
Notice!

I've found that this book project has been showing up on more and more search engines lately and is also being directly linked to for the information it contains(1). I therefore find it necessary to warn all persons viewing this document that it is a work in progress, and as such it contains errors of all kinds, be them in experimental procedures that may cause harm, or in faulty reasoning that would get you slapped by nearly any chemistry instructor. Please for now take the information here with a grain of salt.

Most Importantly!

By reading further you agree not to hold the authors of this document responsible for any injuries/fatalities that may occur from attempting to make any of the products or following any of the procedures that are outlined within. Chemistry inherently possesses a degree of danger and you must understand this, wear gloves and more if the situation calls for it, your safety is in your own hands, not mine!

Also note that this project is open for contribution by any party on the internet. Simply submit a section to Rob.Vincent@gmail.com and it will be added into the text pending editing and such within a few weeks. Any person contributing will have their name mentioned in the credits. Thank you for reading this, and enjoy!

1 Although this document may be directly linked to, it will not work in that manner as I have hotlink protection
documents, however directly linking to the html document is possible, still though I would prefer links be to the
project page.

1.0 Introduction/Statement of Purpose

There are lots of pages scattered across the internet that can serve the amateur chemist in their endeavors, however each one has its own focus. Improvising a distillation apparatus, production of a specific chemical, some go further and tell how to stock a lab at home. There are still other works that are similar in goal to this work and I don't mean to compete with these, just as there are innumerable books available to a 'professional' chemistry student covering the basics this work will just be a further reference in that series, though hopefully very comprehensive. There are other places to look for the information here and a student of chemistry should not be adverse to looking over the basics of chemistry from many different sources, after all some people learn better in one way over another.

It was originally my concept that this work should have as few web references as possible. The reasoning being that they will pass into non-existence in due time. But really at home chemistry is a constantly devolving and changing hobby, an over the counter source for one chemical may be phased out without notice, during work on this project for example sodium hydroxide which was widely available over the counter started to be phased out. Hence despite my wishing that this work be somewhat timeless, that is impossible, links will be provided and they will pass into non-existence and sources for chemicals will be given that likely won't make it to the end of the decade. Still though, it is the goal of this work to explain many of the basic concepts/materials/apparatuses used in chemistry. It is further the goal of this work to depict this in a useful way directly relating to real life and real observations. In doing so this should most closely represent anything you would run across in real life giving you the best idea as to what to expect.

In reading this text you will notice that some things are in **bold face** these are either key points or are sections to which you can refer to for more information on the relevant topic. Words in *italics* are usually shown in this manner to indicate that the definition of that word can be found in section **12** the index under the subsection “Technical Terms”. Additionally as you read through this you will see numbers in superscript with parenthesis around them such as (1) these indicate that at the end of the section you are in there is additional relevant information about the topic at hand, each piece of information matching up with the topic number, this can also include references to web works and book works.

Additionally since this is meant to be a highly readable work, some of the more in depth material including mathematical calculations and other material that might be considered supplemental is occasionally separated from the section where it would normally be contained, either following it immediately or referenced as being compiled into the index. For example, the calculation of the hydrogen ion concentration in an aqueous solution is a useful calculation to perform for some reactions, however it is on the whole dry boring material in most peoples eyes, as such sections such as this will be removed from the more literary part of the text so they do not disturb the flow of the section.

Back to the content there are two sections of experiments for the amateur and by the second half, experienced chemist to perform. Each experiment is intended to develop skills that will be necessary to complete experiments found elsewhere in the future. But not only does this text help to explain itself, but lays out strategies for projects that you devise yourself, sections on real research, and the gritty sections on out of control experiments and contingency plans. Upon reading though all the material presented here you should be able to go out and perform chemistry with a degree of confidence that the at home chemist is not normally afforded. As I said before, this will not be a timeless work, but hopefully it will be a good read even in the future when performing the experiments talked about here in a home setting would be dubbed thoroughly insane (though some might find them insane right now). Never the less I wish the readers the best of luck in all their chemical endeavors even if you never make it past being an armchair chemist(2).

Bromic

1 Example of a reference in the text, this is where relevant information to that passage would be found.

2 An armchair chemist is occasionally used as a derogatory term meaning that the person has no experience with ho work in the real world and draw all of their ‘experience’ from books where the information may conflict wit observations or be entirely wrong, in other words a procedure they come up with may only work in theory not pra may be difficult to carry out in real world conditions, many people start out as armchair chemists.

1.1 Disclaimer

Each person is responsible for his or her own fortunes. It is not reasonable to hold the person writing a hunk of text responsible for your mistake should you choose to pursue their strategies, as stated in the disclaimer at the start of this document, **by reading further you agree not to hold the authors of this document responsible for any injuries/fatalities that may occur from attempting to make any of the products that are outlined within.**

Further, although many of these procedures have been attempted solely for the completion of this project, not every procedure was done or thoroughly researched. Chemistry inherently possesses some danger to it and there is always some chance somehow that things may go wrong. Please use common sense, if you are following a procedure to the letter and something occurs involving excessive heat generation or violent gas evolution and it is not mentioned in a procedure, do not just assume that it is normal, if it scares you take steps to rectify the situation, always have a contingency plan for any procedure you are doing for the first time.

People who are pregnant should not attempt many of the experiments described herein. Additionally chemistry should not be practiced around small children who may interfere with the proceedings, consume chemicals, or become more readily injured though any mishaps. You should not perform a procedure mentioned here until you are familiar with the procedure, the chemicals involved, and the possible extraneous reactions that could take place. Please, for your own safety, consider all safety precautions, gas masks, gloves, aprons, gas scrubbing. Damage done with many chemicals can be forgiving, but over time it can be disastrous, and some never give you a second chance. You only get one life, take precautions now so you do not ruin it in the future.

1.2 Safety Precautions/Gear

Before beginning on any chemical adventure there are certain steps that should be taken. The first thing you should do before starting a new experiment is to find out the properties of any reagents that are being used, along with your intended products. This can be accomplished in many ways, looking up in a chemistry dictionary for example. However the most thorough way to determine the properties of a compound is to look up its Material Safety Data Sheet (MSDS). At the bare minimum one should tie back loose hair when in the laboratory, not eat while doing an experiments and wear closed toe footwear, no sandals. Long pants are always a good thing; as are long sleeve shirts, not loose ones though.

Regardless of the specific dangers of a chemical one should always wear gloves and goggles or a face shield. Some experiments may even require you to wear a respirator when standing up wind is not enough. There are very few chemicals that will go though a good pair of gloves(1). Some solvents will eat them and cause them to become gummy on the outside but a good pair of gloves will last you.

When it comes to the clothing you should wear during chemistry that is somewhat of a matter of debate. The safest thing you could wear would be a full environmental suit, lacking that disposable painting suits made of Tyvek® that cover the full body, even have their own hood attached are widely available. Another choice for a full body suit would be a Nomex® flight suit, widely available on eBay. This is the choice for persons working at high heat or interested in pyrotechnics due to the high heat resistance of this fabric. Common cloth jump suits like the one pictured also work to a lesser degree though posess inherent flammability. However there are other things to consider then fabric.

A chemistry outfit does not necessarily have to be something altogether different from something you would normally wear. The only thing that it has to be is something that you will not care if it gets ruined. What it should also be is something not absurdly flammable like many synthetic fabrics. It should also not be excessively loose or skin tight. If the clothing is too loose it can knock over beakers or drag in reagents, if it is too tight and you get something on it, it might immediately soak though to the skin. You should always be able to remove any effected clothing quickly. In case of spill or fire.

In the case of gloves there are several types. Those that totally cover the digits, the palm, and a majority of the back of the hand are good for most situations. Gloves that only cover the bottom part of the hand and cover the back with webbing are decent for handling solids but not liquids. Disposable latex/nitrile gloves are good for most anything with nitrile being superior to latex, however there is a sweating problem, these kind of gloves can be bought in relative bulk fairly cheaply and are great to have around, the bulk kind are disposable. Finally elbow length gloves are good in situations where you are handling large quantities of reagent or there is a reaction that is causing extraneous conditions, e.g. splashing though boiling so you can easily handle the reaction if need be without splatter hitting you to any extent.

Face protection should be present to prevent splashing into the eyes, goggles like those in the above picture are ideal, they are covered from all sides and have enough air circulation to prevent them from being uncomfortable. Face shields will work and afford some additional protection, however they do not prevent splatter from certain directions, therefore a good pair of goggles are preferred. Just having any form of eye protection does help though, even safety glasses are better then nothing. However if you wear contacts you should take them out before pursuing a chemical reaction, the vapors can cause them to fuse to your eye resulting in a painful removal surgery.

Optional to some, mandatory to others, it really depends on what kind of experiments you plan to do weather or not you need a gas mask. They are available as a full mask, which covers the eyes as well as the mouth and nose, this eliminates the need for additional protective eyewear. The picture above shows a half-mask which only covers the lower part of the face. Masks are also available that utilize one cartridge or two, two providing easer breathing and longer cartridge life. The protection afforded to you by your mask is directly related to the cartridge you use. Some masks which are for military use may be picked up surplus and offer a wide variety of chemical agents to which they can offer some degree of protection, however they vary in their protection from mask to mask and therefore can only be compared on an individual basis. For a more detailed look at gas masks look at section **4.13** on gasses.

As of far all the considerations have been for working at what is known as standard temperature, room temperature, or just normal temperature, defined as 25°C or about 75°F. Now though we delve into the special gear that is necessary for working in conditions other then regular room temperature. For working at high temperature a welding shop is a goodies store, offering welding aprons and gloves that can help up to 1000°C. On the other hand working at cryogenic temperatures might be a seasonal thing, attempting to buy thick winter gloves in the middle of summer might prove to be difficult. Still though even if it is something like a pair of gloves for welding, they are insulated to keep you from the heat, they can be used just as effectively to protect you from colder temperatures, but the reverse is not true, most of the fabrics used in winter gloves and such might combust if brought into contact with hot iron. Working at high temperatures is another aspect of chemistry that depends entirely on you, some people might never need a thermometer that goes over 150°C and some people might work at high temperatures all the time, or cryogenic temperatures for that matter and never get into the realm where a thermometer would be realistic.

Aside from the personal gear that is worn it is also a good idea to have some extinguishing media around in case of fire. A fire extinguisher will work for most situations except some metal fires. In those cases sand (another good thing to have on hand) is used to smother the fire(2). In that case it is best to move away any hazardous chemicals you can get to and let the fire burn itself out. Putting water on a magnesium fire or other highly reactive metal can easily lead to explosion.

Another good thing to have handy are neutralization chemicals. In case of an acid spill you should have a nice wide mouthed container of sodium bicarbonate (baking soda) laying around, tossing this on an acid spill will neutralize its corrosive properties and render it somewhat safer at least so you can clean it up. Base spills are usually less of a problem but sodium bisulfate (used to adjust the pH of pools) can be conveniently located around your lab. Flushing most acids or bases with large quantities of water also helps the situation, however in the case of large spills of concentrated phosphoric or sulfuric acid extra care should be taken as these heat up greatly on combination with water, if you are applying water to these do so very quickly and in large quantity to help remove the heat and prevent *flash boiling.*

Always remember that safety should come first. It is not worth getting a severe burn because you are too cheap to buy some long sleeve welding gloves or respiratory damage because you won't invest in a gas mask. Always consider the possibility of long-term damage and if you think there is something reasonably extra that you can do to prepare for an upcoming reaction spare no expense. It has been learned by many chemists over the years, take the time to do it right the first time, get the right chemicals, set up the apparatus with all the extra safety precautions you think might be necessary but don’t want to take the time to do, keep your patience and don’t rush and things will always turn out a lot better.

(1) One exceptional substance that can easily penetrate gloves is methyl mercury, which can go through many com available glove materials, but considering the toxicity of it, it is better to just not use this compound.

(2) Note that fires of very reactive metals like magnesium will not appreciate being smothered in sand and will conti react even with the sand, sometimes even more vigorously then with the air, there are special mixtures ava commercially to smother these fires which contain among other things magnesium oxide.

1.2(a) Definitions of common medical terms

Lets assume that you are being responsible and looking out for your own health and the heath of those around you and you are looking up MSDS sheets of the chemicals you will be working with. Some of the information on the sheets may be easy to understand, but when it comes to side effects of chemicals you might often come upon words such as ‘pulmonary edema’ and ‘renal failure’ and you wonder to yourself, “What do these words mean?” This can be a stumbling block when deciding on choosing one compound over another, not knowing the potential side effects puts you at a large disadvantage, although you are still encouraged to look up specific definitions for each of these terms, here is a general overview of the major terminology used:

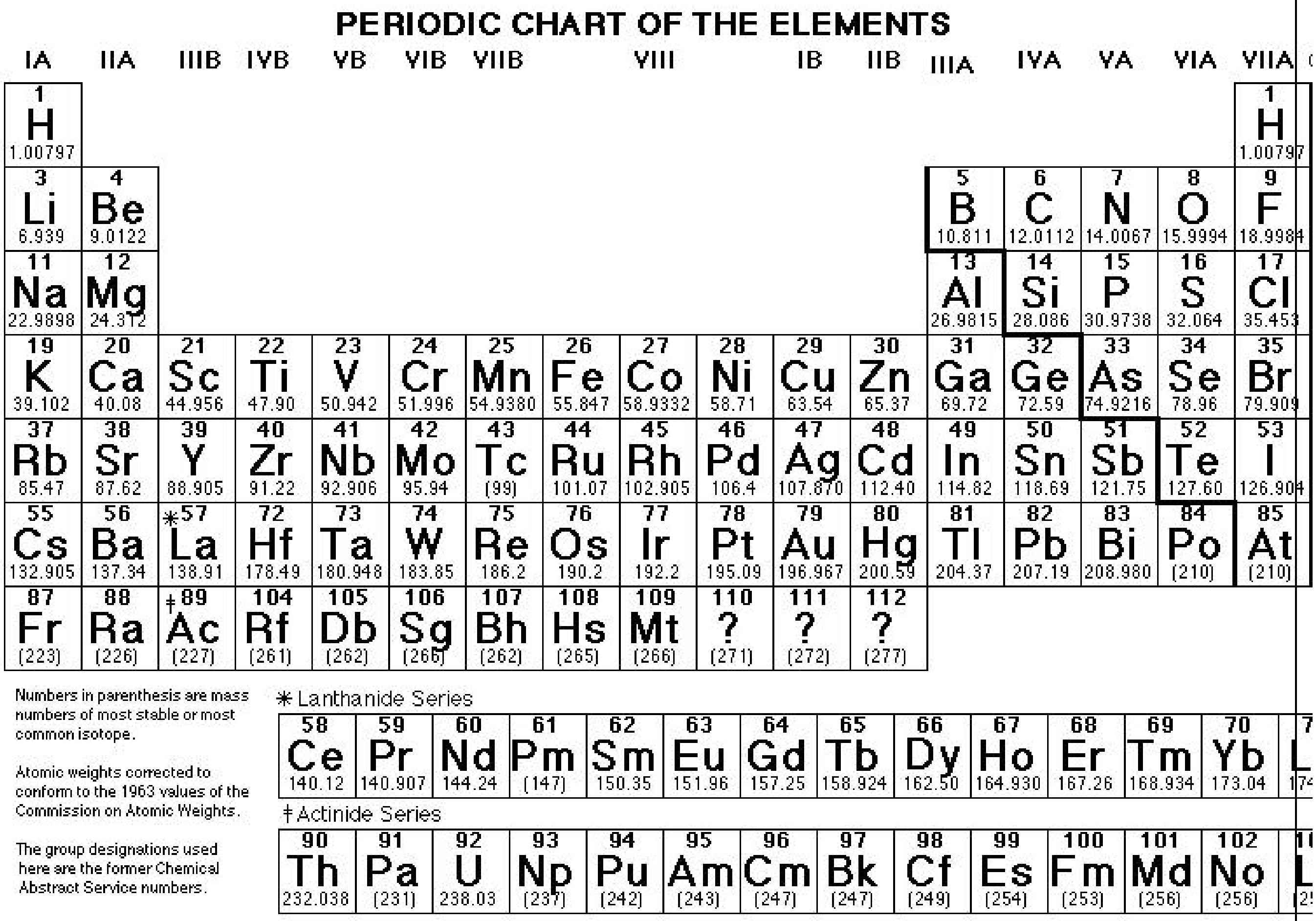
Renal Failure: Often used to designated that a chemical upon entering the body may cause massive damage to the kidneys causing them to shut down usually permanently.

Pulmonary Edema: Corrosive gasses and other chemicals can cause this condition where fluids build up in the lungs, in serious cases this can be so bad that you can drown in your own fluids.

Necrosis: A term used to describe the process of tissue dying and/or rotting while still on the body.

1.3 How to read/write a chemical reaction

If you're just beginning to start a chemistry hobby there are a few skills that you should have. The most useful of these is reading and writing chemical reactions. There are many places on the web and in books and classrooms what will be able to more thoroughly explain this procedure then I will be able to, these skills are easy to explain but take practice to understand, therefore this section is mostly to just show the standard way in which a chemical reaction will be written in this text.



PERIODIC CHART OF THE ELEMENTS

IA	IIA	IIIB	IVB	VB	VIB	VIIB	VIII			IB	IIB	IIIA	IVA	VA	VIA	VIIA
1 H 1.00797																1 H 1.00797
3 Li 6.939	4 Be 9.0122											5 B 10.811	6 C 12.0112	7 N 14.0067	8 O 15.9994	9 F 18.9984
11 Na 22.9898	12 Mg 24.312											13 Al 26.9815	14 Si 28.086	15 P 30.9738	16 S 32.064	17 Cl 35.453
19 K 39.102	20 Ca 40.08	21 Sc 44.956	22 Ti 47.90	23 V 50.942	24 Cr 51.996	25 Mn 54.9380	26 Fe 55.847	27 Co 58.9332	28 Ni 58.71	29 Cu 63.54	30 Zn 65.37	31 Ga 69.72	32 Ge 72.59	33 As 74.9216	34 Se 78.96	35 Br 79.909
37 Rb 85.47	38 Sr 87.62	39 Y 88.905	40 Zr 91.22	41 Nb 92.906	42 Mo 95.94	43 Tc (99)	44 Ru 101.07	45 Rh 102.905	46 Pd 106.4	47 Ag 107.870	48 Cd 112.40	49 In 114.82	50 Sn 118.69	51 Sb 121.75	52 Te 127.60	53 I 126.904
55 Cs 132.905	56 Ba 137.34	✳57 La 138.91	72 Hf 178.49	73 Ta 180.948	74 W 183.85	75 Re 186.2	76 Os 190.2	77 Ir 192.2	78 Pt 195.09	79 Au 196.967	80 Hg 200.59	81 Tl 204.37	82 Pb 207.19	83 Bi 208.980	84 Po (210)	85 At (210)
87 Fr (223)	88 Ra (226)	†89 Ac (227)	104 Rf (261)	105 Db (262)	106 Sg (266)	107 Bh (262)	108 Hs (265)	109 Mt (266)	110 ? (271)	111 ? (272)	112 ? (277)					

Numbers in parenthesis are mass numbers of most stable or most common isotope.

Atomic weights corrected to conform to the 1963 values of the Commission on Atomic Weights.

The group designations used here are the former Chemical Abstract Service numbers.

✳ Lanthanide Series

58 Ce 140.12	59 Pr 140.907	60 Nd 144.24	61 Pm (147)	62 Sm 150.35	63 Eu 151.96	64 Gd 157.25	65 Tb 158.924	66 Dy 162.50	67 Ho 164.930	68 Er 167.26	69 Tm 168.934	70 Yb 173.04

† Actinide Series

90 Th 232.038	91 Pa (231)	92 U 238.03	93 Np (237)	94 Pu (242)	95 Am (243)	96 Cm (247)	97 Bk (247)	98 Cf (249)	99 Es (254)	100 Fm (253)	101 Md (256)	102 No (256)

Pictured above is your basic periodic table (See section **12.1** for a complete legible listing off all the elements in alphabetical order). The periodic table lists elements by increasing atomic number (which usually means increasing atomic weight). Also it has trends in it which can allow you to predict the properties of an element in its uncombined state. The most common grouping that people fall back on is family. That is the vertical groups of elements. For example, on the far right, second to last column, beginning with the letter F, that family is the halogens. In descending order they are Fluorine, chlorine, bromine, iodine and radioactive astatine. Fluorine is the most reactive of the group and the reactivity decreases as you go down.

The ‘atomic weight’ previously mentioned is defined by the quantity called the mole (also referred to simply as mol or a mol). This is a chemistry quantity and what it basically means is ‘one unit’. So, if you look at potassium on the list (K is the symbol for potassium) you will see 39.102 underneath it, what that means is that 39.102 grams of potassium metal is one unit of potassium. All the elements have units with respect to one another. For example one unit of chlorine (Cl on the list) weighs 35.453 grams and it will react with one mol of potassium (39.102 grams) completely to give 1 mol of potassium chloride, KCl which will have weight equal to the sum of the two since mass is not lost to give a total weight of potassium chloride of 74.555 grams, this reaction would be expressed by the following system:

$K + Cl$ ---> KCl

Reactions in this work will be in the same form of *A* + *B* ---> *AB*

Another specific example would be **$2H_2 + O_2$ ----> $2H_2O$** Translating this from chemical speak to common tongue is simple. Looking at a periodic table you find that the letter H represents hydrogen, a colorless odorless gas, and that O represents oxygen, and I would hope you are all familiar with the product H_2O. The prefix, that is the number before the letter signifies the number of whatever it is in front of that take part in the reaction. The numbers behind the letters stand for the number of that element in a compound. Hydrogen and oxygen are gasses that exist not as individual atoms but as two atoms bound together, hence the two behind each of them. So what it is saying is, four atoms of hydrogen, combine with two atoms of oxygen, to produce two molecules of water. Note the distinction between atoms and molecules, O_2 is a molecule containing 2 atoms of oxygen, H_2O is a molecule containing 2 atoms of hydrogen and one atom of oxygen. Chemical reactions of this type are balanced, with each molecule appearing in the same quantity on both sides, if that is not the case the equation is dubbed 'unbalanced' and steps can be taken to rectify it (Note that although a molecule of oxygen contains two oxygen atoms you can still treat it like it is just O in equations, just double the molecular weight to get the weight of the O_2 molecule).

Diatomic Molecules and More

When it comes to a chemical reaction the periodic table gives the symbols only for individual atoms of an element, which is not how many of them are found. For example, many gasses are diatomic molecules (di meaning two in this case, so the molecules are formed from two atoms). What this means is that two atoms of the substance have a bond between them and therefore come in a pair. Here is a quick list of elemental gasses that are diatomic:

H_2, N_2, O_2, F_2, Cl_2, Br_2, I_2

Although bromine and iodine are liquids and solids respectively, in the gas phase they consist of two atoms bonded together. These bonds between the atoms account for a degree of their reactivy, for example, the bond between fluorine atoms is very weak, it breaks readily from light and as a consequence you end up with highly reactive free fluorine. Chlorine will also break its bond to itself from daylight, when the bond breaks it can attack hydrocarbons and chlorinate them. On the other side of the situation the nitrogen-nitrogen bond is actually a triple bond, there are three bonds connecting one nitrogen atom to another. This is actually one of the strongest bonds in chemistry and accounts to some extent for the lack of reactivity of nitrogen (note that it accounts for a large portion of the air we breath). The fact that these are diatomic is not of great importance in chemical reactions because the molecular weight of one atom is still the same, however it is important in calculating the amount of a gas. One mole of gas usually takes up approximately 22.4L of space at standard temperature and pressure. But it’s not one mole of O that takes up that space, it is one mole of O_2 so actually there would be two moles of atomic oxygen for 22.4L of space.

Going further there are other elements that associate so their formulas are usually or can be expressed as different combinations of atoms, explanations are not given here but notable examples include B_{12}, S_8, and P_4.

Exceptions to this rule are nonstoichiometric reactions, reactions that do not have a specific reaction that takes place and a number of products can be formed under different conditions though a main product is usually known or is the desired product of the reaction. This includes a wide variety or organic reactions, a specific example being:

$$\mathbf{C_6H_6 \text{ --}(HNO_3/H_2SO_4)\text{-->} C_6H_5NO_2}$$

Notice how the by product, water is not included in the reaction, and that the amount of HNO_3 (nitric acid) and H_2SO_4 (sulfuric acid) reacting with the C_6H_6 (benzene) to produce $C_6H_5NO_2$ (nitrobenzene) is not a part of the equation, this can be read '*in the presence of*' therefore allowing the reaction to be read, "Benzene, in the presence of nitric acid and sulfuric acid reacts to produce nitrobenzene". Another time that information can appear in between the arrows could be a *catalyst* which only speeds up the reaction, additionally temperature information can appear there as can pressure. Specific reaction conditions are not usually included in the condensed equation and it is not safe to assume that every reaction you see will run at STP (standard temperature and pressure) as a matter of fact there are many reactions not run at STP and because of this you really hardly know anything of reaction conditions when you see a reaction written out, this can be a real pain in the butt.

Now lets say you want to write a chemical reaction. First you should know the chemicals/elements involved along with what you believe to be the products. You write them out in the format previously described, reactants (things reacting to give your desired product) on the left and products on the right, then you attempt to balance the equation by adding to the products or reactants side. If an element only shows up on one side something is automatically off. Just remember that there is not always one correct chemical reaction and just because a reaction looks good on paper does not magically make it the real reaction, a reaction that is nearly impossible may look just as plausible on paper as a simple reaction time tested for the past hundred years anyone can make up a reaction, be wary of reactions you see with no information backing them up.

In addition to the common elements there are also components that the average chemist will come across that almost behave as though they are elements. They are more evident in aqueous solutions in the form of ionic compounds. Collectively they are known as ions but more specifically positively charged species are known as *cations* and negatively charged species are known as *anions*. Most aqueous chemistry (chemistry occurring in water) revolves extensively around cations and anions and it is quite useful to have a ready reference list of cations, anions, and their respective charges, and it just so happens that there are many lists available besides the one here:

Anions:

Acetate	$C_2H_3O_2^-$		Hydrogen Carbonate	HCO_3^-		Hydride	H^-
Arsenate	AsO_4^{3-}		Carbonate	CO_3^{2-}		Hydroxide	OH^-
Arsenite	AsO_3^{3-}		Chloride	Cl^-		Nitrate	NO_3^-
Azide	N_3^-		Hypochlorite	ClO^-		Nitrite	NO_2^-
Bismuthate	BiO_3^-		Chlorite	ClO_2^-		Nitride	N^{3-}
Bisulfate	HSO_4^-		Chlorate	ClO_3^-		Oxide	O^{2-}
Sulfate	SO_4^{2-}		Perchlorate	ClO_4^-		Peroxide	O_2^{2-}
Hydrogen Sulfite	HSO_3^-		Chromite	CrO_3^{2-}		Phosphate	PO_4^{3-}
Sulfite	SO_3^{2-}		Chromate	CrO_4^{2-}		Phosphite	PO_3^{3-}
Thiosulfate	$S_2O_3^{2-}$		Dichromate	$Cr_2O_7^{2-}$		Metaphosphate	PO_3^-
Hydrosulfite	$S_2O_4^{2-}$		Cyanide	CN^-		Phosphide	P^{3-}
Peroxy-disulfate	$S_2O_8^{2-}$		Thiocyanate	SCN^-		Permanganate	MnO_4^-
Bisulfide	HS^-		Cyanate	OCN^-		Iodide	I^-
Sulfide	S^{2-}		Fluoride	F^-			
Borate	BO_3^{3-}		Formate	$HCOO^-$			
Bromide	Br^-		Oxalate	$C_2O_4^{2-}$			

Red = Usually insoluble in water **Blue** = Normally soluble in water **Black** = Follows no trend

Cations:

Hydronium Ion	H_3O^+		Ammonium	NH_4^+		Lithium	Li^+
Sodium	Na^+		Potassium	K^+		Magnesium	Mg^{2+}
Calcium	Ca^{2+}		Barium	Ba^{2+}		Chromous	Cr^{2+}
Chromic	Cr^{3+}		Manganous	Mn^{2+}		Manganic	Mn^{3+}
Ferrous	Fe^{2+}		Ferric	Fe^{3+}		Cobaltous	Co^{2+}
Cobaltic	Co^{3+}		Nickelous	Ni^{2+}		Nickelic	Ni^{3+}
Cuprous	Cu^+		Cupric	Cu^{2+}		Zinc	Zn^{2+}
Silver	Ag^+		Aluminum	Al^{3+}		Stannous	Sn^{2+}
Stannic	Sn^{4+}		Plumbous	Pb^{2+}		Plumbic	Pb^{4+}

Mind you there are more cations and anions then just the ones listed here, these are just common examples. The charge of an unknown cation is usually more easily determined then that of an anion, especially if you are given a name. Charges of anions usually stay constant whereas metals can have differing charges, knowing the anion a metal is coupled with can give you an indication of what the oxidation state of the metal is. In addition some names are currently written out using the stock system. This greatly simplifies things, instead of a name like manganese dioxide you get manganese (IV) oxide, the Roman numeral four indicating that manganese is in the +4 state and therefore knowing that oxygen has a negative two charge you can determine the formula of this compound to be MnO_2. The use of –ous and –ic at the end of some names to differentiate between the higher and lower oxidation states is an older phenomenon and is somewhat being phased out, however tin (stannous +2 and stannic +4) and lead (plumbous +2 and plumbic +4) are somewhat stuck in this system of naming. Regardless, there are many anions, many cations, existing in different situations, some not stable in water, some only found in water, and others only existing in the solid state. Just remember the overall charge of a molecule must remain neutral.

1.4 Units used throughout the text

The system used in this text will be the most accepted system in chemistry academia. The all mighty metric system. Units of weight will often be expressed in grams (g), of volume, in liters (l) and milliliters (ml) and time in seconds (s), hours (h), and days. In addition temperatures will be measured in degrees Celsius (°C).

When it comes to liquids though there are different units that come into play aside from milliliters. The most useful unit is molarity. This is defined as the number of mols of a substance (solute) dissolved in 1 liter of substance (solvent). From here you can convert one molarity solution to another using the formula:

Molarity Initial (Mi) x Volume Initial (Vi) = Molarity Final (Mf) x Volume Final (Vf)

For Example:

Chemoleo wants to make a 1 M NaOH Solution in water. So he weighs out one mole of NaOH, looking at the periodic table he finds the atomic mass of sodium to be 22.9, that of oxygen to be 15.9 and that of hydrogen to be 1.0, adding these together he gets the weight of one mole of NaOH to be roughly 40 g. So after weighing out 40 g of sodium hydroxide prills he adds to them enough water to make the total volume 1 L thus making a 1 M solution. This sits on his shelf for quite some time until one day he finds that he needs 100ml of a .5 M NaOH solution. Having three components of the above equation he can solve for the initial volume of 1 M NaOH he needs to end up with a 100 ml amount of a .5 M solution.

$$1\text{M} \times (V_i) = .5 \times 100 \text{ ml}$$

$$V_i = 50 \text{ ml}$$

So Chemoleo must take 50 ml of his 1 M NaOH solution and add to it 50 ml H2O to bring the total volume to 100 ml of .5 M NaOH solution. Remember to label any reagents you keep laying around Chemoleo.

The molarity unit is exceptionally good for one specific reason, it greatly simplifies calculation involving precise reactions and the amount of reagents you are dispensing.

Molarity is heavily used in *stoichiometry* and is the staple method of labeling many lab reagents. In physics academia a more popular unit is molality, this is a measurement of mols per kilogram solvent, in this way the molality of a solution will not change with temperature whereas molarity will due to the change in volume of the liquid as the temperature fluxuates.

Another method of measurement one will come across is the percent (%) solution. There are different variations on this, the weight/volume method, the volume/volume method, and the weight/weight method. One common example of a % solution would be 6% NaOCl available OTC as bleach.

The chemist known as BromicAcid just bought 3.8 L of a 6% NaOCl solution. In order to keep his lab space organized he must retain the labeling method he has already begun for his other reagents, therefore he must determine the molarity of the 6% NaOCl solution. So the solution is 6% NaOCl by weight, so 6.0 g / 100 g solution. Now, density would come in handy here, however Bromic was unable to find the information on the web and is too lazy to do physical measurements, therefore he is assuming that the density of the solution is close to water so 1.0 g/ml therefore 1000 ml or 1000 g would have approximately 60 g NaOCl. NaOCl has an atomic weight of around 74 g/mol therefore Bromic has 60/74 = .80mols of NaOCl per liter so the molarity equals 0.80M for the solution of NaOCl to store on his shelf.

Often times, as seen above the density of the solution is necessary to determine a more precise molarity calculation from the percent solution. Tables are available online and in the CRC (Handbook of Chemistry and Physics) and elsewhere that give molarity to percent to density conversions that will aid in this task.

Common Percent Solutions to Molarity

Substance Name	Percent Solution (in H2O)	Molarity
Sulfuric Acid H_2SO_4	100% 91% 40%	18.7 M 17.1 M 5.4 M
Nitric Acid HNO_3	70% 90%	15.8 M 21 M
Hydrochloric Acid HCl	20% 28% 38%	6.0 M 8.7 M 12.4 M
Ammonia NH_4OH	4%	2.3 M
Acetic Acid CH_3COOH	5%	.8M
Sodium Hypochlorite NaOCl	6% 10% 12.5%	.85M 1.45 M 2 M

Hydrogen Peroxide H_2O_2	3%	1.25 M

1.5 Discussion of Legality/Words of Encouragement

Practicing chemistry as a hobby has fallen out of fashion in recent years and as such trying to delve into it may cause you to be greeted with skepticism at best and at worst a strange kind of desperation to wipe you off the earth. Trying to keep out of the public eye is usually a necessity for the continued practicing of chemistry. Despite the fact that many reactions are totally legal there always seems to be a way for you to get into trouble if someone decides to make trouble for you. Example, phosphorus in all of its *allotropes* are illegal to own anywhere in the United States. If you attempt and succeed to make phosphorus you are really breaking the law. This is because there are illicit (illegal) uses for this chemical and because of this, it has been outlawed. As such the chemistry of elemental phosphorus, the useful phosphorus halides and other chemicals are taboo unless you break the law.

But the actual list of chemicals that are illegal is very short compared to the chemicals that can be made by a motivated individual. And as such there are plenty of legal target chemicals that one can take aim at. Try not to break the law, many energetic compounds are illegal, as of course drug precursors are but do not let that discourage you from what you can legally do. Chemistry is fun and if you keep up a beginning spirit you will have fun with it for a long time to come. Not only that but it’s useful, giving chemistry a try might be the best thing you do for yourself.

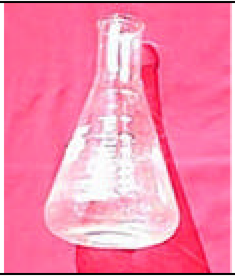
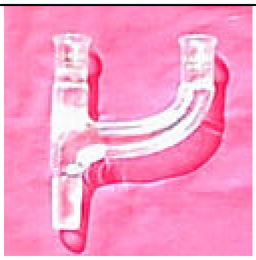
2.0 Reaction vessels

It all starts, sometimes even before the chemicals, with choosing what you will be doing your reactions in. In the beginning it is common to improvise your glassware, just re-using old jars and bottles to store reagents that you procure or produce or to run reactions in. However as time goes on you start to realize you might not be able to heat your bottles without them shattering, and those plastic pop bottles that at one time seemed like a stroke of genius to store things in, are now melting like candles from the corrosive fumes. Well, we all have to start somewhere, and even soda glass has its place, so take the time to read through these varied reaction vessels. Remember glass is the old standby but there are different kinds of glass and there are some reactions that, either though intense heat or specific reagents, are unsuitable to be run in glass.

2.1 Glassware

Most laboratory work is safe to conduct in some sort of glass apparatus. And that’s great news, glass is resistant to most chemical attack; notable exceptions being strong hot bases, and most definitely hydrofluoric acid/some fluorides. Glass also has a high melting point. Glass will deform at high temperature but some types of glass will shatter along the way as you will see from the following descriptions. Another plus is that it is amorphous, and by lacking a crystal structure it is clear, allowing you to see reactions taking place inside the vessel and to allow measuring of liquids using graduation marks found on many pieces of glassware. Glassware is also convenient for storing regents for long periods of time; carrying out complex refluxing; distillation under high heat or pressure/vacuum, glass is the containment choice for nearly every chemist under most situations.

There are many forms of common glassware including beakers, flasks, tubes, test tubes, funnels, pipettes, graduated cylinders and watch glasses. There are also more exotic (and more expensive) glassware products including separatory funnels, ground glass jointed distillation flasks and jacketed condensers. I am only going to explain the purpose of some of the more common glassware that a new home chemist would have.

		
Standard funnel	Beaker	Round Bottom (RB) Flask
		
Graduated Cylinder	Volumetric Flask	Sepretory Funnel
		
Erlenmeyer Flask	Claisen Adapter	90 Deg Vacuum Adapter

		
Still Head with Thermometer	Vigreux Column	2-Neck Flat Bottom (FB) Flask
		
	Liebig Condenser	

Beakers: These are simple cylinders with a pour spout on the lib and a flat bottom. Many times beakers have graduations on the side but be warned that these are not as accurate as from a graduated cylinders. Beakers are used to mix or dissolve substances, as simple heating vessels and sometimes as heating or cooling bath containers.

Flasks: There are two types of flasks; Florence flasks (sometimes called boiling flasks) and Erlenmeyer flasks. Florence flasks have a round body with one or more necks going into them. Some have round bottoms and some have flat bottoms. Round bottomed flasks need stands to hold them up but are stronger so you can use a vacuum with them without fear of an implosion. Erlenmeyer flasks have a cone-like body and are used for simple heating and, with a side nipple, for vacuum filtration. Volumetric flasks are simply Florence flasks with a flat bottom and a very long neck with a mark at the 500ml or 1 liter line. They are used to prepare a solution of known molarity.

Tubes: Tubes are simply glass cylinders. Some are made of Pyrex but most are made of soda glass. By melting and blowing with the help of a burner the home chemist can make simple equipment to help with an experiment. For example he could wrap a cooking thermometer made of metal in glass to increase its chemical resistance or make a simple gas drying tube. Pyrex tubing must be melted with an oxygen rich flame. Tubes can be bent once heated to carry liquids or gas to different glassware.

Test Tubes: Test tubes are simply tubes with a rounded end and a lip made of Pyrex or soda glass. Small reactions can be run in them and small amounts of substances can be stored in them. For example a small bit of potassium metal could be stored in oil in a test tube properly sealed at the top. Because the bottoms are rounded they are susceptible though to being dropped. The bottom can easily crack out in these instances and spill liquids everywhere and care should be taken when using stirring rods and they can also puncture holes in the bottoms of test tubes.

Funnels: Funnels can be used to filter things when you use filter paper or to add liquids or powders into a small area like the mouth of a test tube.

Pipits: Pipits are glass tubes with a small hole in one end and a larger hole in the other. They have very accurate volume measurements on the side of the glass. Pipits are used to suck up liquids, measure small amounts of liquids and place small amounts of liquids somewhere. Never pipit by mouth; always use a rubber pipit bulb.

Graduated Cylinders: These are simply large tubes with a stand on the bottom and a pouring spout. They are used to measure volume and come in sizes from 10ml to 500ml. 100ml cylinders are the most common.

Watch Glasses: These are curved dome-like pieces of glass that can be used to hold powders, cover beakers or make "cold fingers" for sublimation purification of things like iodine crystals.

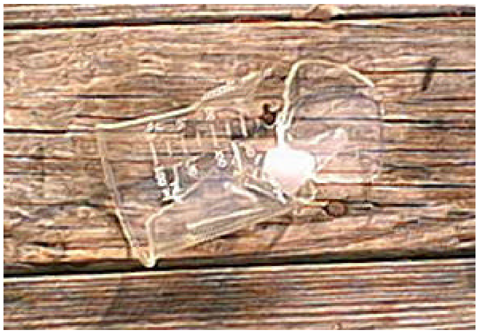
2.1a Pyrex/Borosilicate Glassware

(From left to right …..)

"Pyrex" is a brand of high quality borosilicate glass but the name is used to refer to all sorts of heat resistant glass. Borosilicate glass is simply the type of glass that most high quality labware is made out of, it's most notable difference between normal soda lime glass being a high percentage of boric oxide in the mix which reduces its expansion as it heats. Some of the cheaper glasswares made by companies like Bomex can show lessened resistance to heating and usage, this should be taken into consideration when purchasing glassware of unknown origin. Pyrex and Kimax are good mid priced glassware while Duran is top quality. Although technically any piece of glassware can have ground connections they appear almost exclusively with borosilicate glassware. Ground glassware is simply a larger diameter opening called the female end and a smaller diameter which is frosted on the outside called the male, the male fitting into the female and the area of their connection actually ground to give an even connection. There are many different sizes of joints with which glassware can connect and most countries have their own sizes that are most commonly available.

Other then the glassware intended strictly for purposes of chemistry some glassware available readily from your grocery store is likely Pyrex® these types of glassware include measuring cups and baking dishes, heating these is not recommended by the manufacturer on an open flame but it can be done, there are also glass pans made from these heat resistant glasses, these are great for reducing the volume of aqueous solutions and such to a manageable level though their availability is sometimes limited.

Never assume that your glassware is Pyrex or other another heat resistant type, back in my early days I was planning to boil down about 400ml of $CuCO_3$ in water. At that time I did not have a nice hotplate like I do now so I tried to use the stove. I grabbed a cooking bowl made of glass and poured the greenish mess into it. I then placed it on the stove and started stirring it when after a few minutes it cracked into about four parts. The nasty stuff got all over the oven and dripped onto the floor and the range area. A thousand thoughts started to rush through my head. "$CuCO_3$ + HCl in stomach --> Death?" I cleaned like a cheap animation on a laserdisk stuck in fast forward. Don't assume all glass is Pyrex without looking.



Your glassware will break, how often that happens of course depends on what kind of reactions you do, where you work, and most of all how careful you are when you experiment. But this will happen. Broken Pyrex though is still useful. It can be kept and broken further in order to fill fractioning columns, normally these are filled with Pyrex beads, but broken glassware works as well and the beads used, being Pyrex, are expensive and it can cost over $100 to fill one of these columns with beads but you can just use your broken glassware. Another use is for boiling stones, broken glassware can help to form nucleation points in boiling and keep things boiling smoothly, especially in test tubes. Shards can be used to scrape out beakers, and for filler in reaction vessels, for example shards of glassware can be used to fill a container and keep a powder or chunky solid separate so it doesn't clump together in one mass, or they can also be used in catalyst tubes. The possibilities for broken glassware are unlimited and Pyrex is not something a chemist should throw away lightly.

Ground glassware joints are occasionally subject to a process called freezing. In this, usually, some foreign substance has made its way between the ground joint (Could also be something like vacuum grease or something else you put there) and for some reason or another the glassware becomes fused at the joint and undoing it seems nearly impossible. Storing strong bases in glassware can also freeze the joints from hydroxide getting into the ground joint and somewhat dissolving some of the glass. In any case there are a number of steps you can take to attempt to free your glassware joint so that you can use it once again.

1. Soaking the joint for several hours or even days might help, water is a good first choice, base and acid solutions can be helpful for specific culprits, and organics such as ethanol or stronger non-polars could also free the joint.
2. Next try gentle heating with a blow dryer. Attempt the twist back and fourth while heating.

From there though the processes begins to give a greater possibility of damaging the glassware, such as tapping the glassware, heating with a Ni-Cr wire, or using a torch. Hopefully you will succeed in getting the glassware unfroze, and if you manage that you should take some fine steel wool and gently go over the joint again to free it from any clinging particulates and be sure to clean it well before using again.

Cracks may be difficult to see when looking straight on at an object.

Before heating any borosilicate glassware it is good to check it carefully for defects. Cracks and pits known as stars can lead to catastrophic failure at high temperature due to the expansion of the glass causing tension along the fractures created. Although it is not something to worry about compulsively, if you are using your glassware over high heat containing any corrosives, oxidizing materials, or anything that presents a high hazard situation then beforehand do yourself a favor and give your glassware a quick check over for defects.

2.1b Soda-Lime

In all likelihood this is the type of glassware with which you have had the most contact throughout your life. If it is made out of glass, and there is no need to heat it, it is probably some composition of soda-lime. The number one advantage of soda-lime, over all other types of glassware from an industrial point of view, is the cost of production. For what you get, i.e., a fairly inert transparent material, it has a good cost to usage ratio. So jars are made from it, drinking glasses, vases, light bulbs, and more.

So, it's cheap to produce and therefore you have lots of it laying around, so by that same virtue it is cheap for you to use since it is so widely available. Many people will use soda-lime glass somewhere, sometime, for some reason or another, some people only own soda-lime, while others shun it. Well, it's easy to see where its use can come into play, first, lets look at the advantages of soda-lime:

- Resistant to most chemicals (Exceptions are hydrofluoric acid, fluosilicic acid, concentrated phosphoric acid, or hot/concentrated bases)
- Cheap, and widely available
- Electrical insulator (Can be a good thing or a bad thing.)
- Has a continuous high use temperature of almost 125 C

Now many people will say, wow, soda-lime doesn't look like a bad choice for glassware at all. As a matter of fact, its properties closely match those of the preferred type of glassware for chemical reactions, borosilicate glass. However there is one major exception.

Soda-Lime glass is not meant for heating!

There are people that would argue over this point. Not many though. Aside from gradual heating, in a water bath or just for very short periods this rule will catch up with you. Soda-lime has a terrible time of trying to be conductive toward heat, and as such one part of the glass will try to expand while the other parts do not and you get hairline cracks, usually not even noticing them until it is too late. The average scenario plays out like this.

A chemist is working at home and they have constructed a distillation apparatus consisting of a jam jar into which a tube has been secured into the lid to lead away the vapors. This jar is heated on a hot plate and the liquid begins to boil and distil across the tube. The procedure continues without interruption until the chemist notices some precipitate in the reaction container. Out of curiosity they go to pick up the container to swirl it and better ascertain the identity of the unknown precipitate. But when they go to pick it up the whole top comes away. The bottom has broken off cleanly from the uneven expansion of the glass as it heated, and the liquid filling the flask, hot and boiling, goes everywhere. This is actually a common situation.

For example the picture at left shows a setup for washing gasses, note that the glass components are regular jars over the counter, and the are well suited for this purpose as temperatures and pressures involved are not that great. Also to note is the glass tubes used. Glass tubing is widely available and you have a choice of soda-lime or borosilicate, but in this case the differences between the two are less noticeable. Both can be heated over an open flame to get them to their softening point where they can be bent into a number of shapes suiting your purpose. Although soda-lime is sensitive to heat as long as it is dry the area that is being heated is so small that it can be done safely.

Heating soda-lime is its major weak point and should be undertaken with caution and only where the contents of the container do not pose a significant hazard. However it is good for any of a number of reactions where there is no heating involved, gas generation apparatuses, storage of liquid or solid reagents, or for many other things. Notice that water or aqueous solutions should not be stored in soda lime or any other glass container if there is the possibility of freezing, the expansion of the liquid as it freezes can and often will crack and destroy the containers you have chosen. Nevertheless, soda-lime does have it's place in the home lab, just be sure to consider the fault in heating it.

2.1c Lead Glass, Vycor, Misc.

Vycor™, also known as fused silica or vitreous silica is the holy grail of glassware. Composed entirely of SiO_2 fused together at high temperature, it is very resistant to thermal shock, even more chemically resistant then glass, able to be heated to nearly 1200 °C in use, and does not soften until nearly 1500 °C! This would be the preferred way to go for many chemical vessels. Problem is, owing to its high melting point and exacting standards of preparation fused silica is quite expensive and hard to acquire. Often when you encounter fused silica it will be in the form of a crucible for use in a furnace. Not clear and usually white these crucibles are great for ashing samples for analysis. The absolute cheapest source of fused silica for the home chemist is from online jewelry supply stores. They sell small fused silica crucibles for the explicit purpose of melting precious metals such as gold within the average jewelers shop. These are also often shaped in such a way as to allow direct intense heating of the contents and are thus suitable for direct intense heating of pretty much anything and are considerably cheaper then fused silica crucibles intended for laboratory use.

There are a number of specialty glasses to be found on the free market not listed here. Lead glass is known for being dense and having a high refractive color on cut surfaces. Its use is mainly ornamental, if used in a chemistry lab however there is a high probability that any liquid contained without would leech out a portion of the lead contained and ruin most stored reagents this way. The same holds true for Vaseline glass. This light green glass is also used for ornamental purposes and is quite old usually. The pigment in the glass is uranium oxide and as such this could be leeched with acids leading to ruined reagents. The point of this being that unknown glasses should not be used for the storage of reagents because although they may look aesthetically pleasing they may cause harm to the chemicals contained within.

2.1d Cleaning Glassware

It should be your goal every day, at the end of your experiments to make sure you keep your glassware clean. Dirty glassware can catalyze decompositions, destroy glassware, result in inaccurate measurements, or simply result in you having a solution left in a beaker, unlabeled, of which you have no idea of the contents. Plus if you need to use a piece of glassware that is dirty you usually have to stop what you're doing and wander off to clean the glassware, and you better hope water won't hurt the reaction otherwise you have to dry it too. And things tend to stick in glassware over time, best just to clean it when you use it.

Oft times cleaning glassware is as easy as rinsing out the contents with water, then maybe distilled water and allowing to drip dry. However cleaning organics is another story. Most times in laboratory settings organic compounds are initially rinsed out with acetone, and then water. This combination works well as the acetone removes and dilutes the concentrations of insoluble organics but is itself soluble in water. It's a nice in between solvent for this. Of course you could use a strictly organic solvent like toluene or xylene or even something like methanol too but the first two of these will pose their own problems in terms of their ability to be washed out with simple water.

What about the other times though, how about if something gets stuck inside your glassware. The best advice is to use your brute strength and rub it out. Let it dry and get a cloth and just try and wipe the spot out. This can save a whole world of pain in trying to find the perfect solvent. Even for rings and such inside flasks where they are impossible to get to, improvise, use a pen and bend it in the middle and use that to wipe a piece of paper towel around on the inside. If this does not work, usually it will make it smear or it just might not rub off. Then you have to start to analyze the situation. Is the stain organic? If it is try and use some acetone or ethanol. Next give some acids a try, hydrochloric is a good starter in case it is acid reactive. If you want to step up from here try sodium hydroxide in ethanol and let that soak about an hour (no longer, this mixture can attack glass joints and ruin volumetric flasks). If that doesn't work, boiling nitric will oxidize even carbon to CO_2 which should clear up nearly any mess you can make.

There are other mixtures too though. A mixture of acidified potassium dichromate (usually with sulfuric acid) is a tried and tested method to clean glassware. But remember the carcinogenity of chromium in the +6 oxidation state such as it is here(1), also this is a strong oxidizing agent it can set fire to some organics, cotton in contact with it will develop burn holes quickly if it is fairly concentrated. Another powerful method uses a mixture of concentrated sulfuric acid with strong hydrogen peroxide solution. This solution known as Caro's Acid or "Piranha Solution" is a very strong oxidizing solution and it is the authors recommendation to avoid this as it can explode from contact with easily oxidizeable organics. Another safer alternative might be Fenton's Reagent(2) which is utilized by add a soluble iron salt to a solution of peroxide and acidifying mildly. This can take some time but it will remove most stains. There are many other solutions available including pre-packaged alternatives, many of which involving hydrogen peroxide or solid peroxides such as sodium perborate.

At the very least these methods should loosen stains which will allow you to fall back on physical methods to remove crusted on staining material. Some things however are irremovable such as pitting or etching of the glass and chipping and removal of layers, these may look like stains at first glance but upon close inspection it becomes apparent that the glass is indeed damaged physically. In these cases the glassware should take up a reduced work load and be retired from continuous use.

(1) It is this carcinogenic property of this solution of potassium dichromate in sulfuric acid that has caused it to fall out in many labs. Prepared solutions of this in the home lab may have solid CrO_3 precipitated at the bottom as a brick red this is a very strong oxidizing agent capable of igniting ethanol vapors, take care.

(2) For more information on Fenton’s Reagent try http://www.h2o2.com/applications/industrialwastewater/fentonsreagent.

2.2 Plastics

Plastics are good for storage of some chemical reagents. Nearly all plastics can store non-oxidizing/non-dehydrating/non-reducing aqueous solutions. Such as water, hydrochloric acid, dilute sulfuric acid, sodium hydroxide or other basic solutions (This is the preferred storage medium for many bases), and solutions of inorganic salts. There are however many different kind of plastic, often differentiable by the designation somewhere on containers as to their recycling preference. Once you know exactly what kind of plastic you are dealing with you open up new possibilities as to what you can store in it, for example, some plastics become soft and dissolve in acetone, whereas acetone may be purchased in containers made from a different type of plastic, here are the common types seen on the bottom of most containers, at least in America:

Polymer Name and Abbreviations	Generalized Properties
Polyethylene Terephthalate PETE or PET	
High-density Polyethylene HDPE	
Polyvinyl Chloride V or PVC	
Low-density Polyethylene LDPE	
Polypropylene PP	
Polystyrene PS	

2.3 Ceramic

The best use of ceramics often comes in the form of their heat resistance, especially in light of their price. They are somewhat resistant to acids and less so to concentrated bases. They also find use in laboratory equipment, one common item found made of a ceramic composition is the Buckner funnel. In terms of over the counter ceramic items with a use in the lab, one common item available that is suitable for at least limited high temperature use is the flower pot. Some flower pots are high in magnesium oxide or simply known as high magnesia, these are capable of withstanding high temperatures for short periods of time. For example, such pots, with the drainage hole in the bottom are used with a piece of paper over it, and contained within the pot is a charge of thermite powder. Upon ignition the liquid metal drips out of the bottom of the pot where it can fall into molds to cast simple objects.

Flower pots are the cheaper end of the ceramics front. Things can and do get significantly more expensive , ceramic plates are used in high end bullet proof vests and there are innumerable membranes derived from ceramics that find use in electrolysis and reverse osmosis. Ceramic pots though are quite useful and available. Not only is the thermite trick a use for such a vessel, but arc furnaces and high temperature reductions along with exotherms can be done in these cheap available vessels.

1) Nomenclature:

Before discussing various aspects of high temperature furnaces and other equipment, it is helpful to understand how ceramics actually work. Simply put, ceramics are mostly metal oxides, such as aluminum oxide (Al_2O_3), silicon dioxide (SiO_2), calcium oxide (CaO), etc. To specify an oxide of a metal, commonly the –um, –ium, or -on at the end of the element's name is dropped, and replaced with –a. eg; alumina, zirconia, silica, calcia, etc. Ceramics can also be composed of more complex mixtures, such as kaolin, a type of large grained clay, $Al_2O_3 * 2SiO_2 * 2H_2O$. A list of commonly used ceramic materials and their properties will follow soon enough.

2) Acid, Neutral, and Basic compounds:

Ceramic compounds fall into these three broad categories. Where R represents a metal and O represents oxygen, chemicals with the formula R_2O and RO are bases or fluxes (eg calcia), chemicals with the formula R_2O_3 are neutral compounds (eg alumina), and chemicals with the formula RO_2 are acids or glass formers (eg silica). At high temperatures, which depend on the compound in question, fluxes attack acids or glass formers, lower their melting point, and together form a glass. Soda lime glass, for instance, is easily melted in a furnace because of the large amounts of soda and lime, while pure silica is much harder to melt. Neutral compounds do not flux other compounds and are not easily dissolved by fluxes, with some exceptions such as boria (B_2O_3). While glass is great for making household glass items, pyrex, and pottery glazes, in the furnace itself glass is undesirable.

3)Green strength:

Clay particles will adhere to one another when wet and dried, but most particles will not. If clay is used, the ceramic will probably not need another binder, but if pure alumina, for example is used, some sort of binder will also be needed to keep the powder together until it has heated enough to form a ceramic bond.

4)Firing:

Ceramic objects and ceramic bonds are created by high temperature firing of powders. The powders are usually slip cast, pressed, or extruded into the shape needed, and then dried and heated. At high temperatures the mobility of molecules and ions within ceramic objects increases, and eventually gain enough mobility to diffuse across various grains of the ceramic powder, fusing the separate grains into one monolithic object. This is a ceramic bond created by what I will call solid state sintering. As the grains fuse together, they shrink towards one another, decreasing the porosity of the ceramic object, causing it to shrink. Another type of bond that occurs is called liquid state sintering (by me at least). Here, some of the components of the mixture melt into a glass, which envelops the non-melted particles, and begins to dissolve them. Eventually the solution saturates, and sometimes higher melting point crystals form within the solution, also knitting the ceramic together.

2.4 Teflon ®

Teflon®, is the brand name of a polymer produced by Dupont named PolyTetraFluorEthylene or PTFE for short. A Dupont researcher accidentally discovered this compound when he noticed there was no more pressure on his vessel, which contained tetrafluorethylene gas. He found a snow-white condensation product, which proved to have exceptional chemical resistance.

Teflon is the compound of choice for the amateur chemist when he needs a very resistant and yet not extremely expensive material. The only problem with teflon is that it is a thermoplast and thus it weakens and eventually melts/decomposes when heated too much. Compared to usual plastics its heat resistance is far higher, it can be safely employed between -200°C and +250°C. Another noteworthy fact is that Teflon is insoluble in every solvent below 300°C. Teflon should never be exposed to temperatures above 400°C because it will decompose into several fluorocarbon nasties, which can severely damage your health.

Because of the exceptionally strong fluorine-carbon bond, Teflon resists the most aggressive chemicals, including fluorine gas or ozone. The only applications where it can't be employed are those where it comes into contact with very strong reducing agents and molten hydroxides, because of the fluorine content Teflon can act as an oxidizer in special circumstances. These reductants are mainly alkali and to a lesser degree alkali earth metals. The Air Force uses Teflon + Mg flares (although hard to ignite) to distract heat seeking missiles because they burn hotter than an aircraft exhaust, so be warned.

Teflon is OTC available mainly as tape, for sealing pipe joints in plumbing, and as sheet, for baking without the use of grease. Teflon tape (if it's pure, it should be white) is a very good substitute for joint grease because it won't contaminate your distillate, yet it provides good sealing. Teflon tubing is available on the internet and in other places and is a great choice for leading around halogens in their vapor forum. Many baking sheets are also coated in Teflon but are Most teflon black, so it probably is not pure, but it is the material of choice for applications where elevated temperatures are needed. Note that some baking sheets are made out of ICFLON, an unknown propriety compound.

2.5 Refractory Compositions

Refractory compositions possess an even higher degree of heat resistance then any compound mentioned thus far, except some ceramics into which they overlap. For examples of refractory compositions please see the section 8.4 on working with refectories.

2.6 **Metals**

As with all these other reaction vessels, metals have their own notch were they work the best. The actual value of a metal vessel is of course directly related to what metal it is made out of:

Metal	Working Temperature	Chemical Resistance	Additional Properties	Obtained From
Nickel (Ni)	900 C	Very highly resistant to alkali conditions, resistant to non-oxidizing acids	Can be used to handle fluorine or other halogens.	Nickel can be bought in the form of crucibles from chemistry suppliers
Iron (Fe)	1200 C	Iron will dissolve in acids readily, however is it somewhat more resistant to alkalis, it oxidizes easily.	Iron oxide that forms on the surface of objects adheres loosely flaking off and leading to further oxidation.	Iron end caps for plumbing are cheap and readily available. The shiny end caps are galvanized and have a thin layer of zinc plated on them.
Stainless Steel	1000 C	More resistant to acids and bases then iron alone. Less easily oxidized in general.	Can cause hard to determine contamination to reactions due to varying compositions.	Mixing bowls, measuring cups, and other kitchen containers can often be found to be made of stainless steel.
Copper (Cu)	775 C	Somewhat resistant to acids, equally resistant to bases, better then iron, on par if not slightly better then stainless.	Forms soluble highly colored contaminates. Clean before every use due to oxidation by air. Can be used with fluorine or other halogens.	Copper end caps are available for plumbing; they are perfect for amateur experimenting.
Tin (Sn)	250 C	Weak against acids and bases.	Tin forms an oxide coating when exposed to concentrated oxidizing agents that can prevent it from reacting further.	Unknown, tin cans actually only have a insignificant tin coating, therefore they do not convey the properties of tin entirely.
Aluminum (Al)	550 C	Very weak against acids and bases.	Forms a tenacious oxide coating that prevents further oxidation in strong oxidizing conditions such as HNO_3 >75%	Aluminum end caps and pipes are available in larger home improvement stores.
Silver (Ag)	700 C	Strong against acids and bases.	-NA-	Expensive, hard to find vessels made of silver, used for work with hydrazine.
Platinum (Pt)	1200 C	Very resistant to most anything	-NA-	Very, very, expensive, platinum vessels for

				chemistry are hard to come by as well due to these price constraints.

