## 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<sup>(1)</sup>. I therefore find it necessary to warn all persons viewing this document that  $\mathbf{i}$  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 <u>Rob.Vincent@gmail.com</u> 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 a documents, however directly linking to the html document is possible, still though I would prefer links be to the ma project page.

### **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)

ActiniumAtomic Symbol: AcAtomic Number: 89AtomicWeight:227.0 g/molKnown oxidation state(s): +34

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

AluminumAtomic Symbol: AlAtomic Number: 13AtomicWeight:27.0 g/molKnown oxidation state(s): +34

**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 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.

AmericiumAtomic Symbol:AmAtomic Number:95AtomicWeight:241.1 g/molKnown oxidation state(s): +3+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: 12	1.76 g/mol	Known oxidation state(s): +3	6, +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.

ArgonAtomic Symbol: ArAtomic Number: 18AtomicWeight: 40.0 g/molKnown oxidation state(s): No commonoxidation 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)

ArsenicAtomic Symbol: AsAtomic Number: 33AtomicWeight:74.9 g/molKnown oxidation state(s): +2, +3, +5 (leastcommon)

**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  $20^{\text{th}}$  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.

BerkeliumAtomic Symbol: BkAtomic Number: 97AtomicWeight: 249 g/molKnown 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	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.

<b>Bismuth</b>	Atomic Symbol: Bi	Atomic Number: 83	Atomic
Weight:	209.0 g/mol	Known oxidation state(s): +3, +5	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<sub>3</sub>/NaOH on bismuth trioxide, it will oxidize manganate to permanganate.

# BoronAtomic Symbol: BAtomic Number: 5AtomicWeight: 10.9 g/molKnown 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.

$$\begin{split} Na_{2}B_{4}O_{7(aq)} + 2HCl_{(aq)} + 5H_{2}O_{(l)} &\Rightarrow 2NaCl_{(aq)} + 4B(OH)_{3(s)} \\ \\ &2B(OH)_{3(s)} - (Heat) - > B_{2}O_{3(s)} + 3H_{2}O_{(g)} \\ \\ &B_{2}O_{3(s)} + 3Mg_{(s)} - (Heat) - > 2B_{(s)} + MgO_{(s)} + x[MgB_{2}]_{(s)} \end{split}$$

 $B(s)/MgO(s)/MgB_2(s) - (HCl_{(aq)}) - > B_{(s)} + MgCl_{2(aq)} + B_2H_{6(g)}$ 

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 :

 $B_{(s)} + Cl_{2(g)} \Longrightarrow BCl_{3(g)}$  $2BCl_{3(g)} + 3Zn_{(s)} \Longrightarrow 2B_{(s)} + 3ZnCl_{2(s)}$ 

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<sub>4</sub>] 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 Weight: 79.9 g/mol +5, +7 (rare)

Atomic Number: 35 Known oxidation state(s): -1, +3,

**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:

$$2NaBr_{(aq)} + H_2SO_{4(1)} + H_2O_{2(aq)} \Longrightarrow Na_2SO_{4(aq)} + 2H_2O_{(1)} + Br_{2(1)}$$

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 Weig	ght: 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<sub>2</sub> 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 V	Veight: 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 V	Veight: 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	e Weight: 12.01 g/mol	Known oxidation state(s): -4,
+4 (Carbon ca	n 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. Resorting to the action of both carbon and chlorine gas on an elemental oxide at high temperatures can be an even more powerful reducing agent, able to reduce the inert TiO<sub>2</sub>. 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 Weight: 132.9 g/mol Atomic Number: 55 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<sub>2</sub>, 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 V	Veight: 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
Atomi	c 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 s alt 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.

CuriumAtomic Symbol: CmAtomAtomic Weight:247.1 g/molK

Atomic Number: 96 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 W	eight: 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 W	eight: 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 V	Veight: 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 V	Veight: 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.

