly used to distill liquids, which tend to decompose at their atmospheric boiling points. Vacuum distillation is also used to conveniently distill liquids with relatively high boiling points at a much more efficient temperature. Steam distillation is similar to atmospheric distillation, but steam is used to promote volatility. Steam distillation only works on liquids or solids, which are volatile with steam.

Now it should be noted, that distillation involves the purchasing of some rather pricey equipment. For most people, this equipment is not desired, but not out of reach (as it is available on many online auction sites and stores for reasonable prices). However, purchasing laboratory glassware is a crucial investment, and it can last for a long time as long as you take care of it. If you do not have distillation equipment or the desire to purchase it, you can make some homemade designs of your own from simple and readily available materials. These designs may use pop cans as the distillation flasks, PVC pipes as the condensers or adapters, and tubing for other means. However you design your set-up, it should be similar to the illustrations shown in this manual. If desired, you can use metal stills obtainable at brewer supply stores, but these devices are not suitable for distilling acidic or basic mixtures.

For most laboratory condensers, cold water can be circulated through them by using submersible pumps, which can be purchased at hardware stores, or online stores for ponds, fountains, and man-made landscaping streams at reasonable prices. To do this, purchase a small submersible pump, and then rig it so a piece of ¼" O.D. latex tubing (or something similar) can be attached to the exit port of the pump (where the water jets out). The other end of this latex tubing can then be attached directly to your glass or metal condenser. The submersible pump can be submerged into a large plastic or metal container filled with cold water, and the water circulated over and over again. If the water gets warm during the distillation process, simply throw in some ice. In some cases, the connection of your latex tubing to the glass or metal condenser may not fit 100% snug, so small metal screw clamps can be used to secure the tubing to the condenser (simply use a flat head screw driver or other means to tighten the metal screw clamp, but don't tighten it to tight to the point where it cracks or breaks the side arm of the glass or metal condenser).

In some cases, condensers can be omitted from a distillation apparatus by placing the receiver flask directly into a cold water or ice bath, and then carrying out the distillation under the usual manner. In this case, the cold water or ice baths used to cool the receiver flask may have to be continuously replaced.

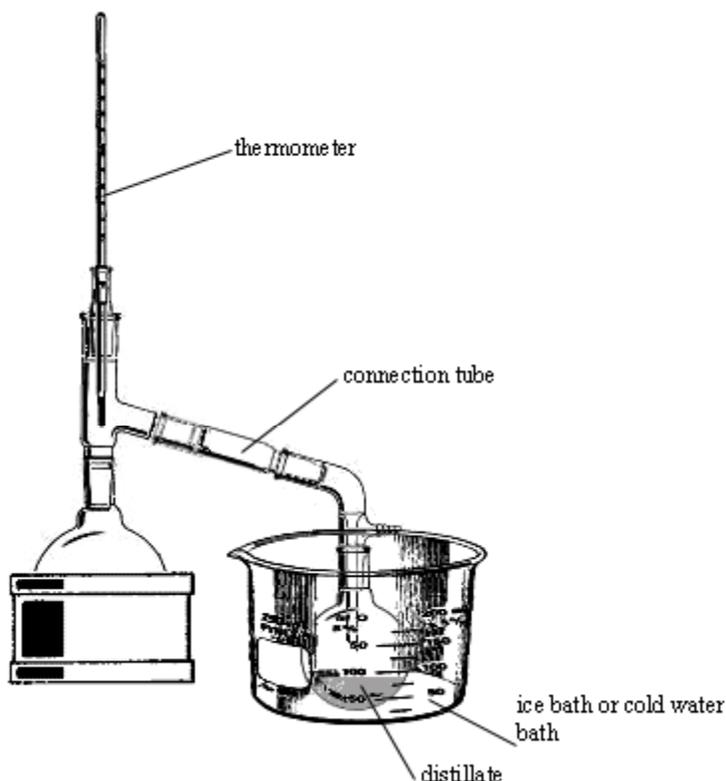


Figure 019. Set-up for short path distillation minus any condensers. The connection tubes can be replaced with latex tubing or equivalent tubing. As usual, the heating mantle can be replaced with a Bunsen burner, hot plate, steam bath, oil bath, stove top, ect., ect.,

1) Atmospheric Distillation (general distillation)

Atmospheric distillation is the most common of the three methods of distillation. The following illustration shows a common distillation apparatus. When liquids are heated they become volatile. The degree of volatility depends on the amount of heat applied to the liquid, the pressure, and the chemicals boiling point. When enough heat is applied to the liquid, the liquid begins to boil. When a liquid boils, intermolecular forces within the liquid break, and the molecules there after convert into the gas phase. During the distillation, this gas passes over into a condenser, where it is condensed back into a liquid by applying a cooler temperature to the gas. A condenser usually filled with circulating cold water acts as the cooling force. When the gas is cooled, it reforms back into a liquid, and then gravity pulls it into a receiver flask where it collects. A typical distillation produces 1 to 50 milliliters of liquid per minute. Most distillations take hours, depending upon the volume of liquid being distilled, and the concentration. Concentrated solutions distill much faster than dilute ones.

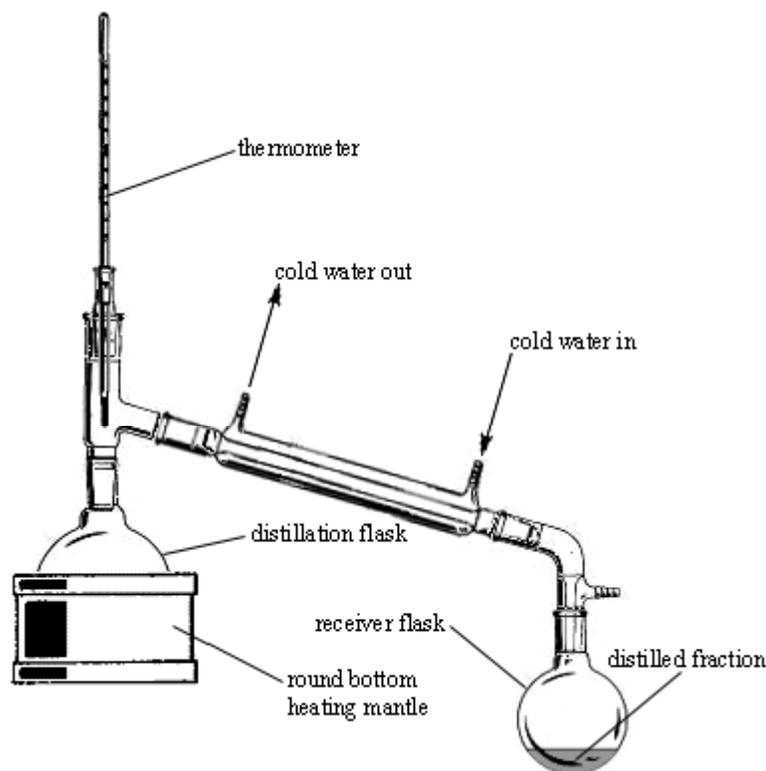


Figure 020. Standard atmospheric distillation apparatus—can be used for most distillation processes. The heating mantle can be replaced with a Bunsen burner, hot plate, steam bath, stove top, ect., ect.,

G. Lab safety

Lab safety is the first step in proper laboratory techniques. For each chemical procedure, read directions carefully, and know precisely what you need to do, before you actually do it. After reading the procedure think about the procedure, and know the hazards associated with it. Know the chemicals used in the procedure and how to properly handle them. Do not attempt to alter the procedure or change chemicals. The best safety is to prevent accidents before they happen.

Carryout all procedures involving volatile chemicals using proper ventilation. Fume hoods work in most cases, but not all. Even in well-ventilated fume hoods, volatile and/or noxious fumes can expand outward contaminating the entire lab. Vapors can travel long distances and cover large areas despite well ventilation.

Under any circumstance, eye protection should be used at all times. Eye protection should include eye goggles that completely seal the eyes; glasses are not proper eye protection. Nitrile gloves, and proper lab coats should be worn at all times.

For general handling of chemicals, including common solvents, reagents, and intermediates, the following checkpoints should be observed:

- 1) Always remember to wear safety goggles at all times. Clothing and equipment can be replaced, but your eyes can't. Contact lenses or glasses are not a substitute for safety goggles. If you get chemicals in your eyes (liquid, gas, or vapor) immediately flush with large amounts of water.
- 2) Immediately wash off any chemical you happen to spill on yourself. Most chemicals are dangerous only if they linger, so take action at once. Concentrated sulfuric acid is not very harmful if washed off immediately, and most acids do little or no skin damage if they are immediately washed off with water.
- 3) In case of an accident such as a fire, save yourself first. Keep fire extinguishers in arms reach, and have an adequate water source within reach. For acid spills, simple baking soda can be used to neutralize it.
- 4) Avoid open flames in a laboratory setting, and do not smoke in the lab. In the event of a fire, calmly but quickly move away from the burning area. Fight the fire only if you are confident the fire can be extinguished.
- 5) Do not eat or drink food products while in the lab. Food and drink can become contaminated by accident, and never use laboratory glassware for eating or drinking.
- 6) Never taste chemicals, and never smell chemicals by sticking your nose right up to the container. Smell chemicals by wafting the vapors with your hand to your nose. Many accidents have occurred when fingers were contaminated in the laboratory and then later used to rub eyes or for eating snacks. Remember to wear gloves at all times. Latex gloves work for most cases, but in some cases

nitrile gloves are recommended. Especially when handling strong acids, or chlorinated solvents. If bare handed, wash hands after touching chemicals and/or their storage bottles.

7) Breathing or handling small amounts of noxious substances does not pose immediate danger, but you should avoid contact with any potentially noxious chemical under all circumstances. Toxic chemicals should be handled with great care, and proper ventilation (fume hoods with maximum settings) should be used. If fume hoods are not available, the toxic chemicals should be handled in well-ventilated rooms with open windows to allow good airflow. Most organic solvents are very volatile and flammable, so proper ventilation should be exercised as well. Always remember, if you can smell a substance, you are breathing it into your lungs.

8) Wear inexpensive clothing when working in a lab. Since there is a possibility of clothing being destroyed in a laboratory accident, a lab coat or an apron should be worn at all times. Do not wear sandals or thong shoes when in the laboratory. Confine long hair and/or loose clothing while in the laboratory. Do not wear shorts, open skirts, blouses, or any other clothing that leaves large areas of skin unprotected.

9) On a final note, never play around with chemicals by mixing or heating them. Always remember, before you mix and/or heat chemicals know what you are doing. Playing around with chemicals can lead to poisonous fumes, fires, and/or explosions.

H. Laboratory equipment

Laboratory equipment is absolutely crucial in the preparation of chemicals and cannot be substituted by anything else. Many of the procedures in this book require some kind of laboratory glassware or equivalent, which can be quite costly. Laboratory glassware comes in many styles, shapes, and sizes from many suppliers. Using glassware only requires a few simple rules, which can go as follows:

1) Most laboratory glassware cannot be heated above 500 Celsius. Quartz glass, which is really expensive, is used in procedures where higher temperatures are needed (up to 1200 Celsius) along with the inertness of glass. Steel, nickel, porcelain, or iron crucibles are used for general heating of solids at high temperatures. General laboratory glassware is used for heating liquids because most liquids will never encounter temperatures exceeding 300 Celsius. 2) Never rapidly heat glass to a high temperature. Exposing glassware to high temperatures all at once can cause cracks and breakage. Cooling hot glassware too quickly can also lead to cracks and breaks.

Always allow the heated glass to cool to room temperature (by itself) before applying it to cold water baths, ice baths, or dry ice baths. Quartz is an exception. It can be heated to 1000 Celsius and then dipped into water. If you get your hands on any quartz glassware, snatch it up like gold and take good care of it. Quartz glassware can be used instead of ordinary laboratory glassware.

3) The following illustrations show some common laboratory glassware and equipment. Most modern glassware contains ground glass joints. Ground glass joints are outer (male) and inner (female) etched surfaces that stick together forming an airtight seal when pushed together. In some cases sealant grease (commonly called vacuum grease) is applied to the joints to allow for easy disconnection. When connecting adapters, do not push them together too hard. Pushing the joints together too hard may lead to a suction effect between the two adapters. This suction effect can make disconnection of the adapters by hand impossible. In some rare cases the joints can be suctioned together so severely that breakage of the adapters while trying to disconnect them results. If adapters become suctioned together, do not use force to separate them. To separate joints that are suctioned together, simply heat the joint directly with a blue flame from a Bunsen burner, being cautious as not to heat the joint too much—when the joint has been heated for a few quick times, simply pull apart the two adapters using a cloth to protect your hands from the hot glass.

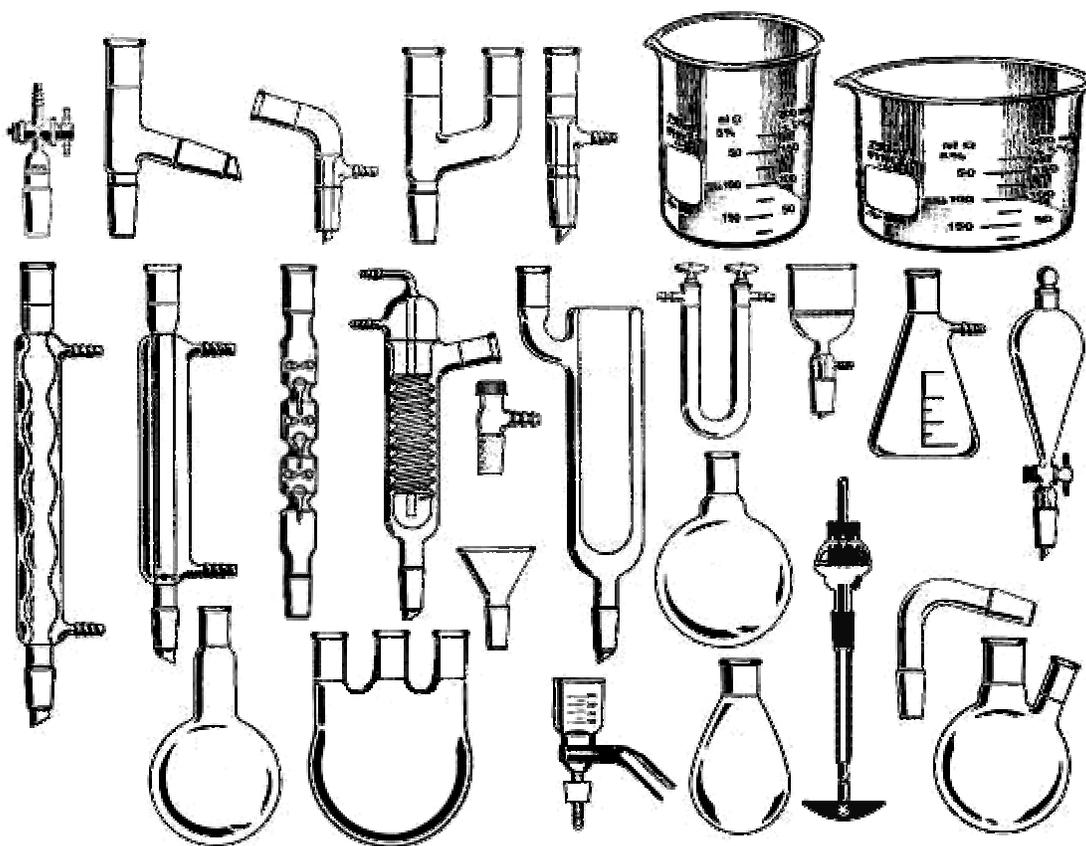


Figure 021. Common laboratory equipment.

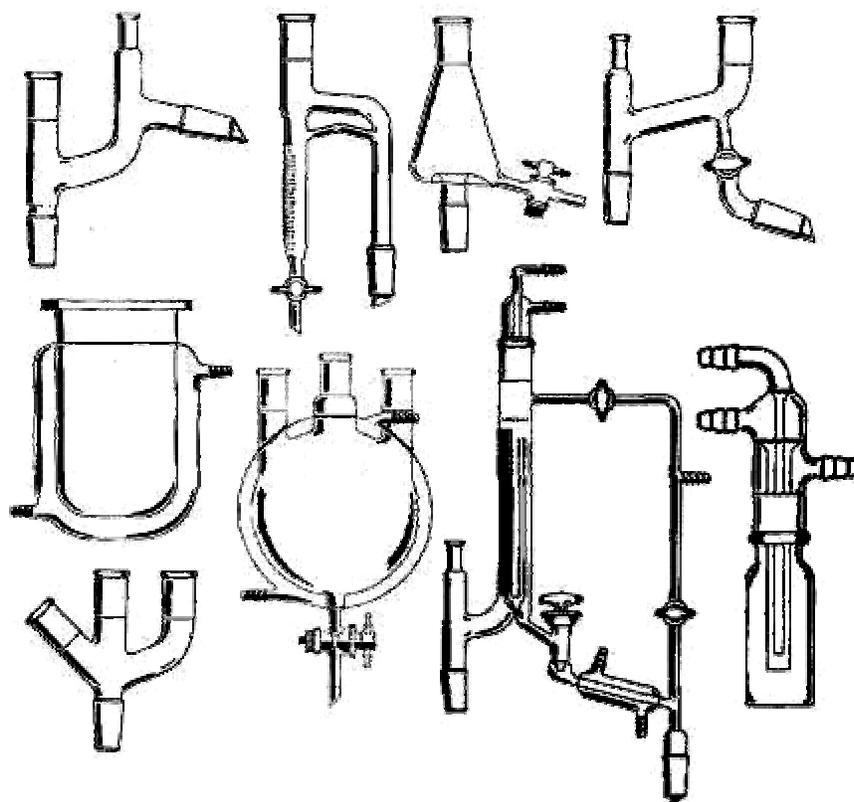


Figure 022. Common laboratory glassware.

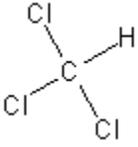
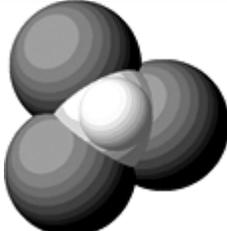
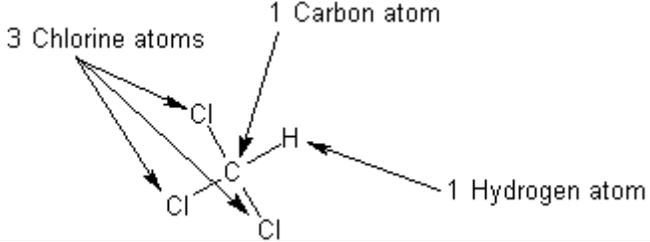
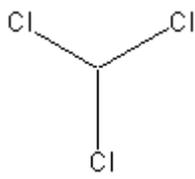
SECTION 3: General Lab Procedures

Part 3: The Preparation of General Lab Chemicals

General laboratory processes involve those chemical reactions where basic chemicals are being reacted, and produced. General lab processes are very simple and easy to setup, and they generally involve relatively safe and simple compounds. Most of these compounds can be easily isolated, purified, and stored.

Preparation 1: Chloroform

Also known as: Trichloromethane, formyl trichloride

| | |
|--|--|
|  |  |
| Chemical structure | 3D Structure |
|  |  |
| Structure make-up | Short hand chemical structure |

Chloroform is a highly refractive, nonflammable, heavy, very volatile, and sweet-tasting liquid with a peculiar odor. It has a boiling point of 62 Celsius, and a melting point of -64 Celsius. Chloroform forms a constant boiling mixture with alcohol containing 7% alcohol, and boiling at 59 Celsius. Commercial chloroform contains a very small amount of ethanol as stabilizer. It is insoluble in water, but miscible with alcohol, benzene, ether, petroleum ether, and carbon disulfide. Pure chloroform is light sensitive, so store in amber glass bottles in a cool place. Chloroform is a suspected light carcinogen, so use proper ventilation when handling. Over exposure to chloroform vapors causes dizziness, and headache. **Note:** Distilling mixtures containing chloroform mixed with one or more strong base (lithium, sodium, or potassium hydroxide) can result in explosion or violent reaction. Always neutralize any base, or extract the chloroform before distilling.

Method 1: Preparation of chloroform from acetone and bleaching powder

(By-products from reaction: Calcium acetate, calcium chloride, and calcium hydroxide)

Materials:

| | |
|--|---|
| 1. 100 milliliters of tap water (3.4 fluid oz.). | 3. 1 milliliter of 95% ethyl alcohol (see entry). |
| 2. 100 grams of acetone (3.5 oz.). Readily available in any hardware store. | 4. 300 milliliters (10.1 fluid oz.) of benzene, toluene, or xylene. Toluene and xylene should be available at most hardware stores. |
| 2. 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles. | 5. 15 grams of anhydrous magnesium sulfate (obtained by heating Epsom salt in an oven, microwave, or Bunsen burner). |

Summary: Chloroform is prepared by reacting acetone with calcium hypochlorite (bleaching powder), and then extracting the mixture with benzene, toluene, or xylene. After extraction, the solvent/chloroform mixture is then distilled to collect the chloroform, which is then re-distilled. After collecting the chloroform after re-distillation, it is mixed with a small amount of 95% ethanol to act as a stabilizing agent.

Hazards: Extinguish all flames before using acetone, which is highly volatile and flammable. *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result.* Chloroform inhalation should be avoided, but is not threatening in mild conditions. Benzene, toluene, and xylene are suspected carcinogens so avoid prolonged exposure to fumes and vapors.

Procedure: Place 100 milliliters of tap water (3.4 fluid oz.) and 100 grams of acetone (3.5 oz.) into a beaker or any suitable container, and then cool this mixture to 0 Celsius using a standard ice bath. Thereafter, slowly add in small portions, 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the acetone solution and maintaining its temperature at 0 Celsius. During the addition of the calcium hypochlorite, rapidly stir the acetone/water mixture, and maintain its temperature below 20 Celsius. After the addition of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 75-milliliter portions (four 2.5 fluid oz. portions) of benzene, toluene, or xylene. After extraction, combine all four portions (if not already done so), and then dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried solvent portion into a distillation apparatus (as illustrated below) and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform in a clean fractional distillation apparatus (as illustrated below) and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place.

Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

Step 1: Initial reaction of calcium hypochlorite with acetone.

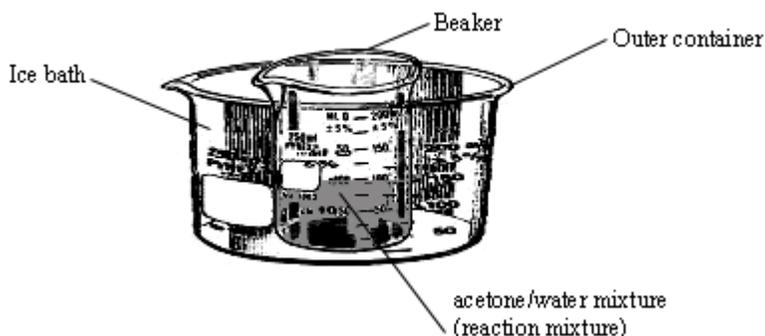


Figure 023. Set-up with ice bath for cooling the reaction mixture. The beaker can be re-placed with a flask or other suitable container. However, the container should not be made of plastic or other polymer that may corrode or dissolve by the acetone or chloroform. The outer container for use as the ice bath, can be glass, plastic, metal, or any other similar container.

Step 2: Extraction process

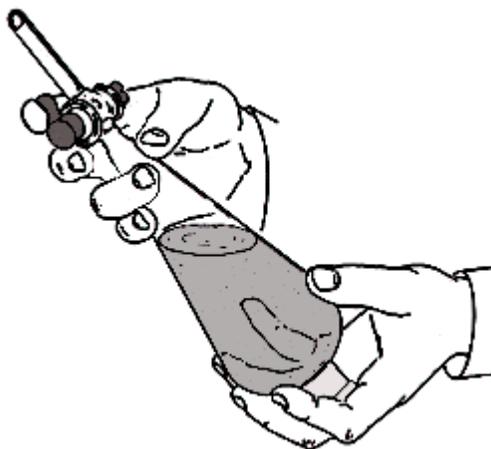


Figure 024. Extract the reaction mixture with benzene, toluene, or xylene.

Step 3: Distillation process to recover the chloroform

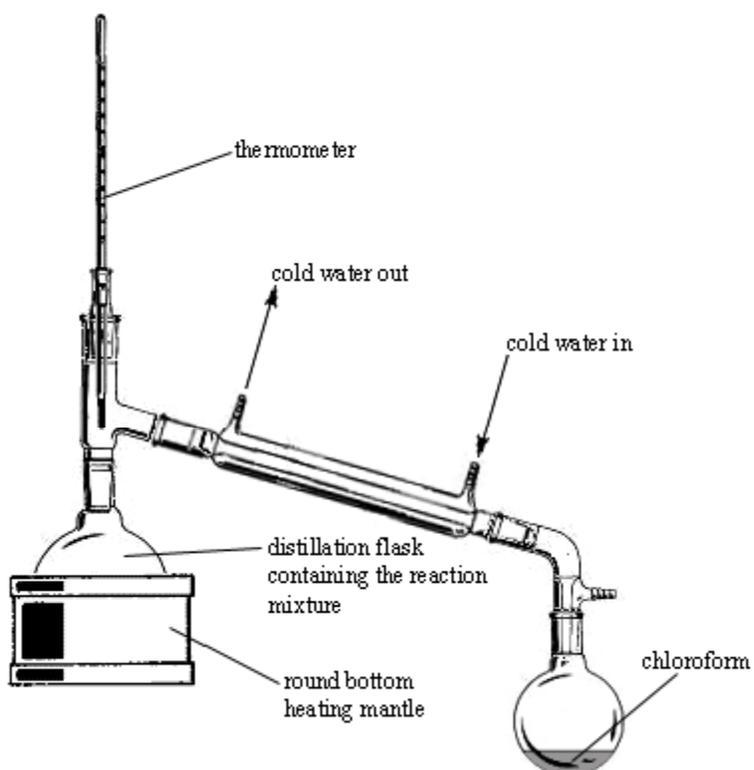


Figure 025. Distillation apparatus for collecting the chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not come into direct contact with the distillation flask (as bumping and foaming may result). The chloroform should be re-distilled using a fractional distillation apparatus for quality and purity.

Step 4: Fractional distillation apparatus for purifying the chloroform

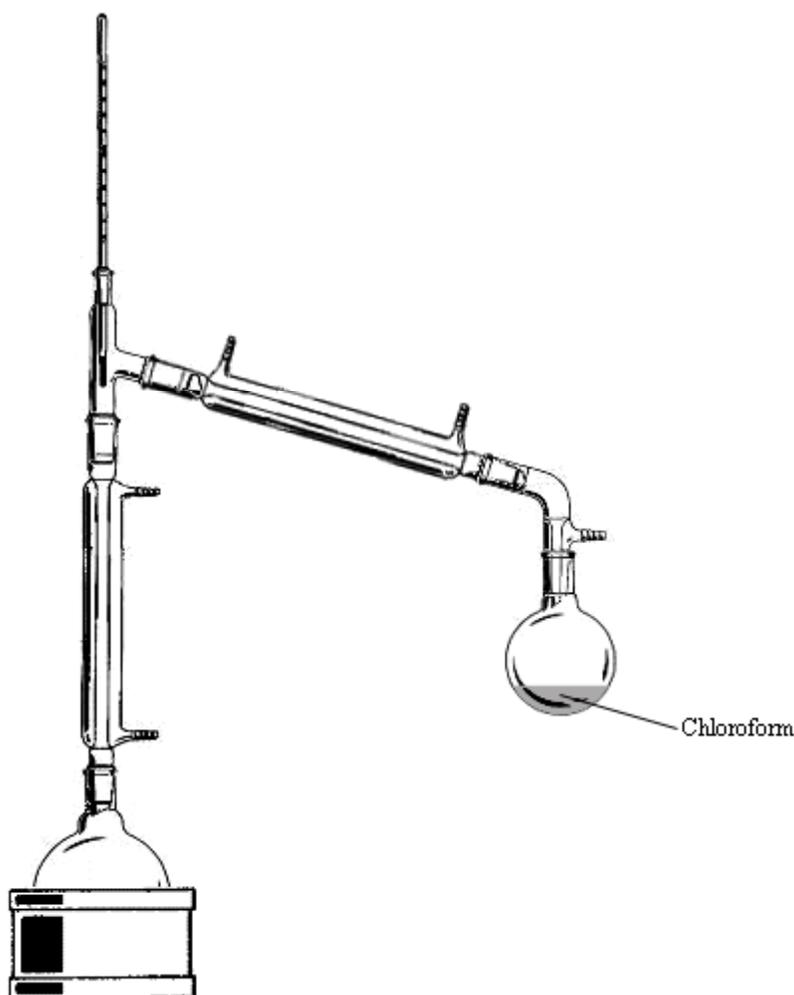


Figure 026. Fractional distillation apparatus for the fractional distillation of chloroform. The heating mantle can be replaced with a Bunsen burner, but the flame should not touch the glass. A hot plate or stovetop can also be used as a heat source if desired.

Final note for method 1

The by-products of calcium acetate, calcium chloride, and calcium hydroxide can be recovered as follows:

1. Filter the extracted reaction mixture (after the extraction process), to filter-off the calcium hydroxide
2. Recover the acetate by treating the filtered reaction mixture with dilute sulfuric acid, and then filter-off the precipitated calcium sulfate. Then, distill the mixture at 110 Celsius to recover the acetic acid formed by the addition of sulfuric acid.

Note: if using hydrochloric acid instead of sulfuric acid, evaporate the left over reaction mixture to dryness after the distillation (to remove acetic acid), so as to recover the calcium chloride. Calcium chloride can be heated using a Bunsen burner so as to form anhydrous calcium chloride, which makes a powerful drying agent. Second note: acetic acid is a useful by-product and can be used in a variety of applications.

Note: If desired, the bleaching powder can be replaced with Clorox bleach or other Clorox like bleaches (that contain sodium hypochlorite only); however, because most bleach products only contain 5% or less of sodium hypochlorite it would take astronomical amounts of bleach to carryout the reaction, but nonetheless, if you would like to try this technique—by all means; just remember to extract the entire huge reaction mixture with extra amounts of benzene, toluene, or xylene to properly recover all of the chloroform)

Method 2: Preparation of chloroform from Rubbing alcohol and bleaching powder

(By-products from reaction: Calcium acetate, calcium chloride, and calcium hydroxide)

Materials:

| | |
|--|---|
| 1. 100 milliliters of tap water (3.4 fluid oz.) | 4. 300 milliliters (10.1 fluid oz.) of benzene, toluene, or xylene. Toluene and xylene should be available at most hardware stores. |
| 2. 150 grams (5.3 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol). | 5. 40 to 50 grams (1.4 to 1.8 oz.) of pickling salt (available in any grocery store). |
| 2. 1181 grams (2.6 pounds) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles. | 6. 15 grams of anhydrous magnesium sulfate |
| 3. 1 milliliter of 95% ethyl alcohol | |

Summary: Chloroform can be made in an identical manner as in method 1; however, we replace the acetone with rubbing alcohol. First of all, the rubbing alcohol needs to be purified, so the salting out process is used. After the salting out process, the refined alcohol, also known as isopropyl alcohol, is then reacted with bleaching powder in a familiar manner. The bleaching powder first reacts with the alcohol, oxidizing it to acetone, from where additional bleaching powder then converts it into chloroform.

Hazards: *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result.* Chloroform inhalation should be avoided, but is not threatening in mild conditions. Benzene, toluene, and xylene are suspected carcinogens so avoid prolonged exposure to fumes and vapors.

Procedure: Into a suitable separatory funnel, place 150 grams (5.3 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol), followed by 40 to 50 grams (1.4 to 1.8 oz.) of pickling salt. Note: make sure the stopcock on the separatory funnel is closed before starting. Thereafter, stopper the separatory funnel, and then shake the entire funnel vigorously for about 5 minutes. Then allow the separatory funnel to stand (upright) for about 30 minutes. Thereafter, drain-off the bottom brine layer (and any excess salt), and thereafter, recover the upper isopropyl alcohol layer. Now, place this recovered upper isopropyl alcohol layer into a suitable container, and then add in 100 milliliters (3.4 fluid oz.) of tap water, and then cool this mixture to 0 Celsius using a standard ice bath. Thereafter, slowly add in small portions, 1361 grams (3 pounds) of 65 to 70% calcium hypochlorite (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs) over a period of 1 hour while stirring the acetone solution and maintaining its temperature at 0 Celsius. During the addition of the calcium hypochlorite, rapidly stir the acetone/water mixture, and maintain its temperature below 20 Celsius. After the addition of the 65 to 70% calcium hypochlorite, continue to stir the reaction mixture at 0 Celsius for an additional thirty minutes. Afterwards, stop stirring and then extract the reaction mixture with four 75-milliliter portions (four 2.5 fluid oz. portions) of benzene, toluene, or xylene. After the extraction, combine all four portions (if not already done so), and then dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place this filtered dried solvent portion into a distillation apparatus (similar to the one in method 1) and then distill at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform into a clean fractional distillation apparatus (as similar to the one in method 1) and distill at 62 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place. See method 1 for the illustrations on extraction, distillation, etc., Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

Final note for method 2:

See method 1.

Method 3: Preparation of chloroform from Rubbing alcohol, potassium dichromate, pickling salt, and dilute sulfuric acid using an electrochemical cell.

(By-products from reaction: sodium acetate, sodium sulfate, and sodium hydroxide)

Materials:

| | |
|---|---|
| 1. 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol) | 5. 375 milliliters (12.6 fluid oz) of warm tap water |
| 2. 148 grams (5.2 oz.) of pickling salt | 6. 225 milliliters (7.6 fluid oz.) of benzene, toluene, or xylene |
| 3. 1 gram of potassium dichromate (available on-line) | 7. 5 milliliters of concentrated sulfuric acid (1 teaspoon) |

| | |
|--------------------------------------|--|
| | (plumbers grade or better) |
| 4. 1 milliliter of 95% ethyl alcohol | 8. 15 grams of anhydrous magnesium sulfate |

Summary: Chloroform can be prepared in a unusual manner by electrolyzing a solution of pickling salt and isopropyl alcohol in the presence of a small amount of sulfuric acid and potassium dichromate using graphite or lead electrodes. The process develops low yields of chloroform, and takes considerable amounts of time, but is effective at producing small amounts of chloroform. After the initial reaction, the mixture is extracted with benzene, toluene, or xylene, and the resulting solvent extracts are then combined, and then distilled to recover the chloroform.

Procedure: Into a suitable seperatory funnel, place 40 grams (1.4 oz.) of 70% rubbing alcohol (just regular rubbing alcohol, no fancy colored stuff or fancy additives—just plain old rubbing alcohol), followed by 20 to 30 grams (0.7 to 1 oz.) of pickling salt. Note: make sure the stopcock on the seperatory funnel is closed before starting. Thereafter, stopper the seperatory funnel, and then shake the entire funnel vigorously for about 5 minutes. Then allow the seperatory funnel to stand (upright) for about 30 minutes. Thereafter, drain-off the bottom brine layer (and any excess salt), and thereafter, recover the upper isopropyl alcohol layer. Now, pour this isopropyl alcohol into a suitable container (see the following illustration for electrochemical cell make-up), and then add in 118 grams (4.1 oz.) of pickling salt, followed by 375 milliliters (12.6 fluid oz) of warm tap water, followed by 1 gram of potassium dichromate, and then followed by 5 milliliters of concentrated sulfuric acid. Thereafter, vigorously stir the entire mixture for about 30 minutes to dissolve all solids. At this point, you will end up with an orangey colored solution. Now, setup your electro chemical cell, and then electrolysize this mixture for 15 hours at 12 volt 6 amp current. Use a battery charger with this setting—these battery chargers are very common and availed in many stores. After 15 hours, unplug the battery charger, and then pour the entire mixture into a beaker, and then extract this entire mixture with three 75-milliliter portions (2.5 fluid oz. portions) of benzene, toluene, or xylene, and after the extraction process, combine all solvent portions, if not already done so. Thereafter, dry this combined solvent portion by adding to it, 15 grams of anhydrous magnesium sulfate (to absorb water). After adding in the magnesium sulfate, stir the entire mixture for about 10 minutes, and then filter-off the magnesium sulfate. Then place this filtered dried solvent mixture into a distillation apparatus (as similar to method 1), and distill over the chloroform at 65 Celsius until no more chloroform passes into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the receiver flask from the distillation apparatus. Then place this distilled crude chloroform into a clean fractional distillation apparatus (as similar to method 1) and distill at 62 Celsius until no more chloroform passes over into the receiver flask. When no more chloroform passes into the receiver flask, stop the distillation, and then remove the chloroform from the receiver flask and then add to it, 1 milliliter of 95% ethyl alcohol. Then store this chloroform in an amber glass bottle in a cool dry place. See method 1 for the illustrations on extraction, distillation, ect., Note: the benzene, toluene, or xylene used in the extraction, can be recovered after the first distillation process.

The electrolysis process

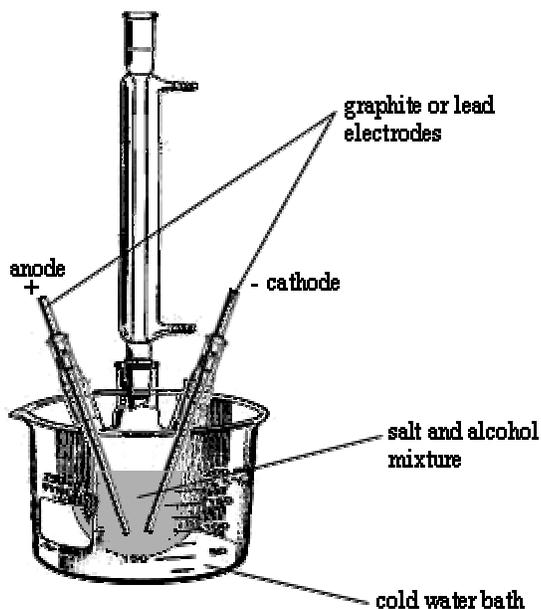


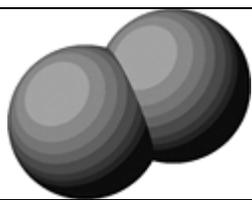
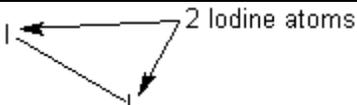
Figure 027. Electro chemical cell for the preparation of chloroform. This apparatus is a laboratory apparatus, and can be replaced with similar apparatus designs. The 3-neck flask shown in the illustration can be replaced with a large canning jar, by cutting three holes in the lid, or something of that nature. Other containers can be used, and you should use your imagination. Note: the red clamp of your battery charger goes on the positive anode, and the black clamp goes on the negative cathode. Graphite rods of various lengths and diameters are available on line for reasonable prices.

Final note for method 3

The by-products of sodium acetate, and sodium sulfate can be recovered as follows:

1. Filter the extracted reaction mixture (after the extraction process), to filter-off any insoluble materials (graphite and/or lead sludge).
2. Recover acetate by adding in dilute sulfuric acid or hydrochloric acid, and then distill the resulting acidic mixture at 110 Celsius to recover the acetic acid. After the distillation process, evaporate the left over reaction mixture to dryness after the distillation (to remove acetic acid), so as to recover the sodium chloride or sulfate (depending on which acid you used). This sodium chloride can be recycled if desired for a new run. Second note: acetic acid is a useful by-product and can be used in a variety of applications.

Preparation 2: Iodine

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | I_2 |
| Structure make-up | Short hand chemical structure |

Iodine forms blackish to purplish-black plates, powder, or granules with a characteristic metallic luster and peculiar odor. It forms a purple vapor when gently heated, and readily volatilizes. It has a melting point of 114 Celsius and boiling point of 185 Celsius. Iodine is soluble in aqueous solutions of potassium or sodium iodide, and is soluble benzene, ethyl alcohol, ether, cyclohexane, and methylene chloride. Iodine is capable of producing eye, nose, and throat irritation so wear gloves when handling and use ventilation. Iodine is readily obtainable by bubbling chlorine gas into a solution of potassium or sodium iodide at room temperature, then filtering-off the precipitated iodine, followed by sublimation to purify the iodine. Iodine can also be obtained by treating solutions of potassium or sodium iodide with bleaching powder, potassium permanganate solutions, or with excess bleach (Clorox), then filtering-off the precipitated iodine crystals, followed by sublimation to purify the iodine.

Method 1: Preparation of Iodine from potassium or sodium iodide and bleach

(By-products from reaction: sodium or potassium chloride)

Materials:

| | |
|---|--|
| 1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide (available on line). | 4. 225 milliliters of methylene chloride (dichloromethane); (7.6 fluid oz.) (available on on-line) |
| 2. 150 milliliters (5 fluid oz.) of cold tap water | 5. 950 milliliters (17 fluid oz.) of warm tap water |
| 3. 500 milliliters (17 fluid oz.) of regular household bleach (Clorox bleach). | 6. 15 grams (1/2 oz.) of anhydrous magnus sulfate |

Summary: Iodine is readily prepared by reacting bleach with potassium or sodium iodide. The reaction is relatively swift, and produces a brownish black precipitate of crude iodine. This crude iodine can be obtained by filtration using gravity or vacuum filtration, or extracted into methylene chloride. The iodine if filtered, is washed and then dried, and then sublimed. If the reaction mixture was extracted, the methylene chloride is removed by distillation, and the resulting left over iodine is then sublimed in the same fashion. **Note: some recent brands of bleach now include sodium hydroxide. Do not buy this brand of bleach, as it will interfere with the reaction. Check the ingredients label of your bleach before purchasing. In some cases, cheap generic bleach is the better product.**

Hazards: Sodium hypochlorite (bleach) is a strong oxidizer and should be kept away from combustible liquids. Iodine is an irritant, so avoid skin contact, and inhalation of fumes or vapors.

Procedure: Into a beaker or other suitable container add 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide, followed by 150 milliliters (5 fluid oz.) of cold tap water. Thereafter, stir the entire mixture for several minutes to dissolve all solids. Thereafter, add in 500 milliliters (17 fluid oz.) of regular household bleach (Clorox bleach). Make sure the bleach is just regular household bleach and has no fancy additives, colors, or the like. The rate of addition of the household bleach to the potassium or sodium iodide solution should be slow, but should only take about 15 to 30 minutes. During the addition of the household bleach to the potassium or sodium iodide solution, casually stir the potassium or sodium iodide solution. After the addition of the household bleach, continue to stir the mixture for about 30 minutes, and then do one of two things: A. filter the mixture to recover the brownish black precipitate formed (crude iodine) using either gravity filtration or vacuum filtration, or B. extract the mixture with methylene chloride (three 75-milliliter portions, three 2.5 fluid oz. portions). If filtering the reaction mixture, allow the brownish precipitate of iodine to collect on the filter paper, and thereafter, allow the filter paper to stand over night and partially dry, or if using vacuum filtration, vacuum dry the brownish precipitate. Thereafter, gently scrape-off the brownish precipitate from the filter paper, into a clean beaker or other suitable container, and then add in 500 milliliters (17 fluid oz.) of warm tap water. Then gently mix the iodine mixture for about 10 minutes, and then once again, filter-off the insoluble iodine using gravity filtration (this is called washing the iodine with water). Note: if using vacuum filtration, after vacuum drying the initial iodine after filtration, simply wash it several times with three 150-milliliter portions (three 5 fluid oz. portions), of warm tap water, and then vacuum dry the iodine. If using gravity filtration, after washing the iodine, and then collecting the iodine by filtration, allow the filter paper containing the iodine to stand over night to partially dry. The next day, gently scrape-off the iodine from the filter paper onto a clean piece of glass or porcelain dish (no metal), and then set this dish aside (no sunlight) for several days to allow the iodine to dry. When the iodine has been dried, either by using vacuum filtration, or by using the slow process of air-drying, place the dry iodine into a sublimation apparatus, as illustrated below, and sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called “double sublimed reagent grade iodine”, which will have a purity of no less than 98% iodine

If you extracted the reaction mixture with methylene chloride, combine all methylene chloride portions once finished (if not already done so), and then dry this combined methylene chloride portion by adding to it, 15 grams (1/2 oz.) of anhydrous magnesium sulfate. Then stir the entire mixture for about 10 minutes (to absorb moisture), and then filter-off the magnesium sulfate. Once the magnesium sulfate has been filtered-off, place this methylene chloride into a standard atmospheric distillation apparatus, and gently distill-off the methylene chloride at 40 Celsius (104 Fahrenheit) until no more methylene chloride distills over. When this is the result, recycle the methylene chloride, and then recover the left over remaining brownish residue of iodine. Then place this recovered iodine into a sublimation apparatus, as illustrated below, and sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called “double sublimed reagent grade iodine”, which will have a purity of no less than 98% iodine

Classic sublimation setup

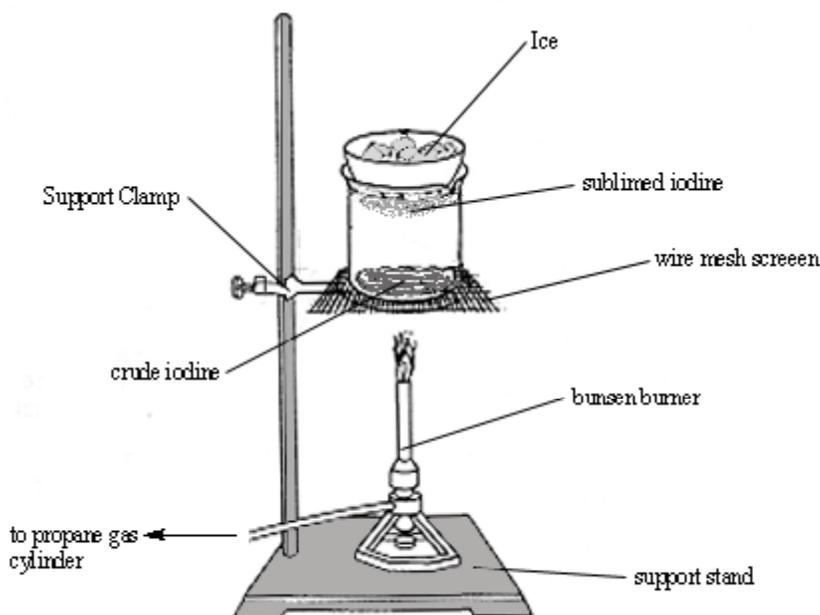


Figure 028. Setup for the sublimation of iodine.