So what's the consensus? If you cannot perform a reaction in glass for one reason or another then you need another choice. Examples of extreme circumstances being, high temperature, reaction mixture attacks glass, or thermal shock might be a problem, metals can be a cheap alternative.

3.0 Lab Techniques (Basic)

This section will cover a number of techniques that can be very useful to most chemists. Refluxing allows for a more complete reaction of reagents by allowing them to react at a temperature very close to or at their boiling points. Distillation is infinitely useful for the separation of volatile reagents from their non-volatile counterparts, or for driving a reaction where the product can easily boil off. Filtering is an essential method of removing solid particulate, this comes in handy when dealing with removing solid catalysts from a reaction mixture, insoluble products, or simply some floating pieces of gunk that come in your reagents bought from hardware stores or your products from nearly any reaction.

Electrolysis is a difficult concept to master but it opens a whole world of possibilities for the manufacture of a number of reagents. Powerful reducing and oxidizing agents are both possible to make though electrolysis, as the process of electrolysis is itself both a powerful reducing and oxidizing agent. Covered in three sections including the rarely covered molten salt electrolysis it is the hope of this text just to give the basic overview to the process, nothing more, as such details would take considerable space.

Shifting gears again this section goes to titrations which are quite useful. Usually used for determining the amount of acid or base present in an unknown they can also be used with some modifications to determine the oxidizing or reducing potential of a solution or even its metal content. Titrations should be preformed with a somewhat expensive piece of equipment called a buret however careful use of a graduated cylinder can give close enough figures for the home chemist. This section continues with tips on heating and cooling and maintaining temperatures during reactions followed by two methods of purification, removing water from gasses, solids, and liquids along with the ever useful recrystalization.

This section rounds out with methods of measuring mass and volume and how useful precise measurements are. All in all these techniques should find use with most chemists who wish to pursue their hobby at home and should hopefully offer a number of alternatives to the professional ways of carrying out these procedures, enough so to allow utilization of these methods in a home environment.

3.1 Refluxing

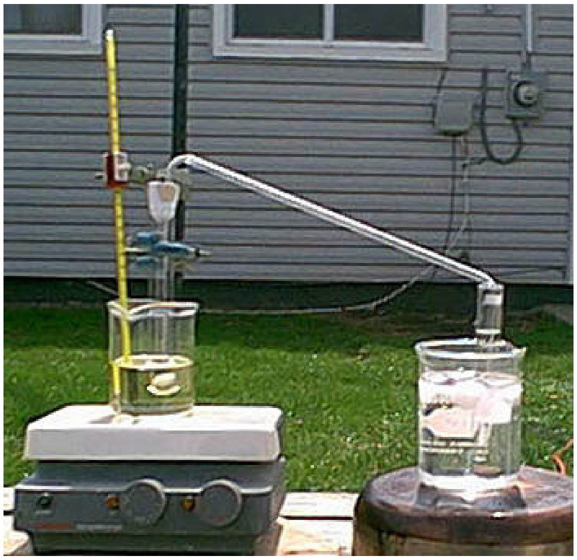
** Before you ever heat up a reaction, you should try to assure yourself that this will not cause the reaction to get out of hand. Always start your reactions cold or at room temperature. If you believe that heat is necessary, heat the reaction after you mix the reagents. **

Refluxing is a common technique, typically used to accelerate reaction rates. The most common method of refluxing a reaction mixture in a flask is to submerge the flask in oil, water, or a sand bath that is at a constant temperature slightly higher than the boiling point of the solvent. Attached to the flask should be a reflux condenser (see section **2.1**) to cool the gaseous solvent when it rises out of the flask. The condenser must be long enough and cool enough (a constant flow of cold tap water is usually sufficient) that the gaseous solvent condenses well below the top of the condenser this is usually apparent, a visual vapor line indicating the height of vapor in the column. This is especially important if long reaction times are necessary, as it will minimize the amount of solvent loss. Always be aware of the temperature of the bath and try to keep it as constant as possible. Monitor your reaction closely to be sure that the gaseous solvent never reaches the top of the condenser!

As the reaction flask heats up some of the liquid(s) will volatize and climb into the reflux condenser. There they intermingle and fall back into the reaction flask. Then once again they boil off and climb into the column and mingle then fall back in, it's somewhat similar to simmering in a culinary process to get the flavors to intermingle. By refluxing for long lengths of time you can react systems with more then one *phase* or react insoluble solids effectively with liquids or simply speed up a reaction. The bubbling helping the mixing of the phases and the heat speeding up the reaction. Refluxing is a very helpful and simple technique that is easy to master and should not be looked upon with apprehension.

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

3.2 Distillation



Distillation is one of the most essential procedures in all of at home chemistry in my honest opinion. When it all comes down to it, distillation is about removing a volatile product from a solution, usually for purification and by the same logic, extraction. The most simple of distillation apparatuses is to the right.

It consists of four essential parts. The tube on the left is the distillation flask (this is also known as the reaction vessel), or in this case the test tube. This leads to a condenser, in this case a glass tube. The condenser serves the essential role of taking the gasses produced from heating the distillation flask and cooling them down, causing you to end up with a liquid. In this case the condenser is the most basic design, air cooled, not very efficient, but made more efficient by a fan blowing on it. Other better condensers utilize running water to cool them or mixtures that can obtain even cooler temperatures.

The condenser tube leads into a receiving flask. This can be cooled as shown in the picture by ice, this is especially necessary if your product that you are distilling over is significantly volatile. The final part of the setup, the one that really makes it work is your heat source. A Corning hot plate is about top of the line for an at home lab but basically anything that gets hot and hopefully has a heating control will work. As usual for heating applications borosilicate is preferred. For a more precise heat control the use of a 'bath' is advised, this is just a beaker full of a fluid (oil in the picture above) that is heated directly, thereby heating your distillation flask more evenly and consistently and if things get out of control, it can act as a heat sink. The bath temperature is usually about 5-10°C higher then the reaction flask temperature as a rule of thumb.

Now, heating a liquid mixture to boiling and condensing the vapor may sound easy, but there are several small but important factors that need to be kept in mind.

First of all, the intent of the distillation is to end up with the most volatile compound of your mixture in the receiving flask and the less volatile compound(s) in the distillation flask. No problem you say, because I am condensing the most volatile component. This is, unfortunately, not entirely true. The other components in your distilling flask also have a vapor pressure, which rises with the temperature. As a result the vapor you are condensing mainly consists of the most volatile compound, but it will also contain a fraction of the less volatile compound(s). This fraction will be large if the boiling points of the compounds don't differ much (less than 40°C) and small when the boiling points are quite different.

For example, distilling wine (usually 12,5 vol% of ethanol in water) will yield a solution which is greatly enriched in ethanol content, but it will still contain a considerable (about 50-40%) amount of water.

Secondly, as your distillation proceeds, the concentration of the most volatile compound decreases and the boiling point of your mixture rises (sic). As a result, your vapor will contain more of the lower boiling compound(s) as the distillation proceeds towards completion.

There are several ways to solve this problem, the most important being *fractional* and *azeotropical* distillation.

Fractional distillations is actually quite simple, the idea is to redistill your distillate until it's pure. Now, if we take our previous example of wine, about three distillations would be needed to attain reasonably pure ethanol. It goes without saying that this is both time and energy consuming. Being the inventive chaps they are, some chemists came up with a clever solution to this problem: The fractionating column. This simple but nifty device allows you to separate close boiling compounds in one run or in considerable less runs than a simple distillation would require.

The fractionating column is placed between the heated flask and your still head with the thermometer. Vapors which pass through it cool down as they rise and eventually condense, the compound(s) with the highest boiling points condense first against the walls and whatever else is filling your column. As a result there are countless condensation cycles taking place in your column. The condensation of the highest boiling compound(s) delivers condensation heat which evaporates the most volatile compound which is running down the wall. As a result the vapor coming out of the top will almost exclusively contain the most volatile component, unless you are distilling an *azeotrope*. One could say that inside the column several successive distillations are taking place simultaneously.

There are several types of fractionating columns and they all have their specific uses. The most commonly used column is the Vigreux column (picture?). It has a relatively small surface area but a high flow rate. The standard 30cm Vigreux column is ideal for separating compounds that have a difference in boiling point of 20°C or more and it can be used for vacuum distillation. Vigreux columns can be made longer or stacked to improve separation, but above a length of one meter one should consider filled columns. Filled columns come in all different sorts and sizes, but they al work on the principle of maximal surface area and therefore they are usually filled with irregularly shaped objects. They retain a lot of liquid and they are also not very well suited for vacuum use. If you are planning to distill compounds with high boiling points or you are using large or long columns you should consider insulating the column to minimize heat loss.

A very important point when performing a fractional distillation is monitoring the temperature, certainly when separating more than two compounds. You'll notice that the temperature suddenly skyrockets when the most volatile compound is depleted from your mixture because of the good separation of your column. Good measuring of temperature can only be achieved by correct positioning of the thermometer, which should be just below the bend towards the cooler, so that it's being immersed in the vapor. (PICTURE!!!!!) Slight deviations of a few mm can cause temperature-reading failures of several degrees centigrade.

Azeotropical distillation could be explained as cheating. The trick here is that a third substance is added to your mixture. This then forms an azeotrope, preferably with the compound you do not need to isolate. An azeotrope is a mixture of two or more substances, which can't be concentrated anymore by distillation because both components have the same vapor pressure at the azeotropic point.

In this case, the azeotrope needs to have a boiling point that differs substantially from the other component in your mixture. Then you can remove the otherwise hard to separate component azeotropically with the third substance. This process is often used during esterification. Toluene is added to the mixture, which forms an azeotrope with water and boils off. After cooling down the water and toluene separate back into two layers, but this is not always the case.

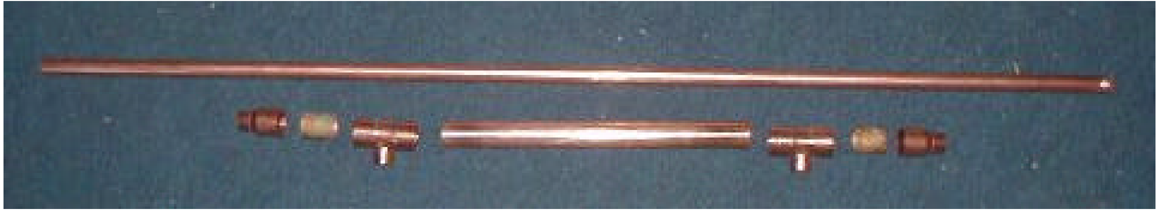
Azeotropes can also complicate a good separation attempt. A good example is nitric acid. Nitric acid forms an azeotrope with water, which contains 69.2% nitric acid. To concentrate further simple distilling won't work and neither does fractional distillation. So what now? The most commonly used method is to break the azeotrope by adding another substance. This is completely the opposite of azeotropical distillation where a substance is added to form an azeotrope, so beware of confusion.

In the case of nitric acid, the most applied method is to add sulfuric acid, as this has such a great affinity towards water it will easily "steal" the water from nitric acid. As a result the nitric acid behaves as if it were all by itself in your flask and thus obtains its' normal boiling point, which is far lower than the hydrated sulfuric acid. One can also snoop the water off by adding a very hygroscopic salt like magnesium nitrate, the only condition being that it does not react with the acid.

The regulation distillation setups used in chemistry labs use ground glassware. The joints are all tapered glass and fit together snugly, with or without the use of a sealant, which can be anything from silicone gel to concentrated sulfuric acid. There are two extreme sizes for the professional distillation setup, the 14/20 sets which would be considered the smaller scale, and the 24/40 sets, considerably larger scale. For example, the largest flask I have seen for a 14/20 set is 250 ml, the largest for a 24/40 is 10 L. The smaller setup has the advantage of taking up less space and using less of the distillate to wet the vessel therefore resulting in a greater yield, it is great for distilling small amounts but the purification of 3.8 L of over the counter paint thinner may be a pain. In contrast a 24/40 setup is perfect for this larger project. But is not good for small amounts, when very small amounts are used the apparatus first needs to be 'wetted' with vapor and as such a majority of your liquid might end up lost on the walls of the apparatus and also the larger setup of course takes up a larger space. It is really up to the chemist and what scale they will be working on to decide. However the 24/40 joints are more readily available online and elsewhere then 14/20. But all of this varies country by country, in Belgium for example the most common joint size is 29/32. Finally in addition to ground glass a time tested method is to use rubber stoppers and glass tubes to connect parts of a distillation apparatus, the drawback being that rubber is attacked by a number of reagents such as oxidizing agents and organic solvents.

Picture 1 A 14/20 distilling apparatus the reaction flask being heated in an oil bath.

The Metal Distillation Apparatus:



Although the materials section goes to great lengths to show that glass is a great all around material in which to perform reactions there is no reason a metal distillation apparatus should not be considered for some undertakings. The most notable advantage of a metal apparatus being the high working temperature and the availability of the materials with which to construct one with. Shown above is a copper condenser in production. Such an apparatus could be used to distil any of a number of solvents for purification purposes. It could also be used for inorganic applications such as distilling sodium, something that would be impossible in normal glassware due to the high working conditions.

Such an apparatus can be simply put together with exoxy and then connected to glassware through rubber stoppers and tubing. Most chemists will limit themselves to the normal glassware distillation apparatus, but metals are more cost effective and they are more durable. Definitely a good investment. Copper is a great choice with which to work, the metal is bendable with the proper tools and it is fairly inert, not to mention most of the connections are readily available as is shown buy the photo above of the distillation apparatus prior to assembly. However distillation apparatus can be made from other metals as well.

This crude apparatus is a condenser fabricated from steel threaded pipes. The connection on the right leads from the distillation vessel and connects to the side of it. The left side actually connects to a jar with the lid attached there to distill into it. And the copper piping around the vessel cools it, as well as additional piping added to simply hold it in place. Crude but effective there are many other ways to prepare distillation apparatuses from metals and over the counter items if one is willing to take the time to make one.

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

1. Check if your compounds form an azeotrope. If not, or if your starting material is below or above the azeotropic concentration, proceed with a normal distillation first. If your compound does form an azeotrope, proceed to step five.
2. Check the difference in boiling points.
3. Check the vapor pressure of the higher boiling component at the boiling point of the lower boiling compound. If this is significant and purity is required, use a column.
4. If you can’t achieve sufficient separation with a column or you don’t have a sufficient column at your disposal, check weather you can use an azeotrope to remove ONE of the components.
5. If your compounds form an azeotrope, which you want to separate, check if

there's something that'll form a stronger azeotrope with it or which has a greater affinity for the compound.

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

Distilling HNO_3 (Nitric Acid) from a mixture of a nitrate salt and sulfuric acid is a time tested way to isolate this useful oxidizing acid. So an adventurous chemist combined an unweighed quantity of $NaNO_3$ (sodium nitrate) into a flask with a large quantity of 94% H_2SO_4 (sulfuric acid) and attached a condenser though which water was run and in turn this ran to a receiving flask. Some boiling stones were also added (pieces of obsidian) to help ease the boiling process and make it smoother. However complications where run into shortly after heat was applied, the black boiling stones started to color the mixture black, and the gas running into the condenser was a dark red, not what would be expected for a clear to off yellow acid. Regardless distillation was continued and in the end the chemist ended up with 20 ml of a dark red volatile distillate. Upon addition to water it decolorized and left an acid solution. Although not obviously apparent to the chemist at the time they had distilled NO_2, a highly toxic gas that is one of the decomposition products of nitric acid. Their ambitiousness and inexperience resulted in them heating the reaction mixture too high and the concentration of their acid only would have allowed 95%+ HNO_3 to make it over, which would have called for a vacuum because high concentration HNO_3 decomposes somewhat more readily then the more common 70% grade. The chemist eventually realized their mistake by simply observing the physical properties of the product ran in contrast to those of the desired product they thought they had. Luckily the chemist made it out unscathed.

Important Safety Concepts:

Heating:

When distilling one should use a safe method of heating, which prevents your glass from cracking and improves its life. It also protects you from a bursting apparatus, which showers you in dangerous chemicals. If heating with a flame try to keep away from that section of your apparatus.

Flames:

Open flames are NOT a safe way of heating. Flames cause localized overheating and are especially a hazard when distilling flammable substances. Flames that are non-diffuse such as flames from a torch can cause extreme stress and failure of glassware, either use flames to heat a bath that your reaction vessel is in, or use a very diffuse flame.

Electric Heating:

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

Baths:

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

SealingJoints:

Sealing your joints is very important. Improperly sealed joints will cause losses but they also pose a safety hazard, because they allow air to enter. This is mainly dangerous when distilling flammable substances or when distilling under vacuum. Joints are commonly sealed with grease. There are several types of grease and they all have their disadvantages. Vaseline is cheap to get and easy to use, but it’s chemical resistance is limited and it will contaminate organic solvents. Other options include silicon oil, but this is expensive and does not provide 100% chemical resistance either, and specialty high vacuum greases composed of higher fluorinated hydrocarbons but they can cost a bundle. Therefore the author recommends cheap, white, teflon tape available from any hardware store to seal pipe joints. This easily fits between the joints and has excellent chemical resistance. Wrap one layer around the ground glass male side and press into the opening opposite, giving a slight twist, do not force or twist too much though or you might crack or otherwise damage your glassware.

• Evaporating to Dryness

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

1. Your intended product will not decompose at the temperatures necessary to volatize the solvent. [Note: Vacuum can decrease the necessary temperature to remove the solvent and prevent decomposition of your product]
2. Any other compounds in your solution besides your intended product are also volatile.
3. Your intended product will not volatize to any major degree at the temperatures used.
4. Your intended product will not explode at the temperatures used/is not *pyrophoric* at these temperatures.
5. An extremely pure product is not required/additional purification will take place.

Examples of simple procedures would be:

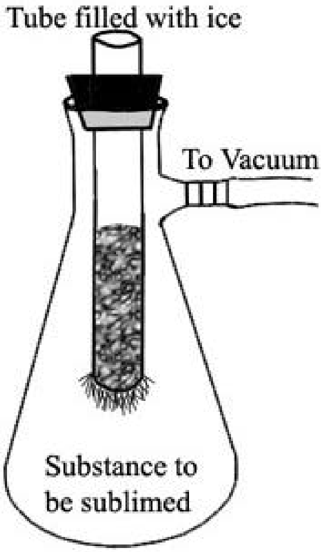
- Making $AgNO_3$ by dissolving silver in HNO_3 and boiling off the HNO_3/Water.
- Neutralizing $BaCO_3$ with HCl then boiling off the water and HCl.

Examples of procedures that will not work are:

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

Things to watch out for consist of azeotrope distillation when involving liquids and carry over of less volatile insolubles. Additionally when a solution has nearly evaporated there may be a heavy precipitate on the bottom, this can cause 'bumping' in the flask which can bounce a flask off a hot plate or even crack it due to the pressure of the vapors rising though the precipitate. To avoid the worst of this you can cool the solution when a precipitate starts to form then filter it then resume heating, or resort to magnetic stirring, or heat at a lower temperature then the boiling point of the liquid. When creating a salt by reacting an acid with a metal/carbonate/hydroxide etc. be sure to use the *stoichiometric* quantity of acid if at all possible, excess acid will only have to boiled off resulting in noxious clouds that kill grass, other plants, your eyes, and lungs (this is assuming the acid is volatile, e.g. H2SO4 will not be easily volatile).

- **Sublimation**



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

Sublimation can be considered a form of short path distillation and can be done providing two criteria are met. 1) The substance to be sublimed must have a high vapor pressure. 2) The substance from which the product is to be sublimed must have a relatively low vapor pressure. Sublimation at room temperature is usually slow, but as stated above, applying vacuum and heating increase the vapor pressure of substances and allow more of the molecules to escape in the gas phase and therefore allow more of them to recondense on the conveniently placed cooler areas of a vessel. The picture shown here is just one way to setup a sublimation apparatus using an Erlenmeyer flask with a side arm to which vacuum is applied. The crystals cooling on the test tube inserted into the rubber stopper and full of ice. When the sublimation is complete the stopper is removed and the crystals are scraped off. Other simpler apparatuses, can easily be improvised just knowing the normal parts of sublimation, basically you need a partially enclosed environment, a cooler part of a vessel, and a warmer part (a simple example being a jar with a bowl sitting in the opening full of ice, with mild heat applied to the bottom). But even at room temperature sublimation will occur as stated, in which case crystals will usually sublime to the top of a test tube.

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

3.3 Filtering

So, you have a mixture of a liquid and a solid that you want to separate. Filtration is the answer. It's easy, fast, and effective. The only things you really need are a funnel and a piece of filter paper (a coffee filter will work too, or even a wad of cotton). To determine which type of filtration is best, you need to know whether you want to keep the solid or the liquid. There are three common types of funnels that are useful to a chemist: liquids, powder, Buckner. A liquid funnel has a long, narrow spout and is usually best for simple filtrations, using a cotton wad or a properly folded filter paper or coffee filter. However because the spout and subsequent area that drains into the spout is so small it is only good for filtering a tiny amount of precipitate otherwise it may become too packed to filter, even with vacuum filtration. Powder funnels have a much wider spout and are useful for filtering things that might clog a smaller spout funnel. Buckner funnels are the best for isolating solids, but they work best with a vacuum (more on that below).

You may be wondering, what good is filtration anyways? I can always *decant* my mixture can't I? Well, yes you can, but there are some inherent problems associated with decanting. 1) If the solid material left at the bottom of your flask was your goal, what you have left will contain appreciable reaction mixture, and some of your solid will probably be lost during the actual decanting process. Or 2) If the liquid is your goal appreciable liquid will be left in your solid that could otherwise be obtained by filtration, and also small particulate may come over during the actual decanting process ruining the purity of your new reagent or the solid may not totally settle out. Filtration is almost always preferable to decanting. Also before selecting a filtering method one should consider a few things:

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

Number one is important because gravity filtration is not a good method to filter large quantities of liquid, both due to the time involved and the possibility of clogging of filter paper, in this case vacuum filtration is more appealing.

2. Am I trying to recover my liquid component?

If you are trying to recover your liquid component and not your solid component, sand or diatomaceous earth could be put onto the filter paper to increase the efficiency of the filtration and speed it up.

3. Am I trying to recover my solid component?

If the solid component is your goal then special care should be taken to make sure your solid is recoverable in high quantity from the filtration method of your choice.

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

The possibility of crystal masses clogging filter paper is exceptionally high in the case of really fine precipitates, which can cause a complete stop to filtration, in addition large quantities of crystals can do the same or overflow my filtration method.

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

If there are ultra fine particulates in the filtrate there are very fine filters that could be used to remove them. Letting the solution rest for a few days can 'age' the precipitate resulting in a more manageable solution or filtering though sand or diatomaceous earth or even a fine sintered glass filter can remove many fine particles.

6. Do I have to filter it while hot?

If you have to filter a solution when hot the first addition of the solution to the filter paper can result in crystallization on the filter paper resulting in almost no filtration ability, which can be catastrophic in some situations where a very hot solution is teetering around. Pre-washing the filter paper with hot solvent or heating the glass/porcelain parts of the apparatus in an oven can help here.

7. Does my substance to be filtered contain components that may not take nicely to filter paper?

Finally, my last point to consider, how will the actual substance passing though the filter paper affect it? Most of the time it won't affect it greatly, but some copper complexes can attack cellulose, as can strong sodium hydroxide solutions or strong oxidizing solutions like concentrated nitric acid, passing these though a normal filter paper could spell doom for the reaction and will at the least call for you to filter again, for oxidizing agents try filtering with glass wool(1).

So how do I filter my substance? Well that depends on what type of filtration you want to take advantage of (see below). But the normal procedure is to slowly decant your liquid into the funnel without adding the solid at the bottom. In this way the filtration can proceed smoothly, because the solid particles will slow it greatly. The liquid mix is added in small increments to your filtration method, if you are taking advantage of the insolubility of a salt yields may be increased by cooling the mixture until it is near the freezing point to depress the solubility. After each addition you should wait until the addition is nearly though the filter paper. Then add more, keeping up the process till all of your solution is used. Once only a small amount of liquid is left in your beaker swirl around the liquid and dump it in all at once to get your solid onto the filter paper.

When all of your liquid has been added the, solid left in the filter paper is washed with cold solvent (whatever your solution is) is added to the flask you were filtering from and swirled around to remove any solid left and this is added to the filter cake to wash it and add this extra little bit of solid to the batch. The cake can then be washed with additional aliquots of solvent. After your solid is thoroughly washed it can be removed from the filtration apparatus and spread out on filter paper or in watch glasses to dry. The basic components of a filtration system are a membrane to separate the filtrate from the non-filtered solution, and a place for each to go. There is room for emergency improvisation here, the author of this text has for instance seen a beaker with a coffee filter over it held in pace with a rubber band inverted over a collection vessel and heated externally with a torch, the increasing vapor pressure in the flask forcing the liquid though the filter. Although this is not a recommended method it just goes to show filtration is a mechanical process and can easily be modified to be accessible in your situation.

(1) In the case of filtering oxidizing solutions you can give glass wool a try, or alternatively fiberglass insulation, this first be cleaned by stirring with hydrochloric acid and drying, if your product however looks bad you may want to different source for your glass wool substitute.

3.3a Selection of filter paper

First and foremost: size. The filter paper you use **must** fit your funnel or bad things will happen. The filter paper should fit entirely within the sides of the funnel. For a Buchner funnel, the filter paper should cover all of the holes on the flat portion, but the edges of the filter paper should not touch the vertical sides of the funnel (it won't seal under vacuum otherwise). If you don't want to buy all kinds of different sizes of filter paper, remember that a big piece can become a small piece, but not vice versa.

The type of filter paper you use really depends on the size of the solid material you are filtering away. For most applications coffee filters are sufficient, but coffee filters are relatively thin and can't take much abuse. If you can get real filter paper, do it (The stuff from Whatman is the best, and not all that expensive). For larger crystals a wad of cotton packed into a liquid funnel works very well. If you have a Buchner funnel, you need a flat piece of filter paper so coffee filters are no good unless you cut them up to fit your Buchner. For filtration of very fine particles, like charcoal dust, you can find very high efficiency filter paper, but the cost is rather high. If you really need it, the best stuff is made from Teflon (a slightly cheaper alternative is a nylon filter paper but this is still quite expensive and a special commodity) and can be found through chemical suppliers or chromatography suppliers. Using Teflon filter paper requires vacuum filtration. You can sometimes sidestep the need for Teflon filters by using a "filter aid" such as diatomaceous earth, sold by the trade name "Celite." Celite is essentially very, very fine dirt, but it really works well! (See the vacuum filtration section for details on its use). As a final note, all filter paper, no matter how expensive, is really only good for one use. It's not worth ruining an experiment to save a few cents by reusing a piece of filter paper.

3.3b Gravity Filtration

Gravity filtration is slower than vacuum filtration, but requires less equipment and is generally more effective. Gravity filtration is better for isolating the liquid phase than the solid phase, but if you don't have a vacuum, it will still work reasonably well for isolating the solids. To perform a gravity filtration, you need a funnel (liquid funnel is best, but a powder funnel is okay too so long as you use filter paper and not cotton), filter paper, and a flask to collect the liquid. There are many ways to fold a flat piece of filter paper in to a cone shape. The best way is called "fluting" and consists of folds in opposite directions along the diameter of the filter paper (FIGURE). A simpler, although somewhat less effective method, is to fold the circle in half and then fold the semi-circle in half again. This wedge can be opened to form a cone, such that half of the cone has a one-ply layer of paper and the other half has a 3-ply layer. Fluting the filter paper will give a significantly faster flow rate! Fold the filter paper whichever way you choose and then place it inside the funnel. Clamp your receiving flask in place (important!) and then set the funnel on top. Slowly pour the mixture into the filter paper cone. The liquid level should never be higher than the top edge of the filter paper or the mixture will spill over and go through unfiltered.

Gravity filtration is relatively slow (remember, fluted filter paper has a faster flow rate, it's worth the extra folds!) so just add enough of your mixture to get close to the top and then let all of the liquid flow through before adding more. This may seem unnecessarily slow, but if you slip and add too much of the mixture and some gets through unfiltered, you're just going to have to start over again. Once you've poured in all of your mixture, rinse your reaction vessel with the solvent and then rinse the solids thoroughly. Let the solvent drain completely before adding more to rinse. If the solids are slightly soluble in the solvent, be sure to use ice cold solvent and try to use as little as possible. Gravity filtration cannot be used reliably for solids that precipitate as 'gels' such as iron or aluminum hydroxide, these are very difficult to remove water from and easily clog filter papers if not assisted by vacuum. Also note that filtering organic liquids of low density or just in general through filter paper can prove difficult without vacuum as their low density gives little incentive to pull them through the filter paper and in addition filter paper contains water as papers are hydrophilic and as such they can 'intimidate' organic liquids from readily passing through them.

3.3c Vacuum Filtration

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

Vacuum filtration is the fastest way to filter a mixture, but it's not always the most effective. If you want to isolate the solid material, this is the best way to go. You will need a Buckner funnel, a piece of filter paper, a rubber sleeve for the funnel, an Erlenmeyer flask with a vacuum sidearm, and a vacuum pump. First, put the rubber sleeve around the spout of the Buchner funnel. The sleeve should fit snuggly and be larger than the opening at the top of the Erlenmeyer (we want it air-tight, remember). Clamp your Erlenmeyer in place (important!) and then set the Funnel on top of the Erlenmeyer and drop in the filter paper. Wet the filter paper with a little bit of the solvent from your reaction (this will help the vacuum seal). Connect the hose from your vacuum to the tubing adapter on the Erlenmeyer. Turn on the vacuum and then slowly pour the mixture onto the filter paper. The solvent should be sucked through quickly. Try to keep the filter paper moist since it won't seal when it's dry and the solid can be sucked through around the edges of the paper. Once all your mixture has been poured onto the filter rinse your reaction vessel with solvent a couple of times and then rinse the solid a few times with a little solvent (if the solid is slightly soluble in the solvent, be sure to use ice cold solvent and try to use as little as possible). Pull air through the solid for a minute to dry the solid. Then turn off the vacuum and you're all set.

For filtration with Celite, set up the filtration apparatus as described above, then add an even layer of Celite 2-3 cm thick over the top of the filter paper. Turn on the vacuum for a few seconds; the layer of Celite should compress a little. Wet the Celite with solvent and then turn the vacuum on again briefly. Repeat the process of adding a little solvent to keep the Celite wet and then turning on the vacuum to pack it down. This process is essential to separating very fine dust from your mixture. When you are satisfied with the packing of the Celite, turn on the vacuum and slowly pour your mixture onto the filter. Try not to disturb the Celite too much, you want it to stay as even as possible. Next rinse the reaction vessel and the Celite. I repeat, rinse the Celite. A lot. There is a lot more to rinse than in a regular filtration. My favorite method for this is to turn off the vacuum, add a volume of solvent that is roughly the same as the volume of Celite (the solvent shouldn't go through the Celite filter with the vacuum off) and then suck it through by turning on the vacuum. I usually repeat this process 2 or 3 times. Finally, if you are using Celite as a filter aid, it is all but impossible to recover your solid material, so be aware!

3.4 Electrolysis

An amateur chemist can easily define electrolysis as any reaction that calls for the direct application of an electric current to a chemical, either on its own, or in solution, for purposes other then heating. Our point here is to understand how we can transform chemicals using electricity. The key word is "ions". What is an ion? A quick and dirty definition would be: "It's an electrically charged atom or molecule". The positively charged are called "cations" and the negatively charged are called "anions". Ions have different chemical and physical properties than the original atom or molecule. All anions have their own name, so Br^- is baptized Bromide and NO^{3-} is Nitrate. Cations with more than one possible charge also have names, so Cu^+ has one electrons missing and it's baptized Cuprous, Cu^{++} has lost two of them and becomes Cupric. [See table in section **1.3** for a list of common cations and anions]

Many chemicals, including all salts, are made of opposite charged ions "glued" together by electrical forces. And in these 'ionic compounds' electrolysis can help unglue them. There are basically three schools of electrolysis that you should familiarize yourself with.

Pure Compound / Molten Salt Electrolysis	This is the simplest form of electrolysis. Not in its practice, but in concept. A pure compound. ionic in nature, i.e., consisting of a cation and anion, is heated until it becomes liquid. Ionic liquids are good conductors of electricity and therefore are able to be electrolyzed directly. Once molten, electricity is applied and the compound breaks down into its constituent parts. For example, molten sodium chloride when subjected to DC electrolysis will break down into liquid sodium metal and chlorine gas.
Aqueous Electrolysis	The most common form of electrolysis for the at home chemist. An ionic salt is dissolved in water and a current applied. Depending on the reduction and oxidation potentials of your cations and anions you get different products. For example, electrolysis of water, with a little salt added to aid in conductivity will yield hydrogen and oxygen gas, excess salt and a higher current will yield hydrogen and chlorine gas.
Non-Aqueous Electrolysis	A compound is dissolved in any liquid other then water and current applied. Different products are possible under different conditions. Products are possible with non-aqueous electrolysis that are impossible in aqueous electrolysis or would require high temperatures for molten salt electrolysis. For example, it is possible to obtain lithium metal as a deposit in the electrolysis of lithium chloride in pyridine, whereas lithium metal would react instantly in water and molten salt electrolysis would require temperatures of several hundred degrees

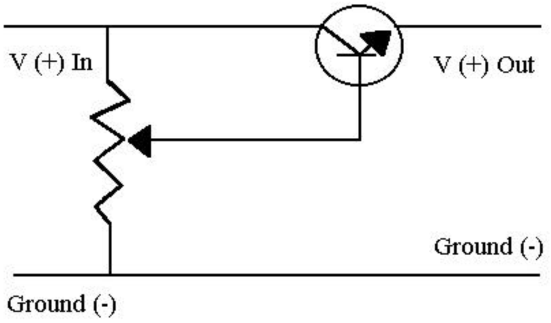
	Celsius.

There are two different types of electricity available. There is alternating current (AC) and direct current (DC). Alternating current is the type of electricity that comes out of the wall, this is not good for electrolysis, alternating current changes which side of your cell is your cathode and which is your anode about 60 times a second, this means that almost nothing can be accomplished with it(1). If you have a cell full of water and something to make it conductive and put two electrodes into it, plugging it into the wall the only thing you will do is heat your solution to boiling with resistance heating, make a random explosive gas mixture above your water, and deform your electrodes. Therefore your only real choice for productive electrolysis is DC.

So where does DC come from? I will not detail the electronics here. There is plenty of information elsewhere on the internet. But you usually have two sources, converting the output of your wall adaptor to DC, or using batteries.

- **Batteries:** Not very useful, except for simple demonstrations. Those little square 9V batteries make me laugh! If you insist on using batteries, be a man, use 4 “C” size (big) batteries or a lantern battery or even a car battery, but if you go with the car battery then you’ll need a charger, and you could just use that directly anyways.
- **Adapters:** These are very common these days and most households have one or two spare ones from old equipments that broke. 6 to 9 volts is fine for most experiments and they usually deliver above 0.5A. Another common source of power that falls into this category packing a little more punch is the car battery charger. A good one can supply from 0 – 12 V and from 0 – 55 A, and can be procured cheaply from second-hand stores. Old ATX power supplies from computers are marvellous for electrolysis because they yield high amperages (up to 20A) at low voltages (5 and 12V).
- **Build your own simple power supply:** If you have some skills on electronics, you can built your own power supply just using a transformer and a single 1N4001 diode.

Build your own variable power supply: An extra potentiometer (100Kohms) and a power transistor like TIP 31 (3A, 40W), TIP 41 (6A, 60W)or TIP3055 (15A, 90W; a.k.a. 2N3055), can give you control over the voltage supplied by the batteries, the adapters or your home built power supply. Don’t expect precision or stability though:



A power source capable of delivering at least 0.5 ampere would be nice. The product yield per hour depends on the current, so your current should be proportional to your hurry and your electrode surface area (too much current per square centimeter may cause unwanted results).

The electrolysis process is sensitive to the voltage applied, but if you want a "rule of thumb" number, 9V will perform most tricks.

An exercise in calculating yields involving electrolysis.

Let's say that you want to make bromine, and just to simplify things let's say you have some lead (II) bromide laying around to perform molten salt electrolysis on.

First off you divide your reactions into half reactions, the half reactions, when added together cancel out but separately they give the number of electrons necessary and help in visualization.

$Pb^{2+} + 2e^{-} \Rightarrow Pb_{(l)}$

$2Br^{-} \Rightarrow Br_{2(g)} + 2e^{-}$

Notice how the number of electrons on one side of the equation match the number of electrons on the other side of the other equation. That is because while one thing is reduced (gains electrons) another thing must be oxidized (loses electrons) a good way to remember this is the mnemonic OILRIG [Oxidation Involves Loss, Oxidation Involves Gain (of electrons)]. Couple this with the mnemonic RedCat [Reduction occurs at the cathode] and you can figure out where your products will be produced at. When wires are color coated in their normal manner the cathode is the black wire, the negative (-) wire. By default then the red wire is the positive (+) wire, is the anode.

Now that you know your reaction, what kind of power source are you using? Maybe you're using a simple wall adaptor, possibly from a phone charger and the phone broke. Looking on the plastic you might even find that it is 6.2 V and .5 A. That's all the information you need off that. The next step is to calculate the number of coulombs (C

) of electricity that pass though the molten mass. A coulomb is a unit of measurement specific to things involving electricity, it is equal to one amp times one second. So it actually measure the quantity of charge moving though the cell. So let's say that we are going to run this for 30 minutes:

$.5A \cdot .5h \cdot (3600s/1h) \cdot (1C/1A{\cdot}s) = 900\ C$

To explain the above equation you can see the that .5A came from the power of the power supply, the .5 hours came from the time the cell was running, the other numbers are conversions to the number of seconds in an our and the number of amp-seconds in a coulomb. This is the generic representation and you can just plug in your numbers in the above equation to get your own unique answers. Now we see from the two equations way overhead that for every one mol of Br_2 generated two mols of electrons are simultaneously brought into existence. Now we need a new unit of measurement for electrolysis, a Faraday (F). A Faraday is the number of electrons necessary to reduce one mol of a single charge unit. A Faraday is equal to 96,500 C. From here we use a new equation:

$g.\ Br_2 = 900\ C \cdot (1\ F / 96{,}500\ C) \cdot (1\ Mol\ Br_2 / 2F) \cdot (159.8\ g\ Br_2 / 1\ Mol\ Br_2) = 1.5\ g\ Br_2$

In the above equation we took the number of coulombs that we got from the first equation and multiplied it by the conversion for Faradays to coulombs and that by the mols of Br_2 and the number of Faradays involved, e.g., in this case two mols of electrons are involved and therefore there are 2 F. This is all multiplied by the grams of Br_2 per mol to give 1.5 g of Br_2 produced overall. To get lead you could convert the grams of Br_2 produced and make it into mols, then you could multiply that by the grams per mol of lead or just substitute that information into the last part of the above equation.

Well, most of you are thinking, "Only 1.5 g…. what the heck, I wanted a liter!" Well, this is not the setup you would use for massive Br_2 production. But electrolysis is good for producing small quantities of hard to obtain chemicals, and you could increase your yields by either increasing the amps of your power supply or by running the setup longer.

Aside from a power supply the second most important consideration are the electrodes. Many of the metals are similarly conductive for all intents and purposes and therefore it is better to consider them in terms of their chemical resistance relating directly to whatever environment you are planning to perform electrolysis in and the ease of procurement of the electrode material.

- **Copper** can be easily obtained from common wires. Copper has a wide resistance to many environments.

- **Zinc** can be found as the outer metallic shell of common batteries (the cheap ones, called carbon-zinc). Not good for acidic environments or basic environments.
- **Carbon or graphite:** These are very useful electrodes, since they do not oxidize as anodes. Well, they don't last forever; they are attacked by oxygen, originating CO_2, or maybe just disaggregate in the solution, but are much, much, cheaper than platinum electrodes, so they are widely used. The most common are pencil's graphite. These have very different compositions, and may or may not last long. In fact, they may even be very bad conductors. Another source of carbon electrodes is the carbon rod that every carbon-zinc battery has inside. They are better than pencil's graphite. The best carbon electrode I found is a rod of graphite covered with a copper layer found in solder's supply shop. When you strip the copper with ferric chloride (or electrolysis), a resistant graphite electrode is left. Another option is graphite from electric motors sliding contacts. They are small, but easy to find and quite resistant. I have a couple from large polisher that are 23x16x6 mm.
- **Lead** is used often as an inert electrode. You will probably find it in a sporting goods shop: fishing weights, gunshots in general, airgun bullets etc(3). It melts easy and you can cast your electrodes melting it with a blowtorch and a scoop.
- **Nickel:** Coins from some countries contain an appreciable amounts of nickel(2) and can be used as electrodes, additionally nickel can be procured from scrap yards or specifically for electrolysis. Nickel is an excellent material for electrolysis of highly basic solutions.
- **Iron:** Iron is attacked readily under acidic conditions and somewhat slower under basic, but it is commonly available and may find some use in a pinch.
- **Platinum:** If you have the money, it's the most resistant anode I know. Fine wire is ok, but for larger surfaces, other metals plated with platinum can be used.
- **Silver/Gold:** A cheaper alternative to platinum somewhat more reactive. Available from coin suppliers in the form of collectable currency either can be melted and cast into the appreciable shapes desired. Silver wire can be purchased from jewelers as can platinum.
- **Misc. Electrodes:** Mercury(4), lead dioxide(5), rare earth oxides plated on titanium, tantalum, there are hundreds of possible electrodes that one may come across that are not covered here.

(1) AC electrolysis is really a good way to fry electrodes. Nothing will survive AC electrolysis of hydrochloric acid, platinum will succumb to this, which is a good way to prepare soluble platinum compounds ironically. Even carbon destroyed.

(2) Older American nickels contain decent amounts of nickel but older Canadian coins contain an even higher amount. amount of nickel in coins in your country can usually be found easily by searching online.

(3) Lately there has been a push in some areas to replace these lead items with the more environmentally friendly bism this time though products that have been replaced with bismuth usually proudly proclaim it as being environme friendly so determining what you have is still relatively easy, lead in these items is also usually alloyed to a small p with other elements such as antimony.

(4) Mercury electrodes are famous in the mercury cathode cell for the preparation of sodium hydroxide. In this ce mercury electrode forces the reduction of sodium cations to sodium metal rather then the production of hydrogen, the is a mercury-sodium amalgam, containing a few percent sodium metal, this reacts with water slow enough to allow re of it, to look further at this phenomenon search 'over voltage' online.

(5) Lead dioxide electrodes must be formed carefully, they have their claim to fame in the preparation of perchlora
electrolysis of chlorates by the armature chemist.

3.4a Molten Salt Electrolysis

Performing electrolysis on a molten binary salt usually yields predictable products providing you have a simple anion coupled to your metal ion. Another advantage is that electrochemistry can produce many elements that would not be possible to produce under aqueous conditions. The most common examples including producing the alkali and alkali earths electrochemically. Of course the most prohibitive feature of molten salt electrolysis is the high temperatures usually employed. The corrosiveness of molten salts and some of the products produced by the electrolysis, especially at the temperatures employed these factors usually result in molten salt electrolysis being beyond the reach of the beginning chemist. However, many chemists would like to do molten salt electrolysis, and they can, the procedures outlined within this section will cover the basic points for this method of electrochemical production.

Working with Molten Salts

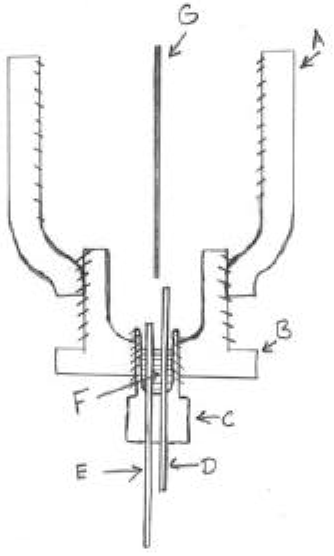
Great care must be exercised when working with molten salts. For the most part this is due to their high melting points. Think about what happens when drop of water hits hot grease, the spattering and violence of the sudden evaporation of the water, now consider molten salts can be several hundred degrees Celsius hotter. Water hits these salts like a bullet, sending liquid everywhere. Due to the temperatures it can greatly dehydrate anything organic and it can also start fires on wood and paper and such. It should also be noted that molten ionic salts are good solvents for…. Whatever, they attack a multitude of things and some they flat out destroy. Specific examples like molten sodium hydroxide (above) destroy wood and such on their own, let alone at high temperatures, getting a quantity of this on your skin would be disastrous. When working with molten salts your apparatus should be firmly placed on a flat surface, and once the salt is molten it should not be moved and you should limit how much you 'mess with it' as many mistakes can occur. Wear welding gloves that can easily be removed if you get your salt on them and avoid inhaling the fumes coming off the melt too much, they can cause sickness and throat pain and loss of vision (as I once experienced). Finally, when you are done with a reaction, allow the salt to cool to room teperature on its own, in case of emergency the salt can usually be dumped in sand.

Nearly everything in your electrolysis cell will have to be metal due to the temperatures employed, although less used, ceramics may also be employed for some applications. Some lower temperature cells may safely employ Teflon (not for use with molten alkali's) or even some epoxies or cements. The type of cell construction depends entirely upon the types of products produced. The physical state of the products and their inherent reactivity after genesis is of the utmost importance and of equal related importance is weather the products thus formed will react with each other or even with the melt. So here is your quick checklist:

1. What is the best melt, and *eutectic* composition or a straight composition and at what temperature does it run?
2. What products will be formed through electrolysis, if an eutectic mixture, will only one product be formed at the anode and one at the cathode? Are you sure of both your anode and cathode products (check their potentials against charts(1)).
3. At the temperature employed, what will be the physical state of the products formed?
4. If the products are solid then they will not mingle, however if they are either liquid or gas, will they react back with each other and thus make it necessary to divide the cell or otherwise separate the products from one another?
5. Will the products formed react with the melt(2), and if so, at what speed will the reaction occur?
6. What corrosive properties might your melt have, testing though melting small portions of it on a small scale is usually necessary before making a full size vessel, if the metal can hold up to the melt for over an hour without pitting or significant loss of weight it is usually good, but products formed from electrolysis may still attack the vessel.
7. How is the cell going to be heated to the melting point? Flames involve additional precautions when working with cells that produce flammable gasses but are not out of the question. Resistance heating, that is the heat generated though the electrolysis of the melt can supply a large quantity of heat and may maintain your cells fluidity although the melt must still be initially melted, optimally an embedded heating coil with a variable power setting will keep the cell temperature exactly where you want it at.

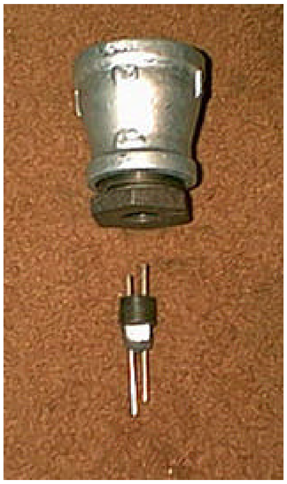
Assuming you have two products that are going to be produced that are going to react back with each other (which is usually the case) there are several cell designs from which to choose depending on the physical form of the products and the density of liquid products compared to the melt. The ultimate variable however in any electrolysis procedure is the electrodes. The surface area of an electrode exposed to the melt, their distance from one another, and the amount of current flowing though them more often then not makes or breaks an electrolysis reaction. Unless one finds highly accurate detailed sources for performing electrolysis on a molten salt they will not have the electrode distances or current densities. And even if you do happen to find them, they usually employ apertures that are beyond what many normal people are capable of producing. The only advice the author can give you on this subject is to try different things and experiment, that is what this is all about anyways, keep a journal so you can notice trends, cells such as the first one detailed pose the extra difficulty of the electrodes being stationary, the misplacement of an electrode in this circumstance is a tragedy, requiring shutting down the cell and possible part replacement, experiment with variable electrode cells before switching to the fixed electrodes for this reason.

Sample Design #1 (In Theory)



A : This is the body of the cell, it is a reducing adaptor for plumbing, this one goes from 2 inches at the top to 1.5 inches at the bottom, both the top and the bottom are threaded. **B** : This is a plumbing piece called a brushing, it reduces from 1.5 to .5 inches, the bottom hole being threaded. **C** : This a plug, .5 inches in diameter that fits the hole in the brushing, these three pieces comprise the containment body of the cell. **D** : This is the anode, it is inserted into a hole drilled into the plug (*C*) and does not touch the plug, thus the hole is somewhat large, if it touched the plug it would short out the cell and perform electrolysis elsewhere. **E** : This is the cathode, it is inserted in the same way as the anode (*D*) the reason for it being shorter then the anode (see picture) is that in being shorter it has a higher current density, the cathode is where reduction happens, it's where metals are formed, and just as a general rule of thumb higher current density gives better yields of metals. **F** : This a material that the hollow part of the plug was filled with, to prevent the molten contents of the cell from leaking out, to hold the electrodes in place, and to prevent them from touching the base of the plug. It can be any material inert to the cell, high heat resistant epoxy, fireplace mortar, concrete. Just be sure to position the electrodes so they don't touch the metal of the cell before setting them permanently in this design. **G** : This a piece of metal that sets loosely inside the cell, it is rectangular with the sides of the metal touching the sides of the cell, but it does not extend to pass between the electrodes, if it did this it would short out the cell. By being here it separates the products formed.

(In Practice)



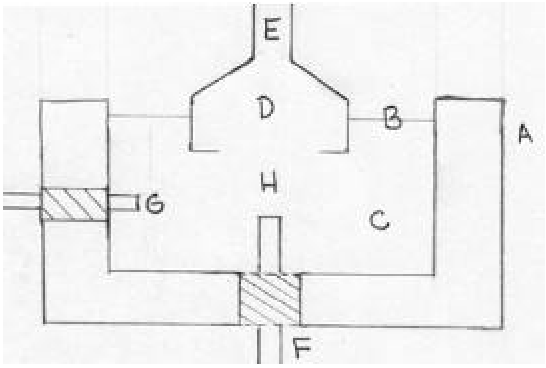
Removable Plug shown Removed

Overview of cell with Divider in Place w/ plug

Additional additions to cell design: What this cell lacks is a heating mechanism. Flame heating is somewhat out of the question unless you have multiple heat sources, because you can't heat from beneath and hot spots in an electrolysis melt are usually undesirable. Wrapping the cell in an electric heating coil from a stove top coil range then embedding it within some refectory is reasonable for supplying most temperatures up to 500 °C. Initially however it may be necessary to heat a quantity of your electrolyte separately over a flame to liquefy it, then add it to a preheated cell with the current ready to run, immediately upon the liquid entering the cell it will want to solidify unless the temperature is sufficient, however by the molten salt coming into contact with instantaneous electrolysis it should hopefully keep it molten(3), from there the cell can be fed periodically with solid electrolyte until the desired level has been reached.

What is this cell good for? : For one the products produced by electrolysis must be lighter then the melt (which is usually the case) and they must be liquids or gasses. Plus they must not react substantially with the melt. If the electrodes in the bottom are sufficiently distant from one another then products that would normally react with each other can possibly be obtained. Upon their formation they rise though the melt and before they have a chance to move around much the metal divider prevents them from crossing over to the other side. In this way efficient separation at the top of the cell is possible. For example, if sodium chloride and calcium chloride are electrolyzed in this cell sodium metal will rise to the top above the cathode and chlorine gas would rise to the top above the anode. The gas would require special handling, such as copper metal piping and such, and have to be taken care of with special precautions. On the other side of the divider, liquid sodium metal would pool and be able to be ladled off.

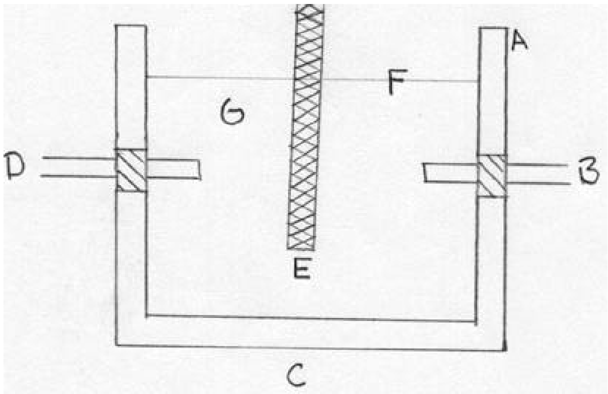
Sample Design #2



Stripped down version of the Downs cell for sodium production from sodium chloride.

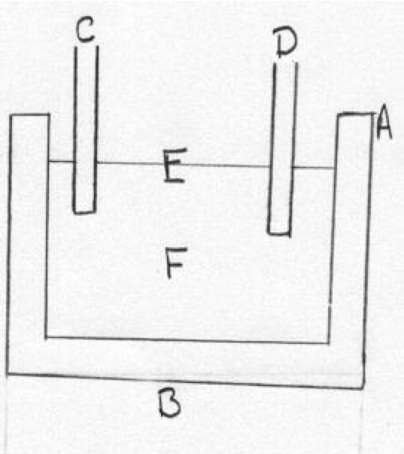
A : Cell body inert or resistant to high temperatures and molten electrolyte. **B** : Electrolyte level in cell, rises above inverted funnel (D). **C** : Molten electrolyte. **D** : Inverted funnel located below the electrolyte level (B) and directly above the anode (F) to collect gaseous products (H) and move them away from the cell to deal with them separately and to prevent them from reacting with the cathode products. Or it may be the intended product such as in a fluorine cell. **E** : Pipe leading away gasses from anode. **F** : Anode, inserted though bottom of cell and carefully positioned to avoid touching the metal lining of the cell itself, held in place with a resistant non-conductive material such as cement, some epoxies, silicates, mortar, etc. (In the Downs cell(4) this would be where the chlorine gas was produced.) **G** : Cathode (In the Downs cell this is where the sodium would be produced, there would be an inverted funnel above this too where the sodium could be drawn off during the electrolysis) the electrode is held in place same as the anode.

Sample Design #3



A : Cell body, resistant to heat and electrolyte. **B** : Anode or Cathode held in place with a resistant material and set so as to prevent it from coming into contact with the metallic cell body. **C** : Direct heat such as a hot plate or torch can be more easily applied to this cell design. **D** : Anode or Cathode held in place with a resistant material and set so as to prevent it from coming into contact with the metallic cell body. **E** : Wire gauze cell divider. **F** : Electrolyte level. **G** : Electrolyte

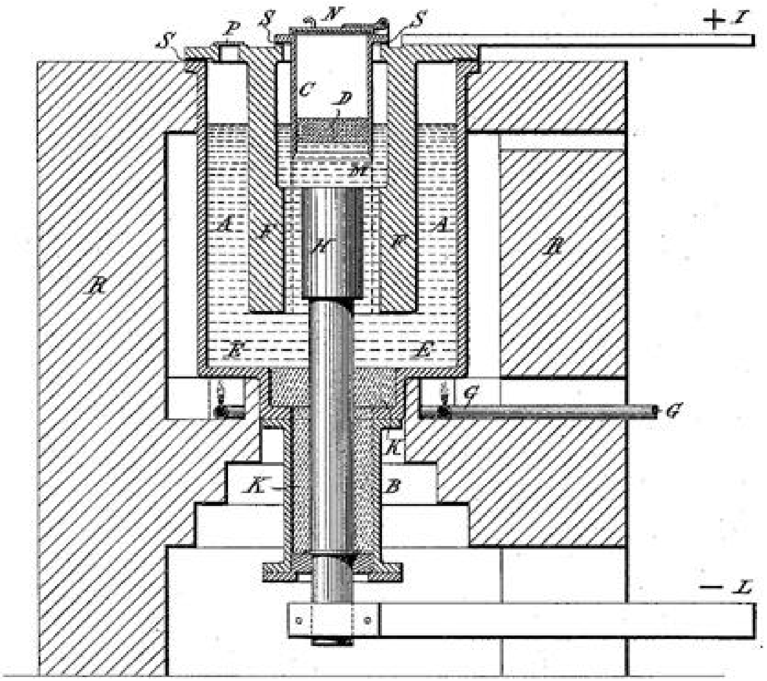
Sample Design #4



The most basic electrolysis cell.

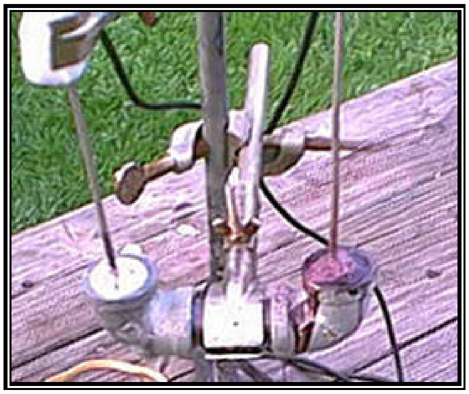
A : Cell body, heat resistant and resistant to the molten electrolyte. **B** : Direct heat can be applied from below in this cell. **C** : Anode or Cathode, suspended above the cell by some mechanism and dipping into the melt a variable amount. **D** : Anode or Cathode, suspended above the cell by some mechanism and dipping into the melt a variable amount. **E** : Electrolyte level. **F** : Molten Electrolyte.

Classic Cell Example:



The Famous Castner Cell (Patent No. 452,030 May 12, 1891)

This cell runs with straight sodium hydroxide, originally run in an iron pot (**S**), and heated by a ring of flames (**G**), set in brickwork (**R**). The sodium hydroxide (**A**), is melted and kept about 20 C above the m.p. (318C) of sodium hydroxide. The cathode made of nickel [or iron or copper] (**H**), comes up though the bottom of the pot. Holding it in place is a cake of solid sodium hydroxide (**K**) cast into the cell when it was cool. This cell has multiple anodes (**F**) that drop down around the cathode from above. A cylindrical vessel (**N**), floats in the fused alkali above the cathode, and the sodium (**D**), and hydrogen liberated at the cathode collect here, the sodium is protected under the hydrogen atmosphere (**C**) which is allowed to escape and is burned off at the exit. This are can be opened at periodically to remove sodium that gathers here. Metal gauze (M), separates the anode from the cathode. Oxygen that is simultaneously produced escapes from vent (P).



Potassium hydroxide electrolyzed with nickel electrodes in a steel U-Tube

Despite all these ideas a simple design such as above, where the anode and cathode areas are simply in a U-Tube (named such because it is in the shape of a U) affords enough separation to work for moderate periods of time, notice the distinct color difference between the anode (right) and cathode (left). Such a cell could be easily constructed from materials at a hardware store and heated at any point where the melt solidifies directly.

Famous examples of cells using molten salts are abundant, but the most famous examples include; (1) The aforementioned Castner cell which uses a melt of sodium hydroxide to yield hydrogen gas, oxygen gas, and sodium metal. (2) The Downs Cell(4) which uses an eutectic of sodium and calcium chloride to give sodium metal and chlorine gas. (3) The Hall-Héroult process whereby aluminum is produced from molten Al_2O_3 with cryolite to depress its melting point to a manageable 950 °C (manageable in industry at least). There are other examples but these stick out among the rest.

(1) Note, the electrolytic potentials in a molten salt are totally skewed with respect to the aqueous potentials found every
these can only be used as an extremely rough guide.

