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Synthesis of alum lab answers

Target Background procedure Experimental techniques: gravitational filtration: plain filtration using filter paper in a funnel to separate solids from a liquid allowed to flow freely (under gravity) through funnel vacuum filtration: filtration using filter paper in a specially designed funnel (Büchner funnel) to separate solids from a liquid drawn through a funnel during vacuum recrystal insulation and purification of a solid by causing it to trigger slowly from solution Theoretical concepts: theoretical yield: amount of product expected based on the amount of limiting reagent used percentage yield: amount of product actually recovered expressed as a percentage of the theoretical yield Formula for alum is $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$. Calculate yields: mol Al1 mol alum474.32 g alum Theoretical yield = gram Al used $x \frac{\text{mol alum}}{\text{mol Al}} \times 100\% = \text{g alum}$ 26.98 g Al1 mol Almol alum actual yield, g% yield = $\frac{\text{actual yield}}{\text{theoretical yield}} \times 100\%$ Procedure Note: the following is not a procedural control list: contained in the lab handout. This is a brief summary of this procedure, including tips, suggestions, and deviations from the procedure described in the handout. It is not a substitute for reading the procedure in the handout before you get to the lab. The synthesis will be performed in the following stages: Work in pairs. Aluminium foil solution: $2 \text{ Al(s)} + 2 \text{ OH}^-(\text{aq}) + 6 \text{ H}_2\text{O} \rightarrow 2 [\text{Al}(\text{OH})_4]^-(\text{aq}) + 3 \text{ H}_2(\text{g})$ Weigh 0.45-0.50 g Al foil, using the tare function on the balance and do not allow the foil to come into contact with your hands or balance pan. Pick up the mass to +/- 0.01 g. Place the foil in a reaction cup marked with your initials. During the smoke cap, 13.0 ml of 3.0 M koh is poured into the reaction cup. Use only the glass part of the pipe rod to wet Al. Use the rubber cop to push back to the solution any Al that sticks to the side of the cup. On the data sheet, describe the reaction mixture after all Al dissolves. Gravitational filtering of the reaction mixture Place a funnel in the funnel holder and place a marked beaker under the funnel. Fold a 7 cm piece of filter paper as described in the labutdeling sheet. Moisten the filter paper with 2-3 ml of distilled water to make it stick to the funnel and discard this water. Transfer the reaction mixture to the filter paper in the funnel. Rinse the reaction cup twice with 2 ml of distilled water and add each rinse to the filter paper. Collect and save this floating filtrate. Rinse the residue on the filter paper with 2-3 ml of distilled water and join this rinse until the filtrate. Once all the liquid has been emptied from the filter paper, dispose of the paper and contents of the correct waste container. Completing the formation of alum: Slowly and gently stir 22.5 ml 4 M H_2SO_4 (aq) into the filtrate in 5 ml servings. Take into account and record changes to the filtrate. Using a hob, heat the acidic filtrate until all solids dissolve. Reaction steps involving sulphuric acid: $\text{Al}(\text{OH})_4^-(\text{aq}) + \text{H}_2\text{SO}_4(\text{aq}) \rightarrow \text{Al}(\text{OH})_3(\text{s}) + \text{HSO}_4^-(\text{aq}) + \text{H}_2\text{O}$ $\text{Al}(\text{OH})_3(\text{s}) + 3 \text{ H}_2\text{SO}_4(\text{aq}) \rightarrow \text{Al}^{3+}(\text{aq}) + 3 \text{ HSO}_4^-(\text{aq}) + 3 \text{ H}_2\text{O}$ The general reaction to form alum is: $\text{Al}^{3+}(\text{aq}) + \text{K}^+(\text{aq}) + 2 \text{ SO}_4^{2-}(\text{aq}) + 12 \text{ H}_2\text{O} \rightarrow \text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}(\text{s})$ Crystallization Use pliers, remove the filtrate cup of a thread of gauze on the laboratory bench. Let it start to cool while preparing an ice bath, an ice water mixture in a 400 ml cup. Place the filtrat cup in the ice bath, making sure that no water from the ice bath enters the filtra cup. Place 3 ml of 95% ethanol in a test tube, and put the tube in another ice bath, secured with a test tube clamp. Let the filtrat cup sit undisturbed in the ice bath until crystals of the alum are observed. If no crystals have formed after 30 minutes, contact the teacher. Vacuum filtration While the solution is cooling, set up a filtration unit. Note: You will not use the second taped filter flask shown as a trap in the labutdeling sheet. Determine the mass of a 7 cm circular filter paper. Then place the paper flat in the Buchner funnel so that it covers all holes. Moisten the paper. Then connect the filter flask to a water aspirator with rubber tubes. Test the assembly by turning on the aspirator. When crystals stop forming in the cooling solution, ask the teacher to initialize the data sheet, giving approval to start filtering the alum. Let