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 f documents, however directly linking to the html document is possible, still though I would prefer links be to the ma project page.

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 s houldn'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

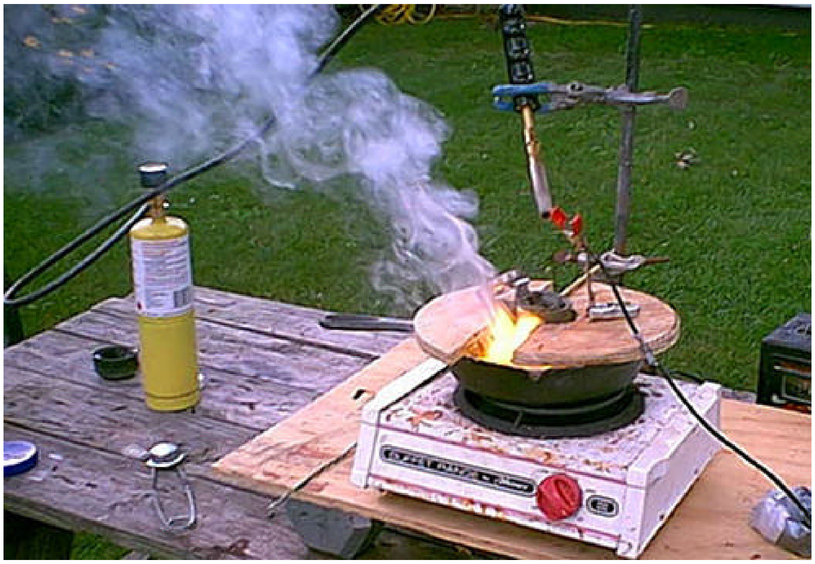
Peroxides by definition are chemicals that contain an oxygen-oxygen bond which puts oxygen in the –1 oxidation state, overall –2 as usual though since peroxides are by virtue of their nature diatomic. Most inorganic peroxides possess at least moderate stability, and if they do indeed prove to be unstable, 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 o f 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 o f 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 t he 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