FermiumAtomic Symbol: FmAtomic Number: 100Atomic Weight: 254 g/molKnown oxidation state(s): +3

**Hazard information:** Highly radioactive, most stable isotope only has a half-life of 3 hours.

Fluorine Atomic Symbol: F Atomic Weight: 19.0 g/mol Atomic Number: 9 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 V	Veight: 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 W	eight: 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 W	/eight: 70.0 g/mol	Known oxidation state(s): +2, +3

Hazard information: NA

Additional information on Gallium: 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 W	eight: 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 (au	ric)	

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 V	Veight: 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 Weight: 4.0 g/mol common oxidation states Atomic Number: 2 Known oxidation state(s): No

**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  $\gamma$  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 W	/eight: 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 V	Veight: 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<sub>4</sub>, H<sub>2</sub>O, and others, hydrogen exhibits the -1 state in metallic hydrides for the most part such as NaH and LiAlH<sub>4</sub> (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<sub>2</sub> and Br<sub>2</sub>. Some extra precautions should be taken with hydrogen due to its high degree of flammability 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
A	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.

IodineAtomic Symbol: IAtomic Number: 53Atomic Weight: 126.9 g/molKnown 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 (rare)	Atomic Symbol: Kr Weight: 83.8 g/mol	Atomic Number: 36 Known oxidation state(s): +2	
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 informa	ation: Would be highly radioacti	we as the most abundant isotope only	

has a half-life of 8 seconds.

Lead Atomic W	Atomic Symbol: Pb /eight: 207.2 g/mol	Atomic Number: 82 Known oxidation state(s): +2,
+4	0 0	
Lithium	Atomic Symbol: Li	Atomic Number: 3
Atomic W	eight: 6.94 g/mol	Known oxidation state(s): +1
Lutetium	Atomic Symbol: Lu	Atomic Number: 71
Atomic W	eight: 175.0 g/mol	Known oxidation state(s): +3
Magnesium	Atomic Symbol: Mg	Atomic Number: 12
Atomic W	/eight: 24.3 g/mol	Known oxidation state(s): +2



Manganese Atomic Symbol: Mn Atomic Weight: 54.9 g/mol +3, +4, +6, +7

Atomic Number: 25 Known oxidation state(s): +2,



Atomic Symbol: Md Mendelevium Atomic Weight: 258.1 g/mol

Atomic Number: 101 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		Atomic Number: 80
Atomic Weight: 200.6 g/mol		Known oxidation state(s): +1
(diatomic), +	2	
Molybdenun	n Atomic Symbol: Mo	Atomic Number: 42
Atom	ic Weight: 95.5 g/mol	Known oxidation state(s): +2, +3
Neodymium	Atomic Symbol: Nd	Atomic Number: 60
Atom	ic Weight: 144.2 g/mol	Known oxidation state(s): +3
Neon	Atomic Symbol: Ne	Atomic Number: 10
Atom	ic Weight: 20.2 g/mol	Known oxidation state(s): No
Common Ox	idation States	
Neptunium	Atomic Symbol: Np	Atomic Number: 93
Atom	ic Weight: 237.1 g/mol	Known oxidation state(s): +5
<mark>Nickel</mark>	Atomic Symbol: Ni	Atomic Number: 28 Atomic
Weight: 58.2	7 g/mol Know	vn 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 Weight: 92.9 g/mol		Atomic Number: 41		
		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 Weight: 190.2 g/mol +3, +4, +6, +8		Atomic Number: 76 Known oxidation state(s): +2, Atomic Number: 8	
Oxygen Atomic (rare)	Atomic Symbol: O Weight: 16.0 g/mol	Atomic Number: 8 Known oxidation state(s): -2, +2	
Palladium Atomic	Atomic Symbol: Pd Weight: 106.4 g/mol +4	Atomic Number: 46 Known oxidation state(s): +2,	



PhosphorusAtomic Symbol: PAtomicNumber: 15Atomic Weight: 31.0 g/molKnown 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 P2O5, 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 P4 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 PC13, PC15, POC13, 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.