water flow through the aspirator to establish a vacuum in the filter flask. Only then does it begin to transfer the supernatural liquid from the beaker to the Buchner funnel. Use a rubber cop to transfer all the crystals to the Buchner funnel. Rinse the crystals with 1-2 ml of cooled ethanol. Stop filtering when liquid no longer drips from the funnel and into the flask. Do not use the water aspirators to dry the crystals. (Excessive use of the aspirators causes flooding in the science building.) Disconnect the hose from the aspirator and then turn off the water. Place the crystals and filter paper in a weighed cup and store them in the cabinet for the next laboratory period. You won't turn into lab reports until the next lab period, when you want to weigh your dry crystals and complete the data sheet. You will be well advised, though, to complete all parts of the laboratory report that can be completed. Don't forget to bring lab handouts back to the next lab period, because the lab report is due in the next lab period. Back to Chemical Principles Lab Schedule. Authors D. L. McCurdy, V.M. Pultz and J.M. McCormick* Last Update: 21. As we have discussed in class, there are rules, such as the Law of Conservation of Mass, where chemical reactions occur, and it took a long time to understand these basic rules. Although we know a lot about chemical reactions, chemists still find new chemical reactions and new ways to assemble atoms in molecules and molecules in more elaborate structures. In this and the next laboratory exercise, you learn some of the basics of how chemists perform chemical reactions and how they characterize the chemical substances involved in these reactions. To fully describe a chemical reaction, one must know the identity of both the products and the reactants, and the proportions in which the reactants are combined and the products are formed. Although it may seem a trivial exercise to identify the reactants, this is not always the case. Needless to say, identifying the reactants in a complex reaction mixture can be very difficult, which is why we will only work with chemical reactions where the reactants are known. The description of a chemical reaction consists of a number of steps: 1) that perform the reaction, 2) isolate the product(s), 3) cleans the product(s), 4) and characterizes the product(s) and determines its purity. Isolation and purification of the products is based on physical properties such as the ability to form crystals, boiling point, melting point, solubility, etc. Characterization of the products can be either quantitative or qualitative. In quantitative characterization, the chemical formula and structure (that is, how the atoms are connected) are determined. The former is usually achieved with the help of element analysis, mass spectroscopy, X-ray crystallography or any spectroscopic method. Sometimes it is sufficient to show only that certain ions or elements are present in a sample, and in this case, a chemist will perform a qualitative test. Qualitative tests often use chemical reactions that result in a visible change (formation of an insoluble solid, a color change or development of a gas) as a way to quickly show whether a particular chemical species is present or not. When the chemical reaction's products are fully characterized, and the balanced chemical reaction is known, we can calculate a theoretical and one percentage yield. We make these final characterizations of the reaction because it is important to know how effectively the reaction converts reactants into products. Chemists always try to strike a balance between the cost of the reactants, the value of the products, the time a reaction requires and the cost of unwanted by-products that need to be handled as hazardous waste. A reaction, even if it provides a valuable product, can be useless because it has low yields, takes too much time or generates too much waste. In this experiment, you will prepare and characterize (potassium aluminium sulfate dodecahydrate, $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$). The first step in this synthesis, which you will perform in week 1, is to react metallic aluminium with a concentrated solution of potassium hydroxide (KOH) to form the potassium salt in tetrahydroxaluminate skin color, $[\text{Al}(\text{OH})_4]^-$. The balanced chemical equation for this oxidation reduction reaction is the second step in the procedure is to convert $\text{KAl}(\text{OH})_4$ to alum when adding sulfuric acid (H_2SO_4) in an acid base reaction. Under experimental conditions, the alum has limited solubility in water, and then it is triggered from the solution. The balanced chemical reaction that occurs in this step is the General Balanced Chemical Reaction for converting aluminum to alum, shown below, can be achieved by adding together the balanced chemical equation for each step (Help me). The second and third weeks of this