(2) An eutectic mixture of KOH and NaOH might be tempting to use for the preparation of the alkali metals due to it
melting point (~150 °C) however neither Na or K will be the product, instead you end up with a mixture of the tw
mixture, the sodium-potassium eutectic can spontaneously catch on fire at room temperature and is a liquid at
temperature, you don't want to think about how it will behave at 150 °C with oxygen and hydrogen being produ
around it!

(3) A note on this, the initial addition of molten salt to the electrodes can be a dangerous affair, without the electrolyte o

electrodes there is a period where the electrolyte added may only touch between the electrodes in a small space, when
the case the electrolyte may pop and short out the cell, sending molten salt out everywhere, during an attempt at sc
metal this happened to the author from a small quantity of sodium being generated causing a small disaster, be w
when working with molten salts.

(4) The Downs Cell uses a mixture of calcium chloride with sodium chloride to reduce the melting point of the sodium ch
from 801 °C to 580 °C, it contains roughly 33% NaCl and the remainder is $CaCl_2$, more NaCl is added as the re
progresses to replace the NaCl being electrolyzed. The resulting Na metal contains a very small calcium metal imp
The actual eutectic between NaCl and $CaCl_2$ lies at a different point giving an even lower melting point but the melt
the melt used in the industry. The Downs Cell is still the main method to produce sodium metal, replacing the Castne
quite some time ago due to NaCl being cheaper then NaOH, however the lower melting point of the Castner Cell ma
more attractive to the amateur chemist then the Downs Cell, coupled with the fact that no chlorine is generated in a C
Cell.

3.4b Aqueous Electrolysis

By Tacho.

1- Ions:

Water and other solvents called "polar solvents" do something interesting: their molecule has two poles, a positive charge on one side and a negative charge on the other. When you put a salt in water, the salt's negative ions are surrounded by water molecules pointing their positive side to it. Of course, the positive ions get surrounded by water molecules pointing their negative side to it. The result is that the salt dissolves in a liquid "soup" of ions that we will call "solution" or "electrolyte" from now on.

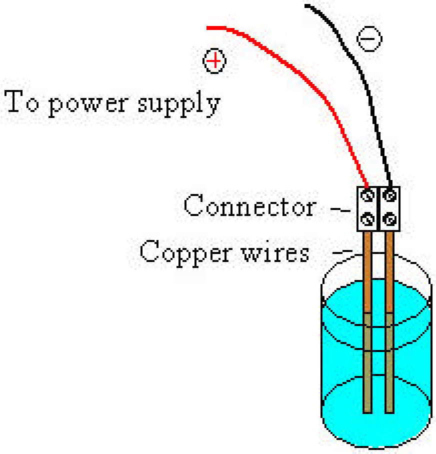
There must be enough anions to neutralize all cations. The whole thing must be neutral. Yes, it can built up charges, and momentarily have an imbalance, but mother nature will find a way to put thing back the way she likes it.

In the solution, you don't have the individual salts anymore. Just ions. So, if you put two salts in water, say... sodium chloride and potassium bromide, you only have sodium cations, potassium cations, chloride anions and bromide anions. You can extract from this solution the original salts or sodium bromide and potassium chloride! If you could substitute both of those anions for the hydroxide anion, than you could extract sodium hydroxide and potassium hydroxide from the same solution.

That's what this work is all about: the substitution of ions in aqueous solutions using electrolysis. We do that by pushing electrons in and out of atoms or molecules using electricity.

2- How?

Suppose I want to get a copper salt, dissolve one of my electrodes. I mount the following setup, put distilled water in the flask and turn on the power:



What do I get? Nothing! Why? Pure water practically does not conduct electricity! So we must add something that will make water conductive but won't be part of the reaction. Let's say we want copper sulfate, so let's choose something with a sulfate ion attached to an "inert" ion. MgSO4 should work fine. Magnesium sulfate is Epson salt that every pharmacy should have. For now, take my word that Mg won't be part of the reaction. Lets dissolve 3g of MgSO4 (the hydrate is okay.) in 30ml of distilled water and try again.

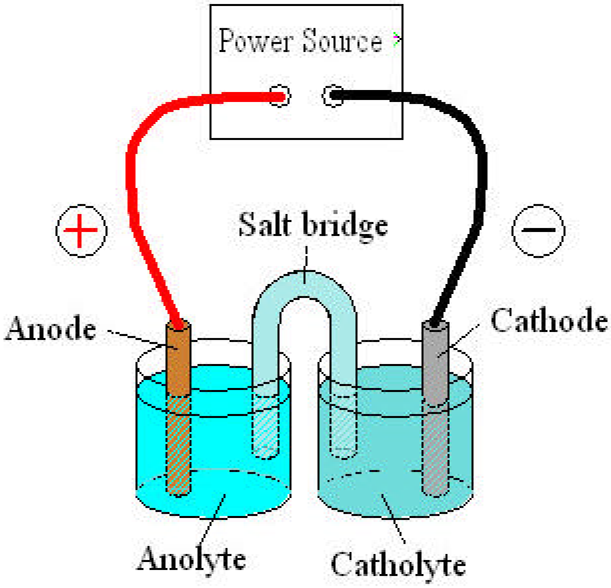
Immediately you'll see bubbles on the cathode and some blue tint by the anode. Cool! Copper ions should be blue! What is in those bubbles? The only thing in this soup that becomes a gas when it gains electrons (that's what cathodes do, they give electrons) is hydrogen. Water molecules get broken and hydrogen bubbles away.

But wait... after a couple of minutes we notice that something is very wrong! The blue tint is solid and it's precipitating! Copper sulfate doesn't do that! What is happening?

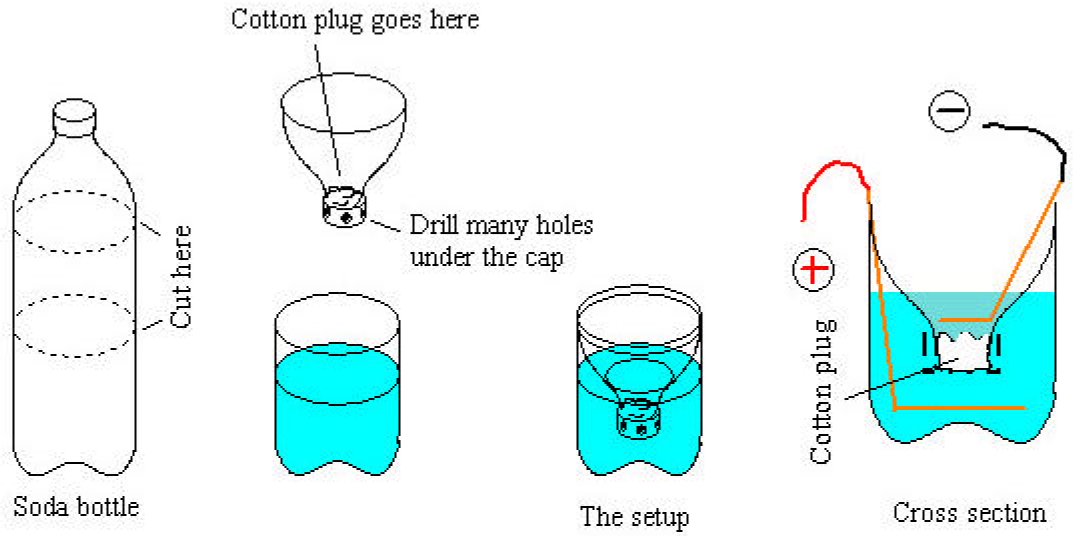
Here goes the explanation: When the hydrogen of water receives an electron, it joins a friend to become a gas molecule and bubbles away. It leaves behind the other half of the water molecule: OH- ion. This ion meets Mg+ ions or Cu++ ions and forms an insoluble hydroxide that precipitates (actually copper ions form more complex insoluble compounds, but let's pretend it's plain hydroxide). If you carefully put a piece of indicator paper close to the cathode while its bubbling, you will see that it's alkaline.

So how do we make our copper sulfate?

The little snotty armchair chemist now smiles and says with his squeaky voice: "I know! I know! All you have to do is to use two half-cells connected by a salt bridge! It's so simple! Like this:"



In the real world, however, this setup is not efficient. If you use the 10g $MgSO_4$ in 100ml water solution, under about 10 volts, you just can't see any bubbles! That's because ions are not agile movers, the salt bridge is a long way for them. Like any electric circuit, shorter paths increase current. So, a better design for the amateur would be:



I tested this for the copper sulfate production and it works. For some reason, it works best using the lower part as the anode cell, where the sulfate forms. The solution by the anode is called anolyte and now is a mixture of Mg^{2+}, Cu^{2+} and SO_4^{2-} ions. How to separate pure CuSO4 from it? If you look up the solubility information for the two salts you will find that 100 ml of cold water will dissolve 31 g of hydrated copper sulfate whereas it will dissolve 71 grams of magnesium sulfate therefore if you ran your electrolysis long enough then heated to reduce the volume of the solution, then cooled, the first thing to come out of solution should be the copper sulfate, this is just one possibility, one of the difficult parts of electrolysis involves this problem applied to systems involving many more ions.

In the above setup the cotton acts as a membrane to prevent the passage of certain ions. Another type of system accessible to the at home chemist consists of a flowerpot, unglazed and previously soaked in a strong acid placed into a larger container. The flowerpot and the larger container are filled so they contain the same level of electrolyte and an electrode is inserted in the flowerpot and another in your main solution outside. The electrons will migrate though the flowerpot and electrolysis will commence but your anode liquid will be separated from your cathode liquid, the strong acid soak necessary to facilitate the permeability of the flowerpot to ions, remember that strong acids are incredibly conductive. This is yet another way to separate your anolyte to prevent extraneous reactions.

I substituted the two copper wires of the first setup (the one with the connector) by two pencil leads (graphite) and put them in a pure copper sulfate solution procured from a gardening shop. When I turn on the power, quickly something starts building up in the negative electrode. That’s copper. It’s probably powdery and maybe too dark to look metallic, but it’s copper. That’s the principle behind electroplating. Don’t expect shiny metallic deposits though(1). What’s happening is that the Cu^{2+} cations in the solution gain a couple of electrons and become Cu, the metal.

3- Giving names to things happening.

What is happening in the anode is called oxidation. It doesn't matter if that there is no oxygen involved! Loosing electrons by the anode is called oxidation and don't you argue! Usually this oxidation results in metals becoming ions, ions gaining oxygen atoms, oxygen evolving in bubbles or halogen ions becoming the element.

What is happening in the cathode is called reduction. It's usually a metal ion becoming a metal, ions loosing oxygen atoms, elemental halogens becoming ions or hydrogen evolving in bubbles.

4- What happens when?

Question: What happens when I have many different metal pieces in the same electrolyte, connected and acting as anodes? Do they all go into solution at once? Do some of them go first? And what happens when I have many different metal cations by the cathode? Do they all get reduced together forming an alloy?

Answer: There is a priority list. Theoretically, if you have two different metal pieces, like copper and zinc connected to the positive pole of a battery and immersed in an electrolyte, all zinc will go into solution before the first atom of copper gets oxidized.

Here is an incomplete list (potential table) that shows the tendency of an atom or molecule or ion to gain or loose electrons. If it shows a reduction potential of–2.71 for something, the oxidation potential for the same something will be +2.71:

Oxidized creature	Reduced creature	Reduction potential:
Li^+	Li	-3.04
K^+	K	-2.92
Ca^{2+}	Ca	-2.76
Na^+	Na	-2.71
Mg^{2+}	Mg	-2.38
Al^{3+}	Al	-1.66
2 H_2O	H_2(g) + 2OH-	-0.83
Zn^{2+}	Zn	-0.76
Cr^{3+}	Cr	-0.74
Fe^{2+}	Fe	-0.41
Cd^{2+}	Cd	-0.40
Ni^{2+}	Ni	-0.23
Sn^{2+}	Sn	-0.14
Pb^{2+}	Pb	-0.13
Fe^{3+}	Fe	-0.04
$2H^+$	H_2(g)	0.00
Sn^{4+}	Sn^{2+}	0.15
Cu^{2+}	Cu^+	0.16
ClO_4^- + H_2O	ClO_3^- + $2OH^-$	0.17
AgCl(s)	Ag + Cl^-	0.22

Cu^{2+}	Cu	0.34
$ClO_3^- + H_2O(l)$	$ClO_2^- + 2OH^-$	0.35
$IO^- + H_2O(l)$	$I^- + 2OH^-$	0.49
Cu^+	Cu	0.52
$I_2(s)$	$2\ I^-$	0.54
$ClO_2^- + H_2O$	$ClO^- + 2OH^-$	0.59
Fe^{3+}	Fe^{2+}	0.77
Hg_2^{2+}	$2Hg(l)$	0.80
Ag^+	Ag	0.80
Hg^{2+}	$Hg(l)$	0.85
$ClO^- + H_2O$	$Cl^- + 2OH^-$	0.90
$2Hg^{2+}$	$Hg_2^{2+}(aq)$	0.90
$NO_3^- + 4H^+$	$NO(g) + 2H_2O$	0.96
$Br_2(l)$	$2Br^-$	1.07
$O_2(g) + 4H^+$	$2H_2O$	1.23
$Cr_2O_7^{2-} + 14H^+$	$2Cr^{3+} + 7H_2O$	1.33
$Cl_2(g)$	$2Cl^-$	1.36
$MnO_4^- + 8H^+$	$Mn^{2+} + 4H_2O$	1.49
$H_2O_2(aq) + 2H^+$	$2H_2O(l)$	1.78
Co^{3+}	Co^{2+}	1.82
$S_2O_8^{2-}$	$2SO_4^{2-}$	2.01
$O_3(g) + 2H^+$	$O_2(g) + H_2O$	2.07
$F_2(g)$	$2F^-(aq)$	2.87

One should be able to get all the information one needs from this table, but lesser creatures like me always get confused trying to draw practical conclusions from it! So I organized the following two specific tables for things that can happen at your anode and things that can happen at your cathode from practical observations:

Things that can happen at your anode (where oxidation takes place, positive pole in the electrolytic cell) in order of priority:

Priority a.k.a. Oxidation Potential in Volts:	**Situation:**	**What I think should happen:** ***What happened when I tried it:***
higher than +0.76	If your anode has alkali metals, aluminum or magnesium ...	...Alkali metals react with water, and can't be electrodes in aqueous solutions. Aluminum behaves strangely as an anode(2) magnesium will likewise react with the solution and little relevant electrolysis can be preformed with it.
+0.76	If your anode has zinc...	...Zn^{2+} ions go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain anions in the electrolyte like carbonate, hydroxide or hypochlorite. The Zn in the anode "dissolves" in the electrolyte.

		It works. Nice and easy. Using a two half- cell setup with MnSO4, the zinc anode dissolves into Zn^{2+} ions, a colorless clear solution. Insoluble impurities may show themselves at this point.
+0.74	If your anode has chromium...	...Cr^{3+} (chromic) ions go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain ions in the electrolyte like borate or cyanide. The Cr in the anode “dissolves” in the electrolyte.
+0.41 or +0.04	If your anode is made of iron...	...Fe^{2+} (ferrous) ions go into solution at “priority” of +0,41V, or Fe+3 at “priority” of +0.04V. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain exotic ions in the electrolyte. The Fe in the anode “dissolves” in the electrolyte. **See -0.77 priority.** *Funny things happen with an iron electrode. In my MgSO4 cell, at voltages lower than about 5 volts, a yellow/brown tint and a greenish precipitate appear due to iron ions(?), but if the voltage is above that, oxygen evolves from the anode and almost no color shows in the solution.* *If a piece of zinc from a battery is strapped to the anode, almost no brown color evolves .Makes sense.*
+0.40	If your anode is cadmium...	...Cd^{2+} ions go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain ions in the electrolyte like carbonate or borate. The Cd in the anode “dissolves” in the electrolyte, take note of cadmiums toxicity.
+0.23	If your anode has nickel...	...Ni^{2+} go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain ions in the electrolyte, like carbonate or borate. The Ni in the anode “dissolves” in the electrolyte.
+0.14	If your anode has tin...	...Sn^{2+} (stannous) ions go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain ions in the electrolyte. The Sn in the anode “dissolves” in the electrolyte. **See -0.15 priority.**
+0.13	If your anode has lead...	...Pb^{2+} (plumbous) ions go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds many common ions in the electrolyte, like sulfate, chloride or carbonate. The Pb in the anode “dissolves” in the electrolyte. Another heavy metal you should be careful about.
0.00	If you make the incredible “hydrogen bubbling on platinum” electrode...	...Literature is unanimous that hydrogen would become an H^{+} ion, this is the standard by which this system of potentials was based.
-0.15	If your anolyte (the solution of ions around your anode)	...The Sn^{2+} is oxidized and becomes Sn^{4+} (stannic) ions.

	has Sn^{2+} (stannous) ions...	
-0.16	If your anolyte has Cu^+ (cuprous) ions...	...The Cu^+ become Cu^{2+} (cupric) ions.
-0.17	If your anolyte has ClO_3^- (chlorate) and OH^- (hydroxide = alkaline medium)...	...The ClO_3^- ion becomes the ClO_4^- (perchlorate) ion and some extra water is formed. *Perchlorate is the sweet darling of pyrotechnics. I have not done it, but the whole electrolysis sequence Cl^- -> ClO^- -> $ClO2^-$ -> $ClO3^-$ -> $ClO4^-$ is well described on the internet(3).*
-0.22	If your anode has silver and your anolyte has Cl^- ions...	...Insoluble AgCl (Silver Chloride) is formed. *The AgCl forms an insulating white layer on the silver piece that reduces the current sharply. It resembles white (well, maybe cream) paint that darkens if exposed to sunlight for long.*
-0.34 or -0.52	If your anode has copper...	...Cu^{2+} will go into solution. Or Cu^+ at –0.52. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain ions in the electrolyte, like carbonate or hydroxide. The Cu in the anode "dissolves" in the electrolyte. *My results with a MgSO4 cell are described in the text.*
-0.35	If your anolyte has ClO_2^- (chlorite) ion and OH^- (hydroxide = alkaline medium)...	...The ClO_2^- ion becomes the ClO_3^- (chlorate) ion and some extra water is formed. *See note for chlorate (oxidation potential –0.17).*
-0.49	If your anolyte has I^- (iodide) and OH^- (hydroxide = alkaline medium)...	...The I^- ion becomes IO- (hypoiodite) ion and some extra water is formed.
-0.54	If your anolyte has I^- ion.	I_2 is formed. *It does. In a potassium iodide solution, using pencil's graphite electrodes, brown iodine color rapidly develops around the anode while hydrogen bubbles form at the cathode.* *Interesting note: If you apply 60Hz AC directly from the transformer (6V) into the electrodes, a yellow color slowly develops, showing this reaction is not fully reversible. At least mine wasn't.* *Also interesting: according to this list of priorities, a copper anode should not generate iodine, instead, the copper should dissolve into copper ions. That did not happen in my test. Brown color developed near the copper anode with no blue tint. Go figure!*
-0.59	If your anolyte has ClO^- (hypochlorite) and OH^- (hydroxide = alkaline medium)...	...The ClO^- ion becomes the ClO_2^- (chlorite) ion and some extra water is formed. *See note for chlorate (oxidation potential –0.17).*
-0.77	If your anolyte has Fe^{2+} (ferrous) ion...	...The Fe^{2+} becomes Fe^{3+} (ferric).

-0.80	If your anode has Hg Mercury...	...Hg^{2+} go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds the certain ions in the electrolyte. The Hg in the anode "dissolves" in the electrolyte. Dangerous heavy metal, by the way. *Mercury, besides being toxic, has some difficult to understand chemical behaviors such as its ability to introduce overpotentials and such.*
-0.80	If your anode has silver...	...Ag^+ go into solution. It may form insoluble salts (precipitate or form chunks in the anode) if it finds certain anions in the electrolyte, like hydroxide, carbonate, iodide, bromide or chloride. Silver has many insoluble salts. The Ag in the anode "dissolves" in the electrolyte.
-0.90	If your anolyte has Cl^- (chloride) and OH^- (hydroxide)...	...ClO^- (hypochlorite) ion is formed. And some extra water. *See note for chlorate (oxidation potential –0.17).*
-1.07	If your anolyte has Br^- (bromide) ion...	...Bromine (liquid) is formed(4). *It does. In a sodium bromide solution, using pencil graphite electrodes, brown bromine color rapidly develops around the anode while hydrogen bubbles form at the cathode.* *This one is fully reversible: If you apply 60Hz AC directly from the transformer (6V) into the electrodes nothing seems to happen, although bromine and hydrogen are being oxidized and reduced 60 times per second!* *Also: If you use a copper anode, no brown color develops, only a bluish precipitate (bromide? hydroxide?). This is expected, since copper is above the bromide ion in the priority list.*
-1.23	**whatever aqueous...**	...At this point, in aqueous solutions, water molecules nearby the anode are split in O_2 (gas), that evolves from the anode, and 4 H^+ ions, that remain in the solution. Theoretically, no oxidation should take place beyond this potential, because water should get oxidized first. Ha ha! So much for theory! Something called overpotential makes, among others, the next two oxidations possible:
-1.36	If your anolyte has Cl^- (chloride)...	...Chlorine gas evolves. *This one sure works. The electrolysis of a concentrated NaCl solution gives off hydrogen and chlorine. In theory, if you keep the voltage low enough, you can get a small amount of oxygen and no chlorine. But that may depend on the anode composition.*
-1.49	If your anolyte has Mn^{2+} (manganous) ion...	...Mn^{2+} becomes MnO_4^- (permanganate) ion. *I have not tested this one personally, but I read in a book a detailed description of a laboratory procedure to obtain potassium permanganate by electrolysis. The book seems trustworthy.*

Things that can happen at your cathode (where reduction takes place, negative pole in the electrolytic cell) in order of priority:

Priority	**Situation:**	**What I think should happen:**

a.k.a. Reduction Potential in Volts:		***What was actually observed:***
1.49	If your catholyte (the solution of ions around your cathode) has MnO_4^- (permanganate ion) and the solution is acidic...	...MnO_4^- turns to Mn^{2+} (manganous) and some water is formed.
1.07	If your catholyte has Br_2 (liquid bromine)...	...Br^- (bromide) ion is formed. *As I said before, if you apply 60Hz AC directly from the transformer (6V) into graphite electrodes in a sodium bromide solution, nothing seems to happen, although bromine and hydrogen are being oxidized and reduced 60 times per second! So, it works!*
0.96	If your catholyte has NO_3^- nitrate ion and your solution is acidic…	...NO (Nitrogen Monoxide) gas and some extra water are formed.
0.90	If your catholyte has ClO^- (hypochlorite) ion...	...Cl^- (chloride) and OH^- (hydroxide) are formed.
0.85	If your catholyte has Hg^{2+} ions...	...Hg (metallic mercury) is formed.
0.80	If your catholyte has Ag^+ ions...	... Ag (metallic silver) is formed.
0.77	If your catholyte has Fe^{3+} (ferric) ions...	...Fe^{3+} becomes Fe^{2+} (ferrous) ion
0.59	If your catholyte has ClO_2^- (chlorite) ion...	...ClO_2^- becomes ClO^- (hypochlorite) ion
0.54	If your catholyte has I_2 (iodine) dissolved...	...Iodine becomes I^- (iodide) ion. *As I said before, if you apply 60Hz AC directly from the transformer (6V) into graphite electrodes in a potassium iodide solution, a yellow color slowly develops, showing this reaction is not fully reversible. But the fact that is a slow development shows that most iodine was oxidized and reduced 60 times per second. So it works!* *However, a practical method of turning elemental iodine into I- is beyond my reach . I tested a solution of ethanol, iodine and MgSO4 and another with ethanol, iodine and NaCl. None seem to have any change under low voltage, At higher voltages, hydrogen evolved but no change in color. The anode was graphite.*

0.52 or 0.34	If your catholyte has Cu^+ (cuprous) or Cu^{2+} (cupric) ions...	...Both get reduced to metallic copper. Cu^+ at 0.52 priority and Cu^{2+} at 0.34 priority. *This one is easy. But to get a shiny smooth deposit is an art*(1). *As expected, when I did electrolysis using graphite electrodes in a solution of copper sulfate and zinc chloride, only copper deposited in the cathode (I tested it regularly dipping the electrodes in 20% HCl where copper does not react, but zinc bubbles). Only when almost (!) all blue tint from copper ions have gone from the solution, zinc starts depositing. Chlorine evolved from the anode, by the way, so by default, zinc sulfate was left in solution.*
0.49	IO- (hypoiodite)...	...Becomes I- (iodide) and OH-.
0.35	ClO_3^- (chlorate) ion...	... becomes ClO_2^- (chlorite) ion
0.17	ClO_4^- (perchlorate) ion...	...becomes ClO_3^- (chlorate) ion.
0.15	Sn^{4+} (stannic)...	...becomes Sn^{2+} stannous ion.
0.00	If your catholyte is acidic and therefore has H^+ ...	H_2(g) is generated. Therefore, none of the following metal ions can be reduced in acidic electrolyte. Which makes sense, since these metals are attacked by acids. In fact, they are attacked by acids exactly because they have a lower reducing potential then H^+, but that's another story.
-0.04	Fe^{3+} (ferric) ...	metallic Fe deposits at the cathode. *No it doesn't. At least not in my ferric chloride solution with graphite electrodes. I could get no deposit. This could be due to the acidity of ferric chloride solutions though.*
-0.13	Pb^{2+} (plumbous)...	metallic Pb deposits at the cathode.
-0.14	Sn^{2+} (stannous)...	metallic Sn deposits at the cathode.
-0.23	Ni^{2+}...	metallic Ni deposits at the cathode.
-0.40	Cd^{2+}...	metallic Cd deposits at the cathode.
-0.41	Fe^{2+} (ferrous)...	metallic Fe deposits at the cathode.
-0.74	Cr^{3+} (chromic)...	metallic Cr deposits at the cathode.
-0.76	Zn^{2+}...	metallic Zn deposits at the cathode. *It works. The deposit from a zinc chloride solution is powdery and dark, but it's zinc allright.*
-0.83	**Whatever aquoeous...**	Water by the cathode gest split in H_2(g) that evolves from the cathode and + 2OH-, that remain in the solution. **End of the line.** No exceptions and no reductions from here!
	Aluminum ions, Magnesium ions and alkali metals ions...	Forget it! Not in aqueous electrolytes! Water gets reduced first(5)!

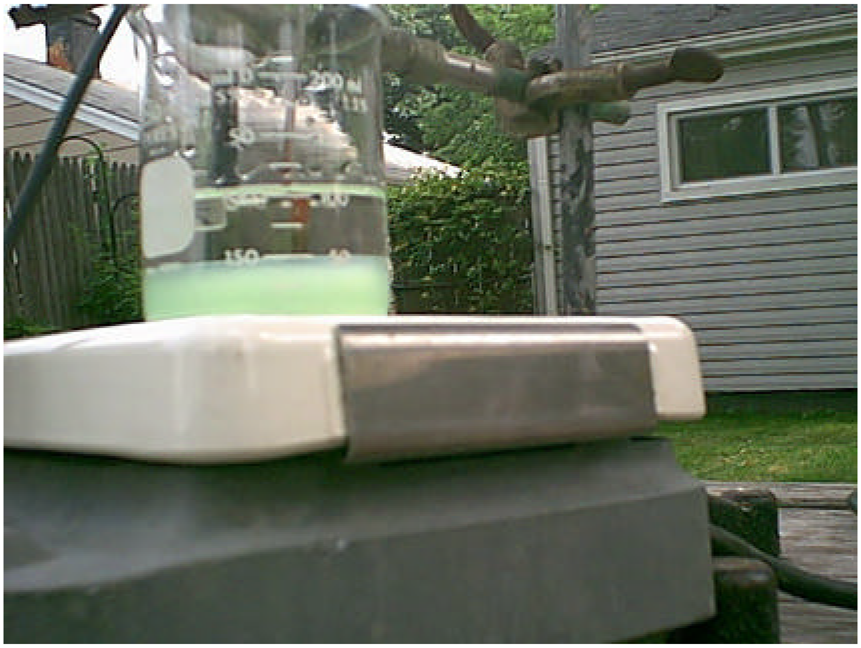
5- Practical notes for amateur experiments:

C) What can be made using aqueous electrolysis?

Electrolysis can be inefficient and slow. Consumes lots of electricity and takes a long time to produce low yields that, in most cases, must be submitted to other procedures to isolate the pure products. If you are hoping to make a bottle of bromine or iodine in one sunny Thursday afternoon, you will be very disappointed. On the other hand, it's simple and within the reach of any amateur. It' a good option when you need just a little bit of a chemical; instead of buying half a kilogram of an expensive, polluting and carcinogenic salt, make a little bit as needed. Also, it's a way of obtaining chemicals that you just can't buy.

- Salts in general can be obtained like sulfates, chlorides, nitrates, chlorates and perchlorates;
- Hydroxides and oxides;
- Metal powders and metal deposits on surfaces or purification of a metal(6);
- Gases like oxygen, hydrogen and chlorine;
- Bromine and Iodine;
- Dilute acids;
- Organics like ethane, chloroform, etc.

A Small Mistake and a Learning Experience



In an attempt to prepare chloroform a quantity of acetone was mixed with water and some sodium chloride. The mixture was subjected to electrolysis using a carbon electrode and a nickel electrode. The reasoning behind the experiment was simple at the time; (1) Electrolysis of the sodium chloride would give chlorine gas at the anode and hydroxide at the cathode. (2) The mixture was being subjected to magnetic stirring so most of the formed chlorine would be dissolved and reacted with the hydroxide being produced to give a solution of sodium hypochlorite (the active ingredient in bleach). (3) The formed sodium hypochlorite would react with the acetone present in a haloform reaction (**5.3c**) to give the desired chloroform. This however was not what happened, instead the nickel electrode went into solution as the electrolysis progressed and 'salted out' the acetone. This term is used to describe the lack of solubility of the nickel chloride in acetone causing the water and acetone to become insoluble in one another. As a result the reaction that I hoped for had ceased. (Notice the green color indicative of nickel salts in the bottom water layer which is denser then the acetone layer above.) It should also be noted that chlorine gas can react with acetone directly giving chloroacetone, a potent lachrimator, which could have definatley caused me some breathing problems if formed in decent quantities. So although the reaction sounded good at first, there were definitely some bugs to work out before I would be able to run it again.

(1) As some have concisely stated, electroplating can be an art. Developing a uniform coating of metal of significant thi
on nearly any substrate requires technique, and a decent investment in time and effort of calibrating a standard techni
work with your equipment, providing you don't just rush out and buy a setup specifically for electroplating. The
advanced form of this is a process known as *electroforming*, whereby an object is coated with a thick mechanically
layer of metal, non-metal objects can be formed from wax or other materials then rendered conductive with some k
paint then electrolyzed and coated with enough metal to make them useable for machining purposes. Ho
electroforming in the home lab is considerably more difficult then electroplating.

(2) When aluminum is used as an anode certain characteristics are expressed that are beyond the scope of this text, infor
relevant to this can be found using key word combinations such as "Anodized Aluminum" or "Anodization".

(3) There are many places on the web covering the whole series from chloride to chlorate to perchlorate through electrol
home, searching is quite easy and rewarding, it can be done and people have succeeded in making large amou
perchlorates in this manner, http://www.geocities.com/CapeCanaveral/Campus/5361/chlorate/chlorate.html is a nice
start out at.

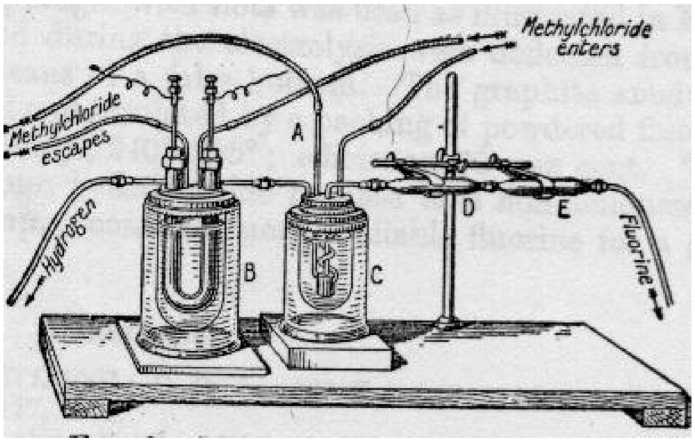
(4) If the anode and cathode are separated even by a sheet of paper the bromine can be collected due to its density comp
water. It will sink to the bottom of the apparatus and a small beaker can be placed there under water, the bromin
actually collect in the beaker. For an example of the experiment look at http://www.crscientific.com/article-bromine.html

(5) As stated previous, using a mercury cathode a mercury-sodium amalgam can be made containing a small amount of s
metal, this however is more a curiosity as there is only a small amount of sodium, and winning it from the ama
involves some dangerous manipulations such as distillation of the amalgam, that much mercury at those ki
temperatures can contaminate everything within a large radius, around an entire house!

(6) A standard procedure industrially for copper but can be applied to other metals. The metal to be refined is placed
anode where it will dissolve in the solution as electrolysis prceeds, if the metal has a fairly low reduction potential
will also re-deposit at the cathode before other impurities in the anode will carry over, details can be found far and w
the internet through simple searching: http://doccopper.tripod.com/copper/er.html

3.4c Non-Aqueous Electrolysis

Sometimes water as a solvent just won't cut it. Rarely this is due to the limited solubility of a compound in water, more often then not electrolysis of a substance in a non-aqueous environment is necessary due to the reactivity of some compound that is being solvated or a compound being produced with the water in which it is dissolved, therefore excluding it as a solvent. Usually in these cases it is some metal that is being formed, the alkali metals being the prime example, that would react with the water, but it need not always be a cation that is being reduced, shown here is a picture of a cell for the preparation of fluorine, not only would the preparation of fluorine be inhibited by its high potential in aqueous solutions, but it would react readily with water, therefore the electrolysis of a salt, usually potassium fluoride is done in anhydrous hydrogen fluoride.



Moissan's original cell for electrochemical fluorine production.

The actual electrolysis takes place in the cell marked B. Contained within was a U-tube made of platinum metal. The stoppers on the top were composed of shaped calcium fluoride and the anode and cathode were both platinum. The cell ran on a mixture of potassium fluoride and hydrogen fluoride at low temperatures. The temperature of the cell being maintained by the methyl chloride that entered the area surrounding the U-tube. Jar C also contains methyl chloride which cooled down the resulting gasses to condense out any hydrogen fluoride that may have made its way through. The bulbous parts (D) and (E) contain sodium fluoride, which forms the acid salt NaF*HF when in contact with hydrogen fluoride to remove the last of the hydrogen fluoride. The whole tubing system leading from the fluorine production area was composed of platinum in this original setup.

When it comes to solvents available for use, the requirements are a little more stringent then just any liquid available that contains water. As a matter of fact finding a good non-aqueous solvent is near impossible, at least one that has the wide range of possibilities as water, therefore selection of a solvent is highly dependent on what you want to accomplish, and each solvent that you have the option of using has its own reactivates and conditions that it best works under that must be taken into account [Note that the reductive or oxidative potential of anions and cations is influenced by their solvent and therefore the table in the previous section on aqueous electrolysis cannot be followed strictly for electrolysis in non-aqueous mediums]. The solvents that do work for non-aqueous electrolysis well are hard to come by, and few of them are over the counter, still, here is a brief listing (Note that when it comes to availability, the reference is to the availability of the anhydrous substance, and of course the availability of substances depends heavily on your country):

[Solvents highlighted in blue have some merit of being used in electrolytic procedures beyond the curiosity stage.]

Liquid	Availability	Properties
CH_3CN Acetonitrile	Somewhat	Not as toxic as one would infer from the

	Difficult to Acquire	cyanide group, it is widely studied for use as a non-aqueous medium, it dissolves a nice variety of ionic compounds to some extent.
H_2SO_4 (Sulfuric Acid)	Somewhat Available	The usefulness of sulfuric acid for non-aqueous electrolysis is limited, more often then not, it is the reagent in a technique to produce peroxysulfuric acid or ozone.
$(CH_3CH_2O)_2CO$ Ethyl Carbonate	Varies Greatly	Coming into its own as a solvent in lithium ion batteries, somewhat inert and works well for increasing the conductivity of a solution.
HF Hydrogen Fluoride	Very Difficult to Acquire	Hydrogen fluoride is incredibly dangerous and preparing it at home in the anhydrous state would be ridiculously dangerous, it finds very limited use except as a solvent for the preparation of high oxidation state compounds (See Moissan's cell at the opening of this section).
SO_2 Sulfur Dioxide	Somewhat difficult to Acquire (Could be made)	Sulfur dioxide is a widely investigated non-aqueous solvent. It is however a gas at room temperature and is very irritating to work with.
CH_3SOCH_3 Dimethyl Sulfoxide (DMSO)	Somewhat Available	Displays a wide solvating ability and conducts fairly well, although somewhat easily attacked by oxidizing agents, still a good non-aqueous solvent.
NH_3 Ammonia	Difficult to Acquire (Could be made)	Ammonia is a gas at room temperature so cooling is necessary to work with it. Ammonia solvates many things including the alkali metals, it can also produce them through electrolysis of their salts, along with other interesting things, such as hydrazine.
HCOOH Formic Acid	Difficult to Acquire (Could be made)	Has a significant polar character and will dissolve many things that water will, finds limited use in the non-aqueous electrolysis field.
CH_3COOH Glacial Acetic Acid	Difficult to Acquire	
$HCON(CH_3)_2$ Dimethyl Formamide (DMF)		
C_5H_9NO N-Methylpyrrolidone		

$C_6H_5NO_2$ Nitrobenzene	Difficult to Acquire (Could be made)	Nitrobenzene is slightly more polar then most aromatics and is therefore more useful then many in electrolysis. It has the famous reputation of allowing for the preparation of the alkali metals by electrolysis of an alkali halide fused with aluminum halide. The downsides of this solvent being the acquisition of it coupled with its toxicity.
C_5H_5N Pyridine	Difficult to Aquire	Somewhat able to dissolve ionic compounds, the classical example of its abilities is the preparation of lithium metal by electrolysis of a solution of lithium chloride in pyridine(2).
$(-CH_2CH_2CH_2CH_2O-)$ Tetrahydrofuran THF	Can be acquired and purified from OTC sources.	THF is used in some OTC applications involving PVC pipe and can be purified from there. It does well in organometallic applications because it has a somewhat pronounced polar character and thus works well for fun with electrolysis.
$(CH_3CH_2)_2O$ Diethyl Ether	Difficult to Acquire (Could be made)	Shows only a small solvating ability, not a good choice for electrolysis as many dissolved salts do not conduct.
CH_3NO_2 Nitromethane	Difficult to Acquire	
CH_3COCH_3 Acetone	Widely Available	
$CH_3CH_2NH_2$ Ethyl Amine		
Aromatics (Toluene, Xylene, Benzene)	Widely Available	Aromatics are had to work with in that they hardly dissolve anything considered ionic, let alone make conductive solutions, but things can be done(1).

Electrolysis of lithium chloride in DMSO with Nickel Electrodes

(1) Although if something is solvated in an aromatic hydrocarbon it really doesn't contain any ions, electrolysis of alum bromide in toluene with an amine to take up the liberated bromine will give deposits of aluminum metal, see US 3,997,410 for an example. This is due to complexes generated between the aluminum and bromine giving compou the sort $AlBr_4^+$ which can carry the current through the solution leading to productive electrolysis.

(2) In practice this electrolysis from lithium chloride to lithium metal is very sensitive and does not give good result solutions are only slightly conductive as well, facilitating the need for very large surface areas on the electrodes.

3.5 Titration

When someone says titration the first thing that comes to the average persons mind is determination of acid concentration. The regular acid/base titration to determine acid concentration in a solution using a base or acid of known concentration is a useful and widely known titration method. However titrations can be used to determine other things, such as metal ion concentrations and oxidizing power of a solution. Titrations at home are possible and with just one moderate investment you will be able to perform a variety of useful titrations for some time to come.

In case you are completely new to the concept of a titration here is a complete overview of the process. Let's say that you have bought some battery acid from your local auto supply and you have no clue of what the concentration is. Reading the label you realize the ingredients are sulfuric acid and water, no added products. So, how much sulfuric acid do you have? In an acid base titration you would take your acid of unknown concentration and react it with a base of known concentration until the acid has been completely reacted (or vice verse with a base of unknown concentration). There are two key points to consider, your standard, and your indicator.

<u>Standard Solution</u>

Your base or acid that you are reacting against your unknown should be of a known concentration, this solution of a known strength that you are basing your calculations off of is known as your standard solution. A standard solution must either be purchased from a chemical supplier of prepared. Many liquids over the counter can vary in their acid of basic strength for one reason or another so you cannot take them at label value and can therefore not use them as standard solutions. For example, the amount of ammonia and OTC ammonium hydroxide can vary by several percent, it evaporates, the plant that made it had a slight variation with your batch, these things are meant for cleaning, not for acid-base titrations after all. So the best thing to actually start with is usually a solid product that when dissolved in water will yield an acid or basic solution whose concentration can easily be calculated. The solid should be something non-hydroscopic or at least have a known water content (i.e. known water of hydration) better yet it should be dried shortly before preparing the solution if you are unsure how much water it may contain, not at exhaustive temperatures but maybe 150 °C for a few hours, assuming the compound will not decompose by then.

Most solids that you will come across are only weakly basic or acidic, for example, sodium carbonate of sodium hydrogen sulfate. However for an acid-base reaction to be complete and to give good results you should always titrate with at least one of the titratrants being strong, either a strong base or strong acid. Therefore the first thing you should titrate against should be a strong acid or base, assuming for example you decide to go with sodium bicarbonate as your primary, it would be wise to titrate it against some over the counter hydrochloric acid, in this way you will get better results and you will additionally have standardized a strong acid for use in standardizing other bases.

Now, to prepare your standard you are going to need a scale and a graduated cylinder or another container that can measure liquid fairly accurately. If your scale doesn't measure that accurately, i.e. no decimal placed, you might want to make a large amount of solution in order to minimize the relative errors. If your are desperate you can find the solubility of a substance at room temperature and saturate a solution with it at 25°C then decant off the saturated solution, this will contain a known amount of your solid, however it may deposit solid on cooling and therefore many induce error so the solution should be used immediately to standardize a different solution. With your standard solution prepared you are part way to the desired goal of being able to determine the concentration of an unknown.

Sample Standard Preparation of a Sodium Carbonate Solution

Taking your grams of sodium carbonate you would put it into a flask that is able to measure volume exactly, a volumetric flask or if all you have is a graduated cylinder, that (Note, you cannot use a beaker for this, beakers can be significantly different then the volume expected). So, lets say that you weigh out 25 grams of anhydrous sodium carbonate and you dissolve this in water and bring the volume up to total 250 ml. 25 g Na_2CO_3 / 106 g/mol = .235 mol then you have 250 ml of solution which is .25 L, dividing the mols Na_2CO_3 by the liters you get mol/L which is M and therefore you have your solution of known concentration, in this case it being .94 M Na_2CO_3.

Indicators

How do you know the acid has been completely reacted. With the Na_2CO_3 you can get a very inaccurate idea of when the reaction is done by watching to see when it stops bubbling. However the best method would be to add a chemical indicator. There are many acid-base indicators, but for going from an acidic solution to being able to tell when it's basic the quintessential indicator is phenolphthalein which would have to be purchased, it is used in a dilute solution. In the presence of acid or in neutral conditions solutions containing this organic molecule are clear, but around a pH of 8 the solution sharply turns pink. (Note that only a few drops of indicator in dilute solution are needed).

There are naturally occurring indicators, a number of natural extracts will change color depending on the pH of the solution and could therefore potentially be useful for home preparations of acid-base indicators. The most famous of all of these natural indicators is the Red Cabbage Indicator(1), that specific phrase pulling up many many hits on google some specifically on the preparation of the solution. Because a color change can be relative though it would normally be practice, when using such an indicator that undergoes such a wide color range to use a standard solution of known acidity of bascisity and subject the indicator to it, that way you can compare that color with the color of your solution that you are titrating until they are equal. However for a simple determination these small details can be ignored.

Performing the Titration

Your equipment in this operation is a piece of glassware called a buret. It is most easily described as a tall thin buret with a stopcock on the bottom where the liquid comes out. It is filled to a specific level with the liquid you are titrating with and a flask containing a known amount of your unknown solution with a small amount of indicator is put below it. The stopcock is turned and the flask is slowly rotated to stir while the titrating liquid enters your unknown. Once the color starts to become apparent in areas the flow of the titrating liquid should be lessened and it should be added as drops until the liquid turns to its indicator color and stays that way for 30 seconds. Congratulations, knowing the initial volume of liquid in your buret and the final you can determine the amount of liquid of a known concentration that it took to react with your unknown. For example, let's use the .94 M Na_2CO_3 solution I mentioned above and let's keep with our sulfuric acid example.

The initial volume of the .94 M Na_2CO_3 in your buret was 0.00 (They start at 0 and increase as they go down) and the initial volume of sulfuric acid in a beaker beneath it is 20 ml. On your first attempt 43.10 ml of Na_2CO_3 was needed to turn the indicator pink. On your second attempt 41.2 ml was needed to do the same, and on your third attempt 41.27 ml was needed (Multiple titrations followed by averaging of results usually gives better results, the less accurate the lab equipment the more titrations necessary to off set it). First, what is the reaction?

$$Na_2CO_{3(aq)} + H_2SO_{4(aq)} \Rightarrow Na_2SO_{4(aq)} + H_2O_{(l)} + CO_{2(g)}$$

Although the process is complicated by H_2SO_4 being diprotic so there are two protons that must be neutralized and because of this complete conversion to Na_2SO_4 cannot be totally confirmed, it is somewhat safe to assume in this case. So the stoichiometry is 1 : 1 so the number of mols Na_2CO_3 used equals the number of mols H_2SO_4 in solution. If this had been hydrochloric acid instead the ratio would be 1 mol sodium carbonate to 2 mols HCl so the number of mols HCl would be double the number of mols Na_2CO_3 in the amount of solution used. But back to the example. Here are the calculated molarities.

(Molarity of titrating solution) x (Volume of titrating solution in ml) x 1/1000 (converts volume in ml to volume in liters) = mols titrating solution

[(mols titrating solution) x (conversion ratio [1/1 in this case])] / (volume of solution being titrated in ml x [1/1000])

So we get:

[.94 M Na_2CO_3 x 43.1 ml x 0.001 x 1 (conversion ratio)]/(20 x .001) = 2.02 M H_2SO_4

[.94 M Na_2CO_3 x 41.2 ml x 0.001 x 1 (conversion ratio)]/(20 x .001) = 1.94 M H_2SO_4

[.94 M Na_2CO_3 x 41.27 ml x 0.001 x 1 (conversion ratio)]/(20 x .001) = 1.94 M H_2SO_4

Adding them all together and dividing my three we get an average molarity of 1.96 M so bottle it and save it for later now that you know.

Some of you might be thinking now, why sodium carbonate? Well, this is a good choice for the at home chemist for a few reasons 1) It is usually a somewhat pure product, especially if heated first to drive off excess moisture and decompose bicarbonate 2) It is readily available 3) It is measurable without difficulty But there are some aspects that it lacks that would make it ideal:

1) Sodium bicarbonate is not a strong base and therefore cannot react completely with an acid :

This is a bad thing, only a strong base can completely titrate a weak acid, however sodium bicarbonate decomposes to carbon dioxide which drives the reaction foreword, to ensure that every last bit of CO2 has been driven out of solution though and ensure the most accurate endpoint possible immediately after a color change is noted that stays for more then a few seconds, it helps to heat the solution being tested to drive off dissolved carbon dioxide which causes a slightly acidic solution.

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

more information on acid-base chemistry.

3.6 Temperature control/Measuring

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

Your basic reactions are readily controlled with water in some form or another. Water has a high specific heat and it can absorb a lot of heat before rising in temperature, or conversely, it can hold enough heat to warm another mass significantly before loosing its full heat. So a cold water bath might be good for cooling, a warm/hot water bath for heating, and ice is always good to have around just in case. However the other time tested method of cooling mixtures even cooler are eccentric mixtures. KOH with ice can achieve extremely low temperatures, HNO3 and ice can too. As can the dry ice/acetone slurry that is occasionally used. Even cooler mixtures can be obtained with hydrocarbon baths with liquid nitrogen added periodically. Liquid nitrogen could even be used directly, much lower then this is hard to accomplish in a home lab, but the lowest temperature coolant you will run across would be liquid helium, but I doubt you'll find this in any local super market.

The Cooling Effect of Winter:

Many of the people who read this text will experience some form of winter cooling and people further south and north of the equator will experience even further cooling and snow. Winter is a good time to perform those exothermic reactions that would usually require external cooling or precooling of the reagents, just sit them outside. Many chemicals are also less volatile under these conditions, bromine is more friendly the colder you get so if you were trying to make somewhat large quantities of bromine winter would at least make it more manageable. The preparation of chloroform from acetone and sodium hypochlorite is aided by allowing the reagents to come to temperature outside then mixing and cooling in a snow bank, it’s cheap and it’s effective.

There is one little catch about the snow though that many people often forget, it may be cold but it’s also an insulator. Where the snow is touching the container to be cooled it is doing it’s job, but in short order if it was actually warm enough to warrant external cooling then the snow around the vessel will melt and make a nice insulating air pocket. For this reason snow is best used as a slush around the reaction vessel to be cooled with occasional stirring, in this way heat is conducted further away faster and there is no insulating effect.

On the opposite side of the temperature scale you are shooting for heating. Most any heating you will be doing will be the work of either electric heating, as in a hot plate, or chemical heating in the form of combustion. Commonly electric heating concepts can get to 250C or so, lab grade hot plates can get even higher. But to get really high you will have to use combustion, lacking a suitable furnace that is. There are different kinds of torches, and different kinds of burners, Meker burners, Bunsen burners, and more each have their own limitations. Common butane torches can reach 700 - 800C but to get higher the use of MAPP gas can bring you there with an appropriate torch head. Methane is also a good carrier of potential energy so hooking directly into your home gas line has its advantages.

Measuring these temperatures though pose their own difficulties. Common thermometers are fine for common temperatures, -30C - 300C can be found on many thermometers such as candy thermometers, but beyond this on either side of the temperature scale it becomes necessary to deviate from the norm. Bimetal thermometers that hook into electrician tools can reach 1000C but you will have to invest in a good probe and the thermometer itself is sensitive to chemical attack. There are three different types of probes, the most common is the K type, these can cost quite a bit, and you need to also purchase the reader to plug it into, and not all K type probes are the same, they have different ranges, always stay within the manufacturers temperature recommendations to prevent destroying your expensive probe. Another type of thermometer useful for high temperature is the infrared thermometer. Just point and click and you get an non-invasive temperature of up to 1000C for upper class models, but you will pay high for this too.

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

3.7 Removing water from gasses/solids/liquids with drying agents

Referring to the section on dehydrating agents and desiccants, section 4.6 you can find a list of compounds that are good for removing water. Both dehydrating agents and desiccants (which are also known as drying agents) can be used to remove water from a system, however the action of dehydrating agents is strong enough that it can attack chemical bonds (beyond any normal chemical reactions that may be associated with a compound) and this extra reactivity should be considered. Drying a chemical is a common procedure for matter in all of its common forms. The basic premise is to find a compound that really likes water, more so then the chemical you want to dry, and adding that chemical in such an amount to tie up the water present, then removing the desired compound from the desiccant which has since been used to remove the water. Some chemicals hold water very strongly and their dehydration proves to be very difficult, but generally procedures for removing water are simple and can be generalized as such:

Removing water from a solid: The most common method to remove water from a solid is to heat it to drive off the water or use heat in combination with vacuum. But this is not always be the case, and when a compound is subject to decomposition from heating, drying agents can be a reasonable thing to try. The same method to keep a solid dry can also be used to dry a solid that already contains water, the use of a desiccator. A desiccator is just an air tight container, with a desiccant (drying agent) at the bottom and an area suspended above the drying agent where a solid sample can sit. As the sample sits above the drying agent it looses water to the environment, in which almost no water is present, this water is then absorbed by the desiccant at the bottom. This process can occasionally take weeks, so patience is a key to a very dry sample. A less used method is also available to remove the water from organic compounds involving dissolving in the minimum of hot organic solvent in which water is insoluble, it will then, in theory, float to the top where it can be pipeted off and the solvent then removed by evaporation. Of course using non-aqueous environments and anhydrous reagents can ensure water does not get into the reaction environment at all and therefore not in the product to begin with although this can be next to impossible for some reactions.

Removing water from a liquid: Once a majority of water has been removed from a liquid usually through careful distillation or washing a fairly insoluble liquid with a saturated salt solution, drying agents can be good for getting out that last percent. The usual procedure is to take the liquid and add a bit of drying agent that is insoluble in it to the liquid. It is then stirred and if the drying agent at the bottom seems clumpy then more is added until the drying agent is free flowing, like dry sand between your hands. That is when most of the water has been removed. In some substances that are exceptionally hydroscopic though this process can take quite a while, the drying of ethanol with anhydrous copper sulfate can take days or weeks for example. The drying agent in this case does not have to be a solid, sulfuric acid can be used to dry some solvents that do not react with it and are not soluble in it such as bromine and acetonitrile.

Removing water from a gas: Gas drying tubes are available for plugging on the end of distillation apparatuses. What they are is a short length of wide glass tube, followed by a bulge that tapers down to thinner glass tube. The thinner part usually is attached to the exit/entry point for the gasses in the apparatus by a small length of rubber tubing. A piece of cotton is crammed in the larger part and the bulbous part filled with a desiccant such as calcium sulfate and another piece of cotton pressed on top of that, gasses entering the apparatus are thus treated to remove some of the water. In gas generation removing water from the gas produced is sometimes refered to as scrubbing (see the section on gasses), in this case the gas is bubbled through concentrated sulfuric acid or through a hygroscopic solid to remove water, sometimes three and four times before the gas is ultimately consumed.

3.8 Recrystalization

When the purity of a product is in question and you can spare a little bit in the quest for a more absolute product something can usually be worked out with recrystalization. The basic principle is to pick out a solid that you want purified, say, ammonium nitrate.

The next step is to find a suitable solvent for it. The solvent should posses the following properties:

1. Be able to dissolve a large quantity of the desired product when hot and have only a low solubility when cold.
2. Not affect the product be it by causing it to decompose or react with it.
3. Be useful under atmospheric conditions, not possess properties that are affected by heating and cooling.

Finding such a solvent is usually difficult. Many places do not list the solubility of a substance in anything but water, let alone finding hot and cold solubilities of a substance of different solvents on the same page. As such some trial and error may be involved, or research can help, finding out what solvent a pioneer in the field used to recrysatalize one of your products may be a good start.

Once your solvent is picked out, in the case of ammonium nitrate water can be used. The first step is to heat the water to a high temperature but not quite boiling, then to saturate the solution with as much of the solute as can be dissolved, if there is still solid solute in the solution either it can be spooned out or more solvent can be added. After the substance is all dissolved and while still hot a quick filtration can be used to remove insoluble materials such as glass/sand particles. Careful though, a hot saturated solution can crystallize on a cooler solid surface quickly and can plug filter paper, have something to scrape the filter paper with handy.

After your hot solution has been quickly filtered you let it cool. Usually once a certain temperature is reached crystals will automatically start to come out of solution. However on occasion a solution may become super-saturated, i.e. the solution should have crystals forming but there is nothing for them to form on, that is one way to look at it. In this case one of two things can be done, a crystal of the original compound can be added, this is called a seed crystal, and new crystals will grow off it. Or you can scrape the inside rim of your container right at the liquid air interface, this agitation can cause the growth of crystals.

Allow the solution to keep cooling but don't mechanically cool it too low, if for example you cool a water solution to near 0C then most of the impurities may crystallize out with your intended product defeating the purpose of recrystalization. But after you get a good crop of crystals then filtration is the logical next step. Vacuum filtration is the best as you may be filtering off a large quantity of solid but gravity filtration may work depending on your circumstances. Your freshly grown crystals should be washed while still in the filter with a few quantities of cold solvent to remove adhering particulates.

Now that you have your crystals they may need to be dried, in a desiccator or under high vacuum are the two normal choices, the desiccator being the most readily available to the amateur chemist. But this step may not be necessary, check in a chemistry book to see if the salt you produce is hydrated, if that is the case heating the salt will usually be necessary to create the anhydrous product, if your product is disquecent or hygroscopic a desiccator may be a good first choice, keeping it there for a few days may prove to be a good move, followed by immediate storage in an air tight container to prevent the re-entry of water.

In addition to cooling a solution to cause a precipitate of crystals another solvent can be added to the solution. In this case the solubility of a solid is less in both the solvents then it is in either one alone.

3.9 Measuring Weight and Volume

Quantitative chemistry requires one to know the amounts of reagents that are to be mixed for each reaction. Measuring weight is most easily done with a digital scale, those used for weighing mail are well within the price range of most chemists and more expensive ones are available if you want to splurge. A good scale should at least go down to measure by the gram, but down to the first decimal place is much better. Such scales usually go up to one kilogram or so and therefore cover a wide range of useful measurements. However if a digital scale is not available to you, there is always the old standby of a good old-fashioned balance. Using a balance requires a set of weights or other objects of known mass to balance against. The substitutes for a weight set are many but need to be a series of objects that are of a known mass and are nearly identical to one another, even something such as sheets of paper can work, or metallic currency, the weight of which is available with some searching online for each country. Knowing this one can make a rudimentary scale and balance against a known weight of currency and obtain the correct weight of your desired reagent. [Note that water at room temperature is roughly 1.0 g/ml and as such since it is easier to measure volume you can make a counter weight in a balance scale to the exact specification you want by adding the correct amount of water.]

Measuring volume is considerably easier as most grocery stores carry objects for just this task. In America however volumes are measured in the system of cups, quarts, pints, gallons, etc. The conversions for these measurements are readily avalible online and with some searching, even in American supermarkets one can find measuring tools that have some scale in milliliters. In a pinch one can use a container of known volume, be it a pop can or a milk jug, just for a rough approximation of the volume it contained.

4.0 Lab Reagent Types (Intro, discuss overlap, and generalization)

Rather then attempt to give you a chemical, name it, give you its properties, have you memorize those and move on to the next one I have organized this area to help you learn chemical properties more readily. There is a bit of generalization here and some overlap, however rather then learning the chemical then the properties, the purpose of this is to tell you the properties then give you a list of the chemicals that posses these properties along with a bit of relevant data for each. In doing this it is easier to go into deeper detail on exactly the designated title means not only under STP (Standard

Temperature and Pressure) but also under extraneous conditions that you might be required to work with them at.

4.1 Acid / Base Theory (Aqueous Solution)

pH

"pH"; everybody knows the term but what does it really stand for? And perhaps more importantly what is it good for?

pH stands for "potential of Hydrogen" (from the original German term "*potenz*"). It is a measure for the activity of hydrogen and because the activity of hydrogen in water equals the acidity of that water the pH effectively denotes the acidity of a solution. When hydrogen cations (H^+ ions) are introduced into water they react with water to form the *hydronium* ion (also referred to as the *oxonium ion*) which is denoted as H_3O^+(aq). The hydronium ion is the ion that gives acidic solutions their acidic nature. The direct opposite of the hydronium ion is the hydroxide ion (denoted as OH^-) which makes water alkaline. Water always contains H_3O^+ ions and OH^- ions (hydroxide ions) but in pure water they are in equilibrium which means they cancel each other out for as the acidity of the water is concerned. When an acid is added to the water the equilibrium shifts to the acidic end of the spectrum which means more H_3O^+ ions are present in the solution than OH^- ions. When a base is added the equilibrium shifts to the alkaline end of the spectrum which means more OH^- ions than H_3O^+ ions are present in the solution. A pH from 0 to 7 means the solution is acidic (so more hydronium ions than hydroxide ions); a pH of 7 means the water is neutral (there are as many hydroxide ions as there are hydronium ions present in the solution) and a pH from 7 to 14 means the solution is alkaline (more hydroxide ions than hydronium ions).

