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 \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 1 documents, however directly linking to the html document is possible, still though I would prefer links be to the ma project page.

5.0 Simple Reactions

Types of reactions:

1) Single Displacement: $A + BC \mathbf{P} AC + B$

Example: $2HCl(aq) + 2Zn(s) \blacktriangleright H2(g) + ZnCl2$

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

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

3) Combination Reaction: A + B **Þ** AB

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

4) Combustion Reaction:

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

5) Decomposition: AB \mathbf{P} A + B

Example: $(COO)_2 Fe_{(s)} \blacktriangleright 2CO_{2(g)} + Fe_{(s)}$

6) Acid-Base Reaction:

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

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

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

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

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

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

 $NaOH(s) - H2O \rightarrow Na+(aq) + OH-(aq)$

 $HCl(g) - H2O \rightarrow H+(aq) + Cl-(aq)$

5.2 Basic Chemistry Experiments [With Step By Step Explanations]

5.3 Basic Chemistry reactions [Simplified Explanations]

5.3a Preparation of Sodium Acetate

$CH_3COOH_{(aq)} + NaOH_{(aq)}$ **D** $CH_3COONa_{(aq)} + H_2O_{(1)}$

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

5.3b Preparation of Zinc Hyposulfite

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

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

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

5.3c Preparation of Chloroform

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

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

.75 mol of NaOCl (880 ml 6% NaOCl, 510 ml 10% NaOCl, 375 ml 12.5% NaOCl) is placed into a reaction vessel capable of holding it's volume plus 100 ml, the vessel if plastic will most likely suffer some attack, therefore glass is preferred. If possible the NaOCl is chilled prior to reaction to 0 °C or ice cubes are added, not many though as chloroform is slightly soluble in water and extra ice will decrease yield. To this chilled NaOCl is added 11 ml (slightly less then .25 mol) of acetone if possible the reaction mixture is kept cool in an ice bath, without the initial chilling and continued cooling the formed chloroform and reacting acetone will volatize off due to the increasing temperature although you will still get a product. The reaction is allowed to proceed for 25-50 minutes with agitation during the addition and at least once during the reaction. The induction period is 1-3 minutes if the mixture is pre-cooled but if it is warm it can be as short as 5 seconds. Finally after the reaction has been allowed to run to completion the top layer of the reaction liquid is poured off, this contains a mixture of sodium acetate which is benign, sodium hydroxide which also poses no hazard, but it does contain some partially chlorinated acetone and dissolved chloroform, do not dispose of in a manner that would allow it to run into rivers or into the water supply. The chloroform will have settled to the bottom in a blob that is more or less insoluble in water. This can be sucked out with a pipette or poured into a tall thin vessel where the water layer and chloroform layer become more distinct and separated. Your impure chloroform contains traces of carbon tetrachloride, water, and acetone. It can be purified further by shaking with a small quantity of water saturated with NaCl, dried in contact with H2SO4, and distilled. Yield will be based on the concentration of hypochlorite used and the temperature of the reaction mixture but should always be greater then 55%. In a series of trials using 10% hypochlorite the average yield of chloroform was consistently around 70% (9 ml),

accounting for solvation of chloroform (.8 g / 100 ml) yields approached 100% [98.5 being the highest].

5.3d Preparation of Benzoic Acid

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

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

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



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



solubility and some NaCl will come out.]

The resulting benzoic acid is placed onto a paper towel with the first batch to let it dry. It takes about three days at room temperature in a normal humidity to dry the

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