exercise will be devoted to characterizing the alum. Alum is an ionic compound, which means that the melting and boiling points are likely to be too high to be measured conveniently. More, most spectroscopic methods would not provide useful information. Therefore, we will rely on chemical agents to show that we actually formed alum in our reaction. This procedure duplicates how chemists characterized chemical reactions to the late 20th century, and in some cases, chemical agents for characterization are still the only methods available. In week 2 you will perform qualitative tests to demonstrate the presence of K^+ , and sulfation (SO_4^{2-}) in the alum. You will also make a quantitative decision to determine the percentage water at mass in the alum. The qualitative test for sulfate uses the insolubility of barium sulfate (BaSO_4). When an aqueous solution of a barium salt (usually BaCl_2) is mixed with an aqueous solution containing sulfate, a white bottom outlet of insoluble BaSO_4 is formed according to the net ionic equation: $\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})$. A positive test for SO_4^{2-} is therefore observation of a white bottom shot when an aqueous BaCl_2 solution is mixed with the aqueous test solution. When placed in a flame, many elements give the flame a distinctive color, an effect that can be used to determine both which elements, and how much of each individual, can be found in a sample. Potassium produces a distinctive lavender flame that we can use as a qualitative test for the presence of potassium. Potassium flame is often difficult to see because sodium, which is often present as impurity, has an intense yellow flame that masks other colors. The potassium flame can be seen in the presence of sodium by seeing the flame through a dark blue cobalt glass filter, which absorbs the yellow light from Na, but allows the light from K to pass. When placed in a flame, aluminium does not change the color of the flame and a visual flame test cannot therefore be used to The presence of Al. Alum is a hydrate, which means that there is a compound that has water molecules trapped in the solid. Hydrates will release some, or all, of their water of hydration upon heating. If the chemical reaction between Al and KOH produces alum as a product, we expect heating of the product to result in a decrease in the sample weight corresponding to the loss of 12 water molecules per formula unit of alum. Thus, if one knows the starting mass of the alum, and the amount (mass, and therefore the number of moles) of anhydrous alum left after all the water is powered by, one can calculate the amount of water that was present in the alum (by the law of preservation of mass). A comparison of the experimentally specific water of hydration and the number expected from the chemical formula can then be used as evidence for the formation of the desired product. The process by which the water of hydration is driven is described by the chemical equation shown below, where Δ written above the arrow indicates that the heat was applied to the reactant(s). A quantitative analysis for Al^{3+} will be done in Week 3. Normally, Al^{3+} is colorless, which means that it does not absorb light in the visible part of the spectrum. So we will add a dye called aluminone that will react with Al^{3+} in the solution to provide a colored complex ion. For a sufficiently diluted solution, the amount of light absorbed by a chromophoria (a chemical species that absorbs light) present in the solution provided by the Beer Act, $A = \epsilon \cdot b \cdot C$, where A is the absorbent (how much light the sample absorbs relative to a solution that does not contain chromophoria), ϵ is molar absorptivity (also known as an extinction coefficient: ϵ depends on the compound and wavelength of light), b is path (how much sample light must pass through) and C is the concentration of chromophoria (More info). According to Beer's Law the intensity of color depends almost on the amount of aluminon- Al^{3+} complex present. So if we knew ϵ for the complex ion formed between Al^{3+} and aluminone, we could do a single absorbance measurement and know $[\text{Al}^{3+}]$ in a solution, and therefore how much Al was in the original alum sample. Unfortunately, this is neither a precise nor accurate way to make this decision. It is imprecise because it is only a single measurement and it is inaccurate because 1) we do not know stoichiometry for the reaction between aluminone and Al^{3+} and 2) commercially available dye is not pure (ϵ can not be determined). So we need a way to increase the precision of the method and to overcome the problem with accuracy. The problems with the colorimetric method are solved using a calibration curve, giving the ratio of absorbance to concentration. A calibration curve constructed by preparing samples with known concentrations of the analyte (in our case Al^{3+}) and