The highest attainable pH at STP (standard temperature and pressure) is 14 and the lowest attainable pH at STP is 0. When the temperature and pressure do not conform perfectly to STP the minimum and maximum pH will vary accordingly. This is however not essential knowledge for hobby-chemists and as such I will not go into it any further.

Two kinds of acids and two kinds of bases.

There are two kinds of acids and bases; **strong** and **weak**. The strong versions do not form equilibriums in water but simply completely dissociate. The weaker versions will form an equilibrium in water and as such they will generally not be nearly as acidic or alkaline as the strong version.

Strong acids	In solution
Hydrochloric acid (HCl)	H_3O^+(aq) + Cl^-(aq)
Sulphuric acid (H_2SO_4)	H_3O^+(aq) + HSO_4^-(aq)
Nitric acid (HNO_3)	H_3O^+(aq) + NO_3^-(aq)
Perchloric acid ($HClO_4$)	H_3O^+(aq) + ClO_4^-(aq)
Hydroiodic acid (HI)	H_3O^+(aq) + I^-(aq)

Hydrobromic acid (HBr)	$H_3O^+(aq) + Br^-(aq)$

Weak acids	**In solution**
Ethanoic acid (*Acetic acid*)	$C_2H_3OOH(aq)$
2-hydroxy-1,2,3-propanetricarboxylic acid (*Citric acid*)	$C_3H_4OH(COOH)_3(aq)$
H_3BO_3 (*Boric acid, aka* $B(OH)_3$)	$H_3BO_3(aq)$
HF (*Hydrofluoric acid*)	$HF(aq)$
H_3PO_4 (*Phosphoric acid*)	$H_3PO_4(aq)$

The first difference that catches the eye is the fact that strong acids are denoted as free constituent ions and that weak acids are denoted as {weak acid}(aq). This is because the strong acids **always** completely dissociate in water whereas the weak acids only very partially dissociate in water.

Strong base	**In solution**
KOH (Potassium hydroxide)	$K^+(aq) + OH^-(aq)$
NaOH (sodium hydroxide)	$Na^+(aq) + OH^-(aq)$
$Ca(OH)_2$ (calcium hydroxide)	$Ca^{2+}(aq) + OH^-(aq)$
$Ba(OH)_2$ (barium hydroxide)	$Ba^{2+}(aq) + OH^-(aq)$
$Na(C_2H_5O)$ (sodium ethoxide)	$Na^+(aq) + OH^-(aq) + C_2H_5OH(aq)$

Weak base	**In solution**
K_2SO_4 (potassium sulfate)	$2K^+(aq) + OH^-(aq) + HSO_4^-(aq) \leftrightarrow 2K^+(aq) + SO_4^{2-}(aq)$
K_2CO_3 (potassium carbonate)	$2K^+(aq) + OH^-(aq) + HCO_3^-(aq) \leftrightarrow 2K^+(aq) + CO_3^{2-}(aq)$
KF (potassium fuloride)	$K^+(aq) + OH^-(aq) + HF(aq) \leftrightarrow K^+(aq) + F^-(aq)$
Na_2HPO_4 (sodium biphosphate)	$Na^+(aq) + OH^-(aq) + H_2PO_4^-(aq) \leftrightarrow Na^+(aq) + HPO_4^{2-}(aq)$
NH_3 (ammonia)	$NH_4^+(aq) + OH^-(aq) \leftrightarrow NH_3(aq)$

The obvious difference here is that in the case of the weak bases they are denoted as equilibriums when in solution whereas the strong bases (which do not form equilibriums) are denoted simply as their respective constituent ions in solution (so with "(aq)" at the end).

Calculations.

The pH of a solution can be calculated as follows:

$$\mathbf{pH = -log[H_3O^+]}$$

please note here that $\mathbf{[H_3O^+]}$ denotes the hydronium concentration and that the addition of the p (for potential) effectively means you take the negative logarithm of the value without the p (in this case negative logarithm of the hydronium concentration). So when I take 2 moles of the "strong acid" HCl(g) and add enough water to make a solution of 1L of water I will have a 2M solution of hydrochloric acid. The pH of that solution would be –log(2) = -0.30 M = $-3 * 10^{-1}$. Which not only means the solution would be rather acidic but also immediately shows that the pH of a solution might be realistically expected to be between ~ -1,5 and ~ 15,5 rather than between the limits that are predicted by the rule. Don't worry it really doesn't make much of a difference when you're using these formulae.

An important conclusion that can be drawn from this formula is that one can easily calculate the hydronium concentration (and as such the amount of hydrogen cations in solution) by means of the following calculation:

10^{-pH}

So when a solution has a pH of 3,7 the $[H_3O^+] = 10^{-3.7} = 0{,}0001995 = 2 * 10^{-4}$ mole/L (can also be denoted as $2 * 10^{-4}$ M).

Ka and pKa

The degree to which an acid will dissociate in water is denoted by means of its **Ka**. The Ka is basically the dissociation constant of the acid in water. Ka's can be denoted in a simpler form by taking their negative logarithm to yield the **pKa**.

$$Ka = \frac{[H_3O^+][X^-]}{[HX]}$$

pKa = -log(Ka)

These formulae can be used to calculate the amount of hydrogen cations and the and the pH of a solution of a certain strong acid. The calculations for a base can be performed in the same way but using the Kb in stead and the result of the calculation would yield the $[OH^-]$ which can be transformed into the pH by means of the following formulae:

$-\log[OH^-] = pOH$

14,00 – pOH = pH

This is because pH + pOH for a certain solution at STP must always equal 14,00. Also the Ka of a base can be calculated by means of the following formula:

pKa + pKb = pKw (which means KaKb = Kw = 10^{-14})
pKw = 14 (which means Kw = 10^{-14})

10^{-pKa} = Ka

To calculate the pH of a solution of a weak acid (or the pH of the solution of a weak base) the following formulae can be applied:

$$Ka = \frac{[H_3O^+][X^-]}{[HX]-[X^-]}$$

This is because only a small portion of the weak acid will dissociate. You can calculate by calling $[X^-]$ and $[H_3O^+]$ x and filling out the equation. Some simple calculus should yield the $[H_3O^+]$. It is useful to have a calculator handy in that case because it will be rather laborious to calculate x.

<u>E.g. a 0,1M solution of formic acid (HCOOH)</u>
pKa = 3,79 ➔ Ka = 1,6 * 10^{-4}
[HCOOH] = 0.1

Fill out the formula, do the math and the answer will prove to be 3.9 * 10 ^{3}. (hence the pH will be ~2,4)

4.2 Acids (Organic/Inorganic)

The most loose definition of acids that most people are familiar with defines an acid as a chemical that is able to donate a hydrogen cation to water. In doing this it generates the H3O+ cation which is the acidic component of water. However this does not cover every acid under every circumstance by a long shot. Never the less, water is a common solvent and in defining an acid it is easier to use definitions governed by water then add in the exemptions later for non aqueous systems.

Common Acids	
Acetic Acid H_3COOH	Commonly known as vinegar, this acid forms no confirmed azeotrope with water. It is somewhat strong in concentrated form, dissociating to an appreciable extent. Acetate salts are usually soluble and are therefore a good source of metal ions in solutions, however solutions are slightly basic.
Hydrochloric Acid HCl (Muratic Acid)	Sold as a solution in water of HCl gas, hydrochloric acid is a strong mineral acid. The commonly available forms are 20% (The azeotrope), 38% (concentrated with a density of 1.19 g/cm^3) It will attack anything in the reactivity series above hydrogen, most chlorides are at least slightly soluble.
Nitric Acid HNO_3	Not commonly available, which is a shame considering how useful it is. Nitrates of metals are all soluble so it

	provides a good ability to solvate a cation of your choice. It is a strong oxidizing acid, able to oxidize metals readily at room temperature evolving nitrogen oxides.
Sulfuric Acid H_2SO_4	The staple acid of at home chemistry. Very difficult to obtain in some countries but relatively easy to find in America. Most sulfates are soluble in water although there are some notable exceptions ($CaSO_4$, $BaSO_4$, and $PbSO_4$). Concentrations vary widely form common battery acid (~30%) to additional acid anhydride dissolve in 100% H2SO4. Weakly oxidizing.
Sulfamic Acid NH_2SO_3H	Somewhat readily available and stronger then many organic acids. It forms may highly soluble salts, in the pure form it is a solid.
Boric Acid $B(OH)_3$	Very weak acid. Borates are readily available in the cleaning industry, dehydrates easily to boric oxide. Boric acid readily forms boric esters, which burn to give beautiful colors.
Hydrofluoric Acid HF	Weak solutions are available over the counter for cleaning rims of cars and such (~3%). Hydrofluoric acid is very toxic and highly concentrated solutions can kill very rapidly if splashed on the skin. As an acid though it is somewhat weak compared to hydrochloric, as a pure compound it is a liquid near room temperature.
Cyanuric Acid $HOCHC(OH)NC(OH)N*2H_2O$	Somewhat weak acid available for adjusting the pH of pools.
Phosphoric Acid H_3PO_4	Concentrated phosphoric acid is a fairly strong acid. It readily attacks metals forming phosphates, which are on the whole soluble in water. The more concentrated the solution the more syrupy it is until it becomes a solid. Phosphoric acid will not boil, it will continuously loose water even past where it is 100% dehydrating to other forms of phosphoric acid such as pyrophosphoric acid. Avalible for cleaning metal and for marine cleaning.

Metal Activity Series:

Time to introduce you to the metal activity series. Although important to other chemistry concepts it answers one question regarding acids that people ask most often. “What will an acid dissolve?” Below is a list of elements, towards the end of the list is hydrogen, and anything to the left of it will dissolve to some extent in acid. Those in the lighter color to the immediate left dissolve slowly-very slowly, going to the darker color even more to the left we find elements that will not only displace hydrogen from an acid but will react with steam. Finally those furthest to the left will readily react with water and their subsequent reaction with acid would only be described as intensely violent.

This is a standard activity series, some series will have elements in slightly different relation to one another but this is the basic order.

Li K Ba Ca Na Mg Al Mn Zn Cr Fe Cd Co Ni Sn Pb (H_2) Cu Ag Hg Pt Au

So you're looking at the list and you wonder, "What about those elements to the right of hydrogen?" A good question, those elements will not displace hydrogen from acid and as a consequence they could be considered inert in that respect. But that would be a mistake to assume they would remain inert in all respects. There is a way around this inertness, the addition of an oxidizing agent. The principle, let's say for example you have a piece of copper that has some surface oxidation, now let's say you put it into some hydrochloric acid, immediately the oxidized layer dissolves off tinting the acid a green/blue color. Pulling out the copper it looks fresh and clean, no oxidation. So, the oxidized layer dissolved, if you were to leave it out the oxygen in the air would re-oxidize that top layer, you could dip it back into the hydrochloric acid, and dissolve yet more of the copper. In this case the atmospheric oxygen is the oxidizing agent, bubbling air though HCl while dissolving copper accomplishes this. But another way would be to add an oxidizing agent to your acid, an even better way would be to have an oxidizing acid. Perchloric acid ($HClO_4$) and nitric acid (HNO_3) are both oxidizing agents as well as acids [Hot concentrated H_2SO_4 is also an oxidizing agent], as a matter of fact nitric acid almost always functions as an oxidizing agent unless coupled with a very reactive metal, magnesium will actually liberate hydrogen for the first few seconds of reacting with nitric acid but after that it will be preferably oxidized first. Oxidizing acids will not dissolve some elements that have insoluble oxides, the formation of the oxide forms a protective layer pacifying the metal to further attack, a good example is aluminum in concentrated HNO3, also tin can be pacified in this way under some conditions.

Oxidizing Acids

By being an oxidizing agent the acid must simultaneously be reduced in the reaction. Therefore when copper is subjected to the action of nitric acid copper is oxidized and the nitrate anion is reduced to any of a number of nitric oxides depending on the conditions under which the oxidation took place. Here are some examples of the reactions of nitric acid:

$$2HNO_{3(aq)} + Mg_{(s)} \Rightarrow Mg(NO_3)_{2(aq)} + H_{2(g)}$$

Rarely occurs, only happens initially with magnesium or even more reactive metals [Na, K, Li, etc.], not important.

$$3Cu_{(s)} + 8HNO_{3(aq)} \Rightarrow 3Cu(NO_3)_{2(aq)} + 4H_2O_{(l)} + 2NO_{(g)}$$

This is an example of nitric acid acting as an oxidizing agent when dilute.

$Cu_{(s)} + 4HNO_{3(aq)} \Rightarrow Cu(NO_3)_{2(aq)} + 2H_2O_{(l)} + 2NO_{2(g)}$

This is an example of nitric acid acting as an oxidizing agent when concentrated. Notice the ratio of nitric acid molecules reacting with copper compared to the dilute reaction above. [Note, a picture of this reaction is shown in the picture in the preceding section]

$P_{4(s)} + 20HNO_{3(aq)} \Rightarrow 4H_3PO_{4(aq)} + 20NO_{(g)} + 4\ H_2O_{(l)}$

Concentrated nitric acid can also oxidize elements such as phosphorus, silicon, sulfur, and occasionally carbon, especially when heated.

$Fe_{(s)} + 6HNO_{3(aq)} \Rightarrow Fe(NO_3)_{3(aq)} + 3\ H_2O_{(l)} + 3NO_{2(g)}$

When metals capable of multiple oxidation states are dissolved in concentrated nitric acid they will usually take the highest normal oxidation state, in this case iron becomes +3 in preference to +2.

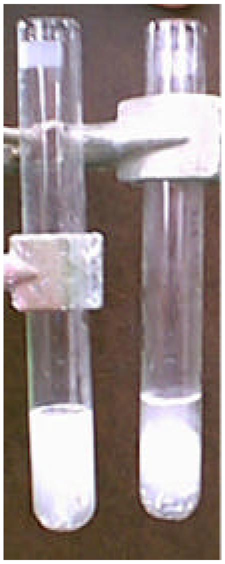
Similarly, when copper or mercury, some of the more reactive of the metals that follow hydrogen in the activity series, come into contact with hot concentrated sulfuric acid they can be oxidized and the sulfuric acid reduced.

$Cu_{(s)} + 2H_2SO_{4(l)} \Rightarrow 2H_2O_{(l)} + SO_{2(g)} + CuSO_{4(aq)}$

Perchloric acid is encountered to a considerably lessened extent in the laboratory, it has a nasty reputation for exploding for no reason, generating out of control reactions, creating fire hazards, and making unstable salts.

Another thing to consider when pondering weather a metal will dissolve in an acid is weather the salt formed would be soluble. One would not logically think that silver would dissolve in hydrochloric acid independent of its unreactivity simply based on the fact that the silver chloride thus formed is totally insoluble. Even a piece of barium metal tossed in H2SO4 may become pacified which is an amazing thing considering it would react very rapidly with water. Oxidizing ability aside there is another method to measure the strength of an acid, the pH scale and the pKa scale, which were discussed in the opening section.

4.3 Bases

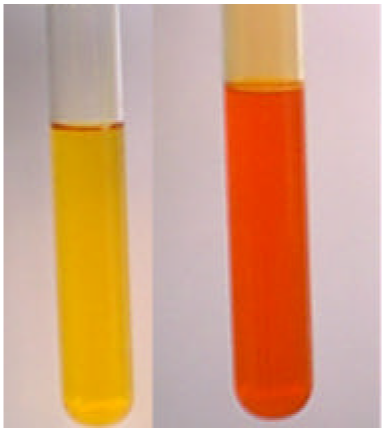


As shown in the above picture bases can rapidly attack some metals just as acids can. To the left in the above picture some aluminum turnings have been placed into a weak potassium hydroxide solution, to the right a weak acid solution is also attacking a similar amount of aluminum. Hydroxides can attack a number of metals, especially when hot and concentrated, however the reactivity shown with aluminum, zinc, and magnesium can be considered special cases for the common metals.

Common Bases	
Sodium Hydroxide NaOH	Avalible over the counter as lye, sodium hydroxide serves the purpose of being the no-nonsense base, addition of sodium hydroxide to an aqueous solution automatically increases the hydroxide ion concentration and brings only the sodium cation along with it.
Sodium Carbonate Na_2CO_3	Sodium carbonate is available as "Washing soda" it is usually the decahydrate (*10H2O) but that does not interfere with calculations as long as it is accounted for. Be wary of other impurities though. Sodium carbonate is a great base because the reaction with acidic components is driven foreword strongly by the loss of carbon dioxide from solution.
Sodium Bicarbonate $NaHCO_3$	Less basic in solution then sodium carbonate but still able to neutralize acids well. It is safer on the skin and is therefore the choice base to have laying around in case of an acid spill.
Ammonia NH_4OH	Ammonia gas can simply be bubbled into solution to increase its pH. That is a great advantage to ammonia. Also it can be forced from solution after its purpose has been served, the gas itself will react with acids even if they

	are not aqueous either.
Trisodium Phosphate Na_3PO_4	Basic in water solution due to the equilibrium present between the phosphate anion and the hydrogen phosphate anion and the dihydrogen phosphate anion which take up hydrogen from the water and therefore leave hydroxide anions. This base is available as prills for a stripping agent in painting.

4.4 Oxidizing Agents



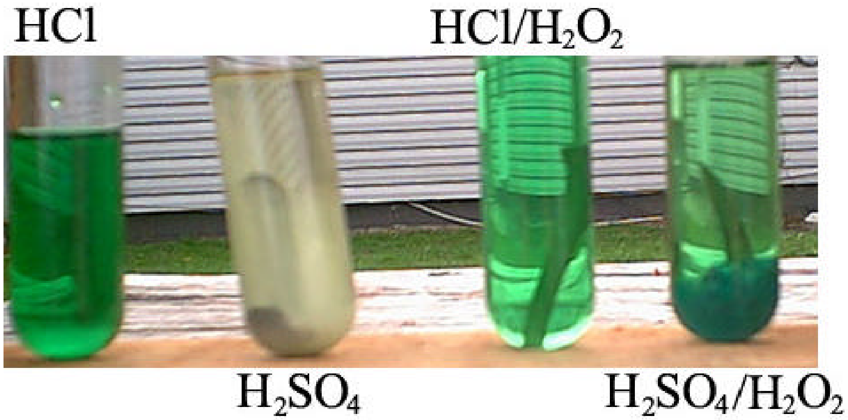
The case to the left shows the effect of hydrobromic acid on hydrogen peroxide. Whereas acidic peroxide solutions are one of the possible oxidizing agents that one can pick from, using hydrobromic acid/H2O2 solutions is not advisable. The hydrobromic acid will act as a catalyst to decompose the H2O2 resulting in lessened yields, and in addition, the oxidation potential of the mix is enough to oxidize Br- anions to elemental bromine. This is clearly shown, initially the H2O2 and the HBr solutions were clear, when mixed they immediately turned yellow, and upon standing for a minute or so the mix was a deep red with bromine vapors clearly stagnant above it. Just goes to show you that you need to consider even the smaller things when attempting oxidation reactions.

Common oxidizing agents	
Potassium Perchlorate $KClO_4$	Solid, white powder, non-hygroscopic, very slightly soluble in water, usually has to be bought from a pyrotechnics supplier or made via electrolysis.
Sodium Nitrate $NaNO_3$	White powder soluble in water, hygroscopic, slightly saline/bitter taste (don't taste it!). Acid solutions will attack noble metals such as copper. Occasionally available during the summer months as fertilizer.

Nitric Acid HNO_3	Clear - Yellow/Green liquid. Available in various concentrations, >70% show remarkable oxidizing capabilities, lower concentrations available over the counter for hydroponics.
Hydrogen Peroxide H_2O_2	Clear liquid, available in various concentrations from 2% to 99% solutions greater then 50% should be treated with care as combination with many things can cause them to explode. Greatly attacks tissue.
Potassium Dichromate $K_2Cr_2O_7$	Bright orange solid, soluble in water. Solutions of potassium dichromate with sulfuric acid were once one of the most routine things to clean lab glass with. Potassium dichromate is considered carcinogenic.
Sodium Hypochlorite $NaClO$	Clear-Yellow/Green liquid strong chlorine type smell. Surprisingly good widely available oxidizing agent. Considerably more powerful in concentrations greater then 12.5% and especially when hot.
Sodium Chlorate $NaClO_3$	White solid available as a weed killer in some areas. Toxic and hygroscopic it has powerful oxidizing powers as a solid, when heated on its own it undergoes self oxidation-reduction to perchlorate and chloride.
Potassium Permanganate $KMnO_4$	Bright purple solid possessing great oxidizing ability as a solid and in either basic or acidic solution. Found as a treatment for water in areas where iron is a problem.

Aqueous Oxidations:

Hydrogen Peroxide Solutions



Shown above is an attempt to dissolve nickel metal under various conditions. Although not totally apparent the HCl solution and the H2SO4 solution showed little attack. The HCl/H2O2 solution did show some attack. However it was the

H2SO4/H2O2 solution that showed incredible results. As you can see the entire top of the nickel in the test tube to the far right has eroded to a point. In addition the whole bottom of the test tube is full of nickel (II) sulfate crystals. The mix of H2O2 with H2SO4 also looks entirely different from just H2SO4 acting alone, seen in the second to left picture, a cloudy mixture formed in that instance unlike the superb green mixture formed from H2SO4 reacting in tandem with H2O2. The reason for this is H2O2 increases the process by oxidizing the noble metal, once the surface is oxidized the oxide dissolves in the acid, and once it dissolves in the acid the H2O2 can oxidize the surface again.

4.4a Molten Salt Oxidations / Solid State Oxidations:

With molten salt oxidations one can force metals into oxidation states that would be very difficult to achieve in the aqueous phase and would be considerably less stable if formed in that way as well. The actual chemistry of such oxidations is usually complex but there are only two simple needs to perform most of these oxidations, an alkali metal hydroxide, and an oxidizing agent usually an alkali metal nitrate where the gaseous visages of the oxidizing anion might readily leave the melt and the remaining cation will not interfere. Several reactions go on in such melts, but mixtures involving potassium hydroxide make a good example:

$$2KOH \Leftrightarrow H2O + K_2O$$

$$4KOH + 3O_2 \Leftrightarrow 4KO_2 + 2H_2O$$

Now, peroxides and superoxides are strong oxidizing agents in their own right, but the oxide anion O^{2-} is incredibly basic as is the superoxide $O^{-1/2}$ (note that potassium peroxide is fairly unstable, and does not exist appreciably in the molten state). The oxidizing agent in the melt, usually something like potassium nitrate helps to drive this equilibrium, acting as a very convenient source of oxygen. At these temperatures things like potassium nitrate are very reactive, a cotton glove for instance, coming into contact with molten KNO_3 will burst into flames, but at room temperature KNO_3 could be safely handled with ones bare hands. All in all, the very basic electron rich environment can best stabilize a number of high oxidation state compounds, which can then be used in further chemical endeavors.

The melts used for these oxidations are fairly corrosive; vessels of nickel, platinum, and silver are best. Glass is out of the question, as the strong bases present will attack it. However for the roughest oxidations, disposable vessels of steel or commonly available pipefittings can work. These will contaminate products obtained but compounds formed under these conditions are not going to be very pure anyways. Here are a few examples of high oxidation state compounds that can be made by these methods:

Ferrate $[FeO_4]^{2-}$: Ferrates will decompose almost instantly if in acid solution, quickly in neutral solutions, and slower in basic solutions. Kept free of moisture and stored without access to air, ferrates will keep for several weeks or months. They are made by fusing ferric oxide with KOH and an oxidizing agent and are purple/red in color. Ferrates can be precipitated from an aqueous solution as the slightly soluble barium salt or by concentrated potassium hydroxide solution.

Bismuthate $[BiO_3]^-$: Bismuthate as with many other high power oxidizing agents is available from chemical suppliers usually only as a purity of 85% or so, further refinement being unnecessary due to some of the brute force type oxidations done with it. Out of these four this is the second most commercially available oxidizer listed. It can oxidize manganese ions in solution to permanganate and is usually found for sale as the sodium salt. It can be prepared by fusing bismuth trioxide with potassium hydroxide as long as the mix is exposed to air.

Chromate $[CrO_4]^{2-}$: This is the most widely available oxidizer listed here. Chromates are toxic and should be handled with care. They are also the weakest oxidizer on this list. They are usually formed by fusing chromium (III) oxide, acidification of a solution of chromate will lead to the formation of dichromate which will usually precipitate if the concentration is high enough and the temperature lowered afterward, dichromates being more useful then chromates. Industrially this process is used to make dichromate by fusing chromite ore ($FeCr_2O_4$) with potassium hydroxide in the presence of oxygen, after oxidation the mixture is dissolved in water and acidified, the chromate being converted to dichromate and the ferrate going to soluble Fe^{3+}.

Manganate $[MnO_4]^{2-}$: Manganates are green in color and the product of fusing manganese dioxide. They are fairly unstable and upon addition to water and acidification yield a solution of permanganate. Subsequent filtering and crystallization allowing for the production of permanganate at home, which is useful as well.

When an oxidation is completed there are two courses of action, if the product is stable to the atmosphere it can be poured onto a sheet of steel and allowed to cool quickly, then broken with a hammer and stored. However if it is not then it must be covered in the crucible while covered and once cooled chipped out and stored. In either case these melts are very strong oxidizing agents and must never come into contact with anything organic or flammable. In addition these melts can **NEVER** be poured directly into water while in the molten state as they can very often explode. Temperature control is not a major issue with most molten oxidations but things should still never be heated too strongly, molten oxidations work best in the range from 375– 550 °C and the amount of time to hold the reactants there depends strongly on what you are trying to oxidize and the amount you have in the mixture. If you desire to produce an oxidizing agent that is very unstable at high temperatures considering an eutectic mixture can help greatly, a 50/50 mixture of NaOH/KOH has a significantly lower melting point then either component alone.

Aside from the inherent risks of holding oxidizing mixtures at high temperatures, the use of nitrates can lead to the formation of nitrogen oxides which are extreme hazards therefore the actual heating step should be preformed while you are not in the company of the reaction vessel. Additionally the subsequent dissolution and acidification of some of these mixtures can also lead to nitrogen oxide release if the reaction yielded a large amount of nitrites as can often be the case. Not to mention that the oxidation reactions also have the possibility of going awry, if excessive frothing or sparks start to come from a reaction mixture that is your key to exit the area. One final note, chlorates and perchlorates can be used for these oxidation reactions, however acidification of a solution of chlorate can yield explosive quantities of chlorine dioxide and additionally the reaction itself can run away in the presence of certain metal oxides.

4.5 Reducing Agents

The most common class of reducing agents one runs across are usually the active metals. In pyrotechnics aluminum, magnesium, and occasionally zinc are used with strong oxidizing agents such as perchlorates and nitrates to give spectacular exothermic reactions. However on a more controlled level these metals can also be used to give reliable reactions even in reactions involving aqueous reactants. Additionally there are a number of organic reducing agents which are most popular in the field of organic chemicstry. Both organic and strictly inorganic reducing agents are of great utility in the chemistry lab however they are more difficult to obtain then oxidizing agents in most cases and in the case of the metals, usually difficult to get into a workable form.

Reducing Agent	Source	Example of Use
Aluminum Al	Pyro suppliers, scrap yards, foil	Powdered aluminum is a powerful solid state reducing agent (Thermite reactions), it can also work to reduce cations in the aqueous phase and can be amalgamated with mercury for organic reductions.
Magnesium Mg	Scrap yards, camping suppliers (fire starter), pyro suppliers, cell phone pieces and in some other high end applications (bike	Powdered magnesium is a stronger reducing agent then aluminum, it reacts slowly with water reducing it. Magnesium can reduce a number of

	frames etc.)	inorganic compounds such as NaOH works in thermite type reactions.
Hydrogen H2	Reaction of a strong mineral acid with an active metal (aluminum, magnesium, iron)	Reductions with hydrogen gas usually take place at elevated temperatures when concerning inorganics and under high pressures with organics, it is very useful but difficult for an amateur to use.
Lithium Li	Purchased from a chemical supplier, some batteries contain lithium, home electrolysis in non-aqueous medium or of electrolysis of lithium chloride/bromide eutectic.	Lithium metal finds use either alone or in compounds for the reductions of organic compounds, lithium dissolved in ammonia or n-butyl lithium being incredibly strong organic reducers.
Sodium Na Potassium K	Purchased from chemical supplier, made at home via electrolysis or reduction of salts.	Considering the high reactivity for each of these they find little practical use in but are occasionally called upon. A liquid eutectic is formed between these two elements with is unbelievably reactive.
Carbon C	Graphite, sugar carbon (formed by heating sugar till it decomposes), coal, charcoal	Carbon is an excellent reductant however its use requires very high (>900C) temperatures.
Sulfite SO_3^{2-}	Some OTC sources, bubbling sulfur dioxide into basified water.	Sulfites are oxidized to sulfates and in the process function as weak reducing agents.
Citric (Ascorbic) acid C6H8O7*H2O	Vitamin C tablets, sold as citric acid for flavoring, extracted from lemon juice	Weak aqueous reducing agent, works better at higher temperatures, good for making metal powders of somewhat nobel metals (e.g., nickel, chromium, silver, etc.)

Runaways can happen with reductions as well as oxidations.

Chemistry can be fun and educating in many ways but one should always remember to be careful. Sometimes one just forgets to do the background work first, before moving to do the actual experiment.. That happened to me some time ago.

I had previously prepared few moles of nitrotoluene and planned to reduce some of it to toluidines. So, I remembered the standard Sn and Fe reductions of aromatic nitro compounds with HCl and did a few calculations on the amounts of reactants required. Ended up using 1mol of nitrotoluene and the appropriate amounts of 40 micron, hydrogen reduced Fe powder and 37% aqueous HCl. The nitrotoluene was mixed well with the Fe powder and a little distilled water in a 500ml erlenmeyer flask. I was first going to use a magnetic stirrer to efficiently keep the iron powder suspended but remembered that iron is magnetic, luckily before I dumped in the stirbar.. HCl was put into an addition funnel and was added drop by drop to the mixture, which I swirled continuously. I expected a fast temperature rise but there was none. HCl addition was continued a bit faster and then the temp finally started to rise. Stopped the addition for a moment and swirled strongly. After continuing the addition the temp didn't rise much more so I decided that I'd add half of the remaining HCl now and the rest after ten minutes or so. That was big mistake. Soon the temp started to rise fast. I swirled the flask as strongly as I could but it didn't seem to help. I took the thermometer off as it was nearing the limit (100C). I put the flask in to an ice bath and swirled vigorously. The flask started to feel _very_ hot at that time and I was ready to dump it in the bath, but I was too late. Suddenly the mix started to boil and shoot itself out of the flask. I had to let go off the flask as it was so hot and then the reaction got so vigorous that it shot all the remaining liquid out in a geyser like fashion on the floor. I quickly took a bottle of water and poured it in and over the flask. After I had the reaction tamed I decided to go outside and take the annoying gasmask off for a while. I immediately smelled the slightly irritating smell of nitrotoluene and realised that all the reaction mixture had probably boiled out of the flask with the steam.. The whole neighbourhood smelled of nitrotoluene for a few hours, as it wasn't windy. The flask seemed ruined but I got it eventually cleaned with some HCl and sodium ethoxide solution. The worst part was cleaning the mixture off the coarse concrete floor.. And the smell stayed for ages in my lab.

That certainly thought me a lesson to always find about different reactions and the possible mistakes that could be made during it. And not to start doing a new reaction with that much material. Starting from mill moles is much more recommended to get a feel for the reaction, before scaling up!

4.6 Dehydrating Agents/ Desiccants

In the case of the above picture nickel chloride is shown. The kernel on the right being an anhydrous lump, and the green solution on the left being the same amount solvated in water. This is just one example of a hygroscopic salt that changes color when hydrated. Mind you, by being hygroscopic, a salt is not at the same time disquecent. A disquencent salt will pull enough water from the air to put itself into a solution, an example being NaOH or CaCl2, a salt that is hygroscopic, but not disquecent, will form a stable solid hydrate that is more easily handled. Another example of a color changing salt that forms a stable hydrate is copper sulfate, which is colorless when anhydrous but turns blue when its water removing capacity has been used up, it can be reactivated for use by heating for an extended period of time.

The main use of desiccants is to remove water, usually from a liquid or gas to render that liquid or gas largely free of water in reactions where water might inhibit a desired reaction, interfere with a reaction, or cause extraneous byproducts. Drying agents can form very strong bonds to water, strong enough to take water from a chemical bond, such as the reaction between concentrated sulfuric acid and sugar, where the sulfuric acid will remove the water from the molecule $C_6H_{12}O_6$ leaving behind just carbon in a very pleasing visual display, phosphorus pentoxide, and hot NaOH are the only other drying agents on this list that posses this strength of drying power, agents of this type are referred to as dehydrating agents.

Chemical Name /Formula /Formula of hydrate	Form of anhydride / Form of hydrate	Details on Agent
Sulfuric Acid H_2SO_4 $H_2SO_4*xH_2O$	Dehydrating Acid Heavy Liquid	Heavy liquid, dehydrating action most apparent at high concentrations 90%+, concentrations higher are possible by dissolving the acid anhydride (SO_3) in concentrated H_2SO_4, such solutions (called oleum) possess additional dehydrating strength, but remains liquid, will dehydrate sugar to carbon. Great for drying liquids.
Phosphorus Pentoxide P_2O_5 H_3PO_4 or H_3PO_3	Solid/Powder (becoming plastic like/liquid) Dehydrating Agent	Solid/Powder formed by burning phosphorus in air. Disquecent, pulling water from air making a crust on the surface forming differing phosphor acids, phosphinic acid, phosphoric acid, etc. Very strong dehydrating agent, forms N_2O_5 from concentrated HNO_3.
Magnesium Sulfate $MgSO_4$ $MgSO_4*7H_2O$	Drying Agent Solid Forms stable hydrate	White powder, commercially the heptahydate $*7H_2O$ is available, however this can be dehydrated in an oven maxed out for a few hours. There is no color change upon hydration or dehydration. $MgSO_4$ is cheap and decent for drying some gasses and liquids however its action is not very strong. The hydrated salt is a solid.
Calcium Chloride $CaCl_2$ $CaCl_2*2H_2O$ (But will go further)	Drying Agent Solid (anhydrous) Liquid (hydrated)	$CaCl_2$ is widely available for use as deicing or as a drying agent for use in basements. It comes in the form of solid prills that will suck moisture from the air until they turn into a puddle. The drying action of this solid is similar to anhydrous $MgSO_4$ but the liquid hydrated state may run back into reactions.
Copper Sulfate $CuSO_4$ $CuSO_4*5H_2O$	Drying Agent Solid (Colorless) Green/Blue	Widely available for killing roots in sewer lines or preparable by dissolving copper in hot sulfuric acid, it can be made anhydrous by heating. Solid that changes color when its drying action is used up. Good for dying alcohols and such, action is stronger then

	when hydrated	$CaCl_2$ or $MgSO_4$ and it is regenerateable over high heat.
Calcium Sulfate $CaSO_4$ $CaSO_4*2H_2O$	Drying Agent Solid White Forms a stable hydrate	Available over the counter as the semi-hydrated ‘Plaster of Paris’ or as drywall, which is the dihydrate. Made anhydrous by heating is possesses decent drying abilities but its ready availability and low cost make it somewhat desirable, color changing versions are available from chemical supply companies.
Magnesium Perchlorate $Mg(ClO_4)_2$ $Mg(ClO_4)_2*6H_2O$	Drying Agent Solid but liquid when hydrated	One of the kings of the drying agent world, magnesium perchlorate possesses exceptional drying ability. However it can explode when exposed to solvent vapors or intense heat and therefore it has fallen into disuse, concentrated sulfuric acid or phosphorus pentoxide often substituted for it.
Sodium Hydroxide $NaOH$ $NaOH*xH_2O$	Dehydrating Agent Solid but liquid when hydrated	Available over the counter, store bought NaOH contains some impurities and is of a variable composition of sodium oxide and water. It is a good drying agent for taking the last bit of water out of liquids in which it is insoluble but does not have the ability to dry large amounts well, just small amounts of a liquid/solid/gas thoroughly.
Calcium Oxide CaO $Ca(OH)2$	Drying Agent Solid forming a stable hydrate	Widely available for adjusting the pH of soil, the calcium hydroxide thus formed is only slightly soluble, although regenerateable through heating it is more often then not simply used once.

The activity of a dehydrating agent, the ability of it to pull water from its surroundings is not usually something to be directly gauged, but there is a great difference in each drying agents ability to pull water and trap it. For example, sulfuric acid when concentrated will char wood and turn sugar to coal. Whereas calcium chloride will do neither. These aspects can make a great difference in their usage and what reaction they may be unsuited for.

4.7 Poisonous Reagents

A poisonous reagent might be easily classified as a chemical that requires only minimal unintentional contact to cause adverse effects. Such a definition is better suited for this class then simply a chemical that can cause harm, after all, there is a lethal dose for table salt and alcohol, so it is better to only classify those chemicals that could easily cause harm to oneself thought an accident as poisonous. From here poisonous chemicals are further divided into two categories, not independent from one another. Those chemicals that are cumulative poisons, and those that are not.

A cumulative poison is a poison whose presence in the body is not immediately eliminated and it accumulates in the system, i.e., it would be easy for a person to take in more of this poison, however infrequently, then their body will expel. This can be referred to as the half-life of a substance. Examples of cumulative poisons are lead salts, fluoride, radioactive strontium, and others. Cumulative poisons can also have almost no half life in the body, but are instead cumulative in the effects they cause, long term lung damage can result from inhalation of even minute amounts of some chemicals and upon repeated exposures that damage might become severe enough to cause emphysema or other conditions.

One mistake people take into account when handing a potentially poisonous substance and assessing its lethality is to consider the time frame over which it is lethal. Poisons that can kill in minutes such as hydrogen sulfide and hydrogen cyanide are often viewed with considerable more trepidation then other gasses like nitrogen dioxide, simply because nitrogen dioxide may not kill instantly. It still caries with it significant danger, all three of them do, and it is not a matter of will it kill you instantly or eight hours from now, it’s a matter of if the chemical you are dealing with is dangerous and taking the necessary precautions to ensure that should an accident happen the least harm will befall you. A poison is a poison, and aside from physical differences they should all be treated with the same careful consideration.

Let’s say, for example you have dissolved silver in excess nitric acid and currently have the beaker sitting in the middle of an open table. Now, you have chosen to precipitate the silver and simultaneously neutralize the excess nitric acid with sodium carbonate. You make your carbonate into an aqueous solution and add it drop wise slowly to avoid excess spattering. The next day you wake up from a restful sleep to find that your arms are covered with tiny black dots, and so is your face. Invisible drops of silver chloride solution were thrown from the beaker, carried by the wind, and otherwise deposited on your person. Had that been a highly toxic chemical you actually may have been beginning to feel the effects, a similar neutralization of a barium salt solution with excess carbonate may well make you sick within a few hours.

Always pay careful attention to chemicals that you work with that may be poisonous. And do not use them unless you feel you have to. Vapors can travel surprisingly far, heating poisonous solids may release similar vapors, some reactions may cause the breakdown of chemicals into more poisonous alternatives. The best ways to deal with a poisonous substance depends on its current form.

My poisonous substance is a **liquid** or is in solution:

If your substance is in water, its poisonous ability is slightly lessened, water has a weak ability to penetrate, chemicals that do however are lipophilic. Examples are carbon tetrachloride, DMSO, ethanol, ether, poisons solvated in these pose a greater hazard then those chemicals alone usually. However, should your substance be a liquid that is comprised of heavy metals directly bonded to carbon, organo-metiallic compounds, I cannot stress enough the danger involved, these will give heavy metals the most direct path into your body and straight to your brain. Wear gloves and don't cause the solution to foam, avoid heating of solutions that contain liquid poisons unless they are completely enclosed within a glassware setup.

My poisonous substance is a **solid**:

As long as the substance is totally dry, if it gets on your skin it can be gingerly wiped off and the area afterward washed with copious amounts of water. Always be careful of handling poisonous solids like cyanides in windy areas or using heavily powdered derivatives which may take flight even without the presence of wind. Keep track of weighing paper and such that comes into contact with your substance and be sure to use powder funnels for the transfer to keep it off the lips of containers.

My poisonous substance is a **gas**:

The most dangerous form of a poisonous substance. A gas mask is recommended should you have access to cartridges that effectively filter out the gas. Look to the section on gasses (4.10) for further information. Remember that even if your mask filters the dangerous component your neighbors will have to live without a mask and are at risk as well. Always consider your environment in these reactions. Note that not all gasses have smells to indicate their presence and they can be generated in large amounts should a liquid solution containing them is heated.

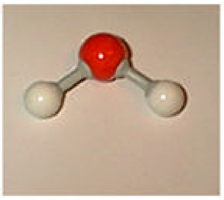
4.8 Solvents

Common Solvents:

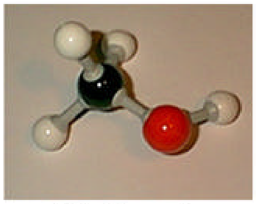
Specific Solvents: (Red = Flammable; Blue = Non-Flammable; Green = Burns with difficulty)

Density of the solvent is listed immediately following the solvent formula.

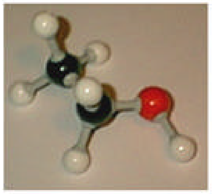
Oxogenated Solvents



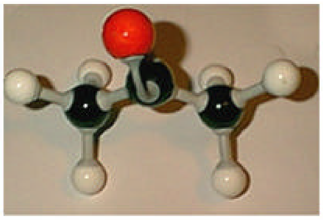
Water



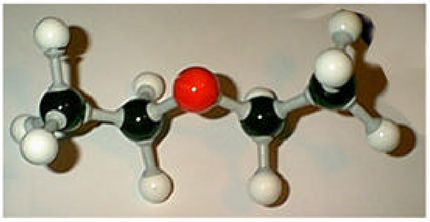
Methanol



Ethanol



Acetone



Ethyl Ether

Water H_2O: [1.0 g/ml] This is it, your ace in the hole. Water is by far the most useful, cheap, and widely available solvent you have. Known as the universal solvent, water also has the very convenient liquid range from 0 °C to 100 °C, it has a few somewhat painful points that become apparent when trying to remove waters of hydration or working with hygroscopic materials but the good definitely outweighs the bad. Water will solvate most ionic compounds, as the saying goes, like dissolves like. Other solvents with properties similar to water include hydrazine and DMF but these are somewhat less available and considerably more dangerous.

Water also has the advantage of being non-flammable, and noticeably inert to attack. The preparation of water in a laboratory setting is simple and if it were not so widely available it would be a easy task, the combustion of hydrogen in oxygen, some acid base combinations react to produce a quantity of water, and thermal decomposition of weak hydroxides. Water is also relatively easy to purify from general inorganic contaminates, simply by distillation. However when it comes to organics a number of solvents form azeotropes with water that must be broken before the two solvents can be effectively separated.

Tap water can work for most reactions but it is better to used distilled water, it is available at most grocery stores and pharmacies for a reasonable price.

Diethyl Ether $CH_3CH_2OCH_2CH_3$: [0.71 g/ml] The good: Ether is fairly inert, solvates a nice variety of compounds, has a low boiling point (34.5 °C) and a very low freezing point (-116.2 °C) so you can drive it off completed reactions, ether was used extensively in chemistry until recently so reactions using it can be copied exactly, plus it can be made with relative ease. The bad: Ether is highly flammable, it can form unstable peroxides in contact with oxygen and if the ether is boiled down containing a high peroxide concentration it can explode. Diethyl ether is commonly referred to simply as ether or ethyl ether, if something mentions using ether this is the ether it means [Note there is also a solvent referred to as petroleum ether, this is not the same thing.]. The preparation of ether falls into the advanced chemistry category, not in principle, but in practice:

$$CH_3CH_2OH =[H_2SO_{4\ (Concentrated)}]\Rightarrow CH_3CH_2OCH_2CH_3 + H_2O$$

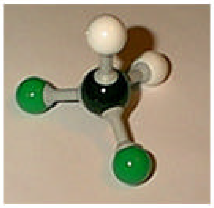
So, ethanol heated with concentrated sulfuric acid gives ether and water. The sulfuric acid is necessary, not just a catalyst, its affinity for water is one of the driving forces of this reaction, the ethanol is added to the hot acid, which is well beyond the boiling point of the ethanol, the ether being distilled as it is formed. The manufacture of ether at home is a tradeoff with safety and privacy, ether is a somewhat watched chemical in many places and ordering it form a supplier (assuming you find a chemical supplier that is willing to sell it) may set up red flags which could lead authorities to assume you are manufacturing illicit chemicals at your home, but on the safety aspect, lacking the proper glassware this procedure is exceptionally dangerous, even with the proper glassware there is a degree of danger. Luckily there is usually a substitutable solvent for ether depending on the reaction, still, it is a useful solvent.

Acetone CH3COCH3: [0.79 g/ml] One good use for acetone is the cleaning of labware. It acts as an in-between solvent, grease that may not come off with water can be pretreated with acetone to remove the bulk of the grease, then washed clean with water, acetone being soluble in water and able to solvate many non-polar molecules. It is also good for this purpose for the reason that it has a high vapor pressure, once a piece of glassware is washed with acetone the film left dries out quickly and the glassware is ready for use. Acetone also finds a place as a reactant, and solvent medium with a low melting point of –94.3 °C and a boiling point of 56.2 °C it is more reactive then many other solvents though. It is flammable and subject to chlorination, polymerization, and the haloform reaction among others. Preparation of acetone on a home scale can be done from the pyrolysis of calcium acetate, but is not necessary, it is available in many over the counter products for removing paint from fingernails and additionally as a solvent in hardware stores, most of these products labeled pure or 100% acetone. Methyl-Ethyl ketone sold under the acronym MEK is another ketone available on the market, it’s properties are similar to acetone and it will also undergo the haloform reaction. It is sold for stripping paint.

Methanol CH3OH: [0.79 g/ml] If something is soluble in water, it is also usually soluble, to a lesser extent, in methanol. From methanol formaldehyde and formic acid can be made, and there are other reactions in which it can readily participate. Methanol is flammable but not incredibly so and is somewhat widely available for a number of purposes, gas line defroster, hardware store solvent, and windshield washer fluid. As a reaction medium it suffices for some reactions, it has a boiling point of 64.5 °C and a freezing point of –97.8 °C, for many reactions though there are better mediums to conduct them. The addition of methanol or ethanol to saturated inorganic solutions in water usually results in the precipitation of some or nearly all of the solvated salt. Overall though it is a good reagent to have laying around. Consumption of methanol can result in blindness and should be avoided. It is not particularly hazardous as an inhalation hazard or contact hazard but precautions should still be taken.

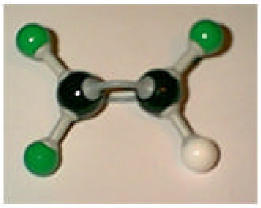
Ethanol CH_3CH_2OH: [0.82 g/ml] The properties of ethanol are similar to those of methanol, the boiling point and freezing point are shifted further up scale but other then that they behave closely to one another. Ethanol and methanol are very difficult to make anhydrous, ethanol forms an azeotrope with water that contains a somewhat high percentage alcohol. But to go beyond that drying agents / dehydrating agents start to become necessary. The prolonged action of anhydrous copper sulfate on concentrated ethanol is one way to make a nearly anhydrous product. Ethanol is available over the counter for consumption in percentages up to 95% but it can be expensive from this source, but the purity is somewhat guaranteed, it is additionally available over the counter denatured as a painting supply, however the denaturants can vary, ketones, methanol and other things can be added and other impurities can be present since it is not intended for human consumption after all.

Halogenated Solvents

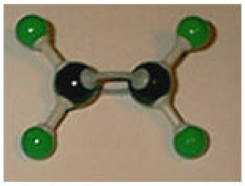


Methylene Chloride

Chloroform



Trichloroethylene



Tetrachloroethylene

Chloroform $CHCl_3$: [1.5 g/ml] While technically not a common solvent as it is not usually commercially available over the counter, the preparation of chloroform is easy enough for the amateur chemist and the reagents are easily acquired, a preparation is included in this text (Under section 5). Chloroform is toxic enough to where you should avoid unnecessary inhalation of the vapors and any skin contact but is relatively safe overall. Chloroform was used for years as the common solvent for organic material, the extraction of everything from albumin to zein. Chloroform is slightly soluble in water to the extent of about 8g/L but it still forms nice layers when added to water.

Improperly stored chloroform, notice the layer between the water and the bottom chloroform layer.

When stored usually 1% by volume ethyl or methyl alcohol is added to retard its decomposition. Chloroform normally decomposes to phosgene (COCl2, section 4.12), the addition of these alcohols leads to the formation of the carbonate ester which helps to slow decomposition. Chloroform can be stored under water but doing so can result in additional decomposition, even the oxygen from air can cause the decomposition of chloroform, but not at a rapid rate. Chloroform has a liquid range of nearly 100 °C, from –63.5 °C to 61.2 °C, it was at one time used as an anesthetic but that use was discontinued due to the toxic effects of chloroform. It is relatively inert in reactions, but contact with solid hydroxides and strong oxidizing solutions should be avoided.

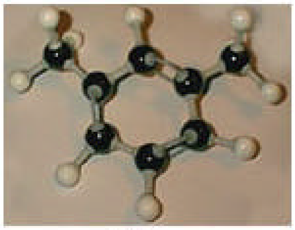
Methylene Chloride CH2Cl2: [1.3 g/ml] A surprisingly useful solvent for extracting desired compounds in the liquid phase, it possesses some unique solvent properties. Chlorinate hydrocarbons are somewhat toxic though not excessively so, still methylene chloride should be treated with respect. Methylene chloride is non-flammable, boiling point of 40.1 °C and a freezing point of –97 °C. The preparation of methylene chloride in a home environment is only feasible by the chlorination of methyl chloride or methane which is a quite dangerous, it is therefore fortunate that with some searching methylene chloride can be procured from over the counter sources specifically in paint thinners and removers, careful distillation from these mediums can provide a product of suitable purity for most reactions.

Tetrachloroethylene CCl2CCl2: [1.60 g/ml] Available as a component of some paint strippers tetrachloroethylene is a somewhat limited solvent. It also finds use as a reactant, it has a boiling point of 121 °C and a freezing point of –22.4 °C. As with all chlorinated hydrocarbons there is a degree of toxicity to this compound and like most it is non-flammable.

Trichloroethylene CCl2CClH: [1.46 g/ml] It is imperative the one never mix trichloroethylene with a strong base, doing so will likely result in the formation of dichloroacetylene, a carcinogenic compound that causes nerve damage. Trichloroethylene is available over the counter in can form in auto part stores, under pressure, for cleaning auto parts.

Aromatic Hydrocarbons

Toluene



Xylene

Toluene C6H5CH3: [0.87 g/ml] An aromatic hydrocarbon, good all around non-polar solvent, that until recently had a wide availability as a solvent for tough to remove paint. Reactive to halogens and other instances it is far from inert, but suffices for reactions nonetheless. Tolune has a boiling point of 110.7 °C and a freezing point of –94.5 °C.

Xylene C6H4(CH3)2: [0.86 g/ml] Properties similar to toluene but has a wider availability still. Xylene is also more reactive, the duel methyl groups activating it further then the one on toluene. It is a mixture of isomers, ortho, meta, and para xylene, and cannot be bought as purely one component as xylene will exchange its methyl groups and within a few days or weeks form a mixture of xylenes again.

Misc. Hydrocarbons: There are literally hundreds of hydrocarbons available on the market to the amateur chemist. They are used in many everyday applications most notably in nearly all combustible fuels, gasoline, kerosene, diesel, and all other sort of combustible. Pure hydrocarbons are decent for removing organics from an inorganic phase, but take special note of the additives that may be added to these hydrocarbons to make their combustion more manageable, especially in mixtures intended for the combustion engine.

Solubility Table:

M = Miscible (soluble in all proportions) **Ss** = Slightly Soluble / Somewhat Soluble
Is = Insoluble

-----	H_2O	CH_3OH	Acetone	Ether	Ethanol	$CHCl_3$	CH_2Cl_2	CCl_2CCl_2	CCl_2CClH	Toluene	Xylene
H_2O	-----	**M**	**M**	**Ss**	**M**	**Ss**	**Ss**	**Is**	**Ss**	**Is**	**Is**
CH_3OH	**M**	-----	**M**	**M**	**M**						
Acetone	**M**	**M**	-----	**M**	**M**	**M**					
Ether	**Ss**	**M**		-----	**M**	**M**				**M**	**M**
Ethanol	**M**	**M**	**M**	**M**	-----	**M**					
$CHCl_3$	**Ss**	**M**		**M**	**M**	-----				**M**	**M**
CH_2Cl_2	**Ss**	**M**		**M**	**M**		-----				
CCl_2CCl_2	**Is**	**M**		**M**	**M**			-----			
CCl_2CClH	**Ss**								-----		

Toluene	Is									-----	M
Xylene	Is									M	-----

Purifying Solvents:

Not a necessary step if your solvents are purchased from a lab supply. However if you purchase your solvent over the counter and it was not being marketed with a purity somewhere on the bottle you can not trust it entirely. As a matter of fact there is a suspicion that should be associated with many of the chemicals your purchase over the counter unless it is something that explicitly states the purity or is meant for human consumption as a pure product (e.g. citric acid). Even if you make a solvent on your own additional purification is usually necessary, for instance, chloroform produced via the haloform reaction is often contaminated with water, insolubles, and acetone for starters. Ether produced from ethanol and sulfuric acid often contains unreacted ethanol, water, and sulfuric acid/sulfur dioxide contamination. So in these cases there also exists a degree of contamination that must be accounted for.

The normal first line of defense for a solvent is to shake with a drying agent, decant, and distill. Non-volatile products such as anything dissolved in your solvent will not carry over and by using a fractioning column and paying careful attention to the temperature of the distillate purity can be increased significantly in one run. There is one danger here though, aside from the inherent flammability of most solvents, peroxides may be present in your solvent, either from impurities or in the case of ethers, the solvent itself. Therefore it is helpful to shake with some kind of reducing agent initially or to simply discontinue distillation with a noticeable amount of liquid left in the distilling flask (due to your intention to separate impurities one should not distill to dryness anyway, not that distilling to dryness should be done in any other situation).

Mixing Solvents:

Mixing solvents of different properties to give a solvent system of a desired property is to some extent hit and miss to the amateur chemist. But mixed solvents do have their definite advantages. They can cause the precipitation of an undesirable compound while keeping your desired compound in solution and vice versa. Additionally they can simultaneously put two compounds in solution to react that might otherwise be in different phases. Some solvents also have catalytic properties on a reaction and their inclusion in the reaction medium might speed things up greatly. Despite these great positive aspects mixed solvents are often overlooked because despite some solvent system looking good on paper (and other solvent systems having nearly impossible to predict properties) many of them just don’t give the expected results upon actually trying the experiment. Which results in wasted solvent, wasted time, and wasted reagents. None the less they do come into play on occasion, the following are some examples.

4.9 Transition Metals

- **Dissolving (Somewhat) Nobel Metals:**

Although many metals will simply dissolve, given enough time in hydrochloric acid or sulfuric acid or even acetic acid, there are some, which won't care in the slightest about being put into these environments. Although not strictly limited to the following, here are some examples of metals that may require a bit of special treatment to put into solution: (1) Silver; (2) Copper; (3) Bismuth; (4) Nickel; (5) Mercury

Although we are focusing on metals like those just listed, there are many other metals with which you would find difficulty putting into solution, like tungsten, tantalum, and in their own category gold and the platinum metals (platinum, iridium, rhodium, and a few others.), which have their own difficulty, associated with their dissolution. Here though are presented a few methods that may aid in dissolving the metal of your choice to form a solution of suitable cations.

Displacement- The principle here is simple, you find a readily available salt containing a metal cation that has a high reduction potential such as copper sulfate ($Cu^{2+}_{(aq)} + 2e^- \Rightarrow Cu_{(s)}$ V=.52) and add to it solid pieces of the metal whose salt you desire, such as lead ($Pb_{(s)} \Rightarrow Pb^{2+}_{(aq)} + 2e^-$ V=.13) The net reaction, lead going into solution is favored by the difference in voltage between the two. This particular reaction is complicated by the formation of lead sulfate on the surface of the lead reacting which only has a limited solubility, to rectify this a fish aquarium bubbler could be led into the area where the lead is reacting, the agitating action of the bubbles continually removing the sulfate layer and deposited copper from the lead allowing it to react further. Of course this is not good for putting metals into solution with very high potentials such as

Electrolysis- As electrolysis of a solution proceeds reduction occurs at the cathode and oxidation at the anode. In a solution containing only a little electrolyte such as NaCl with nickel electrodes the usual reaction is the formation of hydrogen and oxygen, if the concentration of NaCl is increased some chlorine my form in place of oxygen, however if in place of water a strong acid solution is used, such as $HCl_{(aq)}$ and the electrolysis preformed with sufficient current and voltage a significant portion of your cathode may be quickly reduced and put into solution, in this experiment the relevant equation being:

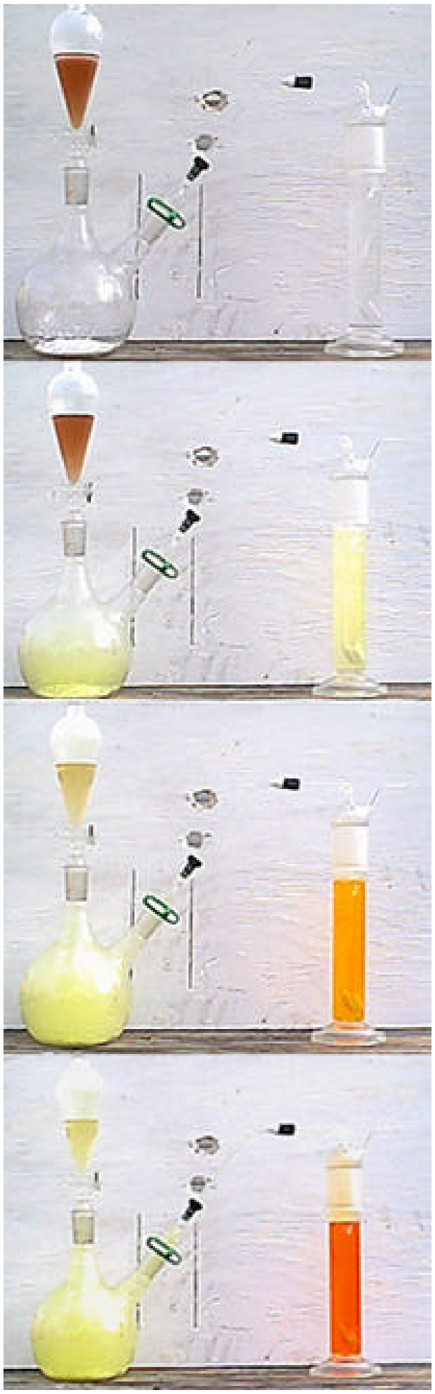
$$Ni_{(s)} \Rightarrow Ni^{2+}_{(aq)} + 2e^-$$

Products at the anode include oxygen and chlorine gas, hydrogen is also produced at the cathode from reaction with finely divided nickel and such, but the overall effect in this case would be the quick production of nickel chloride, and by coiling your cathode to increase the cathode surface in the electrolyte and decreasing the surface area of your anode in the solution you can very efficiently make a concentrated solution of many cations, evaporation in the case of hydrochloric acid would volatize off remaining acid leaving a somewhat pure product.