Final note for method 1: None

Method 2: Preparation of Iodine from potassium or sodium iodide and bleaching powder

(By-products from reaction: calcium hydroxide, and potassium or sodium chloride)

Materials:

| | |
|--|---|
| 1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide | 3. 70 grams (2.5 oz.) of 65 to 70% calcium hypochlorite (bleaching powder) (commercially available; sold under a variety of brand names for use in swimming pools and hot tubs). Can also be found online at many pool and spa chemical suppliers and can be purchased without hassles. |
| 2. 250 milliliters (15 fluid oz.) of warm tap water | 4. 200 milliliters (6.8 fluid oz.) of tap water |

Summary: Iodine can be prepared in a modified process whereby bleaching powder is used instead of Clorox bleach. The reaction is similar, but first, a solution of hypochlorous acid is prepared by simply mixing the bleaching powder with warm water. The warm water decomposes the bleaching powder into hypochlorous acid and calcium hydroxide. This mixture is then filtered, and the resulting hypochlorous acid is then reacted with a solution of potassium or sodium iodide. After the reaction, the precipitated iodine is then recovered by filtration, washed, dried, and then sublimed to yield high purity iodine.

Hazards: *Calcium hypochlorite is a powerful oxidizer, and should never be mixed with concentrated sulfuric acid; explosions will result. Do not mix hypochlorous acid with ammonia or ammonium salts—as the high explosive nitrogen triiodide is formed.*

Procedure: Into a suitable beaker or container, place 250 milliliters (8.4 fluid oz.) of warm tap water, followed by 70 grams (2.5 oz.) of bleaching powder. Thereafter, stir the entire mixture for about 1 hour, and then filter-off the precipitated solids (composed of calcium hydroxide). Note: vacuum filtration works best, but if all you have is gravity filtration, place a small amount of clean sand in the bottom of the filter paper before filtering. This will help aid in the filtration process, which can be a slow one. After the filtration process, you will have a yellowish relatively odorless liquid. This yellow liquid will be composed of hypochlorous acid. Now, prepare a potassium or sodium iodide solution by adding and dissolving 50 grams (1.7 oz.) of potassium iodide, or 45 grams (1.6 oz.) of sodium iodide into 200 milliliters (6.8 fluid oz.) of tap water, and then slowly add this iodide solution to your yellowish hypochlorous acid mixture over a period of about 10 to 15 minutes. After 10 to 15 minutes, stir the mixture for about 30 minutes, and then filter-off the precipitated brownish solids (composed of the crude iodine). Once the brown solids have been filtered-off, wash them with several portions of cold water (use the same technique as in method 1 for washing), and then vacuum dry or air-dry these brownish solids. Thereafter, place this dried iodine into a sublimation apparatus, as illustrated in method 1, and then sublime the iodine to purify it. This sublimation process should be repeated once more to produce what is called “double sublimed reagent grade iodine”, which will have a purity of no less than 98% iodine.

Final note for method 2: None

Method 3: Preparation of Iodine from potassium or sodium iodide and chlorine gas

(By-products from reaction: calcium hydroxide, and potassium or sodium chloride)

Materials:

| | |
|--|--|
| 1. 50 grams (1.7 oz) of potassium iodide or 45 grams (1.6 oz) of sodium iodide. | 3. 30 grams (1 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work). Sold on various online lab supply stores. |
| 2. 240 milliliters (8 fluid oz.) of tap water | 4. 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid. (Available in hardware stores as muriatic acid (at least 31% by weight—read label). |
| 2. 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). All are available on on-line auction sites, or other on line sites. | |

Summary: Iodine can be prepared from chlorine gas instead of household bleach or bleaching powder, but it requires a more elaborate setup, and time. Nevertheless, it can be used to prepare large quantities of iodine (to produce more iodine, simply double any

mass or liquid quantities involved). The chlorine gas used in the operation can be prepared by dripping hydrochloric acid onto a strong oxidizer, or by electrolyzing a solution of hydrochloric acid. The liberated chlorine gas, in any case, is then bubbled into a solution of potassium or sodium iodide. After the reaction, the iodine is filtered-off, washed, dried, and then sublimed in the usual manner.

Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, which can cause skin irritation.

Procedure: Setup the chlorine generating apparatus as illustrated below. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated below, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion. Also, observe the chlorine generator utilizing an electrochemical method. The electrochemical method is sufficient to produce laboratory quantities of gas (see the following illustration).

Now, into a suitable beaker, flask, or container, place 50 grams (1.7 oz.) of potassium iodide or 45 grams (1.6 oz.) of sodium iodide, followed by 150 milliliters (5 oz.) of tap water. Thereafter, stir the entire mixture for about 30 minutes to dissolve all solids.

After you have the chlorine generator set-up, place into the reaction flask, 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 30 grams (1 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) into 40 milliliters (1.35 fluid oz.) of tap water. If you are using an electrochemical cell, fill your cell with 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid, followed by 200 milliliters (7 fluid oz.) of cold tap water.

Once your chlorine generator is setup (if using the hydrochloric acid drip method), begin dripping the hydrochloric acid solution, onto the oxidizer contained in the reaction flask. If using an electrochemical cell, begin the electrolysis by plugging in your battery charger. In either case, the chlorine gas that is evolved should then be bubbled into your potassium or sodium iodide solution (previously prepared). The chlorine gas will react with the potassium or sodium iodide to form iodine, which will precipitate as a brownish-black solid. When no more chlorine gas passes over, in either case, the reaction is complete. Thereafter, filter-off the precipitated brownish-black iodine solids, as usual, using either vacuum filtration or gravity filtration, and then wash them with cold water, and then vacuum dry or air-dry the solids. Once the iodine is completely dry, sublime it as illustrated in method 1. Repeat the sublimation process one more time to yield high purity reagent grade iodine.

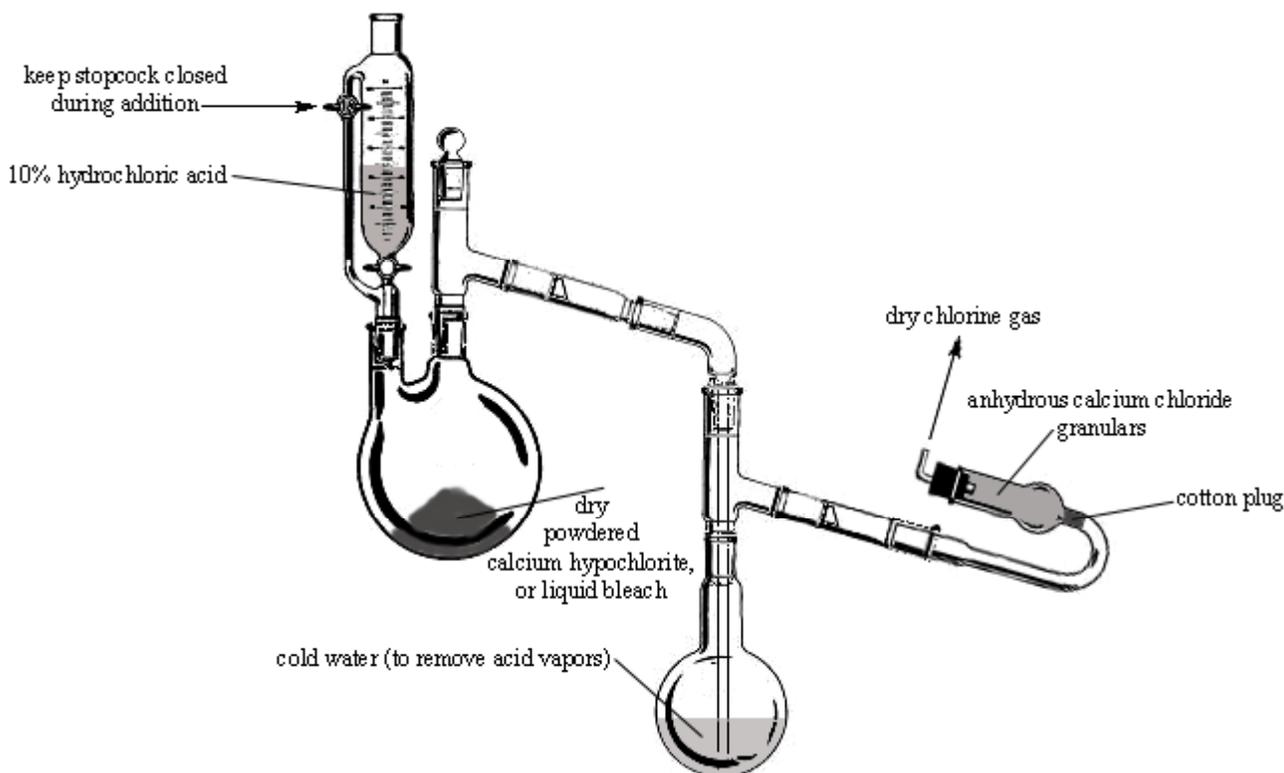


Figure 029. Apparatus for preparing chlorine gas—the hydrochloric acid drip method. The out let tube where the dry chlorine gas comes out, should be attached to a latex tube, and this tube then submerged into the potassium or sodium iodide solution.

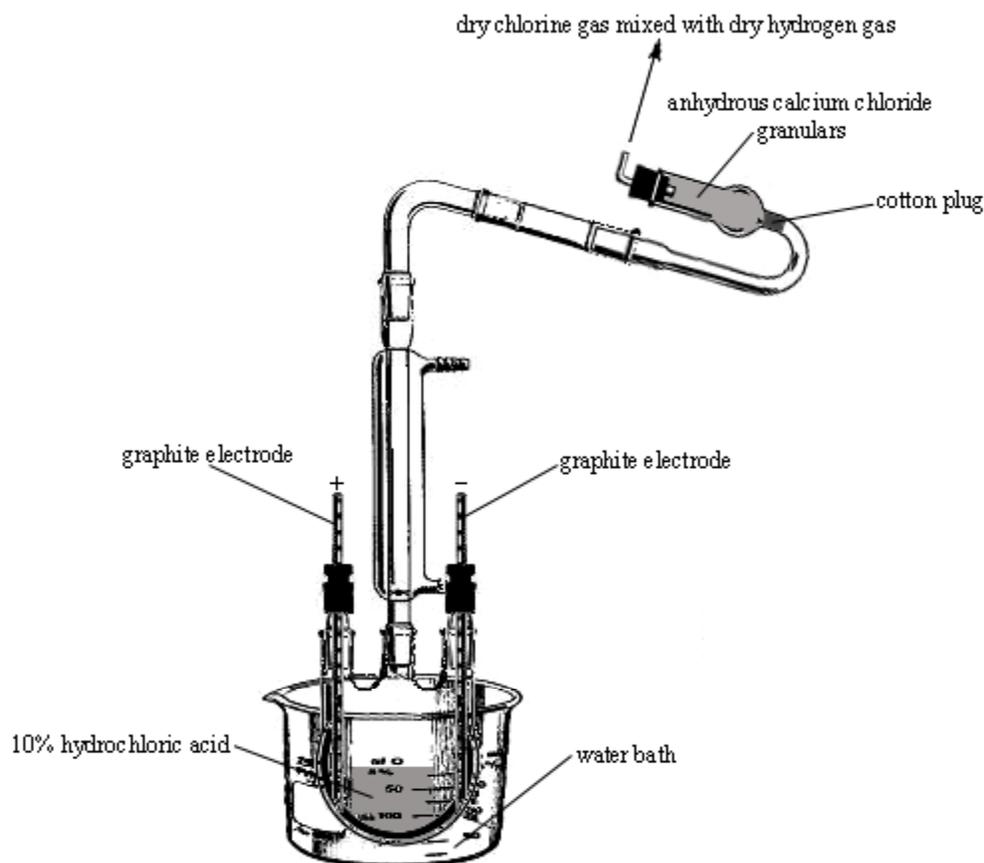
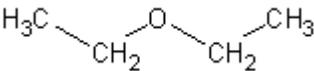
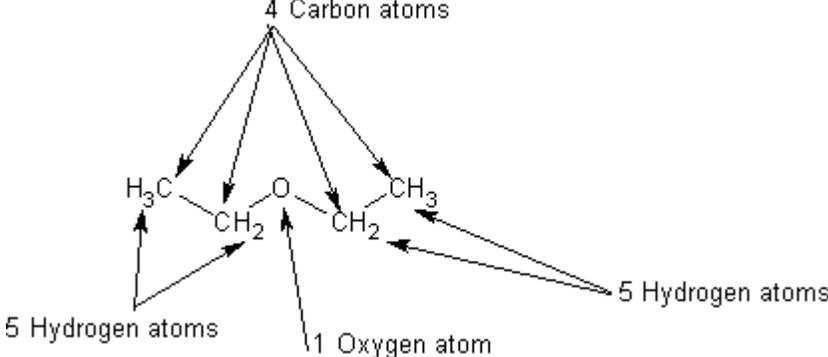
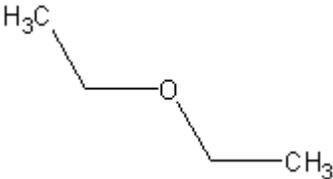


Figure 030. Apparatus for the electrolysis of hydrochloric acid. **Warning:** If the apparatus is exposed to direct sunlight, an explosion will occur—as hydrogen gas is also liberated (hydrogen gas at the negative electrode, and the chlorine gas at the positive anode). Chlorine explodes when mixed with hydrogen and then exposed to sunlight (only when exposed to sunlight). The detonation propagates downwards, so there is no immediate danger from fragments. As a reminder, the electrolysis of hydrochloric acid is perfectly safe as long as the apparatus is protected from direct sunlight, magnesium light, halogen lamps, or UV lamps (cover all windows, ect.). Note: the black clamp of your battery charge goes on the (-) electrode, and the red clamp of the battery charger goes on the (+) electrode. Use a current of 6 volt 2 amp or what ever works best for you. If using 6 volt 2 amp, it should take about 2 hours for the chlorine evolution to cease.

Final note for method 3: None

Preparation 3: Diethyl Ether

Also known as: Ether, Ethyl oxide, Anesthetic, Ethoxyethane

| | |
|--|---|
|  |  |
| Chemical structure | 3D Structure |
|  |  |
| Structure make-up | Short hand chemical structure |

Diethyl Ether, other wise known as just ether, is a mobile, very volatile, highly flammable liquid, which produces explosive vapors. It has a sweetish, pungent odor, and a burning taste. Ether forms explosive peroxides when exposed to air—ether containing peroxides will detonate if heated, shattering the glass vessel. Before heating mixtures containing ether, the peroxide test should be conducted. To test for peroxides, add five drops of ferrous chloride solution to the ether mixture. If a red or black color appears, peroxides are present. *Note: This test will not work properly if there are oxidizing agents in the ether mixture.* Ether can be stabilized by the addition of small amounts of naphthols, but this does not protect ether 100% from peroxide formation. Ether has a melting point of -116 Celsius, and a boiling point of 35 Celsius. Ether and air mixtures are explosive, so extinguish all flames and do not smoke when handling it. Protect ether from static electricity, which can also cause fire. Ether is insoluble in water, but miscible with alcohol, benzene, chloroform, and many oils. Do not mix 99% nitric acid with ether, as detonation will take place. Inhalation of ether vapors can produce intoxicating effects. These effects include feelings of euphoria, well-being, relaxation, and a general state of high. These effects can also lead to feelings of drunkenness. Ether is a narcotic in high concentrations, but is not habit forming. Store ether in tightly sealed bottles in a cool place (preferably in a refrigerator). For prolonged storage, store ether over sodium sulfite and keep in a bottle filled to the top (to minimize the air space). Ether can be prepared by heating 95% ethanol and 98% sulfuric acid (1 to 1 ratio) to 100 Celsius, and simultaneously condensing the distilled-off vapors of ether. The ether is then purified by re-distillation. Ether is a widely available commercial chemical, but its status as an anesthetic may limit its sale to the general public.

Method 1: Preparation of diethyl ether from vodka and concentrated sulfuric acid

(By-products from reaction: none)

Materials:

| | |
|--|---|
| 1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,) | 2. 300 milliliters (17 fluid oz.) of concentrated sulfuric acid (available at several places, but is readily available at janitorial and plumbing supply stores, where it sold as 93 to 98% purity. The 93% acid is 98% concentrated acid, but with iron, and can be used with satisfaction). |
|--|---|

Summary: Diethyl ether can be conveniently prepared by distilling a mixture of vodka with sulfuric acid. The reaction takes appreciable amounts of time, ranging from how much vodka you use to begin with, but produces a steady stream of diethyl ether, which distills over during the reaction. After the initial reaction, the recovered diethyl ether is re-distilled for quality and purity.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics.

Procedure: Into a suitable distillation apparatus, place 1250 milliliters (42.2 fluid oz.) of store bought vodka (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.,), followed by slowly and carefully adding in 300 milliliters (10 fluid oz.) of concentrated sulfuric acid. The concentrated sulfuric acid can be of any brand, and you may use plumbers brand concentrated sulfuric acid with satisfactory results. Note: The addition of the concentrated sulfuric acid to the vodka generates excessive heat, so use caution. After adding in all the concentrated sulfuric acid, boil the mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill over during this time.

After 6 to 8 hours, remove the heat source, and allow the sulfuric acid mixture in the distillation flask or pot to cool to room temperature. The diethyl ether can then be recovered from the receiver flask, and then re-distilled using a fractional distillation apparatus (similar to the one illustrated below), and distilled at 40 Celsius. The result after re-distillation will be around 500 milliliters of pure ether. Note: the sulfuric acid mixture, which may be colored blood red if iron was present in the acid (plumbers grade), can be boiled at 100 celsius in a suitable glass container to boil-off some of the water to concentrate the sulfuric acid. The point is, the sulfuric acid can be recycled over and over again (even if it is colored blood red or contaminated with other impurities), as it is only a catalyst in the reaction, and is not consumed or chemically changed in any way.

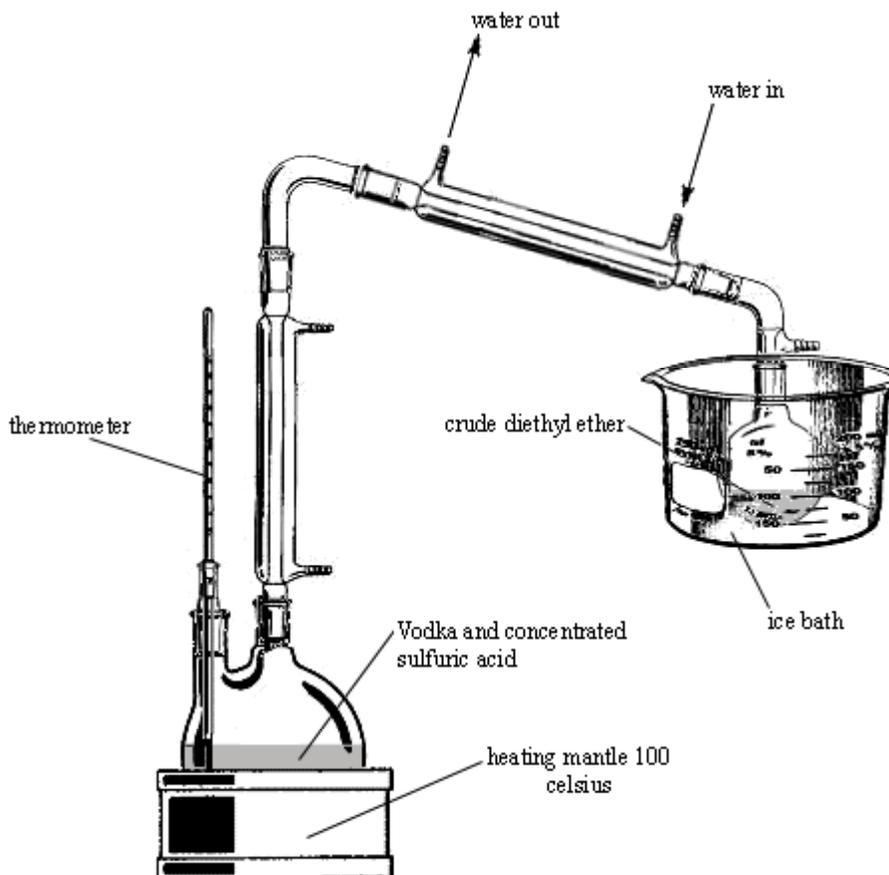


Figure 031. This apparatus can be replaced with any standard distillation apparatus, or standard fractional distillation apparatus; Alcohol stills can also be used, but may undergo corrosion in the distillation pot due to the presence of sulfuric acid. The diethyl ether should be re-distilled using a standard fractional distillation apparatus, or equivalent. After re-distillation, the ether should be stored in a proper container such as a metal can, or glass container, sealed air-tight, and filled to the top (to minimize air gaps), and these containers should be stored in a refrigerator until use. Note: the heating mantle portrayed in the illustration can be replaced by a Bunsen burner, hot plate, stovetop, steam bath, or oil bath if desired.

Final note for method 1: None

Method 2: Preparation of diethyl ether from vodka and battery acid

(By-products from reaction: none)

Materials:

| | |
|---|---|
| 1. 1250 milliliters (42.2 fluid oz.) of store bought vodka, usually 80 proof (use the cheap stuff, i.e., Popov, Kirov, Skoll, ect.) | 3. 500 milliliters (17 fluid oz.) of fresh battery acid |
| 2. 50 grams (3.5 oz.) of pickling salt | |

Summary: Diethyl ether can be prepared by reacting ethyl alcohol with battery acid. However, the vodka employed as the alcohol source must first be distilled, and then salted out. To do this, the vodka is first distilled in the usual manner, and the alcohol, which collects in the receiver flask, is then treated with salt to abstract any water. After the water has been removed, the alcohol portion is

then distilled in the presence of fresh battery acid, whereby the diethyl ether slowly forms in the usual manner, and is carried over. Re-distillation forms a purified liquid.

Hazards: Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: Into a standard distillation apparatus, alcohol still, or other means, place 1250 milliliters (42.2 fluid oz.) of vodka, and then distill the alcohol at 80 Celsius for about 4 hours. After 4 hours, remove the heat source, and then recover the alcohol that distilled over into the receiver flask or receiver container. Now, place this alcohol (which will be composed of about 90%+ ethyl alcohol), into a large separatory funnel or equivalent, and then add in 50 grams (3.5 oz.) of pickling salt. Thereafter, vigorously shake the entire mixture for about 10 minutes. After 10 minutes, allow the mixture in your separatory funnel or equivalent, to stand for about 30 minutes. Thereafter, drain-off the bottom water layer and excess salt, and thereafter, recover the upper alcohol layer. Now, place this recovered upper alcohol layer into a standard distillation apparatus, as illustrated in method 1 or similar, and then add in 500 milliliters (17 fluid oz.) of fresh battery acid. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent in the same manner as in method 1 (distill at 40 Celsius). Note: the battery acid mixture left over, can be recycled, but it must be boiled to drive-off at least 1/3 of the total volume (to remove water).

Final note for method 2: None

Method 3: Preparation of diethyl ether from distilled ethyl alcohol (95% alcohol; ABS alcohol; ever clear), with concentrated sulfuric acid or battery acid

(By-products from reaction: none)

Materials:

| | |
|---|---|
| 1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol; ABS alcohol; ever clear | 3. or 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear |
| 2. 100 milliliters (3.3 fluid oz.) of concentrated sulfuric acid (plumbers grade works great) | 4. or 300 milliliters (10 fluid oz.) of fresh battery acid |

Summary: Diethyl ether can be prepared by reacting 95% ethyl alcohol with concentrated sulfuric acid, or battery acid. The 95% ethyl alcohol is commonly called ABS alcohol, or ever clear. The alcohol is simply mixed with concentrated sulfuric acid or battery acid, and then distilled in the usual manner. The diethyl ether that forms, slowly distills over in the usual manner.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

Procedure: If using concentrated sulfuric acid, place 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear), into a standard distillation apparatus, as illustrated in method 1 or similar, and then slowly and carefully add in 100 milliliters (3.3 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent in the same manner as in method 1 (distill at 40 Celsius). Note: the sulfuric acid mixture left over, can be recycled, but it should be boiled to drive-off some of the water.

If using battery acid, place 500 milliliters (17 fluid oz.) of distilled ethyl alcohol (95% ethyl alcohol; ABS alcohol; ever clear) into a standard distillation apparatus, as illustrated in method 1 or similar, and then add in 300 milliliters (10 fluid oz.) of fresh battery acid. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Then re-distill this diethyl ether using a standard fractional distillation apparatus or equivalent in the same manner as in method 1 (distill at 40 Celsius). Note: the battery acid mixture left over, can be recycled, but it must be boiled to drive-off at least 1/3 of the total volume (to remove water).

Final note for method 3: None

Method 4: Preparation of diethyl ether from denatured alcohol with concentrated sulfuric acid or battery acid

(By-products from reaction: none)

Materials:

| | |
|--|---|
| 1. 550 milliliters (18.5 fluid oz.) of denatured alcohol—ethyl alcohol (available in most hardware stores, paint stores, and automotive stores). | 3. or 550 milliliters (18.5 fluid oz.) of denatured alcohol |
| 2. 100 milliliters (3.3 fluid oz.) of concentrated sulfuric acid (plumbers grade works great) | 4. or 300 milliliters (10 fluid oz.) of fresh battery acid |

Summary: Diethyl ether can be prepared by reacting denatured alcohol with concentrated sulfuric acid, or battery acid. The alcohol is simply mixed with concentrated sulfuric acid or battery acid, and then distilled in the usual manner. The diethyl ether that forms, slowly distills over. The diethyl ether should then be soaked in water, separated from the water phase, and then fractional distilled for purity.

Hazards: Wear gloves when handling concentrated sulfuric acid, and avoid skin contact, contact with clothing, and ingestion at all cost. Concentrated sulfuric acid will corrode, char, and dissolve many substances including some plastics. Wear gloves when handling battery acid, and avoid skin contact, contact with clothing, and ingestion. Battery acid contains about 25% sulfuric acid by weight, and is very corrosive.

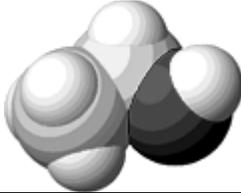
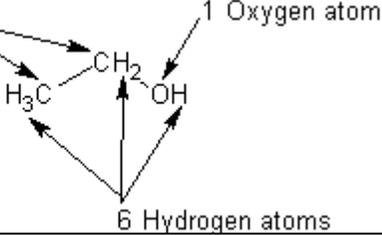
Procedure: If using concentrated sulfuric acid, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus as illustrated in method 1 or similar, and then slowly and carefully add in 100 milliliters (3.3 fluid oz.) of concentrated sulfuric acid (plumbers grade works great). Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Now, place this diethyl ether into a separatory funnel, or similar means, and then add in an equal volume of warm tap water. Then shake the entire mixture for about 10 minutes, and then allow the separatory funnel or equivalent to stand for 30 minutes. Then drain-off the bottom water layer, and then collect the upper ether layer. Finally, place this collected upper ether layer into a clean fractional distillation apparatus, and distill the ether at 40 Celsius, as in method 1. Note: the sulfuric acid mixture left over, can be recycled, but it must be boiled to drive-off some of the water.

If using battery acid, place 550 milliliters (18.5 fluid oz.) of denatured alcohol into a standard distillation apparatus, as illustrated in method 1 or similar, and then add in 300 milliliters (10 fluid oz.) of fresh battery acid. Thereafter, boil this mixture at 100 Celsius for about 6 to 8 hours, and allow the mixture to distill during this time. After 6 to 8 hours, remove the heat source, and then recover the diethyl ether from the receiver flask or receiver container. Now, place this diethyl ether into a separatory funnel, or similar means, and then add in an equal volume of warm tap water. Then shake the entire mixture for about 10 minutes, and then allow the separatory funnel or equivalent to stand for 30 minutes. Then drain-off the bottom water layer, and then collect the upper ether layer. Finally, place this collected upper ether layer into a clean fractional distillation apparatus, and distill the ether at 40 Celsius, as in method 1. Note: the battery acid mixture left over, can be recycled, but it must be boiled to drive-off at least 1/3 of the total volume (to remove water).

Final note for method 4: None

Preparation 4: Ethyl Alcohol; Ethanol

Also known as: Absolute alcohol; Grain alcohol; Ethyl hydrate

| | |
|--|---|
| $\text{H}_3\text{C}-\text{CH}_2-\text{OH}$ |  |
| Chemical structure | 3D Structure |
| <div style="display: flex; justify-content: space-between;"> 2 Carbon atoms 1 Oxygen atom </div>  <div style="text-align: center;">6 Hydrogen atoms</div> |  |
| Structure make-up | Short hand chemical structure |

95% Ethyl alcohol

95% Ethanol is a clear, colorless, very mobile, flammable liquid with a pleasant odor, and a pungent, burning taste. It has a boiling point of 78 Celsius and a melting point of -114 Celsius. 95% Ethanol slowly absorbs water from the air, and dilute ethanol solutions are slowly oxidized by air forming brown colored solutions containing mixtures of aldehydes, and carboxylic acids; mainly acetic acid. 95% Ethanol is miscible with water, and many organic solvents. 95% ethyl alcohol is called absolute ethanol because ethyl alcohol forms a binary azeotrope containing 95.57% ethyl alcohol by weight with a boiling point of 78 Celsius. Distillations cannot produce 99% ethanol because of this azeotrope. Ethyl alcohol is usually sold as denatured ethyl alcohol (mixed with small amounts of toxic chemicals to make non-drinkable) due to US government tax regulations. 95% Ethyl alcohol is toxic, and ingestion can cause alcohol poisoning. Dilute mixtures of ethyl alcohol (Vodka, Gin, Rum, Jack Daniels, beer, wine) produce intoxicating effects when ingested (these intoxicating effects can be increased if the dilute ethanol mixture is injected). 95% ethanol can be made by fermenting starch or sugars with yeast, followed by double distillation. 95% Ethanol is manufactured on an industrial scale by the petroleum industry from ethylene gas, sulfuric acid, and water. 95% Ethanol is a widely available commercial chemical sold under a variety of names. 95% ethanol can be obtained from double distillation of alcoholic beverages such as vodka, gin, or rum.

99% Ethyl alcohol

99% Ethyl alcohol is a colorless, very mobile and flammable liquid with a pleasant odor. Pure ethanol is tasteless. It rapidly absorbs water from the air, from which it forms an azeotrope of 95% ethyl alcohol. It is miscible with water, alcohol, ether, and many common organic solvents. 99% ethanol is toxic, and ingestion can cause poisoning. It is prepared by reacting ethylene gas with sulfuric acid, followed by distillation in the presence of minute amounts of water. It can also be made by double distillation of fermented cocktails, followed by treatment with metallic sodium to remove the water of azeotrope. 99% Ethyl alcohol can be obtained by salting out vodka (see 99% isopropyl alcohol), treating the recovered upper ethyl alcohol layer with large amounts of anhydrous magnesium sulfate, filtering, and then distilling the mixture to recover 99% ethyl alcohol.

Method 1: Preparation of 95% ethyl alcohol from liquor, wine, or beer

(By-products from reaction: none)

Materials:

| | |
|--|---|
| 1. 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money) | 3. or 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor |
| 2. or 2.5 liters or more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7% | 4. 50 grams (1.7 oz.) of baking soda |

Summary: 95% ethanol can be prepared by double distilling liquors, wines, or beers. After the first distillation, the crude ethyl alcohol (which will contain up to 90%+ ethyl alcohol), is treated with baking soda to remove odors, filtered, and then fractionally re-distilled producing 95% ethanol.

Procedure: If using liquors, place 1 liter or more (1.86 quarts or more) of 60 to 180 proof liquors (any kind will work, but you should use the cheap stuff to save money), into a clean distillation apparatus or similar as illustrated below. If using wines, place 2.5 liters or

more (2.64 quarts or more) of wine (any will work) with an alcohol concentration of at least 7%, into a distillation apparatus (similar to the one illustrated below). If using beer, place, 5 liters or more (1.3 gallons or more) of any beer, ail, brew, or malt liquor into a distillation apparatus or similar device as illustrated below. Then, despite which alcoholic beverage you use, distill your alcoholic beverage or concoction at 90 Celsius until no more liquid passes into the receiver flask (will take 4 to5 hours for the liquors, 4 to 8 hours for the wines, and 6 to 10 hours for the beers). When no more liquid passes into the receiver flask, remove the heat source, and then remove the receiver flask from the distillation apparatus. Then place 50 grams (1.7 oz.) of baking soda into the receiver flask, and then boil the contents to 100 Celsius for about 5 minutes. Thereafter, remove the heat source, and allow the contents to cool to room temperature. Afterwards, filter the mixture to remove the insoluble baking soda, and then place this filtered mixture into a clean fractional distillation apparatus (similar to the one illustrated in 3. Diethyl ether, method 1). Then distill at 80 Celsius until no more liquid passes into the receiver flask.

First distillation

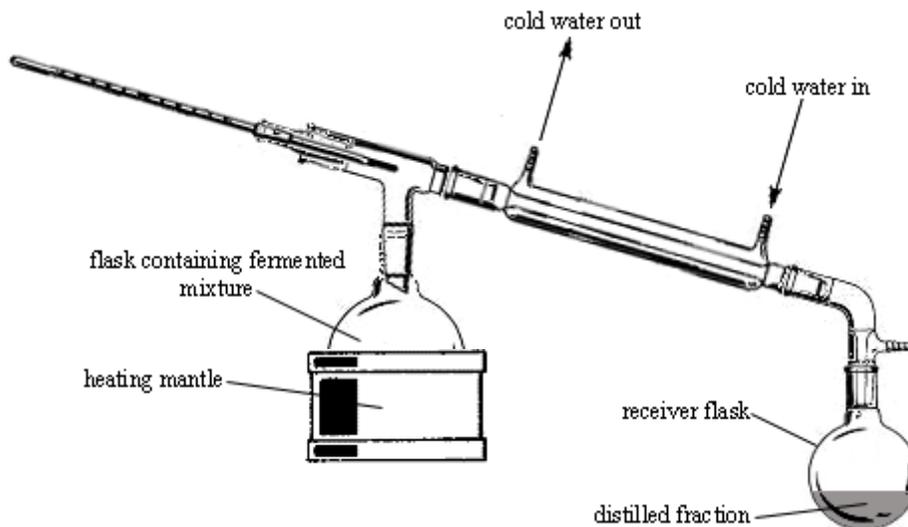


Figure 032. Simple distillation apparatus for the distillation of liquors, wines, or beers to yield 90%+ ethyl alcohol.

Final note for method 1: None

Method 2A: Preparation of 95% ethyl alcohol from table sugar or common food grade starch (corn starch)

(By-products from reaction: to complex for discussion here)

Materials:

| | |
|---|--|
| 1. 1 kilogram (2.2 pounds) of table sugar | 7. or 2 liters (2.1 quarts) of hot tap water |
| 2. 3 liters (3.2 quarts) of tap water | 8. or 10 drops of concentrated hydrochloric acid (muriatic acid will work). |
| 3. 5 drops of concentrated hydrochloric acid (muriatic acid will work) or 5 drops of concentrated sulfuric acid (plumbers grade will work). | 9. or 1 small spoon full of baking soda |
| 4. 115 grams (4 oz.) of baking soda | 10. or 1 oz. of regular bakers yeast, or 1 oz. of brewers yeast |
| 5. 5 to 10 grams (just under ½ oz.) of regular yeast (bakers yeast or preferably brewers yeast | 11. 50 grams (1.8 oz.) of baking soda |
| 6. or 500 grams (17.6 oz.) of regular store bought food starch not the "starch" used for clothing | 12. Charcoal (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect., |

Summary: 95% Ethyl alcohol can be obtained on a lower yield by hydrolyzing table sugar with dilute acid, then fermenting the resulting mixture with yeast to form an ethyl alcohol solution. This ethyl alcohol is then distilled to obtain a crude ethyl alcohol product. This crude product will be contaminated heavily with by-products so multiple distillations and treatments with baking soda will be needed in order to fulfill proper purification. Baking soda is mixed with the distilled liquid to absorb odors and the like. 95% Ethyl alcohol can also be obtained by the fermentation of glucose obtained from starch. In this case regular food starch is hydrolyzed

with acid to form a glucose solution, which is then fermented in the usual manner. The fermented mixture is then double distilled under the usual techniques.

Procedure:

If using table sugar:

Dissolve 1 kilogram (2.2 pounds) of table sugar into 3 liters (3.2 quarts) of tap water. Then rapidly stir this sugar mixture to dissolve all solids, and thereafter, heat this sugar solution (using a hot plate, stovetop, Bunsen burner, ect.,) to 80 Celsius. When the sugar solution reaches about 80 Celsius, stir the sugar solution and add in 5 drops of concentrated hydrochloric acid or 5 drops of concentrated sulfuric acid (hydrochloric acid works best), and then continue heating and stirring the sugar solution for thirty minutes (at the end of 15 to 30 minutes, the sugar solution should have a slight grape sugar like odor). After thirty minutes, remove the heat source, and allow the sugar mixture to cool to room temperature. Then add in 5 grams (2/10th of an oz.) of baking soda to neutralize the acid. Afterwards, pour the sugar solution into an empty bottle (such as a clean empty plastic milk jug), and then add in 5 to 10 grams (just under 1/2 oz.) of regular yeast (bakers yeast or preferably brewers yeast). Then stir the mixture for several minutes to insure good dispersion of the yeast. Then plug the bottles opening with cotton or similar materials (but not air-tight), and then place the bottle into a cool place away from light. Then allow the sugar mixture to ferment for about 4 weeks. After 4 weeks, remove the cotton or similar material from the bottles opening, and then pour the contents of the bottle into a distillation apparatus (similar to the one illustrated in method 1). Then distill at 100 Celsius for 4 1/2 hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a clean distillation apparatus (as similar to the one illustrated in 1. Chloroform, method 1, step 3) and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the crude ethyl alcohol from the receiver flask just like before. Finally, place this crude ethyl alcohol mixture into a clean fractional distillation apparatus (like the one illustrated in 3. Diethyl Ether, method 1) followed by 50 grams (1.8 oz.) of baking soda, and then distill the mixture at 80 Celsius until no more ethyl alcohol passes into the receiver flask (takes about 3 to 4 hours). Once the ethyl alcohol has distilled over for this final time, recover the ethyl alcohol from the receiver flask, and then filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether.

If using starch

Into a large beaker or other container, place 500 grams (17.6 oz.) of regular store bought food starch (not the "starch" used for clothing). Thereafter, add in 2 liters (2.1 quarts) of hot tap water, and then add in 10 drops of concentrated hydrochloric acid. Thereafter, heat this starch suspension for about 2 hours at the boiling temperature of water. During the heating process, stir the mixture as much as possible. Keep heating the mixture until the bulk of the starch dissolves. Note: The purpose for the acid is to hydrolyze the starch into glucose. During the heating process, the insoluble starch suspension will slowly disappear to give a clear solution; this is because of the starch being broken down into glucose, which is very soluble in water. When most of the insoluble starch has been dissolved, remove the heat source, and allow the resulting solution to cool to room temperature. When the solution cools, add in 1 small spoon full of baking soda (to neutralize the acid), and then pour the entire mixture into a clean empty plastic milk jug. At this point, you should now have a plastic milk jug filled with just over 2 liters of glucose solution, containing a small amount of baking soda. Now, pour in 1 oz. of regular bakers yeast, or 1 oz. of brewers yeast. Note: brewers yeast works best, and can be purchased from many locations; bakers yeast is satisfactory. When the yeast has been added, don't shake the container; just allow the yeast to settle on its own. After adding in the yeast, plug the top of the plastic milk container with a cloth, cotton, or any thing that is not airtight, but will keep dust out. Thereafter, place your milk jug into a cool dark place, and let it ferment for about 2 to 4 weeks. After 2 weeks, check on it occasionally. If after 2 weeks, you don't see any bubbles rising up, your home brew is finished. In some cases, it may take as long as 4 weeks to complete the fermentation. Note: During the fermentation process, the yeast catalytically reacts with the glucose forming ethyl alcohol, and carbon dioxide. The carbon dioxide bubbles to the surface, and the alcohol remains in solution. Once the fermentation process is complete, it's now time for the hard part—the mixture has to be distilled. To do this, assemble an apparatus similar to the one in method 1, and then distill at 100 Celsius for 4 1/2 hours. After which, remove the heat source, and then recover the crude ethyl alcohol mixture from the receiver flask. Then pour this crude ethyl alcohol mixture into a clean beaker or other container, and then add in 50 grams (1.8 oz.) of baking soda. Thereafter, boil this mixture for about 5 minutes, and thereafter, remove the heat source, and allow the mixture to cool to room temperature. Afterwards, filter the mixture to remove any insoluble baking soda, and then place the filtered mixture into a clean fractional distillation apparatus (as similar to the one illustrated in 3. Diethyl Ether, method 1), and distill at 80 Celsius until no more liquid passes into the receiver flask (takes about 3 to 4 hours). When no more liquid passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the

receiver flask. Then, filter it through a filter (gravity filtration) containing a layer of charcoal. Note: simply place ordinary charcoal briquettes (crush them up into pieces if necessary) into the bottom of the filter paper (store bought charcoal will work, but only ordinary charcoal—nothing added, i.e., no quick light stuff, ect., ect.). Pass the ethyl alcohol through this filter several times to purify it—the charcoal will help remove impurities, odors, and the like. After the charcoal filtration process, your 95% ethyl alcohol is ready for use. Note: if desired, this alcohol can be used to prepare ether.

NOTE: If you don't have access to laboratory glassware (which most people don't), you will need to use your imagination and come up with a homemade "still" of your own. Use your creativity to design a cheap home made "still". One method is screwing a PVC pipe of appropriate size into the opening of a clean soda can, and then inserting the other end of the PVC pipe into an empty plastic container (such as an empty milk jug). Note: Do not drink the distilled alcohol fraction as is; its 90% alcohol and ingestion can cause poisoning.

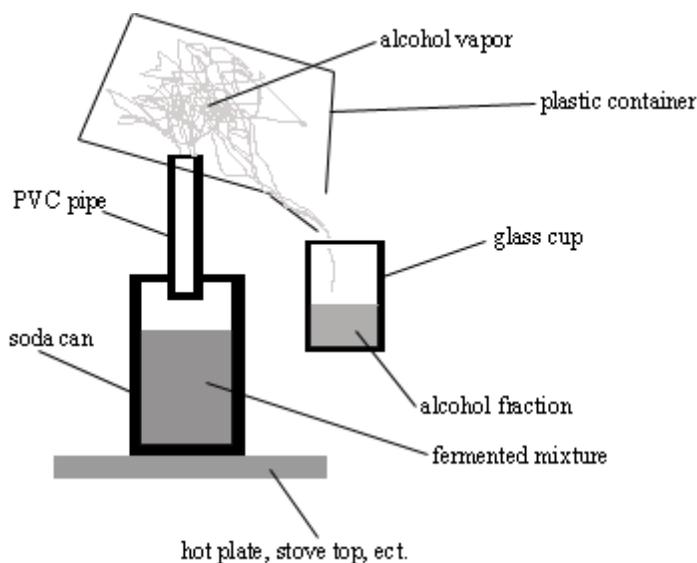


Figure 033. Crude setup for the distillation of ethyl alcohol. This drawing is merely a suggestion, and should be used to get you to use your imagination to come up with something of your own. There are numerous possibilities.