then measuring the absorbance of these samples. If the Beer Act holds, a calibration curve is a straight line, as we can get an equation from a regression analysis. Now, if we measure the absorbance of a sample containing an unknown amount of analyt, it becomes a simple matter of replacing this value in the equation for our calibration curve and solving for the concentration. Because more than one measurement was used to construct the calibration curve, we improve our precision. A calibration curve also improves accuracy because only the concentration of analytes changes (everything else, such as stoichiometry between aluminone and Al^{3+} and the purity of the dye, is constant). When you set up the portable lab notebook for this training treat each week of the exercise as a separate experiment. So, every week will have its own title, declaration of purpose, etc. Please note that some of your results will actually be determined within a subsequent week. Be sure to read the experimental procedure carefully and be aware that there are a number of potential dangers. There are also several places in this exercise where you will wait for something to happen. You can significantly shorten your time in the lab by working on another part of that week's training these times. Also, make sure you have completed all the calculations for a given week's work before you get to the lab. If you do not get to the laboratory prepared, you will not be able to complete week 2 and week 3 exercises in the allotted time. Experimental week 1 (More info) Synthesis of potassium aluminium sulfate dodecahydrate Get a piece of aluminium foil weighing about 0.5 g and weighing it accurately (to the nearest 0.001g). Cut the weighed foil into many small pieces. The smaller the pieces the faster the reaction will go due to the increased surface area exposed to the KOH solution. Put the small pieces of aluminium in a 100 ml cup. Add enough hot water to a Styrofoam cup so that when 100 ml of cup is placed inside, the cup is completely surrounded by water, but the water does not spill out of the cup or into the cup. If water from the hot water bath spills into the beaker, there will be a drastic increase in the yield of the alum. Place 100 ml of cup containing the aluminium in warm water in the Styrofoam cup and transfer everything to the hood. Slowly and gently add 25 ml of the 1.4 M KOH solution to the aluminium. Caution! No open flames can be present in the laboratory while the reaction between KOH and Al takes place. Stir the solution with the glass pipe rod and cover it with a bell glass. Repeat the stirring for a few minutes until the entire aluminium dissolves. If the reaction slows down, replace the water in the bath with fresh warm if the reaction becomes too powerful, remove the beaker from the water bath until the reaction subsides. Caution! Avoid inhaling the gas developed during this reaction. The gas is not toxic at this concentration, but a fine fog of the corrosive KOH solution is formed by the gas evolution. When the aluminium is completely dissolved (do not worry if the solution appears cloudy or contains black spots), gravity filters the reaction mixture into a 50 ml beaker through fluted filter paper (the instructor will demonstrate). Dispose of the used filter paper in the laboratory trash. Caution! The filter paper will be wet with the corrosive KOH solution. So wash your hands after handling the wet filter paper. Get approx. 5 ml 9 M H_2SO_4 in the 10 ml graduated cylinder. Use a plastic pipe to slowly and gently add the H_2SO_4 solution to 50 ml beaker containing the filtered solution. Do not immerse the pipette in the filtered solution! Continue adding the H_2SO_4 solution until no more precipitation forms are triggered. This should require no more than about 5 ml of the H_2SO_4 solution. Do not add too much H_2SO_4 , otherwise the dividend will suffer (More info). After the H_2SO_4 add-on, gently touch the new mixture with the stirring rod and record your observations. Caution! The H_2SO_4 solution is highly corrosive and the reaction between H_2SO_4 and KOH is very exothermic (denies heat). Prepare a cold bath. Place the 50 ml beaker with the filtered reaction solution in the ice bath. Do not induce any of the water from the ice bath in the cup. Also place a ing tube containing 15 ml of 95% ethanol in the ice bath. The ethanol solution will be used to wash the remaining H_2SO_4 from the alumcluses crystals. After a crop of crystals has formed, set up a vacuum filtration device (Help me). Under no circumstances do the rubber hose push

more than 1/4 on the side arm of the filter flask and do not leave the hose attached to the flask while the flask is advertised. While the vacuum is on, carefully remove some of the supernatant (the solution over a solid) from your crystals using a pipette and wet filter paper. This will help the paper stick to the filter and prevent leaks.