Mixtures of acids with oxidizing agents-

Fusion with hydroxides-

Specialty acid combinations-



4.10 Halogens

Pictured at left is a wonderful example of one of the trends followed by the halogens. As you move down the group the oxidizing power of each element decreases. So it stands that the halogen preceeding each halogen should have the oxidizing power to oxidize the element beneath it and in this way replace it. In this instance chlorine is being generated in the flask on the left and goes up through a piece of glass tubing and into a gas washing bottle. The bottle contains a concentrated solution of sodium bromide and as the chlorine goes through it a simple reaction takes place:

$$Cl_{2(g)} + 2NaBr_{(aq)} \Rightarrow 2NaCl_{(aq)} + Br_{2(aq)}$$

The bromine thus generated being slightly soluble in water and coloring the solution red to indicate its presence. It follows that chlorine would be able to easily do this to iodide in solution and that bromine would also be able to do this to iodine but no to chlorine. These three halogens also have other predictable trends, they tend to form soluble compounds with the exception of their silver salts and their copper (I) salts and they each have a series of oxoacids which are similar to one another. Because of these similarities in some reactions halogens are generically represented with an X, for example CH_3X would stand for either methyl chloride, methyl iodide, methyl bromide, or less likely methyl fluoride. When written in reactions and such the halogens are written F_2, Cl_2, Br_2, and I_2, the term for this being diatomic, which means the halogens do not go around as free atoms but rather each halogen is bonded to a neighbor, in this case by a single bond, e.g., Cl-Cl the bonds however are somewhat weak and UV light will usually do the trick to rupture these bonds and leave a reactive radical containing an unpaired electron, the strength of this bond is weakest with fluorine and strongest with iodine. Each of these halogens also have a whole series of oxidation states although they are not the same for each halogen they include –1, +1, +3, +5, and +7 oxidation states.

	HX	HOX	HOXO	$HOXO_2$	$HOXO_3$

Fluorine	Hydrofluoric Acid	Hypofluoric Acid	DNE	DNE	DNE
Chlorine	Hydrochloric Acid	Hypochloric Acid	Hypochlorous Acid	Chloric Acid	Perchloric Acid
Bromine	Hydrobromic Acid	Hypobromic Acid	Hypobromous Acid	Bromic Acid	Perbromic Acid
Iodine	Hydroiodic Acid	Hypoiodic Acid	Hypoiodous Acid	Iodic Acid	Periodic Acid

But what about fluorine? Fluorine is the odd element out, it does not follow these trends. Fluorine has one major oxidation state –1 there are few compounds where this comes into question but those instances are few and far between. Fluorine is the most electronegative element in the periodic table and will react with most elements at room temperature. Whereas the other elements have series of oxoacids of the form HOX, HOXO, HOXO2, and HOXO3 where X is the halogen in question fluorine only displays hypofluoric acid fleetingly by passing flurorine over moist glass at low temperatures. Above this fluorine would be in a higher oxidation state and as far as modern chemistry can show these forms do not exist.

Really though chlorine is the best example of this series, all of the oxoacids having a ‘simple’ method of preparation, chlorites (salts of hypochlorous acid) and hypochlorites (salts of hypochloric acid) being the most unstable of the series. Whereas perbromic acid is only prepared with great difficulty by passing fluorine through a basified bromate solution and iodine compounds of the form HOI and HOIO being preparable only in dilute solutions, periodic acid also displaying some deviance from the remainder of the family by having a formula of H_5IO_6, two water molecules having found their way into the molecule and sticking there pretty good.

Aside from being the basis of many of the most prevalent acids in chemistry the halogens also serve to offer a diverse group of oxidizing agents with fluorine and iodine at the extremes and the oxoacids forming an even larger middle. Many halogen salts are fairly soluble in aqueous inviorments and are somewhat stable to oxidation and reduction. Another shining point of the halogens comes in their organic reactivity, reacting directly with alkanes to give hydrogen halides and substituted alkanes (halogenated hydrocarbons) which are great starting points in organic synthesis. Really the halogens comprise the most utilitarian family in the periodic table and their use in the lab should not be underestimated.

Fluorine

Chlorine

Molecular Weight 35.45

Slightly soluble in water, soluble in non-polar solvents

Green/Yellow dense gas

-1, +1, +3, +5, +7 Oxidation States

Chlorine is somewhat simple to prepare and there are a number of methods to do so, even from over the counter products. Just as with bromine the pool industry is a great help in the preparation of chlorine, simple acidification of many pool chlorinators such as sodium hypochlorite (a liquid chlorinator that is also the active ingredient in bleach), calcium hypochlorite (a powder sold in packs often under the name "Shock") and trichloroisocyanuric acid and trichlorotriazinetrione (yet more pool chlorinators) can be instant sources of this highly noxious gas. Additionally it can be prepared by electrolysis of concentrated aqueous salt solutions with relatively high current densities, as well as by the oxidation of chlorine in the –1 state such as in hydrochloric acid, usually with permanganate. These methods will all give 'wet' chlorine of some purity or another that should be dried before use, chlorine being usually dried by passing through concentrated sulfuric acid.

Bromine **Br_2**

Molecular Weight 79.904 $_{g/mol}$

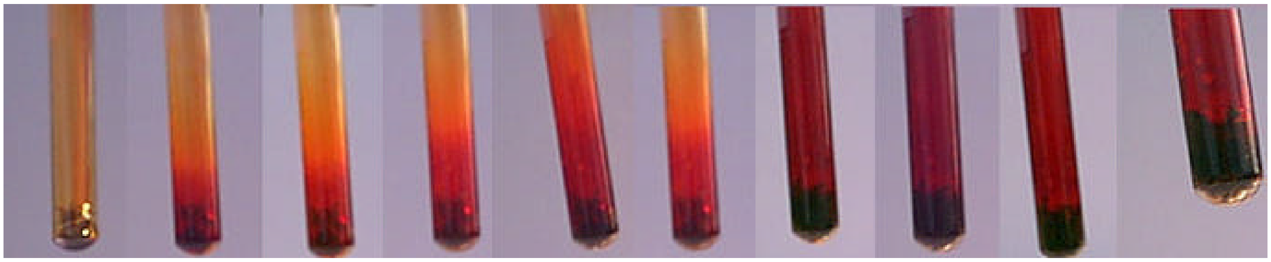
Slightly soluble in water, soluble in non-polar solvents.

Red vapor, liquid can be nearly black when present in large amounts.

-1, +1, +3, +5, +7* Oxidation States (* = very difficult to prepare)

One of bromine's claim to fame is of course that it is only one of two common elements that are liquids at room temperature and pressure. Because it is a liquid it is also somewhat easily storable by the amateur chemist, making it the strongest of the readily storable halogens. Bromine finds a wide variety of uses in the home lab. It can create a large number of bromides by direct reaction with elements, pictured below is the reaction between bromine and aluminum turnings:

$$2Al_{(s)} + 3Br_{2(l)} \Rightarrow 2AlBr_{3(s)}$$



In this case the reaction has a special utility, whereas aluminum bromide could also be prepared from hydrobromic acid and aluminum, the hydrate is formed and the product is nearly impossible to dehydrate. The anhydrous form is nearly the only good form of aluminum bromide to use in organic chemistry, and as such straight bromine dried by shaking with sulfuric acid is one viable way to produce this useful compound. There are many ways to produce bromine, but the most reasonable methods of course use the most readily available materials. Thankfully bromine compounds have found some utility in the pool and spa industry and as such the most readily accessible sources of bromine involve either sodium bromide or in the form of complex organic compounds that hydrolyze in water to give hypobromous acid (HOBr) such as tribromo-s-triazinetrione, treatment with acid will convert these readily to the bromide anion and put you in the same predicament as you would have been had you used NaBr from the beginning.

When going from sodium bromide to free bromine there is really only one key ingredient, an oxidizing agent under acidic conditions. If the reaction were under basic conditions then the bromine would react immediately to from hyprobromite and bromates. The acidic environment also helps the oxidizing agent do its job. Nearly any oxidizing agent will work, peroxide, manganese dioxide, hypochlorite, permanganate, even oxygen bubbled through an acidic solution of bromine can work were it not for the bubbling of gasses going though the liquid sweeping away the bromine as it were formed. Note however bromines low boiling point of 59 °C. But even well below this bromine will readily volatize off and fill an area with its choking fumes. As such preparations of bromine should have ice added, and take place as cold as feasible to prevent it from filling the area and reducing yields.

Disposing of bromine either dissolve in water or on its own is simple. Just adding the solution to an aqueous solution of a base will do the job readily (see picture at right). However the base must be completely dissolved, if you attempt to add bromine water to a hydroxide producing agent recently tossed into some water you will not get nearly the same reactivity. Always wear gloves when working with bromine as its contact with skin will discolor it and leave painful lingering sores which are slow to heal. A reducing agent such as citric acid solutions or sodium thiosulfate solutions can be used to treat recent areas of contact with bromine.

Bromine attacks most every metal including many from the platinum group. But dry bromine can have a hard time of attacking some other metals, notably some metals that would seem quite reactive such as magnesium, lead, iron, zinc, and even sodium. Aside from these metal compounds bromine will attack non-metals such as sulfur and also forms a large series of interhalogen compounds, interesting examples being bromine triflouride and bromine pentaflouride, the pentaflouride exploding on contact with water. Bromine chloride finds more use as a chemical intermediate oxidizing agent. Bromine itself is a good oxidizing agent, in inorganic preparations it has powerful oxidizing properties by adding to hot solutions of hydroxide, in this manner ferrates and bismuthate can be produced. In organic preparations bromine is less reactive then chlorine and is thus more selective in brominations. Brominated organic compounds being of great utility in synthesis operations.

On distillation from an aqueous solution bromine carries over roughly 2% of its weight in water. This is not a real azeotrope but it is something to be noted. Normally bromine can be generated whereupon it sinks to the bottom of the solution it was generated in where it can be pipetted off from. This can be used directly for some things but further purification can be achieved through non-distilative measures. Normally by first washing with a small amount of water, then shaking with concentrated sulfuric acid and finally by filtering though glass wool and storing under water or sulfuric acid.

Iodine

4.11 Alkali Metals

Not only are the alkali metals interesting for their reactivates and unusual properties, they exhibit definite trends as you move down the period which are easy to memorize.

- Lithium is the hardest alkali metal, but it can still be cut with a knife, as you move down the period from lithium to cesium the metals get softer and their melting points go down as well, cesium is a liquid only slightly above room temperature.
- Lithium is the least reactive of the metals, cesium the most, francium is only present on the earth in gram amounts at any time but it likely continues this trend.
- All alkali metals exhibit the +1 oxidation state as their main and only common oxidation state.
- Atomic radius increases as you move down the period, cesium, the largest stable element in the periodic table can stabilize the triodide anion I3- due to its size.
- The increasing reactivity of the alkali metals can be seen in their oxide formation, lithium forms predominately the normal oxide Li2O, sodium mainly the peroxide Na2O2, potassium mainly the superoxide KO2, and rubidium and cesium form almost entirely the superoxide. Additionally they form somewhat stable ozides

CsO_3 by passing ozone over their hydroxide and separating the formed ozide from remaining hydroxide by solubility differences in liquid ammonia.

- All are soluble in liquid ammonia or hydrazine yielding brilliant blue solutions that contain 'solvated electrons'. These are powerful reducing agents.
- The alkali metals react with water along the lines of $M(s) + H_2O(l) \text{--->} MOH(aq) + 1/2\ H_2(g)$ Lithium is somewhat manageable, sodium will usually ignite and can ignite clouds of hydrogen above it leading to explosion, by the time you get to cesium it will detonate.
- Lithium is the least dense metal of the period, it will float on most any oil you try to protect it under, cesium is the most dense.

Distinguishing between the different alkali metals in solution can be incredibly difficult chemically as they all behave very similarly and most salts are very soluble. The easiest test to distinguish between the alkali metal cations in solution is the simple flame test. A circle of wire, preferably inert, e.g. platinum, is dipped into a concentrated solution of the salt. It is then put into a high flame and the color of the flame observed.

Lithium = Red
Sodium = Orange/Yellow
Potassium = Purple
Rubidium = Red - Violet
Cesium = Blue

However the tests can be easily false, the colors can be over-run by other colors generated, and sodium, the most common contaminate of the other alkali metal salts due to most of them being produced from it, can easily over-shadow the other more sensitive colors of potassium and the like, leading to a false positive for sodium.

Lithium

Lithium is very light, it will actually float in oil.

Like other elements that start off their group lithium shows some properties that differ from the normal properties of the rest of the table. When exposed to air lithium will form a black coating of nitride Li_3N which reacts with water to produce ammonia.

One of the only other metals that will do this is magnesium metal appropriately heated, which similarly forms the nitride. Nearly all lithium salts are hydrated and removing the waters of hydration are nearly impossible on some of them, particularly the chlorate and perchlorate that decompose before a majority of the water has been removed. Lithium perchlorate actually contains more oxygen, on a volume-to-volume basis, then liquid oxygen. The hydroxide of lithium forms a stable hydrate $LiOH*8H_2O$ and does not dissolve in water if left exposed to the atmosphere, but it will form the carbonate like the other alkali metals, reacting with atmospheric CO_2. The carbonate of lithium is the least stable carbonate of the alkali metals and decomposes around 1000C.

Lithium metal has a high electrode potential and therefore it is becoming popular in some batteries (this also means that it can be very reactive in the liquid state, it will destroy glassware when liquid). Lithium is one of only a handful of metals that have actually become more expensive over the last 20 years. Lithium is usually stored on top of oil under an argon atmosphere.

Sodium

This is the most common alkali metal that we run across. Sodium carbonate, sodium chloride, sodium vapor lights, sodium hydroxide, sodium bicarbonate, the list goes on. Sodium is the most abundant of the alkali metals in the earths crust and it shows in our preference to it, that and the fact that we need sodium chloride in our daily dietary intake.

Potassium

Potassium follows the trend set up between sodium and lithium in that it is more reactive then either of them, being further down the group. Therefore when it burns in air, shown left (Notice the purple flame that is also indicative of potassium ions in a flame test), it produces not only the oxide and peroxide, but predominately the superoxide. KO2 is a very powerful oxidizing agent and it proves to be a nuisance when storing potassium. When a block of potassium is stored under mineral oil or another inert substance unless it is in an air tight container and the liquid has been degassed the potassium can pick up oxygen and form the superoxide. This leaves a yellow-orange coating on the surface of the pieces of potassium. This in and of itself is usually no problem however upon cutting into a piece of potassium covered in this coating it can force the superoxide into the unreacted potassium and in worst cases this can cause an explosion, sending flaming bits of potassium everywhere and causing severe damage to the individual performing the manipulation.

Potassium superoxide and the superoxides of other higher alkali metals react with water along the following equation:

$$2KO_2(s) + 2H_2O(l) \longrightarrow 2KOH(aq) + H_2O_2(aq) + O_2(g)$$

Potassium superoxide is also used in space capsules for the duel purpose of sequestering CO_2 from the astronauts breath and to generate additional oxygen. It is also used in some self contained breathing apparatuses:

$$4KO_2(s) + 2CO_2(g) \longrightarrow 2K_2CO_3(s) + 3O_2(g)$$

Sequestering an additional water molecule and CO_2 molecule by the following:

$$K_2CO_3(s) + CO_2(g) + H_2O(g) \longrightarrow 2KHCO_3(s)$$

Another interesting property of potassium is that it forms a carbonyl compound $K(CO)_6$ however it is not the most stable of carbonyl compounds by a long shot. It can explode for no reason at all at STP therefore any reaction that generates elemental potassium, or uses elemental potassium, in the presence of carbon monoxide (i.e. reduction of the carbonate with charcoal) should be treated with caution as the in situ preparation of potassium carbonyl may cause explosions.

Potassium-sodium alloys are liquids at STP and more reactive then either metal individually. Industrially potassium is prepared by distilling it from a mixture of sodium metal and potassium chloride. The replacement of potassium with sodium in the reaction is not immediately sensible due to potassium being the more reactive, however the reaction works due to the potassium formed having a significantly lower boiling point then the sodium therefore the reaction is pushed foreword as the potassium boils off. The Castner cell also works with potassium hydroxide in place of sodium hydroxide and actually gives better yields and a lower melting solid. However potassium is more flammable and reactive therefore this reaction is less favored. The electrolysis of the chloride is also less favored due to higher working temperatures of the eutectic. Thermite type reactions also work for the production of potassium, reducing potassium oxide or hydroxide with magnesium works, but aluminum forms aluminates that decrease yields significantly. The most common potassium salt available to the amateur chemist is potassium chloride, it is widely available for use in water softeners and as a salt substitute in health food areas.

4.12 Functional Groups of Organic Chemistry

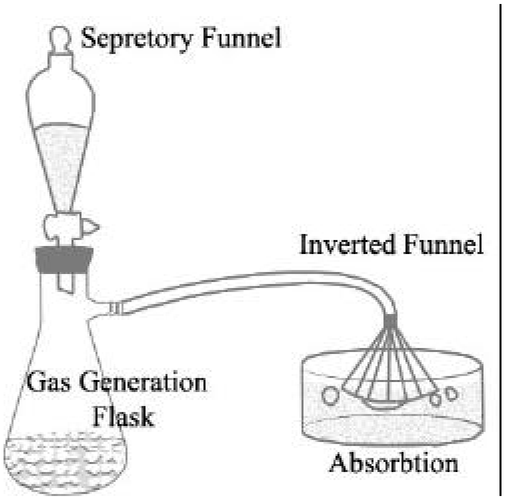
4.13 Gasses

Working with gasses can be quite important to a chemist. They not only posses a great variety of reactivity, but many of them are also easy to make. Dealing with gasses is of course different then working with liquids or solids but the change over is easy as long as you take it step by step. Your main concern is actually probably going to be how the flow of gasses though your system is going to affect fluids in your system, and how those fluids will respond if pressure is applied in the opposite direction if for some reason gas generation slacks off or stops. By nature gasses can be everywhere around you, and unlike a solid or liquid which has to first act on the skin before causing ill effects gasses can go straight from the air into the blood stream. Therefore gasses possess and added degree of possible danger. All gasses are asphyxiants in large amounts (except oxygen) but many possess additional hazards. The author of this text recommends against working with several gasses based on the extreme toxicity of those compounds. However there are still a number of other gasses that can be safely worked with and handled. Please treat all gasses with respect and plan out your reactions and apparatuses before hand and you will be rewarded with safety.

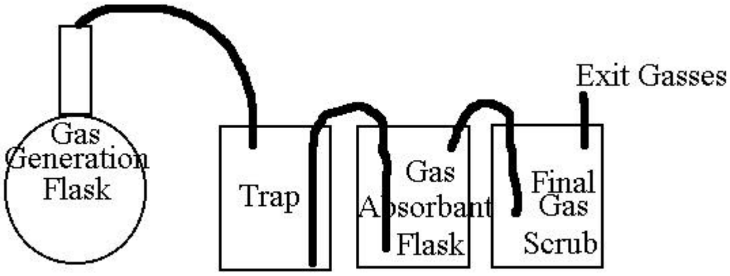
A gas bubbler, as show on the left can be quite the useful tool for an at home lab. However the construction of such a device is exceedingly simple and therefore purchasing a piece of equipment like the one shown can be avoided. The basic principle is simple, gasses come in through the tube on the left, bubble though a solution contained in the body of it, going though a glass frit on the way to disperse them better thereby creating more surface area causing the gasses to be absorbed/washed better, and finally exit though the tube on the right which comes nowhere near the water thereby preventing the liquid in the container from being transferred to the next container.

If you expect to be working with gasses a lot you might want to ask yourself if you want to create a fume hood. Working in a fume hood is like working in a box made of glass with only one exit for any gasses, though a tube away from you. Usually drawn though the tube with a fan and treated to neutralize them, fume hoods are indispensable for some, but not many, applications.

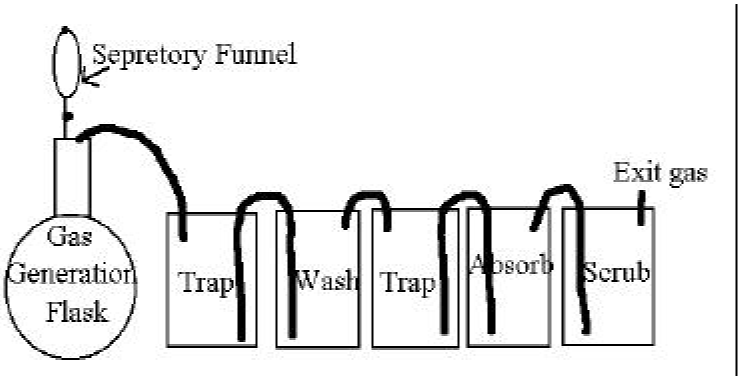
Gas generation and handling setups:



Above is a very simple gas generation apparatus. However since it lacks a trap (which would prevent fluid from coming back into the flask) it would only be good for low temperature generations, which would prevent a dangerous suckback. Something where the addition of the liquid in the sepretory funnel would reliably keep the gas coming along and where your intention is to dissolve your gas in a liquid in which it is fairly soluble (because anything that doesn't dissolve is just going to get into the air around you).



This apparatus goes two steps further, one, it incorporates a trap to prevent liquid from flowing back into the possibly hot flask that may or may not react with water, and two, it has a final gas scrub which may possess a different liquid then the original absorbent flask to make sure to destroy harmful vapors.



Even one step further, this setup has an initial trap, then a wash to take out impurities that may hurt the reaction, followed by another trap and finally the absorbance and the scrubbing step. Also there is a separatory funnel for the addition of another liquid to the reaction flask to keep the reaction going.

Tips for working with gasses:

- When working with flammable gasses it is necessary to heat any part of your vessel with a non-flame, non-sparking heat source. The smallest spark can trigger an explosion and special care should be taken to remove sources of ignition from your work area.
- In the case of gasses that are highly unstable (arsine, diborane, phosphine, silane, hydrazine) and can decompose exothermically when heat is applied and if it is a case where that gas must be disposed of by incineration, it may be necessary to run the gas into a container containing damp sand, up though the bottom and though a tube at the top, this will negate the possibility of a sudden explosion of gasses flashing back and detonating your whole reaction setup.
- If working with a gas of very high toxicity (most of them on the list) and inhalation occurs, do not delay, **get medical help immediately**, gasses are absorbed fast and a delay of a few minutes could mean your life, call a poison control, or the local emergency services.
- Make sure your reaction apparatus is air tight beforehand, and keep some duct tape around for emergency fixes (real emergencies only, if some of these start to leak you might just evacuate before it gets to the duct tape)
- Flammable/Explosive gasses should never be generated in the same reaction as one that also produces oxygen or nitrous oxide, the possibility of explosion is too great.

Gas Masks:

Gas	Filter Properties
Hydrogen Sulfide H2S	Filters designed specifically to protect against hydrogen sulfide can only do so for very limited periods of time and are designated rescue filters. Being that they have a life of less then 10 minutes, are not designed for excessive concentrations, and are expensive, they are not economically feasible. Hydrogen sulfide is extremely toxic and has killed many individuals working in amateur labs because it quickly deadens the sense of smell. Common logic would dictate that you just not work with this foul smelling gas.
Organic Solvents	There are filters specifically designed to block out VOC (Volatile Organic Carbons) these work fairly well and are the most commonly available filters in my experience as they are used widely in painting. They last for an extended period of time and are designed for constant use. Good for working with solvents of

	all kinds but especially carcinogenic solvents such as halogenated hydrocarbons and benzene derivatives. (Methanol is notoriously difficult to filter out, however it does not present much of an inhalation hazard.)
Hydrogen Chloride Chlorine Acid Gasses	There is a specific filter known as an acid gas filter. It will block out hydrogen halides (except fluorides) and elemental halogens along with other acidic gasses such as SO2, although they cannot reliably block out nitrogen oxides.
Hydrogen Cyanide HCN	Regular carbon filters can block out HCN, however one exposure can ruin the filter and therefore make you susceptible to anything else you attempt to protect yourself from. As with H2S, it is better not to use this gas/liquid in your experimentation, as it is highly toxic.

To put on a gas mask you place it over your face, straps behind your head, the exact technique is not as important as the tests you do before giving it the all clear. First is the positive pressure test, find the exit hold for the air, usually right in the middle of the mask, cover it with your hand and breath out, if the air only 'farts' out from around the edges of the mask you are good. Next is the negative pressure test, cover the intakes on your cylinders with your hands and inhale, no air should come in, the mask should attempt to deform inward to compensate for the uptake of air. If you pass both of these tests your mask is correctly positioned on your face. If either one of these tests prove negative, reposition your mask and try again.

Dealing with exit gasses:

In all instances read the below information and thoroughly acquaint yourself with a gas before working with it. You are responsible for your own actions and therefore should take extra precautions, gasses are inherently dangerous so use them with care, extra information regarding the scrubbing of gasses is in the information of the main text regarding specific gasses.

Name of Gas:	**Disposal by incineration:**	**Disposal by scrubbing**
Acetylene	**Yes**	**Possible (Oxidizing Agents)**
Air	**NA (Vent)**	**NA (Vent)**
Ammonia	**Yes**	**Yes (Acids)**
Arsine	**Not Recommended**	**Yes (Oxidizing Agents)**
Butane	**Yes**	**No**
Carbon Dioxide	**NA (Vent)**	**Possible (Bases)**
Carbon Monoxide	**Yes**	**No**
Carbonyl Chloride	**No**	**Yes (Hot Aqueous Bases)**
Chlorine	**No**	**Yes (Bases)**
Chlorine Dioxide	**Could Cause Explosion**	**Yes (Bases)**
Boranes	**Yes**	**Yes (Basic Oxidizing Agents)**
Ethylene	**Yes**	**Yes (Oxidizing Agents)**

Fluorine	**No**	**Yes (Anything)**
Hydrogen	**Yes**	**No**
Hydrogen Cyanide	**Not Recommended**	**Yes (Bases)**
Hydrogen Fluoride	**No**	**Yes (Bases)**
Hydrogen Halides	**No**	**Yes (Bases)**
Hydrogen Sulfide	**Yes (But Generates SO_2)**	**Yes (Bases/Oxidizing Agents)**
Methane	**Yes**	**No**
Nitric Oxide	**Not Recommended**	**Yes (Bases)**
Nitrogen	**NA (Vent)**	**NA (Vent)**
Nitrogen Monoxide	**No**	**Yes (Strong Bases)**
Nitrous Oxide	**Yes**	**No**
Nobel Gasses	**NA (Vent)**	**NA (Vent)**
Oxygen	**NA (Vent)**	**NA (Vent)**
Ozone	**Not Recommended**	**Yes (Reducing Agents)**
Phosphine	**Yes**	**Yes (Oxidizing Agents)**
Propane	**Yes**	**No**
Silane	**Yes**	**Yes (Oxidizing Agents)**
Solvent Vapors	**Yes**	**No**
Sulfur Dioxide	**No**	**Yes (Bases)**

Scrubbing Exit Gasses: When scrubbing exit gasses care should be taken to ensure complete neutralization of the toxic effects. The greater the toxic effect the more drastic measures should be taken. Severely toxic chemicals should go though no less then two scrubbing solutions, but preferably three or more. The concentration of the solution should not be incredibly high or low but will depend on the gas to be neutralized. If it will help the addition of an acid base indicator may tell when some acidic or basic gasses have used up the neutralization power of a given solution.

Igniting/Incinerating exit gasses: When igniting exit gasses it is good to flush the system with an inert gas first (carbon dioxide, butane, propane, methane, argon, etc.) even steam will work for this purpose. This is so once flammable vapors come over and they are ignited the vessel does not contain any oxygen gas which may flash back into the system and blow up your glassware. If flushing the system is not feasible, let the apparatus run and generate gas, then from a distance blow away the gasses with a fan and ignite after the vessel is full of the gas in question (this method is not advised). Sometimes the gasses will escape the vessel at a speed sufficient to maintain a constant flame. However it is best to run the gasses though a tube that connects to a pipette and run them straight into a flame or into the intake of a flame. In this way if the gas evolution slacks off and the escaping gasses stop combusting but start exiting again afterwards they will still be burning as they exit. Also even spontaneously flammable gasses are not spontaneously flammable in all concentrations, without external burning some will make it all the way to the inside of your lungs and wreak havoc.

Specific Gasses: (Green = Water Solution Basic Red = Water Solution Acidic) WS = Water Soluble

Acetylene HCCH: The most common source of acetylene for the home chemist is by the action of water on calcium carbide.

$$2H_2O_{(l)} + CaC2(s) \Rightarrow HCCH_{(g)} + Ca(OH)_{2(s/aq)}$$

Acetylene is quite the unsaturated molecule, being that it contains a triple bond between the carbons. This can be acted on by a number of reagents, most of the halo acids adding across it forming vinyl halides and it is easily oxidized or polymerized. Acetylene is ridiculously explosive if allowed to accumulate in one area. If liquefied it can undergo hazardous polymerization and as such cylinders of acetylene sold for welding actually contain acetylene dissolved in acetone. The cylinders of MAPP gas available from hardware stores contain an derivative of acetylene among other things. Acetylene has the normal asphyxiation hazard that many of the gasses here have. It is more reactive then your average hydrocarbon and is readily produced as stated before, calcium carbide still being somewhat easily available specifically for the purpose of making acetylene, especially over the internet. Acetylene should not be lead into basified solutions or acidified, doing so can make explosive acetylides or carcinogenic vinyl halides. Acetylene can only really be disposed of by incineration and then the vessel from which the acetylene comes from must be purged of oxygen otherwise the flame could flash back inside and detonate the vessel

Air: Air is a mixture of gasses of approximate composition by volume; (78%) Nitrogen, (21%) Oxygen, (1%) Argon, (<1%) Other gasses, CO2, Ne, He, etc. [Exact composition of the air around you varies with your elevation and your surroundings, however these numbers are relatively constant] Unless you take steps to the contrary, you will be working in air. Most reactions can be carried out with exposure to the atmosphere, there are many that cannot be though, be sure to take into consideration the properties of the atmosphere you make for your reaction, and how it will react with your reaction before beginning any involved chemical reaction. Never forget that the water content varies daily and that it can impact many reactions to a great extent.

Ammonia (NH_3) WS: Ammonia is not only a foul smelling gas, but also a low temperature reaction solvent. It does the most interesting trick often referred to as the "Solvated Electron" trick in which it will dissolve the alkali metals which turns the ammonia a beautiful blue color and upon concentration makes the ammonia look golden like liquid metal. Still though, these reactions take place in liquid ammonia, so below –33 °C. Ammonia can be liquefied at home but it is a hassle. Ammonia gas in the presence of water attacks a number of metals but most notably copper with which it forms a dark blue complex, note that ammonia is flammable and can be explosive if initiated like the lighter hydrocarbons. Ammonia gas is poisonous and its basicity does not combine well with ones eyes and it causes terrible burning and tearing and eventually blindness. Commercial ammonia solutions available from grocery stores and such contain roughly 4% ammonia by volume. Solutions of ammonia in water are also referred to as ammonium hydroxide due to the equilibrium:

$$NH3_{(aq)} + H2O_{(l)} \Leftrightarrow NH_4OH_{(aq)}$$

However this is an equilibrium reaction and the equilibrium actually lies to the left. Ammonium hydroxide itself is not isolatable in the pure state, it is known through its salts and in solution. Ammonia is a very useful reagent to have around. It forms numerous complexes and has a wide variety of reactions to exploit. Strong solutions of ammonia can be made with the help of an ammonia salt, in this case ammonium sulfate:

$$(NH_4)_2SO_{4(aq)} + 2NaOH_{(aq)} \Rightarrow 2NH_{3(g)} + Na_2SO_{4(aq)} + 2H_2O_{(l)}$$

The combination of a solution of ammonium sulfate and sodium hydroxide giving off noticeable ammonia gas readily. This reaction can also be done in the solid phase but it can become violent. The gasses being produced in both instances being channeled through water to dissolve as much as possible then scrubbed with a solution of nearly any acid, but the stronger the better.

Arsine (AsH_3) WS: Arsine has a garlic-like odor similar to phosphine. Stibine (SbH_3) is similar to arsine and this may be used as a guide for it as well. Initial exposure to arsine produces few symptoms, headache, nausea, nothing to make a person worry too severely. However several hours or so after what some would call a mild exposure, a breath or two, vomiting and cramping set in and depending on the dose kidney failure, CNS depression, and death. Arsine is the most toxic way for the body to come into contact with arsenic. Because of its severe toxicity arsine should be avoided. To destroy arsine from exit gasses it is prudent to run the gasses though at least two washes of sodium hypochlorite, calcium hypochlorite, potassium permanganate, bromine water, or sodium hypobromite solutions. Be sure their volume is sufficient to provide excessive decomposition ability for more arsine then you can think might be produced. Do not run arsine into any incineration, fine As_2O_3 will be produced creating a terrible wide spread inhalation hazard. Arsine is really terrible, it will cause long-term reproductive damage, and cancer concerns, do not tinker with it. It will decompose to its elemental constituents at around 400°C providing there is not oxygen present to support its combustion. Electrolysis of solutions that contain arsenic cations under acid conditions can produce arsine, this is not meant to be a preparation however, it is a warning.

Boranes: Boron-hydrogen compound chemistry is extensive, although difficult to facilitate at home. Most of the boranes can be broken down in some way to the building block molecule borane (BH_3), which has never been isolated on its own, diborane (B_2H_6) for example is the dimmer of borane. Diborane is like all the volatile boranes is toxic, inhalation of boranes results in headache, dizziness, unconsciousness, fluid in the lungs, and finally death. Mixes of borane are spontaneously flammable in moist air, which can cause manipulations involving it to result in explosions. Boranes burn with a green flame producing powdery B_2O_3 that lays a fine dust on all surroundings. The main use of diborane is in the preparation of sodium borohydride, a moderately strong reducing agent that can be safely recrysatalized from water.

The preparation for boranes is similar to the preparation of silane or of hydrogen sulfide. Acid is slowly dripped over solid magnesium boride (MgB_2) resulting in the formation of this spontaneously flammable gas (Note, many boranes are produced, diborane produced is almost instantly hydrolyzed to boric oxide, picture at left shows a milliliter of HCl being dripped onto some magnesium boride). Concentrated phosphoric acid has been shown to give better results in the preparation of boranes. One borane in specific, B4H10, being produced in the largest amounts, this can be converted to diborane by careful distillation at liquid air temperature, under which conditions it dissociates and gives a distillate of diborane.

Being that some boranes are pyrophoric it is easy to assume they might be easily oxidized, that is in this case very true. Atmospheric oxygen and even water will oxidize many of the boranes (though some of the higher anaolges are resistant to oxidation). Therefore all handling vessels must be free of moisture and especially oxygen (however it would be impossible to exclude moisture from the initial flask as it is necessary for the reaction and a part of most acids). Note that diborane was once considered for use as a rocket fuel but the produced boric oxide was too abrasive on the rocket cones, to say this another way, its oxidation is very exothermic and such an exothermic reaction anywhere near you would be disastrous. The industrial process for producing diborane involves the following reaction.

$$BF_{3(g)} + 6NaH_{(s)} \Rightarrow B_2H_{6(g)} + 6NaF_{(s)}$$

Additionally the reaction between sodium borohydride and elemental iodine in THF or other ethers produces diborane. Although simpler both of these reactions are complicated by the use of two somewhat difficult to obtain chemicals, whereas the magnesium boride used in the first reaction can be theoretically obtained quite readily by a thermite reaction (Note, unless fine reactants are used this reaction is hard to initiate/maintain due to the excess magnesium):

$$4Mg_{(s)} + B_2O_{3(s)} \Rightarrow 3MgO_{(s)} + MgB_{2(s)}$$

Boric oxide and magnesium shavings being more available then the boron triflouride and sodium hydride used in the industrial process.

Being that boranes are very toxic gases, any emissions from systems that may contain boranes should be appropriately dealt with as to reduce their danger. Although these substances can be pyrophoric, one cannot assume that boranes leaking from a reaction vessel will oxidize before it has a chance to do any damage to their person.

Therefore one should do either or both of the following; 1) Lower boranes will react with water to produce boric acid and hydrogen gas readily, bubbling diborane though a a tall column of $NaOH/H_2O_2$, using an efficient bubbling mechanism (e.g., a glass frit) should greatly diminish the concentration of diborane. 2) Running the exit gasses of a system into the air intake of a burner. Much care should be taken with boranes, as many of them are toxic, and potentially explosive, and spontaneously flammable, this is yet another gas that poses such a danger as to make the author of this work suggest against working with it.

Butane $CH_3(CH_2)_2CH_3$: Butane is relatively uncreative to may chemical conditions. It makes a workable inert gas for some situations but the actual production of butane on a lab scale from other chemicals is needlessly complicated as it can be purchased for refilling lighters and some camping supplies easily. Butane containing exit gasses should be run into the intake of a torch to prevent butane vapors from accumulating around your work area and causing an explosion hazard. Although butane does not prevent as specific inhalation hazard it can act as an asphyxiant gas, especially if used in an enclosed area.

Carbon Dioxide CO_2 WS: Carbon dioxide has no detectable smell. You breath out carbon dioxide and plants take it in and as such it does not possess some terrible toxicity. But in concentrated form it is hazardous for your health. Therefore when working with dry ice or a carbon dioxide cylinder or another source that could provide a large amount of carbon dioxide quickly into a small space one should take care and have adequate ventilation. Aside from commercially available dry ice (solid carbon dioxide) and cylinders that are available for the soft drink industry, it is also marketed as a water solution seltzer. Production of carbon dioxide in the home lab is very simple. The addition of a markedly acid solution to a carbonate or bicarbonate, either solvated in water or simply as a powder will generate copious volumes of carbon dioxide, the production of which is controlled by the rate of acid addition:

$$CO_3^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \Rightarrow CO_{2(g)} + H_2O_{(l)}$$

Carbon dioxide produced in this way will contain significant quantities of water vapor that must be removed using a desiccant and additionally if your acid has a volatile component (e.g., HCl, HNO_3) a portion of the acid may carry over as well and need its own separate scrubbing. The use of carbon dioxide as an inert atmosphere is covered in section 8.4 an example of a preparation of carbon dioxide for lab purposes would entail a setup consisting of a sepretory funnel on the gas generation flask for the addition of acid at will. The exit gasses for this vessel would be scrubbed for volatile acid components such as HCl and HNO3 by passing though a carbonate solution which will in turn make more CO2 and passing those gasses though strong H2SO4 to remove any water that may be present. A minimum of one trap is required between the volatile acid scrubbing chamber and the sulfuric acid chamber as suck back of H2SO4 into saturated carbonate would not be favorable. Additional traps could be placed though out but the controlled addition of acid to the solution should ensure that gas continuously flows away from the reaction flask. Carbon dioxide is also the product of complete combustion but the carbonate method works better for a reasonable supply. Carbon dioxide is a gas slightly heavier then air, it possesses no oxidative properties except with strong reducing agents under heat and it does not act as a reducing agent, upon dissolution in water it makes a slightly acid solution of carbonic acid.

Carbon Monoxide CO: A colorless, odorless, poisonous gas. It's toxic action is produced by its strong bonding with the oxygen carrying constituent of human blood, hemoglobin. Carbon monoxide inhalation is treated with oxygen, however since it has no odor, and the usual warning signs of poisoning are lethargy and headache, which can easily be overlooked cases of carbon monoxide poisoning usually go untreated resulting in chronic poisoning or fatal poisoning. The most common reason for carbon monoxide poisoning is from a faulty furnace in the home.

Carbon monoxide is the product of incomplete combustion of carbon containing molecules. It's use in chemistry is actually quite extensive, however for the beginning chemist it is really not a gas of interest. It does however act as a decent reducing agent, and in environments where carbon is the reducing agent under extreme conditions carbon monoxide can be the main product, therefore taking precautions to remove the gas is an important measure. Other then generating insanely toxic carbonyls of the transition metals though it does not have the wide variety of uses that would render it highly important. It's preparation is simple, the addition of concentrated sulfuric acid to formic acid results in its dehydration and subsequent generation of carbon monoxide gas.

$$HCOOH_{(aq)} + H_2SO_{4(l)} \Rightarrow H_2SO_4{*}xH_2O_{(aq)} + CO_{(g)}$$

Gas generated in this way is suitable directly for a number of applications provided the addition of the reagents is controlled. The exit gasses of apparatuses that use carbon monoxide or produce carbon monoxide should always be lead into the entry of a flame to burn the gas away entirely. Carbonyl compounds can also be pacified in this manner. However allow me to reiterate the main fact here, carbon monoxide is highly poisonous and gives no warning to its presence, apparatuses using it should be checked and rechecked to ensure they are air tight and exit gasses are properly incinerated, the author of this work recommends against intentionally using carbon monoxide in any preparations.

Chlorine Cl_2: (See Section 4.9)

Chlorine Dioxide ClO_2 ws: Chlorine dioxide is surprisingly soluble in water, making a green solution. Its actual reaction with water is slow and solutions of ClO_2 in water are stable for some time. Chlorine dioxide is very toxic and destroys lung tissue, eye tissue, and skin wholesale upon contact when concentrated. It is used industrially for disinfecting and bleaching of paper and as long as the gas is heavily diluted with an inert gas, usually CO2, it is somewhat safe to handle, it is usually produced at the point of consumption for this reason though. When concentrated as a gas chlorine dioxide can explode for no apparent reason or in the presence of traces of organic material. The usual process to make ClO2 on the spot involves the reaction between sodium chlorite and sulfuric acid:

$$5H_2SO_{4(l)} + 5NaOCl_{2(aq)} \Rightarrow 4ClO_{2(g)} + 2H_2O_{(l)} + NaCl_{(aq)} + 2Na_2SO_{4(aq)}$$

Sodium chlorite being the salt of the unstable acid HOCl2. This process is no where near free from danger, the previous method of manufacture of ClO2 was to heat a mixture of sodium chlorate and oxalic acid, this produces CO2 gas simultaneously which dilutes the ClO2 produced, but even with this advantage there have been numerous reports of this preparation going awry and exploding without ascertainable reason. The hydrolysis of ClO2 in water yields a number of products depending on the temperature and time of reaction but the products include chlorate, chlorite, hypochlorite, chloride, and perchlorate. Scrubbing of exit gasses containing ClO2 should be done with strong NaOH, reducing agents should be avoided due to danger of explosion, and apparatuses venting this gasses should be wrapped in screen and shielded from light to lessen the possibility of exploding, exit gasses containing it should never be burned as it may flash back into the system and cause a massive explosion. ClO2 is a very dangerous gas to work with and preparations involving its use and isolation should be avoided.

Ethylene CH2CH2: A colorless gas with a slightly sweet odor. Once used for anesthesia it is exceedingly flammable and explosive under the right conditions. It also has a unique property of acting to ripen fruit despite it being such a simple molecule. Although it can act as an asphyxiant is does not possess any excessive toxic effects on the human body. Unlike acetylene hazardous polymerization is less likely however this gas is quite reactive toward oxidizing agents. Here are a few examples:

$$CH_2CH_{2(g)} + Br_{2(aq)} \Rightarrow CH_2BrCH_2Br_{(l)}$$

$$CH_2CH_{2(g)} \text{--(}KMnO_4/NaOH_{(aq)}\text{)-->} CH_2OHCH_2OH_{(aq)}$$

That double bond that ethylene has is quite prone to addition and it is likewise reactive as illustrated above. It has some use in synthesis for such reasons but no use as an inert atmosphere. The production of ethylene is one step further then the production of diethyl ether. In the case of ether two molecules of ethanol are condensed and a water molecule is lost, ethylene goes one step further, another water molecule pulled out of diethyl ether so to speak according to the following reaction:

$$2CH_3CH_2OH_{(l)} \text{ --(High Heat/Excess } H_2SO_{4(l)}\text{)-->} 2CH_2CH_{2(g)}$$

High temperatures (>140 °C) and a significant excess of sulfuric acid (at least a 4x molar excess) to ethanol produce favorable conditions for ethylene formation. It is recommended that you add some fine sand to the reaction mixture to make a slurry that prevents some of the bumping associated with this reaction. As with working with other highly flammable gasses heating should be accomplished by an oil bath held on an electric/non-sparking heating source. The ethylene thus produced after being put though a weak basic wash is suitable for most any purpose.

To scrub ethylene from the exit gasses from a reaction either significant bubbling though an alkaline permanaganate solution or leading the exit gasses into the intake on a flame will work. Although highly flammable, ethylene is less dangerous then some of the gasses mentioned here and can be used with a measure of safety.

Fluorine F_2 WS (reacts): (See Section 4.9)

Hydrogen H_2: Hydrogen gas is highly flammable although otherwise not terribly reactive. With heating it will form hydrides with some metals and it is a good reducing agent at high temperatures but the kind of run-away reactions that can be expected from some reactions are simply in the lacking with hydrogen (except that whole highly flammable thing). Hydrogen is a very light gas that clears the reaction area very quickly owing to its ability to take flight. The simplicity of making hydrogen gas in the laboratory is only matched by the simplicity of making carbon dioxide. Section 4.2 on acids covers the hydrogen activity series, anything in the list above hydrogen will displace hydrogen from an acid and will therefore produce hydrogen gas as the metal is solvated. If the only acids you have are weak acids you need a somewhat reactive metal, aluminum or magnesium will displace hydrogen from acetic acid (it works with the other metals too but due to dilution the reaction is slow), but usually somewhat stronger acids are available. A great reaction to produce hydrogen gas is to drip hydrochloric acid onto magnesium scrap, the reaction is fast but it is therefore over fast and allows for a more precise control over the speed of hydrogen generation, other good choices might be common steel wool, aluminum foil, or zinc in any form (Do not use nails, you do not want to make a shrapnel bomb!).

$$Mg_{(s)} + 2HCl_{(aq)} \Rightarrow MgCl_{2(aq)} + H_{2(g)}$$

The gas thus produced would have to be scrubbed for two things, volatile acid components ($HCL_{(g)}$) and for water that vaporizes, it depends on how pure you want this gas as to how much scrubbing you are going to do on it. Running the gas though a sodium hydroxide mixture will eliminate acidic components and running it though a final sulfuric acid wash will remove gas and make the hydrogen suitable for most any application. Exit gasses containing hydrogen are best disposed of by incineration.

Hydrogen Cyanide HCN WS: This is actually a borderline gas/liquid, approx. Bp is 26C so slightly above room temperature, it has the odor of bitter almonds, however only 1/3 of the population can smell it due to a genetic defect. But that is not important, what is important is that this is one of the top four most toxic gasses listed in this countdown. Not only is it toxic, but it is prone to explosive polymerization if not properly stabilized and it is also flammable/explosive on its own. Hydrogen cyanide is more often then not accidentally made when a chemist dabbling in cyanides decides to acidify the solution, big mistake:

$$NaCN_{(aq)} + H_2O_{(aq)} \Leftrightarrow NaOH_{(aq)} + HCN_{(aq)}$$

I say big mistake because that equilibrium lies too far to the right for my own comfort to begin with owing to the weakness of hydrocyanic acid and when acid is added separately it forces the equilibrium to the right by taking up the sodium hydroxide and therefore hydrogen cyanide is formed, although very soluble in water the acid continuously comes acidified solutions, and if significantly acidified a significant eruption of HCN can occur. Hydrogen cyanide has incredible knockdown power, one whiff can instantly put a person on the floor unconscious with no hope of recovery and death following within two or three minutes. Scrubbing hydrogen cyanide from exit gasses is a simple affair as it is so easily oxidized bubbling though a sodium hypochlorite solution or other mild to strong oxidizing agent will easily oxidize the cyanide anion to cyanate OCN^- a less toxic version. Hydrogen cyanide really is a chemical that I recommend others not even work with, as such the information here is more of a warning then anything amyl nitrate (inhalation) and sodium thiosulfate (ingestion) are somewhat of folklore medicines to help combat cyanide poisoning.

Hydrogen Fluoride HF WS: Another borderline gas/liquid, approximate Bp is 19C, slightly below room temperature. As with all soluble fluorides hydrogen fluoride would be considered toxic to start out with, however it goes one step beyond, if you get a splash of hydrogen fluoride on your arm the accepted scenario involves your flesh dissolving to the bone, then your bone dissolving, followed by several minutes of pain as you get terrible fluoride poisoning, finally ending with your heart stopping. But that’s liquid hydrogen fluoride, nasty stuff anhydrous.

In dilute solutions (<3%) it is fairly safe to work with, concentrations of this magnitude are available in over the counter products. They are still toxic but not to the same degree. As a gas anhydrous hydrogen fluoride is almost just as bad, eating away at lung tissue and poisoning your body. At least it’s not flammable. Hydrogen fluoride of all concentrations will attack glass show in the following equation:

$$SiO_{2(s)} + 6HF_{(aq)} \Rightarrow SiF_6H_{2(aq)} + 2H_2O_{(l)}$$

The ability of hydrofluoric acid to attack glass increases steadily with concentration to about 99.9%, supposedly totally anhydrous hydrogen fluoride will not attack glass, however notice that water is created in the attack on glass, therefore even a stray water molecule could catalyze the reaction. It will eat glass to a significant extent eliminating the ability to use glass vessels when handling hydrogen fluoride, either as a solution or as a gas.

$$CaF_{2(s)} + H_2SO_{4(l)} \Rightarrow CaSO_{4(s)} + 2HF_{(g)}$$

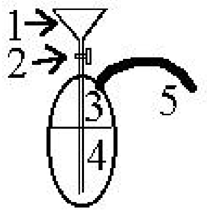
The formation of hydrogen fluoride by heating calcium fluoride with concentrated sulfuric acid is favored by the volatility of the hydrogen fluoride thus formed and continued heating to keep driving it off. Additionally hydrogen fluoride could be had by removing the water from over the counter solutions, either by distillation (be sure to check for azeotropes) or by dehydration via a strong desiccant.

As an aqueous solution hydrogen fluoride is a weak acid, this is to be expected if you look at the trend setup by the other hydrogen halides, but it is actually much weaker then could be predicted. This is because the bond between hydrogen and fluorine is incredibly strong and therefore ionization happens to a considerably lessened extent. It will dissolve many metals though and as it is doing so the heat of the reaction is probably vaporizing HF out of the solution and into your air.

Please, hydrogen fluoride is not only a terrible contact poison but cumulative poison, please consider this information a warning as to its dangers.

Hydrogen Halides except Fluoride HCl, HBr, HI WS: These are all noxious smelling gasses at room temperature and pressure. Their solution in water form the recognized acids hydrochloric acid, hydrobromic acid, and hydroiodic acid. Inhalation of these gasses can cause damage to the lungs and mucus membranes. Skin contact with concentrated vapors will result in discoloration and possibly necrosis. Hydrogen bromide presents a problem different from the other two in that bromine is not normally utilized by the body, hydrogen bromide inhalation, resulting in the increase of bromide in the body results in increase lethargy and in extreme instances, death. Although not exceedingly toxic these gasses all cause damage on the physical level, which if enough could cause death.

All of these gasses are soluble in water to significant extents, hydrogen chloride the least soluble and hydrogen iodide the most. Their acid solutions provide the most effective ways to generate the gasses. Dehydration of the solutions, especially if they are initially concentrated will result in the formation of the free halogen halide.



Above is an apparatus ideal for the formation of a hydrogen halide gas from a hydrogen halic acid. In such an apparatus the acid to be dehydrated is put into 1 which is a funnel closed off from the rest of the system by the stopcock 2. This funnel extends downward with a stem that has a very small opening, almost like capillary tubing 3. This extends down to the bottom of the vessel and deep under the concentrated dehydrating acid (sulfuric for hydrogen chloride, phosphoric for bromide or iodide) 4. The gas upon formation bubbles though the sulfuric acid and out of the gas tube 5. The opening in the tubing is small enough that it draws down more acid until the stopcock is closed. This apparatus will generate large quantities of water-free hydrogen halide. The simplified version is just a container full of sulfuric acid into which your hydrohalic acid is dripped in with a sepretory funnel. The disadvantage of this version is the water spray and evaporation which calls for an additional wash of the exit gasses.

The hydrogen halides can also be generated from a chemical reaction. Hydrogen chloride can be generated (although not controllably) by the reaction between sodium chloride and concentrated sulfuric acid:

$$NaCl_{(s)} + H_2SO_{4(l)} \Rightarrow NaHSO_{4(s)} + HCl_{(g)}$$

Although for hydrogen bromide and hydrogen iodide this reaction is not as feasible. Some of the hydrogen bromide formed will be oxidized by the sulfuric acid to free bromine and for the reaction between sodium iodide and sulfuric acid most if not all of the hydrogen iodide produced is oxidized to iodine. However distillation of weaker solutions of sulfuric acid with these salts can result in aqueous azeotropes of the acids. The hydrogen halides of these salts can be generated by the reaction of a sodium salt with concentrated phosphoric acid, which lacks the oxidizing power to release the free elements.

HBr and HCl can also be generated as side products from organic halogenations. Usually ½ of the initial halogen is consumed in the reaction and the other half is released as the hydrogen halide, iodine however does not posses the power necessary to perform most organic halogenations and therefore this is not a good way to make hydrogen iodide.

Exit gasses containing these acidic gasses can easily be scrubbed by bubbling them though concentrated sodium hydroxide solution. Subjecting these gasses to high heat will dissociate them to some extent and oxidizes some of them part way but results in a multitude of products.

Hydrogen Sulfide H_2S WS: Yet another incredibly toxic gas, this one more so then hydrogen cyanide. It has it's own unique smell, which everyone has probably smelled at one time or another, the smell of rotten eggs. However, although the smell of hydrogen sulfide becomes prominate at levels below lethal levels it has one major trick, it deadens the sense of smell quickly so the smell goes away and you think you're okay, but actually you might just be about ready to die. Many beginning chemists have died from hydrogen sulfide thinking that because they could not smell it they were okay. Smell is only a way of detecting hydrogen sulfide if it is generated unexpectedly, if that happens leave the area. You do not want to mess around with this chemical.

The treatment of many sulfides with acid is the cause of hydrogen sulfide production usually. As with hydrogen cyanide, hydrogen sulfide dissolves in water forming a weak acid solution, and adding acid to a salt of hydrogen sulfide drives the equilibrium to the production of hydrogen sulfide gas. Sulfides can easily be made by the direct combination of an active metal with elemental sulfur followed by heat. They can also be the product of high temperature reductions of sulfates.

$$CaSO4_{(s)} + H2_{(g)} \Rightarrow CaS_{(s)} + H2O_{(g)}$$

$$S^{2-}{}_{(aq)} + 2H_2O_{(l)} \Leftrightarrow HS^{-}{}_{(aq)} + OH^{-}{}_{(aq)} + H_2O_{(l)} \Leftrightarrow H_2S_{(aq)} + 2OH^{-}{}_{(aq)}$$

In the first equation calcium sulfate is treated with hydrogen at a high temperature in the absence of oxygen, the products are gaseous water and calcium sulfide. The second equation shows the equilibrium that exists in a neutral solution of hydrogen a sulfide salt of of hydrogen sulfide. Addition of a base adds hydroxide, which appears on the far right, this drives the equation to the left, and as long as plenty of base is present sulfides are relatively safe. However if acid is added that will destroy the base and it will protonate the sulfide anion floating around it the solution, both of these will drive the equation to the right and produce hydrogen sulfide gas.

The allure of hydrogen sulfide is there though. It is useful in the laboratory, as an agent to detect certain metal compounds, as a reducing agent and also to generate some interesting acids.

$$Br_{2(l)} + H_2S_{(g)} \Rightarrow 2HBr_{(aq)} + S_{(s)}$$

If this reaction were to be carried out under a layer of water and stirring were applied it would not stop there, the sulfur formed would react with the bromine formatting S_2Br_2 which would react with the water resulting in the oxidation of sulfur converting it to SO_2 and making two more molecules of hydrogen bromide, most of which would dissolve in the upper layer of water making a concentrated hydrobromic acid solution. Solutions of formic, acetic, and other acids can be made in this manner. Exit gasses containing H_2S can be burned or scrubbed with two washes of concentrated basic solutions or alkali oxidizing agents such as $KMnO_4$. But the danger of carrying out such operations usually render this unfeasible, hydrogen sulfide kills, the author of this work recommends against not working with this toxic chemical.

Methane CH_4: A common gas with which most people are familiar, methane is a simple asphyxiant gas with the side pitfall of being an explosion hazard. It has no uncommon reactivities and behaves fairly inertly. Natural gas piped to homes is mostly methane with other agents added for smell and a small percentage of other hydrocarbons. Methane in home synthesis is somewhat of an extreme measure do to its lack of reactivity. Chlorinating methane should yield carbon tetrachloride, chloroform, methylene chloride, and methyl chloride. However the careful control of temperature and necessary supply of chlorine gas are usually outside of the normal working scope of the home lab. It is interesting to note the simple procedure by which methane can be generated in the home lab:

$$CH_3COONa_{(s)} + NaOH_{(s)} \Rightarrow CH_{4(g)} + Na_2CO_{3(s)}$$

By simply heating anhydrous sodium acetate with sodium hydroxide the reaction commences generating methane gas and sodium carbonate. Methane can be formed as a product of the electrolysis of some mixtures containing organic components or by the action of water or acid on aluminum or beryllium carbide. If a reaction is run in which methane provides an ‘inert’ atmosphere or a reaction that involves the production or use of methane is run, the exit gasses should be lead into the intake of a burner and incinerated to prevent and explosion hazard.

Nitogen dioxide NO_2 WS (reacts): This compound reacts with water to form nitric acid and nitric oxide gas. Due to this fact it is incredibly toxic, think about it, it goes into your lungs and makes nitric acid which in turn causes your lungs to secrete fluid which causes pulmonary edema which means you're going to die. Nitrogen dioxide has a biting odor caused by its hydrolysis upon contact with fluids within your nose, also due to its hydrolysis you can taste it. It has a high boiling point and can be easily condensed at home, as a liquid/solid it is colorless in theory due to the formation of the dimmer N2O4, however it is actually more often then not brown-red as is the gas.

$$4HNO_{3(aq)} + Cu_{(s)} \Rightarrow 2NO_{2(g)} + Cu(NO_3)_{2(aq)} + 2H_2O_{(l)}$$

The easiest production of NO_2 involves the action of concentrated nitric acid upon copper metal. However if the reason you seek this toxic gas is for the production of nitric acid then that really does not seem like a feasible method of production. Alternative methods of production include the moderate temperature decomposition of heavy metal nitrates (although complicated by the formation of oxygen), or by the action of a strong electrical discharge on a mixture of oxygen and nitrogen to produce a mixture of nitrogen oxides followed by a means of separation such as condensation.

$$6NO_{2(g)} + 3H_2O_{(l)} \Leftrightarrow 3HNO_{3(aq)} + 3HNO_{2(aq)} \Leftrightarrow 4HNO_{3(aq)} + 2NO_{(g)} + H_2O_{(l)}$$

As you can see nitrogen dioxide disproportionates in water to form nitrous acid and nitric acid, however the nitrous acid thus formed is unstable and decomposes resulting in the formation of yet another molecule of nitric acid and a two molecules of nitric oxide. Giving the overall equation showing that six molecules of nitrogen dioxide will react with two molecules of water to form four molecules of nitric acid. It should be noted that nitric oxide is easily oxidized to nitrogen dioxide by the action of atmospheric oxygen with no other stimulus. Solutions of high concentration nitric acid with excess NO_2 dissolved within are known as red fuming nitric acid. Incineration of exit gasses containing nitrogen dioxide should be avoided in combination with any flammable gasses as nitrogen dioxide can support combustion and cause a flash back of fire into your reaction regardless of a lack of oxygen.

Exit gasses containing nitrogen dioxide should be appropriately scrubbed using a strong sodium hydroxide solution. The formed sodium nitrate and sodium nitrite may be recovered for future uses by evaporation of the scrubbing solution afterwards. Nitrogen dioxide is a very poisonous gas, if it is to be used safety measures should be planned out in advance and it should be used entirely in a closed reaction system, the operator of such a system should wear a respirator of some sort and exit gasses should be double washed in concentrated sodium hydroxide solutions, this is not a gas to take lightly.

Nitrogen N_2: Approximately 78 % of the air you breath is, by volume, nitrogen gas. Nitrogen is a diatomic molecule that has a very strong triple bond connecting the two nitrogen atoms. It is inert to a wide variety of common applications except electrostatic discharges. It is purified from air by liquefaction of air followed by fractional distillation. It is a widely available gas in the chemistry industry and can be found for welding applications. Laboratory preparation of nitrogen is very complicated on average, one method being by the action of a strong oxidant on an aqueous ammonia solution, or by the careful heating of ammonium nitrite, or by the careful decomposition of an azide. Other methods also exist but the best is going to be simply removing other components from air. Running normal air into a metal tube full of copper wool and heated externally very hot with a torch will remove the oxygen present if the tube is significantly long enough. You are left with nearly 98% nitrogen content with 1% or so of argon. Nitrogen forms a good inert atmosphere for many reactions. It is an asphyxiant gas like any other, however there is no need to take extra precautions with exit gasses that may contain nitrogen only worry about other things that may be there.