Final note for method 2: None

Method 2B: Preparation of 95% ethyl alcohol from table sugar, brown sugar, molasses, or juice (improved process)

(By-products from reaction: to complex for discussion here)

Materials:

| | |
|---|--|
| 1. 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar | 5. 2 to 4 liter (2.1 to 4.2 quart) container of grape juice, apple juice, or apricot juice |
| 2. 3 liters (3.1 quarts) of warm tap water | 6. 1 kilogram (2.2 pounds) of molasses |
| 3. 1 packet of store bought yeast (7 grams or 1/4 th of an ounce) | 7. 3 liters (3.1 quarts) of warm water |
| 4. 200 grams (7 oz.) of pickling salt | |

Summary: Ethyl alcohol can be prepared by fermenting sugar, brown sugar, juice, or molasses using yeast in a similar manner as in method 2A. In this improved process, the yeast is thoroughly ground into a small amount of warm water prior to mixing with the sugar, brown sugar, juice, or molasses mixture. The fermentation is rapid and only takes about 2 to 3 days under the normal conditions. After the fermentation process, in either case, the fermented mixture is distilled to recover a crude alcohol product. This crude alcohol product is then salted out by mixing it thoroughly with pickling salt to form a two-phase mixture. This two-phase mixture is then separated, and the alcohol fraction is then re-distilled to recover a refined alcohol fraction.

Hazards: None.

Procedure: 95% Ethyl alcohol can be prepared by fermenting sugar, brown sugar, molasses, or juice using yeast in a similar manner to method 2A. However, in the case of sugar, instead of hydrolyzing the sugar, it is fermented directly using store bought yeast used

for baking. Now, if using sugar or brown sugar, dissolve 1 kilogram of sugar (2.2 pounds) or 1 kilogram (2.2 pounds) of brown sugar into 3 liters (3.1 quarts) of warm tap water contained in a washed out empty milk gallon jug, and then prepare a yeast base mixture by doing the following: 1. take 1 packet of store bought yeast (7 grams or 1/4th of an ounce) and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like. Thereafter, pour the yeast mixture into the sugar solution, and then place a paper towel or cloth over the mouth of the gallon jug, and then allow the mixture to ferment for about 3 to 4 days at room temperature. Note: once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 48 to 62 hours. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. After the fermentation process, the mixture needs to be distilled using a similar apparatus as in method 1. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the crude alcohol mixture should then be "Salted out" using the following process: 1. Place the crude alcohol mixture into a separatory funnel (see extraction), and then add in 200 grams (7 oz.) of pickling salt, and then shake the mixture vigorously for about 5 to 10 minutes. Thereafter, allow the mixture to settle and stand for about 15 minutes, whereupon a two-phase mixture will develop. The upper layer will be the alcohol layer, and the water layer containing dissolved pickling salt and other impurities, will be the lower layer. Now, simply drain-off the lower water layer, and discard, or you can keep it to recover the dissolved salt, and then drain-off or pour out the upper alcohol layer. Then place this alcohol layer into a clean distillation apparatus of the usual design, as illustrated in method 1, and then distill the alcohol at 78 Celsius. The resulting alcohol may not be exactly 95%, but is well suitable for use in applications as a solvent.

If using molasses, or juice, simply prepare a yeast mixture by taking 1 packet of store bought yeast (7 grams or 1/4th of an ounce) and place it into 1/4th of a cup of warm water, and then using a spoon or similar utensil, grind and thoroughly mix the yeast into the water until a uniform mixture results, which is free from clumps, chunks, or the like, and then pour this yeast mixture directly into a large (2 to 4 liter, 2.1 to 4.2 quart) container of grape juice, apple juice, apricot juice, or a molasses solution, and then allow the mixture to ferment for about 3 to 4 days. As in the above process, once the yeast gets going, in about an hour or so the evolution of carbon dioxide will become vigorous. The total amount of time for the fermentation process ranges from about 48 to 62 hours. When the evolution of carbon dioxide gas bubbles slows down and drops-off to a trickle, the fermentation process is relatively complete. Note: if using juice, pour out some of juice before adding the yeast to create a gap or space between the juice and the mouth of the container to prevent foaming and frothing from escaping the bottle once the yeast kicks in. Second not: If using molasses, dissolve 1 large container of molasses (about 1 kilogram, 2.2 pounds), into 3 liters (3.1 quarts) of warm water contained in a clean empty milk jug or equivalent. If using molasses, you will want to carryout the procedure in a well-ventilated area as the odor of molasses will be heavy. After the fermentation process, the fermented juice or molasses mixture needs to be distilled using a similar apparatus as in method 1. The distillation process will take about 5 to 6 hours for completion. After the distillation process, the crude alcohol mixture should then be "Salted out" using the following process: 1. Place the crude alcohol mixture into a separatory funnel (see extraction), and then add in 100 to 200 grams (3.5 to 7 oz.) of pickling salt, and then shake the mixture vigorously for about 5 to 10 minutes. Thereafter, allow the mixture to settle and stand for about 15 minutes, whereupon a two-phase mixture will develop. The upper layer will be the alcohol layer, and the water layer containing dissolved pickling salt and other impurities, will be the lower layer. Now, simply drain-off the lower water layer, and discard, or you can keep it to recover the dissolved salt, and then drain-off or pour out the upper alcohol layer. Then place this alcohol layer into a clean distillation apparatus of the usual design, as illustrated in method 1, and then distill the alcohol at 78 Celsius. The resulting alcohol may not be exactly 95%, but is well suitable for use in applications as a solvent.

Method 3: Preparation of 99% ethyl alcohol from 95% ethyl alcohol and metallic sodium

(By-products from reaction: sodium hydroxide and a small amount of sodium ethoxide)

Materials:

| | |
|--|--|
| 1. 500 milliliters (17 fluid oz.) of 95% ethyl alcohol | 2. 40 grams (1.4 oz.) of metallic sodium |
|--|--|

Summary: 99% ethyl alcohol is obtained by treating 95% ethyl alcohol with metallic sodium. The metallic sodium reacts with the water in the binary azeotrope, freeing the alcohol. Once the water has been neutralized (forming sodium hydroxide and sodium ethoxide), the mixture is distilled to recover the anhydrous ethyl alcohol.

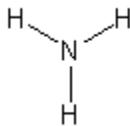
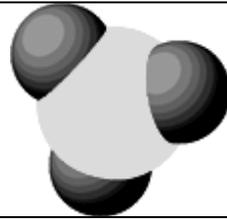
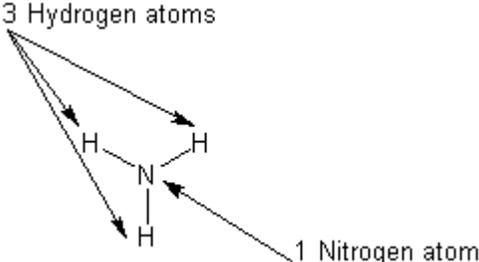
Hazards: Wear gloves when handling metallic sodium, and avoid skin contact and ingestion. Metallic sodium reacts violently with water and other chemicals, so use caution.

Procedure: Into a suitable beaker or container, place 500 milliliters (17 fluid oz.) of 95% ethyl alcohol, and then place the beaker or container into an ice bath, and chill to about 5 to 10 Celsius. Thereafter, slowly add in, in very small portions, 40 grams (1.4 oz.) of metallic sodium over a period of about 1 hour. After the addition of the metallic sodium, stir the entire alcohol mixture for about 30 minutes, and then place it into a standard distillation apparatus (as similar to 1. Chloroform, method 1, step 3), and distill the ethyl

alcohol at 80 Celsius. Note: your distillation apparatus should be completely dry before proceeding. When no more ethyl alcohol passes into the receiver flask, remove the heat source, and then recover the ethyl alcohol from the receiver flask. The resulting alcohol will be about 99% pure, and should be stored in a suitable container protected from moisture.

Final note for method 3: None

Preparation 5: Anhydrous Ammonia

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
| <p>3 Hydrogen atoms</p>  <p>1 Nitrogen atom</p> | <p>NH₃</p> |
| Structure make-up | Short hand chemical structure |

Ammonia, anhydrous

Anhydrous ammonia is a colorless gas with a very pungent odor. It has a melting point of -77 Celsius, and a boiling point of -33 Celsius. Ammonia is regarded as nonflammable, but mixtures with oxygen can ignite (when passed over red-hot platinum resulting in nitrogen dioxide—see nitric acid *vide supra*). Anhydrous ammonia is a corrosive alkaline gas that is very soluble in water. It is also soluble in alcohol, chloroform, and ether. Liquid ammonia is a good solvent for many elements and compounds. Commercial anhydrous ammonia is supplied in the form of a compressed gas in steel tanks, or in the liquid form supplied in steel tanks. Ammonia is also widely sold in water solutions. Anhydrous ammonia is the 4th largest chemical produced in the US. Anhydrous ammonia is a widely available commercial chemical. Anhydrous ammonia is prepared on an industrial scale from hydrogen and nitrogen at high pressure and temperature in the presence of platinum. The average ammonia plant produces 1000 tons of liquid ammonia per day.

Method 1: Preparation of anhydrous ammonia from store bought commercial ammonia solution (for cleaning) with lye and acid

(By-products from reaction: sodium chloride or sodium or potassium sulfate)

Materials:

| | |
|---|---|
| 1. 156 grams (5.5 oz.) of 10% ammonia (household ammonia) | 3. 38 grams (1.3 oz.) of sodium hydroxide or 53 grams (1.9 oz.) of potassium hydroxide |
| 2. 100 grams (3.5 oz.) of 35% hydrochloric acid, or 47 grams (1.7 oz.) of 98% sulfuric acid | 4. 38 milliliters (1.3 fluid oz.) of tap water or 53 milliliters (1.8 fluid oz.) of tap water |

Summary: Anhydrous ammonia can be prepared by treating an ammonium salt with a warm solution of sodium hydroxide, or potassium hydroxide. In the following procedure, the ammonium salt is either ammonium chloride or ammonium sulfate, which are first prepared by reaction of household ammonia with the corresponding acid. Thereafter, the resulting ammonium salt is then treated with sodium hydroxide, or potassium hydroxide to yield ammonia gas, which is then dried to yield the anhydrous gas.

Hazards: Wear gloves when handling 50% sodium hydroxide, or 50% potassium hydroxide. The alkaline solutions may cause painful skin burns on prolonged exposure, and itching sensation on short exposure. Use proper ventilation when making ammonia gas, and avoid inhalation of the vapors.

Procedure: Place 156 grams (5.5 oz.) of 10% ammonia into a flask and then rapidly add 100 grams (3.5 oz.) of 35% hydrochloric acid, or 47 grams (1.7 oz.) of 98% sulfuric acid while stirring the 10% ammonia solution. After the addition of the acid, continue stirring for ten minutes, and then recrystallize the ammonium salt from solution (simply boil-off the water until crystallization begins). After all the desired ammonium salt has been collected, dry it using vacuum drying, or let air-dry. Afterwards, place the dry ammonium salt into an apparatus as illustrated below, and then prepare a sodium hydroxide solution by dissolving 38 grams (1.3) of sodium hydroxide into 38 milliliters (1.3 fluid oz.) of water, or 53 grams (1.9 oz.) of potassium hydroxide into 53 milliliters (1.8 fluid oz.) of water (much heat is produced when dissolving sodium or potassium hydroxide into water). After the sodium or potassium hydroxide solution has cooled some, add it to the dry ammonium salt, drop-wise, over a period of about thirty minutes. During the addition of the sodium hydroxide or potassium hydroxide, ammonia gas will be steadily evolved.

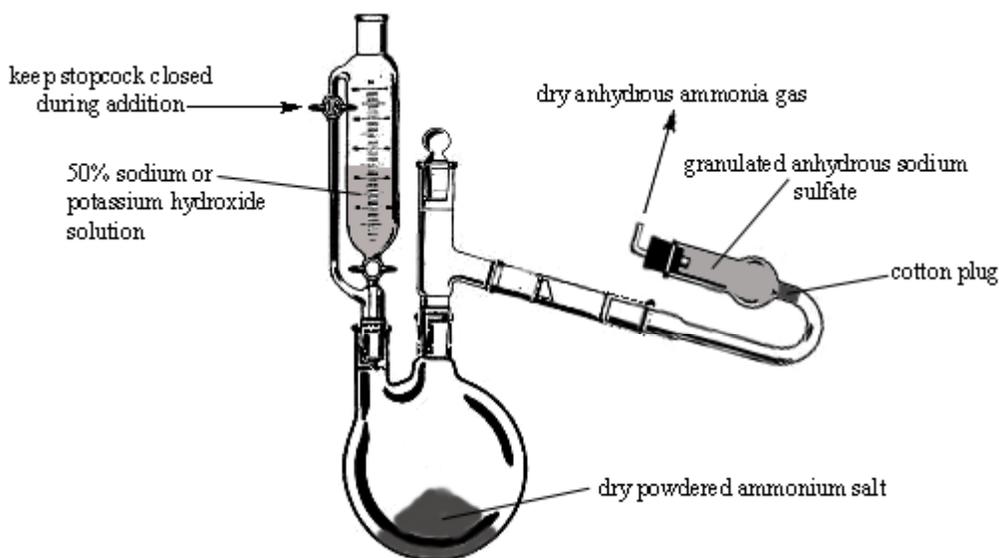
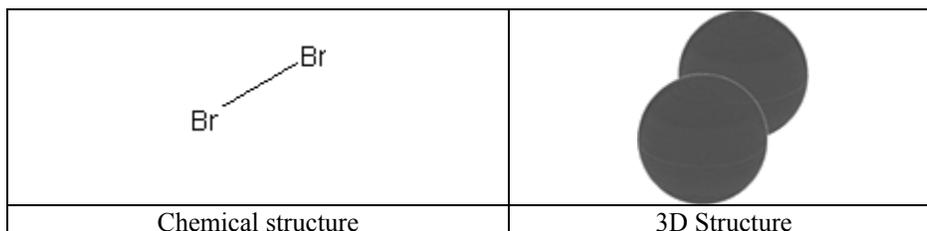
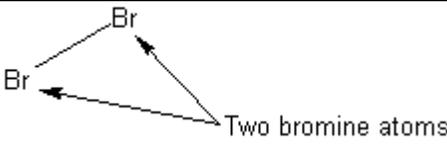


Figure 034. Apparatus for the preparation of anhydrous ammonia.

Final note for method 1: Like most of the apparatus illustrated in this book, the apparatus illustrated in figure 027 can be replaced by any similar set-up. It should also be noted that the illustrations in this book are merely suggestive drawings, and do not represent the actual design. Design and function of apparatus can vary, and the illustrations given in this book should be used to define or describe the necessary set-up only to the point of design.

Preparation 6: Bromine



| | |
|---|-------------------------------|
|  | Br ₂ |
| Structure make-up | Short hand chemical structure |

Bromine

Bromine is a dark red, highly fuming liquid, which is very volatile. Its fumes are toxic, corrosive, and strongly irritating. Bromine has a melting point of -7 Celsius, and a boiling point of 59 Celsius. It is insoluble in water, but freely soluble in alcohol, ether, chloroform, and carbon disulfide. It is soluble in alkali bromide solutions. Bromine is less reactive than chlorine, but just as toxic. Keep bromine stored in glass stoppered bottles, and store in a cool place (refrigerator) away from sunlight. Bromine is prepared by passing chlorine gas into a solution of sodium bromide, and then simultaneously evaporating-off the bromine. The bromine vapors are then condensed. Bromine is commercially available but shipping regulations may restrict its sale.

Method 1: Preparation of bromine from sodium bromide and chlorine

(By-products from reaction: sodium chloride or potassium chloride, and small amounts of monobromochloride – ClBr -)

Materials:

| | |
|--|---|
| 1. 36 grams (1.3 oz.) of potassium bromide or 31 grams of (1.09 oz.) of sodium bromide | 5. 40 milliliters (1.35 fluid oz.) of tap water |
| 2. 150 milliliters (5 oz.) of tap water | 6. 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid |
| 3. 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Note: if using bleach, some recent brands of bleach now contain sodium hydroxide. Avoid these brands of bleach as they will disrupt the reaction. Generic bleach may work best. | 7. 200 milliliters (7 fluid oz.) of cold tap water |
| 4. 30 grams (1 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) | |

Summary: bromine is readily prepared by bubbling chlorine gas into a solution of sodium or potassium bromide in water. The reaction is very mild, and liquid bromine is steadily evolved, forming at first, a deep red liquid that gives off red fumes. The bromine vapors are carried over and condense into liquid bromine.

Hazards: Use maximum ventilation when performing this procedure as both chlorine and bromine are toxic and very irritating to the eyes, nose, and throat—use caution.

Procedure: Setup the chlorine generating apparatus as illustrated in Preparation 2: Iodine, method 3. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion. Also, observe the chlorine generator utilizing an electrochemical method. The electrochemical method is sufficient to produce laboratory quantities of gas (see the illustration).

Now, into a suitable beaker, flask, or container, place 36 grams (1.3 oz.) of potassium bromide or 31 grams of (1.09 oz.) of sodium bromide, followed by 150 milliliters (5 oz.) of tap water. Thereafter, stir the entire mixture for about 30 minutes to dissolve all solids.

After you have the chlorine generator set-up (just like in preparation 2: Iodine, method 3), place into the reaction flask, 15 grams (1/2 oz.) of manganese dioxide, or 240 grams (8.5 oz.) of a 5% sodium hypochlorite solution (Clorox bleach), or 13 grams (1/2 oz.) of potassium permanganate, or 19 grams (0.67 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 30 grams (1 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) into 40 milliliters (1.35 fluid oz.) of tap water. If you are using an electrochemical cell, fill your cell with 40 milliliters (1.4 fluid oz.) of concentrated hydrochloric acid, followed by 200 milliliters (7 fluid oz.) of cold tap water.

Once your chlorine generator is setup (if using the hydrochloric acid drip method), begin dripping the hydrochloric acid solution, onto the oxidizer contained in the reaction flask. If using an electrochemical cell, begin the electrolysis by plugging in your battery charger. In either case, the chlorine gas that is evolved should then be sent to the apparatus as illustrated below for the preparation of bromine. The chlorine gas will react with the potassium or sodium bromide forming bromine. When no more chlorine gas passes over, in either case, the reaction is complete.

After the initial reaction, the collected liquid bromine should be fractionally distilled to free it from dissolved gases and moisture. The distilled bromine should then be stored in amber glass bottles in a cool place.

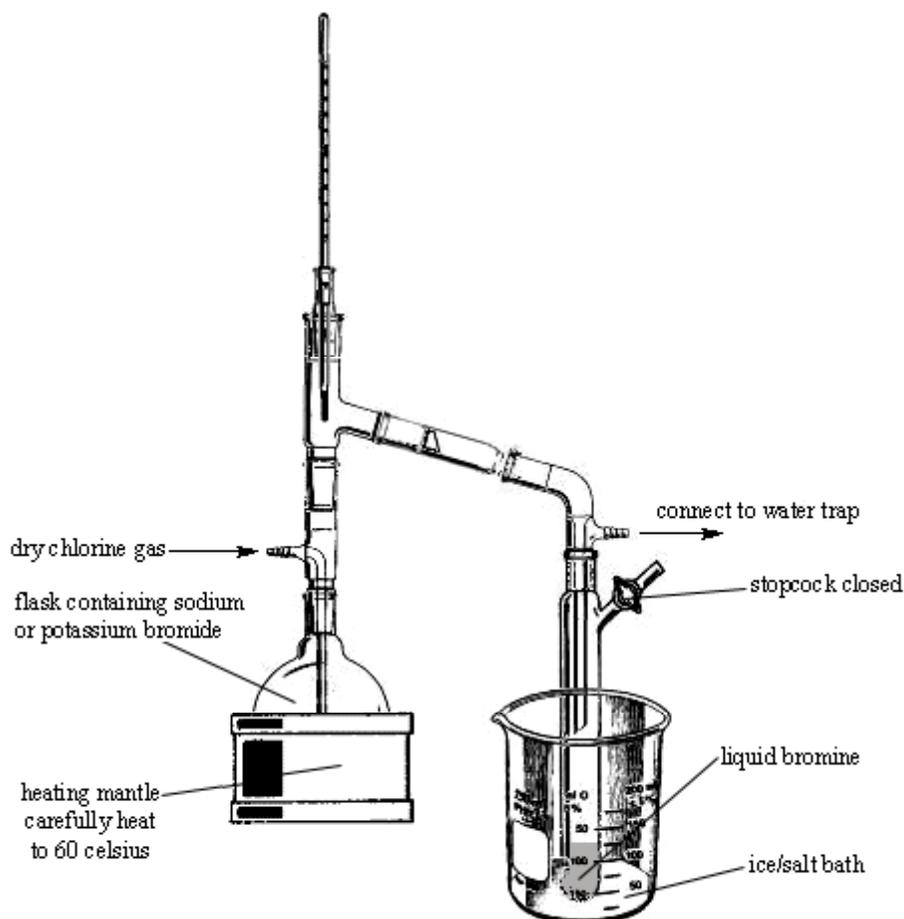
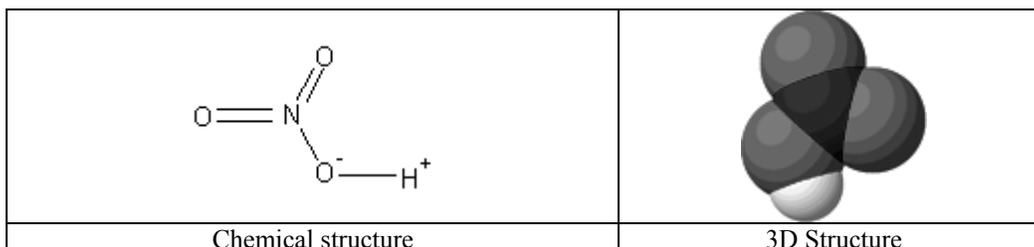
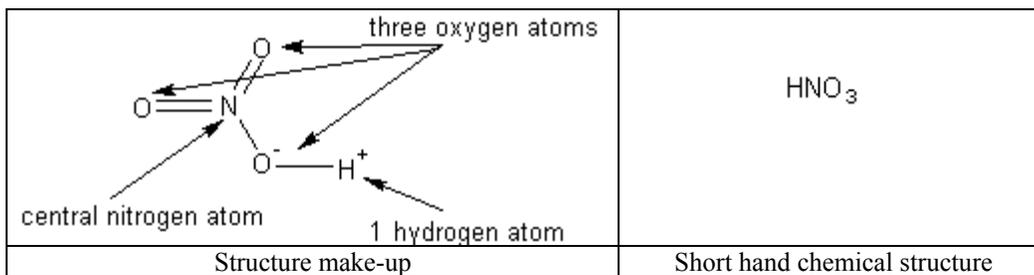


Figure 035. Apparatus for preparing bromine. The bromine should be fractionally distilled at 60 Celsius. Note: the apparatus should be connected to a water trap containing a sodium carbonate solution to neutralize any bromine vapors that may escape. Second note: This apparatus is merely an example, and is not intended to be the “exact” set-up.

Preparation 7: Nitric Acid





70% Nitric acid (65 to 68% acid) *Aqua fortis*; *Salpetersaure*

70% Nitric acid is referred to as concentrated nitric acid, and has a concentration of 65 to 71% nitric acid by weight (simply called 70% nitric acid). 70% nitric acid is a colorless liquid, which fumes in moist air yielding a choking vapor. Its melting point is -41 Celsius with a boiling point of 85 Celsius. When boiling 70% nitric acid, it forms a constant boiling mixture with water forming 68% nitric acid with a boiling point of 120 Celsius. 70% nitric acid should be stored in airtight glass bottles away from sunlight. It stains fabrics, and animal tissue a bright yellow. In contact with the skin, it produces a yellow stain. 70% Nitric acid is a powerful oxidizing acid. It reacts violently with many different substances including alcohols, turpentine, charcoal, and organic refuse. 70% Nitric acid begins to turn yellow in contact with strong light sources such as sunlight or magnesium light. 70% Nitric acid is a widely available commercial acid. It is the 7th largest manufactured chemical in the US. Many chemical dealers will not sell 70% nitric acid to unlicensed persons because of its use in manufacturing high explosives. Concentrated nitric acid is prepared on an industrial scale by the oxidation of ammonia with air in a special ammonia burner, utilizing platinum as catalyst. ***Wear gloves and use proper ventilation when handling 70% nitric acid. 70% Nitric acid can cause skin burns and can chemically react with clothing. Use caution when handling.***

90% fuming nitric acid

90% Nitric acid is a yellow to brownish-red, strongly fuming liquid. It is a very poisonous and corrosive liquid, which evolves poisonous, choking fumes of nitrogen oxides. It is a powerful oxidizer, and should be kept stored in glass, or Teflon containers, in a cool dry place and away from light (preferably in a refrigerator). 90% Nitric acid reacts violently with many substances. The acid is commercially available, but shipping regulations and dealer restrictions may limit its sale. In most cases, 90% nitric acid is not available to the general public. ***Wear gloves and use maximum ventilation when handling this substance.***

99% Nitric Acid

99% nitric acid is a colorless (when freshly prepared, i.e., white fuming acid), highly fuming, and poisonous liquid. It turns yellow to dark-red on standing, and has a melting point of -41.59 Celsius. 99% nitric acid is a powerful oxidizer, and it reacts violently with many substances. For the most part, the acid should be used right after it's preparation, but the fuming acid can be stored in tightly sealed amber glass bottles in a cool place for a week or more. 99% Nitric acid is commercially available, but shipping regulations and dealer restrictions will definitely restrict its sale and shipment to most people. ***Warning! 99% Nitric acid is a very poisonous and corrosive liquid, which evolves large amounts of poisonous fumes. Wear gloves and proper laboratory clothing (lab coat; boots, face shield) when handling this substance and use maximum ventilation.***

Method 1: Preparation of 70% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

| | |
|---|---|
| 1. 194 grams (6.8 oz.) of 98% sulfuric acid | 4. 1050 milliliters of methylene chloride (2.2 pints) |
| 2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate | 5. 27 milliliters (1 fluid oz.) of tap water |
| 3. 260 milliliters (8.8 fluid oz.) of cold water | |

Summary: 70% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water, and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid. This methylene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then carefully distilled to remove the methylene chloride and leave behind 70% nitric acid.

Hazards: Methylene chloride/99% nitric acid mixture is very volatile and dangerous—wear gloves, and use proper ventilation. 99% nitric acid is highly toxic, and corrosive, which evolves poisonous fumes—use maximum ventilation. Perform the distillation using proper ventilation, and protect from direct sun light.

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water—if a precipitate forms, never mind it). Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.0 fluid oz. portions) of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then add 27 milliliters (1 fluid oz.) of tap water to the methylene chloride. Next, place the entire methylene chloride (two phase) mixture into a distillation apparatus and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. *Note: After all the methylene chloride has been removed, the 70% nitric acid may have a slight yellowish to red tint. If this is the case, don't worry—go ahead and use this nitric acid as is.*

Method 2: Preparation of 90% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

| | |
|---|---|
| 1. 194 grams (6.8 oz.) of 98% sulfuric acid | 3. 260 milliliters (8.8 fluid oz.) of cold tap water |
| 2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate | 4. 1050 milliliters of methylene chloride (2.2 pints) |

Summary: 90% Nitric acid can be obtained by treating potassium or sodium nitrate with sulfuric acid, mixing with water and then extracting the acid mixture with methylene chloride. The result is a methylene chloride solution containing 99% nitric acid. This methylene chloride/99% nitric acid solution is then mixed with a calculated amount of water, and then distilled to remove the methylene chloride and produce 90% nitric acid.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 90% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. *Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.*

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in an ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water. If this happens, never mind it). Then remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.07 fluid oz. portions) of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so), and then add 7 milliliters (0.23 fluid oz. or 1.4 tsp) of water to the methylene chloride. Then place the entire methylene chloride (two phase) mixture into a distillation apparatus (see the illustration in method 3 below), and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

Method 3: Preparation of 99% nitric acid from 70% Nitric acid or lower concentrations of nitric acid utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: none)

Materials:

| | |
|--|---|
| 1. 94 milliliters (3.1 fluid oz.) of 98% sulfuric acid | 3. 1400 milliliters of methylene chloride (3 pints) |
| 2. 106 milliliters (3.6 fluid oz.) of 70% nitric acid | |

Summary: 99% Nitric acid can be prepared from 70% nitric acid or any other lower concentration of nitric acid by adding the nitric acid to excess sulfuric acid, and then extracting with methylene chloride. The upper methylene chloride layer is recovered, and then carefully distilled at 40 Celsius to remove the methylene chloride and recover the 99% nitric acid.

Hazards: Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. *Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.*

Procedure: Into an appropriate sized beaker place 94 milliliters (3.1 fluid oz.) of 98% sulfuric acid, and then 106 milliliters (3.6 fluid oz.) of 70% nitric acid. Then extract this acid mixture with seven 200-milliliter portions (seven 6.7 fluid oz. portions) of methylene chloride. Afterwards, combine all seven portions of methylene chloride (if not already done so). The result will be a 99% nitric acid solution in methylene chloride. This methylene chloride/nitric acid mixture can be used directly in general chemistry applications (if desired), or separated to recover pure 99% nitric acid. To separate the mixture, place the mixture into a distillation apparatus (as illustrated below), and then carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. **Note:** *Take caution when distilling and use proper ventilation because decomposition of the nitric acid might result forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid.*

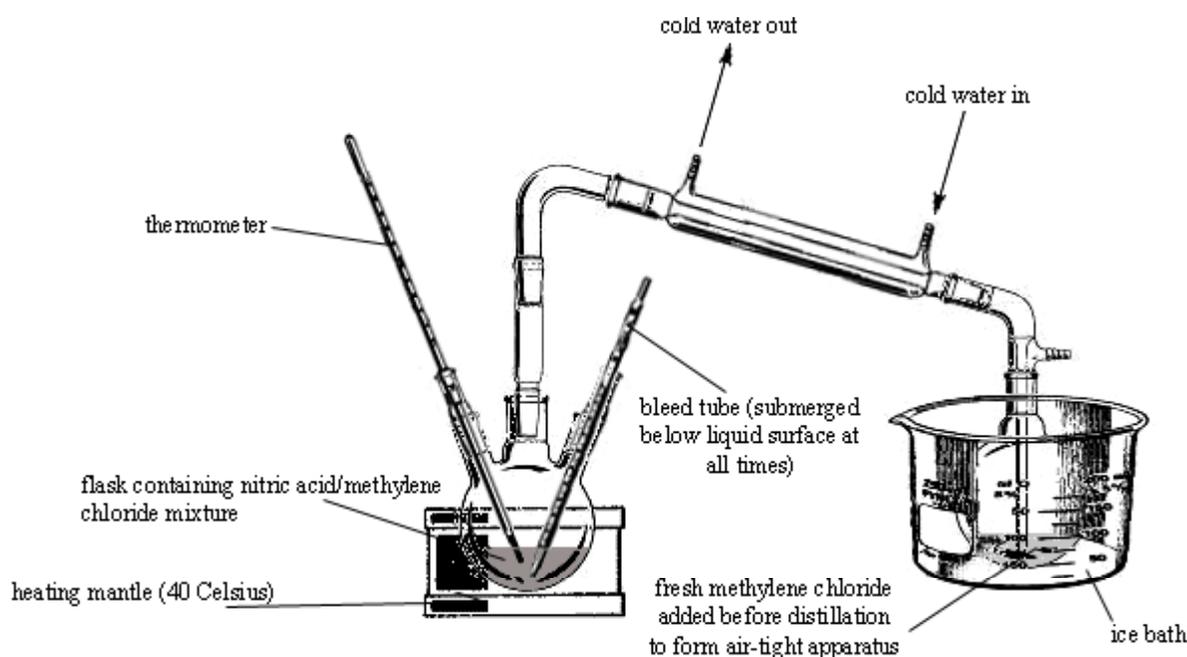


Figure 036. Apparatus for the distillation of methylene chloride to collect pure nitric acid. The receiver flask should contain some fresh methylene chloride so as to form an air-tight seal by submerging the exit tube below the liquid surface. When the methylene chloride has been removed, the pure nitric acid will remain in the distillation flask.

Note: Any concentration of nitric acid can be used instead of just 70% nitric acid. When using nitric acid concentrations below 70% by weight, simply mix the dilute nitric acid with excess sulfuric acid. For example, mix 57 grams (2.01 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 60% nitric acid, mix 67grams (2.4 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 50% nitric acid, mix 77 grams (2.7 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 40% nitric acid, mix 87 grams (3.06 oz.) of sulfuric acid with 53 milliliters (1.8 fluid oz.) of 30% nitric acid, mix 97 grams (3.4 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 20% nitric acid, or mix 107 grams (3.8 oz.) of 98% sulfuric acid with 53 milliliters (1.8 fluid oz.) of 10% nitric acid. After anyone of these mixings, extract the acid mixture with seven 100-milliliter portions (seven 3.4 fluid oz. portions) of methylene chloride. Thereafter, the result will be the same as in the above procedure. To isolate the 99% nitric acid, follow the directions in the above procedure.

Method 4: Preparation of 99% nitric acid from potassium or sodium nitrate utilizing sulfuric acid and extraction with methylene chloride

(By-products from reaction: sodium or potassium sulfate)

Materials:

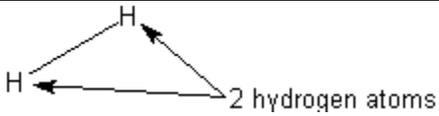
| | |
|---|---|
| 1. 194 grams (6.8 oz.) of 98% sulfuric acid | 3. 260 milliliters (8.8 fluid oz.) of cold tap water |
| 2. 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate | 4. 1050 milliliters of methylene chloride (2.2 pints) |

Summary: 99% Nitric acid can also be obtained by treating potassium or sodium nitrate with sulfuric acid, adding water, and then extracting the acid mixture with methylene chloride to form a solution of 99% nitric acid in methylene chloride. The methylene chloride is then carefully distilled off to recover the pure 99% nitric acid.

Hazards: Take caution when distilling and use proper ventilation because decomposition of the nitric acid might take place forming reddish-brown fumes of nitrogen oxides. If the nitric acid begins to decompose during the distillation, don't worry and continue the distillation. After all the methylene chloride has been removed, the result might be a reddish-brown highly fuming liquid. This reddish-brown fuming liquid is suitable for use as 99% nitric acid. Carryout the distillation with extreme caution. Do not heat the methylene chloride/nitric acid mixture above 40 Celsius, and use proper ventilation. *Nitrogen oxide gases will develop so be prepared. Carryout the distillation away from direct sun light.*

Procedure: Place 194 grams (6.8 oz.) of 98% sulfuric acid into a beaker, and then place the beaker in a ice bath and cool to 0 Celsius by means of an ice bath. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 100 grams (3.5 oz.) of potassium nitrate or 84 grams (3 oz.) of sodium nitrate over a period of 1 hour while stirring the 98% sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 260 milliliters (8.8 fluid oz.) of cold tap water while continuously stirring the 98% sulfuric acid mixture and maintaining its temperature at 0 Celsius. Afterwards, remove the ice bath and then extract the acid mixture with seven 150-milliliter portions (seven 5.07 fluid oz. portions) of methylene chloride. Then combine all seven portions of methylene chloride (if not already done so), and then place the methylene chloride into the distillation apparatus as illustrated in method 2, and carefully distill at 40 Celsius until no more methylene chloride is collected in the receiver flask.

Preparation 8: Hydrogen gas

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | H ₂ |
| Structure make-up | Short hand chemical structure |

Hydrogen

Hydrogen is a colorless, tasteless, highly flammable and explosive gas. Hydrogen is a strong reducing agent when in the presence of a suitable catalyst such as platinum, palladium, nickel, and the like. Hydrogen gas is very difficult to handle and store, but can be stored in compressed cylinders; these cylinders are often hard to purchase and ship, but hydrogen can easily be prepared by reacting dilute hydrochloric acid with zinc, or aluminum, and collecting the resulting hydrogen gas. Note: iron should be avoided as it contains impurities leading to the formation of metal hydrides such as arsine and stibine, which contaminate the hydrogen (gives the hydrogen a strange metallic like garlic odor). Hydrogen can also be obtained by the electrolysis of acidic or basic water solutions, or by treating a 25% sodium hydroxide (lye) solution with aluminum foil—this latter reaction is utilized by several dry drain opener products to open and clear clogged drains.

Method 1: Preparation of Hydrogen gas from hydrochloric acid or sulfuric acid and a metal, or by reaction of lye with aluminum foil.

(By-products from reaction: metal chlorides, or sodium aluminate)

Materials:

| | |
|--|---|
| 1. 150 grams (5.3 oz.) of concentrated hydrochloric acid | 6. 10 grams (0.35 oz.) of aluminum |
| 2. 13 grams (1/2 oz.) of aluminum | 7. 17 grams (1/2 oz.) of zinc |
| 3. 45 grams (1.6 oz.) of zinc | 8. 50 grams (1.8 oz.) of lye (sodium hydroxide), |
| 4. 75 milliliters (2.5 fluid oz.) of ice cold tap water | 9. 150 milliliters of ice-cold tap water |
| 5. 50 grams (1.8 oz.) of concentrated sulfuric acid | 10. 11 grams (0.4 oz.) of aluminum (soda pop cans or aluminum foil) |

Summary: Hydrogen gas is easily prepared by reacting hydrochloric acid or sulfuric acid with zinc or aluminum, or by reacting lye with aluminum foil. If using hydrochloric acid or sulfuric acid, avoid metal foils or finely divided metals such as aluminum foil, or zinc dust, as the reaction will be quite violent. For aluminum, aluminum pop cans house the best source, as they are cheap and readily available.

Hazards: Hydrogen is highly flammable and explosive. Mixtures of hydrogen with air when confined will explode when ignited.

Procedure:

Using Hydrochloric acid:

Into the reaction flask of the below illustration, place 150 grams (5.3 oz.) of concentrated hydrochloric acid. Thereafter, slowly add, in small portions at a time, 13 grams (1/2 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 45 grams (1.6 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

Using Sulfuric acid

Into the reaction flask of the below illustration, place 75 milliliters (2.5 fluid oz.) of ice cold tap water, and then carefully and slowly add in 50 grams (1.8 oz.) of concentrated sulfuric acid. Thereafter, allow the acid mixture to cool (as adding sulfuric acid to water generates excessive heat), and then slowly add, in small portions at a time, 10 grams (0.35 oz.) of aluminum, preferably aluminum pieces made by carefully cutting-up a clean aluminum can, or 17 grams (1/2 oz.) of zinc. After adding each piece of aluminum or zinc, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

Using Lye (sodium hydroxide)

Into a clean beaker, or other suitable glass container, place 50 grams (1.8 oz.) of lye (sodium hydroxide), followed by 150 milliliters of ice-cold tap water. Note: the addition of sodium hydroxide to water generates excessive heat, so allow the solution to cool before using. Thereafter, place the cooled alkaline mixture into the reaction flask of the below illustration. Then slowly add, in small portions at a time, 11 grams (0.4 oz.) of aluminum, preferably aluminum foil pieces. After adding each piece of aluminum, a steady stream of hydrogen gas will develop. Use a cold-water bath to maintain the temperature of the reaction mixture at a safe level. The hydrogen gas that is evolved can be used for many applications. Have some fun with the hydrogen by filling up a party balloon with the gas. Then carefully make contact with the balloon with the flame of a candle!

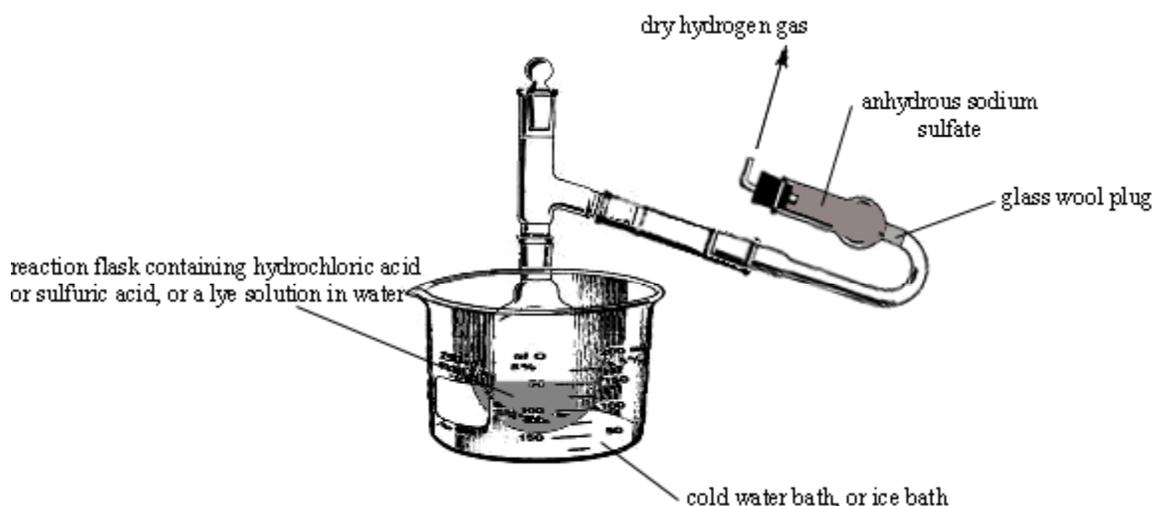
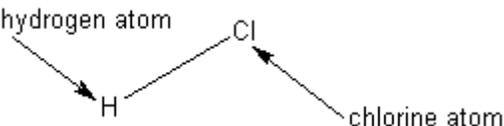


Figure 037. Apparatus for the generation of hydrogen gas.

Electrolysis of water

To collect hydrogen gas by utilizing the electrolysis of water, set-up a similar apparatus as illustrated in Preparation 2: Iodine, method 3 for the electrolysis of hydrochloric acid. However, replace the graphite anode (+) with iron, and replace the hydrochloric acid with an Epsom salt solution prepared by adding and dissolving 100 grams (3.5 oz.) of Epsom salt into 500 milliliters (17 fluid oz.) of warm tap water. Thereafter, electrolysis the mixture at a current ranging from 6 to 12 volt at 2 to 6 amp (use a battery charger). During the electrolysis, a simple, yet moderately complex array of chemical reactions will take place producing hydrogen gas and a steady mushy precipitate of hydrated iron hydroxide (greenish to yellowish-black). The hydrogen gas that is evolved can be used for many applications in the usual manner.

Preparation 9: Anhydrous Hydrogen Chloride gas

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | HCl |
| Structure make-up | Short hand chemical structure |

Anhydrous Hydrogen chloride

Anhydrous hydrogen chloride is a very corrosive, non-flammable gas, with a highly irritating vapor. Hydrogen chloride is very soluble in water, forming a fuming liquid; vide supra, hydrochloric acid. It has a melting point of -114 Celsius, and a boiling point of -85 Celsius. It is prepared by the reaction of concentrated sulfuric acid upon table salt (preferably pickling salt), or by the action of chlorine upon organic compounds. The latter being the chief source of hydrochloric acid.

Method 1: Preparation of Anhydrous Hydrogen Chloride gas from pickling salt (sodium chloride) and sulfuric acid

(By-products from reaction: sodium sulfate, and sodium bisulfate)

Materials:

| | |
|---|--|
| 1. 50 grams (1.8 oz.) of concentrated sulfuric acid | 2. 60 grams (2.1 oz.) of pickling salt |
|---|--|

Summary: Anhydrous hydrogen chloride is readily prepared by reacting concentrated sulfuric acid with pickling salt. During the reaction, hydrogen chloride gas is steadily evolved.

Hazards: Perform the reaction in a well-ventilated area, as hydrogen chloride vapors are highly irritating to the nose and throat.

Procedure: Simply place 50 grams (1.8 oz.) of concentrated sulfuric acid into the addition funnel, and then place 60 grams (2.1 oz.) of pickling salt, into the reaction flask. Thereafter, slowly drip the concentrated sulfuric acid onto the pickling salt. During the sulfuric acid addition, hydrogen chloride gas will be steadily evolved. Once the gas has been dried, it can be used in the manufacture of anhydrous aluminum chloride, in the preparation of hydrochloride salts, and/or the preparation of anhydrous metal chlorides.