PlatinumAtomic Symbol: PtAtomic Weight:195.1 g/mol+4+4PlutoniumAtomic Symbol: Pu<br/>Atomic Weight:239.1 g/mol

+4, +5, +6

Atomic Number: 78 Known oxidation state(s): +2,

Atomic Number: 94 Known oxidation state(s): +3, Polonium Atomic Symbol: Po Atomic Weight: 210.0 g/mol +4

Potassium Atomic Symbol: K Atomic Weight: 39.1 g/mol

Praseodymium Atomic Symbol: Pr Atomic Weight: 141.0 g/mol

Promethium Atomic Symbol: Pm Atomic Weight: 146.9 g/mol

Protactinium Atomic Symbol: Pa Atomic Weight: 231.0 g/mol

Radium Atomic Symbol: Ra Atomic Weight: 226.0 g/mol

Radon Atomic Symbol: Rn Atomic Weight: 222.2 g/mol +4, +6 (rare)

RheniumAtomic Symbol: ReAtomic Weight:186.2 g/mol+2, +3, +4 (stable), +5, +6 (stable), +7 (stable)

Rhodium Atomic Symbol: Rh Atomic Weight: 102.9 g/mol

RubidiumAtomic Symbol: RbAtomic Weight:85.5 g/mol

RutheniumAtomic Symbol: RuAtomic Weight:101.1 g/mol+4, +5, +6, +8

Samarium Atomic Symbol: Sm Atomic Weight: 150.4 g/mol

Scandium Atomic Symbol: Sc Atomic Weight: 45.0 g/mol

Selenium Atomic Symbol: Se Atomic Weight: 79.0 g/mol +4, +6 Atomic Number: 84 Known oxidation state(s): +2,

Atomic Number: 19 Known oxidation state(s): +1

Atomic Number: 59 Known oxidation state(s): +3

Atomic Number: 61 Known oxidation state(s): +3

Atomic Number: 91 Known oxidation state(s): +5

Atomic Number: 88 Known oxidation state(s): +2

Atomic Number: 86 Common oxidation statse: +2,

Atomic Number: 75 Known oxidation state(s): +1,

Atomic Number: 45 Known oxidation state(s): +3

Atomic Number: 37 Known oxidation state(s): +1

Atomic Number: 44 Known oxidation state(s): +3,

Atomic Number: 62 Known oxidation state(s): +3

Atomic Number: 21 Known oxidation state(s): +3

Atomic Number: 34 Known oxidation state(s): -2, +2, Silicon Atomic Symbol: Si Atomic Weight: 28.1 g/mol Atomic Number: 14 Known oxidation state(s): -4, +4



Silver Atomic Symbol: Ag Atomic Weight: 107.9 g/mol +2 (rare)

Sodium Atomic Symbol: Na Atomic Weight: 23.0 g/mol

Strontium Atomic Symbol: Sr Atomic Weight: 87.6 g/mol

Sulfur Atomic Symbol: S Atomic Weight: 32.1 g/mol +4, +6

TantalumAtomic Symbol: TaAtomic Weight:181.0 g/mol+3, +5

TechnetiumAtomic Symbol: TcAtomic Weight:99.0 g/mol+5, +6, +7

TelluriumAtomic Symbol: TeAtomic Weight:127.6 g/mol+2, +4, +6

Terbium Atomic Symbol: Tb Atomic Weight: 158.9 g/mol +4 Atomic Number: 47 Known oxidation state(s): +1,

Atomic Number: 11 Known oxidation state(s): +1

Atomic Number: 38 Known oxidation state(s): +2

Atomic Number: 16 Known oxidation state(s): -2, +2,

Atomic Number: 73 Known oxidation state(s): +2,

Atomic Number: 43 Known oxidation state(s): +4,

Atomic Number: 52 Known oxidation state(s): -2,

Atomic Number: 65 Known oxidation state(s): +3, Thallium Atomic Symbol: Tl Atomic Weight: 204.4 g/mol +3