Nitric Oxide (Nitrogen Monoxide) NO: Some references refer to this as nitrogen monoxide, others refer to nitrous oxide as nitrogen monoxide, check the context in older text to be sure which is which. The nitrogen of this molecule has one unpaired electron, which should cause it to at least dimerise forming N2O2, however it does not have a tendency to do this greatly until a low temperature. As a gas nitrogen monoxide is colorless, and somewhat reactive. It will react almost instantly with air forming brown nitrogen dioxide (see above). Nitric oxide is only very slightly soluble in water forming a very small amount of nitrous acid. Nitrogen monoxide can act as either a reducing agent or an oxidizing agent, resulting in the formation of the nitrate anion or nitrogen dioxide respectively.

Nitric oxide is usually formed by the action of dilute nitric acid on copper metal. However it can also be formed by reacting sodium nitrate, ferrous sulfate, and sulfuric acid, which is a convenient laboratory preparation. In all cases the gas must be washed to remove traces of other nitrogen oxides that may be present:

$$2NaNO_{2(s)} + 2FeSO_{4(aq)} + 3H_2SO4_{(aq)} \Rightarrow 2NO_{(g)} + Fe_2(SO_4)_{3(aq)} + 2NaHSO_{4(aq)} + 2H_2O_{(l)}$$

Solutions of nitrous acid formed by the acidification of a nitrite salt in general will decompose somewhat rapidly to a solution of nitrate and nitrogen monoxide gas:

$$3NO_2^-{}_{(aq)} + 3H^+{}_{(aq)} \Leftrightarrow 3HNO_{2(aq)} \Rightarrow HNO_{3(aq)} + 2NO_{(g)} + H2O_{(l)}$$

The last step being somewhat irreversible due to the low solubility of NO gas and that it is leaving the solution, therefore driving the reaction to that step. That is one of the reasons why $HNO_{2(aq)}$ is made in situ as needed and not an item to be laying around on a stock shelf. The addition of acid to a nitrite is the way to go, the addition of dilute sulfuric acid to a solution of barium nitrite followed by filtration leading to a somewhat pure solution.

Nitrogen monoxide will support combustion and will attack rubber and possibly lead to an explosion if mixed with potentially oxidizeable gasses. Being that nitrogen monoxide converts to highly poisonous nitrogen dioxide on exposure to atmospheric oxygen your main concern upon venting from a reaction mixture will be the probable inhalation of nitrogen dioxide. However nitrogen monoxide can be scrubbed by passage though a concentrated sodium hydroxide solution, and passing though an open flame may help destroy this molecule, although passage though two or three vats of hydroxide is recommended. As long as you are careful in its use and clean up nitrogen monoxide only poses a hazard to those who disrespect it, if you are working in an area and sudden circumstances cause the evolution of nitrogen monoxide/dioxide gas occur just leave the area, no sense in risking your life.

Nitrous Oxide N_2O: Nitrous oxide will readily support the combustion of any of a number of possibly combustible molecules, nearly anything that will burn in air will burn readily in nitrous oxide:

$$H_{2(g)} + N_2O_{(g)} \Rightarrow H_2O_{(g)} + N_{2(g)}$$

The formation of exceedingly stable dinitrogen being one of the motivating factors in its oxidizing ability. Nitrous oxide has the famous synonym “laughing gas” and is somewhat widely used in the dental profession. It is also used as a foaming agent/propellant for whipped cream due to its high solubility in lipids. Nitrous oxide on its own poses no real danger except the usual asphyxiation hazard, however as stated before it is a potent oxidizing agent therefore its presence in the laboratory should still be monitored and general venting of the gas should be avoided (also in view of its sedative properties).

The classic method of preparation of nitrous oxide is the controlled thermal decomposition of ammonium nitrate:

$$NH_4NO_{3(s)} \Rightarrow N_2O_{(g)} + H_2O_{(g)}$$

During the decomposition extra care must be taken to prevent water from condensing around the top of a flask generating the nitrous oxide and dripping back in, this can increase the production of other nitrogen oxides or may crack a flask. Large amounts of ammonium nitrate should be avoided (>50g) or else the ability of the mass to transfer thermal energy may become inhibited which could result in localized super heating and possibly a run away deflagration. Other safer methods, such as the reaction between hydroxyl amine and sodium nitrate in solution or reacting ammonium nitrate with a small amount of sodium chloride and dilute nitric acid in solution, either one of these reactions requires external heating in a water bath to achieve decent N_2O production.

Nitrous oxide should never be produced for human consumption. As an exit gas it should be subjected to thermal decomposition or possibly passed though a bath containing a strong reducing agent.

Nobel Gasses; Helium He/Neon Ne/Argon Ar/Krypton Kr: Helium is readily available mixed with air though party supplies, neon is available though companies catering to neon light production, argon is available to welders, and krypton is a specialty gas. All these gasses are nearly totally unreactive and are good for blanketing a reaction environment. The latter two of them are heavier then air and subsequently do a better blanketing job. As exit gasses these posses the regular asphyxiation hazard but nothing more unless they are carrying with them other potentially hazardous substances which require additional treatment.

Oxygen O_2: Making up roughly 20% of the air we breath oxygen is all around us. It's reactions are numerous but its most noted reaction is a combustion reaction, usually the definitive case between oxygen an a hydrocarbon such as butane:

$$2CH_3(CH_2)_2CH_{3(g)} + 13O_{2(g)} \Rightarrow 8CO_{2(g)} + 10H_2O_{(g)}$$

The combustion of such hydrocarbons at least requiring a spark to initiate but once started continuing almost instantly in the case of gasses mixed with oxygen and somewhat more manageably at the liquid gas interface or liquid/gas/solid interface with other forms. If oxygen had been deficient in the above reaction mainly carbon monoxide would have formed in preference to carbon dioxide but only because oxygen had been deficient, carbon monoxide itself will burn if it is mixed with oxygen and the proper spark of activation energy is applied.

Oxygen is a very good and cheap oxidizing agent. Bubbling air though hydrochloric acid with nickel metal immersed in it for example will lead to the dissolution of the nickel somewhat rapidly. At elevated temperatures oxygen attacks many metals, magnesium will readily burn in oxygen as will many finely divided/powdered metals (nickel, iron, zirconium, zinc, etc.). Zirconium powder burning in pure oxygen can achieve temperatures in excess of 4500 °C! Oxygen can also bring out an even higher oxidation state in some elements then even elemental fluorine can. For example, combination of oxygen and osmium metal at room temperature leads to the slow formation of osmium tetroxide which gives osmium a +8 charge, by contrast the reaction of osmium at high temperature with fluorine can only yield the somewhat unstable compound OsF_7 (osmium heptafluoride) which decomposes to the more stable osmium hexafluoride.

Dioxygen serves many important oxidizing roles in the lab. But it can also be a nuisance, some materials are so easily oxidized that even a moments exposure to oxygen can contaminate them. A container containing nickel carbonyl most clearly shows this, opening and closing the opening of it for a moment almost immediately causes solid nickel particles to form on the surface of the previously clear solution. Oxygen also leads to explosion hazards and flammability concerns. Liquid oxygen increases these risks, for example, a charcoal briquette soaked in liquid oxygen and then ignited supposedly explodes with the force of a stick of dynamite. However explosives of this type are unpredictable and not commonly used in industry.

Although air contains a relatively decent oxygen percentage it may become necessary to produce oxygen on a lab scale. Purification from air is out of the question for most any amateur chemist due to oxygen being the most reactive component of it, however industrially oxygen is prepared from air by condensing it to a liquid and fractionally distilling off the oxygen (-183 °C). On a home scale oxygen can be prepared by numerous methods, the simplest is the catalytic decomposition of hydrogen peroxide.

Adding many different things to hydrogen peroxide will decompose it to oxygen and water. With dilute solutions (<30%) and with monitoring of the temperature (Keeping it less then 70 °C using an ice bath as necessary) this preparation is free of danger. The gas must first be bubbled though concentrated sulfuric acid or another dehydrating agent to remove any water that may be present but afterwards it is of sufficient purity to use. A great catalyst for this is produced by mixing manganese dioxide with sodium silicate until the solution becomes thick then slowly add dilute acid with stirring. The resultant chunks of silica with manganese dioxide imbedded in them are put onto a strainer and washed to remove fine particulates and the granular precipitate is excellent for decomposing peroxide and is fully recoverable.

The classical procedure of making oxygen is the controlled decomposition of a chlorate or perchlorate at medium (150 – 150 °C) temperatures:

$$2KClO_{3(s)} \Rightarrow KClO_{4(s)} + O_{2(g)} + KCl_{(s)} \Rightarrow 2KCl_{(s)} + 3O_{2(g)}$$

The decomposition can be stopped at the middle stage producing perchlorate and chloride and oxygen gas or additional or prolonged heating will result in total chloride conversion and maximum production of oxygen gas. The addition of a small amount of manganese dioxide also helps to make the reaction run smoother. However working with nearly molten chlorates/perchlorates is not a nice experience and there is always a chance that the reaction could run out of control. Therefore this method is mentioned for curiosities sake. And also because it is the methodology behind many pyrotechnic mixtures.

Oxygen gas can be safely vented only if there are no additional potentially flammable gasses being released in the vicinity, however if a large quantity of oxygen gas is vented continuously or all at once there is always additional danger associated with it. Exit gasses containing oxygen can be lead into the intake of a burner and will only add to the combustion of the hydrocarbons therein.

Ozone O_3: A step up from oxygen in terms of oxidizing ability, ozone will attack may organic compounds with vigor if it comes into contact with them. In very small amounts ozone actually has a pleasant smell, it can be smelt around power lines or when running electrical equipment that can spark such as electric drills. However in higher concentrations the pleasant smell goes away and you are left with a nauseating biting smell. Ozone is capable of innumerable oxidizations, many of which are at least difficult with oxygen and near impossible under the same reaction conditions. For example, ozone will oxidize sulfur dioxide to sulfur trioxide at STP, where as for substantial conversion with oxygen at a minimum an efficient catalyst and somewhat elevated temperatures should be used.

The formation of ozone by chemical means is difficult, especially under controlled circumstances in reliable yields. A number of reactions will produce ozone in small amounts, two such reactions being the oxidation of which phosphorus with oxygen, or the decomposition of manganese heptoxide. However the amounts generated from partial oxidation of phosphorus are small and manganese heptoxide is an unstable explosive liquid.

Electrolysis is another way of making ozone. The electrolysis of perchlorates under alkaline conditions can yield ozone. However the most time tested and reliable way to make ozone is to run oxygen gas though an electric discharge. If nitrogen is present nitrogen oxides may be formed. But a good electric discharge system with a slow steady supply of oxygen flowing though it can yield mixtures of oxygen and ozone containing up to 20% ozone. Ozone is almost always made on site where it is needed and therefore it would be almost impossible to purchase it.

Ozone has its place in chemistry, ozides of the alkali metals can be made with it and extensive oxidations can be carried out with it. It will only be in your exit gas in all likelihood if you put it in your reaction to begin with. If that is the case ozone can be destroyed by bubbling the exit gasses though aqueous solutions of alcohols or reducing agents. High temperatures can destroy ozone but leading it into the intake of a burner will destroy it though combustion. Ozone is highly toxic and as such should be treated with care.

Phosgene $COCl_2$ (Carbonyl Chloride) WS (reacts): Yet another gas who once found use as a war gas, phosgene is produced industrially from carbon monoxide and chlorine gas by the simple reaction:

$$CO_{(g)} + Cl_{2(g)} \Rightarrow COCl_{2(g)}$$

Carbonyl chloride finds use industrially in the production of polycarbonates. It has a somewhat high boiling point of 8.7 °C and therefore could be liquefied very easily if that was your intention. However it is very toxic to human beings, upon entering the human body it hydrolyzes to hydrochloric acid and carbon dioxide, the hydrochloric acid attacking the lungs and eyes causing blindness and pulmonary edema. Its danger should not be underestimated and any undertaking that could produce phosgene should be treated with the utmost care and caution. Phosgene can be produced from the oxidation of chloroform or carbon tetrachloride. Containers of chloroform should have ethyl or methyl alcohol added, their presence destroys phosgene by converting it to hydrochloric acid and ethyl or methyl carbonate (non-toxic). These chemicals should be present to the extent of about 1% the total volume, additionally chloroform should be stored in dark bottles dry and without opening often to limit the supply of air in the containers.

Exit gasses that contain phosgene should be run through at least two washes of strongly basified water. Turning it into chloride and carbon dioxide. Additionally phosgene could be burned but its combustion would release hydrogen chloride. Addition of an oxidizing agent to the basic wash is not known to have any additional positive effect.

Phosphine PH_3: Unlike ammonia phosphine only acts as a base in the presence of fairly strong concentrated acids. Phosphine is only very slightly water-soluble and a solution of phosphine in water is not appreciably basic. Phosphine has a particular smell similar to molded garlic or rancid fish depending on the individual. The odor is not a very good indicator as it is toxic below the odor threshold. Phosphine is actually incredibly toxic, and causes terrible delayed damage to the kidneys and liver. Similar to arsine poisoning, a person subject to phosphine inhalation may feel nauseous or ill for a few hours but the symptoms may clear up, only to have the person struck down in bed again a few days later, with death possibly following shortly thereafter.

Similar to the production of hydrogen sulfide by the action of acid on an appropriate sulfide, the action of acid on many phosphides (of which only zinc phosphine is commonly found) will usually yield phosphine. An older demonstration for the properties of phosphine was to take a retort and put some white phosphorus, water, and sodium hydroxide into it and seal it. Place it on a hot surface and put the beak of the retort so it dipped just under water. Phosphine would be produced and bubble up, and as it broke the surface it would spontaneously ignite and create perfect smoke rings that rose up one after another. But, just like hydrogen sulfide, although phosphine is a useful chemical as a reducing agent and for other reasons, due to its toxicity the author must advise against using phosphine for any purpose.

The scrubbing of exit gasses containing phosphine can be accomplished by nearly any oxidizing agent, hypochlorites, permanganate, ferric salts, even nickel salts will be reduced by phosphine. The most though method though to dispose of phosphine is to run it into an open flame, this will create a fine dispersion of phosphorus pentoxide and therefore it is advisable to remain a distance away from the flame, because although not poisonous it may cause damage to the throat and lungs.

Propane $CH_3CH_2CH_3$: Propane follows along the same lines as the other hydrocarbons in this list (Butane and Methane) in that it is relatively unreactive to many conditions except oxidation once a certain activation energy is achieved. It is widely available at a decent purity and can be used as an inert gas in some situations. It is an asphyxiant gas and is flammable so should not be allowed to accumulate. Therefore care should be taken to ignite the exit gasses as they pass though otherwise they may accumulate.

Silane SiH_4: Yet another spontaneously flammable gas at STP when exposed to oxygen:

$$SiH_{4(g)} + 2O_{2(g)} \Rightarrow SiO_{2(s)} + 2H_2O_{(g)}$$

Described as having a 'repulsive odor' silane has few if any common uses in chemistry. Industrially it is used to produce amorphous silica. Of course that means that when it ignites on its own it will make a cloud of very fine silica which if inhaled will do damage to the lungs, inhalation of fine silica can cause cancer actually. Silane is highly reactive, there are methods in which a chemist could utilize it, for example, reacting it with chlorine would produce silicon tetrachloride, an interesting chemical with a wide variety of uses. But who would want to use such a toxic spontaneously flammable chemical? The normal method of producing silane is to react magnesium silide with acid:

$$Mg_2Si_{(s)} + 4HCl(aq) \Rightarrow 2MgCl_{2(aq)} + SiH4_{(g)}$$

Mg_2Si could in theory be made by direct combination of the elements followed by heating. However it is easiest to produce it in situ by a thermite type reaction between an excess of magnesium and silicon dioxide:

$$3Mg_{(s)} + 2SiO2_{(s)} \Rightarrow 2MgO_{(s)} + Si_{(s)} + Mg_2Si_{(s)}$$

It is not recommended to use a great excess of magnesium in this mixture, the above equation showing slightly more then is used in practice. If too much magnesium is used, the already difficult to ignite mixture may become impossible to initiate. The mixture thus obtained could be used directly to produce silane.

When present in the exit gasses of a reaction silane can easily be destroyed by running though water, however somewhat basic solutions work even better. The exit gasses may also be destroyed by burning,

Solvent Vapors: In general solvent vapors should not be vented as most every solvent is highly flammable with the usual exception of water. As such the usual method of disposal of solvent vapors is by incineration. The problem with incineration occurs with halogenated hydrocarbons. Their incineration entails the production of hydrogen halides which are one of the gasses specifically described in this test to dispose of in its own way, incineration not being one of them. If your exit gasses do contain hydrogen halides it may be good to attempt to take them out by running them into a beaker full of ice, to re-liquefy them and therefore not have to deal with their gasses. They can be burned, but as stated the production of another nuisance gas is usually not a good reason to burn them. Many of the more halogenated hydrocarbons will not burn easily though (Chloroform, carbon tetrachloride). Although against environmental regulations one may be forced to vent them. However the author of this texts highly recommends finding another method of dealing with the exit gasses of these.

Sulfur Dioxide SO_2 WS: Sulfur dioxide is a useful easily condensed gas with a boiling point of –10 °C. There are a number of preparatory methods for this gas including the most obvious of burning sulfur:

$$S + O_{2(g)} \Rightarrow SO_{2(g)}$$

However this method is difficult to control for a constant source of the gas, as such the addition of oxidizing materials may be necessary to provide a reliable burn rate, in the olden days ‘sulfur candles’ were designed to accomplish the reliable burning of sulfur to sulfur dioxide. In some instances it has been proposed to use some brands of road flares for the preparation of sulfur dioxide but the gasses thus produced usually include some water and carbon dioxide as well. Additionally sulfur dioxide can be prepared from non-combustive methods, notably the reaction between hot concentrated sulfuric acid with copper metal which gives sulfur dioxide and water as the exit gasses. Another notable method involves the reaction between a bisulfite salt ($-HSO_3$) and an acid such as sulfuric or hydrochloric. What this reaction actually does is to drive an equilibrium in the solution toward sulfurous acid which cannot exist in solution in concentrated form, due to this when the concentration increases it leaves the solution as sulfur dioxide. This method is one of the easiest for small amounts of sulfur dioxide to be prepared. Sulfur dioxide is also found in some high-end batteries condensed to a liquid.

Sulfur dioxide is a good reducing agent both in aqueous solution and in other conditions. In aqueous solution sulfurous acid is first formed:

$$SO_{2(g)} + H_2O_{(l)} \Rightarrow H_2SO_{3(aq)}$$

This acid is easily oxidized to sulfuric acid and in the process reduces a number of compounds, for a simple example, copper salts are reduced to elemental copper. Additionally sulfur dioxide is used in the preparation of sulfuric acid by the contact process, it is passed over heated vanadium pentoxide on a material with a high surface area and sulfur trioxide passes out. This process is difficult to duplicate on a home scale though efforts have been made, additionally some transition metals serve as catalysts in the reaction of sulfur dioxide with water to form sulfuric acid, notably manganese salts though they can only achieve concentrations of ~40% before acid production comes to a stand still.

In addition to being used as a reducing agent and in the production of sulfuric acid, sulfur dioxide can also be used as a reagent for the preparation of other reagents, for example, the reaction of sulfur dioxide with carbonyl chloride at 200 °C (which, may I note is a very dangerous reaction) can be used for the preparation of thionyl chloride, a very selective and powerful chlorinating reagent in organic synthesis. There are a number of applications for sulfur dioxide in the lab, however it is very irritating to the eyes and throat. Exit gasses containing sulfur dioxide should be lead through basified water to remove traces of the gas.

Xenon Xe: The heaviest stable noble gas. Xenon has a place of its own due to its incredible reactivity. It will react with a number of very powerful oxidizing agents directly. Although of no consequence to many chemists at home it will react with elemental fluorine, oxides of fluorine, complexes of platinum with oxygen and fluorine, and a few others. But for most everything else it is a great inert gas. For additional properties see the other inert gasses. Xenon is available from specialty gas suppliers.

5.0 Simple Reactions

Types of reactions:

1) **Single Displacement: A + BC Þ AC + B**

Example: 2HCl(aq) + 2Zn(s) Þ H2(g) + ZnCl2

2) **Double Displacement: AB + CD Þ AC + BD**

Example: $2KBrO_{3(aq)} + BaCl_{2(aq)}$ Þ $Ba(BrO_3)_{2(s)} + 2KCl_{(aq)}$

3) Combination Reaction: A + B Þ AB

Example: $8Fe_{(s)} + S_{8(s)}$ Þ $8FeS_{(s)}$

4) Combustion Reaction:

Example: $C_6H_{12}O_{6(s)} + 6O_{2(g)}$ Þ $6CO_{2(g)} + 6H_2O_{(g)}$

5) Decomposition: AB Þ A + B

Example: $(COO)_2Fe_{(s)}$ Þ $2CO_{2(g)} + Fe_{(s)}$

6) Acid-Base Reaction:

Example: $HCl_{(aq)} + NaOH_{(aq)}$ Þ $NaCl_{(aq)} + H2O_{(l)}$

5.1 Shifting the equilibrium, a basic reaction technique, and an essential one : Le Chatelier's Principle

A very large percentage, if not all the reactions that you will run across involve some sort of equilibrium. Since I stated that nearly every reaction involves an equilibrium many of the reactions that are considered productive have the equilibrium lie to one side of the arrow though some mechanism. Here are a few examples, which will help to explain this concept:

1. $NaCl_{(s)} + H_2O_{(l)} \Leftrightarrow NaOH_{(aq)} + HCl_{(aq)}$
2. $NH_4OH_{(aq)} \Leftrightarrow NH_{3(g)} + H_2O_{(l)}$
3. $Ca(NO_3)_{2(aq)} + H_2SO_{4(l)} \Leftrightarrow CaSO_{4(s)} + 2HNO_{3(aq)}$
4. $Na_{(s)} + KCl_{(s)} \Leftrightarrow NaCl_{(s)} + K_{(g)}$

The examples above illustrate different reactions, which can occur and are shifted though some process, some of which can be shifted back afterwards. Let me explain the reasoning behind each reaction now.

1. Sodium chloride, common table salt when dissolved in water undergoes an equilibrium reaction, although a vast majority of the sodium chloride dissolves forming sodium cations and chloride anions, a small percentage reacts with the water forming sodium hydroxide and hydrochloric acid. The reason for this actually relates to the individual components NaOH and HCl.

NaOH(s) –H2O→ Na+(aq) + OH-(aq)

$$HCl(g) \xrightarrow{H2O} H^+(aq) + Cl^-(aq)$$

5.2 Basic Chemistry Experiments [With Step By Step Explanations]

5.3 Basic Chemistry reactions [Simplified Explanations]

5.3a Preparation of Sodium Acetate

$$\mathbf{CH_3COOH_{(aq)} + NaOH_{(aq)} \Rightarrow CH_3COONa_{(aq)} + H_2O_{(l)}}$$

One hundred milliliters, over the counter 5% acetic acid (.8 M vinegar) is placed into a beaker. After this 3.0 g of solid sodium hydroxide in prill form is weighed out and put directly into the beaker with the acetic acid [Note: 6.5 g of sodium bicarbonate may be substituted for the sodium hydroxide] , the mixture is stirred occasionally until all the sodium hydroxide has dissolved and then the mixture is placed on a hotplate and heating is commenced on a medium setting. The water is boiled off at a medium pace until the solution is less then 10 ml in volume and the heat turned down even further, the solution is observed carefully until it appears there is no further change in volume, it is removed from heat and allowed to cool. Shortly after removal of heat the solution should begin to solidify (however sodium acetate has a tendency to super saturate, meaning that scraping the sides of the beaker may be necessary to induce crystallization), the solid is allowed to stand until it comes to room temperature and then it is removed with the aid of scraping and placed into a storage container, yield is roughly 9 g or 100% of the trihydrate $CH_3COONa*3H_2O$ and is somewhat impure, it can be purified by recrystalization from a minimum amount of hot ethanol or methanol.

5.3b Preparation of Zinc Hyposulfite

$$\mathbf{SO_{2(g)} + Zn_{(s)} \text{ --(H2O)--> } ZnS_2O_{4(aq)}}$$

Zinc hyposulfite is an interesting and powerful reducing agent who's preparation is somewhat simple. Begin by putting together one of the setups listed in section 4.11 for the collection of a soluble gas and fill the gas collection area with 100 ml of distilled water to dissolve the gas. As seen above the gas to be solvated is SO2, please look to the section on the preparation of SO_2 to setup this part of the apparatus. Please pressurize the apparatus initially with CO2 by the addtion of acid to bicarbonate or a similar procedure to make sure the apparatus is air tight except the exit lines. Sulfur dioxide although not lethaly toxic has a strong physical destructive potential to the eyes, nose, and lungs. Please be careful in the generation of gas and the scrubbing of exit vapors to protect yourself. Finally once you are ready to begin and have your 100 ml of water ready to collect your gas slowly start SO2 evolution. The gas dissolves in the water somewhat rapidly, keeping your solution cool externally helps and at 0C your water should dissolve nearly 23 g of SO2 when it is saturated making a solution of sulfurous acid H2SO3.

Upon saturation allow your vessel to stand before taking it apart. To avoid the pain of SO2 gas you can have a large bucket of water prepared, and providing your glassware is cool your whole apparatus, minus your SO2 collection water can be slowly lowered into the water and taken apart under water. Finally after you are able to pay your sulfurous acid solution the attention it deserves put it into an ice bath to keep it cool for the next step. Take roughly 25 ml of your cold solution and add it to a small flask to which you have a lid. Add 8 grams of zinc in the form of powder and shake the solution stoppered until the solution obtains a uniform clear color again. Quickly filter this mixture and place the filtrate in a darkened bottle to prevent decomposition, repeat this in 25 ml portions for the remaining volumes. You are now left with a solution of usable zinc hyposulfite, use within a few weeks for the best results.

5.3c Preparation of Chloroform

CH3COCH3 + 3HOCl + NaOH Þ CH3COONa + CHCl3 + 3H2O

Chloroform is a useful solvent that is relatively safe and can be easily made from readily available reagents. It also found wide use in chemistry in older days and as such many older preparations call for the use of chloroform and with this reagent they can be followed as they were intended to be. You'll notice that the reagent in this reaction is actually HOCl, hypochlorous acid, this is the result of hypochlorous acid being such a weak acid that when sodium hypochlorite is in solution, the equilibrium favors the formation of HOCl and NaOH rather then NaOCl. Regardless, the reaction constitutes the reaction between 3 mols of sodium hypochlorite and 1 mol of acetone.

.75 mol of NaOCl (880 ml 6% NaOCl, 510 ml 10% NaOCl, 375 ml 12.5% NaOCl) is placed into a reaction vessel capable of holding it's volume plus 100 ml, the vessel if plastic will most likely suffer some attack, therefore glass is preferred. If possible the NaOCl is chilled prior to reaction to 0 °C or ice cubes are added, not many though as chloroform is slightly soluble in water and extra ice will decrease yield. To this chilled NaOCl is added 11 ml (slightly less then .25 mol) of acetone if possible the reaction mixture is kept cool in an ice bath, without the initial chilling and continued cooling the formed chloroform and reacting acetone will volatize off due to the increasing temperature although you will still get a product. The reaction is allowed to proceed for 25 – 50 minutes with agitation during the addition and at least once during the reaction. The induction period is 1 – 3 minutes if the mixture is pre-cooled but if it is warm it can be as short as 5 seconds. Finally after the reaction has been allowed to run to completion the top layer of the reaction liquid is poured off, this contains a mixture of sodium acetate which is benign, sodium hydroxide which also poses no hazard, but it does contain some partially chlorinated acetone and dissolved chloroform, do not dispose of in a manner that would allow it to run into rivers or into the water supply. The chloroform will have settled to the bottom in a blob that is more or less insoluble in water. This can be sucked out with a pipette or poured into a tall thin vessel where the water layer and chloroform layer become more distinct and separated. Your impure chloroform contains traces of carbon tetrachloride, water, and acetone. It can be purified further by shaking with a small quantity of water saturated with NaCl, dried in contact with H2SO4, and distilled. Yield will be based on the concentration of hypochlorite used and the temperature of the reaction mixture but should always be greater then 55%. In a series of trials using 10%

hypochlorite the average yield of chloroform was consistently around 70% (9 ml), accounting for solvation of chloroform (.8 g / 100 ml) yields approached 100% [98.5 being the highest].

5.3d Preparation of Benzoic Acid

$$\mathbf{C6H5COONa_{(aq)} + HCl_{(aq)} \text{ Þ } C6H5COOH_{(s)} + NaCl_{(aq)}}$$

As of writing this, sodium benzoate is a product currently available over the counter and even easier to find over the internet. It is used in food preservation and canning. And it has its own uses, notably in the preparation of benzene. However the conversion of sodium benzoate to benzoic acid has its own advantages, benzoic acid being useful for the preparation of other benzoates. Never the less this simple procedure is useful for the beginner in the methods of filtering and the difficulty in scaling up.

230 grams (~1.60 mol) of sodium benzoate was weighed out and placed in a beaker with 375 ml of distilled water. After stirring and a time of approximately 30 minutes the solution was considered saturated and a very small amount of undissolved solids was left. The solution was filtered into a 1 L Erlenmeyer flask and subjected to magnetic stirring. [Note that the solubility of sodium benzoate in water is approx. 66 g/100 ml] While stirring 250 ml of a 7.8M solution of HCl was measured out. This equates to approximately 1.9 mols of hydrochloric acid. One half of this hydrochloric acid is added rapidly as the mixture stirs and it quickly becomes too thick for a stir bar to handle. It is stirred manually for minute more and the resulting thick solution is filtered through a Buckner funnel with the solid portion washed each time with a portion of water, use about 100 ml of water to wash in total. [Note that there is enough product here to fill several Buckner funnels unless they are very large in size.]

The solid is removed from the Buckner funnel and placed on a paper towel to dry. Once all the solid has been filtered from the liquid it is again placed in the 1 L flask. Again under stirring the remainder of the HCl is added and the mixture again quickly becomes thick form the formation of the benzoic acid. After stirring a few more minutes to ensure a complete reaction the solid is again filtered from the liquid. Be sure to thoroughly wash the product this time as some NaCl will be in the solid product and water will efficiently remove it. [Note, the solubility of NaCl in water is about 35.7 g/100ml and the reaction will generate about 94 grams of sodium chloride so it is well within the solubility range, however once the excess HCl is in solution it depresses the solubility and some NaCl will come out.]

The resulting benzoic acid is placed onto a paper towel with the first batch to let it dry. It takes about three days at room temperature in a normal humidity to dry the product. It must be checked on daily and efforts must be taken to crush the pieces of benzoic acid into smaller pieces to make sure they dry entirely. It should be apparent when the product is dry because it will cease to stick together. [Note, once it becomes fairly dry it is quick to take to the air, the powder is fairly irritating to the nose and throat so care should be taken and a dust mask should be considered.] In one such trial of this reaction 175 grams of product were obtained resulting in a yield of 91%. The product must be stored in a glass or plastic container as, over time, it can attack metals. Also note that the drying can be sped up by heating but benzoic acid does have a low melting point.

6.0 Practical Concerns for running an amateur lab

Although it is not a nice issue to bring up, it remains true that in most places around the world, especially industrially advanced, chemistry at home is frowned upon. For example, in a place like the United States chemistry was a widely practiced hobby until the 70’s where environmental concerns and safety considerations made chemistry seem forbidden and dangerous. Saying that you are performing a chemistry experiment may shock people in your area and might force them to call the authorities.

The legality of the procedures that you perform usually will fall into questionable territory. Outside of using common over the counter reagents for their intended over the counter purpose you are walking a thin line. The use of sulfuric acid in the form of drain cleaner to act as a catalyst might not seem like a breach of the law but some chemicals will flatly state on the back of them that it is illegal to use that chemical for anything other then the instructions listed on the opposite side. Therefore it may be prudent to move all obtained chemicals to new containers, and if possible between the move, purify them.

Aside form these legality issues of chemical possession and use, you come to the disposal issue. You will not be able to recover every bit of product from every reaction, you won't be able to continuously run any series of reaction without generating waste along the line somewhere. You will have to dispose of this waste you create, and although the disposal is completely up to you, you should always dispose of chemicals in the most environmentally friendly way possible, disposing of certain chemicals, by dumping on the ground, flushing down a toilet, or throwing in the trash is a major crime that can bring about jail time, and or extraneous fines.

Also the illegal dumping of chemicals can cause immediate destruction to your local ecosystem. Your grass and trees may die, accidental releases are also a problem. The unintended release of large quantities of noxious gasses can also kill grass and make your neighbors life miserable. Increasing their likelihood on calling the authorities. Although you shouldn't have to sneak around in the middle of the night, which would make others more suspicious you shouldn't perform your reactions in the public eye. Doing so just increases that chance that someone will see you and object to what you are doing. And if that happens disastrous consequences can result, people with small children are often the most objective over chemicals, but, it is a reasonable response on their part.

Considering all of these aspects it is usually a good thing to not flash around the fact that you have a chemistry hobby. Your chemicals should be kept under lock and key just in case anyone wants to get to them just for the fun of mixing something up. This whole situation is a sad one in some respects but it is the way that you should follow your hobby, carefully disposing of waste, not willingly divulging more information then you have to, and working out of the public eye.

6.1 Starting up and Stocking your Lab Space (The essentials)

Here is a table of some of the basic items that should be in any laboratory, those highlighted in **green** are indispensable, you can do chemistry without them, but nothing that would be considered quantitative or productive. Those in **orange** are very useful and should be purchased at first convenience, and finally those in yellow are quite useful, you will want to own these after awhile.

Item	Description	Source
Test Tubes	Roughly 20 – 30 ml in volume, borosilicate is best.	Chemistry Supply
Beakers	A variety is best, small (100 ml), large (500 ml), medium (250 ml) are most used.	Chemistry Supply

Test Tube Holder	Holding more then one test tube, allows for viewing of reactions and hands free manipulation.	Can be made at home or purchased.
Graduated Cylinder	A 100 ml cylinder is the staple for precise measurements of liquid.	Chemistry Supply
Watch Glass	A concave circle of glass, for covering beakers or evaporating liquids to obtain crystals.	Chemistry Supply (Many objects can suffice as watch glasses)
Thermometer (glass)	The normal range is from –10C to 150C the higher and lower the better.	Grocery store (Candy Thermometer), Chemistry Supply
Pipette / Eye Dropper	For dispensing small quantities of liquid or sucking up different layers of liquid.	Pharmacy, Chemistry Supply Store, Grocery Store
Funnel (glass)	For pouring liquid and for filtration.	High end cooking stores, chemistry supply
Scale	Digital or balance, should be able to measure to the gram.	Office supply stores for weighing mail, internet
Vacuum Source	To create suction for filtration or distillation.	**Hand**: Auto store (for break lines), Internet **Mechanical**: Salvaged Compressor or Medical/Chemical Supply
Heating Source	Torch, coil heater, hot plate, alcohol lamp, etc.	Most any place
Stirring Rods	Glass is the best	Chemical Supply, can be improvised easily.
Scoops	To remove reagents from jars for weighing or use.	Long plastic spoons can be readily purchased at grocery stores.
Reagent Bottles	To keep the things you make and acquire	Keep your eye out.

These items are all useful as you will find out in your experiments. But the necessary chemicals to do reactions are really non-existent. This is because each chemical usually opens many possibilities for experimentation. To start out though it is good to have one acid, any acid will do, even vinegar, and one base, sodium bicarbonate for cooking works for this. That is already a good start. With acetic acid you can create acetates, which are luckily one of the classes of compounds that are widely soluble. With it you can dissolve aluminum, magnesium, iron, zinc and some other metals and you can also add hydrogen peroxide, even the concentration available for cleansing wounds and it will attack even more metals. Form here you can evaporate solutions o f metal salts and collect them for later use. Each chemical created opening more possibilities to make still more chemicals.

6.2 Legality

6.3 Storage of chemicals, Separation of reagents

6.4 Disposal of waste materials generated

Treating wastes containing hydrazine with excess sodium hypochlorite converts the hydrazine to harmless nitrogen gas.

When it comes to disposal the home chemist must do what they can to ensure their chemicals are disposed of in the most heath conscientious and environmentally friendly manner possible. Though there are usually some compromises to be had that is the overall goal in the disposal of hazardous reagents. To get straight to the point let us start by taking at look at inorganic chemicals. Believe it or not some metal cations are themselves hazardous, for example barium wastes can cause sickness readily upon skin contact if they are appropriately soluble, so the solution is to render them insoluble, treating a soluble barium salt with sulfuric acid or a sulfate leads to the precipitation of highly insoluble barium sulfate, a chemical so insoluble that it is actually intentionally consumed prior to X-rays of the gastrointestinal tract to outline the details. There are also anions that are inherently hazardous, the most famous being the cyanide anion (CN^-), cyanide can be converted to the considerably less toxic cyanate anion by simply treating a soluble cyanide solution with excess bleach. I therefore present these tables, remember to consider both your cation and anion in the method of disposal. These are no where near perfect solutions to the problem of waste management, however they are the best us chemists can often do, attempting to turn the chemicals to the least soluble compounds where they can do no harm or into the naturally occurring form of the compound. [Note: For those of you with septic systems take extra care, you do not want to kill the bacteria involved in destroying your organic wastes, be careful with what you flush.]

Cation disposal options:

Cation	Disposal	Cation	Disposal
Aluminum (Al^{3+})	Although aluminum salts are implicated in the cause of	Antimony (Sb^{3+})	Conversion to the sulfide*.

	Alzheimer’s, they are usually safe for disposal.		
Arsenic (As^{3+})	Conversion to the sulfide*.	Barium (Ba^{2+})	Conversion to the sulfate, carbonate**, or sulfide*.
Beryllium (Be^{2+})		Bismuth (Bi^{3+})	
Boron (B^{3+})	Boron salts are not inherently toxic, though it doesn’t follow as set of a trend as some of the other elements.	Cadmium (Cd^{2+})	
Calcium (Ca^{2+})	Calcium salts do not pose any inherent danger due to the cation.	Chromium	
Cobalt (Co^{2+})		Copper (Cu^{2+})	Though copper salts are somewhat toxic, it is acceptable to dispose of them in the municipal sewer system, as evidenced by over the counter $CuSO_4$ root killer. Conversion to the sulfide is another possibility.
Gold (Au^{+})		Iron (Fe^{2+})	
Iron (Fe^{3+})		Lead (Pb^{2+})	
Lead (Pb^{4+})		Lithium (Li^{+})	Soluble lithium salts are neutrotoxic, reaction with steric acid gives insoluble soaps.
Magnesium (Mg^{2+})	Magnesium salts do not pose any inherent danger due to the cation.	Manganese	
Mercury (Hg^{+})	Conversion to the sulfide.	Mercury (Hg^{2+})	Conversion to the sulfide, reduction may be necessary.
Molybdenum		Nickel (Ni^{2+})	Reduction to the base metal, alternately conversion to the carbonate** or phosphate.
Silver (Ag^{+})	Reaction with a soluble chloride, bromide, or idodie.	Sodium (Na^{+})	Sodium salts do not pose any inherent danger due to the cation.
Strontium (Sr^{2+})		Tantalum	
Thallium (Tl^{+})		Tin (Sn^{2+})	
Tungsten		Zinc (Zn^{2+})	Zinc salts do not pose any inherent danger due to the cation.

* = In the case of a soluble salt, the solution containing the cations my be treated with a soluble sulfide (such as sodium sulfide) or with hydrogen sulfide gas (Dangerous, see section *4.13 Gasses* for the entry on hydrogen sulfide), usually in the case of the sulfide it is preferred as the disposable form of the cation as it is both highly insoluble (except in highly basic or oxidizing environments) and that it is the naturally occurring mineral in

which the element is found. In all instances where the sulfide is the preferred final form it will precipitate from the solution whereupon filtration is possible to remove it.

**=Conversion to the carbonate is accomplished by first making the solution neutral or slightly basic and then adding a solution of a soluble carbonate such as sodium carbonate. Also the solution can be made fairly basic with hydroxide (assuming the hydroxide is soluble) and then carbon dioxide can be bubbled into the solution to precipitate the carbonate.

Anion Disposal Options:

Acetate ($C_2H_3O_2^-$)	Acetate poses no unusual hazard for humans.	Azide (N_3^-)	Azides can be unpredictably explosive, in solution they can be treated with sodium hypochlorite to convert them to nitrogen.
Bisulfate (HSO_4^-)	Although acid in nature it poses no unusual hazard.	Sulfate (SO_4^{2-})	Sulfates are fairly benign.
Sulfite (SO_3^{2-})	A weak reducing agent, sulfites can usually be disposed of with no further treatment.	Thiosulfite ($S_2O_3^{2-}$)	
Sulfide (S^{2-})	Sulfides should not be treated with acid as hydrogen sulfide may be evolved, many sulfides are insoluble enough to be safely disposed of, sulfides of the alkali metals should be treated with a solution of cation that will form an insoluble sulfide, such as copper.	Borate (BO_3^{3-})	Borates can be mildly basic but nothing of major concern.
Bromide (Br^-)	The bromide anion can have a sedative effect on the human body but that is only in large concentration, overall bromide is safe for disposal.	Hydrogen Carbonate (HCO_3)	Only the alkali metals and a few others can form the hydrogen carbonate salt, this salt is safe enough to be disposed of in nearly any manner.
Carbonate (CO_3^{2-})	Aside form the alkali metals and some of the alkali earths the carbonates are fairly insoluble, the carbonate anion is non-hazardous.	Chloride (Cl^-)	The chloride anion is non-hazardous and can be disposed of in nearly any manner.
Hypochlorite (ClO^-)	Hypochlorite is a good oxidizing agent but can safely be dumped down drains in normal amounts.	Chlorite (ClO_2^-)	
Chlorate (ClO_3^-)		Perchlorate (ClO_4^-)	The perchlorate anion is suspect as having a long term enviormental impact, excess perchlorate can be disposed of by heating to decomposition with charcoal (**Note**, some perchlorates are violently

			explosive on their own, take caution!)
Chromate (CrO_4^{2-})		Dichromate ($Cr_2O_7^{2-}$)	
Cyanide (CN^-)	Soluble cyanide solutions can be treated with excess hypochlorite solutions to oxidize them to the considerably less toxic cyanate.	Cyanate (OCN^-)	Can be disposed of as long as it is not in any area where it may be consumed by animals and humans.
Fluoride (F^-)	Soluble fluoride compounds are toxic, treatment of fluoride solutions with calcium cations is recommended to precipitate the fairly safe CaF_2.	Formate ($HCOO^-$)	Formate is toxic however provided the amounts are not excessive it may be flushed down the drain as products that contain it are disposed of in this way.
Oxalate ($C_2O_4^{2-}$)	Conversion of soluble oxalates to calcium oxalate (insoluble) is one method, all oxalates are subject to thermal decomposition so just heating to ~250 °C should do the trick, be warned CO may be produced.	Hydroxide (OH^-)	Aside from basicity the hydrodroxide anion has no terrible toxicity and can safely be flushed, even concentrated solutions can be dumped providing there are no hazardous cations associated with it.
Nitrate (NO_3^-)	Nitrates are good for the grass, you can dump them outside in some situations though concentrated solutions will kill the grass. Do not dump where it can go into rivers or streams, destroy by heating to dryness and mixing with carbon and igniting.	Nitrite (NO_2^-)	Addition of acid will destroy nitrite in solution but converts it to toxic NO_2, heating to dryness and mixing with carbon and igniting is another option. Nitrite should not be dumped.
Peroxide (O_2^{2-})	Strong peroxide solutions can get out of hand during destruction procedures. Strongly basifying a peroxide solution will destroy it in a few hours, adding MnO_2, iron salts and some other catalysts (such as iodine) will also destroy inorganic peroxide as can reducing agents.	Phosphate (PO_4^{3-})	Do not dispose of in massive amounts in waters that may lead to rivers, phosphate can lead to bacterial blooms in waterways. Other then that phosphate is fairly environmentally benign, it helps flowers bloom brighter, assuming the pH is properly adjusted it can be dumped.
Permanganate (MnO_4^-)	Initial reduction of permanganate can be accomplished by just allowing a solution of it to stand for several days or with reducing agents. After reduction you are left with soluble manganese salts and manganese dioxide which can easily be disposed of.	Iodide (I^-)	Toxic in large amounts but safe to dump.

When in doubt, organic materials can be readily destroyed by the addition of Feonton's Reagent, which is a mixture of strong (>20%) hydrogen peroxide with a soluble iron +2 compound. It is believed that the power of this solution to oxidize nearly anything organic (benzene rings and chlorinated hydrocarbons readily succumb to this treatment) is the result of the formation of the hydroxyl radical HO* in solution which is highly reactive.

6.5 Considering your neighbors/neighborhood in every reaction

Usually when considering a reaction you will consider your own safety, a gas mask, gloves, and you might weigh the possibility of catastrophic error against your life and decide that the reaction is feasible. But before doing any reaction think about how these things relate to your neighbors. You can't have a faulty distillation apparatus spewing out ammonia gas just because you are wearing a gas mask. Your neighbors do not have that luxury. Maybe your reaction could run away and explode, you might be willing to risk that, but you cannot assume your neighbors would. However there is a linear relationship with what kind of risks you can take and how far away from you your nearest neighbor is, it stands to reason that if you live in a remote area you can get away with doing nearly any chemical adventure you can fathom for yourself, likewise if your neighbor is only a dozen meters away then what you do becomes more dependent on how it will affect them.

Not really something to be covered in detail here, it is more of a general awareness that you are not alone in your endeavors, your waste disposal, privacy, and your major risks should all be contemplated with your neighbors in mind. Not only for your sake, for fear of an errant call to the police, but for theirs as well. If you are comfortable with your neighbors you might tell them that chemistry is your hobby, it will make them feel safer then looking in their backyard and seeing you stroll around in a hazmat suit with a self-contained breathing apparatus strapped to your back and assuming the government has sent an agent to probe around the neighborhood.

6.6 Privacy & Security

7.0 Choosing your own experiments

A good way to figure out what experiments you want to do on your own is to keep track of what experiments you see. Whenever you're reading a book or surfing the internet and you come across an interesting reaction, one where you have all the reagents or they are readily available, an experiment where you could make a product that could be useful in creating another product down the road, or maybe just something that changes color in just the right way to catch your fancy, copy it down into a book. Be sure to write down whatever important reaction details it gives (temperature, pressure, stirring, etc.) and a complete chemical reaction if it is available. Also be sure to include some bibliographical information as to where you found this source so if you ever have to cite it or go back to it you won't have any difficulties.

7.1 Researching

As has been said time and time again, you do not want to re-invent the wheel. If someone has already gone before you and discovered the best way to a compound or already tried a modification you have on your mind it is very helpful to find this information before actually digging into something. The internet is a great guide but books are the best resource, try to find a local library, even college libraries do not usually require you to be a student to use their resources.

7.1a Internet

The internet is a good starting point for nearly any chemical research to be done. The one thing it is great for is giving an overview of the current knowledge available. However as many people here are aware there is no policy against posting complete and utter garbage on the internet and passing it off for the gospel truth. There are no checks and balances on the internet to prove that the information that you have is correct. However it is usually easy to get a feel for a site and determine weather it seems legitimate. On top of superficial looks, most good information that you find online is either backed up with direct experimental evidence by the individual posting it, or better yet, references to written works. Now, both of these can be faked as well, they simply add credence to whatever information you are reading. Further research can be done from here, looking up anything you can find on the individual posting this information and determine their credibility and also looking up references in paper form, which is something you should probably do anyway if the information that you are looking for has any true importance to you.

Finding good information on the internet requires that you use search engines with very large databases, at the time of writing this google.com and yahoo.com being some of the largest in the English speaking world. However the more languages you know the better, this allows you to use other search engines in different languages, not to mention the obvious fact that you are capable of reading those languages.

Whole books can be found online if you only search, *.pdf files being the most common format for them, however *.djvu is also an up and coming format. Some of the more advanced search engines allow you to specifically search for file types under the advanced search options, a button for which is always located somewhere near the actual search button. This should not be something done consistently though, as there is much good information to be found in normal html files online. The normal methods to refine a search still hold true when searching for chemistry information:

Problem:	Solution:
Too Many Results	Results should be narrowed by taking advantage of quotation marks around phrases that are unique to your subject or multi word chemical names, i.e., “Potassium Cyanide” vs. Potassium Cyanide. Some of the more common chemicals can also show up frequently on pages that are hardly related to chemistry, a little hard chemistry can help sort them out. For example, lead chromate is a chemical that is often found in paints as a yellow pigment, however by searching for PbCrO4 many results (although some useful results) will be filtered out. Other key words that help to give chemistry specific results include yield, precipitate, solvent, cation, anion, oxidized, reduced, and innumerable other words, trying different combinations can always help to reduce the amount of irrelevant information you come across.
Too Few or no Results	Determine synonyms for your name or reaction of interest. For example mercuric chloride can also be known as mercury (II) chloride. Try chemical formulas and if using quotation marks remove them. Check your spellings and try to trace your steps backward by looking at pages that mention the processes leading up to the process of interest or pages that m ention things relating to your compound of interest to best determine changes in your search that you may have to make to increase results, always give a different search engine a try if you are having trouble finding anything.

Really though there are many sites online that can teach you to search for documents of all kind using search engines. Just remember these key things:

1) Many compounds have more then one name to describe them, especially organic compounds, but also inorganics such as $FeSO_4$ (Ferrous Sulfate vs. Iron (II) Sulfate vs. Iron (II) Sulphate vs. Ferrous Sulphate).

2) Chemical formulas can hurt or help your searches, be sure to try both, also they can be helpful for finding the names of unknown compounds.

3) Using words that are directly related to the reaction you are wondering about or products or precursors to a product in your search can help to find more pinpoint results.

7.1b Library

7.1b1 Following up on footnotes

7.1c CAS

7.2 Scaling up and Scaling Down

7.3 Being through

7.4 The Importance of Keeping a Log

When I first started doing experiments I would figure out how much of A to mix with B, write it down on a little scrap of paper, then read off that when I did the experiment. Later though I would wonder, "Did I mix 25 ml of A with 15 ml B, or was it 15 ml A with 25 ml B?" Or I would try to explain to someone exactly what I did different then them, only to find that whatever I didn't different wasn't something important enough to stick in my mind. It took nearly two years of running experiments for me to come to the conclusion that I needed some permanently bound book to write nearly everything that crossed my mind in, one reference work by me that I could add to, alter, and make notes in, and come back to again and again. And once I realized it for myself I made sure to stick to writing in it, even when it was less continent to drag it out then it was to just grab a scrap of paper and make a note.

7.5 Trouble Shooting

7.6 Words of encouragement, stories of "Try Try Again"

7.7 Lengthy Story about procedure with details

8.0 Advanced Techniques

8.1 Working at high temperatures

8.1a Advanced Heating techniques

8.1b Fuel

1) Charcoal/Coal:

Charcoal and coal are the simplest and initially least expensive fuels to use for a furnace. All one needs is a blower to force air into the furnace, and charcoal to put in the furnace. Although a hair dryer will work, it will not work as well as more powerful blowers, such as the blower from a dryer. Charcoal is easier to obtain than coal, and can easily be made from wood, but because it is much lighter, small glowing fragments shoot continually into the air from the furnace, reaching heights of several meters. Both charcoal and coal provide enough heat for practically any furnace use, from melting metals to forging iron. The main problem with both is the mess; charcoal will land in crucibles full of molten metal, and can be skimmed out, but is still a hassle.

2) Wood:

Wood can be used instead of charcoal and coal if they are unavailable, and works very well as a fuel if split into small chunks. Wood has been used for centuries to fire pottery to a glowing yellow-white heat, and if used correctly can easily get that hot. The problem is that it burns so rapidly that more must continually be added, which is awkward, and it tends to land on top of crucibles and other apparati in the furnace, and burns above them, meaning that they don't receive much of the heat. This can be solved by making a furnace or kiln with a chamber below or to the side of the main chamber into which the wood is added and through which the air blows on its way to the main chamber so that all of the heat reaches the furnace charge.

3) Oil:

Oil may be the cheapest fuel in the long run because it can be obtained from restaurants for free, but it takes special equipment to burn. Oil will not easily ignite as a liquid because of its very small surface area. This can be solved in two ways; either the oil is atomized into small drops or it is vaporized by heat. The oil can be atomized by stretching it into a thin film, which is then broken up. This is accomplished by injecting the oil through a special nozzle into the furnace under pressure, which is the approach that most commercial oil burners use, by stretching the oil as a thin film over a sphere with a miniscule hole out of which high pressure air is blown (babbington burner), or by rotating the oil in a cup; as it is flung off of the walls of the cup, an air blast breaks it up into a spray. (rotary cup atomizer) The problem with this approach is the special equipment required. The other approach is much simpler. A smaller chamber or the furnace itself is preheated using wood or other fuels, and oil is then dripped or injected into the chamber along with air from a blower. The oil hits the walls of the chamber, vaporizes, and combusts; the process is self-sustaining. The problem with vaporizing oil is that there are almost always cracks through which the partially burnt vapor leaks, causing a very unpleasant and probably unhealthy stench. Also, this method takes several minutes of preheating, and is difficult to get running smoothly. There is a commercial product called the Isomax Blue Flame Burner ® which is a tube with a side passage used to recirculate preheated air. Propane is used to preheat the system, and then oil is injected into the side tube; it vaporizes, creating a blue flame as it returns to the main tube, and then is shot into the furnace. Part of the flame is recirculated into the secondary tube, and used to vaporize more oil.

8.1c Refectories

1) Binders and aggregates:

Refractory compositions have two essential parts; binders, which are the glue used to hold the composition together, and aggregates, which are the main bulk of the composition. One could make a composition composed solely of a binder, of course, but as binders are usually more expensive than aggregates, and aggregates add desirable properties such as thermal insulation they are almost always used. Certain aggregates work best with certain other aggregates and binders; large amounts of both acidic and basic components should not both be used in the same refractory composition, as the composition may melt. A small percentage of components, which are fluxed and melt are sometimes actually beneficial because they form a glass gluing the rest of the particles together (perlite for example).

2) Commercial refractory compositions:

Commercial refractories mainly use binders based on three categories of anions; aluminates, phosphates, and silicates. Calcium aluminate, or ciment fondue, is created by the heating of calcia and alumina by charcoal in a blast furnace; the two components melt, are poured into ingots, crushed, mixed dry with the aggregate, and later cured by the addition of water. Calcium or aluminum phosphate binders are created by adding reactive alumina, or calcia to the refractory aggregate, and then adding orthophosphoric acid solution, forming a metal phosphate and water. Some compositions using alumina and phosphoric acid remain puttylike and unhardened until being fired. Furnace cements sometimes use alkaline silicates (eg sodium silicate) as a binder. Other refractory compositions, such as are used in arc furnaces for steelmaking, use phenolic resins which decompose to glassy carbon as a binder. There are many different commercial refractory aggregates; most are neutral compounds because of their greater compatibility, such as alumina, silicon carbide, etc. Where the binding agent is not ceramic (such as phenolic resins) the acidity or basicity of the aggregate is not important, and thus basic oxides with very high melting points such as CaO or MgO can be used.

3) Homemade refractory composition:

Obviously one simply duplicate a commercial refractory composition, but it is usually more feasible to use inexpensive compounds, such as clay. Various recipes have been made which use Portland cement, which contains a high percentage of calcium silicates. The calcium oxide in Portland cement is obviously a flux at high temperatures, and thus lowers the melting point of the composition, but cement does allow the composition to cure at room temperature, whereas pure clay only reaches its full strength after being fired to its maturing point. The outer parts of a clay based refractory furnace will never reach full strength, but this is usually not a problem. Essentially the recipe consists of approximately 15-30% clay; ball clay, kaolin, or fireclay work, but not bentonite, which looses its strength at high temperatures. The rest of the composition is composed of aggregates such as sand, whose main advantage is its low cost, and perlite, which decreases the strength of the composition because of its porous nature, but greatly increases the insulation value. Paper, wood chips, sawdust, or other combustibles can also be added to increase the insulation value as they burn out. Obviously the composition used will be based on what is available; if one can obtain alumina or other higher quality refractory materials, they should of course be utilized.

Crucibles:

1) Commercial crucibles:

Commercial crucible come in many different formulations and sizes. The most common are small porcelain, fused silica, fused magnesia, or graphite crucibles which used for chemical analysis, and mostly larger clay/graphite or silicon carbide crucibles which are used for metalcasting. Porcelain is a mix of kaolin, flint (a natural form of silicon dioxide), and feldspar, and when fired creates a mix which matures around 1300-1450 deg. C, forming a clay body with a large amount of glass. Fused silica and fused magnesia crucibles are fused from almost pure silica or magnesia powders at very high temperatures. Graphite crucibles are almost pure graphite, and are assumably created from graphite and carbon forming resins such as pitch or phenolic resins. Clay graphite crucibles are formed of a complex mixture of chemicals, obviously including clay and graphite, but also silicon carbide and more silica than clay alone has. It is not known by the author whether or not the silicon carbide is created in situ from silica and carbon when the crucible is fired, or is merely added as a powder. There are many varieties of silicon carbide crucibles, but many are created by bonding silicon carbide particles with phenolic resins. Crucibles are formed into their shape by processes including presses or slip casting.

2) Homemade crucibles:

Since the average mad scientist does not have access to the materials needed to create fused silica or fused magnesia crucibles, they are limited to creating clay bonded crucibles or resin bonded crucibles. Although crucibles can be made using a potter's wheel, slip-casting is a much easier, more reproducible, and much more accurate process. In order to slip-cast a crucible, a slip is made. This consists usually of clay, to which is added 0.25-0.5% (by weight) deflocculant, which prevents the clay particles from adhering to one another. Sodium silicate is commonly used as a deflocculant. When 20% (by weight) water is added, the clay actually forms a liquid slurry (for formulas which involve other materials besides clay, these numbers may need to be adjusted), which is then stirred very thoroughly to eliminate dry pockets and voids. The slip is poured into a simple one piece or multi-piece plaster of paris mold with a cavity in the shape of the ceramic object desired. The plaster of Paris, because of its porous structure, dehydrates the slip, causing the parts of the slip touching the plaster of Paris to become solid again. After several minutes, the plaster mold is inverted, and while most of the slip pours and drips out, the slip adjacent to the plaster remains. After waiting approximately 20 minutes to allow the clay slip to dry out more, the plaster mold is then removed and the clay object dried further.

For an experimental study of refractory compositions see section 13.2

8.1d Furnaces (Intro by VooDooMan)

Well then I see you have ventured into the realm of HIGH TEMPERATURES - EH! Yes it is definitely a great thing to learn about and perhaps even put to use some day! Never the less on we go onto our wonderful trip..............The reason I par took in this particular section is because I my self have extensive knowledge on this area. I have my own Forge / Foundry that I almost every day to smelt bronze, brass, aluminum, and other interesting metals that melt under the melting point of my refractory (approximately 1800C).

Well then, on we go, the basic idea of a forge/smelter/foundry and related, is to produce massive amounts of heat faster then it can dissipate to the surroundings to accomplish productive work. There are several types of furnaces, the first being (Which I believe is the one most used throughout the steel industry) the gas-fueled furnace. The other important type is the coiled electrical unit, more often then not, running on 220V for some serious power. Last but not least is the good old charcoal furnace, although outdated, it is the easiest furnace to get into the high temperature realm with.

When speaking strictly of the gas furnace, the way the flame from a gas burner is injected into the forge is of a critical interest to us. It is projected slightly higher then the bottom of the actual crucible and at an angle (not straight on but sideways so that the heat spirals up and around the crucible for uniform heating of items inside. The electrical unit does much the same thing but since it doesn't s pot heat, it is a better choice for pottery uses, the resistance elements are imbedded into the refractory / firebrick in a circular pattern all the way to the top of the forge.