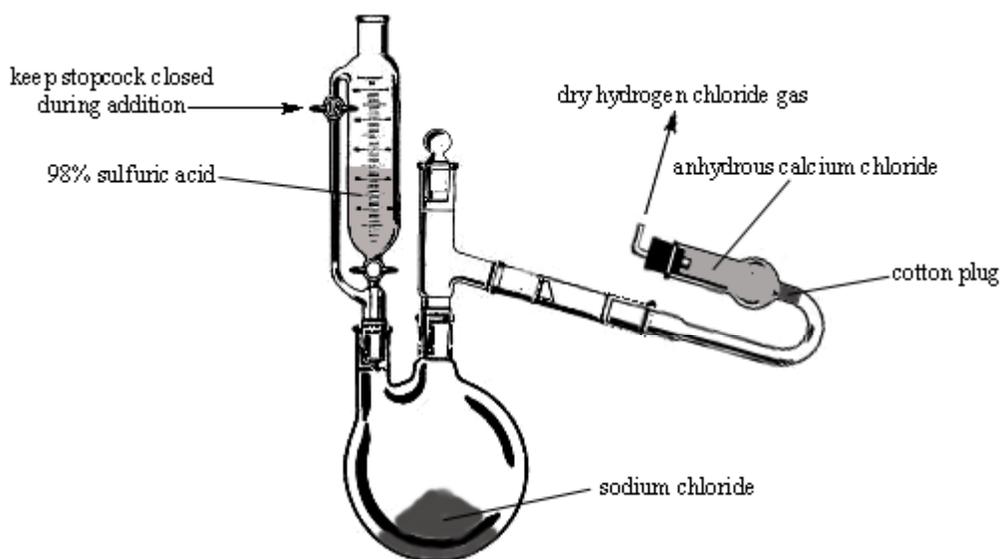
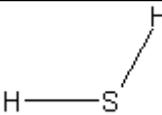
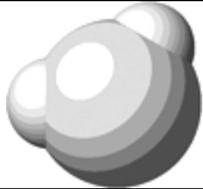
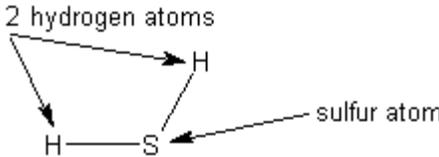


Figure 038. Set-up for the preparation of anhydrous hydrogen chloride gas. Note: gently heating the reaction flask can help speed up the reaction.

Preparation 10: Anhydrous Hydrogen Sulfide gas

| | |
|--|--|
| | |
|--|--|

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | H ₂ S |
| Structure make-up | Short hand chemical structure |

Hydrogen sulfide gas

Hydrogen sulfide is a flammable, poisonous gas with disagreeable odor of rotten eggs. It can be detected by the human nose in extremely small quantities. Hydrogen sulfide has a sweetish taste. Hydrogen sulfide burns in air with a pale blue flame. It has a melting point of -85 Celsius, and a boiling point of -60 Celsius. Hydrogen sulfide is insoluble in water, and not very soluble in alcohol. It is soluble in glycerol, gasoline, kerosene, carbon disulfide, and crude oil. Hydrogen sulfide is a highly toxic gas, and inhalation can be fatal. Use maximum ventilation when handling. It is evolved from many different natural environmental sources including bacterial decomposition of vegetable and animal proteins, natural springs, natural gas deposits, and volcanoes. Hydrogen sulfide can be obtained from the distillation of petroleum. Hydrogen sulfide is prepared in the laboratory by dropping an acid (usually sulfuric or hydrochloric) onto a metal sulfide such as sodium sulfide, or calcium sulfide (calcium sulfide is prepared by roasting calcium sulfate with charcoal at 1000 Celsius). Hydrogen sulfide is commercially available, but shipping regulations may decrease sale.

Method 1: Preparation of Anhydrous Hydrogen Sulfide gas from sulfides and sulfuric acid

(By-products from reaction: metal sulfates)

Materials:

| | |
|---|--|
| 1. 50 grams (1.8 oz.) of concentrated sulfuric acid | 2. 30 to 40 grams (1.05 to 1.4 oz.) of powdered sodium sulfide, calcium sulfide, magnesium sulfide, or pulverized pyrites, or pulverized sulfides ores |
|---|--|

Summary: Hydrogen Sulfide gas is readily prepared by reacting concentrated sulfuric acid with a sulfide material. Sulfides such as sodium or magnesium sulfide are the most commonly used sulfide materials, but pyrites, and sulfide ores can be used. **Note: Sodium, Calcium, or Magnesium sulfide can be prepared by roasting sodium sulfate, calcium sulfate (gypsum), or magnesium sulfate (Epsom salt) with powdered charcoal (natural charcoal; no added chemicals or quick light stuff) at 1000 Celsius (1800 Fahrenheit) for several hours. The resulting blackish-gray mass can then be cooled and then placed into the reaction flask for reaction with the sulfuric acid.**

Hazards: Use proper ventilation when using hydrogen sulfide, and avoid chronic exposure, as hydrogen sulfide is toxic.

Procedure: Simply place 50 grams (1.8 oz.) of concentrated sulfuric acid into the addition funnel, and then place 30 to 40 grams (1.05 to 1.4 oz.) of powdered sodium sulfide, calcium sulfide, magnesium sulfide, or pulverized pyrites, or pulverized sulfides ores into the reaction flask. Thereafter, slowly drip the concentrated sulfuric acid onto the sulfides. During the sulfuric acid addition, hydrogen sulfide gas will be steadily evolved.

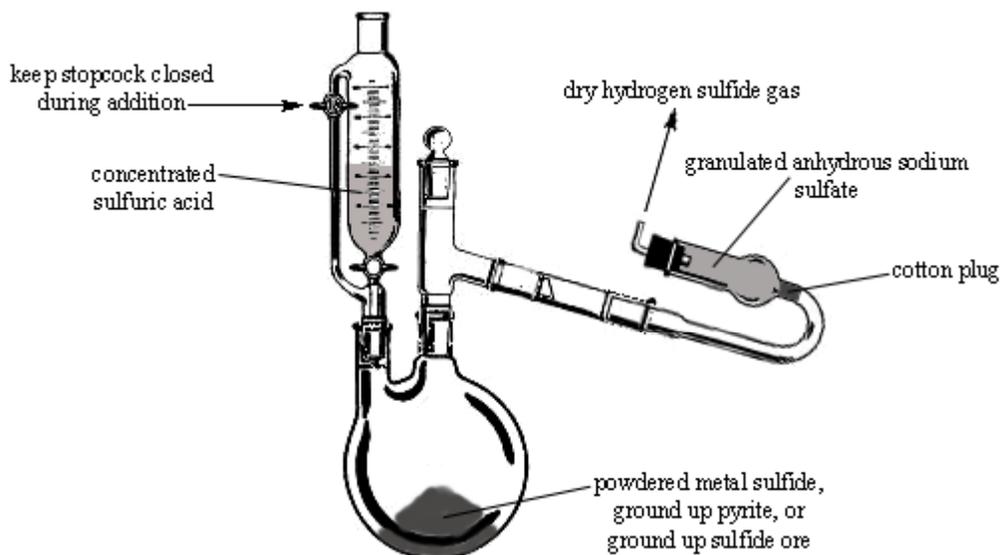
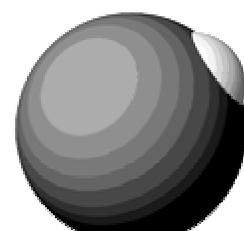
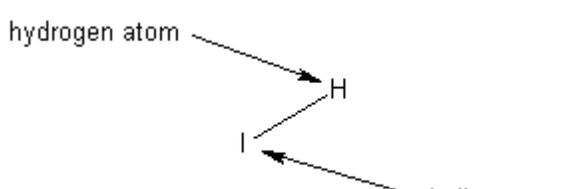


Figure 039. Set-up for the preparation of anhydrous hydrogen sulfide gas. Note: gently heating the reaction flask can help speed up the reaction.

Preparation 11: 46%

Hydroiodic acid

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | HI |
| Structure make-up | Short hand chemical structure |

Hydroiodic acid

Hydroiodic acid is a colorless to yellowish to brownish liquid. It is colorless when freshly prepared, but rapidly turns yellow due to oxidation. Hydroiodic acid should be used within 2 weeks of its preparation, and it will turn brown on standing due to oxidation; however, these brownish solutions can be regenerated by adding in hypophosphorus acid. Hydroiodic acid forms an azeotrope with water containing 57% acid by weight with a constant boiling temperature of 127 Celsius. Hydroiodic acid is readily prepared by bubbling hydrogen sulfide gas into a suspension of iodine in water, and then filtering-off the precipitated sulfur.

Method 1: Preparation of 46% Hydroiodic acid from Iodine and Hydrogen Sulfide gas

(By-products from reaction: sulfur)

Materials:

| | |
|--|---|
| 1. 37 grams of iodine crystals (1.3 oz.) | 3. 5 grams (0.17 oz.) of hydrogen sulfide gas |
| 2. 36 milliliters (1.2 fluid oz.) of water | |

Summary: Concentrated hydroiodic acid can be prepared by reacting iodine crystals with hydrogen sulfide gas in the presence of water. The acid should be used within a week of preparation.

Hazards: Hydrogen sulfide is highly toxic, and inhalation should be avoided. Hydroiodic acid is corrosive and can be absorbed through the skin; wear gloves when handling.

Procedure: Into a suitable reaction flask (equipped with motorized stirrer or other stirring means—see the following illustration), and gas inlet tube, place 32 grams (1.1 oz.) of iodine crystals, followed by 36 milliliters (1.2 fluid oz.) of water. Then place this reaction flask into an ice bath, and chill to about 0 Celsius. Thereafter, bubble into this iodine mixture, 5 grams (0.17 oz.) of hydrogen sulfide gas. During the addition of the hydrogen sulfide gas, rapidly stir the iodine mixture and maintain its temperature at 0 Celsius. After the addition of the hydrogen sulfide gas, continue to rapidly stir the entire reaction mixture for about 30 minutes at 0 Celsius, and then filter-off the precipitated sulfur. Now, add to this filtered reaction mixture, 5 grams (0.17 oz.) of iodine crystals, and then store this reaction mixture (which will be the 47% hydroiodic acid) in a refrigerator at 5 Celsius until use.

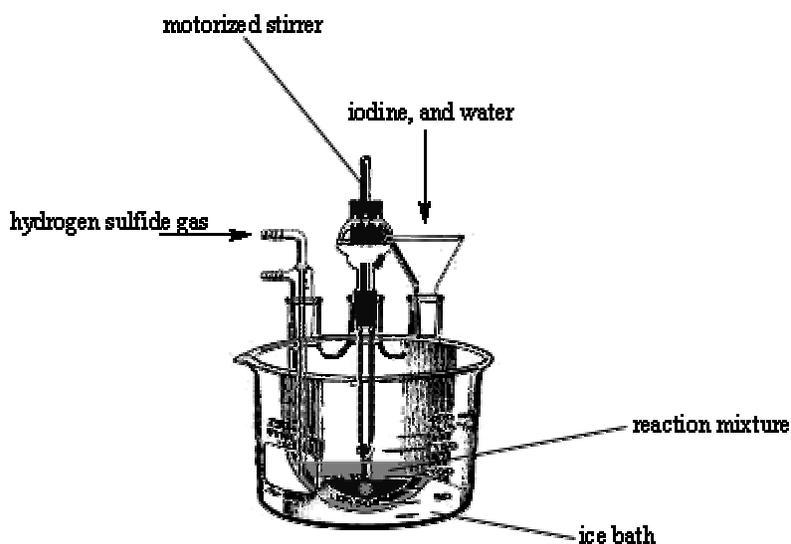
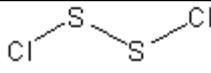
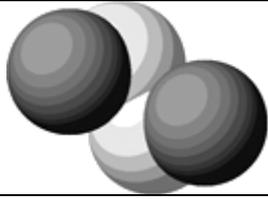
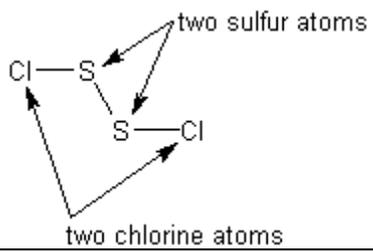


Figure 040. Set-up for the preparation of concentrated hydroiodic acid.

Preparation 12: Sulfur monochloride

| | |
|--|--|
| | |
|--|--|

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | S_2Cl_2 |
| Structure make-up | Short hand chemical structure |

Sulfur monochloride

Sulfur monochloride forms a non-flammable, light amber to yellowish red, fuming oily liquid, which has a penetrating odor. It has a melting point of -77 Celsius, and a boiling point of 138 Celsius. It is soluble in alcohol, ether, carbon disulfide, toluene, carbon tetrachloride, and many oils. It reacts with water yielding sulfur dioxide, hydrogen chloride, and other products. Over exposure may cause skin irritation. It is conveniently prepared by passing dry chlorine into molten sulfur, and then recovering the sulfur chloride by distillation.

Method 1: Preparation of Sulfur monochloride from sulfur and chlorine

(By-products from reaction: none)

Materials:

| | |
|---|---|
| 1. 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene | 3. 26 grams (0.9 oz.) of dry chlorine gas |
| 2. 24 grams (0.8 oz.) of powdered sulfur | |

Summary: Sulfur monochloride is readily prepared by reacting dry chlorine gas with powdered sulfur. The reaction is carried out in an inert solvent such as methylene chloride or benzene. After the reaction is complete, the reaction mixture is quickly filtered, and then distilled to remove the methylene chloride or benzene solvent.

Hazards: Use caution when handling chlorine gas, and use proper ventilation.

Procedure: Into an appropriate flask (as illustrated in the following illustration), place 300 milliliters (10 fluid oz.) of dry methylene chloride, or benzene, and then add in 24 grams (0.8 oz.) of powdered sulfur. Thereafter, place the flask into a cold-water bath, and then pass 26 grams (0.9 oz.) of dry chlorine gas into the mixture over a several hour period while rapidly stirring the reaction mixture. During the addition, keep the reaction mixture below 30 Celsius. After the addition of the chlorine gas, quickly filter the reaction mixture to remove any insoluble materials, and then place the filtered reaction mixture into a distillation apparatus (not illustrated below; the distillation apparatus can be any simple apparatus), and then boil-off the methylene chloride at 40 Celsius, or if using benzene, boil at 80 Celsius to drive-off the benzene. When no more methylene chloride or benzene distills over, remove the heat source, and then allow the remaining oily liquid of sulfur monochloride to cool to room temperature. Then pour the remaining oily liquid into any suitable dry amber glass bottle, and store in a cool dry place.

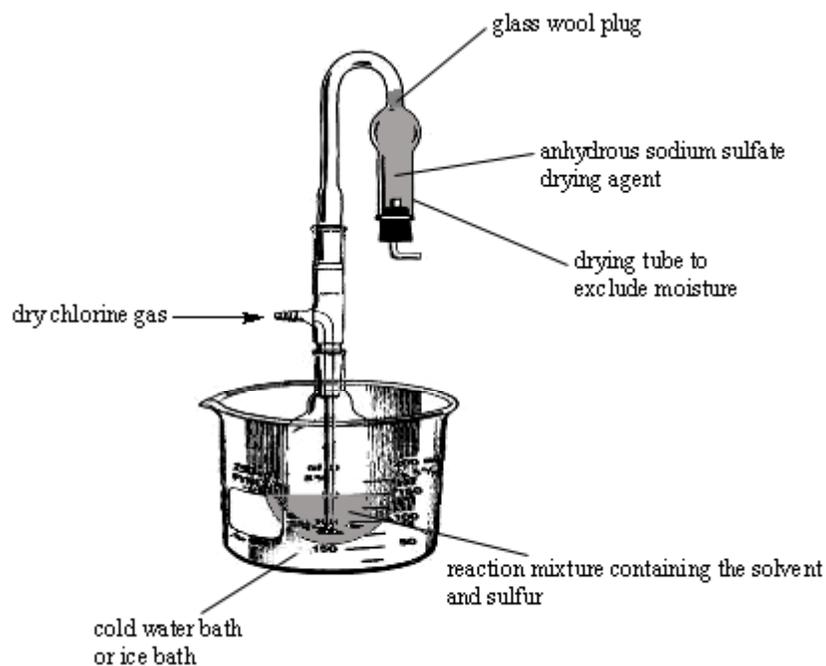
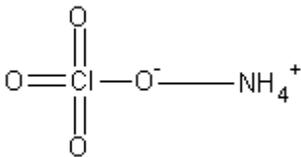
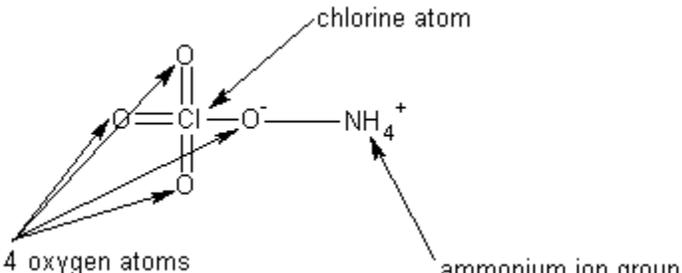


Figure 041. Suggestive apparatus for the preparation of sulfur monochloride from sulfur and chlorine.

Preparation 13: Ammonium perchlorate

| | |
|---|----------------------------------|
|  | Unavailable |
| Chemical structure | 3D Structure |
|  | NH ₄ ClO ₄ |
| Structure make-up | Short hand chemical structure |

Ammonium perchlorate

Ammonium perchlorate forms colorless orthorhombic crystals or a white powder or granules. The crystals decompose when heated into oxygen and ammonium chloride. The salt is readily soluble in water, but relatively insoluble in most organic solvents. Ammonium perchlorate is widely used in solid rocket propellants. The salt is a strong oxidizer, and should be kept away from combustible materials and sources of possible ignition. Store ammonium perchlorate in tightly sealed bottles in a cool place, and away from sunlight.

Method 1: Preparation of ammonium perchlorate from household ammonia and sodium perchlorate utilizing carbon dioxide

(By-products from reaction: sodium bicarbonate)

Materials:

| | |
|---|--|
| 1. 470 grams (1 pound) of sodium perchlorate monohydrate | 4. 1300 grams (2.8 pounds) of dry carbon dioxide gas |
| 2. 1000 milliliters (1 quart) of tap water | 5. or 1000 grams (2.2 pounds) of dry ice |
| 3. 900 grams (1.9 pounds) of a household ammonia solution | |

Summary: Ammonium perchlorate is prepared by adding an ammonia solution to a concentrated solution of sodium perchlorate, and then passing carbon dioxide into the solution over several hours to precipitate the sodium as the water insoluble sodium bicarbonate. The ammonium perchlorate remains in solution, and the solution is then filtered to remove the water insoluble sodium bicarbonate. Thereafter, the ammonium perchlorate is then collected by recrystallization in the usual means.

Hazards: Wear gloves and use proper ventilation when handling ammonia.

Procedure: Dissolve 470 grams (1 pound) of sodium perchlorate monohydrate into 1000 milliliters (1 quart) of tap water. Then quickly add in 900 grams (1.9 pounds) of a household ammonia solution (obtained from grocery stores, sold as “crystal clear” ammonia), while stirring the sodium perchlorate solution. Afterwards, Cool the mixture to 0 Celsius by means of an ice bath. Then bubble 1300 grams (2.8 pounds) of dry carbon dioxide gas into the mixture over a several hour period while keeping the reaction temperature at 0 Celsius and stirring, or add in pieces, 1000 grams (2.2 pounds) of dry ice. During the carbon dioxide gas addition, sodium bicarbonate will slowly precipitate (if dry ice is used, precipitation of the sodium bicarbonate will be more rapid). After all the carbon dioxide has been added, continue to stir the reaction mixture for 90 minutes at 0 Celsius, and then filter the reaction mixture to remove the insoluble sodium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

Method 2: Preparation of ammonium perchlorate from dry ammonia gas and sodium perchlorate utilizing carbon dioxide

(By-products from reaction: sodium bicarbonate)

Materials:

| | |
|--|--|
| 1. 470 grams (1 pound) of sodium perchlorate monohydrate | 4. 1300 grams (2.8 pounds) of dry carbon dioxide gas |
| 2. 1000 milliliters (1 quart) tap water | 5. or 1000 grams (2.2 pounds) of dry ice |
| 3. 96 grams (3.4 ounces) of anhydrous ammonia gas | |

Summary: This procedure is identical to method 1, except the house hold ammonia is replaced with dry anhydrous ammonia gas. Ammonium perchlorate is prepared by bubbling dry ammonia gas into a concentrated solution of sodium perchlorate, and then passing carbon dioxide into the solution over several hours to precipitate the sodium as the water insoluble sodium bicarbonate. The ammonium perchlorate remains in solution, and the solution is then filtered. Thereafter, the ammonium perchlorate is then collected by recrystallization.

Hazards: Wear gloves and use proper ventilation when handling dry ammonia gas, which is highly irritating to the nose and throat.

Procedure: Dissolve 470 grams (1 pound) of sodium perchlorate monohydrate into 1000 milliliters (1 quart) water. Then bubble 96 grams (3.4 ounces) of anhydrous ammonia gas into the sodium perchlorate solution while stirring the sodium perchlorate solution. Afterwards, Cool the mixture to 0 Celsius by means of an ice bath. Then bubble 1300 grams (2.8 pounds) of dry carbon dioxide gas into the mixture over a several hour period while keeping the reaction temperature at 0 Celsius and stirring, or add in pieces, 1000 grams (2.2 pounds) of dry ice. During the carbon dioxide gas addition, sodium bicarbonate will slowly precipitate (if dry ice is used, precipitation of the sodium bicarbonate will be more rapid). After all the carbon dioxide has been added, continue to stir the reaction mixture for 90 minutes at 0 Celsius, and then filter the reaction mixture to remove the insoluble sodium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

Method 3: Preparation of ammonium perchlorate from house-hold ammonia and potassium perchlorate utilizing carbon dioxide

(By-products from reaction: potassium bicarbonate)

Materials:

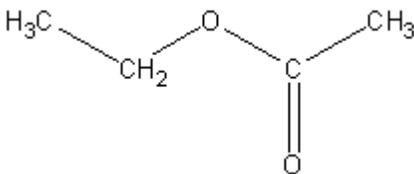
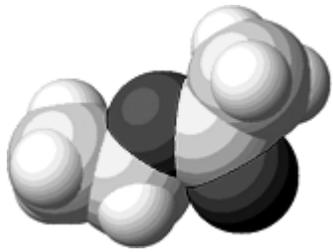
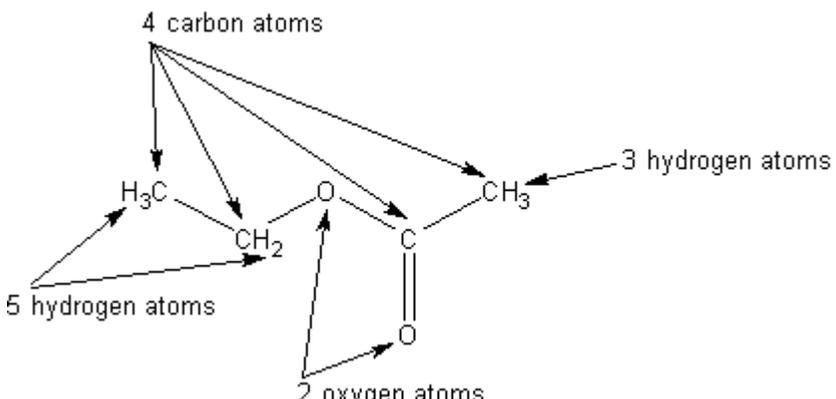
| | |
|---|---|
| 1. 57.6 grams (2 oz.) of potassium perchlorate | 4. 200+ grams (7 oz.) of dry carbon dioxide gas |
| 2. 1000 milliliters (1 quart) tap water | 5. or 200+ grams (7 oz.) of dry ice |
| 3. 90 grams (3.1 oz.) of a household ammonia solution | |

Summary: This procedure is identical to method 1 and 2; expect the sodium perchlorate monohydrate is simply replaced with potassium perchlorate. The ammonium perchlorate is prepared by adding an ammonia solution to a hot dilute solution of potassium perchlorate, and then passing carbon dioxide into the solution over several hours to form potassium bicarbonate and ammonium perchlorate. After the reaction, the mixture is chilled, and then filtered to remove the potassium bicarbonate precipitate. The ammonium perchlorate remains in solution, and is then collected by recrystallization in the usual means.

Hazards: Wear gloves and use proper ventilation when handling ammonia.

Procedure: Dissolve 57.6 grams (2 oz.) of potassium perchlorate into 1000 milliliters (1 quart) of boiling tap water. Then quickly add in 90 grams (3.1 oz.) of a household ammonia solution (obtained from grocery stores, sold as “crystal clear” ammonia), while stirring the potassium perchlorate solution. Afterwards, while boiling the solution, bubble 200+ grams (7 oz.) of dry carbon dioxide gas into the mixture over a several hour period while boiling and stirring the mixture, or add in pieces, 200+ grams (7 oz.) of dry ice. Use caution when adding dry ice to boiling water. During the carbon dioxide gas addition, potassium bicarbonate will slowly form, and some it will dissolve in the boiling water, and some will precipitate (if dry ice is used, the formation of potassium bicarbonate will be much faster). Note: during the boiling process, water will evaporate. As the water evaporates, more needs to be added, so maintain the volume of the water at about 1000 milliliters during the whole boiling process. After all the carbon dioxide has been added, continue to stir the reaction mixture at 100 Celsius for about 10 to 15 minutes, and then remove the heat source and allow the mixture to cool to room temperature. Thereafter, place the cooled reaction mixture into an ice bath, and chill to 0 Celsius and allow this mixture to stand at this temperature for about 90 minutes. Thereafter, filter the reaction mixture to remove the precipitated potassium bicarbonate. After filtration, recrystallize the ammonium perchlorate from the filtered mixture at 100 Celsius. Note: during the recrystallization process, do not over recrystallize. This means when most of the ammonium perchlorate has been collected by filtration, do not boil-off all the remaining water to the point where dry solid remains. Dry ammonium perchlorate may decompose if heated above 70 Celsius. If boiling the mixture at 100 Celsius during the recrystallization process causes some decomposition of the ammonium perchlorate, place the filtered reaction mixture into a shallow pan, and heat the pan at 50 Celsius while blowing air over the surface of the liquid. Do this until dry solid remains (a small portable cooling fan can be used).

Preparation 14: Ethyl acetate

| | |
|---|---|
|  |  |
| Chemical structure | 3D Structure |
|  | $C_4H_8O_2$ |
| Structure make-up | Short hand chemical structure |

Ethyl acetate

Ethyl acetate is a clear, volatile, and flammable liquid with a pleasant, fruity odor. It has a pleasant taste when diluted. Ethyl acetate slowly decomposes by moisture, so it should be kept in air-tight bottles and in a cool dry place. It has a boiling point of 77 Celsius, and a melting point of -83 Celsius. Ethyl acetate is moderately soluble in water (1 milliliter in 10 milliliters of water), but is miscible with alcohol, acetone, chloroform, and ether. It forms an azeotropic mixture with water (6% by weight with a boiling point of 70 Celsius). Ethyl acetate can be prepared by distilling a mixture of ethanol and acetic acid in the presence of a few drops of sulfuric acid. Ethyl acetate is a valuable solvent for many chemical reactions.

Method 1: Preparation of ethyl acetate from Vodka, battery acid, and vinegar

(By-products from reaction: none)

Materials:

| | |
|--|--|
| 1. 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar | 4. 5 drops of concentrated sulfuric acid or 10 to 15 drops of battery acid |
| 2. 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity | 5. 15 grams (1/2 oz.) of anhydrous magnesium sulfate |

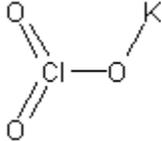
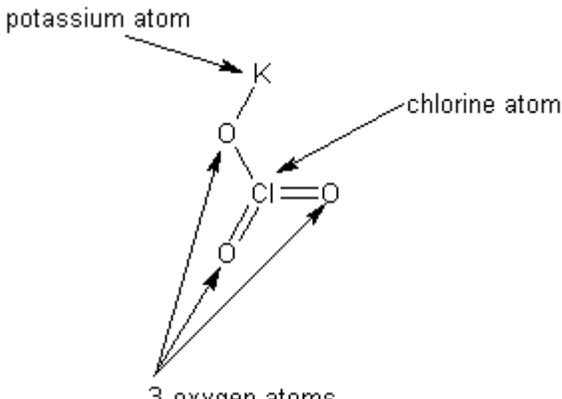
3. 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof

Summary: Ethyl acetate is readily and easily prepared by distilling a mixture of vinegar and ethyl alcohol in the presence of a small amount of sulfuric acid. The sulfuric acid is the catalyst. The resulting distilled product is then treated with anhydrous magnesium sulfate and re-distilled to remove water.

Hazards: Use caution when handling sulfuric acid.

Procedure: Into a large distillation apparatus, or similar distillation apparatus of any desired type or design, place 1000 milliliters (1 quart) of standard 5% acidity distilled crystal clear vinegar or 180 milliliters (6 fluid oz.) of concentrated vinegar of 30% acidity, followed by 120 milliliters (4 fluid oz.) of cheap Vodka of 80 proof, and then followed by 5 drops of concentrated sulfuric acid, or 10 to 15 drops of battery acid. Thereafter, heat and distill the mixture at 77 Celsius until no more ethyl acetate distills over—the time may vary from 1 to 3 hours. After the distillation period, the crude ethyl acetate should be thoroughly mixed with about 15 grams (1/2 oz.) of anhydrous magnesium sulfate, and stirred for about 30 minutes at room temperature. Finally, filter-off the magnesium sulfate, and then fractionally distill the filtered liquid at 77 Celsius to obtain a refined product of at least 99% ethyl acetate.

Preparation 15: Potassium chlorate.

| | |
|---|---|
|  |  |
| Chemical structure | 3D Structure |
|  | $KClO_3$ |
| Structure make-up | Short hand chemical structure |

Potassium chlorate

Potassium chlorate forms colorless to white crystals, white granules or powder. The crystals have a melting point of 368 Celsius, and are relatively insoluble in water and most organic solvents. However, the crystals are soluble in boiling water. Potassium chlorate forms explosives mixtures with combustible materials. These mixtures readily ignite from friction, heat, flames, or sparks. Potassium chlorate should be kept away from iodides and tartaric acid.

Method 1: Preparation of Potassium chlorate from sodium chlorate

(By-products from reaction: sodium chloride)

Materials:

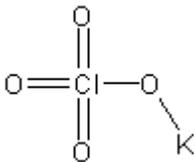
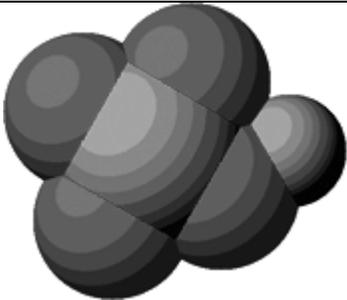
| | |
|--|--|
| 1. 220 milliliters (7.4 fluid oz.) of warm water | 3. 105 grams (3.7 oz.) of potassium chloride |
| 2. 150 grams (5.2 oz.) of sodium chlorate | 4. 250 milliliters (8.4 fluid oz.) of warm water |

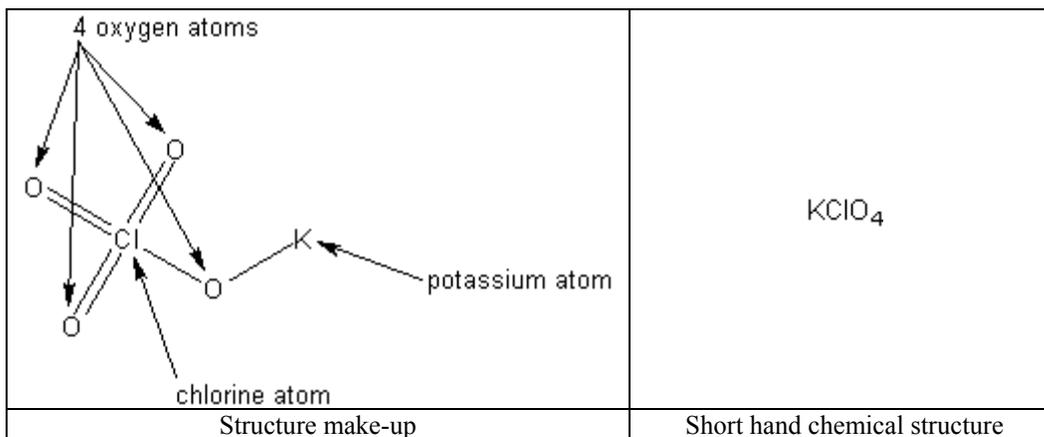
Summary: Potassium chlorate is readily prepared by condensing potassium chloride with sodium chlorate in solution. After the initial mixing, the potassium chlorate is readily collected by filtration.

Hazards: None.

Procedure: Into a suitable beaker, flask, or any other suitable container, place 220 milliliters (7.4 fluid oz.) of warm water, and then add in 150 grams (5.2 oz.) of sodium chlorate. Thereafter, stir the mixture until all the chlorate dissolves. Now, prepare a second solution by adding and dissolving 105 grams (3.7 oz.) of potassium chloride into 250 milliliters (8.4 fluid oz.) of warm water in a suitable beaker, flask, or container. Then, quickly mix both solutions, and then stir the combined mixture for about 30 minutes. Thereafter, cool the mixture to 0 Celsius by using an ice bath, or by placing the beaker, flasks, or container containing the combined mixture into a refrigerator and allow it to stand for 1 hour or so. Afterwards, filter-off the precipitated potassium chlorate, and then vacuum dry or air-dry the crystals. Afterwards, the potassium chlorate can be recrystallized from boiling water if desired, but this is not necessary in most cases as the chlorate will have a good purity as is.

Preparation 16: Potassium perchlorate

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
| | |



Potassium perchlorate

Potassium perchlorate forms colorless to white crystals, or white crystalline powder, or granules. The crystals begin to decompose when heated to 400 Celsius into oxygen and potassium chloride. Potassium perchlorate is sensitive to concussion, and forms explosive mixtures with combustible materials such as sulfur and charcoal. The crystals have a low solubility in water (1 gram per 65 milliliters of water) at room temperature, but the crystals are somewhat soluble in boiling water (1 gram in 15 milliliters). Potassium perchlorate is insoluble in the usual solvents.

Method 1: Preparation of Potassium perchlorate from potassium chlorate

(By-products from reaction: potassium chloride)

Materials:

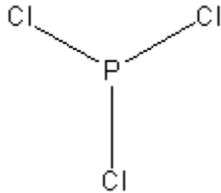
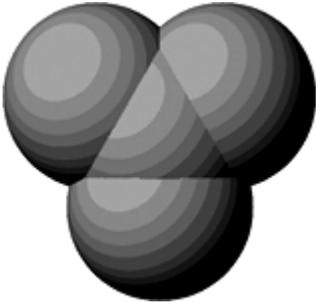
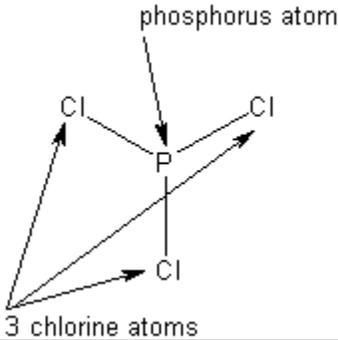
| | |
|--|---|
| 1. 100 grams (3.5 oz.) of potassium chlorate | 3. 200 milliliters (6.7 fluid oz.) of water |
| 2. 50 grams (1.7 oz.) of potassium chloride | |

Summary: Potassium perchlorate is readily prepared by fusing potassium chlorate in the presence of potassium chloride. The potassium chloride is used to decrease decomposition of the perchlorate and provide a stabilized uniform mixture. After the initial reaction, the potassium perchlorate is easily collected by thoroughly mixing the cooled mixture in water to dissolve the potassium chloride and any unreacted chlorate, followed by filtration to recover the insoluble perchlorate.

Hazards: None.

Procedure: Into a suitable crucible, place 100 grams (3.5 oz.) of potassium chlorate, followed by 50 grams (1.7 oz.) of potassium chloride. Thereafter, briefly shake the crucible to mix the two salts. Then gently and slowly heat the mixture using a Bunsen burner to about 370 Celsius. Be sure to carefully monitor the temperature using a temperature probe or equivalent. Shortly thereafter, a gentle gas evolution of oxygen will take place. Keep heating the mixture at about 380 Celsius until the evolution of gas ceases. After the evolution of gas has ceased, immediately remove the heat source, and allow the mixture to cool to room temperature. Then, using a knife or other sharp and hard instrument, fracture and pulverize the solid mass in the crucible, and then place this pulverized mass into a suitable beaker, or container. Thereafter, add in 200 milliliters (6.7 fluid oz.) of water (at room temperature), and then rapidly stir the entire mixture for about 1 hour to dissolve the potassium chloride. After stirring for 1 hour, filter-off the insoluble potassium perchlorate, and then vacuum dry or air-dry the crystals.

Preparation 17: Phosphorus trichloride

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | PCl_3 |
| Structure make-up | Short hand chemical structure |

Phosphorus trichloride

Phosphorus trichloride forms a colorless to slightly colored fuming liquid with a melting point of -112 Celsius, and a boiling point of 76 Celsius. The liquid is highly reactive and decomposes rapidly in the presence of water or alcohol. The liquid should be kept in airtight bottles and stored in a cool dry place. It is soluble in benzene, methylene chloride, ether, and hexane. Avoid inhalation of the fumes and skin contact.

Method 1: Preparation of Phosphorus trichloride from trisodium phosphate, chlorine, calcium chloride, and charcoal

(By-products from reaction: calcium oxide, calcium chloride, tetrachlorethane, trichloroethane, chlorine, acetylene)

Materials:

| | |
|--|--|
| 1. 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate | 6. 100 grams (3.5 oz.) of finely powdered wood charcoal |
| 2. 750 milliliters (25.2 fluid oz.) of warm water | 7. 250 milliliters (6.9 fluid oz.) of methylene chloride |
| 3. 250 grams (8.8 oz.) of anhydrous calcium chloride | 8. 200 grams (7 oz.) of extra dry chlorine gas |
| 4. 750 milliliters (25.2 fluid oz.) of cold water | 9. 100 grams (3.5 oz.) or so of aluminum oxide |
| 5. 1500 milliliters (50.7 fluid oz.) of warm water | |

Summary: Phosphorus trichloride can be prepared in a three-step process starting with the formation of calcium phosphate. This crude technical grade of calcium phosphate is prepared by mixing aqueous solutions of trisodium phosphate and calcium chloride. The mixing causes the water insoluble calcium phosphate to precipitate. The precipitate is readily filtered-off, washed, and then dried in the usual manner. Thereafter, the crude calcium phosphate is then roasted at high temperature in the presence of carbon, whereby it gets reduced to calcium phosphide. Note: some elemental phosphorus may be liberated in the reaction, so use proper ventilation. The calcium phosphide crude mixture is then chlorinated to form a mixture of compounds, one of which being the desired phosphorus trichloride. After the chlorination process, the reaction mixture is filtered, and then distilled using a conventional distillation apparatus to remove the methylene chloride solvent. Finally, the remaining crude liquid left over after removal the methylene chloride, is then fractionally distilled to recover the liquid product of phosphorus trichloride. The distilled phosphorus trichloride can be re-distilled if desired.

Hazards: Use good ventilation when working with chlorine gas, and avoid inhalation of the fumes. Use caution when heating the calcium phosphate as small amounts of white phosphorus may be evolved.

Procedure:

Step 1: Preparation of calcium phosphate

Into a suitable beaker or similar container, place 250 grams (8.8 grams) of technical grade (store bought) trisodium phosphate, and then add in 750 milliliters (25.2 fluid oz.) of warm water, and then stir the mixture to dissolve all of the trisodium phosphate (technical grade). Note: technical grade and store bought trisodium phosphate (TSP) contains sodium carbonate and maybe some sodium hydroxide to prevent the product from caking. This sodium carbonate and any other alkaline product needs to be neutralized before the initial reaction. To do this, first, place 250 grams (8.8 grams) of the dry TSP product from the packaging container into a suitable crucible or similar container, and then roast it at 150 to 250 Celsius for 1 hour to remove several moles of water of hydration. Thereafter, break-up the re-solidified TSP product from the crucible or similar container, and then dissolve it into 750 milliliters (25.2 fluid oz.) of water contained in a suitable beaker or container. Thereafter, drip hydrochloric acid, battery acid, or sulfuric acid into the trisodium phosphate solution until the evolution of carbon dioxide gas ceases. Once this point is achieved, the sodium carbonate has been neutralized. Now, to this trisodium phosphate solution, add in a calcium chloride solution prepared by adding and dissolving 250 grams (8.8 oz.) of anhydrous calcium chloride into 750 milliliters (25.2 fluid oz.) of cold water—heat will be evolved upon dissolving. Note: anhydrous calcium chloride is obtained by placing a sample of at least 300+ grams of technical grade calcium chloride, available in hardware stores and grocery stores sold as “ice melter”, into a crucible or similar container, and then roast the technical grade calcium chloride at 200+ Celsius to remove the water of hydration. Thereafter, the anhydrous calcium chloride can be dissolved into the 750 milliliters (25.2 fluid oz.) of water, once the anhydrous salt has cooled. Upon mixing the two solutions of trisodium phosphate and calcium chloride, a precipitate of crude calcium phosphate tribasic, mixed with a small amount of other calcium phosphates, will form a chunky and messy precipitate. Thereafter, filter-off this chunky precipitate using gravity filtration, or preferably vacuum filtration, and then wash this precipitate with three 500 milliliter (three 16.9 fluid oz. portions) portions of warm water. Thereafter, vacuum dry or air-dry the washed filtered-off precipitate. Finally, place the dried calcium phosphate into a crucible or similar container, and then roast it at 100 to 150 Celsius for an hour or so to remove any moisture or water of hydration. Thereafter, pulverize the heated calcium phosphate into a fine powder, once it has cooled.

Step 2: Preparation of calcium phosphide

Now, into a zip lock bag, or similar plastic container, place the pulverized calcium phosphate (prepared in step 1), followed by 100 grams (3.5 oz.) of finely powdered wood charcoal (average charcoal used for cooking, with no quick-light garbage or other chemical additives), and then shake the bag thoroughly for about 10 to 15 minutes to thoroughly mix both solids. Thereafter, place this mixed powdered mixture into any high heat resistant crucible, such as stainless steel crucible, nickel, or quartz crucible, or equivalent, and then blast this mixture at 1000 Celsius using a high temperature Meeker Bunsen burner or equivalent for about 1 hour or so. If a high temperature Bunsen burner is unavailable, you can use a fire or some other means of heat. After the roasting process, remove the heat source, and allow the hot mixture to cool to room temperature. Now, at this point, the blackish-gray mixture will be composed of calcium phosphide, calcium oxide, a little calcium carbide, unreacted calcium phosphate, and un-reacted charcoal. Keep this mixture as is for use in step 3.

Step 3: Preparation of phosphorus trichloride

Into a suitable apparatus, as illustrated below, place the crude product obtained in step 2, and then add in 250 milliliters (6.9 fluid oz.) of methylene chloride. Then begin the motorized stirrer on moderate speed, and then bubble into the mixture, 200 grams (7 oz.) of extra dry chlorine gas over a period of 2 to 6 hours while stirring the reaction mixture on moderate speed. Note: dry chlorine can be obtained by passing the chlorine through multiple anhydrous calcium chloride drying tubes. During the addition of the chlorine gas, phosphorus trichloride will form, and will be taken-up into the methylene chloride. The by-products, will form insoluble precipitates along with the carbon. After the addition of the chlorine gas, the mixture needs to be filtered, to remove the insoluble materials, and then the resulting filtered mixture needs to be passed several times, through a silica gel column, filled with 100 grams (3.5 oz.) or so of aluminum oxide (see illustration). Thereafter, the mixture should be placed in a distillation apparatus, and first, distilled at 40 Celsius to remove the methylene chloride. Thereafter, place the remaining mixture into a fractional distillation apparatus, and fractionally distill the product mixture at 76 Celsius to collect the phosphorus trichloride. Note: during all distillations, keep a drying tube attached to the apparatus to keep moisture out.