Thorium Atomic Symbol: Th Atomic Weight: 232.0 g/mol

Thulium Atomic Symbol: Tm Atomic Weight: 168.9 g/mol

Tin Atomic Symbol: Sn Atomic Weight: 118.7 g/mol +4

Titanium Atomic Symbol: Ti Atomic Weight: 47.9 g/mol

TungstenAtomic Symbol: WAtomic Weight:183.9 g/mol+4, +5, +6

Uranium Atomic Symbol: U Atomic Weight: 238.0 g/mol +4, +6

Vanadium Atomic Symbol: V Atomic Weight: 50.9 g/mol +3, +4, +5

Xenon Atomic Symbol: Xe Atomic Weight: 131.3 g/mol +4, +6, +8

Ytterbium Atomic Symbol: Yb Atomic Weight: 173.0 g/mol +3

Yttrium Atomic Symbol: Y Atomic Weight: 88.9 g/mol

Zinc Atomic Symbol: Zn Atomic Weight: 65.4 g/mol Atomic Number: 81 Known oxidation state(s): +1,

Atomic Number: 90 Known oxidation state(s): +4

Atomic Number: 69 Known oxidation state(s): +3

Atomic Number: 50 Known oxidation state(s): +2,

Atomic Number: 22 Known oxidation state(s): +3, +4

Atomic Number: 74 Known oxidation state(s): +2,

Atomic Number: 92 Known oxidation state(s): +3,

Atomic Number: 23 Known oxidation state(s): +2,

Atomic Number: 54 Known oxidation state(s): +2,

Atomic Number: 70 Known oxidation state(s): +2,

Atomic Number: 39 Known oxidation state(s): +3

Atomic Number: 30 Known oxidation state(s): +2



A ball of Zinc and turnings produced from it.

Zirconium	Atomic Symbol: 7	Zr
Atomic Weig	ht: 91.2 g/mol	
+3, +4	-	

Atomic Number: 40 Known oxidation state(s): +2,

### 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 **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.

 $Cu^{2+}_{(aq)} \Rightarrow Cu_{(s)}$ 

### <u>Filtrate</u>

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.

### <u>Catalyst</u>

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<sub>3</sub> 169.87 grams 1 mol HCl 1 mol AgNO<sub>3</sub>

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 twocomponents, 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.

### 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<sub>2</sub>) can also become supercritical but at 7,38MPa and 31.1°C. All liquids c an become supercritical at the right pressure and temperature. Supercritical liquids/gases are used in the industry as high-efficiency solvents. E.g. Supercritical CO<sub>2</sub> 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) <sub>2</sub>	6.7 x 10 <sup>-4</sup> g/100ml	Calcium Sulfate	CaSO <sub>4</sub> or CaSO <sub>4</sub> * 2H <sub>2</sub> O	.241 g/100ml
Barium Sulfate	BaSO <sub>4</sub>	2.22 x 10 <sup>-4</sup> g/100ml	Silver Chloride	AgCl	8.9 x 10 <sup>-5</sup> g/100ml
Silver Bromide	AgBr	8.4 x 10 <sup>-6</sup> g/100ml	Silver Iodide	AgI	3 x 10 <sup>-7</sup> g/100ml
Magnesium Hydroxide (AS)	Mg(OH) <sub>2</sub>	9.0 x 10 <sup>-4</sup> g/100ml	Aluminum Fluoride	AlF <sub>3</sub> or AlF <sub>3</sub> *3H <sub>2</sub> O	
Barium Carbonate (AS)	BaCO <sub>3</sub>	2.0 x 10 <sup>-3</sup> g/100ml	Barium Chromate (AS)	BaCrO <sub>4</sub>	3.4 x 10 <sup>-4</sup> g/100ml
Barium Citrate	$\begin{array}{c} Ba_{3} \\ (C_{6}H_{5}O_{7})_{2} \\ *7H_{2}O \end{array}$	4.06 x 10 <sup>-2</sup> g/100ml	Barium Oxylate (AS)	Ba(COO) <sub>2</sub>	9.3 x 10 <sup>-3</sup> g/100ml
Barium Phosphate (AS)	$Ba_3(PO_4)_2$		Barium Sulfite	BaSO <sub>3</sub>	$\begin{array}{c} 2.0 \text{ x } 10^{-2} \\ \text{g} / 100 \text{ml} \end{array}$
Bismuth Hydroxide (AS)	Bi(OH) <sub>3</sub>	1.4 x 10 <sup>-4</sup> g/100ml	Cadmium Carbonate (AS)	CdCO <sub>3</sub>	
Camium Hydroxide (AS)	Cd(OH) <sub>2</sub>	2.6 x 10 <sup>-4</sup> g/100ml	Cadmium Oxalate (AS)	$Cd(COO)_2$	$3.3 \times 10^{-3}$ g/100ml
Calcium Carbonate (AS)	CaCO <sub>3</sub>	1.45 x 10 <sup>-3</sup> g/100ml	Calcium Fluoride	CaF <sub>2</sub>	1.6 x 10 <sup>-3</sup> g/100ml
Calcium Hydroxide (AS)	Ca(OH) <sub>2</sub>	1.8 x 10 <sup>-1</sup> g/100ml	Calcium Phosphate (AS)	$Ca_3(PO_4)_2$	$2.5 \times 10^{-3}$ g/100ml
Calcium Metasilicate (AS)	CaSiO <sub>3</sub>	9.5 x 10 <sup>-3</sup> g/100ml	Cesium Aluminum Sulfate	CsAl(SO <sub>4</sub> ) <sub>2</sub> * 12H <sub>2</sub> O	3.4 x 10 <sup>-1</sup> g/100ml
Cobalt (II) Carbonate (AS)	CoCO <sub>3</sub>		Cobalt (II) Chromate (AS) (OxA)	CoCrO <sub>4</sub>	
Cobalt (II) and (III) hydroxide (AS)	Co(OH) <sub>2</sub> & Co(OH) <sub>3</sub>		Copper (I) & (II) Carbonate (AS) (Am)	Cu <sub>2</sub> CO <sub>3</sub> & CuCO <sub>3</sub>	
Copper (I) Halides (AS) (Am)	CuX		Copper (I) & (II) Hydroxide (Am)	CuOH & Cu(OH) <sub>2</sub>	
Lead Phosphate (AS) (BS) (OxA)	$Pb_3(PO_4)_2$	1.4 x 10 <sup>-5</sup> g/100ml	Mercury (I) Chloride	Hg <sub>2</sub> Cl <sub>2</sub>	2.1 x 10 <sup>-4</sup> g/100ml