Now then onto the actual guts of what makes a forge or foundry have a special zing to it. Any retard can heat a crucible to hot temperatures in a tin can and say look mommy I melted aluminum. Stop there, that is exactly the wrong idea. The whole idea of the forge is to keep hot temperatures in and only in with minimal heat escape when needed. That is why in most cases the refractory is several inches thick lining more then ¾ of a forge and only letting the crucible sit into it and a little room for air to be heated.

The composition of firebrick and refractory (pour able type of firebrick) are all-similar yet each has its own unique characteristic. They are more then often several types of sand mixed in with special customer wanted materials for special jobs. Also insulators like perlite or vermiculite are added.

There has been new talk of using recycled oils like from your car to heat the crucible of a gas forge, I am unable to comment on this part but I am sure with some good PSI being pumped into the oil and squeezing it out a large flare it is more then possible to create such a burner with very low cost!

Although most of this talk was from a metal casting point of view, there are several ways these processes can come into hand wile working with chemicals. For example, to create mangallium the metal that is used in go-getters or fountains or strobe pots and related. It is a mix of magnesium and aluminum melted together at high temperatures to essentially fuse together to form the material.

To sum it all up, of course the world is endless and there will be new advancements for our fuels and burner designs with new and improved refractory to keep an insane amount of temperatures inside. But for now the use of ordinary gasses I believe is our best bet. It is the easiest to setup and make and the fuels are extraordinarily cheap to buy.

There most simple and versatile furnace design is a bucket lined with refractory, with a hole in the side near the bottom for an air blast (tuyere) and a lid also lined with refractory, with a hole in the center for exhaust gasses. This can be used for metal casting, calcining of various materials, and for conducting experiments in closed retorts, which can be hung through the hole in the lid and piped off to the side. Other designs are better for other things, which may be discussed later.

8.2 Fractional Distillation

Distilling hydrobromic acid (HBr) seemed to be simple enough so the amateur chemist thought that they would give it a shot. After mixing together the reagents in the prescribed manner the mix was put into a 1 L flat bottom flask connected to a still head, then to a lebig condenser and in turn to a vacuum adaptor and then a 500 ml round bottom receiving flask. A quick look in a book revealed that the Bp of the azeotrope would be 122.5 C. Heating was begun with magnetic stirring to keep the mix agitated. Some time later the mix began to boil. The first of the vapor to touch the thermometer bulb at the top of the still head only brought the temperature up in the mid 90's. The liquid condensed and dripped into the receiving flask and over the course of the next hour the temperature continued to climb, when it reached 118 C the chemist removed their current receiving flask and replaced it with a new once, they then set aside the forerun for future analysis and watched as the temperature continued to climb to 122 C where it held steady for over an hour, during this time over 100 ml of distillate came over. Shortly thereafter though the temperature began to drop as vapors no longer reached the bulb, signaling that the HBr azeotrope had finally finished distilling over and distillation could be discontinued.

8.3 Catalyst Tubes

The purpose of a catalyst tube is simply to increase the rate at which a reaction happens. They are used widely in industry, most notably in the production of sulfuric acid using a vanadium pentoxide catalyst. But the different chemicals coated onto the catalyst themselves along with the sheer number of materials that can be coated onto are as limitless as the possibilities that using a catalyst can open. But there are always things to consider, contact time with the catalyst, approximate surface area of the catalyst, most effective temperature and how to keep it there, not to mention renewing the catalyst, keeping it clean, finding a tube to hold it all in, running the gasses or liquids through it at a certain flow, etc. As you can see there is quite a bit to this little aspect of chemistry, despite this, catalyst tubes are something that a chemist should familiarize themselves with regardless of if they intend to utilize the information or not.

8.4 Inert Atmospheres

There are some reactions out there where you just don't want the air coming into contact with your product or your reactants. The solution for this is the removal of the components you don't want mixing with your solution. That is where inert gasses come into play. By being inert that does not necessarily mean that the gas itself it inert under nearly all conditions. Just that it is not going to precipitate in the reaction going on. Two extreme examples of this would be argon, which is for all intents and purposes, totally inert, and propane, which is not considered inert to most due to its high flammability, however hydrocarbons are fairly inert, just not with respect to oxygen and an ignition source, propane therefore provides a cheap and readily available inert gas, however it does have its problems.

When working with an inert gas the standard procedure is to run it through a closed system for a length of time to flush out any previous gasses, then introduce your reagents quickly, preferably under the inert gas to prevent recontamination of the environment. Then sealing the system back off except for an exit and slowly letting the gas continue running though it. There is the concern of what to do with the exit gasses, which will depend on your reagents and your 'inert' gas but that is a specific problem. Although less useful inert gasses can also be used outside of a closed environment. All inert gases are by nature asphyxiants and therefore the gasses should not be vented into an enclosed area. They can aid evaporation if slowly run over a hot solution and they can also be used just being sprayed into a beaker to provide some protection, argon which is heavier then air is especially suited for this.

- **Argon** : Argon is a good blanketing gas approximately 38% heavier then air and will sink into the nooks and crannies of a distillation apparatus. It is not known to combine with anything at STP, although it does form HArF when irradiated with hydrogen fluoride at -255C but it decomposes about -245C so you shouldn't have to worry about extraneous reactions. Couple that with the fact that it is widely available for welding applications (although there is a bit of a startup fee including a cylinder, which you can rent, and a regulator) argon is well suited for most applications. As an exit gas you only have to worry about any contaminates in it, argon itself can be vented to the environment with no ill effects.
- **Butane / Propane** : Butane, commonly available as a refill gas for lighters, and propane, commonly available for just about everything are both gasses at STP, both are easily liquefied under pressure and vapors from either of these gasses are heavier then the surrounding air. As such they collect in low spots, unlike argon both of these gasses are highly flammable in the presence of oxygen, as such systems using them should be thoroughly flushed first and should not contain oxidizing agents or compounds that can yield free oxygen/halogens. In addition care must be taken with exit gasses containing these flammable products, they should be lead directly into a burner of some sort where they can be burned without hassle.
- **Carbon Dioxide** : Available in cylinders for the carbonation of beverages or as 'dry ice' relatively pure carbon dioxide has its sources outside of the lab setting. This gas is also denser then air, however it is more reactive then the others. It will ruin Grignard reactions, react with hydroxides and strong bases of all sorts and

more, but it does have its uses, just be extra wary of reactivity. CO2, although not incredibly toxi,c can cause damage if inhaled in a concentrated form, but as long as it is used in vented conditions it is relatively safe.

- **Freon** : Freon is a tempting source for an inert atmosphere. First because it is widely available and second because the regulating equipment is sold right along side it. But that is where the benefits end, freon is flammable, and somewhat more reactive then these other inert gasses, it has oils in it and as for purity, well, it's a grab bag. Use it at your own risk.
- **Helium** : Helium is the most widely available of the noble gasses and can be picked up from many places for the purpose of filing balloons, it even comes with its own cylinder. However the purity of such forms is questionable, unless it is in the large industrial containers it is usually mixed with a certain percentage of air. Beyond this helium is an extremely light gas that will not blanket a majority of your vessel, however extended flushing can help to overcome this, the price of commercial helium does not make it economically feasible in light of its light nature.
- **Nitrogen** : Has a limited availability as a welding gas. Also available in the liquid form, but be wary of frost bite. Nitrogen is the staple inert gas for the organic chemist, usually on tap in fume hoods. It is fairly non-reactive, cheap and easy to transport. Nitrogen is roughly equal to air in terms of density due to it making up about 80% of our atmosphere. There is no need to worry about venting nitrogen gas.
- **Neon / Krypton / Xenon** : Availability of these nobel gasses is considerably more limited. They posses properties nearly identical to argon for reference. The exception to this being Xenon, which is the most reactive of this group, it will react with fluorine and some other high oxidizing molecules, but not to any extent that the amateur chemist should have to worry about.
- **Sulfur Hexafluoride** : Fairly inert gas used in blanketing the setup used for the electrochemical production of magnesium metal. Not widely available, dense gas, not exceedingly toxic, TLV 1,000 PPM.

Taking care of exit gasses depends on what exactly is contaminating them. Disposing of radioactive chemicals by incineration of exit gasses is not advisable but the decomposition of organic material by leading exit gasses through a flame works in most cases. If there is a specific component in the gas that you expect to survive the flame then take the extra step to bubble the exit gasses through an appropriate solution to neutralize the offending contaminate. Occasionally a reaction will be called for to run under a specific gas that does not seem to be inert, it may well not be. If something is run under a chlorine atmosphere you can be that it is probably necessary for the reaction to commence.

When discontinuing use of the inert atmosphere the vessel must become slowly accustomed to regular atmosphere again, unless the apparatus is to be disassembled under an inert atmosphere in a fume hood and cleaned there. Unless the reaction products are explosive (boranes, phosphines, etc.), or exceedingly pyrophoric, simply removing the tube for the exit gas and turning off the flow of inert gas will suffice to bring the vessel back into atmospheric conditions as the gasses inside diffuse out and the gasses from the outside slowly work their way in. Explosive mixtures between the air and flammable gasses may form so it is necessary to allow the apparatus to cool to room temperature before hand.

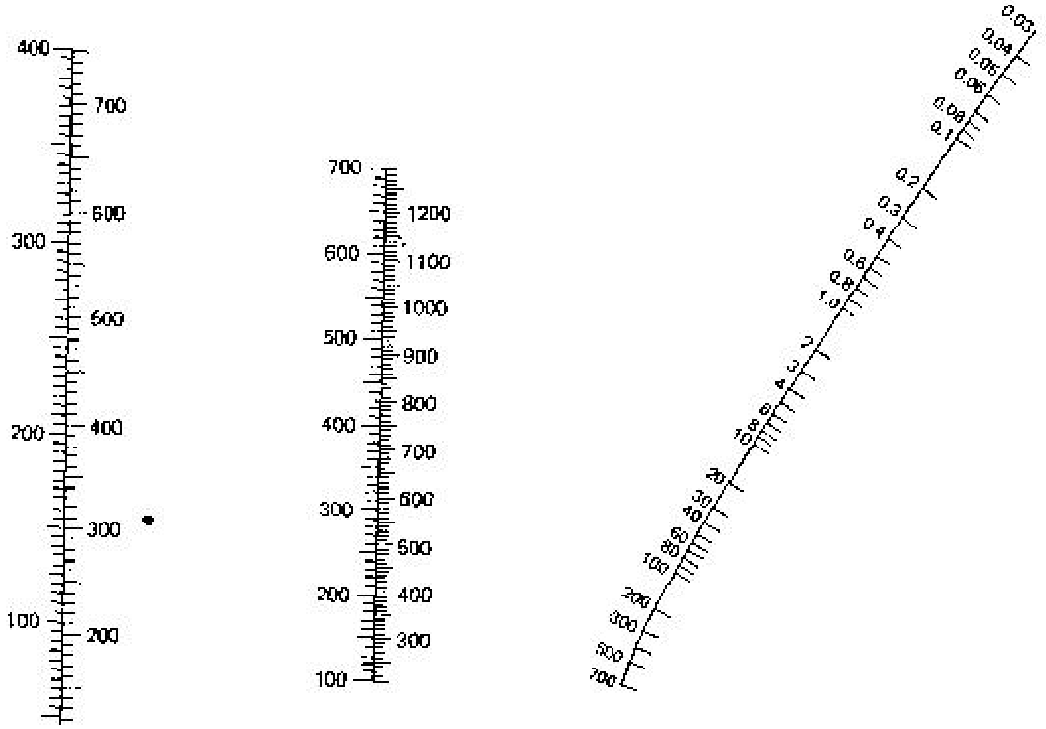
8.5 Solvent/Solvent extraction systems

8.6 Vacuum Pumps and working under Vacuum

Vacuum distillation is necessary when your compound decomposes when being boiled at STP. The premise of boiling is that the vapor pressure of your substance increases as it heats and when it gets to the boiling point, the vapor pressure of your substance is equal to the atmospheric pressure, by reducing the pressure you reduce the atmospheric pressure and therefore reduce the temperature needed to boil the liquid. It simply is distilling under reduced pressure and for the most part the techniques mentioned earlier regarding distillation can be safely applied here as well. There are however a few important observations. Boiling points of different components don't necessarily change in a linear way when the pressure is altered. Simply said, it could mean that whilst the difference in vapor pressure is 200mbar at STP, it could be 50mbar or 400mbar under reduced pressure. Note that there are a number of units with which pressure is measured:

	mm Hg/Torr	Atmospheres	Pascal's	PSI	in Hg/mbar
Atmospheric Pressure	760 torr	1 atm	101325 pa	14.69	~1001

Usually there is some indication, as to which pressure unit a book or text is using, and conversion between pressure units is a simple task involving just a constant, however these constants will not be listed here as they are many in number, however finding them online is a simple matter.



Using the above table called a nomograph one can figure out around where a liquid would boil. To use it you can copy it to your favorite paint program and on the right hand side you find the pressure at which you plan on distilling (in torr) and draw a line from that point through the line in the middle at the point of your boiling point at standard pressure and the intersection point on the left is in the region of your new boiling point. Simple and easy and it can give you an idea of how powerful your vacuum pump is.

The main considerations when distilling under reduced pressure is the quality of your glassware and pumps. Your lab glass needs to be borosilicate glass and it should be in prime condition. Cracks or even scratches severely compromise the strength of your glass under vacuum. Only round-bottomed flasks can be used during vacuum distillation, no flat bottom flasks or Erlenmeyer's! What could possibly happen you think? Well, imagine a flask filled with boiling ethanol imploding. It suddenly comes into contact with a surge of fresh air and whoooosh...flaming inferno all over and you being sprayed with glass pieces. That's why. Not to mention what would happen with acids, poisonous or otherwise hazardous substances.

So your glassware is in perfect shape. On to the other problems, boiling stones don't work under vacuum and boiling under vacuum can be very aggressive, so aggressive that the bumping can crack your glass, after which it implodes. Again, there are two solutions to this problem. A Claisen adapter (picture?) with a capillary tube that is immersed in the liquid and then provides bubbles is a way to go, or magnetic stirring, the authors personal preference.

Bumping

Bumping is the ominous word used to describe a sudden ‘bump’ in the distilling flask. This can physically cause the flask to jump up and usually results in liquid being projected from the liquid surface, occasionally all the way into the still head and over into your receiving flask, ruining a distillation. Bumping is caused by a number of factors in these situations but it comes down to a large bubble of gas forming and violently making its way to the surface of your liquid. If bumping occurs (and it will occasionally) the boiling flask should be removed from the heat for a few moments until the bumping is passed and then placed back on the heat, hopefully at a reduced level. If it occurs again, repeat. There are a few things that can be done to reduce/eliminate bumping:

- The distilling flask should be heated slowly and if possible the whole flask should be heated, not just heat from the bottom but form the sides too, as in a heating bath or heating mantle.
- Stirring should be constantly applied, usually the faster the better, be it magnetic or by an overhead stirring unit.
- A capillary tube can be immersed in the liquid to the bottom which can lead bubbles up and away before they become large and destructive.
- If your liquid is very dense small amounts should be distilled at one time, denser liquids have a greater tendency to bump.

It goes without saying that your joints should be sealed well. This can be accomplished by grease or commercially available teflon tape or specially designed teflon joint fitters, which are expensive. Don’t neglect this aspect, because a joint which fails after a while is another possible doom for you and your set up.

Special care should also be taken in the way you fix your apparatus. You must avoid any stress or strain caused by hanging flasks, flask that are being pushed up because they float in the water bath, etc. Carefully balance your apparatus before applying your vacuum.

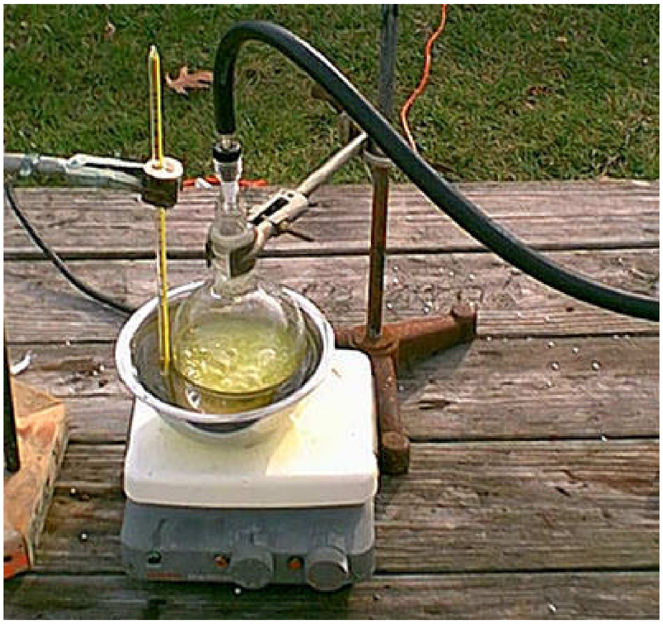
Heating is not an easy task when using vacuum. Flames or any other forms of localized heating are a NO GO. A water or oil bath is preferable. You must also realize that your set ups heat conductance is much lower because of the insulating factor of the vacuum. That’s why the author also recommends magnetic stirring of your bath, because this dramatically increases heat transfer. Otherwise you risk a bath, which is at 90C while the inside of your flask is at 30C, which causes a huge amount of stress and is generally energy consuming.

Finally, wrapping your glass with a wire mesh or laminating it with heat resistant plastic protects you from flying glass should something go wrong.

When first starting a procedure involving distillation using vacuum there is a sequence of events that one should follow:

Applying vacuum should be done FIRST, before heating starts. If you start heating first and then apply vacuum, there is a severe hazard that your liquid will flash boil when the pressure drops. Flash boiling usually comes with a pressure spike, insane bumping and a lot of frothing. Short & sweet, it means death to your setup and possibly to you. You also should start up your stirring or bubbling at the same time your heating starts, because if you do it too late, there is again a risk of flash boiling.

When you're done distilling, allow the apparatus to cool down first, then allow air to enter. If done otherwise, hot solvent vapor can come into contact with fresh air and reach the explosive range...which again means flaming inferno all over and flying glass.



Vacuum is being applied to this flask to remove ammonia as it is formed to drive the reaction foreword, the product remains in the flask.

9.0 When things go wrong

A mixture of barium chloride and potassium bromate explodes from the beaker creating a hazardous situation.

Accidents happen, you live long enough and you realize this, not just about chemistry but in every situation there is a probability for failure that people must live with. You hope for the best and take measures to prevent foreseeable catastrophes, but you cannot prepare for every occurrence, you'd never get anything done because you would always be worrying and planning, you don't want to over-worry about the details but you want to remain far from reckless.

Since preventing every accident might well drive you batty, cost large sums of money, and require you to work at a snails pace, lucky for most people, a good balance of hazard prevention coupled with some tips on managing a possible disaster will help to get you though most reactions.

9.1 Contingency Plans

In any reaction that has an abnormal risk it is always good to have a contingency plan. How can you determine if a reaction has an abnormal risk? In my opinion if you have to worry about a reaction then it has an abnormal risk, however you shouldn't have to plan for every possible screw up. Here is an example, you add a piece of sodium metal to some ethanol with the intent to make sodium ethoxide. However you find that your ethanol must be contaminated with a large water percentage, too bad you found this out when you added your Na to your ethanol. It's boiling and bubbling and H2 is coming off it like there's no tomorrow. If that H2 builds up the the reaction keeps up the Na will ignite it, possibly detonating your reaction vessel and thus spraying flammable liquid everywhere, most likely on fire itself, what are you going to do? Your normal plan to dump the offending reaction on the ground and spray it with water seems to be bad but it's always been your backup before. So you toss your reaction solution on the ground and spray it with your hose, the H2O hits the sodium and **Boom** not only does the sodium explode and spray the surrounding area with little chunks, but the ethanol was scattered too and is now burning merrily all over your grass and house and, wait, your arm's on fire.....

Don't tell me you didn't see this coming, Na can be quite nasty when removed from its anhydrous environment that it stays so comfortable in. You didn't have a back up plan and from here things could get even nastier. So, the obvious solution is to come up with a backup plan. So what could you have done in retrospect.... Sand is good for metal fires, that could have been a decent idea, maybe instead of water you could have tossed it in ice, that might have been slightly better but not a lot, how about having some real anhydrous ethanol on hand to dissolve excess sodium in, you know, like they do in professional labs. Regardless, it's always good to figure out what might go wrong before hand then to figure out why things went wrong after the fact. MSDS sheets can give a good indication of what to do incase a reagent gets out of control. In addition just knowing a chemicals properties can help.

9.2 Don’t Mix .... or....

9.2a Explosive mixtures involving oxidizing agents

9.2b Unstable Peroxides

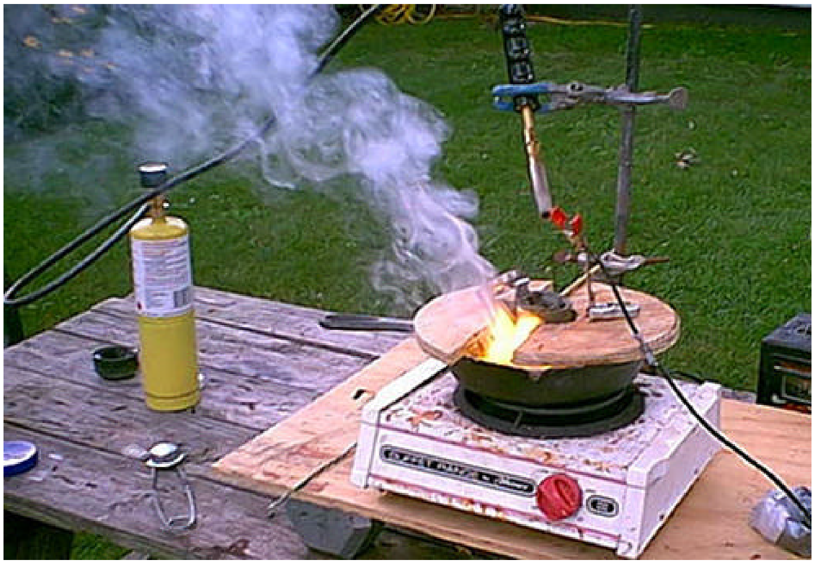
Peroxides by definition are chemicals that contain an oxygen-oxygen bond which puts oxygen in the –1 oxidation state, overall –2 as usual though since peroxides are by virtue of their nature diatomic. Most inorganic peroxides possess at least moderate stability, and if they do indeed prove to be unstable, they usually decompose without fanfare. For example, sodium peroxide Na_2O_2 has a defined melting point of several hundred degrees Celsius and does not decompose till far after that. Common hydrogen peroxide will break down with time but is comparatively stable except in highly concentrated from. Organic compounds however tend to form somewhat dangerous peroxides. Note that not every organic peroxide is dangerously unstable, however many of them are and it is best not to make them by accident or desire.

The figurehead of unstable organic peroxides that form spontaneously would have to be the peroxide of diethyl ether, or just ethers in general. A container of ether is opened, ether vapor escapes and air enters the vessel, it is once again closed and left to sit for awhile. In the absence of inhibiting agents most of the oxygen that entered the container will, in time, react with the ether creating the peroxide compound. A small amount of peroxide in this may go unnoticed as it is soluble and of such a low concentration. However over time it will accumulate, and may be mechanically accumulated unintentially, for example when evaporating ether from a reaction mixture. Peroxides can exist in many solvents that are available over the counter, because they were never intended for chemistry, so peroxide formation is never a major issue for suppliers. But you will notice, if you’re boiling off a large quantity of acetone which usually possesses almost no hazard, there may be sufficient impurities that may have led to peroxide formation, and after a large amount if boiled off these impurities may be of such a high concentration in your flask to represent a real explosive danger, therefore in cases where boiling down solvents of unknown purity and where you are working with ethers it is best to never evaporate to dryness.

Additionally organic compounds should never be mixed with peroxides, inorganic peroxides such as sodium peroxide for certain, but even hydrogen peroxide can possess a great degree of danger if it is sufficiently concentrated (>30%) when mixed with organic liquids. Aside from the radical reactions that cause the formation of peroxides in ethers peroxides may be unintentially created by the action of hydrogen peroxide on some compounds in the presence of acids or bases. Be wary of such complications, especially with ketones.

9.3 Flammability Concerns

Many of the reagents, particularly the solvents used in chemistry are quite flammable. However some posses specific flammability concerns. Highly flammable solvents include ether, which is known to creep along the ground for an ignition source, and carbon disulfide which can ignite from boiling water and a whole world of flammable hydrocarbons like propane and butane. It is good to know if you keep these in an enclosed environment without oxygen they will not spontaneously ignite, however they do get out. So as a common practice flammable liquids should not be heated with an open flame unless you are quite familiar with what might happen.



Someone Should be going for the Fire Extinguisher about Now (or at least turning off the gas!)

Flammability is always augmented if the word pyrophoric is involved. Some metal powders are pyrophoric (zirconium powder being a good example), some other solids are too (white phosphorus), as are some gasses (diphosphine), and liquids. After just a short contact with the atmosphere, either from reacting with ambient moisture or oxygen these may ignite on their own. These are extreme examples though, normally you need a spark to initiate a flammable air mixture (electric apparatuses for chemistry go to great lengths to avoid sparks whenever possible) or at the most an open flame. No smoking near any running reaction that contains anything flammable and note that fumes can travel a long way, just think of people who use paint stripper in one part of their house and the fumes creep along the ground till they come to a furnace or water heater and *BOOM* the doors go flying off the house.

9.4 When to abandon Ship

Although it's best to avoid potentially dangerous reactions that could lead to situations where you may have to 'run for your life' or possibly do whatever is in your power to bring a reaction to a halt at that moment, there are occasions, where, with proper preparations you believe that you can succeed in a reaction with little possibility of things going wrong. Too bad that things have a propensity to go wrong in chemistry. Knowing what could go wrong beforehand and mentally going over what you could do is a thing you should do for every reaction, big or small, in the simpiliest situation a chemist may be planning on dissolving a metal in a non-oxidizing acid, what are the possible things that could go wrong here?

A) The acid could spill what would you do? **Solution:** Sodium bicarbonate is good for acid spills keep some on hand.

B) If you have a very active metal and strong acid the reaction mixture may heat up and boil releasing acid fumes of making a breakage hazard. **Solution:** Adding water will dilute the reactants and cool the reaction, adding the mixture to a large bucket of water may halt it almost entirely. Bases may cause additional heat but could be used to halt the reaction if necessary and if possible removing the remaining metal could rectify the situation.

C) Aside from the acidity of a solution, what about the metal that you are putting into solution, are the cations toxic? **Solution:** Wear gloves, have water near by to quickly wash any contaminated area.

Now, this situation does not justify 'abandoning ship' just what could result in a minor spill or acid burn possibly some broken glassware. The major area in chemistry that has the greatest potential for harm would have to be energetic materials, although not within the scope of this text, these dangers can be moderated by strict adherence to procedures set out, attention to temperature and the ability to quickly cool a reaction mixture down if necessary. Although they possess a great possibility for mishap, energetics are not the only area of chemistry that could result in a serious situation. Organic oxidations can get out of hand, procedures involving very high temperatures can cause mechanical explosions, and of course aside from the possibility of explosions, there is always the poison gas aspect. But what should you do? What should you do if you decide to dissolve matches in hydrochloric acid and suddenly gas above the beaker starts to explode or if your airtight reaction vessel suddenly implodes releasing hydrogen cyanide? Drastic times call for drastic measures and each situation is different, here are some things to consider, but everything is always up to the best judgment of the person involved in the situation, but the number one guiding rule should always be to stay calm.

There are four major schools of catastrophic failure; 1) Poisonous gases are rapidly being evolved and released into your atmosphere. 2) It is a liquid mixture with relatively little water containing an oxidizing agent and something that is being oxidized and is rapidly heating. 3) A closed vessel containing a reactant or system of reactants suddenly begins reacting much faster, evident by incredibly increased gas evolution or noises. 4) A product from your reaction is on fire, is spontaneously flammable, or is exploding as formed. And of these types there are those that can be solved by quenching with large amounts of water, and those that may react faster or cause other extreme circumstances.

If you suddenly find your reaction falling into one of these categories as stated above quenching with large amounts of water may help, in the event of poisonous gasses, dropping the whole reaction vessel into a bucket of water could bring a quick end to any mishap. In the authors own experience he has found it necessary to dig a hole and physically bury a reaction vessel that was producing a pyrophoric toxic substance and the reaction vessel was on fire belching out explosions. Risking your life is not something you want to do, if at all possible in any situation that threatens your life from which you see no immediate solution you should leave the area. But this is not always an option, consider those around you, if you were in the middle of a forest with no one around for quite a distance fleeing is always a good option, but for the majority of us backyard chemists we have neighbors and family close by, the luxury of fleeing from some mishaps is not an option. A small explosion, maybe you could leave that, a small fire, as long as it's not near anything, but anything that threatens a large explosion or release of poison gas is not some minor event. Call the police, call poison control, do what you can, it's easier to never get into this situation but if it comes down to it, it is your mess, and your responsibility to take care of. Consider those around you, you can risk your own life if you so choose, but never theirs. Please take care, keep a bucket of water handy, do what you have to, to make sure you keep yourself and those around you safe, and if things do go wrong in a big way, it is your duty to do whatever it takes to make sure no one is hurt, do whatever is in your power.

But then again, if there is no danger unless you are in the immediate vicinity, go ahead and run if you are endangering yourself. If you get that feeling that something is going to go wrong, you become uncomfortable with a situation and it affects your ability to work though it and your possibility of failure goes up. Just remember, safety first, and more important then glassware and expensive chemicals is human life.

10.0 Finding things locally

To some extent people have a chemical stockpile in their houses even if they are not practicing chemists. For example many households have acetic acid, sodium chloride, sodium bicarbonate, sodium hypochlorite solutions, and more. But that is not the extent of the chemicals available at your local hardware store or super market, or if you want to go even further specialty stores like hydroponics stores can be a uranium mine for the amateur chemist. When ever you visit these places just keep an eye on the shelves and if something catches your eye look over the label for information relating to the compounds contained within. This can give you the best idea of what you have available in your area.

The second best alternative is the internet. Not only are there sites dedicated to chemicals found at home, you can search compounds on google or other places and attempt to find them in some household use. Some are pure some are not and some are easily separated.

10.1 Pure compounds

10.2 Making Vs. Buying

The most logical way to determine if it would be better to buy a chemical rather then produce it yourself is to think about how much of a chemical you want, and how much it would cost to buy in the quantity that you want, now compare this with how much it would cost in terms of materials, danger, AND time to produce the same chemical. If it costs noticeably less to buy it then buying is a good option. But as said before there most of us neglect the time aspect, thinking that if we were to make it then any savings in terms of materials and money would be worth it, not true. The reaction might require refluxing for hours, hot filtrations, pressurized reactions, extensive drying, low yields, and decomposition of products. There are a number of reasons you might choose to buy a chemical rather then buy it. However if you have easy access to the beginning reagents it is usually cheaper to make a chemical yourself due to prohibitively high prices on lab grade chemicals.

An example being the manufacture of barium chloride, barium carbonate is readily available from ceramics stores for low prices, this can easily be reacted with hydrochloric acid to form the desired barium chloride and the mixture evaporated to dryness resulting in a relatively pure product. Whereas to buy barium chloride from an online source might cost 5x as much. This would definitely be an example of making a chemical versus buying it. On the other side of the coin one could think of making something like methyl carbonate, which may require you to first produce phosgene which was once used as a war gas, followed by reaction with alcohol which will produce hydrogen chloride, reaction would have to be under pressure, in terms of reagents, it might prove cheaper then to buy a specialty chemical, but in terms of time, safety, and glassware, opting to buy it would seem to be the smart choice.

There are some chemicals that are almost impossible to buy, or if they are available they charge exorbant amounts to ship them. There are also other externalities, you never want to attract more attention then you have to and if you happen to want a chemical that might have some major illicit uses and might be watched by your local governmental agencies it might be better to make it. What it all comes down to is, there are some things that are cheaper and easier to make, and there are some things that are much better bought. Chemists should not limit themselves to one or the other, making essential reagents can allow you to develop your lab skills better, but others are cheaper to buy then make. Each reagent should be evaluated on an individual basis, but remember that most assortments of chemicals found in a home lab were accumulated there though necessity and great sales along with boredom.

10.3 Extracting compounds

10.3a Basic Principles (Comparing Properties)

10.4 Mail Order

10.5 Notes about purity

11.0 Advanced Experiments (Name Reactions)

12.0 Index (Links throughout will be highlighted and click able to bring you to the specific index entry, e.g. H2SO4 will be highlighted and clicking on it will bring you to a page listing its properties, High temp oxidizing agent, dehydrating agent, different concentrations available.)

12.1 The Elements (See Section 1.3 for a depiction of the periodic table)

Actinium **Atomic Symbol: Ac** **Atomic Number: 89** **Atomic Weight: 227.0 g/mol** **Known oxidation state(s): +3**

Hazard information: Highly radioactive, most stable isotope has a half-life of 22 years.

Aluminum **Atomic Symbol: Al** **Atomic Number: 13** **Atomic Weight: 27.0 g/mol** **Known oxidation state(s): +3**

Hazard information: The presence of aluminum cations in soft drinks is a the suspect to some cases of Alzheimer's. Aluminum dust poses two hazards, it can provide an environment that could possibly lead to an explosive mixture with the air and secondly it can cause irritation to the respiratory system and disorientation. Always wear gloves and a dust mask when working with aluminum in the powder form. Bulk aluminum is safe.

Additional information on Aluminum: Aluminum as a bulk metal is widely used in the building industry. It is easily spotted in a scrap yard for a few reasons, it is relatively light, and forms an oxide coating which is easily scraped off with a knife to reveal the clean metal underneath. Carry a small bottle of vinegar with you if you are hunting for aluminum in a scrap yard to test samples, scrape the surface of the aluminum clean and apply a little of the acid, it will react with aluminum forming bubbles if it is the real deal. Aluminum turnings are also available at some scrap yards. Aluminum powder is available from pyrotechnic suppliers. There are also guides online for turning bulk aluminum to powder. Aluminum powder cannot be made by the decomposition of aluminum formate or oxylate as the finely divided aluminum can react readily with the carbon dioxide produced to form aluminum oxide as the majority product.

Aluminum powder, turnings, and foil.

Industrially aluminum is produced by the Hall process, electrolysis of aluminum oxide held in a molten cryolite [Na_3AlF_6] bath. On a home scale such a process would be demanding at best. On a side interesting note one of the first uses of sodium was as a reductant for producing aluminum from the oxide. This process has since been replaced by the Hall process noted above.

Aluminum is a highly reactive metal, it reacts readily with atmospheric oxygen and would simply rust to a pile if the oxide coating thus produced did not adhere so well. If for example a small amount of mercury is placed on a block of aluminum it continuously alloys with the aluminum rendering the oxide coating ineffective and will allow the oxygen in the air to rapidly oxidize large amounts of aluminum. Aluminum will react with nearly any acid and many bases readily (it will pacify [the surface will become coated in oxide and not react further] in strong concentrated oxidizing acids). Many aluminum salts are soluble and therefore are a good source of choice anions in solution.

Americium **Atomic Symbol: Am** **Atomic Number: 95** **Atomic Weight: 241.1 g/mol** **Known oxidation state(s): +3**

Hazard information: Radioactive element, treat with care.

Additional information on Americium: Americium oxide is the source of ionization energy in the vast majority of smoke detectors. It is a very small piece of this radioactive element.

Antimony **Atomic Symbol: Sb** **Atomic Number: 51** **Atomic Weight: 121.76 g/mol** **Known oxidation state(s): +3, +4, +5 (least common)**

Hazard information: Excessive handling of antimony metal should be avoided as many of the salts formed even those on contact with air could be hazardous. Antimony and its salts have been linked to reproductive damage and cancer.

Additional information on Antimony: Used in alloying, with lead in solder and in other applications, a hardening agent. Antimony is toxic and forms some interesting salts, the pentafluoride is a component of superacids but obtaining this metal in an over the counter way is difficult. Antimony sulfide is used in pyrotechnics. Somewhat of a weak metal antimony has a few interesting allotropes including the exploding antimony allotrope which has yet to be confirmed in recent years.

Argon **Atomic Symbol: Ar** **Atomic Number: 18** **Atomic Weight: 40.0 g/mol** **Known oxidation state(s): No common oxidation states**

Hazard information: Argon is an asphyxiant gas, use with ventilation. Argon directly exiting from cylinders may be cold enough to induce frost bite.

Additional information on Argon: (See section on inert atmospheres **8.4**)

Arsenic **Atomic Symbol: As** **Atomic Number: 33** **Atomic Weight: 74.9 g/mol** **Known oxidation state(s): +2, +3, +5 (least common)**

Hazard information: Excessive handling of arsenic metal should be avoided as many of the salts formed even those on contact with air could be hazardous. Arsenic and its salts have been linked to reproductive damage and cancer. Arsenic can show progressive physical and neurological damage, the progressive signs of arsenic poisoning are well covered. Arsenic trioxide was once known as "Inheritance powder".

Additional information on Arsenic: The only widely available compound containing arsenic is arsenic trioxide, I have seen it marketed for the purpose of killing a variety of insects, in ant traps and less commonly to kill mice. It's use has been phased out since the beginning of the 20th century though. It is also found in some specialty solders and in semiconductors. From its trioxide it could be reduced with an active metal such as magnesium to form the metal. Another available form of arsenic comes in the form of some herbicides and pesticides which contain arsenic organic molecules. Arsenic is a chemically reactive metal with interesting properties especially evident in the covalency of its high oxidation state compounds.

Astatine **Atomic Symbol: At** **Atomic Number: 85** **Atomic Weight: 210.0 g/mol** **Known oxidation state(s): NA**

Hazard information: Highly radioactive, most stable isotope has a half-life of 8 hours.

Barium **Atomic Symbol: Ba** **Atomic Number: 56** **Atomic Weight: 137.3 g/mol** **Known oxidation state(s): +2**

Hazard information: Barium salts are highly toxic, a small amount of a soluble barium salt that makes its way into your body will make you have a very bad day, diarrhea, blood in stool, headache, stomach pains, etc. The metal itself is highly reactive towards water along the lines of sodium and can cause minor explosions and presents a flammability hazard on its own. The free metal will burn the skin if it comes into contact with it. Should be stored under oil, most reactive of the common alkali earth metals.

Additional information on Barium: When exposed to air barium will from an appreciable percentage of the peroxide. Very few barium salts are available to the general public, the few that I know of are barium sulfate which is obtainable from medical clearances (it is used to make the intestines more visible with though xray, it is one of the very few safe barium salts), and barium ferrate, which is present in the coating on VHS tapes. In theory a large quantity of VHS tape could be ashed (heated till it turned to ash) then reduced with an active metal (aluminum or magnesium) then dissolved in water, the barium oxide thus formed would react with the water and convert to the somewhat soluble barium hydroxide which could be extracted by evaporation and crystallization.

Furthermore barium is available in both the hobby of pyrotechnics (carbonate, nitrate, perchlorate, sulfate) and pottery (carbonate) for colorization. These can be scrounged up from local sources or from online sources. Barium metal could be produced by aluminothermic reduction of the oxide or carbonate or hydroxide and subsequent distillation under high vacuum. Reaction of barium oxide and aluminum metal at high heat furnishes an alloy of high barium percentage >50% on cooling. Barium can also be procured though electrolysis of an eutectic mixture of barium salts in the molten state.

Berkelium **Atomic Symbol: Bk** **Atomic Number: 97** **Atomic Weight: 249 g/mol** **Known oxidation state(s): +3, +4**

Hazard information: Highly radioactive, half life sufficiently short to render amateur experimentation futile.

Beryllium **Atomic Symbol: Be** **Atomic Number: 4** **Atomic Weight: 9.0 g/mol** **Known oxidation state(s): +2**

Hazard information: Beryllium salts and beryllium metal dust are highly toxic and carcinogenic.

Additional information on Beryllium: Some aircraft parts, specifically gyroscopes are occasionally made of almost entirely beryllium, easily differentiated by their unearthly lightness. Machining beryllium is dangerous as shavings and powder can cause ‘metal fume fever’ and terrible pain. Beryllium is a reactive metal that forms an oxide coating that prevents further atmospheric attack. It is hard to find on the civilian market though except as the aforementioned use and in a very few copper alloys. Because of the beryllium ions small size and high charge density it forms unique cations when dissolved in water involving several water molecules.

Bismuth **Atomic Symbol: Bi** **Atomic Number: 83** **Atomic Weight: 209.0 g/mol** **Known oxidation state(s): +3, +5 (rare)**

Hazard information: Bismuth is fairly benign and safe to handle, the toxicity of bismuth salts is almost entirely dependent upon the anion to which it is coupled.

Additional information on Bismuth: Bismuth is available as environmentally friendly buck shot for re-loading guns in areas where guns are permitted, but by this route it is fairly expensive. Also it can be found in some areas that sell minerals and collectable rocks, bismuth forms beautiful crystals when solidified from a properly formed melt and are sold as a pure material, again, the price can be exhorbant. The internet is always another choice for bismuth metal if all else fails.

Bismuth trioxide has found use in pyrotechnics and this could be reduced with an appropriate aluminothermic reduction. Also bismuth subsilicate is available as an over the counter stomach soothing remedy, it may be possible, although economically disastrous to extract this small quantity of bismuth. Some bismuth salts, especially those where bismuth is in the +3 state and attached to three different molecules are prone to decomposition in water due to the formation of the stable oxy compound. For example, a solution of bismuth trichloride left to stand may decompose in the following manner:

$$BiCl_{3(aq)} + H_2O_{(l)} \Rightarrow OBiCl_{(s)} + 2HCl_{(aq)}$$

Many compounds will do the same hydrolysis reaction if left in solution too long, bismuth nitrate may form bismuth subnitrate, bismuth chloride may precipitate as bismuth oxychloride and there are many more. Dissolving bismuth is a difficult chore although it comes ahead of hydrogen in the activity series and should theoretically dissolve in acid it does so sluggishly at best, it is necessary to add an oxidizing agent to get a decent rate of solvation of the native metal. And as I just mentioned it is necessary to recover your bismuth salt quickly lest it hydrolyze, the hydroxide is a good choice as it will allow conversion to other appropriate salts at a later date. The bismuthate anion BiO_3^- in which bismuth has a +5 charge is an excellent oxidizing agent prepared by the reaction of dry bismuth trioxide with sodium peroxide or by the action of molten $NaNO_3$/NaOH on bismuth trioxide, it will oxidize manganate to permanganate.

Boron **Atomic Symbol: B** **Atomic Number: 5** **Atomic Weight: 10.9 g/mol** **Known oxidation state(s): +3**

Hazard information: Elemental boron is toxic, dust should be avoided, boron compounds differ widely in their toxicity, for example, the chloride is a strong irritant/corrosive liquid, whereas the acid is the only acid that is actually good for the eyes.

Additional information on Boron: Boron has two widely available salts, borates/metaborates are available to some extant as borax in the cleaning industry. Borax as found in cleaning products usually has the formula $Na_2B_4O_7*5H_2O$ solutions of borax can be treated with a strong acid such as HCl to precipitate out boric acid. Boric acid can also be bought as a somewhat pure substance from pharmacies and also from grocery stores for the purpose of pest control. From boric acid heat can be applied to dehydrate it to boric oxide. And from the oxide elemental boron can be had.

$$Na_2B_4O_{7(aq)} + 2HCl_{(aq)} + 5H_2O_{(l)} \Rightarrow 2NaCl_{(aq)} + 4B(OH)_{3(s)}$$

$$2B(OH)_{3(s)} \text{ --(Heat)--> } B_2O_{3(s)} + 3H_2O_{(g)}$$

$$B_2O_{3(s)} + 3Mg_{(s)} \text{ --(Heat)--> } 2B_{(s)} + MgO_{(s)} + x[MgB_2]_{(s)}$$

$$B(s)/MgO(s)/MgB_2(s) \text{ --(}HCl_{(aq)}\text{)--> } B_{(s)} + MgCl_{2(aq)} + B_2H_{6(g)}$$

In the above reactions we start from the commonly available borax with a precipitation reaction to get to our boric acid. Of if you have boric acid start from step 2. From here the acid is dehydrated and easily goes to boric oxide. The oxide is then pulverized with a hammer or other suitable object and mixed with either magnesium powder or turnings in a stoichiometric amount. The mix is ignited and a thermite reaction ensues, this generates lots of heat but the reaction must be covered loosely immediately to prevent the oxidation of the boron thus formed, at the same time a small amount of magnesium boride is formed as a side reaction. Finally after the reaction cake has cooled and is powdered, it is digested in hydrochloric acid, the magnesium oxide being a basic oxide is readily dissolved in the HCl and the magnesium boride reacts with the HCl to produce diborane. The diborane is a spontaneously flammable gas and therefore small explosions may result, it is therefore advisable to cover the cake first with water then add acid in small amounts to prevent excessive sudden gas evolution. The magnesium chloride stays in solution, the boride is decomposed and what you are left with is boron as a precipitate at the bottom of the reaction vessel.

Product from aluminum reacting with B2O3, how are you going to separate that?

The first question that comes to many peoples mind when they see this thermite type reaction is weather they can substitute aluminum for the magnesium as aluminum powder is more readily made/acquired. Yes, it could be substituted in theory, but there is one drawback, see step 3 where the magnesium boride is formed as a side reaction. It could be assumed, and in this case correctly that aluminum boride [AlB_{12}] would be formed analogously in this reaction. But the problem comes in reaction 4, aluminum boride is very inert, it will not react with the HCl and therefore you end up with very impure boron as you are unable to separate the aluminum boride (in addition the aluminum oxide is very hard to dissolve out). So what you are left with is a neigh insoluble mass of boron, aluminum oxide, and aluminum boride from which the boron is very difficult to remove. One possible removal method would be to run chlorine gas over the heated mass to produce boron trichlroide and run that over heated zinc powder to facilitate the reaction along the lines of :

$$B_{(s)} + Cl_{2(g)} \Rightarrow BCl_{3(g)}$$

$$2BCl_{3(g)} + 3Zn_{(s)} \Rightarrow 2B_{(s)} + 3ZnCl_{2(s)}$$

Although this method facilitates boron powder it makes the reaction considerably more difficult in the manipulation of chlorine gas and boron trichloride. However one could make boron trichloride directly from boric oxide and sodium chloride and run that over the zinc therefore skipping the active metal reduction with magnesium and replacing it with this zinc step.

Boron will form an additional bond at its lone pair making it a negative cation, an excellent example of this is sodium borohydride [$NaBH_4$] in which the boron atom has a negative one charge due to the extra bond to hydrogen.

Boron is an elemental color emitter, its combustion produces a beautiful green color and its esters produce the same.

Bromine **Atomic Symbol: Br** **Atomic Number: 35** **Atomic Weight: 79.9 g/mol** **Known oxidation state(s): -1, +3, +5, +7 (rare)**

Hazard information: Bromine is a highly corrosive red liquid. It will attack rubber, your lungs (causing pulmonary edema), your eyes (causing blindness), and your skin (causing painful ulcerations). Skin exposure should be treated with a reducing agent such as sodium thiosulfate which will help to destroy the bromine before it destroys any more of you. Although it is not highly toxic it does have sedative effects that can result in death due to depression of the central nervous system.

Additional information on Bromine: (See section 4.9 for further information) Bromine is a diatomic molecule and normally appears at Br_2 in formulas, in the gaseous state it maintains these bromine-bromine bonds. Free bromine is found as the diatomic molecule Br_2 that is whenever bromine is free it is always coupled with another bromine molecule. Your best bet to finding commercially available bromine sources is going to be from pool/spa suppliers. Bromination sources include sodium bromide but more often you may find a complex organic compound that actually acts as the brominating agent. If possible the sodium bromide provides the much easier compound from which to extract bromine although the organic compound could yield a combination of bromine and bromine chloride (although this decomposes above 10C, the chlorine gas that makes its way though and comes into contact with your condensed bromine in a receiving flask could react with the bromine there). That is if it is sufficiently gassed with chlorine in powder form at a temperature sufficient to distill off the bromine [>59C].

As for bromine production from sodium bromide. 1) Running chlorine gas though a solution of warm sodium bromide will cause the chlorine to replace the bromine in the compound resulting in free bromine. This reaction really is complicated by working with chlorine gas. 2) Reacting aqueous sodium bromide with an oxidizing agent under acidic conditions can result in the formation of free bromine which can be distilled off:

$$2NaBr_{(aq)} + H_2SO_{4(l)} + H_2O_{2(aq)} \Rightarrow Na_2SO_{4(aq)} + 2H_2O_{(l)} + Br_{2(l)}$$

In the above reaction it is the hydrogen peroxide that acts as the oxidizing agent, other oxidizing substances; potassium permanagnate, potassium bromate; etc. could be used in its place. Additionally different acids could be substituted, hydrochloric acid could be substituted but there is the possibility that it could be oxidized resulting in free chlorine contaminating the reaction. An additional benefit to the addition of concentrated H_2SO_4 is the heat of hydration which allows the mixture to obtain a temperature to distill off the Br_2 formed without the need for significant, if any, additional heating.

Cadmium **Atomic Symbol: Cd** **Atomic Number: 48**
Atomic Weight: 112.4 g/mol **Known oxidation state(s): +2**

Hazard information: Highly toxic, carcinogenic, poisoning from cadmium compounds is rare though due to their ability to induce vomiting rapidly.

Additional information on Cadmium: Cadmium serves very few purposes in the life of the general populous. One of the only sources of any form of cadmium, aside from meager alloys and coatings, is found inside of household rechargeable batteries. This is in the form of a cadmium oxide electrode. Another source of cadmium is in the form of pigments, cadmium sulfide (yellow-brown) and selenide (red) being the main ones. Cadmium sulfide could be dissolved in dilute HCl and the mixture heated to reflux, hydrogen sulfide would be evolved though which is highly toxic. The resulting $CdCl_2$ could be re-dissolved in neutral water and the solution electrolyzed to yield the metal. Cadmium is resistant to alkalis but readily attacked by acids.

Calcium **Atomic Symbol: Ca** **Atomic Number: 20**
Atomic Weight: 40.1 g/mol **Known oxidation state(s): +2**

Hazard information: Flammable as a bulk solid, spontaneously flammable in powder/fine turnings. Calcium is non-toxic but it can cause skin damage if handled without gloves from the basicity of the hydrolyzed metal and the dehydrating action on the skin. Reacts readily with water forming hydrogen gas, which can ignite and explode.

Additional information on Calcium: Calcium is produced most often by the electrolysis of straight molten CaCl2, in this process the cathode must either be barely touching the surface of the melt and slowly raised up or, constantly rotated to provide a cohesive non-porous mass of calcium metal. The addition of up to 15% KCl can depress the melting point of the mixture without noticeable potassium formation at the cathode but at percentages beyond this potassium formation becomes evident. Additionally mixing calcium chloride with chlorides of other alkali earth metals can form eutectics which may prove useful, but despite finding patents on such mixtures, they have found no use in industry. During electrolysis of the molten chloride there is a very small range over which electrolysis can progress successfully, between 780 and 800 C, during this small frame calcium produced will be a solid and the melt will be molten, lower then this and the melt solidifies, higher and the already highly reactive calcium will be molten and almost guaranteed to catch fire. Remember, chlorine gas would be produced at the anode to complicate matters even further.

Chemical reduction of calcium oxide is another route to calcium metal production. When calcium iodide and sodium metal are heated together in a metal vessel at high heat and the mixture allowed to cool, calcium metal crystallizes out. Aluminothermic reduction of calcium oxide with aluminum metal over high heat under high vacuum has been used to isolate calcium metal, however it does not work as well as similar reductions of other heavier alkali earths.

Calcium itself is a great reducing agent due to the low volatility of its oxide and chloride. Heating cesium/rubidium/potassium chlorides with calcium metal under high vacuum will distill over the free metals. Calcium carbide, a somewhat available chemical can also act as a potent reducing agent.

Californium **Atomic Symbol: Cf** **Atomic Number: 94**
Atomic Weight: 251.1 g/mol **Known oxidation state(s): +3, +4**

Hazard information: Highly radioactive element. However the half-life is long enough to work with the element in macroscopic quantities. Cf^{252} is the most widely available isotope and is for sale in milligram quantities. The isotope with the longest half-life is Cf^{251} with a half-life of nearly 900 years.

Carbon **Atomic Symbol: C** **Atomic Number: 6**
Atomic Weight: 12.01 g/mol **Known oxidation state(s): -4, +4 (Carbon can form hybrid orbitals resulting in unique states)**

Hazard information: Small particles of carbon in the form of diamond can prove to be an inhalation/ingestion hazard, in addition finely divided carbon in any of its many forms, such as carbon black, charcoal, coal, etc. can prove detrimental to the lungs of an individual.

Additional Information on Carbon: It is beyond the scope of this work to attempt to describe the entirety of organic chemistry, which would be necessary to somewhat describe the many reactions of carbon containing compounds. Therefore focusing strictly on the elemental, it is available in many different allotropes, the familiar diamond, amorphous such as coal, the spheroid bucky balls (C_{60}), nanotubes, graphite, and a few other minor modifications. All of these forms except bucky balls, are fairly inert to many chemical actions except at high temperatures, at which point they become excellent reducing agents. Finely divided carbon can reduce many oxides to their free elemental state at high temperature. Reaction of carbon at high temperatures with metals can also result in the formation of carbides, whose reaction with water yields the metal hydroxide and acetylene gas, with the exception of beryllium carbide and aluminum carbide, which form true carbides and react with water to form the metal hydroxide and methane gas. Activated charcoal and other high surface area carbon forms are excellent catalyst for a number of operations in chemistry, carbon is oxidized in the presence of oxygen to carbon dioxide and in a deficient oxygen system to carbon monoxide, it can also be oxidized by elemental sulfur at high temperature to carbon disulfide.

Activated Charcoal, Graphite Rods, and Carbon Powder

Usually the preparation of carbon is unnecessary, however amorphous carbon can be made by the reaction between concentrated sulfuric acid and sugar. Additionally it can be made the old-fashioned way by heating wood without access to oxygen in a container to high temperatures and holding it there, both forms contain impurities. Graphite, the only common conductive from of carbon can be salvaged as electrodes in larger batteries, diamonds find little use in chemistry. However bucky balls are starting to be more widely produced, gram quantities are currently available. Bucky balls will actually dissolve in organic solvents yielding brightly colored solutions depending on the exact bucky composition used, (several different spherical structures of carbon have now been made available).

Cerium **Atomic Symbol: Ce** **Atomic Number: 58**
Atomic Weight: 140.1 g/mol **Known oxidation state(s): +3, +4**

Hazard information: Metal is fairly reactive, reducing water and burning readily in the air when pure. Cerium salts are not particularly toxic enough to warrant additional caution during normal use.

Additional information on Cerium: The decomposition of cerium oxalate by heat results in the formation of cerium dioxide, which is available for polishing lenses, especially those to do with telescopes. Cerium compounds are also used within self cleaning ovens and cerium itself makes up a notable percentage of mish metal, which contains many other rare earths, mish metal is used in lighter flints and other places that need an easy source of sparks. Cerium-iron alloys can be prepared in such a way as to make them pyrophoric, they find use in ignition devices. The +4 oxidation state of cerium is not strongly oxidizing despite cerium being one of the few lanthanides with a +4 state. Cerium metal can be made by the electrolysis of molten cerous chloride (mp 848 °C).

Cesium **Atomic Symbol: Cs** **Atomic Number: 55**
Atomic Weight: 132.9 g/mol **Known oxidation state(s): +1**

Hazard information: Cesium metal is incredibly reactive and can explode from prolonged contact with the atmosphere and will explode in contact with water, reaction with ice rapidly even below –100 °C. It is the most reactive of the alkali metals and will easily liquefy slightly above room temperature (28 °C). Cesium salts are moderately toxic.

Additional information on Cesium: Cesium metal can be made by chemical methods such as distillation from a mixture of cesium chloride and calcium metal under a vacuum, or by electrolytic methods with the chloride, bromide, or iodide. Supposedly cesium when viewed in person has a slight gold color to it. Cesium hydroxide is the most powerful of the alkali metal hydroxides, it will readily attack glass. Sources of cesium and its salts over the counter are rare to find. Atomic clocks use a small amount of cesium metal and these ampoules can occasionally be purchased, in biology cesium chloride is used to add to centrifuge tubes and create a density gradient to separate specific components of a mixture. In all of its sources cesium is expensive, it does not find any common use in home chemistry.

Chlorine **Atomic Symbol: Cl** **Atomic Number: 17**
Atomic Weight: 35.5 g/mol **Known oxidation state(s): -1, +1, +3 (rare), +4, +5, +7**

Hazard information: Chlorine gas is toxic, on inhalation it damages the lungs and if the damage is severe enough the throat may close suffocating the individual or the lungs may fill with fluid drowning the individual. Chlorine also attacks the eyes and skin. Chlorides do not possess any noticeable toxicity.

Additional information on Chlorine (See section 4.9 for further information): Chlorine is a diatomic gas and therefore appears in formulas as Cl_2, 22.4L of chlorine gas at STP is actually two mols of chlorine because of this association between molecules. Chlorine forms a series of oxoacids: Hypochlorous acid (HOCl), Chlorous acid (HOClO), Chloric acid (HOClO2), and Perchloric acid (HOClO3). Of these acids perchloric is the most stable and chlorous acid is the most unstable although salts of it can be isolated. Chlorine has a large area of use in the laboratory, although alternatives to the use of free chlorine should always be investigated due to the danger of working with it. Chlorine is a useful oxidizing agent and is one of the simplest ways to make anhydrous metal chlorides (AlCl3, ZnCl2, etc.). Chlorine can be made in many ways, electrolysis of concentrated chloride solutions, electrolysis of molten metal chlorides, acidification of hypochlorites, and other ways as well.

Chromium **Atomic Symbol: Cr** **Atomic Number: 24**
Atomic Weight: 52.0 g/mol **Known oxidation state(s): +2, +3, +6**

Hazard information: Salts of chromium in the lower oxidation states and the free metal are not noteably hazardous, however chromium in the +6 oxidation state, commonly dichromate, is a carcinogenic form of chromium, +6 chromium compounds should be reacted with a reducing agent prior to disposal.

Additional information on Chromium: Finding sources of pure chromium over the counter is a difficult endeavor, alloys of chromium and nickel find use in resistance heating elements and chromium metal is the main constituent (~98%) of the chrome that covers some of the shinier parts of cars. A few chromium compounds are available over the counter, mostly in the form of pigments and glazes for pottery. Chromate and dichromate are useful oxidizing reagents, dichromate can be made from elemental chromium by heating the solid chromium with potassium hydroxide and potassium nitrate while molten over high heat, the procedure is somewhat dangerous.

Cobalt **Atomic Symbol: Co** **Atomic Number: 27**
Atomic Weight: 58.9 g/mol **Known oxidation state(s): +2, +3**

Hazard information: Soluble cobalt compounds are toxic, the free metal is not notably so unless ingested.

Additional information on Cobalt: Originally found in nickel ores cobalt was considered a nuisance to nickel production. Cobalt compounds find limited use in home chemistry, they find use industrially in dyes, inks, and catalysts. The oxide CoO can be found with some searching as a pigment and component in ceramics, this could be reduced to the element by a simple thermite type reaction or a soluble cobalt salt can be electrolyzed, plating out the desired cobalt on the cathode.

Copper **Atomic Symbol: Cu** **Atomic Number: 29**
Atomic Weight: 63.5 g/mol **Known oxidation state(s): +1, +2**

Hazard information: Soluble copper salts are toxic.

Additional information on Copper: Copper (II) salts are mild oxidizing agents and most copper (I) salts are significantly less soluble then their comparative copper (II) salt. Copper is widely available, in wires, currency, electronics, etc. It can be found native in some areas of the world but for the most part it is extracted from ore. Copper is somewhat inert, reacting very slowly with hydrochloric or room temperature sulfuric but readily with nitric acid. Boiling copper with sulfuric acid is one good way to produce sulfur dioxide. Copper (II) sulfate is avalible widely for killing tree roots that end up in sewer lines and this can be the jumping point for making other copper salts or for electrolytic production of copper, sufficiently heated copper sulfate will yield sulfur trioxide which could then be solvated (with difficulty) to form sulfuric acid, leaving behind CuO.