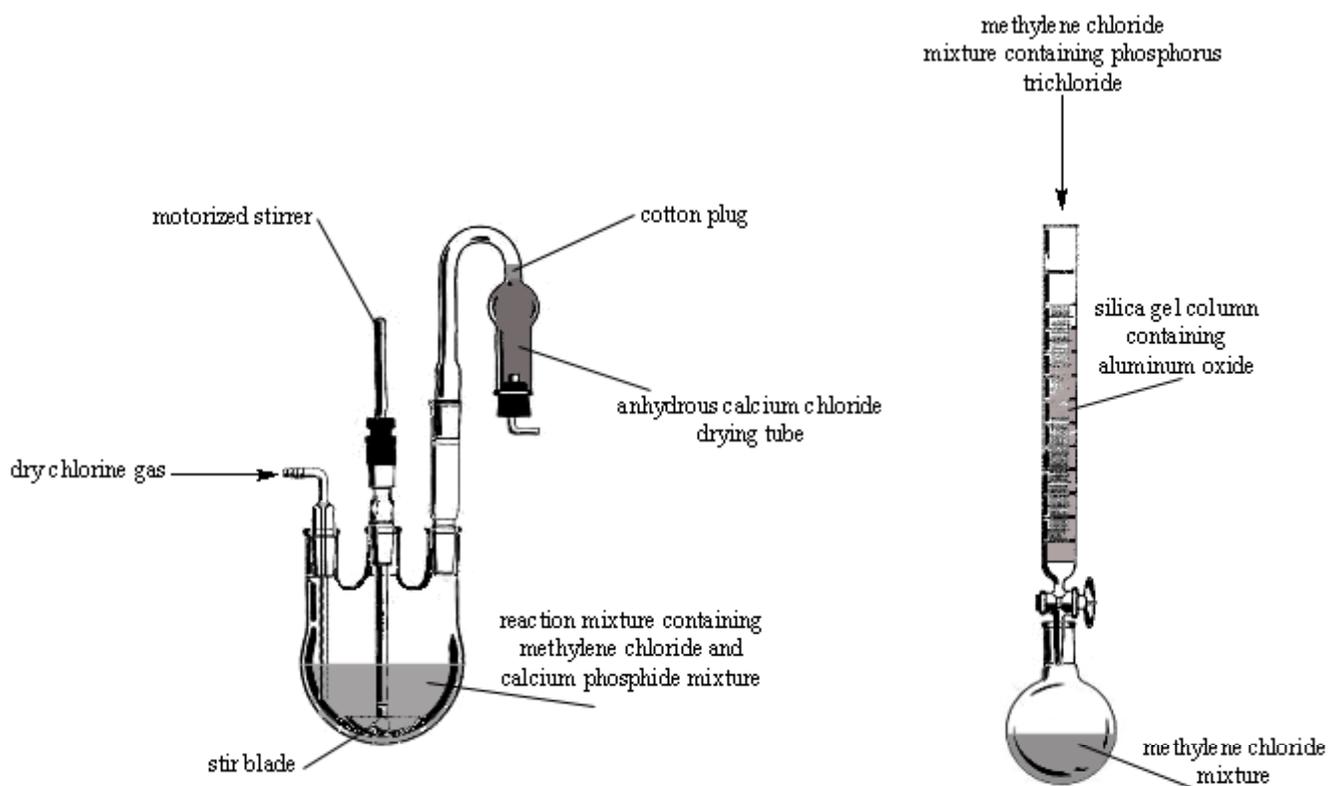


Figure 042. Left illustration: Apparatus for the addition of chlorine gas to a mixture of methylene chloride and calcium phosphide. Right illustration: setup for the purification of the methylene chloride reaction mixture containing the phosphorus trichloride. The aluminum oxide helps remove impurities, and carbon.

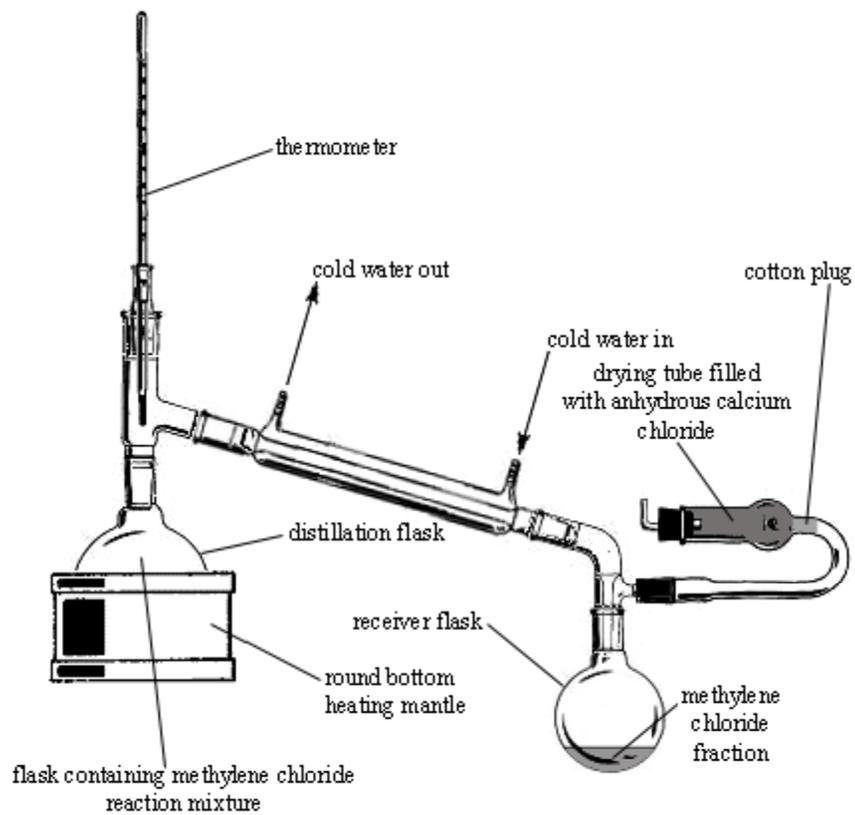


Figure 043. Distillation apparatus for removal of the methylene chloride.

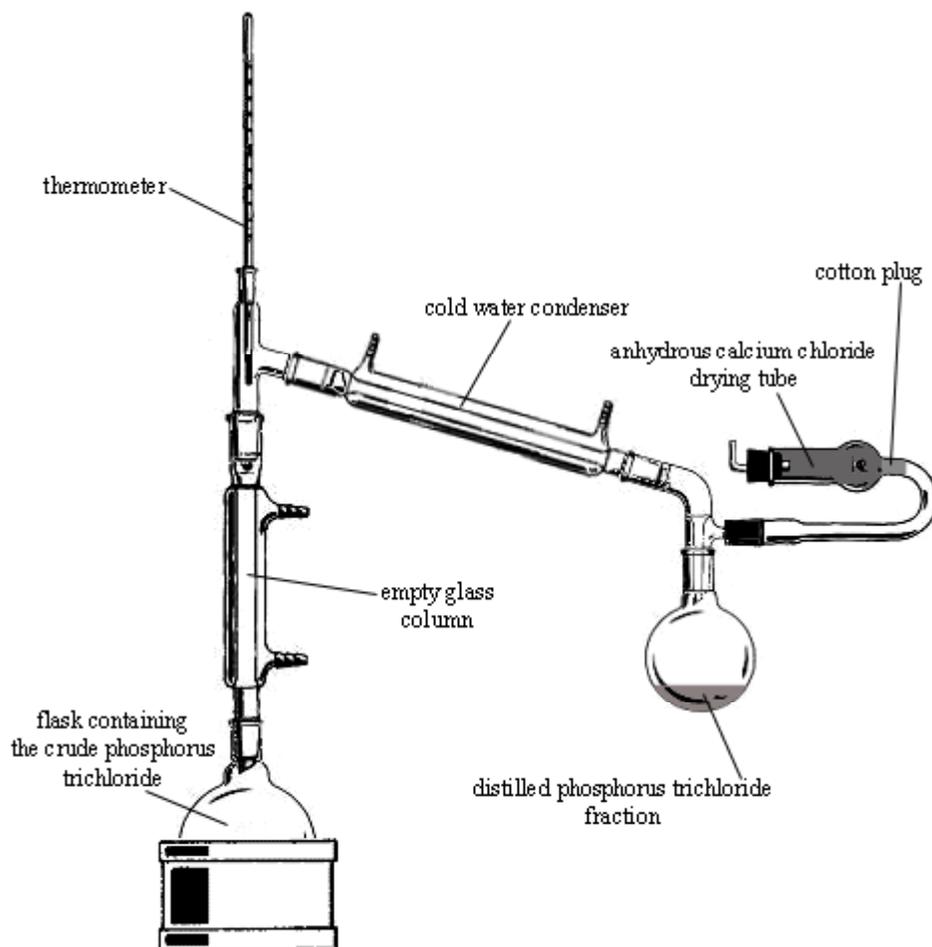
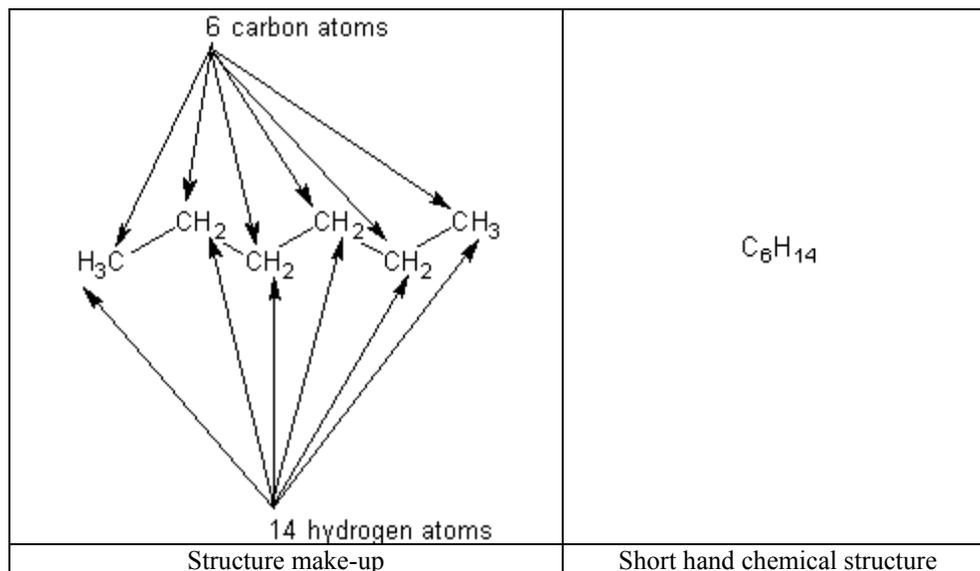


Figure 044. Fractional distillation apparatus for distillation of phosphorus trichloride.

Preparation 18: Hexanes; the distillation of gasoline

| | |
|--|------------------|
| $\text{H}_3\text{C}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_3$ | Mixed structures |
| Chemical structure | 3D Structure |
| | |



Hexanes

Hexanes are a colorless, very volatile liquid with a faint, peculiar odor. It is rarely sold as n-hexane but usually admixed with hexane isomers simply called “hexanes”, but marketed as “hexane”. Hexane has a boiling point of 69 Celsius, and a melting point of -100 Celsius. It is insoluble in water, but miscible with alcohol, chloroform, and ether. Hexane is a major component of gasoline, and can be distilled from the gasoline using a multiple-path distillation apparatus. Hexane is obtained commercially from petroleum, and is a widely available commercial chemical.

Method 1: Preparation of mixed hexanes by the distillation of gasoline

(By-products from reaction: complex mixture of hydrocarbons and gasoline additives)

Materials:

| | |
|---|--|
| 1. 1 to 4 liters (1 to 4.2 quarts) of unleaded premium gasoline | |
|---|--|

Summary: Gasoline can be distilled to yield a variety of products, one of which is the desired hexanes. Hexanes are widely used as a solvent, and are a major component of gasoline.

Hazards: Gasoline is extremely flammable, so use caution.

Procedure: Mixed hexanes are prepared by distilling unleaded premium gasoline using a special multi-path distillation apparatus. To proceed, setup an apparatus as illustrated below. Then fill the distillation flask with 4 liters or less of unleaded premium gasoline, and then distill the mixture at 70 Celsius. This method of distillation takes advantage of the vapor densities of the various components of the gasoline. The vapors of the liquids with the lower densities will condense higher up in the apparatus, and the vapors with the higher densities, such as octane condense lower down in the apparatus. The octane, and heptane fractions will distill over first, followed by the hexane fraction at the second highest condenser mark. The pentane fraction will condense at the highest mark, due its density being less then the hexane, heptane, and octane fractions. Dissolved gases within the gasoline will carry over and will be vented through the top of the system, so the distillation should be carried to in a well ventilated area away from any source of ignition.

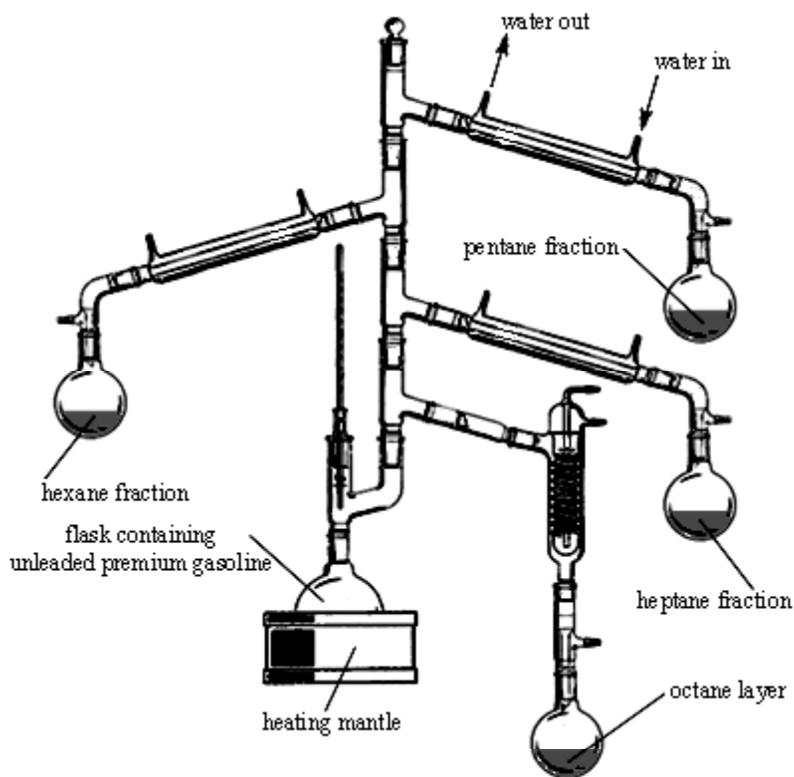
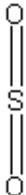
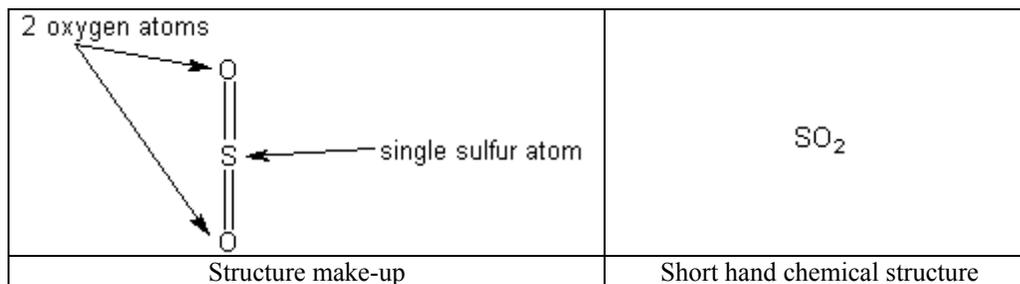


Figure 045. Distillation apparatus for the distillation of gasoline.

Preparation 19: Sulfur dioxide gas

| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
| | |



Sulfur dioxide

Sulfur dioxide forms a colorless gas with a strong suffocating and irritating odor. The gas has a melting point of -72 Celsius, and a boiling point of -10 Celsius. The gas is easily condensed into a colorless liquid. The gas is soluble in water, alcohol, chloroform, and ether. Sulfur dioxide is available in gas cylinders, but it can be prepared in the lab by dripping hydrochloric acid onto excess sodium bisulfite.

Method 1: Preparation of sulfur dioxide gas from sodium bisulfite and hydrochloric acid

(By-products from reaction: water and sodium chloride)

Materials:

| | |
|---|--|
| 1. 100 grams (3.5 oz.) of powdered sodium bisulfite or concentrated Muriatic acid | 2. 100 grams (3.5 oz.) of concentrated hydrochloric acid |
|---|--|

Summary: Sulfur dioxide gas is easily prepared by mixing hydrochloric acid with sodium bisulfite. The reaction is very smooth and generates a steady stream of gas.

Hazards: Use proper ventilation when using sulfur dioxide gas, which is very irritating to the nose and throat. Avoid inhalation of the fumes.

Procedure: Set-up the apparatus as illustrated below, and then place 100 grams (3.5 oz.) of powdered sodium bisulfite into the flask as illustrated, and then place 100 grams (3.5 oz.) of concentrated hydrochloric acid or concentrated Muriatic acid into the addition funnel as illustrated. Thereafter, gradually drip the hydrochloric acid onto the sodium bisulfite over a period of about 30 to 90 minutes. During the acid addition, the sulfur dioxide gas will be steadily evolved and will carry over.

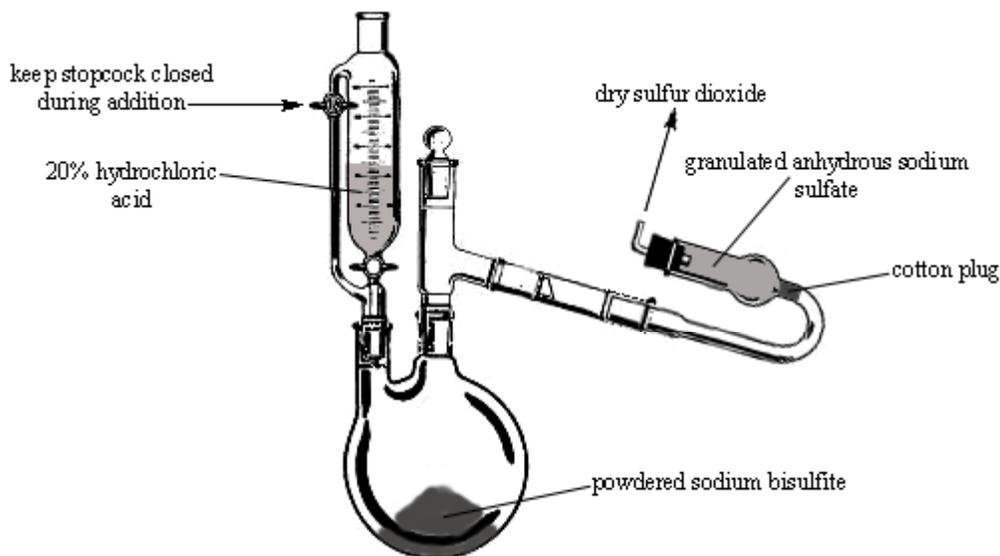


Figure 046. Set-up for the generation of sulfur dioxide gas.

Method 2A: Preparation of sulfur dioxide gas from sulfuric acid and copper

(By-products from reaction: water and copper-II-sulfate)

Materials:

| | |
|--|--|
| 1. 150 grams (5.2 oz.) of concentrated sulfuric acid | 2. 97 grams (3.4 oz.) of copper wire, pipes, pieces, or any other pure copper material |
|--|--|

Summary: Sulfur dioxide gas is readily prepared by reacting concentrated sulfuric acid with copper. The reaction is smooth and produces a steady stream of gas.

Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated sulfuric acid, and use caution.

Procedure: Into the apparatus illustrated below, place 150 grams (5.2 oz.) of concentrated sulfuric acid, followed by 97 grams (3.4 grams) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper as illustrated below. Thereafter, monitor the reaction and do not let the acid mixture get to hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily evolved. The total reaction time can be anywhere from 45 minutes to 2 hours. Heat may or may not be applied to speed up the reaction.

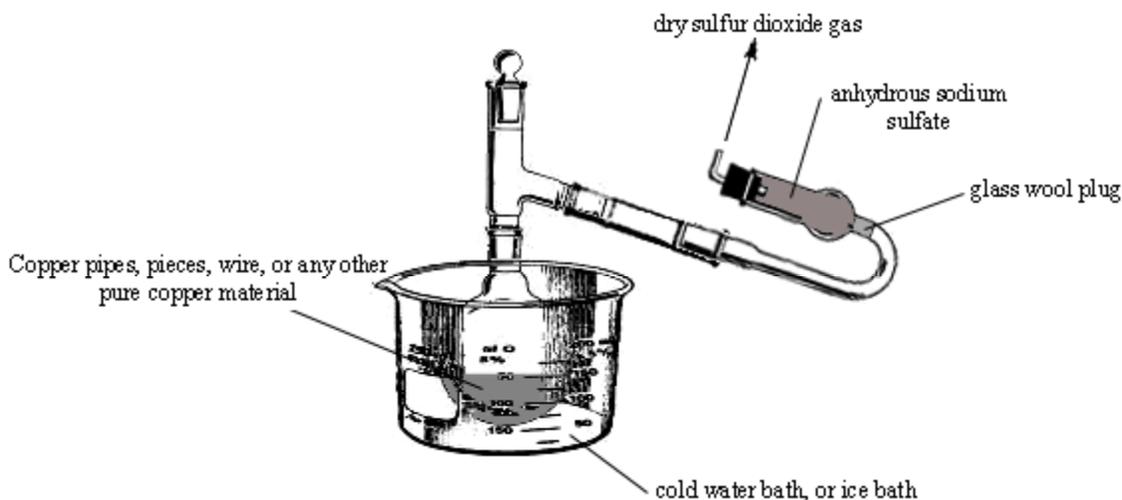


Figure 047. Set-up for the preparation of sulfur dioxide gas from sulfuric acid and copper.

Method 2B: Preparation of sulfur dioxide gas from Epsom salt, hydrochloric acid, and copper

(By-products from reaction: water, magnesium chloride, and copper-II-sulfate)

Materials:

| | |
|--|---|
| 1. 400 grams (14.1 oz.) of Epsom salt | 3. 330 grams (11.6 oz.) of concerted hydrochloric acid or concentrated Muriatic acid |
| 2. 1300 milliliters (1.3 quarts) of warm water | 4. 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material |

Summary: Sulfur dioxide gas can be readily prepared in a modified process using Epsom salt rather than sulfuric acid. In this regards, the concentrated hydrochloric acid partially reacts with the Epsom salt forming the acidic bisulfate ion, and sulfuric acid. These substances then in turn, react with copper to produce sulfur dioxide and the usual by-products.

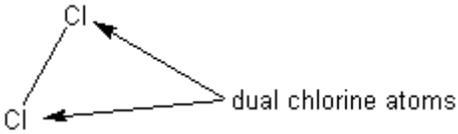
Hazards: Use proper ventilation when using sulfur dioxide gas, and avoid inhalation of the fumes. Wear gloves when handling concentrated hydrochloric acid, and avoid inhalation of the fumes.

Procedure: Into a large beaker or similar container, place 400 grams (14.1 oz.) of Epsom salt, followed by 1300 milliliters (1.3 quarts) of warm water, and then stir the mixture briefly to dissolve all of the Epsom salt. Thereafter, pour this Epsom salt solution into a similar apparatus as illustrated in method 2A, and then add in 330 grams (11.6 oz.) of concentrated hydrochloric acid or concentrated

Muriatic acid, and then blend the mixture for about 10 minutes to form a uniform acidic mixture. Note: an exothermic reaction will proceed, lowering the temperature of the acidic solution. Thereafter add in 103 grams (3.6 oz.) of copper wire, pipes, pieces, or any other pure copper material, and then seal the apparatus using a glass stopper as illustrated below. Thereafter, monitor the reaction and do not let the acid mixture get to hot. During the reaction, a steady stream of sulfur dioxide gas will be steadily, yet slowly evolved. The total reaction time can be anywhere from 2 hours to 4 hours. Heat may or may not be applied to speed up the reaction.

Preparation 20: Chlorine gas (non electrochemical preparation)

NOTE: For electrochemical preparations, see electrochemical preparation 11.

| | |
|---|---|
|  |  |
| Chemical structure | 3D Structure |
|  | Cl_2 |
| Structure make-up | Short hand chemical structure |

Chlorine

Chlorine gas is a yellow gas with a suffocating, and strongly irritating odor. It has a melting point of -101 Celsius, and a boiling point of -34 Celsius. Chlorine is sold as a compressed gas in steel cylinders. It is insoluble in water and not very soluble in alcohol, but soluble in dry benzene, and toluene. Chlorine combines readily with all elements except the noble gases, hydrogen, oxygen, and nitrogen. Chlorine does not occur naturally, but occurs in combined form as chlorides. It occurs in nature (in the form of chlorides) as sodium chloride, potassium chloride, and magnesium chloride. Many finely divided metals will burn in a chlorine atmosphere. Chlorine is a toxic gas, which can be fatal if inhaled for prolonged periods of time. Inhalation of mild quantities of chlorine causes nose and throat irritation followed by excessive mucous congestion. Chlorine is a corrosive gas, which will react with many metals on contact. It is a strong oxidizer and is capable of oxidizing a great many inorganic compounds. Chlorine will explode in contact with hydrogen if direct sunlight is present. Chlorine should be protected from sunlight. It is prepared on an industrial scale from the electrolyses of sodium chloride brine in a system called the chloro-alkali process (sodium hydroxide is a useful by-product). It can be prepared in the lab by reacting hydrochloric acid with calcium hypochlorite or any other strong oxidizer.

Method 1: Preparation of chlorine gas from hydrochloric acid and an oxidizing salt

(By-products from reaction: metal oxides, metal chloride, and water)

Materials:

| | |
|--|---|
| 1. 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine) | 3. 200 milliliters (6.7 fluid oz.) of tap water |
| 2. 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) | |

Summary: This procedure is a duplicate of the procedure for the preparation of Iodine whereby chlorine is generated. Chlorine gas is prepared by dripping hydrochloric acid onto a strong oxidizing salt. During the reaction, chlorine gas is steadily evolved.

Hazards: Use care when handling strong oxidizers like manganese dioxide, potassium permanganate, sodium hypochlorite, and calcium hypochlorite, as they are all highly reactive and capable of reacting with many organic substances. Mixtures of strong oxidizers and combustible materials can ignite and burn violently. Wear gloves when handling hydrochloric acid, which can cause skin irritation. Chlorine is toxic, so handle with care and use caution.

Procedure: Setup the chlorine generating apparatus as illustrated below. Note: your chlorine generator does not have to be as fancy as the apparatus illustrated below, but your system should be similar in design. You can use latex tubing, PVC pipes and other plastic devices; as well, steel pipes can be used, but other metals should be avoided due to corrosion.

After you have the chlorine generator set-up, place into the reaction flask, 75 grams (2.6 oz.) of manganese dioxide, or 1200 grams (2.6 pounds) of a 5% sodium hypochlorite solution (Clorox bleach), or 65 grams (2.3 oz.) of potassium permanganate, or 95 grams (3.3 oz.) of calcium hypochlorite, bleaching powder (65% available chlorine). Then place into the addition funnel, a hydrochloric acid solution prepared by adding and dissolving 150 grams (5.3 oz.) of 35 to 38% hydrochloric acid (muriatic acid of 31% will work) into 200 milliliters (6.7 fluid oz.) of tap water. Note: If using Clorox bleach, avoid brands that contain sodium hydroxide—look on the label to find out if your bleach has sodium hydroxide in it. Sodium hydroxide will screw-up the reaction, which is the main reason the companies add it in!

Once your chlorine generator is setup, begin slowly dripping the hydrochloric acid solution onto the oxidizer contained in the reaction flask. During the reaction, chlorine gas will be steadily evolved. Note: Obviously, you should have another apparatus set-up for reacting the chlorine with something.

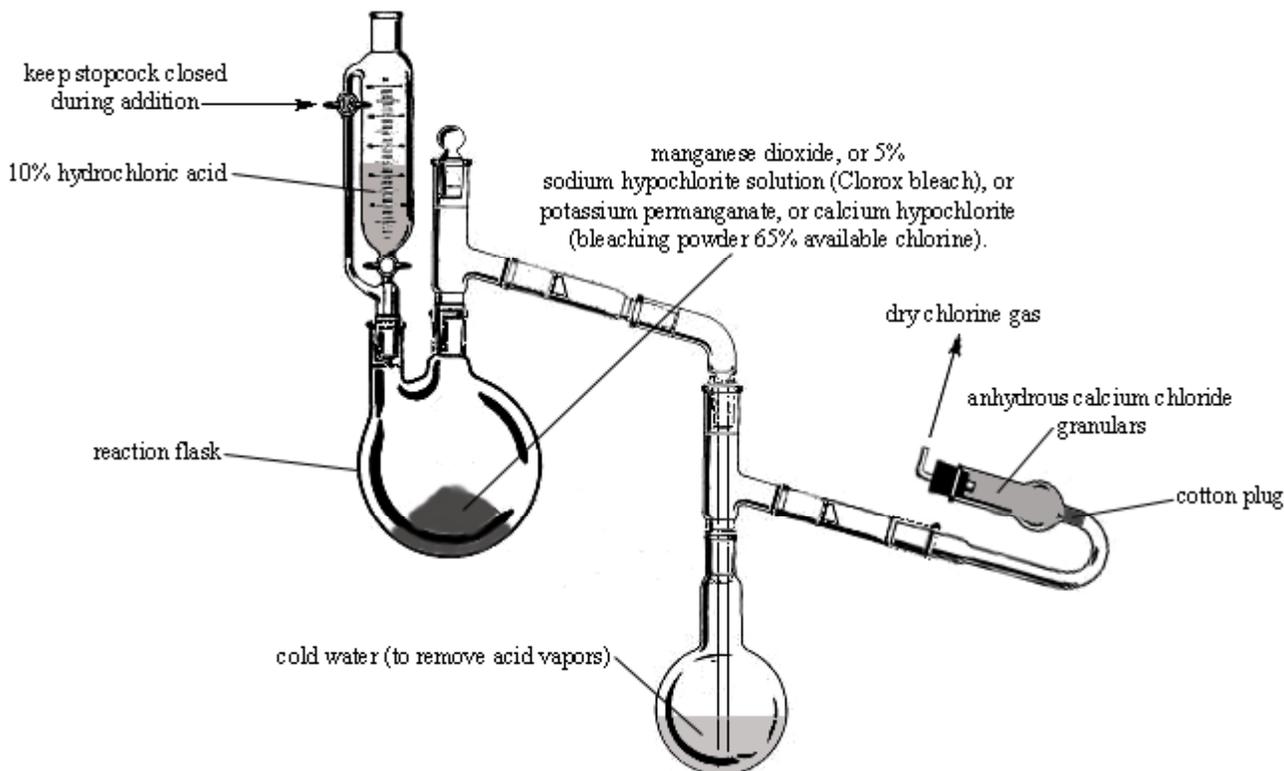
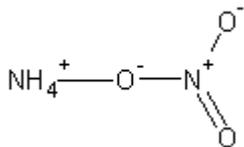
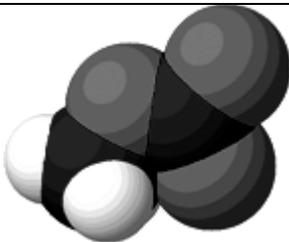
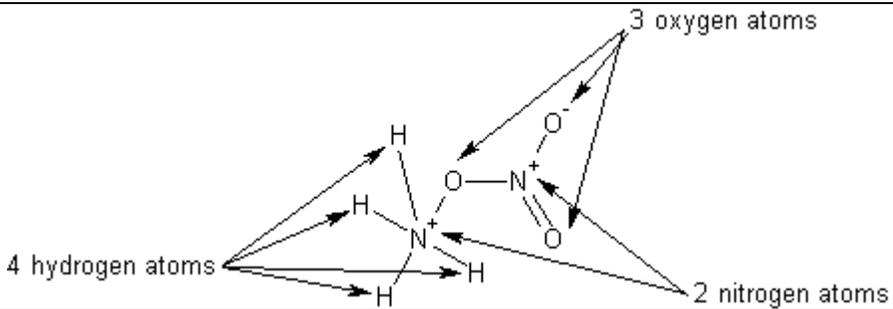


Figure 048. Setup for the generation of chlorine gas.

Preparation 21: Ammonium nitrate

| | |
|---|---|
|  |  |
| Chemical structure | 3D Structure |
|  | NH_4NO_3 |
| Structure make-up | Short hand chemical structure |

Ammonium nitrate

Ammonium nitrate forms odorless, transparent, hygroscopic, and deliquescent crystals or white granules. It decomposes at 210 Celsius into water and nitrous oxide (laughing gas). Ammonium nitrate is very soluble in water, and alcohol. It is widely available commercially, and is available in a number of products including fertilizers and garden products. For use in explosives, AN is commonly used admixed with fuel oil, diesel fuel, TNT, oils, and aluminum for explosives compositions. Ammonium nitrate mixed with oils and fuels are commonly called prills, and are highly stable mixtures widely used in mining and engineering operations. Because ammonium nitrate prills are quite stable for long-term storage and they yield no noxious fumes upon detonation, they are called “safety explosives”. Ammonium nitrate and TNT are commonly used as military dynamites, and blasting charges for military engineering purposes. Ammonium nitrate should be stored in tightly sealed, amber glass bottles, or non-transparent plastic containers in a well-cooled area protected from sunlight.

Method 1: Preparation of ammonium nitrate from ammonia and nitric acid

(By-products from reaction: none)

Materials:

| | |
|--|---|
| 1. 206 grams (7.3 oz.) of 70% nitric acid | 3. Next, bubble 40 grams (1.4 oz.) of ammonia gas or 136 grams (4.8 oz.) of 28 – 30% ammonia solution or 400 grams (14.1 oz.) of a 10% ammonia solution (household ammonia) |
| 2. 600 milliliters (20.2 fluid oz.) of water | |

Summary: Ammonium nitrate is easily prepared by dissolving ammonia gas, or water solutions of ammonia into dilute nitric acid at low temperature. The ammonium nitrate is recovered by recrystallization from the water.

Hazards: Wear gloves when handling 70% nitric acid. 70% Nitric is highly toxic and corrosive. Use proper ventilation when handling ammonia gas, or its solutions.

Procedure: Dissolve 206 grams (7.3 oz.) of 70% nitric acid into 600 milliliters (20.2 fluid oz.) of water. Then cool the diluted nitric acid solution to 0 Celsius by means of an ice bath while stirring continuously. Next, bubble 40 grams (1.4 oz.) of ammonia gas into the nitric acid mixture over a period of one hour while stirring and maintaining the reaction mixtures temperature at 0 Celsius. Or slowly add 136 grams (4.8 oz.) of 28 – 30% ammonia solution, or 400 grams (14.1 oz.) of a 10% ammonia solution into the diluted nitric acid solution while stirring and maintaining the diluted nitric acids temperature at 0 Celsius. Upon completion of the ammonia addition, recrystallize the ammonium nitrate from the reaction mixture, and then vacuum dry or air dry the product. If a rotary evaporator is available, place the filtered reaction mixture there into, and remove the water under high vacuum. Then, recrystallize the ammonium nitrate from methanol (2 grams (0.07 oz.) ammonium nitrate dissolves in 16 milliliters (0.54 fluid oz.) methanol), and then vacuum dry or air-dry the product. The product will weigh about 180 to 188 grams and will be of 99% purity.

Method 2: Preparation of ammonium nitrate from sodium nitrate, ammonia, and carbon dioxide

(By-products from reaction: sodium bicarbonate)

Materials:

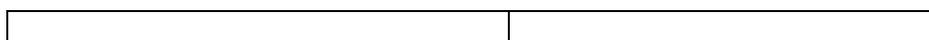
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|--|---|
| 1. 1000 grams (2.2 pounds) of sodium nitrate | 3. 740 grams (26.1 oz.) of 28 – 30% ammonia solution or 2000 grams (4.4 pounds) of 10% ammonia solution or 200 grams (7 oz.) of anhydrous ammonia |
| 2. 2300 milliliters (2.4 quarts) of water | 4. 3530 grams (7.8 pounds) of dry carbon dioxide or 3000 grams (6.6 pounds) of dry ice |

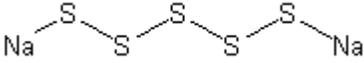
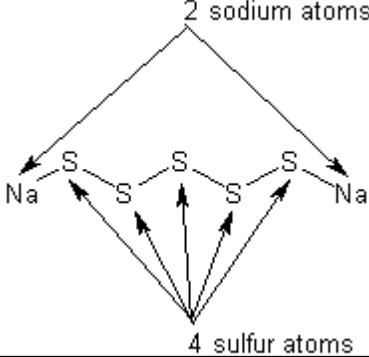
Summary: Ammonium nitrate is easily prepared by dissolving sodium nitrate into water, adding ammonia, and then passing in an excess of carbon dioxide gas (dry may be used if desired). The resulting insoluble sodium bicarbonate is then filtered-off, and the ammonium nitrate is recovered by recrystallization from the water.

Hazards: Use proper ventilation when handling ammonia gas, or its solutions, and avoid inhalation of vapors.

Procedure: Dissolve 1000 grams (2.2 pounds) of sodium nitrate into 2300 milliliters (2.4 quarts) of water. Then add 740 grams (26.1 oz.) of 28 – 30% ammonia solution, or 2000 grams (4.4 pounds) of 10% ammonia solution, or pass 200 grams (7 oz.) of anhydrous ammonia into the nitrate solution while stirring the nitrate solution. Thereafter, place this mixture into an ice bath, and chill to 0 Celsius. Then, pass 3530 grams (7.8 pounds) of dry carbon dioxide into the reaction mixture at a steady rate while stirring the reaction mixture and maintaining its temperature at 0 Celsius, or add in pieces, 3000 grams (6.6 pounds) of dry ice. After the carbon dioxide addition, continue to stir the reaction mixture for 30 minutes at 0 Celsius, and then filter-off the precipitated sodium bicarbonate. Thereafter, recrystallize the ammonium nitrate from the reaction mixture. If a rotary evaporator is available, place the filtered reaction mixture there into, and remove the water under high vacuum. After the recrystallization process, vacuum dry or air-dry the crystals. Then, recrystallize the ammonium nitrate from methanol (2 grams (0.07 oz.) ammonium nitrate dissolves in 16 milliliters (0.54 fluid oz.) methanol), and then vacuum dry or air-dry the product. Recrystallizing the ammonium nitrate from methanol is not necessary if the ammonium nitrate is to be used in making prills. If the ammonium nitrate is to be alloyed with sensitive high explosives such as picric acid, picric acid salts, styphnic acid, or styphnic acid salts, the ammonium nitrate should be recrystallized from methanol.

Preparation 22: Sodium polysulfides



| | |
|---|--|
|  |  |
| Chemical structure | 3D Structure |
|  | Na_2S_4 |
| Structure make-up | Short hand chemical structure |

Method 1: Preparation of sodium polysulfides from sulfur and lye

(By-products from reaction: sodium thiosulfate)

Materials:

| | |
|--|--|
| 1. 250 milliliters (8.5 fluid oz.) of cold water | 3. 50 grams (1.7 oz.) of powdered sulfur |
| 2. 51 grams (1.8 oz.) of sodium hydroxide | |

Summary: Sodium polysulfide is readily prepared by treating a sodium hydroxide solution with excess sulfur. The reaction produces a dark brownish red liquid.

Hazards: Use caution when handling sodium hydroxide, which is capable of producing skin burns.

Procedure: Into a suitable beaker, flask, ect., place 250 milliliters (8.5 fluid oz.) of cold water, and then add and dissolve 51 grams (1.8 oz.) of sodium hydroxide. Note: sodium hydroxide generates excessive heat when dissolved in water so use caution. Thereafter, add in, in small portions at a time, 50 grams (1.7 oz.) of powdered sulfur, and stir the mixture vigorously during the addition. Note: more sulfur may be added if desired. Thereafter, continue to stir the mixture for about 30 minutes. After 30 minutes the mixture is ready for use. Even though the mixture will be contaminated with sodium thiosulfate, it can be used directly in the reduction of nitro compounds.

This method of preparing sodium polysulfides can be used to purify sulfur to high degree. When hydrochloric acid is added to the mixture, pure sulfur precipitates.

Preparation 23: Nitroethane (An advanced procedure)

| | |
|--|--|
| | |
|--|--|

| | |
|--------------------|-------------------------------|
| | |
| Chemical structure | 3D Structure |
| | $C_2H_5NO_2$ |
| Structure make-up | Short hand chemical structure |

Nitroethane

Nitroethane forms a colorless to oily liquid with a pleasant odor. It has a melting point of -50 Celsius, and a boiling point of 115 Celsius. Nitroethane is only very slightly soluble in water, but miscible in alcohol, and ether. It is also soluble in chloroform. Nitroethane forms explosive salts when treated with strong bases. It can be made by reacting ethyl bromide with sodium nitrite.

Method 1: Preparation of nitroethane

Materials:

| | |
|--|---|
| 1. 45 milliliters (1.5 fluid oz.) of ice water | 7. 30 grams (1 oz.) of sodium nitrite |
| 2. 75 milliliters (4 fluid oz.) of 98% sulfuric acid | 8. 27 grams (0.95 oz.) of ethyl bromide |
| 3. 75 milliliters (4 fluid oz.) of 95% ethyl alcohol | 9. 1250 milliliters (42.2 fluid oz.) of ice water |
| 4. 60 grams (2.1 oz.) of potassium bromide or 52 grams (1.8 oz.) of sodium bromide | 10. Five 90-milliliter portions (five 3 fluid oz. portions) of diethyl ether |
| 5. 10 grams (0.35 oz.) of anhydrous calcium chloride | 11. Three 75-milliliter portions (three 2.5 fluid oz. portions) of ice cold water |
| 6. 500 milliliters (16.9 fluid oz.) of dimethylformamide (DMF) | 12. 15 grams (0.52 oz.) of anhydrous magnesium sulfate |

Summary: Nitroethane is readily prepared by condensing ethyl bromide with sodium nitrite. The ethyl bromide can be made on site by the interaction of ethyl alcohol with potassium or sodium bromide in the presence of concentrated sulfuric acid. The ethyl bromide once prepared, can be purified by distillation. Once the reaction between the ethyl bromide and sodium nitrite is complete, the reaction mixture is extracted with ether, the ether removed, and the resulting remaining nitroethane then distilled to recover the purified product.

Hazards: Wear gloves when handling concentrated sulfuric acid, and use caution. Ethyl alcohol, dimethylformamide, and ether are highly flammable. Ether can form explosive mixtures with air, so use caution and extinguish all sources of ignition.

Procedure:

Step 1: Preparation of ethyl bromide

Into a standard flask (see illustration below), equipped with motorized stirrer or other stirring means, addition funnel, and thermometer, place 45 milliliters (1.5 fluid oz.) of ice water. Thereafter, slowly and carefully add in 75 milliliters (4 fluid oz.) of 98% sulfuric acid. Then place this acid mixture into an ice bath, and chill to about 0 Celsius. Afterwards, place 75 milliliters (4 fluid oz.) of

95% ethyl alcohol into the addition funnel, and then slowly add this ethyl alcohol, drop-wise, to the acid mixture. During the addition of the alcohol, stir the acid mixture and maintain its temperature around 0 Celsius at all times. After adding in the ethyl alcohol, slowly add in, 60 grams (2.1 oz.) of potassium bromide or 52 grams (1.8 oz.) of sodium bromide, in small portions at a time, over a period sufficient to keep the reaction mixture at 0 Celsius. After the addition of the bromide salt, continue to stir the entire reaction mixture for about 30 minutes, and thereafter, pour this entire reaction mixture into a distillation apparatus, and distill-off the ethyl bromide at 38 Celsius. When no more ethyl bromide passes over or is collected, stop the distillation process, and then recover the ethyl bromide from the receiver flask. Then add to this collected ethyl bromide, 10 grams (0.35 oz.) of anhydrous calcium chloride, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the calcium chloride. Finally, re-distil this ethyl bromide using a fractional distillation apparatus at 38 Celsius. After the distillation process, collect the ethyl bromide and store it in an amber glass bottle in a refrigerator until use.

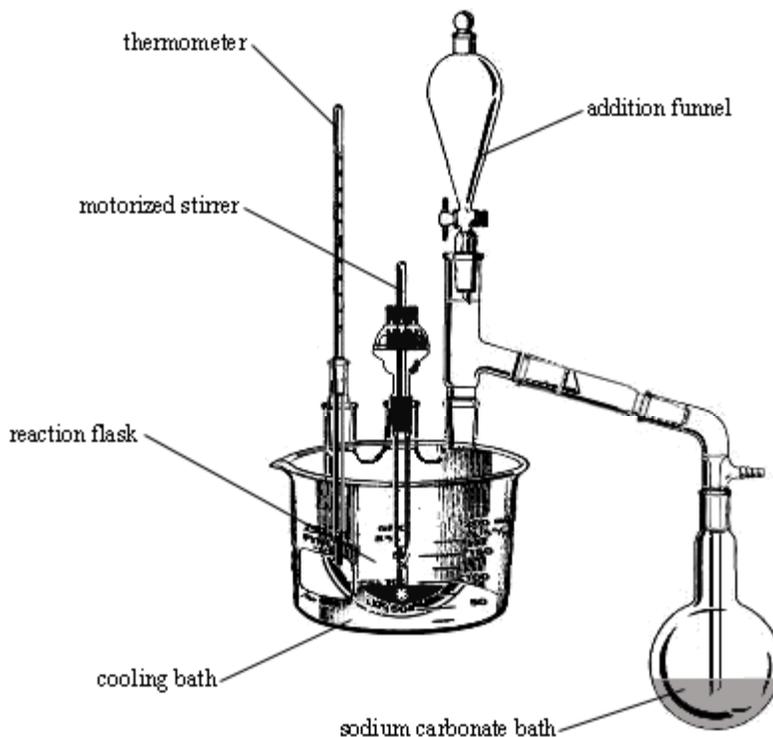


Figure 049. Advanced apparatus for the preparation of ethyl bromide.