Lead (II) Chloride	PbCl <sub>2</sub>	6.73 x	Mercury (II)	HgS	1.0 x 10 <sup>-6</sup>
		$10^{-1}$	Sulfide		g/100ml
		g/100ml			
Lead (II) Sulfide	PbS	$1.2 \ge 10^{-2}$	Copper (I)	Cu <sub>2</sub> S	
(OxA) (AS)		g/100ml	Sulfide (OxA)		
			(Am)		
Copper (II)	CuS	$3.3 \ge 10^{-5}$	Cadmium (II)	CdS	
Sulfide (OxA)		g/100ml	Sulfide (OxA)		
(Am)					
Arsenic Sulfide	$As_2S_3$		Antimony	$Sb_2S_3$	
(OxA)			Sulfide (OxA)		
Tin (IV) Sulfide	$SnS_2$		Aluminum	Al(OH) <sub>3</sub>	
(OxA)			Hydroxide (AS)		
			(BS)		
Iron (II) Sulfide	FeS		Manganese (II)	MnS	
(UXA)			Sultide		
Zinc Sulfide	ZnS		Nickel (II)	N1S	
(OxA)	~ ~		Sulfide (OxA)		
Cobalt (II) Sulfide	CoS		Strontium	$Sr_3(PO_4)_2$	
(OxA)			Phosphate		
Zinc Hydroxide	$Zn(OH)_2$		Chromium (III)	$Cr(OH)_3$	
(AS) (BS)			(BS)		
Iron (III)	Fe(OH) <sub>3</sub>		Copper (II)	Cu(COO) <sub>2</sub>	$2.5 \times 10^{-3}$
Hydroxide			Oxylate (Am)		g/100ml
Bismuth (III)	Bi <sub>2</sub> S <sub>3</sub>		Gold Sulfide	$Au_2S$	
Sulfide (OxA)			(OxA)		
Iron Ferricyanide	Fe <sub>3</sub>		Lead (II)	PbBr <sub>2</sub>	$4.5 \times 10^{-1}$
	$[Fe(CN)_6]_2$		Bromide (AS)		g/100ml
Lead (II)	PbCrO <sub>4</sub>	5.8 x 10 <sup>-6</sup>	Lead (II)	PbCO <sub>3</sub>	$1.1 \times 10^{-4}$
Chromate (BS)		g/100 ml	Carbonate (BS)		g/100ml
(AS)		2	(AS)		
Lead (II)	$Pb(OH)_2$	$1.5 \ge 10^{-2}$	Lead (IV) Oxide	PbO <sub>2</sub>	
Hydroxide (BS)		g/100ml	(AS)		
(AS)					
Lead (II) Oxylate	$Pb(COO)_2$	$1.6 \ge 10^{-4}$	Lead (II) Sulfate	PbSO <sub>4</sub>	4.25 x
		g/100ml			10-3
	11.00				g/100ml
Magnesium	MgCO <sub>3</sub>		Magnesium	MgF <sub>2</sub>	$7.6 \times 10^{-3}$
Carbonate (AS)		<b>–</b> 0 10- <sup>2</sup>	Fluoride (OxA)		g/100ml
Magnesium	$Mg(COO)_2$	$7.0 \times 10^{-2}$	Magnesium	$Mg_3(PO_4)_2$	$2.0 \times 10^{-2}$
Oxylate (BS) (AS)		g/100ml	Phosphate (AS)		g/100ml
Manganese (II)	$MnF_2$		Manganese (II)	$Mn(OH)_2$	$2.0 \times 10^{-3}$
Fluoride (AS)			Hydroxide (AS)		g/100ml
Manganese (II)	$Mn(COO)_2$		Mercury (I) (AS)	$Hg_2Br_2 \&$	
Oxylate (AS)			and (II) Bromide	HgBr <sub>2</sub>	