Caution! The solution is corrosive. Remove 50 ml of cup from the ice bath, gently swirl it to suspend the crystals and pour it into the Büchner funnel. Use the glass tube rod to remove any crystals that stick to the side of the beaker. When the aqueous solution is filtered completely (leaving the crystals on the filter paper), place 2 – 3 ml (the plastic pipettes hold about 3 ml) of the cold ethanol solution in 50 ml cups. Use the glass tube rod to loosen any remaining solids that cling to the side. Swirl to suspend any crystals left in the beaker, and pour the suspension into the filter. Once the ethanol is filtered away, repeat the repeatedly. After the last ethanol wash, let the vacuum run for a minute or two to draw air through the crystals to help them dry. After the ethanol solution has stopped draining out of the funnel, inspect the product. If it looks dry, gently prod it with metal spatula. If it is dry enough to remove from the filter, the fasting will not be very sticky and will have the consistency of fine sand. Break the vacuum by removing the vacuum hose from the side arm of the filter flask, then switch off the aspirator. Transfer fixed and filter paper from the funnel to a pre-weighing glass using the metal spatula, which the teacher will demonstrate. Carefully scrape up an alum that sticks to the side of the Büchner funnel on the bell glass. If the alum is dry, the filter paper will differ from the crystals, and you can remove the filter paper. Carefully scrape any crystals that hold the filter paper on the watch glass. If the alum is still too wet, leave the filter paper and remove it next week. Get the mass of the wet alum. You need to have about 2 g of wet alum (3 g if the mass contains the filter paper) so that you get enough for the next two weeks. If you do not have enough, collect the second crop of crystals and / or redo the synthesis. Keep the crystals from different crops and syntheses separate. Cover the container that holds the crystals with a piece of paper towel and place it in the tray to dry. You may notice that several crystals formed in the filter flask under the sink. This second crop of crystals can also be collected, but if you choose to collect these crystals, they should be kept separate from the main crop. It is always good laboratory practice to keep different crops of crystals separate to the identity and purity of each crop determined (other crops almost always contain more impurities than the first crop and the time needed to clean them sometimes outweighs the additional yield). Collect the second crystal crop by vacuum filtration; wash with several small portions of the cold ethanol solution and dry, as described above. Week 2 Before doing anything else in the lab, get the mass of each crop of alum to the nearest milligram (three decimal places). Make observations on the crystalline product (color, texture, etc.), and record all your observations in your lab notebook. Share your results with your classmates. Qualitative chemical tests Perform the following qualitative tests for SO42 and potassium on the sample. If you collected another crop of alumcrystals, you should perform sulfate and potassium quality tests on both first and second crops (are your two crops qualitatively the same?). Sulfate Test Place some crystals of alum in a 6-test tube. Add distilled water droplets while stirring until the alum dissolves. Add 0.5 M BaCl2 (barium chloride). Record your results. Does alum contain sulfate? Potassium flamemetest The instructor will demonstrate the correct techniques for using the Bunsen burner and heating the needle. In the hood, heat the supplied needle in the flame to remove impurities. When the needle is clean, gently scoop up a small amount of alum at the end of the hot needle. Place the alum in the flame and heat it until the crystals start to melt and the solid glows. Note the color of the flame. If the flame is light yellow (indicating the presence of sodium), try cleaning the needle again, or use the cobalt glass filter. Does this sample contain potassium? Quantitative determination of water by hydration Before you begin this section, make sure that the alum test is pulverized and that you have weighed your alum specimen! Set up a ring rack, ring clamp and porcelain triangle, which the teacher will demonstrate. Clean the melting pot by placing a few drops of 1 M NH3 solution in the empty melting pot and scrub with a paper towel. Caution! This ammonia solution has a strong smell and is corrosive. Rinse the melting pot with distilled water and place the empty melting pot on the porcelain triangle supported by a ring and ring rack. With the majority of the flame left under the bottom of the crucible, heat the melting pot to the bottom lights a dull red. After heating for five minutes, remove the flame and let the melting pot cool to room temperature on the triangle. Caution! Do not touch the crucible with your hand. It is extremely hot and will remain warm for several minutes. Remember that a hot melting pot looks exactly the same as a cool melting pot. Once cooled, you can move the melting pot to the countertop using the crucibles. Do not set a hot melting pot on the countertop, because the temperature difference can cause the melting pot to break. Once you have cleaned the melting pot, it is important that you handle it only with the crucibles. This prevents burns and will eliminate a systematic defect caused by the weight of your fingerprints. Route the cooled melting pot (and cover) to the nearest milligram (three decimal places) and register this mass in the notebook. If the balance does not show three decimal places, you must notify the teacher. Place approx. 