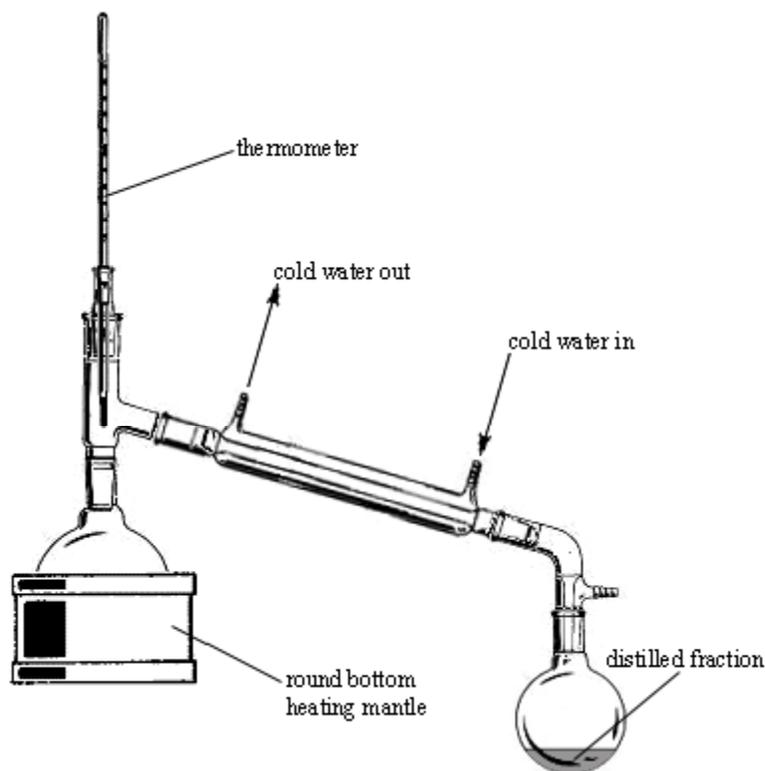


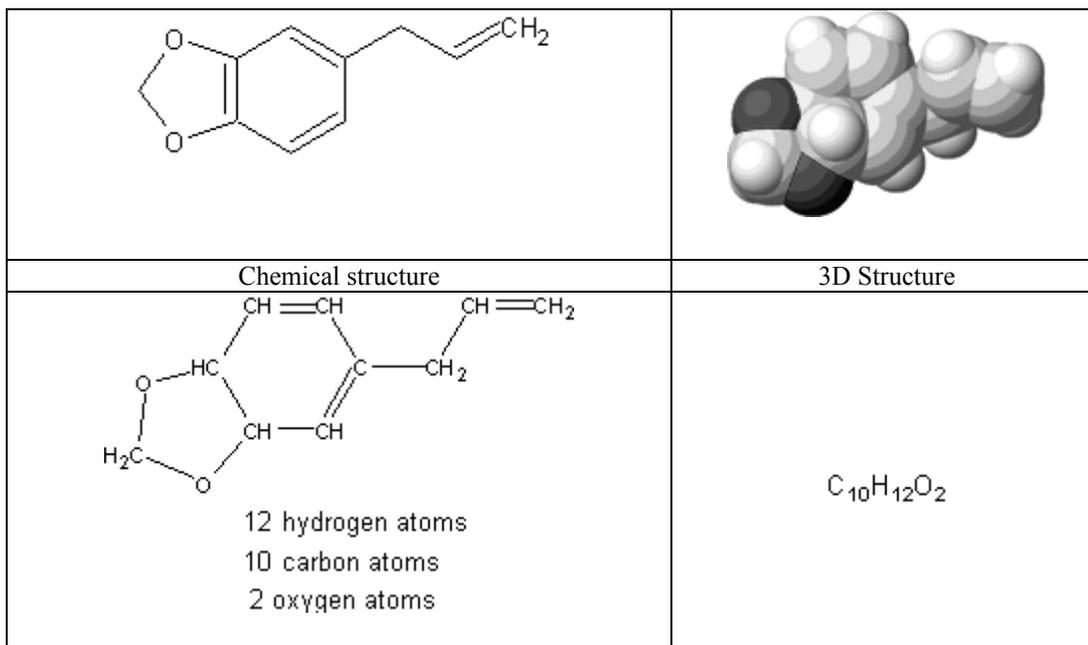
Figure 050. Standard distillation apparatus for the recovery of ethyl bromide.

Step 2: Preparation of nitroethane

Into a standard flask, equipped with motorized stirrer or other stirring means, place 500 milliliters (16.9 fluid oz.) of dimethylformamide (DMF), followed by 30 grams (1 oz.) of sodium nitrite. Thereafter, stir this entire mixture to form a uniform mix, and then place this mixture into a cold-water bath. Afterwards, carefully and gradually add in, 27 grams (0.95 oz.) of ethyl bromide (prepared in step 1), over a period sufficient to keep the reaction mixtures temperature below 25 Celsius at all times. During the addition, rapidly stir the reaction mixture and maintain its temperature below 25 Celsius. After the addition of the ethyl bromide, continue to rapidly stir the reaction mixture for about 6 hours at a temperature below 30 Celsius. After 6 hours, pour the entire reaction mixture into a suitable sized beaker, and then add in 1250 milliliters (42.2 fluid oz.) of ice water. Thereafter, extract this aqueous mixture with five 90-milliliter portions (five 3 fluid oz. portions) of diethyl ether, and after the extraction process, combine all ether portions, if not already done so, and then wash this combined ether portion with three 75-milliliter portions (three 2.5 fluid oz. portions) of ice cold water. Note: after each extraction and washing portion, the ether will be the upper layer each time. After the washing portions, dry the collected washed ether portion by adding to it, 15 grams (0.52 oz.) of anhydrous magnesium sulfate, and then stir the entire mixture for about 10 minutes—thereafter, filter-off the magnesium sulfate. Now, place this dried filtered ether mixture into a distillation apparatus, and distill-off the ether at 40 Celsius. When no more ether passes over or is collected, stop the distillation process, and then recover the left over remaining residue (after it has cooled). Finally, place this left over recovered residue into a distillation apparatus, and distill over the nitroethane at 115 Celsius. When no more nitroethane passes over or is collected, stop the distillation process, and recover the nitroethane (after it has cooled). Then place this nitroethane into an amber glass bottle and store it in a cool dry place until use.

Preparation 24: Safrole (An advanced procedure 2)

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Method 1: Preparation of Safrole

Materials:

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| 1. 13 grams (0.45 oz.) of catechol | 11. with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride |
| 2. 14 grams (0.49 oz.) of allyl bromide | 12. 15 grams (0.52 oz.) of anhydrous magnesium sulfate |
| 3. 22 milliliters (0.74 fluid oz.) of dry acetone | 13. 50 milliliters (1.7 fluid oz.) of methylene chloride |
| 4. 17 grams (0.59 oz.) of finely divided anhydrous potassium carbonate | 14. 250 milliliters (8.5 fluid oz.) of dimethyl sulfoxide (DMSO). |
| 5. 25 milliliters (0.84 fluid oz.) of cold water | 15. 74 grams (2.6 oz.) of 4-allyl catechol |
| 6. 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid solution | 16. 40 grams (1.4 oz.) of sodium hydroxide |
| 7. 50-milliliter portion (1.7 fluid oz.) of diethyl ether | 17. 10 milliliters (0.33 fluid oz.) of methylene chloride |
| 8. 35 grams (1.2 oz.) of sodium hydroxide | 18. 1.5 grams (0.05 oz.) of sodium hydroxide |
| 9. 150 milliliters (5 fluid oz.) of water | 19. 500 milliliters (17 fluid oz.) of water |
| 10. 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid | |

Summary: Safrole can be synthetically prepared by methylating 4-allyl catechol. This 4-allyl catechol is prepared by reacting catechol with allyl bromide, followed by an exhaustive extraction process, and then followed by a pain staking distillation process. Once the 4-allyl catechol has been successfully obtained, it is converted into safrole by refluxing it with methylene chloride and dimethyl sulfoxide in the presence of sodium hydroxide. The resulting reaction mixture is then distilled to remove any unreacted methylene chloride, and the resulting mixture is then steam distilled to collect the oily safrole product, which is then collected via a separatory funnel.

Hazards: Extinguish all flames before using diethyl ether, which is highly flammable, and can form explosive mixtures with air. Use care when handling acetone, which is also highly flammable. Use maximum ventilation when handling allyl bromide, which is very irritating to the eyes, nose, and throat.

Procedure:

Step 1: Preparation of 4-allyl catechol

Into a suitable reflux apparatus, place 13 grams (0.45 oz.) of catechol, followed by 14 grams (0.49 oz.) of allyl bromide, and then add in 22 milliliters (0.74 fluid oz.) of dry acetone. Then stir the entire mixture to form a uniform mixture. Immediately thereafter, gradually add in 17 grams (0.59 oz.) of finely divided anhydrous potassium carbonate, and stir the mixture while adding this potassium carbonate. After the addition of the potassium carbonate, reflux the entire reaction mixture at 60 Celsius for about 3 hours. Note: fit a calcium chloride drying tube to the top of the reflux condenser to keep moisture out from the apparatus. After refluxing for about 3 hours, quickly remove the reflux condenser, and replace it with a conventional cold water condenser, fitted with a receiver

flask, and then distill-off the acetone until no more acetone passes over into the receiver flask. When this point is reached, stop the distillation process, and allow the reaction mixture to cool to room temperature. Thereafter, pour the distilled reaction mixture left over, into a clean beaker, and then add in 25 milliliters (0.84 fluid oz.) of cold water, followed by 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid solution. Then stir the entire acidic reaction mixture for about 10 minutes. Thereafter, extract the entire reaction mixture with one 50-milliliter portion (1.7 fluid oz.) of diethyl ether. After the extraction process, wash the ether portion by adding to it, a sodium hydroxide solution prepared by adding and dissolving 35 grams (1.2 oz.) of sodium hydroxide into 150 milliliters (5 fluid oz.) of water. Note: the addition of sodium hydroxide to water generates much heat, so allow the mixture to cool to room temperature before using. Thereafter, remove the upper ether layer by using a separatory funnel, or by decantation, and then discard or recycle this upper ether layer (will contain diallyl ether). Now to the lower water layer, add in 100 milliliters (3.4 fluid oz.) of 10% sulfuric acid, and upon the acid addition, some oil should separate. After the addition of the sulfuric acid, extract the entire acidic mixture (including any separated oil) with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride. Note: after each extraction, the methylene chloride will be the upper layer. After the extraction process, combine all methylene chloride extracts, if not already done so, and then dry this combined methylene chloride mixture by adding to it, 15 grams (0.52 oz.) of anhydrous magnesium sulfate—thereafter, stir the whole mixture for about 10 minutes, and then filter-off the magnesium sulfate. Thereafter, place the filtered methylene chloride mixture into a distillation apparatus or rotary evaporator, and remove the methylene chloride. When no more methylene chloride is collected, recover the left over remaining oil. Now, to this oil, place it into a reflux apparatus, and heat it to 180 Celsius. Note: during the heating process, the oil will self heat raising the temperature to about 260 Celsius. When this temperature change results, stop the heating process, and then place the oil (which will now be red in color) into a vacuum distillation apparatus (after it has cooled, or simply replace the reflux condenser with the appropriate glass adapters and immediately begin the vacuum distillation process), as similar to the one used for the distillation of safrole as listed above, but use only one condenser and receiver rather than two, and distill the oil at 158 Celsius under a vacuum of 16 millimeters of mercury. When no more oil is obtained at this temperature and vacuum, stop the distillation process, and then remove the left over remaining residue, and discard it. To the collected fraction, re-vacuum distill it using the same apparatus (after it has been cooled, and cleaned), and re-vacuum distill the oil at 158 Celsius, under a vacuum of 16 millimeters of mercury to obtain a refined 4-allyl catechol product.

Step 2: Preparation of safrole by methylating the 4-allyl catechol

Into a suitable 3-neck flask fitted with motorized stirrer, reflux condenser, thermometer, and a mercury bubbler fitted into the left or right female joint of the 3-neck flask to form an apparatus that is excluded from the atmosphere. Note: the top of the reflux condenser should be fitted with a calcium chloride drying tube, and to the other end of the drying tube, a gas inlet tube connected to a cylinder of dry nitrogen should be in place. Before assembling the calcium chloride drying tube with nitrogen purge adapter, charge the 3-neck flask with 50 milliliters (1.7 fluid oz.) of methylene chloride, followed by 250 milliliters (8.5 fluid oz.) of dimethyl sulfoxide (DMSO). Thereafter, begin the nitrogen purge and allow the entire apparatus to be flushed with nitrogen to exclude air and moisture—the mercury bubbler is to allow the nitrogen gas to run through the apparatus, and then escape into the atmosphere without exposing the apparatus to the atmosphere. Once the apparatus has been purged with nitrogen, reflux the contents in the 3-neck flask using a heating mantle to 130 Celsius, and when the temperature of these contents reaches the desired 130 Celsius mark, add in 3.7 grams (0.13 oz.) of 4-allyl catechol (obtained in step 1), followed immediately by 2 grams (0.07 oz.) of sodium hydroxide. Note: the 4-allyl catechol and sodium hydroxide can be quickly added through the top of the reflux condenser by quickly and temporarily removing the calcium chloride drying tube briefly, and then reattaching it after the addition. This process of addition should then be repeated 19 times (3.7 grams of 4-allyl catechol and 2 grams of sodium hydroxide each time for a total of 74 grams (2.6 oz.) of 4-allyl catechol and 40 grams (1.4 oz.) of sodium hydroxide. During each addition maintain the reflux at 130 Celsius. After the last addition, reflux the entire reaction mixture for an additional 10 minutes, and thereafter, add in 10 milliliters (0.33 fluid oz.) of methylene chloride followed by 1.5 grams (0.05 oz.) of sodium hydroxide. Thereafter, continue to reflux the reaction mixture at 130 Celsius for 35 minutes. Finally, after refluxing for the final 35 minute time period, stop the heating and reflux, and then allow the entire reaction mixture to cool to room temperature. Then pour the entire reaction mixture into a distillation apparatus, and distil-off any unreacted methylene chloride. When no more methylene chloride distills over, allow the left over remaining contents to cool to room temperature before removing from the apparatus, and then place these contents into a steam distillation apparatus, and then add in 500 milliliters (17 fluid oz.) of water, and then steam distill the safrole from the mixture to obtain safrole and water in the receiver flask. The oily safrole layer can then be recovered by using a separatory funnel in the usual manner. The safrole can be vacuum distilled at 100 Celsius under a vacuum of 11 millimeters of mercury to obtain a refined product if desired.

SECTION 4: Electrochemical processes

Part C: Introduction to Electro chemical methods in general chemistry

Electrochemical processes involve specially designed cells for use in carrying out unique and un-common chemical reactions that would otherwise be impossible. For most people, electrochemistry will be unfamiliar, and strange; however, electrochemistry is by far the most interesting of the laboratory techniques, and can be learned by anyone with ease. You don't have to be a chemist to carry out your own electrochemical processes, and the cell design, function, and operation is very easy to learn and operate. Electrochemistry is a fun aspect of laboratory procedures, and can be used to produce a variety of interesting substances.

1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL

1. Introduction

A standard diaphragm cell can be made from simple and inexpensive store bought materials. Anyone can easily assemble their own cell, and carry out interesting and useful procedures to make a variety of interesting products. First of all, you need to learn how to assemble your own homemade cell. The first thing you will need to do is go to the store and purchase the following materials:

1. 1 box of Epsom salt (available in any grocery store)
2. 1 standard clay pot (just under 1 quart size; 500 milliliters; try to find one that does not have a hole in the bottom)
3. 1 plastic Tupperware container w/lid (just under ½ gallon size; about 1800 milliliters)
4. 2 lead electrodes (about 6 inches long each).
5. 1 car battery charger; with power output with at least 12 volt 6 amp (available in most stores for \$20.00 to \$40.00)

Note: The lead electrodes should be rectangular with dimensions of 5 to 6 inches long, by ½ inch to ¼ inch in diameter (they don't have to be perfectly round, and can be square or rectangle). The lead electrodes may be circular if desired with dimensions of 6 inches long by 0.30 to 0.50 inch radius (9 millimeter to 12.9 millimeter). The lead electrodes can be made by melting pieces of lead into a make shift mold, made by forming a rectangular shaped mold using aluminum foil. After shaping the mold with the aluminum foil, place the pieces of lead there into, and then use a standard propane torch to melt the lead. After allowing the lead to cool, remove the aluminum foil. Lead can be found in car batteries, and in lead solder for welding copper and in electronics.

Note: The clay pot is a standard clay flowerpot, which can be found at any hardware store or pottery store. The clay pot should not have a hole in the bottom, as most of them do. *If a clay pot with no hole in the bottom is not available, use plumbers putty to seal the hole on both sides.*

2. Assemble your cell

Now that you have your materials, assemble your cell. Use the following illustration to aid you. Note: For the electrodes, suitable sized holes should be cut into the plastic lid. One hole on the left or right side of the plastic lid, and the other directly in the center of the plastic lid. The holes if properly cut, can secure the electrodes firmly (with a tight fit). The clay pot is then placed into the plastic Tupperware container as shown in the illustration. Note: The plastic lid should be able to fit securely onto the plastic Tupperware container without disrupting the clay pot. A small hole should also be cut into the plastic lid to allow for gas venting. Note: This cell is not airtight. To make the cell airtight, the holes in which the electrodes protrude need to be sealed airtight. To do this, a glue gun may be used, or plumbers putty. The plastic lid should also be sealed gas tight. To do this, wrap Teflon tape (available at any hardware store) around the outer lip of the plastic Tupperware container many times, so that when the lid is attached, it forms a firm seal. Note: Other methods may be used to create an airtight cell. Use your own imagination, and see what you can come up with. An airtight seal of your cell is not necessary for most procedures. An airtight seal is only desired if a producing gas is the desired product, as in the production of chlorine gas.

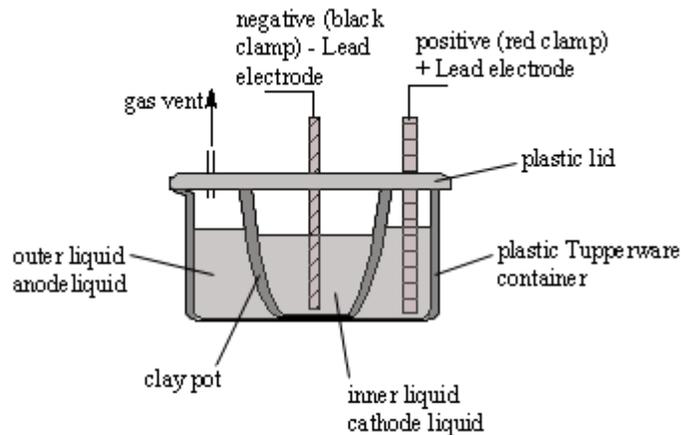


Figure 051. Setup for the assembly of the cell. Note: The lead electrodes can be replaced with graphite, but lead is better to reduce corrosion of the electrodes.

3. Charge your cell

Now that you have setup your cell, the next task is to "charge the cell". What does this mean? Well, to put it into simple terms, the clay pot acts as a salt bridge. In order for this salt bridge to work properly, it must be "Charged" with ions, so that during a particular process, the electrochemical reaction works properly. To charge the cell, carryout the following:

1. Into a beaker or other container, place 1500 milliliters (50.7 fluid oz.) of water. Then add 100 grams (3.6 oz.) of Epsom salt, and stir the water to completely dissolve the Epsom salt.
2. Place 500 milliliters (17 fluid oz.) of this Epsom salt solution into the clay pot. This will be called the "cathode" liquid, designating the "negative" side.
3. Place the remaining Epsom salt solution into the plastic Tupperware container (the outer compartment). This will be called the "anode" liquid, designating the "positive" side.
4. Then secure the plastic lid to the plastic Tupperware container, and then secure both electrodes.
5. Then attach each electrode to their respective electric wire from the battery charger. Note: The battery charger comes with a black clamp, and a red clamp. The black clamp is the "negative", and the red clamp is the "positive". Note: The battery charger only works when plugged in! Before plugging the battery charger in, secure both clamps to the cell. The black clamp goes on the negative (-) lead electrode, and the red clamp goes on the positive (+) lead electrode.
6. When everything is in place, plug the battery charger in, and allow it to run for about 6 hours. Note: During the process, hydrogen gas will be steadily evolved. Carryout the process in a ventilated area, such as a garage, bathroom, or shed.
7. After 6 hours, unplug the battery charger, and remove the clamps from each electrode. Then open the plastic lid, and pullout the clay pot. Note: The clay pot will be filled with a white precipitate. This white precipitate is *magnesium hydroxide*. The cathode liquid will contain a *dilute sulfuric acid solution*. This dilute sulfuric acid solution can be saved, if desired, or mixed with the contents in the clay pot. Mixing both solutions will reform magnesium sulfate (Epsom salt). If you wish to recycle the Epsom salt for future cell charge, mix both liquids. **Note:** To verify, for your own amusement, drop in a little baking soda to the anode liquid.
8. After you have decided what you want to do with both liquids, rinse out the clay pot with tap water, and do the same for the plastic Tupperware container. **Your clay pot is now charged.**

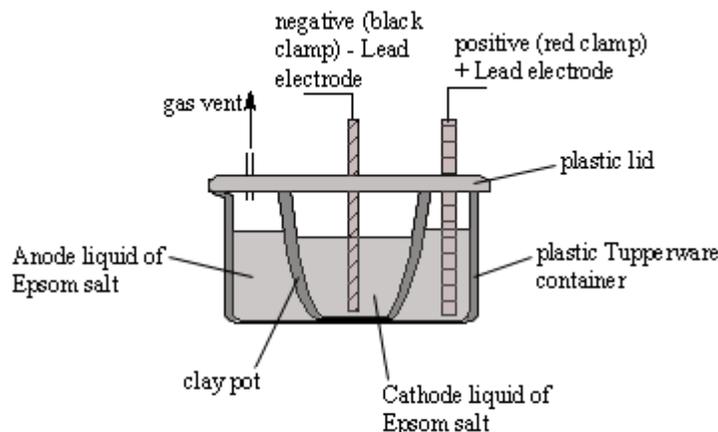


Figure 052. Charging the cell

4. Carryout your first procedure (*The Preparation of ferrous chloride*)

Now you're ready to carry out your first process. The following process will help you familiarize yourself with electrochemical methods, and the endless processes that can be carried out. So let's get started!

With your "charged" cell (just established), reassemble the apparatus as illustrated above. This time, instead of using Epsom salt, we are going to use another common salt, called "pickling salt", which is sodium chloride. Also, the contents in the clay pot will be considered the anode liquid, and the contents in the Tupperware container the cathode liquid. Under most conditions, sodium chloride is relatively inert. Although, when we place this salt into a cell, like the one we have, strange things begin to happen! Now is the time for you to find out what! Go to the store and purchase a box of "pickling" salt. Make sure it's just pure salt with nothing added.

1. Assemble the cell as illustrated above. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then dissolve another 50 grams (1.8 oz.) of salt into 1 liter (33.8 fluid oz.) of water. Thereafter, pour the 300 milliliters (10 fluid oz.) of salt solution into the clay pot (anode liquid). Then pour the 1-liter (33.8 fluid oz.) of salt solution into the outer plastic container (cathode liquid).
2. Now secure the plastic lid to the Tupperware container, and then slide in 1 lead or graphite electrode into the cathode side (outer side). The same lead electrode or graphite electrode you used during the charging process may be used. Now, instead of using a lead electrode or graphite electrode in the anode compartment, we are going to use an iron one. Go to the hardware store, and purchase an iron rod (about ¼ inches in diameter, and about 12 inches long, or ½ inch in diameter by 12 inches long). 3 Foot long iron rods are usually sold in hardware stores for welding purpose. This rod can be cut into segments of 1 foot each, or simply purchase an iron nail, bolt, or equivalent.
3. Using the same plastic lid as before, or using a different one (of the same size with a custom hole designed just for your iron rod), slide in the iron electrode.
4. Then hook up the battery charger clamps to their respective electrodes (black for negative, red for positive; positive for iron, negative for the lead or graphite electrode), and then plug in the battery charger.
5. Allow the battery charger to run for about 6 hours. Note: hydrogen gas will be steadily evolved during the process. Use a well-ventilated area as before.
6. After 6 hours, unplug the battery charger, and then open the lid to the plastic container. Then carefully remove the clay pot, and then filter the contents of the clay pot. The contents of the clay pot will contain ferrous chloride. By this time you will notice that part of your iron electrode has disappeared! Never fear, it's now in the form of ferrous chloride, which is dissolved in the anode liquid. After filtering said anode liquid, you will notice that it has a light greenish color to it. This greenish color is caused by ferrous chloride hydrate.
7. Filter-off the cathode liquid, if desired, and then perform a little experiment. Add a few drops of the anode liquid to a small sample of your cathode liquid. Wham! What you get is a gelatinous precipitate of ferrous hydroxide hydrate. *The cathode liquid will contain sodium hydroxide, commonly called lye.* Use this solution to unclog your sink, or simply discard it. If you want, you can play around with it a little. Take some aluminum foil, and drop it into your cathode liquid. Watch as the sodium hydroxide in the cathode liquid consumes the aluminum foil, producing hydrogen gas.

8. *The anode liquid can be evaporated (by simply boiling-off the water), to yield crystals of ferrous chloride.* Before hand, if you like, add a few drops of your anode liquid to a small sample of Clorox bleach or hydrogen peroxide. Wham! What happened? It turned black! The ferrous chloride was oxidized by the Clorox bleach (or hydrogen peroxide) to ferric chloride. Ferric chloride is commonly used by electricians to etch printed circuit boards!

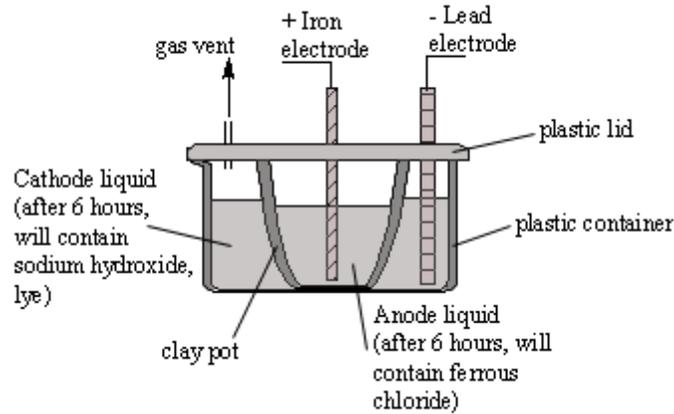


Figure 053. Preparation of ferrous chloride
Carryout a similar procedure (procedure 2)

5. Now that you have learned to make ferrous chloride, you can apply this new skill to making other metal chlorides. Let's try one more using a different metal anode, and then we will move onto something more advanced.

1. Assemble the cell as previously described, using a clean, freshly charged cell. Note: In this procedure, a brand new clay pot should be charged using the aforementioned process for cell charging. If we wanted to carryout another process using another iron anode, we could use the same cell we just finished with (a freshly charged cell can be used for the SAME procedure multiple times without having to recharge said cell). Although, since we are going to use a copper anode in this procedure, we should either recharge the cell we just used (for the ferrous chloride preparation), or we should use a clean, freshly charged clay pot.
2. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then add this solution to the clay pot (anode liquid). Then dissolve 50 grams (1.8 oz.) of sodium chloride into 1 liter (33.8 fluid oz.) of water, and then pour this solution into the plastic Tupperware container (cathode liquid).
3. Then attach the plastic lid, and then insert your electrodes. 1 Lead of graphite electrode for the negative cathode, and a copper electrode for the positive anode. Note: Use copper pipes about 10 to 12 inches long, by ½ inch in diameter for the electrode. If desired, you can use copper wire of up to 20 gauge, wound around a pencil to form a coil, as the anode.
4. Then attach the battery charger clamps to their respective electrodes (red for copper, black for lead or graphite).
5. Then plug in the battery charger, and allow it to run for 6 hours. After 6 hours, unplug the battery charger, and then open the cell. Remove the clay pot from the plastic Tupperware container, and then pour the contents into a clean glass, or beaker. You can discard the cathode liquid if desired, as it will contain sodium hydroxide (lye). Allow the anode liquid to stand for 24 hours, and then carefully decant (pour off), the liquid above the bluish precipitate. The bluish precipitate will be very finely divided cupric hydroxide, which is very difficult to filter-off. Use centrifuge apparatus if you have one. The anode liquid will have an emerald greenish-blue color to it. This color is caused by cupric chloride hydrate. If you want, you can add a few drops of this cupric chloride hydrate to a small sample of your cathode liquid, to obtain an interesting precipitate of cupric hydroxide hydrate. *You can evaporate the anode liquid to dryness (by simply boiling off the water) to obtain brownish black crystals of cupric chloride.*

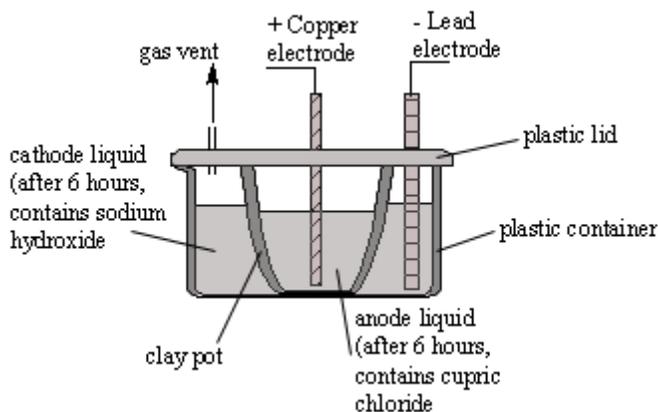


Figure 054. Preparation of cupric chloride

Now that we have run through the two simple processes. Its time to move onto something a little more advanced. To review what we have learned so far, look at the two similarities between the two procedures we have done. In the first procedure we made ferrous chloride by electrolyzing a salt solution using an iron anode. In the second procedure we used a copper anode, and got cupric chloride. Now, you should remember that if we replaced the copper anode with zinc for example, we would get zinc chloride. Note: Any metal can be used with the exception of lead, platinum, and a few others we need not discuss at this point. If you want, try it with aluminum, zinc, nickel, chromium, or magnesium to get the respective chlorides. Note: The sodium chloride can be replaced with sodium bromide, or sodium iodide to make the corresponding bromides and iodides.

6. Now lets try something a little bit more interesting

This time, we will use more then one anode, to carryout a double reaction. In this example, we will make ferric chloride hydrate by using an iron anode, and a graphite anode. The graphite anode should be 8 to 12 inches long, by at least ¼ inch in diameter. Note: these graphite rods can be obtained from numerous arts and craft stores. Look for arts and crafts stores that sell drawing freehand accessories. Graphite is a form of carbon, and is commonly called "pencil lead".

1. Assemble the cell as previously described, using a clean, freshly charged cell. Note: In this procedure, a brand new clay pot should be charged using the aforementioned process for cell charging. If we wanted to carryout another process using another copper anode, we could use the same cell we just finished with (a freshly charged cell can be used for the SAME procedure multiple times without having to recharge said cell). Although, since we are going to use two different anodes in this procedure, we should either recharge the cell we just used (for the cupric chloride preparation), or we should use a clean, freshly charged clay pot.
2. Then dissolve 50 grams (1.8 oz.) of sodium chloride into 300 milliliters (10 fluid oz.) of water, and then add this solution to the clay pot (anode liquid). Then dissolve 50 grams (1.8 oz.) of sodium chloride into 1 liter (33.8 fluid oz.) of water, and then pour this solution into the plastic Tupperware container (cathode liquid).
3. Then attach the plastic lid, and then insert your electrodes. 1 Lead electrode for the negative cathode, a graphite electrode and a iron electrode for the positive anode.
4. Then attach the battery charger clamps to their respective electrodes; Red for graphite and iron (to connect a single battery charger clamp to both electrodes, wrap a copper wire around both the graphite and iron electrodes), and black for lead.
5. Then plug in the battery charger, and allow it to run for 6 hours. After 6 hours, unplug the battery charger, and then open the cell. Remove the clay pot from the plastic Tupperware container, and then pour the contents into a clean glass, or beaker. You can discard the cathode liquid if desired, as it will contain sodium hydroxide (lye). Filter the anode liquid (using a standard plastic funnel, and coffee filters). The anode liquid will have a dark brown to black color to it. This color is caused by ferric chloride hydrate. If you want, you can add a few drops of this ferric chloride hydrate to a small sample of your cathode liquid, to obtain an interesting precipitate of ferric hydroxide hydrate. You can evaporate the anode liquid to dryness to obtain brownish black crystals of ferric chloride hydrate. Note: Do not boil the anode liquid to drive off the water. Doing so will produce highly corrosive hydrochloric acid vapors. If you want, you can boil the anode liquid under appropriate conditions to collect, and then condense the hydrochloric acid for later use.

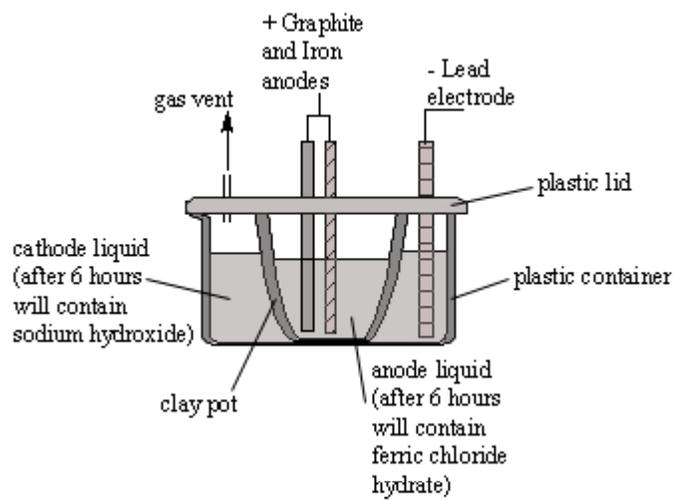
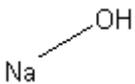
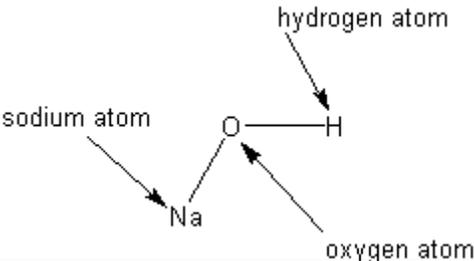


Figure 055. Preparation of ferric chloride

Now you can try different combinations if desired, by using different electrodes, and using multiple electrodes.

Part D: Electro chemical methods in general chemistry

Electro preparation 1: Sodium Hydroxide (Lye)

| | |
|--|--|
|  |  |
| Chemical structure | 3D Structure |
|  | NaOH |
| Structure make-up | Short hand chemical structure |

Sodium hydroxide

Sodium hydroxide forms fused solid pieces, granules, rods, or powder. It rapidly absorbs moisture and carbon dioxide from the air. Solutions of sodium hydroxide are very corrosive to animal tissue, and aluminum. It has a melting point of 318 Celsius. Sodium hydroxide is very soluble in water and alcohol. It generates large amounts of heat when dissolving in water, or when mixed with acid. Sodium hydroxide is toxic. Handle sodium hydroxide with care. Sodium hydroxide is a widely available commercial chemical, which is sold under a variety of names such as “Lye”. Sodium hydroxide is prepared on an industrial scale in a procedure called the “chloro-alkali” process. In the chloro-alkali process, a sodium chloride solution is electrolyzed in a special cell composed of two compartments separated by a porous membrane. Chlorine gas is produced at the positive anode, and sodium hydroxide forms at cathode.

Method 1: Preparation of Sodium Hydroxide from pickling salt (sodium chloride)

(By-products from reaction: chlorine gas and hydrogen gas)

Materials:

| | |
|--|--|
| 1. 500 grams (1.1 lbs.) of table salt (sodium chloride preferable sold under the name “pickling salt”) | 3. 1000 milliliters (2.1 pints) of tap water |
| 2. 1500 milliliters (3.1 pints) of tap water | |

Summary: Sodium hydroxide can be prepared by electrolyzing a sodium chloride solution in a two-compartment cell separated by a porous membrane. Chlorine gas is liberated at the positive anode and hydrogen and sodium hydroxide are liberated at the cathode. *Use*

proper ventilation when running the electrolysis cell because of chlorine and hydrogen gas evolution. Run the cell in an area that is away from direct sunlight.

Hazards: Chlorine gas is produced in this procedure; either properly vent the gas, or neutralize it by bubbling it through a sodium hydroxide or sodium carbonate solution. Carryout this procedure away from direct sun-light, and keep any source of ignition away—hydrogen gas is very flammable and explosive.

Procedure: Prepare the cell shown in the following illustration, and then add 500 grams (1.1 lbs.) of table salt (sodium chloride preferable sold under the name “pickling salt”) to a beaker and then add 1500 milliliters (3.1 pints) of tap water. Then stir the mixture to dissolve the table salt. After which, pour 1000 milliliters (2.1 pints) of tap water into the apparatus cathode compartment. Then pour about 100 milliliters (3.4 fluid oz.) of the sodium chloride solution into the cathode compartment to bring its total volume to about 1100 milliliters. Afterwards, pour the rest of the sodium chloride solution into the apparatus anode compartment. Then put the graphite electrodes in place and electrolysis at 12-amp/12-volt until no more chlorine gas is evolved. When no more chlorine is evolved, stop the electrolysis. Then pour the cathode liquid into a beaker, and then filter to remove any insoluble materials. After filtering, pour the sodium hydroxide solution into a clean stainless steel beaker and then boil-off the water until dry sodium hydroxide solid remains. Do not use glass when boiling-off the water because the sodium hydroxide will corrode the glass and cause it to break.

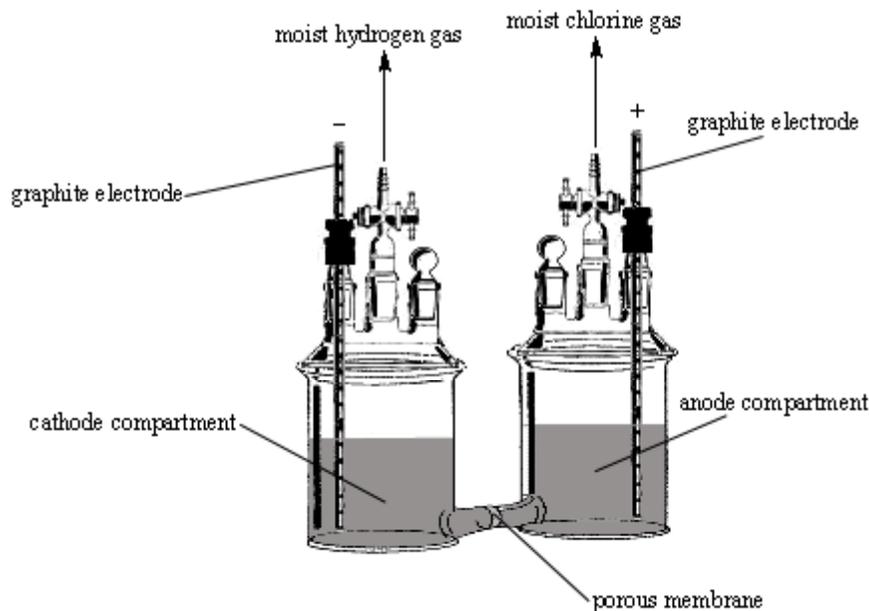
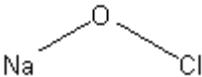
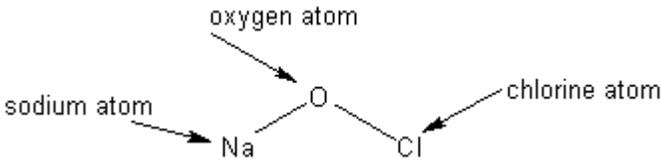


Figure 056. Apparatus for the production of sodium hydroxide. Chlorine gas is liberated at the positive anode electrode, and hydrogen gas is liberated at the negative cathode electrode. The sodium hydroxide is formed at the negative cathode electrode and remains dissolved in water.

Electro preparation 2: Bleach (sodium hypochlorite)

| | |
|--|--|
| | |
|--|--|

| | |
|---|---|
|  |  |
| Chemical structure | 3D Structure |
|  | NaOCl |
| Structure make-up | Short hand chemical structure |

Sodium hypochlorite solution

Sodium hypochlorite solution, commonly called bleach, is a light yellowish liquid with a characteristic chlorine-like odor. It is a powerful oxidizing agent, and is used extensively in disinfections and decontamination procedures. It is quite stable at room temperature, but decomposes when heated forming sodium chlorate and salt. It can be easily prepared using a diaphragm cell, or by passing chlorine gas into a cold dilute sodium hydroxide solution.

Method 1: Preparation of Sodium Hypochlorite solution from pickling salt (sodium chloride)

(By-products from reaction: some chlorine gas, sodium hydroxide, and hydrogen gas)

Materials:

| | |
|---|---|
| 1. 100 grams (3.5 oz.) of pickling salt | 3. 25 grams (1.0 oz.) of pickling salt |
| 2. 350 milliliters (11.8 fluid oz.) of cold tap water | 4. 350 milliliters (11.8 fluid oz.) of cold tap water |

Summary: Bleach can be made using an electrochemical process whereby pickling salt is electrolyzed. During the process, chlorine gas is evolved at the anode, and sodium hydroxide is liberated at the cathode. As the process proceeds, the chlorine reacts with the sodium hydroxide forming sodium hypochlorite (bleach). Some chlorine gas does escape, so use proper ventilation when carrying out this operation.

Hazards: Perform this operation in a well-ventilated area as chlorine gas is evolved. However, if you have an airtight system, the operation can be performed within enclosed areas as long as you neutralize the chlorine gas by bubbling it into a baking soda slurry or lye solution.

Procedure: First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell as been properly charged with Epsom salt, you can proceed with preparation 2: sodium hypochlorite solution.

Now, dissolve 100 grams (3.5 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 25 grams (1.0 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, and then place this salt solution into the anode compartment (+). Now, assemble the cell as illustrated below, using two lead electrodes, and then begin the operation by turning on your power supply, or by plugging in your battery charger. Allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not exceed 40 Celsius.

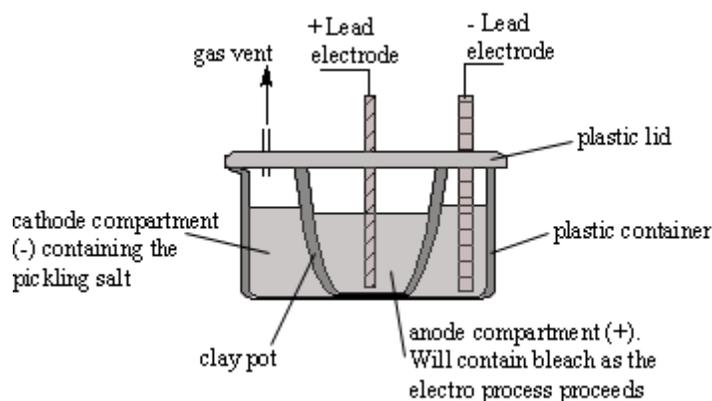


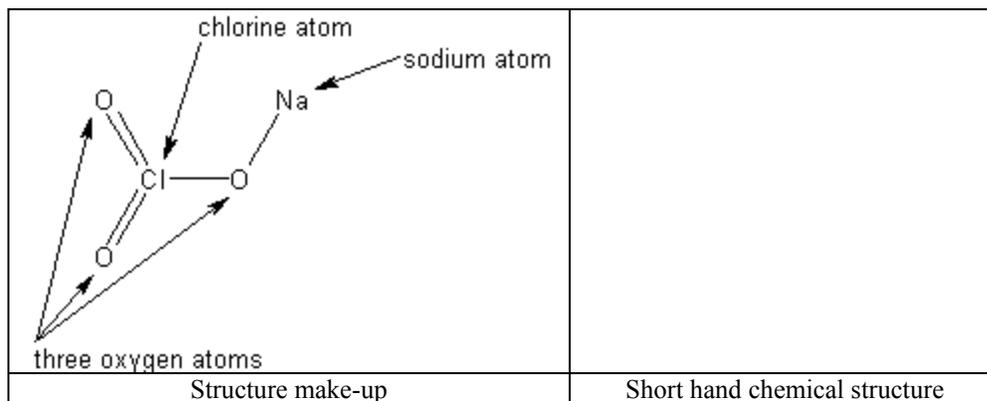
Figure 057. Set-up for the preparation of bleach from pickling salt. The lead electrodes can be replaced with titanium, chromium, or graphite.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the bleach. The cathode compartment will contain some sodium hydroxide. You can discard the cathode liquid if desired, or you can use it for other applications. Your bleach should then be quickly filtered, to remove any insoluble impurities, and then stored in any suitable containers. The bleach solution will be about 20 to 25% concentration. Note: the cathode liquid will probably also contain some bleach admixed with the sodium hydroxide, and un-reacted salt.