Mercury (I) and	Hg <sub>2</sub> CO <sub>3</sub> &		Mercury (II)	$Hg_3(PO_4)_2$	
(II) Carbonate	HgCO <sub>3</sub>		Phosphate (AS)		
(AS)					
Molybdenum (II)	MoBr <sub>2</sub> &		Molybdenum (II)	MoCl <sub>2</sub> &	
and (III) Bromide	MoBr <sub>3</sub>		and (III) Chloride	MoCl <sub>3</sub>	
Molybdenum	$Mo_2S_3$ and		Nickel (II)	NiCO <sub>3</sub>	$9.3 \times 10^{-3}$
Sulfides	$MoS_2$		Carbonate (AS)		g/100ml
Nickel (II)	NiF <sub>2</sub>	$2.0 \times 10^{-2}$	Nickel (II)	Ni(OH) <sub>2</sub>	$1.3 \times 10^{-3}$
Fluoride		g/100ml	Hydroxide (AS)		g/100ml
			(Am)		
Nickel (II)	Ni(COO) <sub>2</sub>		Nickel (II)	$Ni_3(PO_4)_2$	
Oxylate (AS)			Phosphate (AS)		
Potassium	KClO <sub>4</sub>	7.5 x 10 <sup>-1</sup>	Silver Carbonate	Ag <sub>2</sub> CO <sub>3</sub>	$3.2 \times 10^{-3}$
Perchlorate			(Am)		g/100ml
Silver Oxide (AS)	Ag <sub>2</sub> O	$1.3 \times 10^{-3}$	Silver Phosphate	Ag <sub>3</sub> PO <sub>4</sub>	$6.5 \times 10^{-4}$
	_	g/100ml	(Am) (AS)	_	g/100ml
Silver Sulfate	$Ag_2SO_4$	5.7 x 10 <sup>-1</sup>	Tin Phosphate	$Sn_3(PO_4)_2$	
(Am) (AS)		g/100ml			
Zinc Carbonate	ZnCO <sub>3</sub>	$1.0 \ge 10^{-3}$	Zinc Cyanide	$Zn(CN)_2$	$5.0 \times 10^{-4}$
(AS) (BS)		g/100ml	(BS)		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 aluminagrog, 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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- Suggestions regarding topics were provided by 'Magpie (from USA)'
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