Curium **Atomic Symbol: Cm** **Atomic Number: 96**
Atomic Weight: 247.1 g/mol **Known oxidation state(s): +3, +4**

Hazard information: Curium is a radioactive bone-seeking element. It is available in gram quantities but is quite expensive and outside the price range of the average at home chemist.

Dysprosium **Atomic Symbol: Dy** **Atomic Number: 66**
Atomic Weight: 162.5 g/mol **Known oxidation state(s): +3**

Hazard information: Spontaneously flammable in powder form, reacts slowly with water and halogens.

Additional information on Dysprosium: Formed by the reduction of its fluoride with calcium metal, dysprosium is a rare element with which you have little probability to run across. It finds limited use to alter the optic properties of mirrors and glass.

Einsteinium **Atomic Symbol: Es** **Atomic Number: 99**
Atomic Weight: 253 g/mol **Known oxidation state(s): +2**

Hazard information: Rare and radioactive, a man-made element. Found in the green glass left over after an atomic explosion and named after the ever-famous Albert Einstein.

Erbium **Atomic Symbol: Er** **Atomic Number: 68**
Atomic Weight: 167.3 g/mol **Known oxidation state(s): +3**

Hazard information: Flammable in powder form but otherwise less reactive then it's rare earth cousins, salts are toxic.

Europium **Atomic Symbol: Eu** **Atomic Number: 63**
Atomic Weight: 152.0 g/mol **Known oxidation state(s): +2, +3**

Hazard information: Highly reactive, spontaneously flammable and reactive with water.

Additional information on Europium: Used in the pigments on the inside of televisions and as a neutron absorber in nuclear reactors. One of the more useful rare earths but still quite uncommon.

Fermium **Atomic Symbol: Fm** **Atomic Number: 100**
Atomic Weight: 254 g/mol **Known oxidation state(s): +3**

Hazard information: Highly radioactive, most stable isotope only has a half-life of 3 hours.

Fluorine **Atomic Symbol: F** **Atomic Number: 9**
Atomic Weight: 19.0 g/mol **Known oxidation state(s): -1**

Hazard information: Incredibly reactive with nearly anything it will come across, asbestos will glow in a stream of fluorine, water can catch on fire, and soluble fluorides are fairly toxic, hydrofluoric acid is insanely powerful with respect to it's ability to decimate the human body.

Additional information on Fluorine (See section 4.9 for further information):
Fluorine is a diatomic molecule so usually it will appear in a formula as F_2, therefore at STP 22.4 L of fluorine gas is really 2 mol of F instead of one. Chemical production of fluorine was only recently achieved, the first methods to produce fluorine were electrolyitcally from a mixture of anhydrous hydrofluoric acid and potassium fluoride. Fluorine and oxygen both have a tendency to bring out the highest oxidation state of elements with which they combine. If it were safer fluorine would be the friend of the armature chemist, but this is truly a case where the element is so dangerous as to preclude it from most any effort of use. Fluorides are interesting in that they behave differently then most of the other halogens, for example, silver fluoride is soluble in water, whereas the other silver halides are all incredibly insoluble, by contrast calcium fluoride is very insoluble but the other calcium halides show good solubility. Solutions of fluorides are basic by the equilibrium existing:

$$F^-_{(aq)} + H2O_{(l)} \Leftrightarrow HF_{(aq)} + OH^-_{(aq)}$$

The equilibrium shifting to the left due to the weakness of hydrofluoric acid. Weak solutions of hydrogen fluoride are available over the counter for cleaning car rims, and ammonium hydrogen fluoride finds limited use in the arts and crafts area for etching glass. Calcium fluoride is a widely available mineral, its reaction with sulfuric acid being the basis for the production of hydrofluoric acid.

Francium **Atomic Symbol: Fr** **Atomic Number: 87**
Atomic Weight: 223.0 g/mol **Known oxidation state(s): +1**

Hazard information: Highly radioactive, most stable isotope only has a half-life of 3 hours. Less then 25 g on Earth at any given time.

Gadolinium **Atomic Symbol: Gd** **Atomic Number: 64**
Atomic Weight: 157.3 g/mol **Known oxidation state(s): +3**

Hazard information: Salts are toxic, free metal reacts slowly with water forming hydrogen, do not mix gadolinium powder with oxidizing agents.

Gallium **Atomic Symbol: Ga** **Atomic Number: 31**
Atomic Weight: 70.0 g/mol **Known oxidation state(s): +2, +3**

Hazard information: NA

Additional information on Gallium: The free metal serves no commercial purpose other then a means to an end, to make semi-conducting materials such as gallium arside. Most gallium found online for sale and other sources is of a very high purity due to it being used so exclusively in the semiconductor industry. As such it can be somewhat expensive. Gallium melts slightly above room temperature (29 °C) but is reactive so unlike mercury it will become sticky and form a film of oxide on it, gallium metal expands as it freezes like water, gallium has an incredibly long liquid range so it finds some limited use in high temperature thermometers. Gallium and its compounds find no use other then curiosity in the home lab.

Germanium **Atomic Symbol: Ge** **Atomic Number: 32**
Atomic Weight: 72.9 g/mol **Known oxidation state(s): +2, +4**

Hazard information: Germanium salts are slightly toxic.

Additional information on Germanium: Used almost exclusively in the electronics industry, germanium will not react with water but dissolves readily in acids. Germanium itself is a semi conducting material.

Gold **Atomic Symbol: Au** **Atomic Number: 79**
Atomic Weight: 197.0 g/mol **Known oxidation state(s): +1, +3 (auric)**

Hazard Information: Gold salts are toxic but not incredibly so.

Additional information on Gold: Gold is relatively inert, it will not oxidize notably on exposure to moist air and to dissolve it requires aqua regia or other somewhat harsh techniques. Were it not for price it would make a decent electrode in many applications. Gold has a modest melting point slightly over 1000 °C but can be readily fabricated due to its malleability, it can be pounded into sheets so thin that light is visible through them. Most gold salts in solution are unstable with respect to reduction. If for example a solution of gold chloride is allowed to stand in sunlight the gold will precipitate from solution as fine particles. As for the chemical applications of gold for the at home chemist its main role would be probably in apparatus manufacture if it were not for the price. As such it finds little use in the home lab aside from curioso mixture with which to deposit a layer of gold onto glassware for aesthetic purposes.

Hafnium **Atomic Symbol: Hf** **Atomic Number: 72**
Atomic Weight: 178.5 g/mol **Known oxidation state(s): +2, +3, +4, +6**

Hazard information: Hafnium and its compounds are all fairly toxic.

Additional information on Hafnium: Resistant to oxidation and corrosion in general it also finds use in electrodes and is most known for its role as a control rod material in the nuclear industry. Of importance relating to its role in the nuclear industry is the difficulty in separating hafnium from zirconium (which possesses the opposite properties then those that make hafnium desirable). Hafnium is a dense metal with a melting point over 2000 °C, however it is fairly reactive and the free metal as a powder can spontaneously ignite and possibly explode from exposure to the atmosphere.

Helium **Atomic Symbol: He** **Atomic Number: 2**
Atomic Weight: 4.0 g/mol **Known oxidation state(s): No common oxidation states**

Hazard information on Helium: Helium is an asphyxiant gas, use with adequate ventilation.

Additional information on Helium: Up until the 1940's helium was a very expensive commodity, being that it is a noble gas it has no compounds from which it could be won and due to its low molecular weight there is very little in the atmosphere. The price however dropped to less then 3% of its previous price after it was discovered helium could be obtained in high quantities from the gasses escaping certain oil deposits, the source, the natural decay of radio active nuclei in the surrounding bedrock, since then different cavernous areas and such have been tapped and the exit gasses condensed to obtain this useful gas. Helium is quite unreacitve although in the plasma state certain 'compounds' have been identified, particularly with hydrogen. Liquid helium also shows some very interesting properties, even when cooled to 0 K helium remains a liquid, and it must be put under pressure to solidify, another very interesting thing to note here is that the melting of solid helium is exothermic. There are a number of other incredible thermodynamic properties of helium at this state such as the γ transition, which marks its change from a 'superfluid' having zero viscosity to a normal fluid. Helium being so light is nowhere near an ideal choice for an inert atmosphere but it is discussed in section 8.4 on inert atmospheres.

Holmium **Atomic Symbol: Ho** **Atomic Number: 67**
Atomic Weight: 164.3 g/mol **Known oxidation state(s): +3**

Hazard Information: Radioactive element, compounds are toxic.

Additional information on Holmium: Reacts slowly with water this metal is set apart slightly from the other rare earth metals due to some of its magnetic and electrical properties.

Hydrogen **Atomic Symbol: H** **Atomic Number: 1**
Atomic Weight: 1.0 g/mol **Known oxidation state(s): -1, +1**

Hazard Information: Highly flammable asphyxiant gas.

Additional information on Hydrogen: Hydrogen is a colorless diatomic gas so it normally appears in equations H_2, which means that at STP a mol of hydrogen ~22.4 L is actually 2 mol of H. The normal oxidation state of hydrogen is +1, some examples of which include HCl, CH_4, H_2O, and others, hydrogen exhibits the –1 state in metallic hydrides for the most part such as NaH and $LiAlH_4$ (lithium aluminum hydride), metallic hydrides are strong reducing agents and are often more reactive then the free metals. Hydrogen is incredibly common in the universe and is extremely easy to make either by electrolysis or chemical methods (see section 4.13 gasses). There are a number of uses for hydrogen in the amateur laboratory, for the preparation of strong reducing agents (e.g., the preparation of a sodium hydride dispersion by melting sodium under mineral oil with magnetic stirring and bubbling hydrogen through it) or as a direct reducing agent, it can also be a reactant for the final product such as making HBr from H_2 and Br_2. Some extra precautions should be taken with hydrogen due to its high degree of flammablility and if ignition is the key somewhere in an apparatus oxygen must be precluded from the areas you do not want to explode otherwise the fire will flash back through the vessel.

Hydrogenation reactions, where hydrogen adds to a molecule, usually across a double bond, involve the reaction of hydrogen under pressure (this is a necessity) with your molecule in the presence of a catalyst (usually transition metal such as a platinum or palladium compound). When dealing with these pressure reactions there is an inherent danger and hence some of the contraptions in which they are performed are called ‘bombs’.

Indium **Atomic Symbol: In** **Atomic Number: 49**
Atomic Weight: 114.8 g/mol **Known oxidation state(s): +1, +3**

Hazard information: Indium powder and compounds of indium are toxic.

Additional information on indium: Indium is a shiny silvery metal, reactive enough to dissolve in most acids, indium is used as an alloying agent in a number of applications. It also is the ingredient in a number of low melting alloys/eutectics. Indium is a fairly expensive compound, and there are no commonly available indium compounds on the market.

Iodine **Atomic Symbol: I** **Atomic Number: 53** **Atomic Weight: 126.9 g/mol** **Known oxidation state(s): -1, +1, +3, +5, +7**

Hazard information: Iodine is a skin irritant and if consumed can be fatal, the fatal dose being about two grams, iodine anion is an essential component of the human body.

Additional information on iodine (See section 4.9 for further information): Like the other halogens iodine is diatomic and as such appears in formulas as I_2. Iodine is a readily sublimed solid, purplish in appearance, its color more apparent when dissolved in non-polar solvents or when vaporized. The reactivity of iodine follows the trend established and as such it is less reactive then bromine. It can be readily won from compounds using an oxidizing agent as simple as acidic H_2O_2 or NaOCl, however in the latter case it can dissolve again due to the basicity of the environment. Iodine forms a series of oxoacids analogous to chlorine, the periodate showing evidence of polymerization in solution.

Iridium **Atomic Symbol: Ir** **Atomic Number: 77** **Atomic Weight: 192.2 g/mol** **Known oxidation state(s): +1, +2, +3, +4, +6**

Iron **Atomic Symbol: Fe** **Atomic Number: 26** **Atomic Weight: 55.9 g/mol** **Known oxidation state(s): +2, +3, +4 (rare), +5 (unstable), +6 (rare), +7 (rare)**

Krypton **Atomic Symbol: Kr** **Atomic Number: 36** **Atomic Weight: 83.8 g/mol** **Known oxidation state(s): +2 (rare)**

Lanthanum **Atomic Symbol: La** **Atomic Number: 57** **Atomic Weight: 138.9 g/mol** **Known oxidation state(s): +3**

Lawrencium **Atomic Symbol: Lr** **Atomic Number: 103** **Atomic Weight: 262.1 g/mol** **Known oxidation state(s): NA**

Hazard information: Would be highly radioactive as the most abundant isotope only has a half-life of 8 seconds.

Lead **Atomic Symbol: Pb** **Atomic Number: 82** **Atomic Weight: 207.2 g/mol** **Known oxidation state(s): +2, +4**

Lithium **Atomic Symbol: Li** **Atomic Number: 3**
Atomic Weight: 6.94 g/mol **Known oxidation state(s): +1**

Lutetium **Atomic Symbol: Lu** **Atomic Number: 71**
Atomic Weight: 175.0 g/mol **Known oxidation state(s): +3**

Magnesium **Atomic Symbol: Mg** **Atomic Number: 12**
Atomic Weight: 24.3 g/mol **Known oxidation state(s): +2**

Manganese **Atomic Symbol: Mn** **Atomic Number: 25**
Atomic Weight: 54.9 g/mol **Known oxidation state(s): +2,**
+3, +4, +6, +7

Mendelevium **Atomic Symbol: Md** **Atomic Number: 101** **Atomic Weight: 258.1 g/mol** **Known oxidation state(s): +2, +3**

Hazard information: Longest half-life of a mendelevium isotope is just shy of two months. This highly radioactive element is not something you will likely run across.

Mercury **Atomic Symbol: Hg** **Atomic Number: 80** **Atomic Weight: 200.6 g/mol** **Known oxidation state(s): +1 (diatomic), +2**

Molybdenum **Atomic Symbol: Mo** **Atomic Number: 42** **Atomic Weight: 95.5 g/mol** **Known oxidation state(s): +2, +3**

Neodymium **Atomic Symbol: Nd** **Atomic Number: 60** **Atomic Weight: 144.2 g/mol** **Known oxidation state(s): +3**

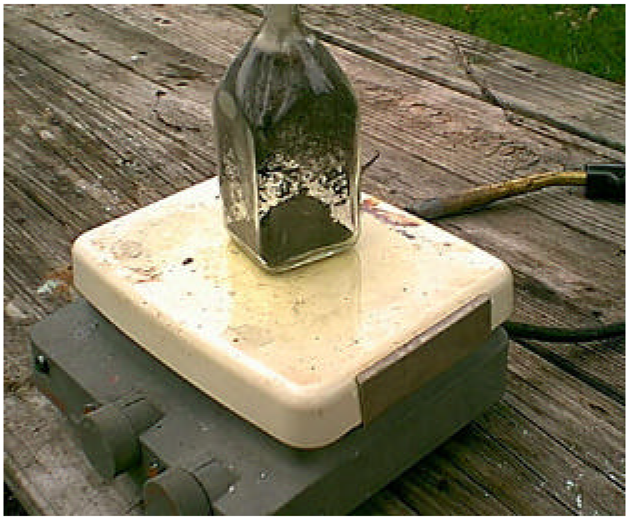
Neon **Atomic Symbol: Ne** **Atomic Number: 10** **Atomic Weight: 20.2 g/mol** **Known oxidation state(s): No Common Oxidation States**

Neptunium **Atomic Symbol: Np** **Atomic Number: 93** **Atomic Weight: 237.1 g/mol** **Known oxidation state(s): +5**

Nickel **Atomic Symbol: Ni** **Atomic Number: 28** **Atomic Weight: 58.7 g/mol** **Known oxidation state(s): +2, +3**

Hazard information: Many nickel salts have been shown to have carcinogenic properties, care should be exercised with them due to these concerns.

Additional information on nickel: Known to some as "Poor Man's Platinum" nickel finds much use in the amateur laboratory. In reference to the aforementioned saying, nickel is useful for the catalysis of a number of reactions in which platinum is traditionally used; it is an excellent hydrogenation catalyst. In addition to this nickel also has favorable physical properties including a high melting point and resistance to oxidation. Also nickel has a premium resistance to bases and good resistance to non-oxidizing acids.



Nickel powder can be formed in a number of ways, most notably by the reduction of a soluble nickel salt in an aqueous medium by zinc powder or citric acid. Additionally it can be formed as is shown at left by the decomposition of nickel oxalate formed by the displacement reaction between sodium oxalate and a soluble nickel salt.

Due to the favorable chemical resistance a number of lab items are available coated in nickel such as the ever-present nickel spatula. Nickel dishes and crucibles are also somewhat common. Nickel also is a major component of the alloys used for handling the halogens including fluorine. The most common oxidation state of nickel is +2 and in solution nickel cations usually appear green. Higher oxidations then +2 are possible, +4 has been documented and higher oxidations have been rumored.

Niobium **Atomic Symbol: Nb** **Atomic Number: 41**
Atomic Weight: 92.9 g/mol **Known oxidation state(s): +2, +3, +4, +5**

Nitrogen **Atomic Symbol: N** **Atomic Number: 7**
Atomic Weight: 14.0 g/mol **Known oxidation state(s): -3, +5**

Nobelium **Atomic Symbol: No** **Atomic Number: 102**
Atomic Weight: 259.1 g/mol **Known oxidation state(s): NA**

Hazard information: Although nobelium has nine known isotopes, none of them have a long enough existence to determine any of the physical or chemical properties of this element.

Osmium **Atomic Symbol: Os** **Atomic Number: 76** **Atomic Weight: 190.2 g/mol** **Known oxidation state(s): +2, +3, +4, +6, +8**

Oxygen **Atomic Symbol: O** **Atomic Number: 8** **Atomic Weight: 16.0 g/mol** **Known oxidation state(s): -2, +2 (rare)**

Palladium **Atomic Symbol: Pd** **Atomic Number: 46** **Atomic Weight: 106.4 g/mol** **Known oxidation state(s): +2, +4**

Phosphorus **Atomic Symbol: P** **Atomic Number: 15** **Atomic Weight: 31.0 g/mol** **Known oxidation state(s): -3, +3, +5**

Hazard information: White phosphorus is spontaneously flammable in contact with atmospheric oxygen and burns to form the acidic oxide P_2O_5, phosphorus is soluble in many organic solvents and as such it is usually stored under water. In addition to this white phosphorus is highly toxic by ingestion or skin contact, areas of contact with the skin of white phosphorus should be treated immediately with a solution of copper sulfate and medical attention should follow. The red allotrope is fairly benign and possesses no extensive toxicological properties, phosphates are an essential part of our daily diet. Phosphorus should never be heated with aqueous base or the generation of phosphine may result.

Additional information on Phosphorus: Phosphorus is one of the ancient elements in that it was discovered well before the modern era, some time in the late 1500's. It was originally obtained by distillation to dryness of putrefied urine and was instantly coveted for the ability to glow in the dark. The next step in phosphorus production came when people realized that phosphorus was in bones, from then bones were the raw material, first being treated with concentrated sulfuric acid to create a solution of calcium super phosphate, which was then filtered and heated to drive off water. The resulting impure super phosphate was treated with coal and heated in clay retorts to liberate phosphorus. Later improvements utilized a mixture of silicon dioxide and coal as the reducing mixture. In modern times the use of bones have been replaced by phosphate rock, and the heating is now done with resistance heating, involving temperatures in excess of 1200 °C. Lower temperature reduction of phosphates can be facilitated however using easily reduced phosphates such as sodium hexametaphosphate and strong reducing agents such as aluminum or magnesium.

White phosphorus is the 'mother' allotrope of all phosphorus, all other allotropes convert to white phosphorus on distillation at standard pressure. It consists of individual P_4 molecules and is very soft, easily cut with a knife, when pure it looks like nearly clear wax. Phosphorus also has a low melting point (44°C) and a reasonably low boiling point (280 °C). As mentioned previously phosphorus occurs in several allotropes, a red allotrope being the most common, it is utilized as a catalyst in the striker pad of match books, it is a polymeric form of phosphorus and is made by dissolving phosphorus in molten lead and keeping it at its melting point for five days or so before removing from the lead in any of a number of ways including electrolysis of the resulting lead. Red phosphorus can also be prepared from white phosphrous simply by the action of light on white phosphorus, as shown in the picture above. It also occurs as a black allotrope which is the most stable, this is formed with difficulty under several hundred times atmospheric pressure and with heating, and occasionally in the presence of a mercury catalyst. Both allotropes sublime under heating and condense as the white allotrope.

Phosphorus finds use in organic synthesis mostly in the form of its inorganic compounds such as PCl3, PCl5, POCl3, and PBr3. The halides being easily formed by direct reaction of phosphorus with the halogen in question. These compounds are fuming highly reactive chemicals, reacting with water to form phosphoric acid and the hydrogen halide for the most part. In the United States phosphorus is a controlled substance and it is illegal for an individual to own in nearly any quantity. This is due mainly to its use in the production of substances of abuse and to a much lesser extent the possibility of producing precursors to nerve gasses.

Platinum **Atomic Symbol: Pt** **Atomic Number: 78**
Atomic Weight: 195.1 g/mol **Known oxidation state(s): +2, +4**

Plutonium **Atomic Symbol: Pu** **Atomic Number: 94**
Atomic Weight: 239.1 g/mol **Known oxidation state(s): +3, +4, +5, +6**

Polonium **Atomic Symbol: Po** **Atomic Number: 84**
Atomic Weight: 210.0 g/mol **Known oxidation state(s): +2, +4**

Potassium **Atomic Symbol: K** **Atomic Number: 19**
Atomic Weight: 39.1 g/mol **Known oxidation state(s): +1**

Praseodymium **Atomic Symbol: Pr** **Atomic Number: 59**
Atomic Weight: 141.0 g/mol **Known oxidation state(s): +3**

Promethium **Atomic Symbol: Pm** **Atomic Number: 61**
Atomic Weight: 146.9 g/mol **Known oxidation state(s): +3**

Protactinium **Atomic Symbol: Pa** **Atomic Number: 91**
Atomic Weight: 231.0 g/mol **Known oxidation state(s): +5**

Radium Atomic Symbol: Ra Atomic Number: 88
Atomic Weight: 226.0 g/mol Known oxidation state(s): +2

Radon Atomic Symbol: Rn Atomic Number: 86
Atomic Weight: 222.2 g/mol Common oxidation statse: +2, +4, +6 (rare)

Rhenium Atomic Symbol: Re Atomic Number: 75
Atomic Weight: 186.2 g/mol Known oxidation state(s): +1, +2, +3, +4 (stable), +5, +6 (stable), +7 (stable)

Rhodium Atomic Symbol: Rh Atomic Number: 45
Atomic Weight: 102.9 g/mol Known oxidation state(s): +3

Rubidium Atomic Symbol: Rb Atomic Number: 37
Atomic Weight: 85.5 g/mol Known oxidation state(s): +1

Ruthenium Atomic Symbol: Ru Atomic Number: 44
Atomic Weight: 101.1 g/mol Known oxidation state(s): +3, +4, +5, +6, +8

Samarium Atomic Symbol: Sm Atomic Number: 62
Atomic Weight: 150.4 g/mol Known oxidation state(s): +3

Scandium Atomic Symbol: Sc Atomic Number: 21
Atomic Weight: 45.0 g/mol Known oxidation state(s): +3

Selenium Atomic Symbol: Se Atomic Number: 34
Atomic Weight: 79.0 g/mol Known oxidation state(s): -2, +2, +4, +6

Silicon Atomic Symbol: Si Atomic Number: 14
Atomic Weight: 28.1 g/mol Known oxidation state(s): -4, +4

Silver **Atomic Symbol: Ag** **Atomic Number: 47**
Atomic Weight: 107.9 g/mol **Known oxidation state(s): +1, +2 (rare)**

Sodium **Atomic Symbol: Na** **Atomic Number: 11**
Atomic Weight: 23.0 g/mol **Known oxidation state(s): +1**

Strontium **Atomic Symbol: Sr** **Atomic Number: 38**
Atomic Weight: 87.6 g/mol **Known oxidation state(s): +2**

Sulfur **Atomic Symbol: S** **Atomic Number: 16**
Atomic Weight: 32.1 g/mol **Known oxidation state(s): -2, +2, +4, +6**

Tantalum **Atomic Symbol: Ta** **Atomic Number: 73**
Atomic Weight: 181.0 g/mol **Known oxidation state(s): +2, +3, +5**

Technetium **Atomic Symbol: Tc** **Atomic Number: 43**
Atomic Weight: 99.0 g/mol **Known oxidation state(s): +4, +5, +6, +7**

Tellurium **Atomic Symbol: Te** **Atomic Number: 52**
Atomic Weight: 127.6 g/mol **Known oxidation state(s): -2, +2, +4, +6**

Terbium **Atomic Symbol: Tb** **Atomic Number: 65**
Atomic Weight: 158.9 g/mol **Known oxidation state(s): +3, +4**

Thallium **Atomic Symbol: Tl** **Atomic Number: 81**
Atomic Weight: 204.4 g/mol **Known oxidation state(s): +1, +3**

Thorium **Atomic Symbol: Th** **Atomic Number: 90**
Atomic Weight: 232.0 g/mol **Known oxidation state(s): +4**

Thulium **Atomic Symbol: Tm** **Atomic Number: 69**
Atomic Weight: 168.9 g/mol **Known oxidation state(s): +3**

Tin **Atomic Symbol: Sn** **Atomic Number: 50**
Atomic Weight: 118.7 g/mol **Known oxidation state(s): +2, +4**

Titanium **Atomic Symbol: Ti** **Atomic Number: 22**
Atomic Weight: 47.9 g/mol **Known oxidation state(s): +3, +4**

Tungsten Atomic Symbol: W Atomic Number: 74
Atomic Weight: 183.9 g/mol Known oxidation state(s): +2, +4, +5, +6

Uranium Atomic Symbol: U Atomic Number: 92
Atomic Weight: 238.0 g/mol Known oxidation state(s): +3, +4, +6

Vanadium Atomic Symbol: V Atomic Number: 23
Atomic Weight: 50.9 g/mol Known oxidation state(s): +2, +3, +4, +5

Xenon Atomic Symbol: Xe Atomic Number: 54
Atomic Weight: 131.3 g/mol Known oxidation state(s): +2, +4, +6, +8

Ytterbium Atomic Symbol: Yb Atomic Number: 70
Atomic Weight: 173.0 g/mol Known oxidation state(s): +2, +3

Yttrium Atomic Symbol: Y Atomic Number: 39
Atomic Weight: 88.9 g/mol Known oxidation state(s): +3

Zinc Atomic Symbol: Zn Atomic Number: 30
Atomic Weight: 65.4 g/mol Known oxidation state(s): +2

A ball of Zinc and turnings produced from it.

Zirconium Atomic Symbol: Zr Atomic Number: 40
Atomic Weight: 91.2 g/mol Known oxidation state(s): +2, +3, +4

Compounds

Technical Terms

Decantation

A method of separation based upon insolubility of a substance in a particular solvent (for instance Fe2O3 in water). The insoluble substance is left to sink to the lower portion of the solvent where after the upper portion of the solvent is poured off.

Destructive Distillation

A process of distillation wherein an organic material (such as wood) is strongly heated in the absence of oxygen. It will decompose into several useful substances which are separated from each other by the distillation process. (E.g. Wood will decompose into volatile gases and charcoal.) This method of distillation differs from “normal” distillation as it is meant to obtain several substances from a single source. Rather than distilling a mix of those substances a single compound is thermolysed to yield several new substances. The original substance is destroyed in the process. Hence; “destructive” distillation.

Suction Filtration

When filtration is not proceeding quick enough it may be sped up by applying a vacuum. The liquid is then "sucked" through the filter (technically it is pushed through the filter by the positive pressure on top of the liquid). Suction filtration is particularly useful when a suspension is being filtered as suspensions tend to hold liquids rather well.

Precipitate

When an insoluble chemical is created in a reaction it will form a very fine powder in suspension. This powder is referred to as a precipitate. Depending on the speed of the precipitation the size of the grains of the powder may vary from extremely small to very large. Artificial diamonds are precipitated carbon crystals; they are precipitated from saturated solutions of carbon in liquid iron. By keeping the speed of precipitation low a single crystal can be grown.

Supernatant

When analysing a system that contains a liquid which is not the desired product and a desired precipitate which has sunk to the bottom, the supernatant is the liquid above the precipitate, the supernatant is usually removed by careful decantation from the solid precipitate until the precipitate remains at the bottom with only a small amount of supernatant which can then be allowed to carefully evaporate or the precipitate can then be filtered.

Cation

Ions with a positive charge (e.g. Fe^{2+}, Cu^{2+} Al^{3+} etc.)

Anion

Ions with a negative charge (e.g. halides such as Cl^-, Br^- and I^- but also P^{3-}, O^{2-} etc.)

Pyrophoric

Substances that spontaneously ignite in air are called "pyrophoric". (E.g. K(s), Na(s), extremely fine metal powders are also pyrophoric. So are boranes, phosphines, silanes and certain metallo-organic chemicals.)

Flash Point

The lowest temperature at which a substance can form an ignitable mixture in air. The lower the flash point the easier a substance can ignite in air.

Explosive Limit

Every substance has 2 explosive limits, an upper- and a lower-explosive limit. When the saturation of the substance has not yet reached the lower-explosive limit it cannot ignite because of a lack of fuel. When the saturation level of the substance in air is exceeded it will not be able to ignite either because there is too much fuel (and thus too little oxygen) for successful (self propagating) combustion. Some substances can have narrow explosive limits such as ammonia 16-25% by volume or very large ranges of explosive concentrations such as hydrazine 3-99% by volume.

Supersaturated

When a solution is "supersaturated" it contains more of a certain chemical in solution than it should be able to contain. Supersaturated solutions are stable as long as they are left undisturbed. As soon as an uneven surface, sufficient motion, or additional components are introduced the excess dissolved chemical will come out of solution in crystalline form.

Supercooled

Some liquids can be cooled below their freezing point without solidifying. They are stable as long as they are left undisturbed. As soon as an uneven surface or sufficient motion is introduced the liquid will solidify.

Anode

A positive electrode (usually the red wire), this is where oxidation occurs in electrolysis.

$Fe^{2+}_{(aq)} \Rightarrow Fe^{3+}_{(aq)}$

Cathode

A negative electrode (usually the black wire), this is where reduction occurs in electrolysis.

$Cu^{2+}_{(aq)} \Rightarrow Cu_{(s)}$

Filtrate

The liquid that is left after a filtration. (E.g. Coffee after hot water is poured upon the ground coffee beans in the filter)

Scrub (gas)

When a gas consists of multiple mixed gases one or more of those gasses may be removed by scrubbing the gaseous mixture. This may be accomplished by leading the gas through a reagent (-solution) that will react with only some of the constituents of the mix. (E.g. leading air containing H2S through a NaOH or KOH solution will remove the H2S gas from the air.) Scrubbers are often used to remove potentially harmful gasses from effluent gases. They are also used to remove gases from air before the air is lead into a reaction chamber if these gases might unduly influence the desired reaction. (E.g. water is removed from air before it is lead into a chamber where it will be used to react with SO_2 to form SO_3 to prevent premature formation of sulphuric acid.)

Wash (precipitate)

A precipitate can be “washed” by adding the solvent that it will not dissolve in and decanting or filtering it. Every time the precipitate is “washed” it will become more pure as the pollutions it contained are dissolved and flushed away.

Superheating

Superheating occurs when a liquid is heated past its boiling point without actually boiling. This can happen by rapidly heating a homogenous liquid in the absence of nucleation sites where bubbles may form. (A nice example is boiling water in a clean glass in a microwave. It will appear as if not boiling (no bubbles are formed) but as soon as a teabag is introduced explosive boiling may occur [see Flash Boiling].)

Flash Boiling

An explosive form of boiling where all of a liquid boils instantaneously. (Rather than nucleated boiling as would normally occur where bubbles are formed) May occur when a superheated liquid is suddenly violently shaken or subjected to a huge amount of boiling-nuclei such as a spoonful of sugar of finely ground tea in the case of micro waved tea water. Flash boiling can also occur if a new clean beaker or test tube is heated containing a liquid un disturbed, the liquid can then attain a temperature above its boiling point [see Superheating] and upon any outside mechanical action can flash boil which may result in hot liquid going everywhere, the addition of boiling stones, or pieces of glass, or mechanical stirring from the start can avoid this problem.

Catalyst

A substance that accelerates a reaction (by lowering the activation energy of that reaction) without being used up that does not influence the equilibrium of that reaction. An example being manganese dioxide and hydrogen peroxide mixing together and resulting in the decomposition of the peroxide to oxygen and water.

Stoichiometry

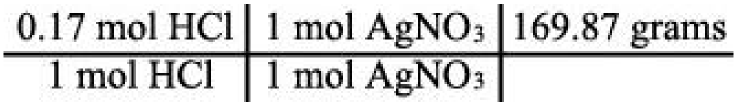
Stoichiometry is a quantitative approach to chemistry. In a stoichiometric calculation the mols of one substance is equated to another factor, for example the mols of another substance or to the volume of a pure liquid required to give a certain concentration. If a reaction is referred to as a ‘stoichiometric’ reaction it usually insinuates that equimolar ratios are used, i.e., the exact amounts of reactants are used as predicted by theory in a reaction and it is usually assumed the reaction goes to completion. Here is an example stoichiometric calculation:

$$AgNO_{3(aq)} + HCl_{(aq)} \Rightarrow HNO_{3(aq)} + AgCl_{(s)}$$

Above is the reaction that we wish to follow, let’s assume we have 100 ml of a 1.70 M HCl solution and we want to convert it all to HNO3, how much AgNO3 do we need to make the reaction stoichiometric?

0.1 ~~L~~ HCl x 1.70 mol/~~L~~ = 0.17 mol HCl

Notice that the liters cancel in the above reaction, hence the strike through, they cancel because on is in the numerator and the other in the denominator, they divide away. Now that we have the number of mols of HCl that are in solution we can carry out the more difficult stoichiometric calculation.



0.17 mol HCl	1 mol $AgNO_3$	169.87 grams
1 mol HCl	1 mol $AgNO_3$	

This is how a majority of American texts will teach stoichiometry. Starting from the left we see the 0.17 mol of HCl we had calculated before, now we need the number of grams that will give us the same number of mols of silver nitrate. So, the next step is the conversion, one mole of hydrochloric acid requires one mole of silver nitrate. So you divide by 1 mol HCl to cancel HCl and that equates to one mol AgNO3 which appears in the numerator. Now, one mole of AgNO3 is equal to 168.87 grams, so divide by 1 mol AgNO3 to cancel it because we want our answer in grams and multiply by the number of grams per mol. So in the above equation everything cancels but the grams, which is the answer you want, in this case it is 28.88 grams that are necessary. Note that there are easier less elaborate systems to get the same number but they are all considered stoichiometric.

Allotrope

A particular form of an element. (E.g. Phosphorous can exist as white and red phosphorous; oxygen can exist as O2 and O3; carbon can exist as graphite, diamond, fullerenes, nanotubes and many more forms.) Specific allotropes of elements may be denoted by stating the element and its allotropic form as follows "Element (allotropic form)". E.g. Diamond may be denoted as $C_{(diamond)}$ and graphite may be denoted as $C_{(graphite)}$. Phosphorous is often denoted as either $P_{(red)}$ or $P_{(white)}$.

Phase (liquid)

In this test the term phase is used to discern between two immiscible liquids. In most cases there is an aqueous phase and an organic phase. Usually the aqueous phase is the bottom phase, and the organic is the top, the determination of which is top and which is bottom made by the density of the two liquids. However there are organics more dense then water, chloroform being a good example (d. 1.5 g/ml) which would form the bottom phase in a strictly water/chloroform mixture. Three phase mixtures can exist but are unstable and can mix together with agitation. Two phase mixtures can often be forced to mix together using strong shaking, at least for short periods of time forming emulsions which can separate back into their two components, although some emulsions are stable. The concept of these different liquid phases comes into use when washing a liquid compound or solution and keeping track of which is your product and which is your waste phase. A good way to differentiate is to simply add a drop of water to your phases and see which phase it ends up in if you are having trouble discerning organic form aqueous phases.

Azeotrope

An azeotrope is a liquid mixture of two or more components which has a unique constant boiling point. This azeotrope may boil at a higher, lower, or intermediate temperature, relative to the constituent liquids, and the liquid retains the same composition as it is boiled. As a consequence, the vapour has the same composition as the liquid and simple distillation will not separate the constituents as it would with most liquid mixtures.

The word azeotrope comes from the Greek "zein tropos", or "constant boiling". An azeotrope is said to be positive if the constant boiling point is at a temperature maximum, and negative when the boiling point is at a temperature minimum. The vast majority of azeotropes are minimum boiling. All liquid mixtures which are immiscible and which form azeotropes are minimum boiling.

Examples of azeotropes:

* Nitric acid (68.4%) / water, boils at 122°C
* Perchloric acid (28.4%) / water, boils at 203°C (negative azeotrope)
* Hydrofluoric acid (35.6%) / water, boils at 111.35°C (negative azeotrope)
* Ethanol (95%) / water, boils at
* Sulphuric acid (98.3%) /water, boils at 330°C
* Acetone / methanol / chloroform form an intermediate boiling azeotrope

(Source: http://en.wikipedia.org/wiki/Azeotrope)

Electrolyte

A substance that may dissociate into ions when dissolved in a solvent. Salts, acids and bases are all by definition electrolytes. In aqueous solutions electrolytes allow for the conduction of electric current.

Eutectic

A eutectic is a mixture of 2 or more elements that has a lower melting point than any of its constituents and usually the lowest melting point between the two substances possible. (E.g. A solution of NaCl in water will freeze at -21.2 °C which is a lower melting point than that of any of its constituents.) An eutectic mixture can be thought of loosely as somewhat of an azeotrope of freezing. If for example a solution has more of component A then B to form the lowest melting eutectic, the component A will crystallize out or otherwise separate first and then the solution further cooled until the eutectic mixture crystallizes out. Other notable eutectics include the eutectic formed between potassium (78% by wt.) and sodium (22% by wt) metals (fp –12.6 °C) and the common eutectics taken advantage of in over the counter solders.

Polar (liquid)

A liquid that consists of polar molecules. A molecule is polar when the electron density is unevenly spread across the molecule. For instance water is a polar liquid because the O is more negatively charged than the 2H's (which are relatively positively charged). Polar liquids may dissolve (some) salts into their constituent ions and are referred to as hydrophilic (water loving) liquids. Because they will mix with water. Generally speaking polar substances will dissolve into polar solvents and not into non-polar solvents.

Non-Polar (liquid)

A non-polar liquid is a liquid that is made up of nonpolar molecules. Non-polar liquids generally have lower boiling points than polar liquids because they are not held together by molecular bonds as tightly as polar molecules. (Think of a polar liquid as many many tiny magnets flowing through each other. They attract each other. A non-polar liquid would be like a lot a tiny tiny plastic bars flowing through each other. They do not attract each other very much at all). They referred to as hydrophobic (water fearing) liquids because they will not mix with polar liquids very well at all. They will not dissociate salts into their constituent ions. Generally speaking non-polar substances will dissolve into non-polar solvents and not into polar solvents.

Supercritical water

At STD water will boil at 100°C (or 212°F or 373,16K) but when the pressure is raised its boiling point will rise with it. The pressure can be raised until a certain pressure is reached at which the water will no longer boil at all. In stead it will reach a next “phase”, the so-called “supercritical” phase. This is not really a phase as it is more of a hybrid between a gaseous and a liquid state. It is considered such because it has properties of both these phases. It has excellent solvent properties like a liquid (supercritical water can dissolve pure gold!) but also possesses excellent diffusability (It can quickly fill up any hole) like a gas. It also has some unique capabilities of its own. Its volume can vary to a great extent in a continuous manner when the pressure and temperature are varied. Water becomes supercritical at a pressure of 22,1MPa and a temperature of 374°C. Carbon dioxide (CO_2) can also become supercritical but at 7,38MPa and 31.1°C. All liquids can become supercritical at the right pressure and temperature. Supercritical liquids/gases are used in the industry as high-efficiency solvents. E.g. Supercritical CO_2 is used to extract caffeine from coffee beans to make de-caf coffee (the leftover caffeine is used for medicinal applications).

13.0 Appendix (Specific Procedures/Additional Experiments)

13.1 Salts (Nearly)Insoluble in cold neutral water:

Calcium Oxylate (AS)	$Ca(COO)_2$	6.7 x 10^{-4} g/100ml
Barium Sulfate	$BaSO_4$	2.22 x 10^{-4} g/100ml
Silver Bromide	AgBr	8.4 x 10^{-6} g/100ml
Magnesium Hydroxide (AS)	$Mg(OH)_2$	9.0 x 10^{-4} g/100ml
Barium Carbonate	$BaCO_3$	2.0 x 10^{-3}

Calcium Sulfate	$CaSO_4$ or $CaSO_4 * 2H_2O$	.241 g/100ml
Silver Chloride	AgCl	8.9 x 10^{-5} g/100ml
Silver Iodide	AgI	3 x 10^{-7} g/100ml
Aluminum Fluoride	AlF_3 or AlF_3*3H_2O	----
Barium	$BaCrO_4$	3.4 x 10^{-4}

(AS)		g/100ml
Barium Citrate	$Ba_3(C_6H_5O_7)_2 * 7H_2O$	4.06×10^{-2} g/100ml
Barium Phosphate (AS)	$Ba_3(PO_4)_2$	------
Bismuth Hydroxide (AS)	$Bi(OH)_3$	1.4×10^{-4} g/100ml
Camium Hydroxide (AS)	$Cd(OH)_2$	2.6×10^{-4} g/100ml
Calcium Carbonate (AS)	$CaCO_3$	1.45×10^{-3} g/100ml
Calcium Hydroxide (AS)	$Ca(OH)_2$	1.8×10^{-1} g/100ml
Calcium Metasilicate (AS)	$CaSiO_3$	9.5×10^{-3} g/100ml
Cobalt (II) Carbonate (AS)	$CoCO_3$	-----
Cobalt (II) and (III) hydroxide (AS)	$Co(OH)_2$ & $Co(OH)_3$	-----
Copper (I) Halides (AS) (Am)	CuX	-----
Lead Phosphate (AS) (BS) (OxA)	$Pb_3(PO_4)_2$	1.4×10^{-5} g/100ml
Lead (II) Chloride	$PbCl_2$	6.73×10^{-1} g/100ml
Lead (II) Sulfide (OxA) (AS)	PbS	1.2×10^{-2} g/100ml
Copper (II) Sulfide (OxA) (Am)	CuS	3.3×10^{-5} g/100ml
Arsenic Sulfide (OxA)	As_2S_3	-----
Tin (IV) Sulfide (OxA)	SnS_2	-----
Iron (II) Sulfide (OxA)	FeS	-----
Zinc Sulfide (OxA)	ZnS	-----

Chromate (AS)		g/100ml
Barium Oxylate (AS)	$Ba(COO)_2$	9.3×10^{-3} g/100ml
Barium Sulfite	$BaSO_3$	2.0×10^{-2} g/100ml
Cadmium Carbonate (AS)	$CdCO_3$	-----
Cadmium Oxalate (AS)	$Cd(COO)_2$	3.3×10^{-3} g/100ml
Calcium Fluoride	CaF_2	1.6×10^{-3} g/100ml
Calcium Phosphate (AS)	$Ca_3(PO_4)_2$	2.5×10^{-3} g/100ml
Cesium Aluminum Sulfate	$CsAl(SO_4)_2 * 12H_2O$	3.4×10^{-1} g/100ml
Cobalt (II) Chromate (AS) (OxA)	$CoCrO_4$	-----
Copper (I) & (II) Carbonate (AS) (Am)	Cu_2CO_3 & $CuCO_3$	-----
Copper (I) & (II) Hydroxide (Am)	CuOH & $Cu(OH)_2$	-----
Mercury (I) Chloride	Hg_2Cl_2	2.1×10^{-4} g/100ml
Mercury (II) Sulfide	HgS	1.0×10^{-6} g/100ml
Copper (I) Sulfide (OxA) (Am)	Cu_2S	
Cadmium (II) Sulfide (OxA)	CdS	-----
Antimony Sulfide (OxA)	Sb_2S_3	-----
Aluminum Hydroxide (AS) (BS)	$Al(OH)_3$	-----
Manganese (II) Sulfide	MnS	-----
Nickel (II) Sulfide (OxA)	NiS	-----

Cobalt (II) Sulfide (OxA)	CoS	-----
Zinc Hydroxide (AS) (BS)	$Zn(OH)_2$	-----
Iron (III) Hydroxide	$Fe(OH)_3$	-----
Bismuth (III) Sulfide (OxA)	Bi_2S_3	-----
Iron Ferricyanide	$Fe_3[Fe(CN)_6]_2$	-----
Lead (II) Chromate (BS) (AS)	$PbCrO_4$	5.8×10^{-6} g/100 ml
Lead (II) Hydroxide (BS) (AS)	$Pb(OH)_2$	1.5×10^{-2} g/100ml
Lead (II) Oxylate	$Pb(COO)_2$	1.6×10^{-4} g/100ml
Magnesium Carbonate (AS)	$MgCO_3$	-----
Magnesium Oxylate (BS) (AS)	$Mg(COO)_2$	7.0×10^{-2} g/100ml
Manganese (II) Fluoride (AS)	MnF_2	-----
Manganese (II) Oxylate (AS)	$Mn(COO)_2$	-----
Mercury (I) and (II) Carbonate (AS)	Hg_2CO_3 & $HgCO_3$	-----
Molybdenum (II) and (III) Bromide	$MoBr_2$ & $MoBr_3$	-----
Molybdenum Sulfides	Mo_2S_3 and MoS_2	-----
Nickel (II) Fluoride	NiF_2	2.0×10^{-2} g/100ml
Nickel (II) Oxylate (AS)	$Ni(COO)_2$	-----
Potassium Perchlorate	$KClO_4$	7.5×10^{-1}
Silver Oxide (AS)	Ag_2O	1.3×10^{-3} g/100ml
Silver Sulfate (Am) (AS)	Ag_2SO_4	5.7×10^{-1} g/100ml

Strontium Phosphate	$Sr_3(PO_4)_2$	-----
Chromium (III) Hydroxide (AS) (BS)	$Cr(OH)_3$	-----
Copper (II) Oxylate (Am)	$Cu(COO)_2$	2.5×10^{-3} g/100ml
Gold Sulfide (OxA)	Au_2S	-----
Lead (II) Bromide (AS)	$PbBr_2$	4.5×10^{-1} g/100ml
Lead (II) Carbonate (BS) (AS)	$PbCO_3$	1.1×10^{-4} g/100ml
Lead (IV) Oxide (AS)	PbO_2	-----
Lead (II) Sulfate	$PbSO_4$	4.25×10^{-3} g/100ml
Magnesium Fluoride (OxA)	MgF_2	7.6×10^{-3} g/100ml
Magnesium Phosphate (AS)	$Mg_3(PO_4)_2$	2.0×10^{-2} g/100ml
Manganese (II) Hydroxide (AS)	$Mn(OH)_2$	2.0×10^{-3} g/100ml
Mercury (I) (AS) and (II) Bromide	Hg_2Br_2 & $HgBr_2$	------
Mercury (II) Phosphate (AS)	$Hg_3(PO_4)_2$	------
Molybdenum (II) and (III) Chloride	$MoCl_2$ & $MoCl_3$	-----
Nickel (II) Carbonate (AS)	$NiCO_3$	9.3×10^{-3} g/100ml
Nickel (II) Hydroxide (AS) (Am)	$Ni(OH)_2$	1.3×10^{-3} g/100ml
Nickel (II) Phosphate (AS)	$Ni_3(PO_4)_2$	-----
Silver Carbonate (Am)	Ag_2CO_3	3.2×10^{-3} g/100ml
Silver Phosphate (Am) (AS)	Ag_3PO_4	6.5×10^{-4} g/100ml
Tin Phosphate	$Sn_3(PO_4)_2$	-----

Zinc Carbonate (AS) (BS)	$ZnCO_3$	1.0×10^{-3} g/100ml

Zinc Cyanide (BS)	$Zn(CN)_2$	5.0×10^{-4} g/100ml

(AS) = Increased solubility in acids (BS) = Increased solubility in bases (OxA) = Soluble in oxidizing acidic conditions (Am) = Can be rendered soluble in the presence of ammonia

13.2 A study of various additives on kaolin based ceramics by Cyrus:

Hypotheses:

The rate of shrinkage upon firing decreases linearly as the percentage of kaolin decreases. The green strength (unfired strength) and fired strength of the rods decreases linearly as the percentage of kaolin decreased. The strongest and most suitable formulation for crucibles is composed of clay and graphite.0

Experimental Procedure:

- **Powder Mixing:**

~Various ceramic powders were weighed out using an Ohaus dial-o-gram scale and placed in a cup. (for example 6.00 g kaolin, 4.00 g silica; 10.00 grams total material was used for most formulations)

~The contents were stirred for several minutes, placed in another cup, combined with enough water to make the mixture very plastic but not enough to make the mixture fluid, and then mixed using a painter's spatula for several more minutes.

- **Extrusion**

~Using the spatula, the ceramic mixture was placed inside of a syringe with a 0.476 cm inside diameter extrusion orifice.

~The slurry was then extruded onto paper towels by gently pressing down on the syringe piston as the syringe was slowly drawn back toward the body at a low angle relative to the paper towel. The bead diameter was kept as close to 0.476 cm as possible.

~The ceramic rods (approximately 5 per formula) were dried and organized.

- **Pre-firing measurements**

~The lengths of 2 rods of each formula were measured in centimeters using a standard ruler accurate to the nearest millimeter, and estimating to the nearest tenth of a millimeter.

~Several rods of each formula were placed on the 3 point flexural strength apparatus and the pressure on the rods was increased gradually until the rod cracked. The force required to crack each rod was recorded.

- **Firing**

~Each ceramic rod was labeled using a glaze composed of black iron oxide, talc, and kaolin, placed in slip cast kaolin/alumina crucibles, and preheated in the oven to 550 deg. F. This drives off any water remaining, preventing the rods from exploding from steam.

~The crucibles were then placed in a furnace, and fired for approximately 1 hr using an oil burner on a low setting, not going above approximately 900 deg. F.

~Wood kindling was then added to the furnace as air was blown through the oil burner for approximately 1.5 hrs. The ceramic rods reached approximately an orange to yellow heat.

- **Post-firing measurements**

~The previously measured rods (M1) were measured again (M2), and the shrinkage was determined by 100*(M1- M2)/M1.

~Several remaining rods of each formula were then placed on the flexural strength apparatus, and the force required to break each was recorded.

- **Variables**

~The independent variables were the percentage of aggregate or additives and the composition of those additives.

~The dependent variables were the percentage of shrinkage of the clay rods when fired, the green or unfired strength of the rods, and the fired strength of the rods.

~The constants were the thickness of the rods, the methods of mixing, extruding, drying, and firing the rods.

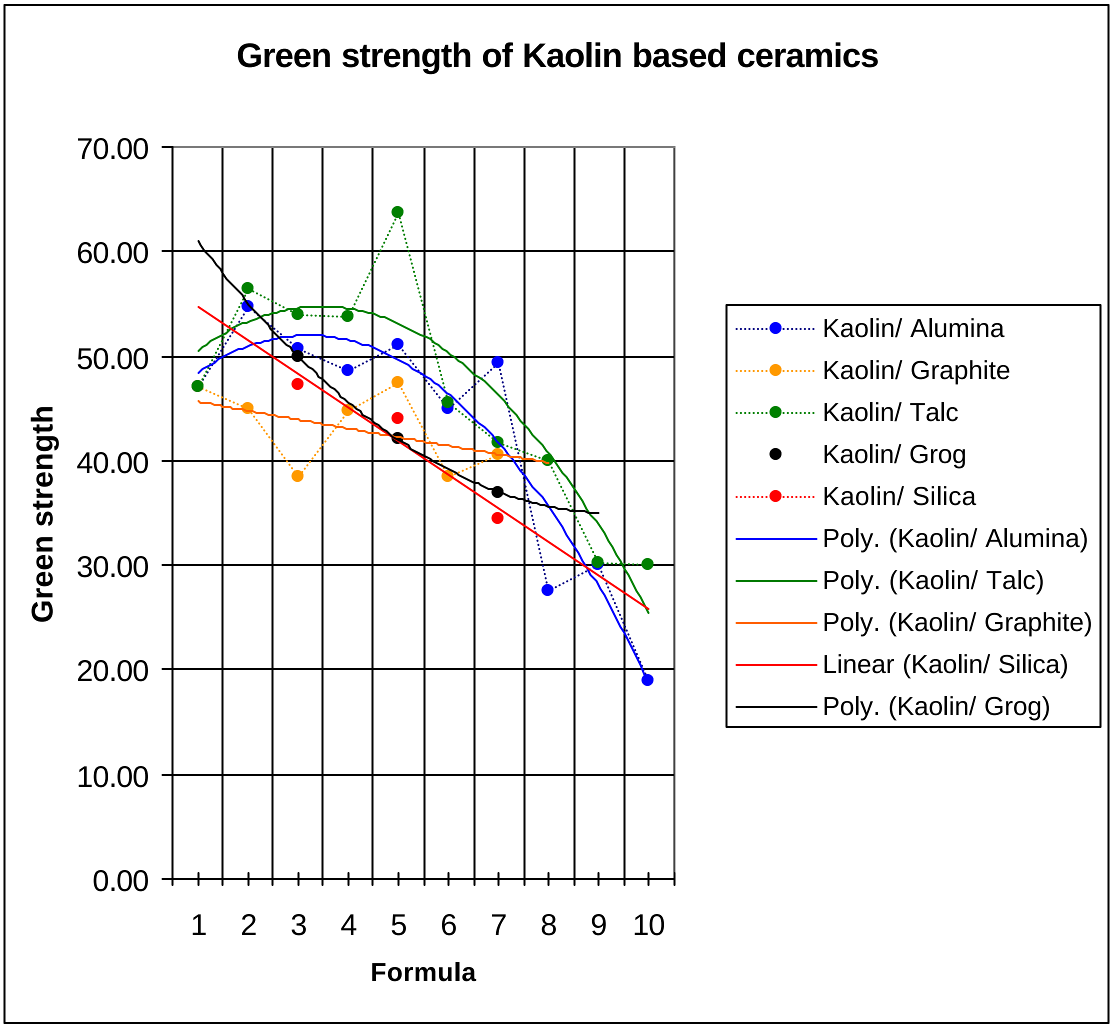
Conclusions:

The first hypothesis, that as the percentage of kaolin decreased the percentage of shrinkage would also decrease linearly, was found to be approximately correct. This is due to the behavior of ceramic compounds at different temperatures. Kaolin is composed of many small flake-like particles; when heated to high temperatures the molecules within these particles vibrate so rapidly that they begin to diffuse across the particles, fusing the particles together. As the temperature further increases the molecules vibrate more rapidly and the particles behave more like a liquid; surface tension draws the particles of kaolin together, causing the ceramic to shrink as a whole. If the temperature increases even further, the kaolin will actually shrink into a puddle and become a liquid. Shrinkage depends greatly on the mobility of molecules, and their ability to contract, which is determined mostly by temperature. Also, once clay has been fired to a maturing temperature, it will not shrink nearly as much when fired to that temperature again; the particles have already fused into a mostly solid mass (matured) and cannot shrink much more. This is why grog decreases the shrinkage of kaolin. While the kaolin in kaolin/grog ceramics does shrink when fired, the grog does not shrink, reducing the total amount of shrinkage. Other additives such as alumina and graphite also decrease the shrinkage of ceramics because they do not shrink significantly when fired. Their molecules are bound tightly together, as indicated by very high melting points, and thus cannot fuse together and shrink as easily as kaolin does. Finally, the larger grained powders, grog and silica, caused the ceramics to shrink less than the smaller grained powders, talc and alumina.

The second hypothesis was that the green and fired strengths of kaolin based ceramics would decrease linearly as the percentage of kaolin decreased. On the whole, the strength did decrease as the percentage of kaolin decreased, but not in a linear fashion. The reason for the reduction of strength is simple in green or unfired ceramics. Kaolin has the unique property that when wet and dried, the particles adhere to one another significantly. Other powders such as alumina and silica will not adhere to one another when wet and dried. Thus, as the percentage of kaolin decreases, the percentage of particles that actually bind to other particles also decreases, causing a reduction in strength. All formulations showed a marked increase in strength at 60% kaolin/ 40% additive. Since no chemical processes are taking place, this increase in strength is purely mechanical; kaolin's mostly flat particles have the most mechanical strength when mixed with 40% of other mostly rounded particles. A variation in sizes and shapes of particles allows the particles to interlock more effectively, making them noticeably stronger. The fired strength of kaolin based ceramics also decreases smoothly, except in the case of kaolin/talc and kaolin/alumina. For example, 50% kaolin/ 50% talc is much stronger than would be expected (see graph). It could be that these rods were extruded improperly and were thicker than normal. In this case, though, the green strength would also probably be noticeably higher, which it is not. It also could be that this ratio of kaolin and talc is near a eutectic point, the ratio of 2 chemicals at which their melting point is lowest. This would also cause the ceramic to shrink more; the shrinkage graph shows that 50% kaolin/ 50% talc shrinks more than would be expected. In the case of alumina, it is not known what caused the peak in strength at 30% kaolin/ 70% alumina. It is probably not a eutectic, because all combinations of kaolin/alumina have very high melting points, and so could merely be an error.

The third hypothesis, that kaolin/graphite formulations would be the best, was completely incorrect. The graphite was burned away by oxygen in the furnace, leaving the ceramics very porous and weak, the green strength was below average, the shrinkage was merely average, and graphite is one of the harder to obtain chemicals used, making it the least practical. This research indicates that the best formulation for mechanical green strength was determined to be 60% kaolin/ 40% additive, but the green strength of a ceramic is not as significant as its fired strength; crucibles would only be used in their fired state. The best formulation for fired strength was determined to be 80% kaolin/ 20% alumina or talc. Fired strength, though, must be balanced with low shrinkage. Several tests have indicated that crucibles with high rates of shrinkage will crack when fired. The best additive to reduce shrinkage is about 70% kaolin/30% grog, and its fired strengths are not much lower than alumina. A solution that may meet all of these requirements would be to fire pieces made of 80% kaolin/ 20% alumina, crush and powder them for use as grog, and then mix that with kaolin and alumina in order to obtain a ceramic crucible with the formula 80% kaolin, 20% alumina, which is the strongest, but also comprised of 70% unfired kaolin and alumina/ 30% fired kaolin and alumina grog, which would have low shrinkage.

This study had several errors, which could easily be fixed in future research. First, using only a simple syringe it was impossible to extrude rods of the same diameter every time, causing some variation. This could be solved by using a proper clay extrusion device, and extruding square cross section rods, instead of round cross section rods, which could be tested for flexural strength more accurately. Second, different amounts of water were added to each formula in order to make the formula easily mixable; this may have affected the green strengths, and could be solved by using a pipette to accurately deliver water. Third, the firing of the ceramic rods was inexact. Neither the exact length nor the exact temperature reached was measured, and different parts of the furnace may have been at different temperatures. Using a large pottery kiln and pyrometric cones to measure the temperature would solve this problem. Although the methods used in this experiment were not always precise, the data itself shows that there are significant and quantifiable differences between the effects of various additives on kaolin based ceramics.



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All contributors were members of the mad science discussion forum [http://www.sciencemadness.org]

- *Sections on Glassware and Pyrex partially written by and based off writings by Reed Winn or 'Quantum (from USA)'*
- *Suggestions regarding topics were provided by 'Magpie (from USA)'*
- *The electrolysis section would have been near impossible without 'Tacho (from Brazil)' who wrote most everything.*
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