Note: Bleach can also be made by bubbling chlorine gas into a solution of lye.

Electro preparation 3: Sodium Chlorate

| | |
|--------------------|--------------------|
| | |
| Chemical structure | 3D Structure |
| | NaClO ₃ |



Sodium chlorate

Sodium chlorate forms colorless, odorless crystals or white granules. The melting point of the crystals is 248 Celsius, but it begins to decompose into oxygen and sodium perchlorate when heated to 300 Celsius. The crystals are highly soluble in water, but relatively insoluble in alcohol, and most common organic solvents. Sodium chlorate is widely used in pyrotechnic compositions, and in the preparation of ammonium chlorate, and perchlorate, which are used in powerful solid rocket fuels.

Method 1: Preparation of Sodium chlorate from pickling salt

(By-products from reaction: sodium hypochlorite, chlorine gas, sodium hydroxide, and hydrogen gas)

Materials:

| | |
|--|---|
| 1. 100 grams (3.5 oz.) of pickling salt | 5. 5 drops of concentrated sulfuric acid |
| 2. 350 milliliters (11.8 fluid oz.) of hot tap water | 6. 100 milliliters (3.4 fluid oz.) of rubbing alcohol |
| 3. 25 grams (1.0 oz.) of pickling salt | 7. 150 milliliters (5 fluid oz.) of warm tap water |
| 4. 350 milliliters (11.8 fluid oz.) of hot tap water | |

Summary: Sodium chlorate can be made using an electrochemical process whereby pickling salt is electrolyzed under slightly different conditions than as in the preparation of bleach. During the process, some chlorine gas is evolved at the anode, and sodium hydroxide is liberated at the cathode. As the process proceeds, the chlorine reacts with the sodium hydroxide forming sodium hypochlorite (bleach); this bleach then spontaneously oxidizes under heat to form sodium chlorate and sodium chloride. Some chlorine gas does escape, so use proper ventilation when carrying out this operation.

Hazards: Perform this operation in a well-ventilated area as chlorine gas is evolved. However, if you have an airtight system, the operation can be performed within enclosed areas as long as you neutralize the chlorine gas by bubbling it into a baking soda slurry or lye solution.

Procedure: First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell has been properly charged with Epsom salt, you can proceed with preparation 3: sodium chlorate.

Now, dissolve 100 grams (3.5 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of hot tap water. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 25 grams (1.0 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of hot tap water, and then add in 5 drops of concentrated sulfuric acid. Then place this acidified salt solution into the anode compartment (+). Now, assemble the cell as illustrated below, using two lead electrodes, and then begin the operation by turning on your power supply, or by plugging in your battery charger. Allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not fall below 50 Celsius. The optimal temperature for maximizing chlorate formation in the cell is around 80 to 90 Celsius.

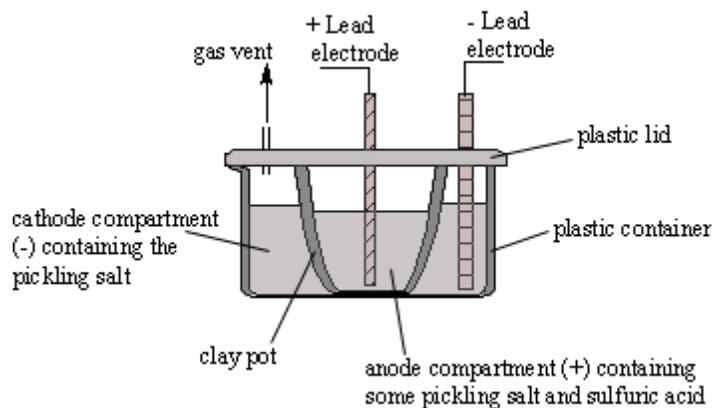
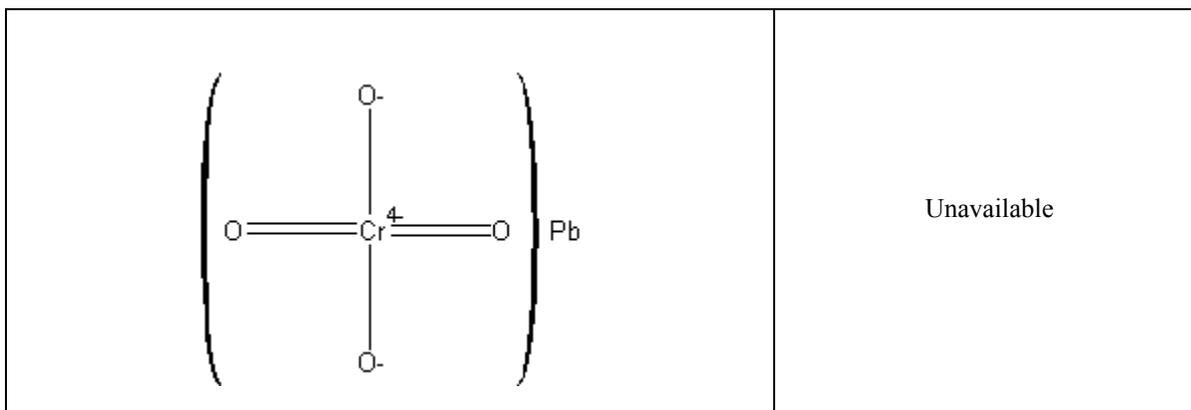


Figure 058. Set-up for the preparation of sodium chlorate from pickling salt. The lead electrodes can be replaced with titanium, chromium, or graphite.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the bulk of the sodium chlorate, as well as some bleach, and small amounts of sodium perchlorate and sodium hydroxide. The cathode compartment will contain sodium chloride, and some sodium hydroxide, bleach, and sodium chlorate. You can discard the cathode liquid if desired, or you can recycle it for another crop of chlorate—to do this, simply fortify it with about 50 to 60 additional grams of salt, and then place it back into the cathode compartment. Your sodium chlorate solution (anode liquid) should be quickly filtered, to remove any insoluble materials, and then gently heated to a boil for about 10 minutes to drive-off any dissolved gasses, and to break down any bleach. Thereafter, for best results, pour the entire anode liquid onto a large shallow pan, and then allow to air dry. Blowing air over the surface of the pan using a cooling fan can help speed-up the process. Once all the water has evaporated, there will be left behind crystals of sodium chlorate, sodium hydroxide, and small amounts of sodium chloride, and sodium perchlorate. Now, scrape-up all the crystals, and then place them into a beaker, and then add to this beaker, 250 milliliters (8.5 fluid oz.) of a rubbing alcohol solution prepared by adding and mixing 100 milliliters (3.4 fluid oz.) of rubbing alcohol into 150 milliliters (5 fluid oz.) of warm tap water. Then gently swirl the beaker or similar container for several minutes, and then filter-off the insoluble crystals of the sodium chlorate. Finally, recrystallize these filtered-off crystals from 150 milliliters of hot tap water (see laboratory techniques guide on how to carryout a recrystallization). The final product after recrystallization will be about 95 to 96% sodium chlorate, well suitable for use in making fireworks, or pyrotechnic compositions.

Electro preparation 4: Lead-VI-Chromate



| Chemical structure | 3D Structure |
|--------------------|-------------------------------|
| | PbCrO_4 |
| Structure make-up | Short hand chemical structure |

Lead-VI-Chromate

Lead-VI-chromate forms yellow to orange-yellow crystals, with a melting point 844 Celsius. The chromate is highly insoluble in water, and all solvents; however, it is soluble in sodium hydroxide or potassium hydroxide solutions, and in dilute nitric acid. Lead chromate is widely used in pigments, and in paint compositions.

Method 1: Preparation of Lead-VI-Chromate from Potassium dichromate

(By-products from reaction: Potassium hydroxide and hydrogen gas)

Materials:

| | |
|---|---|
| 1. 50 grams (1.8 oz.) of pickling salt | 4. 350 milliliters (11.8 fluid oz.) of cold tap water |
| 2. 350 milliliters (11.8 fluid oz.) of cold tap water | 5. 75 grams (2.6 grams) of potassium dichromate |
| 3. 15 grams (1/2 oz.) of potassium dichromate | |

Summary: Lead-VI-chromate can be made by electrolyzing a solution of potassium dichromate and sodium chloride using a lead anode. During the process, lead-VI-chromate is formed on the lead anode, from where it flakes off. Potassium hydroxide is liberated at the cathode, along with hydrogen gas.

Hazards: Perform this operation in a well-ventilated area as some chlorine gas may be evolved.

Procedure: First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell as been properly charged with Epsom salt, you can proceed with preparation 4: Lead-VI-Chromate.

Now, dissolve 50 grams (1.8 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, followed by 15 grams (1/2 oz.) of potassium dichromate. Thereafter, place this salt solution into the cathode compartment (-). Thereafter, dissolve 15 grams (1/2 oz.) of pickling salt into 350 milliliters (11.8 fluid oz.) of cold tap water, followed by 75 grams (2.6 grams) of potassium dichromate. Then place this acidified salt solution into the anode compartment (+). Now, assemble the cell as illustrated below, using two lead electrodes, and then begin the operation by turning on your power supply, or by plugging in your battery charger. Allow the cell to electrolysis for about 15 hours at 6 Volt/6 Amp or equivalent—be sure to monitor the temperature so as it does not rise above 50 Celsius.

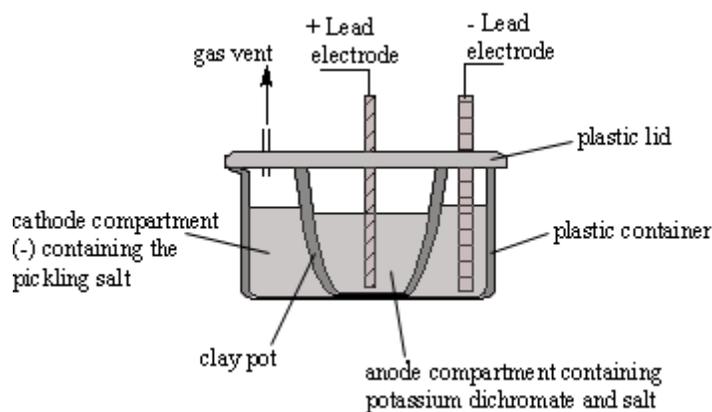


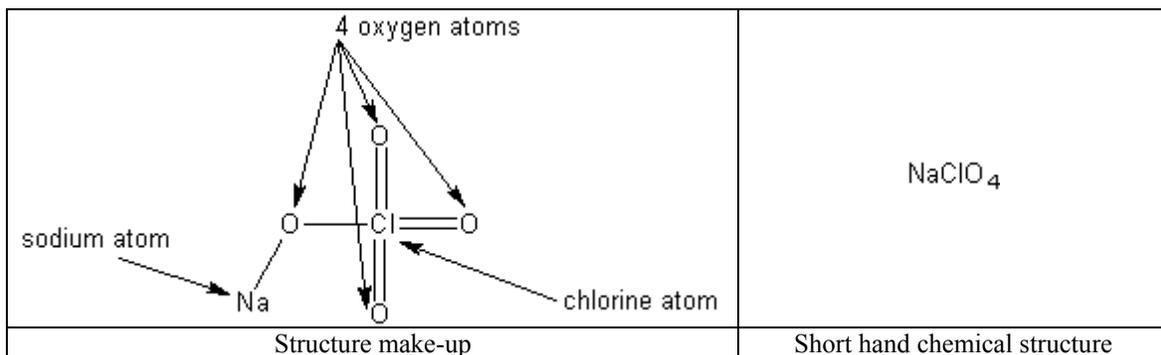
Figure 059. Set-up for the preparation of lead-vi-chromate from potassium dichromate. The positive lead electrode will slowly be corroded in this operation. The Lead-VI-Chromate will form a flaky precipitate that will collect in the anode compartment. The negative lead electrode can be replaced with titanium, chromium, or graphite.

After the 15-hour electrolysis process, unplug your power supply, and then open the cell. Carefully remove the clay pot, and then dump its contents into a clean beaker or similar glass container—the anode compartment will contain the insoluble lead-vi-chromate, and some dissolved sodium hypochlorite, and some sodium chloride and potassium hydroxide. The cathode compartment will contain potassium hydroxide, sodium chloride, sodium hydroxide, and some bleach. You can discard the cathode liquid if desired. The lead-vi-chromate can be easily collected by filtering the anode liquid. The collected lead salt should then be air-dried, and then stored in a suitable container.

Note: Many other metals can be used to replace the lead anode electrode. Such metals include, aluminum, zinc, iron, nickel, copper, and various other metals forming the corresponding metal chromates.

Electro preparation 5: Sodium perchlorate monohydrate

| | |
|---|--------------|
| | |
| Chemical structure (minus the water of hydration) | 3D Structure |
| | |



Sodium perchlorate

Sodium perchlorate forms whitish to colorless deliquescent monohydrated crystals with a decomposition temperature of 130 Celsius. The crystals are very soluble in water, but have limited solubility in the usual solvents.

Method 1: Preparation of Sodium perchlorate monohydrate from sodium chlorate

(By-products from reaction: Hydrogen and oxygen gases)

Materials:

| | |
|---|---|
| 1. 300 milliliters (10.1 fluid oz.) of ice-cold tap water | 3. 500 milliliters (17 fluid oz.) of ice-cold tap water |
| 2. 50 grams (1.8 oz.) of sodium chlorate | 4. 50 grams (1.8 oz.) of sodium chlorate |

Summary: Sodium perchlorate monohydrate can be prepared by electrolyzing a solution of sodium chlorate using a typical diaphragm salt bridge cell utilizing titanium anodes at a temperature of about 0 Celsius. The exact dimensions of the electrodes, the specific distance between the electrodes and the current density can vary widely and plays a role in the yield of sodium perchlorate. Many cell designs and operations exist, but many are sketchy and leave various details subdued; however, the overall operation consists of electrolyzing a solution of sodium chlorate using inert electrodes in a separated cell (diaphragm cell) at ice-cold temperatures.

Hazards: Perform this operation in a well-ventilated area as some chlorine gas may be evolved, and avoid contact with flames as hydrogen gas is evolved.

Procedure: First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell as been properly charged with Epsom salt, you can proceed with preparation 5: Sodium perchlorate.

Into a freshly charged cell, as previously prepared, place 300 milliliters (10.1 fluid oz.) of ice-cold tap water into the anode compartment (clay pot), and then add and dissolve 50 grams (1.8 oz.) of sodium chlorate into this water. Thereafter, place 500 milliliters (17 fluid oz.) of ice-cold tap water into the cathode compartment, and then add and dissolve 50 grams (1.8 oz.) of sodium chlorate there into. Thereafter assemble the cell as illustrated below, and then place the cell into an ice bath, and then begin the electrolysis process. The desired dimensions of the titanium electrodes may vary, but it is recommended to use rectangular bars of 10 to 15 millimeters in width of surface area (0.39 inches by 0.59 inches), and the electrodes should be placed about 127 millimeters apart (5 inches). The current should be 1.5 to 2.7 volts DC current at about 50 amps. Over voltage should be minimum, and the cell temperature should be kept below 5 Celsius at all times. Electrolyze the solution for about 18 to 24 hours.

After the electrolysis process, open the cell and then pour the anode liquid into a clean beaker or similar container, and then recrystallize the sodium perchlorate monohydrate from the solution using the normal techniques. The sodium perchlorate can be recrystallized from 150 milliliters of boiling water in the usual manner if desired.

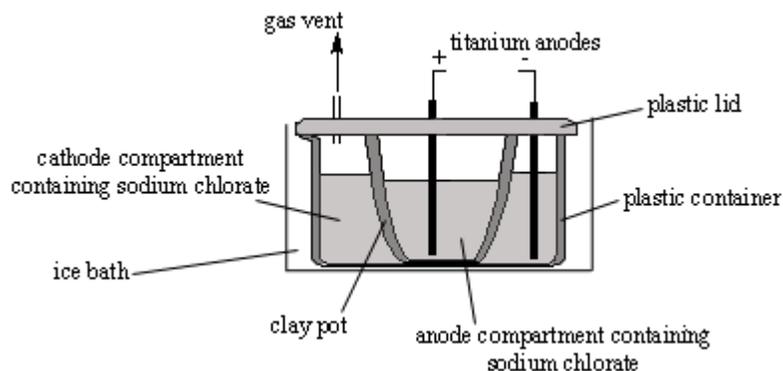


Figure 060. Set-up for the preparation of sodium perchlorate.

Electro preparation 6: Iron oxide(s)

| | |
|---|---|
| | |
| <p>Chemical structure Top: Iron-II-oxide Bottom: Iron-III-oxide</p> | <p>3D Structure Top: Iron-II-oxide Bottom: Iron-III-oxide</p> |
| | <p>FeO Fe₂O₃</p> |

Iron-II-oxide

Ferrous oxide forms a black powder with a melting point of 1360 Celsius. The powder readily oxidizes when heated in air forming ferric oxide. The powder can absorb carbon dioxide from the air, and is a strong base. It is insoluble in water and the usual solvents.

Iron-III-oxide (ferric oxide)

Iron-III-oxide forms a red to reddish to blood red powder with varying chemical composition. It is insoluble in water, and the usual solvents, and iron-III-oxide that has been roasted for several hours is almost insoluble in acids. Iron-III-oxide is a major ingredient in thermite and other incendiary agents.

Method 1: Preparation of Iron-II-oxide and Iron-III-oxide from iron nails or rods using an open cell and pickling salt

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 100 grams (3.5 oz.) of pickling salt | 3. Two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length |
| 2. 400 milliliters (13.5 fluid oz.) of water | 4. 750 milliliters of warm water (25.3 fluid oz.) |

Summary: Iron-II-oxide can be readily prepared by first, electrolyzing a solution of pickling salt using iron electrodes. During the electrolysis process, a messy precipitate of mixed hydrated iron oxides is formed. Thereafter, this precipitate is collected by filtration, and then dried. The dried messy mass is then dried in a desiccator under mild heat for 12 to 24 hours to facilitate formation of the iron-II-oxide. For the preparation of iron-III-oxide, the same electrolysis process is used to form the initial messy mass, and this mass is then collected by filtration and dried in the usual manner. However, instead of drying this mass in a desiccator, it is roasted at high temperature for several hours to facilitate formation of iron-III-oxide, which is formed by the oxidization of the iron-II-oxides.

Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated iron oxide slag using an open electrochemical cell.

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two iron nails, or rods ranging 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding nails, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated iron oxides will precipitate. The color will vary from green to orange-like. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The iron anode will slowly corrode and disappear. The electrolysis process can be slow, and can take any where from 12 to 16 hours to complete, depending on the thickness of the iron nails or rods. Thick nails or rods will take much longer to consume as the electro-chemical reaction proceeds. Thin iron nails or rods should be avoided as they will corrode much faster. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass. Note: the iron nails or rods should be as pure as possible, and steel, galvanized, and plated iron nails or rods should be avoided to avoid the formation of other metal impurities.

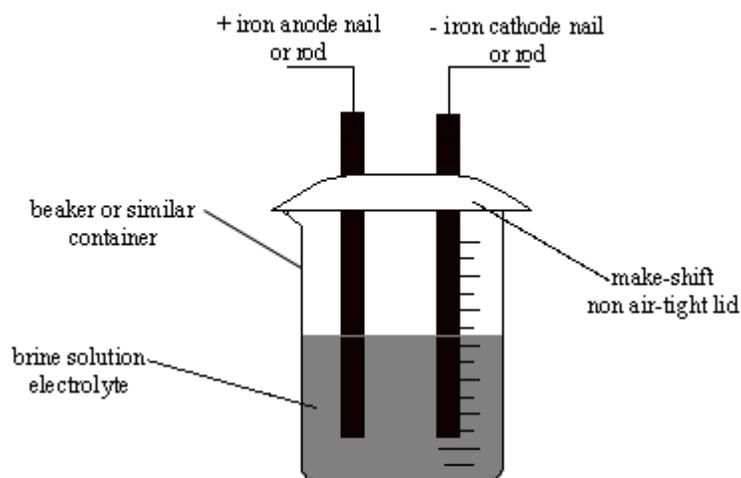


Figure 061. Set-up for the preparation of mixed hydrated iron oxides.

Step 2A: Preparation of iron-II-oxide

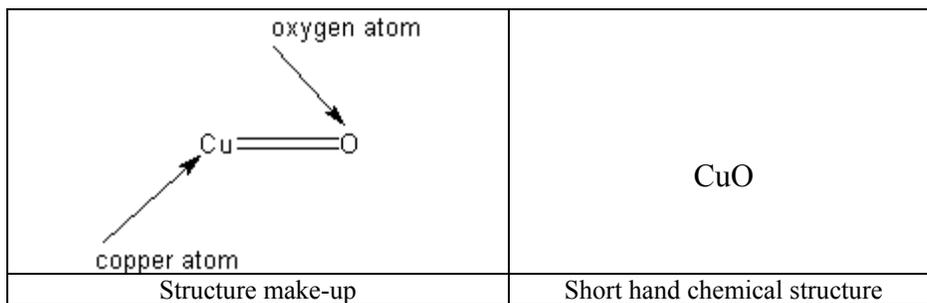
To prepare basic iron-II-oxide, the dried mixed hydrated iron oxide mass (prepared in step 1), needs to be dehydrated under suitable conditions. To do this, place the dried mass into a desiccator, filled with concentrated sulfuric acid, and then gently heat the dried mass to about 50 to 60 Celsius for 12 to 24 hours. After the heating process, the final product should be a jet-black fine mass, which can be ground into a powder if desired. The final dried product should be kept in airtight containers.

Step 2B: Preparation of iron-III-oxide

To prepare iron-III-oxide, all you need to do is place the dried mass of hydrated iron oxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatilized and removed, and the iron oxides are oxidized to iron-III-oxide forming a red powder. After the roasting process, the iron-III-oxide is cooled, and then stored in any suitable container. This iron-III-oxide can be powdered and used in thermite compositions, or pyrotechnics, or used as a catalyst for the oxidation of various gases.

Electro preparation 7: Copper-II-oxide

| | |
|----------------------|--|
| $\text{Cu}=\text{O}$ |  |
| Chemical structure | 3D Structure |
| | |



Copper-II-oxide

Copper-II-oxide forms a black to dark brownish-black amorphous or crystalline powder. The powder is insoluble in water and most organic solvents, but soluble in ammonia solution, acids and alkalis. The powder, after being roasted at high temperature for sometime, may become resistant to acids. Copper-II-oxide is widely used in the manufacture of fireworks and other pyrotechnic compositions for imparting a blue flame.

Method 1: Preparation of copper-II-oxide from copper pipes utilizing an open cell and pickling salt

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 100 grams (3.5 oz.) of pickling salt | 3. Two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length |
| 2. 400 milliliters (13.5 fluid oz.) of water | 4. 750 milliliters of water (25.3 fluid oz.) |

Summary: Copper-II-oxide is formed in a similar manner as for iron-II-oxide. It is prepared, first, by electrolyzing a solution of pickling salt using copper electrodes. During the electrolysis process, a messy precipitate of mixed hydrated copper hydroxides are formed. Thereafter, this precipitate is collected by filtration, and then dried. The dry mass is then roasted at high temperature for several hours to facilitate formation of copper-II-oxide, which is formed by the dehydration and oxidization of the hydrated copper hydroxides.

Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated copper hydroxides using an open electrochemical cell.

Set-up the apparatus illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a multi colored precipitate of mixed hydrated copper hydroxides will precipitate. The color will vary from yellowish to yellowish-orange. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will slowly corrode and disappear. The electrolysis process can be slow, and can take any where from 12 to 16 hours to complete, depending on the thickness of the copper pipes or rods, and depending on how much of the copper oxide you want. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated messy mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the filtered-off mass.

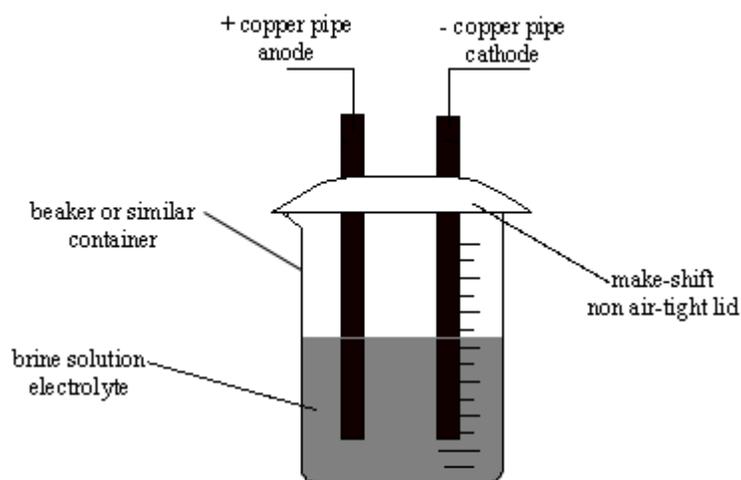
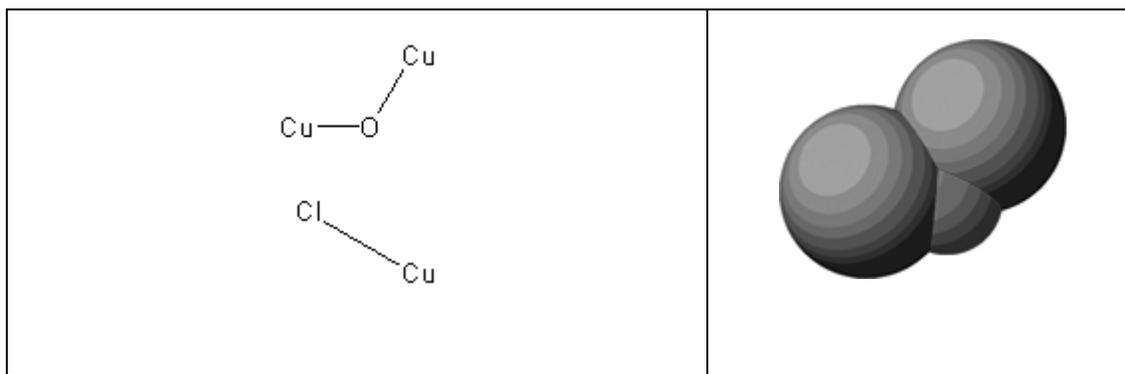


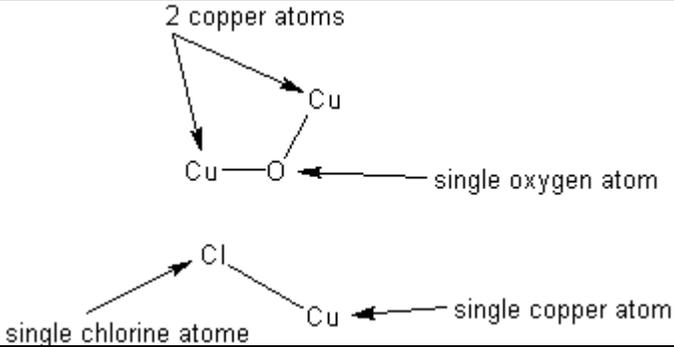
Figure 062. Set-up for the preparation of mixed hydrated copper hydroxides (practically identical to the iron oxide prepositions).

Step 2: Preparation of copper-II-oxide

To prepare copper-II-oxide, all you need to do is place the dried mass of hydrated copper hydroxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatized and removed, and the copper hydroxides are oxidized to copper-II-oxide forming a black powder. After the roasting process, the copper-II-oxide is cooled, and then stored in any suitable container. This copper-II-oxide can be used in pyrotechnic compositions, or used as a catalyst for the oxidation of various gases, such as the oxidation of methanol to formaldehyde.

Electro preparation 8: Copper-I-oxide and Copper-I-chloride



| | |
|---|---|
| |  |
| Chemical structure Top: Copper-I-oxide Bottom: Copper-I-chloride | 3D Structure Top: Copper-I-oxide Bottom: Copper-I-chloride |
|  <p>2 copper atoms</p> <p>Cu</p> <p>Cu—O ← single oxygen atom</p> <p>Cl</p> <p>← single chlorine atom</p> <p>Cu ← single copper atom</p> | Cu_2O CuCl |
| Structure make-up | Short hand chemical structure |

Copper-I-oxide

Copper-I-oxide forms a brick red crystalline powder, which may be yellow, red, or brown depending on method of preparation. The powder is insoluble in water, and the usual solvents, but is readily soluble in acids, and ammonia solutions. The melting point of the powder is about 1232 Celsius, and the dry compound slowly oxidizes to copper-II-oxide on standing. Copper-I-oxide should be used within 1 month of its preparation.

Copper-I-chloride

Copper-I-chloride forms a white crystalline powder, which is stable in dry air, but tends to turn yellow, green, blue, or brown on exposure to moisture, air, and light. Store dry in airtight amber glass bottles. The dry crystals have a melting point of 430 Celsius. The crystals are relatively insoluble in water, with partial decomposition, but are soluble in concentrated hydrochloric acid, and ammonia solutions. Copper-I-chloride can be used in pyrotechnic compositions, for imparting cool effects to the flames. Copper-I-chloride is a very useful catalyst for use in chemistry.

Method 1: Preparation of copper-I-oxide and copper-I-chloride from copper pipes utilizing an open cell and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 15 milliliters (½ fluid oz.) of concentrated hydrochloric acid or Muriatic acid | 3. Two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length |
| 2. 500 milliliters (16.9 fluid oz.) of cold water | 4. 150 milliliters of alcohol (5 fluid oz.) |

Summary: This procedure is rather interesting in the regards that replacing the pickling salt with dilute hydrochloric acid causes something entirely different to happen. In this reaction, the hydrochloric acid reacts with the copper forming copper-II-chloride, which then gets reduced at the cathode forming a brick-red layer of copper-I-oxide, and a small amount of granulated metallic copper. Over at the copper anode, some copper-I-chloride forms, which then falls to the bottom of the cell. This white flaky precipitate of copper-I-chloride can be collected by submerging a small beaker or similar container into the cell underneath the copper anode. The cell has to be stopped multiple times every 5 to 10 minutes in order to scrape-off the copper-I-oxide coating that forms on the copper cathode. If this layer of copper-I-oxide is not scraped-off periodically, the electrochemical reaction will cease, as the circuit is disrupted. The collected precipitate of copper-I-oxide is then washed, and then dried. The copper-I-chloride can be recovered and dried as well.

Hazards: Use caution when handling hydrochloric acid.

Procedure: As in the previous two procedures, set-up the apparatus illustrated below, and then add and dissolve 15 milliliters ($\frac{1}{2}$ fluid oz.) of concentrated hydrochloric acid or Muriatic acid into 500 milliliters (16.9 fluid oz.) of cold water, and then put in place two copper pipes, or rods ranging 5 to 18 millimeters in diameter (0.19 inch to 0.70 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding pipes, and then begin the electrolysis. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. Note: A large volume of hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. The copper pipe anode will corrode and disappear at a steady rate. The electrolysis process will be quite fast, much faster then the previous two procedures, and the power supply will have to be turned-off once every 5 to 10 minutes, and the positive copper anode will have to be quickly and temporally removed, and then scraped-clean using a spatula or any suitable utensil to recover a layer of copper-I-oxide that forms. After each scraping, simply scrape or place the brick red copper-I-oxide precipitate into any beaker or similar container, and store submerged under water temporarily, until all the desired amount of copper-I-oxide is collected. Note: Over time, the electrolyte will be consumed, as the hydrochloric acid is precipitated in the form of copper-I-chloride. To collect the copper-I-chloride precipitate, simply place a small beaker or similar container directly under the copper anode, submerged, so the white flaky precipitate of copper-I-chloride can be collected as it forms. As the electrochemical reaction proceeds, the acid in the electrolyte is consumed by the formation of the copper-I-chloride. Even though the acid gets consumed, the electrochemical reaction can continue as long as necessary by simply adding in a little more concentrated hydrochloric acid or Muriatic acid. The amount of acid added, should not exceed 25 milliliters so as to prevent your power supply form being damaged due to the high conductivity of the cell. However, to maximize power input, you can hook several cells in series to maximize electrical current consumption.

After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated brick-red copper-I-oxide (contained submerged under water in a beaker), and then wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, and then vacuum dry or air-dry the washed solids. Note: do not use heat to dry the copper-I-oxide as it is easily oxidized to copper-II-oxide. Now, for the copper-I-chloride, carefully extract the small submerged beaker (placed under the copper anode to collect the flaky precipitate of copper-I-chloride), using a suitable utensil or equivalent, and then carefully filter-off the white mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off copper-I-chloride with three 50-milliliter portions (three 1.6 fluid oz. portions) of cold alcohol, and then vacuum dry or air-dry the washed copper-I-chloride. Note: copper-I-chloride is rather unstable and tends to change colors when exposed to air and moisture.

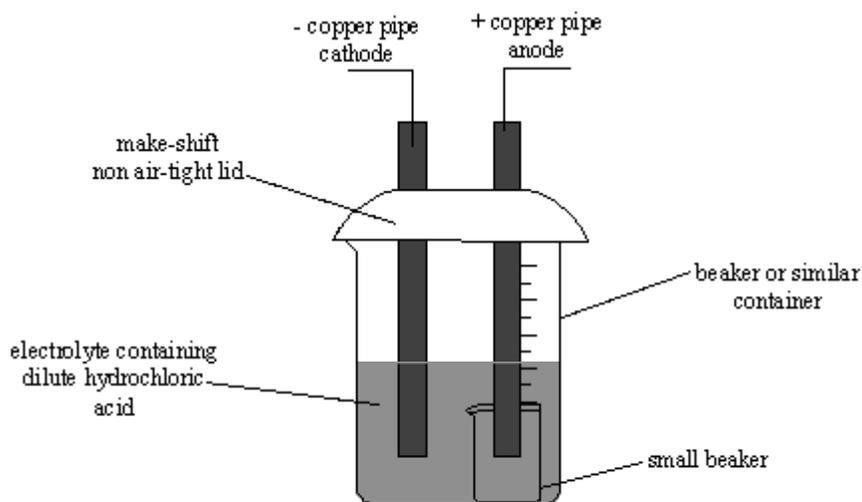


Figure 063. Apparatus for the preparation of copper-I-oxide and copper-I-chloride. The copper-I-oxide forms on the cathode, and then copper-I-chloride collects in the small beaker as a white flaky precipitate. The lid should be non airtight, and is sole used to secure the copper pipes and keep them aligned in a good vertical position without falling over.

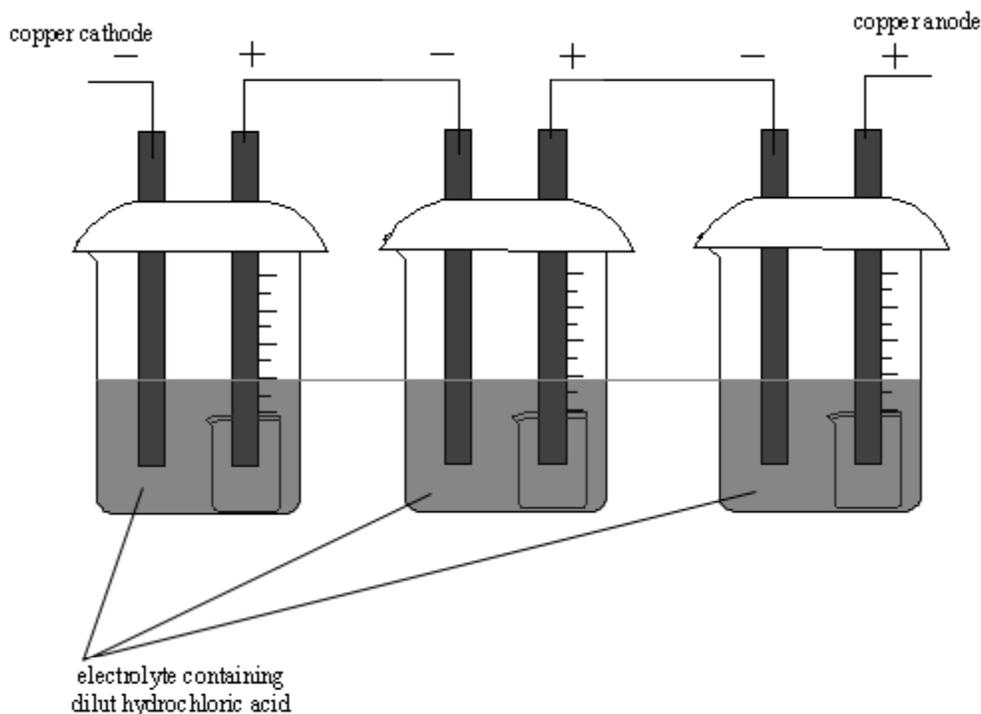
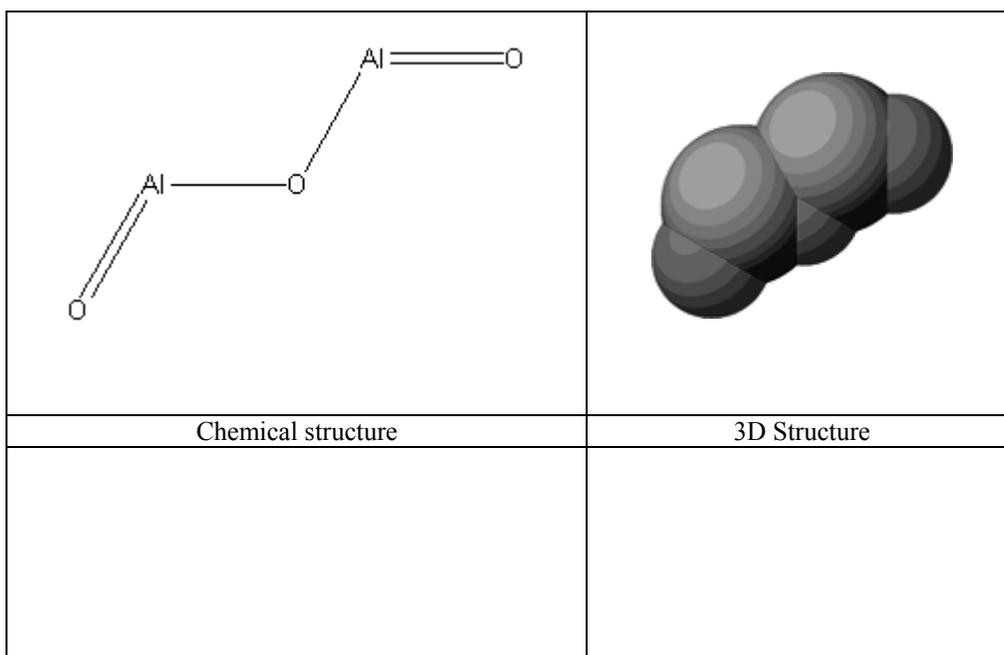
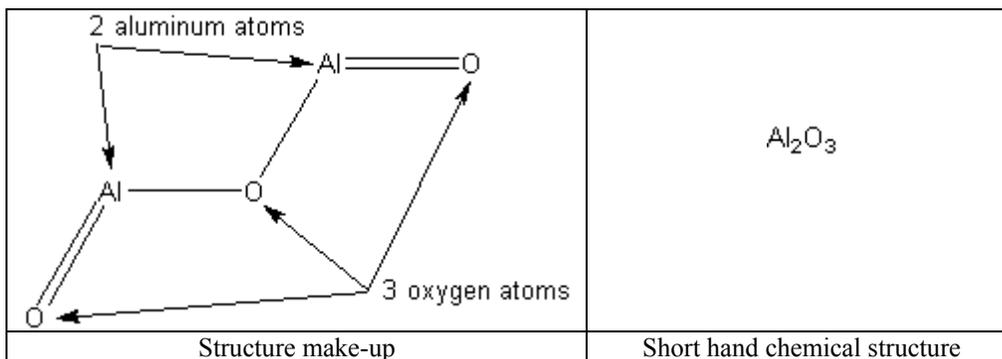


Figure 064. Apparatus for running more than 1 cell on the same power supply to maximum current efficiency. This “in series” type operation can be used for any electrolysis procedure, even if using a diaphragm cell, and is not restricted sole to copper-I-oxide and copper-I-chloride as in this procedure.

Electro preparation 9: Aluminum oxide





Aluminum oxide

Aluminum oxide forms a white powder, granules, or crystalline material. The compound is highly resistant to heat and chemical attack. Aluminum oxide becomes resistant to acids when roasted at 800 Celsius for an hour or so. It is widely used in organic chemistry as a catalyst, filtering aids, chromatography, and purification of organic liquids and solvents.

Method 1: Preparation of aluminum oxide from aluminum, an open cell, and pickling salt

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 100 grams (3.5 oz.) of pickling salt | 3. Two aluminum rods, or nails, ranging from 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length |
| 2. 400 milliliters (13.5 fluid oz.) of water | 4. 750 milliliters of water (25.3 fluid oz.) |

Summary: Aluminum oxide can be readily prepared in a similar manner as for iron-III-oxide utilizing an open cell in an identical manner. To prepare aluminum oxide, a solution of pickling salt is electrolyzed using aluminum electrodes. During the electrolysis process, a fine white precipitate of mixed hydrated aluminum hydroxides is formed. Thereafter, this precipitate is collected by filtration, and then dried in the usual manner. The dried mass is then roasted at high temperature for several hours to facilitate formation of aluminum oxide, which is formed by the dehydration of the mixed hydrated aluminum hydroxides.

Hazards: use caution when using electricity, and do not touch both electrodes at the same time while the electricity is on.

Procedure:

Step 1: Formation of mixed hydrated aluminum hydroxides.

Set-up the apparatus as illustrated below, and then add and dissolve 100 grams (3.5 oz.) of pickling salt into 400 milliliters (13.5 fluid oz.) of water, and then put in place two aluminum rods, or nails, ranging from 5 to 12 millimeters in diameter (0.19 inch to 0.47 inch) by 6 to 9 inches in length, and then place the positive and negative clamps of your power supply onto the corresponding rods or nails, and then begin the electrolysis. Note: do not use aluminum cans or foil, as impurities will contaminate the end product. Use a current of 6 to 12 volt at 2 to 6 amp. Note: a simple house hold car battery charger is well suitable as a power supply. During the electrolysis, a fine white flaky precipitate of mixed hydrated aluminum hydroxides will precipitate. Hydrogen gas will be steadily evolved from the cathode so use proper ventilation and do not allow the gas to accumulate. As in the usual manner, the aluminum anode will slowly corrode and disappear. The electrolysis process can be slow, and can take anywhere from 12 to 16 hours to complete, depending on the current density and amount of time. Very thin aluminum nails or rods should be avoided as they will corrode much faster. After the electrochemical reaction, or after the desired amount of time has passed, remove the power source, and then filter-off the precipitated flaky mass, using preferably vacuum filtration. After the filtration process, wash the filtered-off mass with three 250-milliliter portions (three 8.4 fluid oz. portions) of warm water, in the usual manner, and then vacuum dry or air-dry the filtered-off mass.

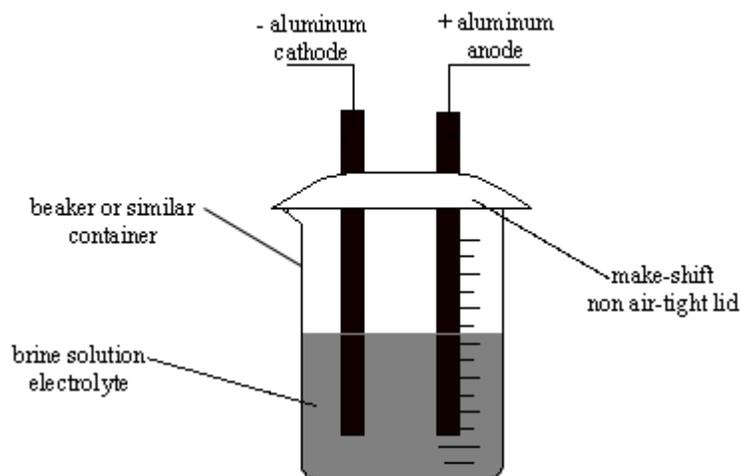


Figure 065. Set-up for the preparation of mixed hydrated aluminum hydroxides. Note: the cathode can be composed of aluminum or graphite.

Step 2: Preparation of aluminum oxide

As in the previous procedures, to prepare aluminum oxide, all you need to do is place the dried mass of hydrated aluminum hydroxides (prepared in step 1) into a crucible and then heat at 600 to 800 Celsius using a typical Bunsen burner for about 3 to 4 hours. During the heating process, water is volatilized and removed, and the hydrate aluminum hydroxides are dehydrated forming a white powder. After the roasting process, the aluminum oxide is cooled, and then stored in any suitable container. This aluminum oxide is well suitable for use as a filtering aid, and for use in silica gel columns for filtration purification.

Electro preparation 10: Preparation of highly purified metals and methods for electroplating

In this cool procedure, you can learn how to make highly purified metals, and the foundations of electroplating.

Method 1: Preparation of flaked highly purified iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|---|---|
| 1. 450 milliliters (15.2 fluid oz.) of cold water | 3. 1 iron, zinc, nickel, gold, chromium, silver, cadmium, |
|---|---|

| | |
|--|--|
| | titanium, cobalt, or zirconium nail, rod, wire or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length |
| 2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid | 4. 1 graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, or graphite rods of 1/4 th inch diameter by 6 to 12 inches in length |

Summary: Highly purified flaked metals can be prepared by electrolyzing a dilute solution of hydrochloric acid utilizing a corresponding metal anode and a graphite cathode. The electrolysis is generally simple, and produces beautiful flakes of the desired metal.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Forming highly purified flaked metals

Set-up the apparatus as illustrated below, and then fill the cell with 450 milliliters (15.2 fluid oz.) of cold water, and then add in 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid. Thereafter, turn on the magnetic stirrer, and then stir the mixture and then attach the power supply clamps to the corresponding electrodes, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current. During the electrolysis, the hydrochloric acid will be consumed forming the corresponding water-soluble metal chloride. Within about 15 to 30 minutes of starting the electrolysis, highly purified corresponding metal begins to form around the cathode. At the same time, the corresponding metal anode will slowly corrode and disappear. Even though the hydrochloric acid gets consumed, the electrolyte does not need to be replaced over time, and the electrochemical reaction can go on as long as there is a corresponding metal anode. In this procedure, the cathode should be composed of a graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, and graphite rods of 1/4th inch diameter by 6 to 12 inches in length are preferred. Continue the electrolysis as long as necessary, and afterwards, the highly purified corresponding metal can be filtered-off, washed, and then dried in the usual manner. The metal anode can be composed of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium. Note: The metal anodes should be rods, nails or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length.

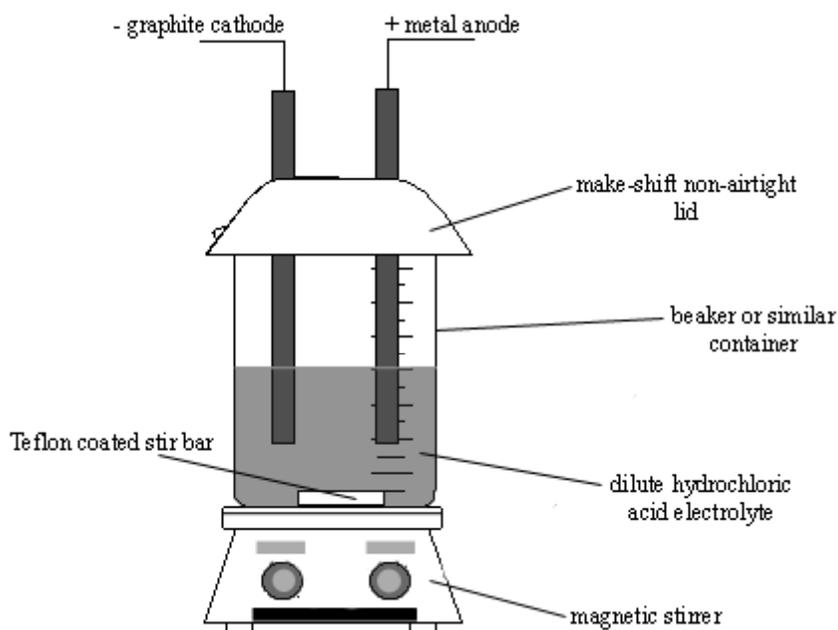


Figure 066. Set-up for the preparation of highly pure flaked metals.

Method 2: Preparation of highly purified metal crystals of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 450 milliliters (15.2 fluid oz.) of cold water | 3. 1 iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium rod, nail or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length |
| 2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid | 4. 1 graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, or graphite rod of 1/4 th inch diameter by 6 to 12 inches in length |

Summary: Highly purified metal growth crystals can be prepared by electrolyzing a dilute solution of hydrochloric acid in an identical manner as in method 1, utilizing a corresponding metal anode and a graphite cathode. The electrolysis is generally simple, and produces beautiful growth crystals of the corresponding metal forming various elongated shapes.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Growing crystals of highly purified metals

Set-up the apparatus as illustrated below, and then fill the cell with 450 milliliters (15.2 fluid oz.) of cold water, and then add in 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid. Thereafter attach the clamps of the power supply to the corresponding electrodes, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current. During the electrolysis, the hydrochloric acid will be consumed forming the corresponding water-soluble metal chloride. Within about 15 to 30 minutes of starting the electrolysis, highly purified corresponding metal will begin to plate, and then form on the cathode. Over time, the metal will “grow” on the cathode forming beautiful elongated crystals reaching towards the anode. At the same time, the corresponding metal anode will slowly corrode and disappear. Even though the hydrochloric acid gets consumed, the electrolyte does not need to be replaced over time, but the electrochemical reaction will have to be stopped periodically to remove metal growth as it will grow to touch the anode, thereby closing the circuit. In this procedure, as in the above procedure, the cathode should be composed of a graphite rod of 5 to 10 millimeters (0.19 inch to 0.39 inch) in diameter, and graphite rods of 1/4th inch diameter by 6 to 12 inches in length are preferred. Continue the electrolysis as long as necessary. The metal anode can be composed of iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium. Note: The metal anodes should be rods, nails or similar objects ranging from 5 to 15 millimeters (0.19 inch to 0.59 inches) in diameter by 6 to 9 inches in length.

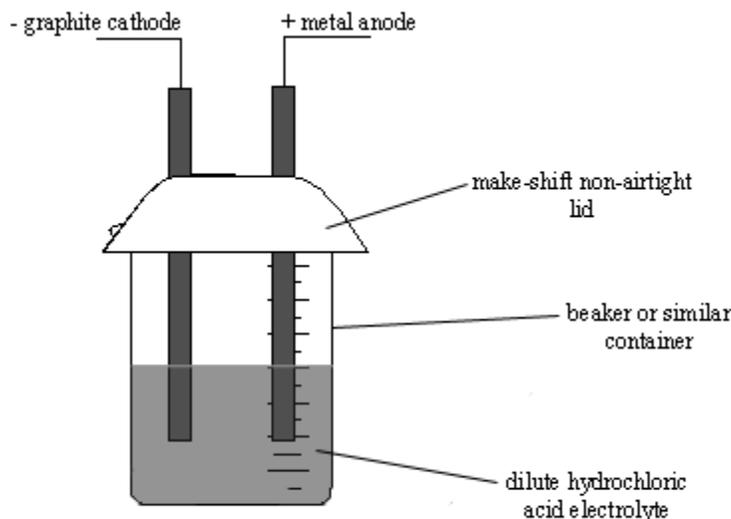


Figure 067. Set-up for growing highly pure crystals of metals.

Method 3: Electroplating techniques using iron, zinc, nickel, gold, chromium, silver, cadmium, titanium, cobalt, or zirconium using an open cell, and dilute hydrochloric acid or water soluble corresponding salts

(By-products from reaction: Hydrogen gas)

Materials:

| | |
|--|--|
| 1. 1000 milliliters (33.9 fluid oz.) of cold water | 4. 50 to 60 grams (1.7 to 2.1 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride |
| 2. 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid | 5. 1000 milliliters (33.9 fluid oz.) of water |
| 3. 10 grams (0.35 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride | 6. 1 gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt rod, wire, or nails or similar objects ranging from 1 to 10 millimeters (0.03 inch to 0.39 inches) in diameter by 6 to 9 inches in length |

Summary: Electroplating is carried out by utilizing identical electrolysis techniques as seen in methods 1 and 2, but instead of forming metal flakes or crystals, the cathode is composed of a desired metal object to be plated. To plate metal objects, the metal object is placed as the cathode, and a solution of either dilute hydrochloric acid and corresponding metal anode, or a solution of a water soluble metal salt utilizing a metal anode or inert anode, is electrolyzed using varying current to plate the desired metal object. The electrolysis process is rapid and can be carried out with success in minimal amounts of time.

Hazards: Use caution when handling hydrochloric acid, and avoid inhalation of the fumes. Extinguish all flames before carrying out the procedure as hydrogen gas is evolved, and use proper ventilation.

Procedure:

Electroplating

Set-up the apparatus as illustrated below, and then fill the cell with 1000 milliliters (33.9 fluid oz.) of cold water. Now, depending on what you want to plate and with what metal you wish to plate the desired object with, add 25 milliliters (0.84 fluid oz.) of concentrated hydrochloric acid into the cell if you wish to plate gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt and you wish to use a metal anode of the metal you wish to plate. For plating iron, zinc, nickel, cobalt, or chromium without the use of hydrochloric acid, dissolve 10 grams (0.35 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride into 1000 milliliters (33.9 fluid oz.) of cold water. Note: You must use the exact metal your plating as the anode of the cell if using only 10 grams of dissolved salt. For example, if using 10 grams of nickel-II-chloride, to plate nickel onto an object, you must use a nickel anode, depending on how many objects you wish to plate (as ten grams of dissolved salt will not produce enough metal to plate very many objects; however, if you are using an inert anode (composed of some other material than the metal your plating, you should dissolve 50 to 60 grams (1.7 to 2.1 oz.) of zinc chloride, ferrous chloride hexahydrate, hydrated nickel chloride, cobalt-II-chloride, or chromium-II-chloride (just so there's enough metal to plate the desired number of objects, ect., ect.), into 1000 milliliters (33.9 fluid oz.) of cold water. Also note: if using hydrochloric acid, and if plating gold, silver, cadmium, titanium, or zirconium, it is preferred you use the corresponding metal anode, for example, if you wish to plate gold, it is preferred you use a gold anode; however, this is not necessary as any water soluble salt of gold, silver, cadmium, titanium, zirconium, ect., ect., can be used. In this regards, simply dissolve 10 to 50 grams of any water soluble salt of gold, silver, cadmium, titanium, or zirconium (depending on how many objects you want plated) into 1000 milliliters (33.9 fluid oz.) of water. Thereafter attach the clamps of the power supply to the corresponding metal anode or inert anode, and then secure the negative clamp of the power supply on the metal item you wish to plate. Now, if you're plating gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt, and your using dilute hydrochloric acid, turn on the power supply, and electrolysis the mixture at 6 to 12 volt at 2 to 6 amp direct current but only until the hydrochloric acid is consumed. This point should take anywhere from 10 to 15 minutes, and needs to be monitored carefully, because once the hydrochloric acid has been consumed, the plating of the dissolved metal will being immediately and rapidly. Thereafter, immediately reduce the current to 0.10 to 0.8 volt at 0.002 to 0.10 amps, and then continue to electrolysis the mixture on short 5 to 6 second bursts to form a uniform metal coating on the desired object. Now, if your plating gold, silver, cadmium, titanium, zirconium, iron, zinc, chromium, or cobalt, and your using the corresponding dissolved salt, briefly electrolysis the salt solution at 0.10 to 0.8 volt at 0.002 to 0.10 amp for brief 5 to 6 second bursts for a couple of minutes or so, depending on the desired thickness of the metal plate, and the number of objects to be plated. Note: The metal anodes should be rods, wire, or nails or

similar objects ranging from 1 to 10 millimeters (0.03 inch to 0.39 inches) in diameter by 6 to 9 inches in length. Metal foils should be avoided.

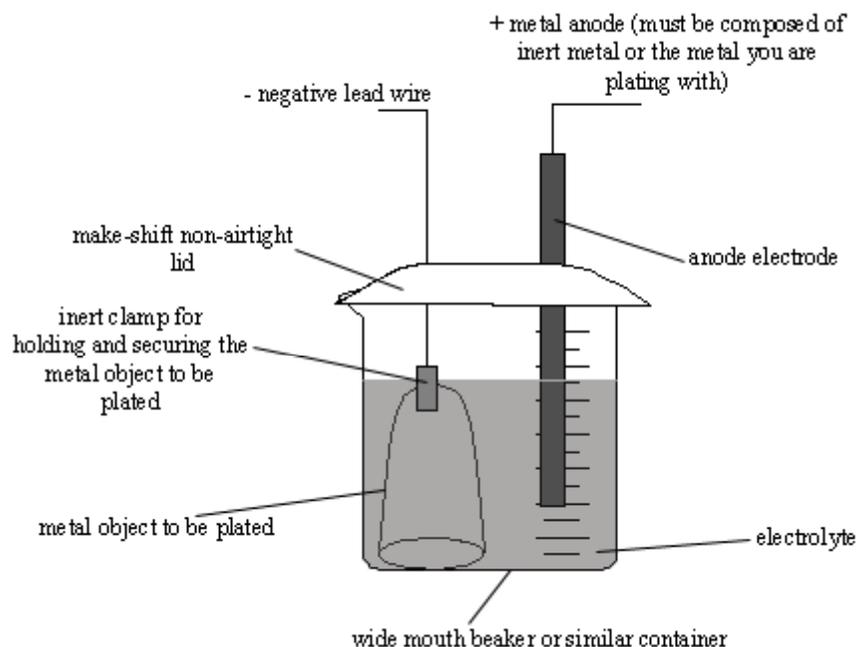


Figure 068. Set-up for electroplating. Multiple set-ups can be used, and you should use a design that best suites you.

Electro preparation 11: Preparation of Chlorine gas

Method 1: Preparation of chlorine gas from dilute hydrochloric acid utilizing an open cell

(By-products from reaction: Hydrogen gas and oxygen gas)

Materials:

| | |
|---|--|
| 1. 400 milliliters (13.5 fluid oz.) of cold water | 2. 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid |
|---|--|

Summary: Chlorine gas is readily prepared by the electrolysis of hydrochloric acid. The reaction generates heat, so the flask or container should be submerged in a cold-water bath.

Hazards: Use proper ventilation when handling chlorine gas, and avoid contact with direct sunlight.

Procedure: Into either of the apparatus listed below, place 400 milliliters (13.5 fluid oz.) of cold water, followed by 100 milliliters (3.3 fluid oz.) of concentrated hydrochloric acid or concentrated Muriatic acid. Thereafter, turn on the power supply and run the cell for about 6 to 7 hours at 2 to 12 volt at 2 to 6 amp. The amperage determines how long the process will take. Higher amperage is preferred. Note: This process will produce about 31 grams of chlorine gas. Note: the graphite electrodes can be replaced with lead if desired. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that the electrolysis of hydrochloric acid utilizing an open cell is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**

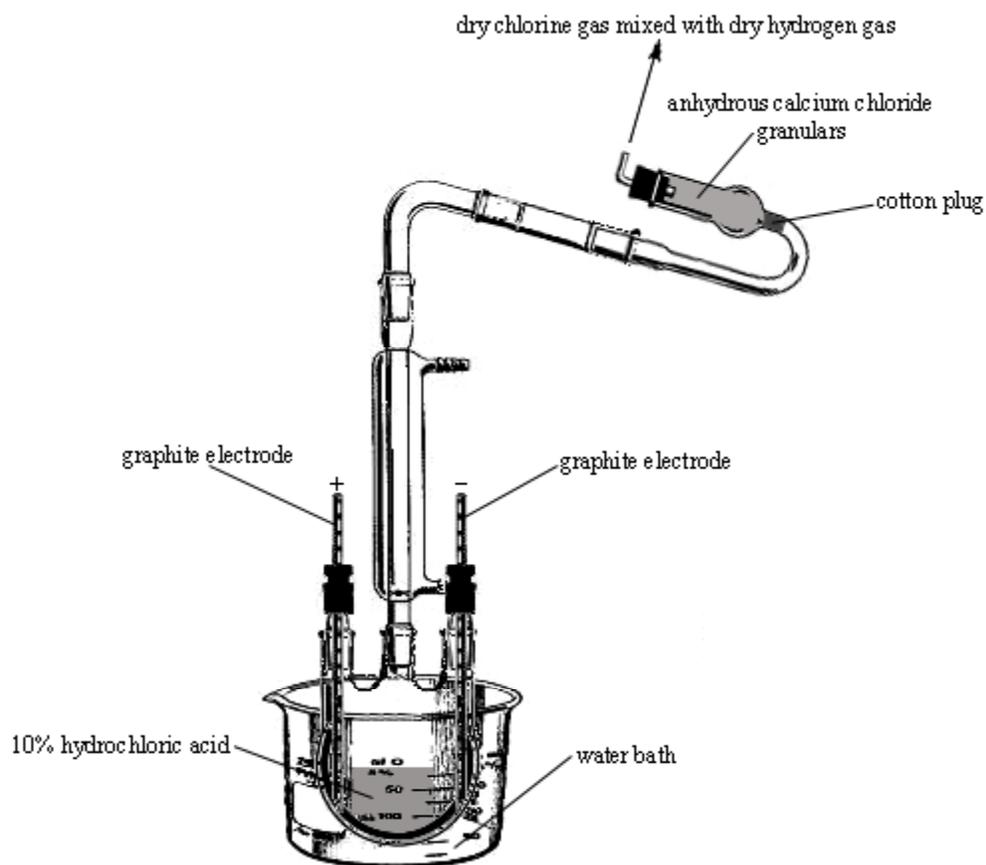


Figure 069. Professional setup for the electrolysis of hydrochloric acid.

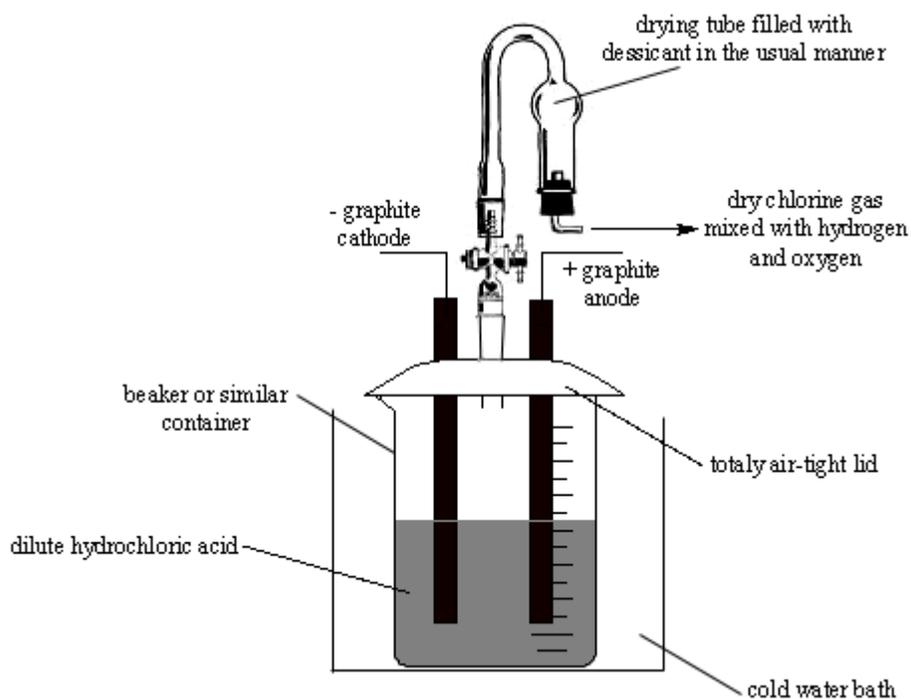


Figure 070. Apparatus for the preparation of chlorine gas. Note: the entire apparatus should be airtight, 100%

Method 2: Preparation of chlorine gas from Epsom salt, and pickling salt utilizing an open cell

(By-products from reaction: Hydrogen gas, oxygen gas, sodium sulfate, and magnesium hydroxide)

Materials:

| | |
|--|--|
| 1. 200 grams (7 oz.) of Epsom salt | 3. 95 grams (3.4 oz.) of pickling salt |
| 2. 1000 milliliters (33.8 fluid oz.) of cold water | |

Summary: Chlorine gas can also be generated by electrolysis of a mixture of Epsom salt and pickling salt. The electrodes used should be made of lead to prevent excessive corrosion. During the reaction, water insoluble magnesium hydroxide will precipitate, and a steady stream of chlorine gas will be evolved over time. This procedure is useful in generating moderate amounts of chlorine gas while using simple and readily available materials.

Hazards: Use proper ventilation when handling chlorine gas, and avoid contact with direct sunlight.

Procedure: Dissolve 200 grams (7 oz.) of Epsom salt into 1000 milliliters (3.8 fluid oz.) of cold water, and thereafter, add and dissolve 95 grams (3.4 oz.) of pickling salt. Now, pour this salt solution into an apparatus as illustrated below, and then electrolysis the mixture at 6 to 12 volt at 6 to 12 amp for a period ranging from 12 to 18 hours. During the electrolysis process, a moderate, yet steady stream of chlorine gas will be steadily evolved and carried over. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that the electrolysis of hydrochloric acid utilizing an open cell is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**

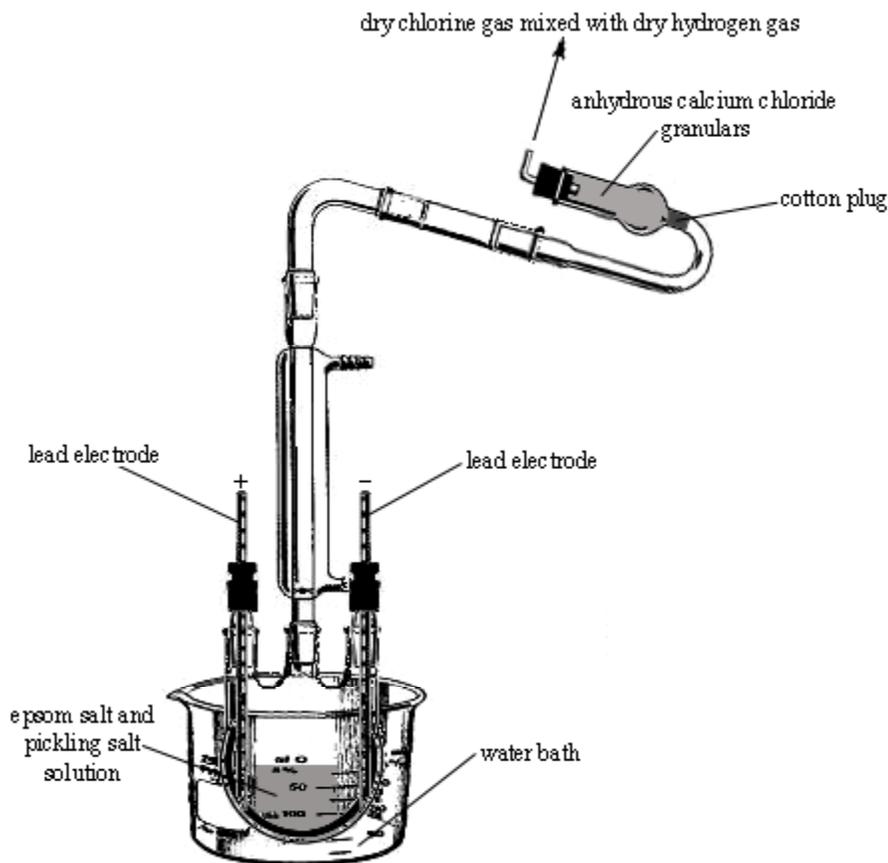


Figure 071. Professional setup for the preparation of chlorine.

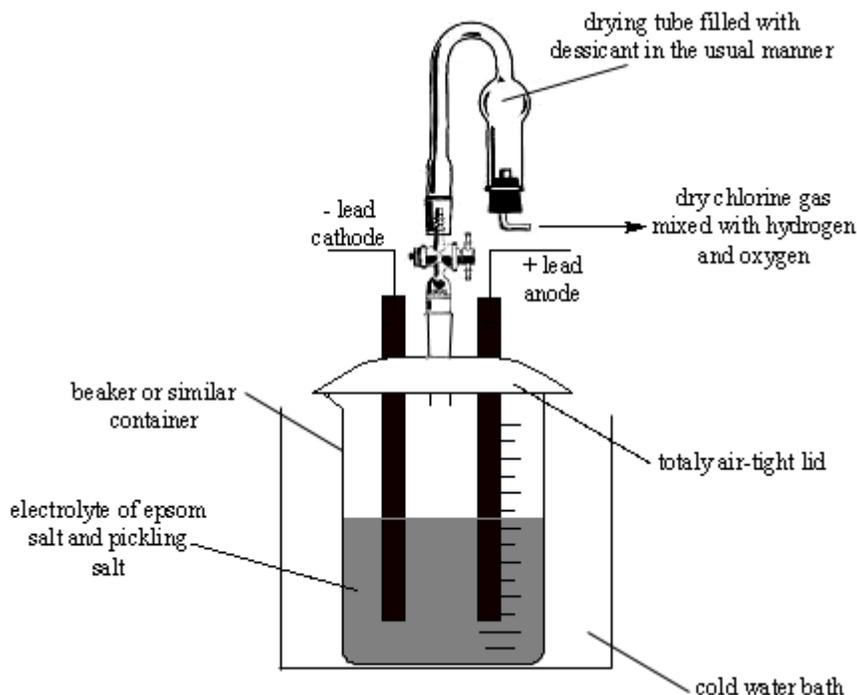


Figure 072. Setup for the preparation of chlorine.

Method 3: Preparation of hydrogen and oxygen free chlorine gas from ferrous chloride (prepared by a diaphragm cell) utilizing an open cell

(By-products from reaction: iron)

Materials:

| | |
|--|--|
| 1. 250 milliliters (8.5 fluid oz.) of warm water | 3. 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm water |
| 2. 10 grams (0.35 oz.) of pickling salt | 4. 50 grams (1.8 oz.) of pickling salt |

Summary: Hydrogen and oxygen free chlorine gas can be readily prepared by electrolyzing a solution of ferrous chloride in an open cell. The ferrous chloride can be pre-prepared by electrolyzing a solution of pickling salt using an iron anode in a diaphragm cell.

Hazards: Use proper ventilation when handling chlorine gas, and avoid inhalation of the fumes.

Procedure:

Step 1: Preparation of ferrous chloride using a diaphragm cell.

First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell has been properly charged with Epsom salt, you can proceed with preparation 11: method 3.

Thereafter, into the freshly charged cell, place 250 milliliters (8.5 fluid oz.) of warm water into the clay pot (anode compartment), and then add and dissolve, 10 grams (0.35 oz.) of pickling salt there into. Then place 500 to 750 milliliters (17 to 25.4 fluid oz.) of warm water into the cathode compartment, followed by adding and dissolving 50 grams (1.8 oz.) of pickling salt. Thereafter, using the appropriate electrodes, attach the clamps of your power supply to the proper electrode, and then electrolyze the cell at 6 to 12 volt, by 2 to 6 amp direct current for about 6 to 12 hours (depending on how much ferrous chloride you want). Thereafter, remove the power source, and then open the cell. Then pour the contents of the anode compartment into a clean beaker. The contents of the cathode compartment, which will contain sodium hydroxide, can be discarded, or the sodium hydroxide can be recovered by boiling-off the water. Now, filter the anode liquid to remove any insoluble impurities, and then place the filtered anode liquid aside temporarily.

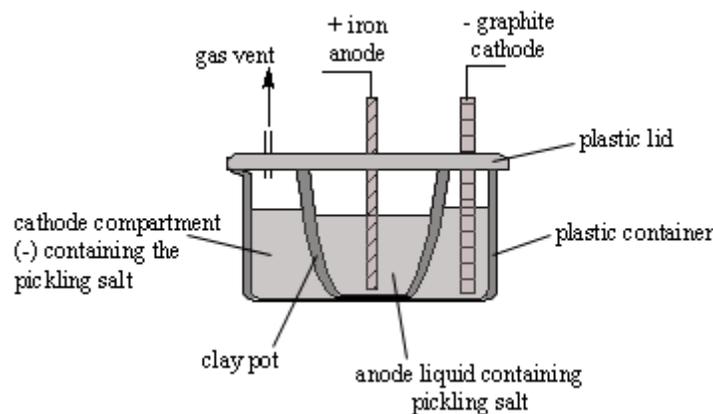


Figure 073. Setup for the preparation of ferrous chloride

Step 2: Preparation of hydrogen and oxygen free chlorine gas.

Now, into an open cell, pour the anode liquid containing the ferrous chloride (obtained in step 1), and then put the electrodes in place. Thereafter, electrolysis the liquid at 6 to 12 volt by 2 to 6 amp direct current for about 6 to 8 hours. During the electrolysis, a steady stream of hydrogen and oxygen free chlorine gas will be steadily evolved, and the metallic iron will slowly plate the cathode. Note: during the start of the reaction, the solution will turn brownish black, hence the formation of ferric chloride. After the electrolysis, the iron cathode, which will be plated with fresh metallic iron, can be recycled by using it as the anode for another electrolysis process using a diaphragm cell to prepare more ferrous chloride as in step 1. The iron anode from that process, in turn, can be reused as the cathode in the open cell as in step 2—the iron can be recycled multiple times.

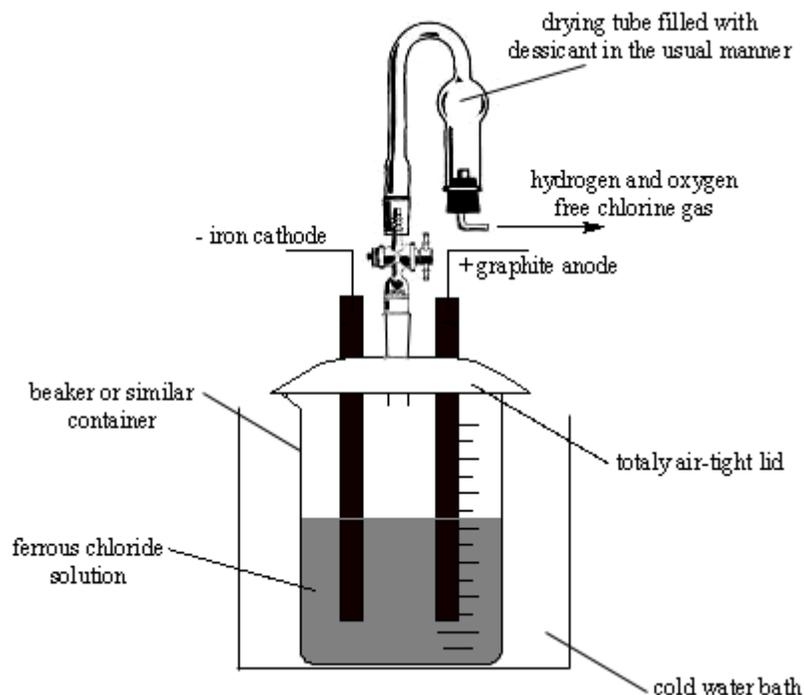


Figure 074. Setup for the preparation of hydrogen and oxygen free chlorine gas. During the electrolysis, metallic iron will slowly coat the iron cathode.

Electro preparation 12: Preparation of isopropyl hypochlorite

Method 1: Preparation of isopropyl hypochlorite from rubbing alcohol, pickling salt, and sulfuric acid using a diaphragm "salt bridge" cell

(By-products from reaction: Hydrogen gas, and oxygen gas)

Materials:

| | |
|--|--|
| 1. 100 milliliters (3.4 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol | 6. 500 to 750 milliliters (17 to 25.4 fluid oz.) of water |
| 2. 50 grams (1.8 oz.) of pickling salt | 7. 5 drops of concentrated sulfuric acid |
| 3. 10 grams (0.35 oz.) of anhydrous sodium sulfate, or magnesium sulfate | 8. Three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride |
| 4. 250 milliliters (8.5 fluid oz.) of cold water | 9. 10 grams (0.35 oz.) of anhydrous sodium or magnesium sulfate |
| 5. 50 grams (1.8 oz.) of pickling salt | |

Summary: Isopropyl hypochlorite is readily prepared by electrolyzing a solution of isopropyl alcohol and pickling salt in the presence of sulfuric acid in a diaphragm salt bridge cell. The electrolysis takes considerable amount time, but produces no significant heat build-up. After the electrolysis process, the anode liquid is filtered, and then extracted with a suitable, low boiling solvent. Afterwards, the solvent is removed via evaporation, and then the desired isopropyl hypochlorite is recovered.

Hazards: Use caution when handling concentrated sulfuric acid. Although this procedure is safe, and produces little or no noxious fumes, take good caution when opening the cell after the reaction. Isopropyl hypochlorite is a highly irritating liquid. Use caution.

Procedure: First of all, assemble the cell as illustrated in, 1. STANDARD DIAPHRAGM "SALT BRIDGE" CELL of section 4. Thereafter, run through the steps 1 to 3 to properly assemble and charge your salt bridge clay pot cell. Once your cell as been properly charged with Epsom salt, you can proceed with preparation 12: isopropyl hypochlorite.

First, we need to "salt out" isopropyl alcohol from rubbing alcohol. To do so, place 100 milliliters (3.4 fluid oz.) of rubbing alcohol containing 70% isopropyl alcohol (avoid colored products), into a separatory funnel, and then add in 50 grams (1.8 oz.) of pickling salt. Then vigorously shake the funnel for about 5 minutes. Thereafter, allow the funnel to stand for about 5 minutes, whereby a two phase mixture will result. Thereafter, drain-off the bottom water layer, and then carefully pour-off or drain-off the upper isopropyl alcohol layer. Finally, place this alcohol layer into a clean beaker or similar container, and then add in 10 grams (0.35 oz.) of anhydrous sodium sulfate, or magnesium sulfate, and then blend the mixture for about 10 minutes. Thereafter, filter-off the sulfate compound.

Now, into the freshly charged cell, into the clay pot (anode compartment), place 250 milliliters (8.5 fluid oz.) of cold water, followed by 50 grams (1.8 oz.) of the dried "Salted out" isopropyl alcohol (obtained above), followed by 10 (0.35 oz.) grams of pickling salt. Then briefly stir the mixture (or before placing into the cell) to dissolve the salt. Then dissolve 40 grams (1.4 oz.) of pickling salt into 500 to 750 milliliters (17 to 25.4 fluid oz.) of water (depending on how big the outer Tupperware container is), and then place this salt solution into the cathode compartment. Thereafter, add 5 drops of concentrated sulfuric acid to the anode liquid. Thereafter, attach the lid, and then begin the electrolysis. Electrolysis at 6 to 12 volt by 2 to 6 amp direct current for about 12 to 18 hours. After the electrolysis process, carefully open the cell, and then pour the contents of the anode compartment into a clean beaker, and then immediately cover the beaker with plastic wrap. The contents of the cathode compartment can be discarded or the sodium hydroxide

dissolved there in can be recovered for other use. **Note: Use caution as the anode liquid will be highly irritating to the eyes, nose, and throat. Use caution, and avoid inhalation of the vapors.**

Now, quickly filter the anode liquid to remove insoluble impurities, and then extract the filtered anode liquid, with three 50-milliliter portions (three 1.7 fluid oz. portions) of methylene chloride. After the extraction, combine all methylene chloride extract portions, if not already done so, and then dry this combined methylene chloride portion by adding to it, 10 grams (0.35 oz.) of anhydrous sodium or magnesium sulfate, and then stir the mixture for several minutes. Thereafter, filter-off the sodium or magnesium sulfate, and then place the dried filtered methylene chloride mixture into a distillation apparatus, and distill-off the methylene chloride at 40 Celsius until no more methylene chloride passes over. Thereafter, remove the heat source, and allow the remaining liquid to cool to room temperature. Thereafter, store the remaining liquid of isopropyl hypochlorite in an amber glass bottle in a cool place and away from sunlight.

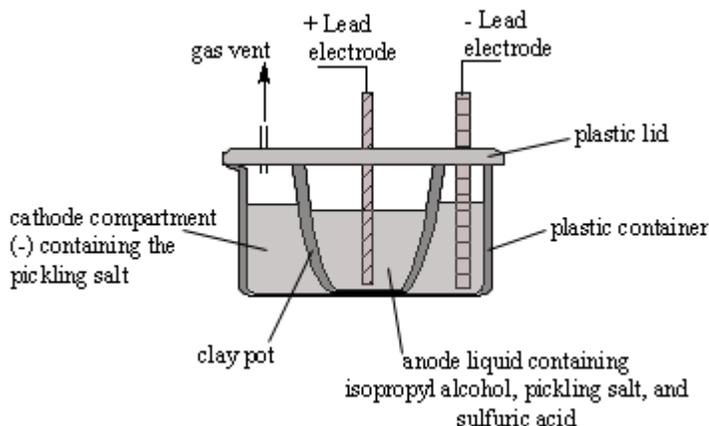


Figure 075. Common set-up for the preparation of isopropyl hypochlorite.

Electro preparation 13: Preparation of Hydrogen chloride gas via electrochemical methods

Method 1: Preparation of hydrogen chloride from Epsom salt, pickling salt, and using an open cell

(By-products from reaction: oxygen gas)

Materials:

| | |
|---|---|
| 1. 300 milliliters (10.1 fluid oz.) of dry hexane | 4. 1000 milliliters (3.8 fluid oz.) of cold water |
| 2. 20 grams (0.70 oz.) of aluminum foil pieces | 5. 95 grams (3.4 oz.) of pickling salt |
| 3. 200 grams (7 oz.) of Epsom salt | |

Summary: Hydrogen chloride can be prepared using electrochemical methods. In this regard, hydrogen chloride can be prepared by first, electrolyzing a mixture of Epsom salt and pickling salt, and then passing the gas mixture produced, into a mixture of hexane containing aluminum foil pieces. The aluminum foil acts as a catalyst, causing hydrogen gas to combine with chlorine gas, forming hydrogen chloride.

Hazards: Use caution when handling chlorine gas, which is toxic—avoid inhalation of the fumes. Extinguish all flames before using hexane, which is highly flammable.

Procedure: Setup the apparatus as illustrated below (place 300 milliliters (10.1 fluid oz.) of dry hexane and 20 grams (0.70 oz.) of aluminum foil pieces into the appropriate round bottom flask as indicated), and then dissolve 200 grams (7 oz.) of Epsom salt into 1000 milliliters (3.8 fluid oz.) of cold water, and thereafter, add and dissolve 95 grams (3.4 oz.) of pickling salt. Now, pour this salt solution into an apparatus as illustrated below, and then electrolysis the mixture at 6 to 12 volt at 6 to 12 amp for a period ranging from 12 to 18 hours. During the electrolysis process, a moderate, yet steady stream of chlorine gas (mixed with hydrogen and oxygen) will be steadily evolved and carried over. **NOTE: This operation should be carried out in a room or shed free of windows, and/or direct contact to sunlight. If direct sunlight, magnesium light, or light from a UV lamp reaches the apparatus, the flask or container will explode! Please be advised that the electrolysis of hydrochloric acid utilizing an open cell is perfectly safe, and can be carried out without worries or hazards, as long as the operation is carried out in the absence of direct sunlight.**

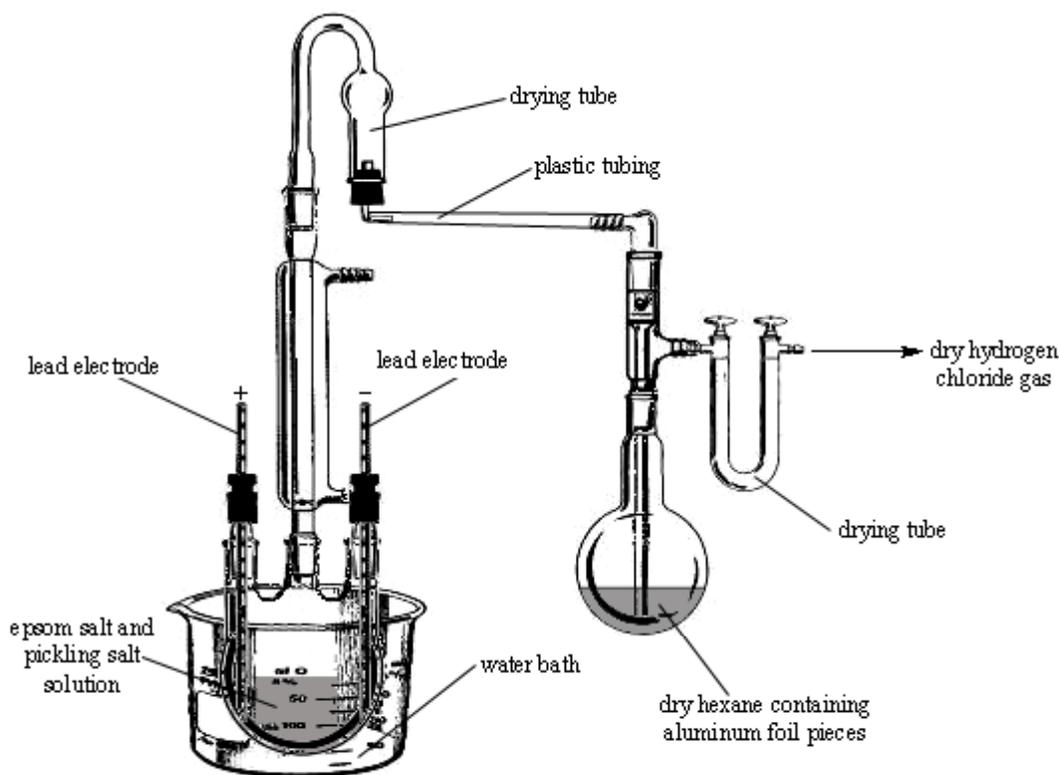


Figure 076. Advanced setup for the preparation of hydrogen chloride. As a gas mixture of hydrogen, oxygen, and chlorine is evolved from the electrolysis process, the gas mixture is bubbled into a mixture of hexane containing aluminum foil pieces. The aluminum foil acts as a catalyst, causing the hydrogen to combine with the chlorine without violence, forming a steady stream of hydrogen chloride. This hydrogen chloride can be used directly or dissolved in water forming hydrochloric acid.

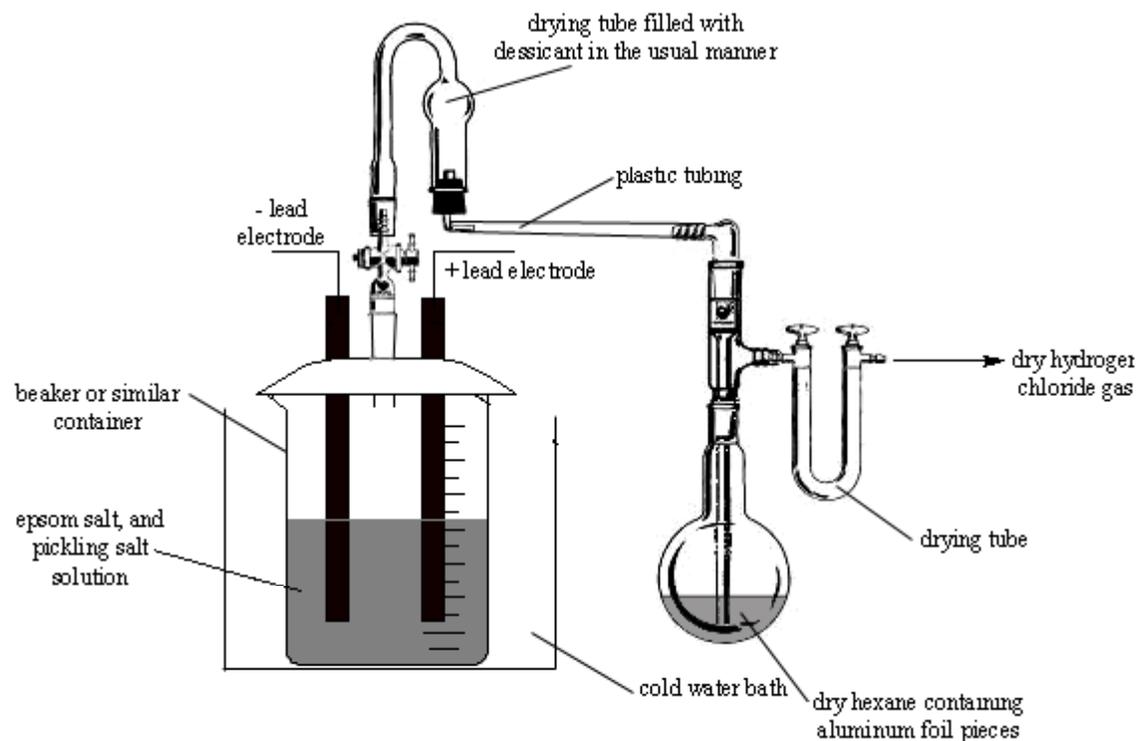


Figure 077. Another setup for the preparation of hydrogen chloride. Note: the lid on the cell should be air-tight.