1.0 g of the alum specimen in a melting pot. Get the mass of the melting pot, cover and alum to the nearest mg and record this in your notebook. Return the melting pot to the porcelain triangle and place the cover slightly on the mullet so that the water vapor can escape. For the first few minutes, gently heat (only the light blue part of the flame touches the melting water) melting pot by keeping the Bunsen burner off to the side. take care of yourself! The water can violently leave the alum at this point, if it is heated too strongly. Move the Bunsen burner so that the tip of the inner blue cone is approx. 3 under the crucible. Heat until melting pot glows red and continue warming for 10 minutes. If at any time you observe that white smoke is given off, or smell an acrid smell, you must cancel the heating immediately (the sulphate breaks down to SO2). Remove the heat and cover the melting pots completely with a lid. Cool the melting pot to room temperature on the triangle (this takes about ten minutes). Route the cooled melting pot (including the cover and contents) to the nearest milligram (three decimal places). Use the pliers to move the melting pot and contents back to the triangle and repeat the heat step for 10 minutes. When this heat step is over, cover the melting pot and let it cool on the triangle to room temperature, then reweigh the melting pot, cover and its contents. Record this second mass in your notebook. If the second mass is within a 50 mg of mass after the first heating, you have run off all the water. If the masses are not within 50 mg, repeat the heating procedure until two consecutive masses agree. Once you have done your final weighing, the inverter melting pot and anhydrous alum should fall out. If it does not, add a little water from a splash bottle and use the metal spatula gently to loosen it. The anhydrous alum can be thrown into the trash or in the sink with plenty of water. Rinse the melting pot with distilled water and dry it before returning it to the tray. Week 3 Before you reach the laboratory, you must have completed the following: 1) prepare a table, for example, Table 1, in the Notebook Results section where you can write your data for the calibration curve, 2) set up the calculations to calculate [Al3+] in Table 1 (the number of significant numbers in each volume is given in Table 1 and in Table 2), 3) prepare an Excel worksheet to graph the calibration curve (save to Y : drive or flash drive), and 4) familiarize yourself with the instrument before the lab; the teacher will undergo spectrometer operations before you start working (click here for instructions for use). Solution numberVolume of Al3+ storage solution Used (ml)Final volume of solution (ml)[Al3+] (M)Absorbance at 525nm10.0050.0021.0050.0032.0050.0043.0050.0055.0050.00Table 1. Example of a table that can be used to present the data for the calibration curve. The teacher will demonstrate how to create solutions using volumetric glass and will review the protocols for using the balances. Colorimetric determination of aluminium2.3 Preparation of the aluminium storing solution Weighs accurately (to the nearest milligram) approximately 0.1 g AlCl3·6H2O using an analytical balance. Quantitatively transfer this solidly to a 100 ml volumetric flask (assume the flask volume is 100.0 ml). Add about 10 ml of distilled water and swirl to dissolve AlCl3·6H2O. If the solid fasting does not dissolve, carefully add small distilled water, swirling between each addition, until it does so. Add distilled water to bring the level of solution in the flask to the mark on the neck (this procedure is called dilute to the mark). Mix well by stopping the flask, then turn and shake the flask. Repeat if necessary. Pipet 3.00 ml of the aluminum solution that you have just made into a 25 ml volumetric flask. Dilute to the mark and mix well. This is the aluminum store solution you want to use to construct the calibration basket. Construction of the calibration basket Number five 50 ml volumetric bottles 1 to 5. Do not add any aluminium storing solution to flask 1. To flask 2 add 1.00 ml of the aluminum stocking solution; to flask 3 add 2.00 ml; add 3.00 ml in flask 4 and 5.00 ml to flask 5. These measurements must be precise, which is why you need to use volumetric pipettes. To each flask then add 20 ml of acetate buffer solution and 5 ml of aluminone solution (in that order) and swirl gently to mix. These volume measurements do not have to be very precise. So you can use your 50 ml and 10 ml graduated cylinders here. Dilute all the solutions to the mark by adding distilled water and mixing well. Leave the solutions for 20 minutes while monitoring the colors of the solutions. Note any changes to your notebook. Follow the spectrometer's instructions for use to prepare the appliance for use. Fill cuvette with the buffer solution to be used as an empty (IMPORTANT! you need to use the same cuvette for both blank and for the samples). Remove any bubbles by gently taping the cuvette with your finger. Under absolutely no circumstances are you pressing a cuvette on a tabletop. Do not handle cuvette with the clear window (your fingerprints will cause an error in the measurement). Before placing the cuvette in the spectrometer, wipe the clear sides of the cuvette thoroughly with a Kim-Dry (do not use a paper towel). When placing the cuvette in the spectrometer, make sure that clear sides align with the light beam and that the cuvette is placed in the spectrometer in the same way each time. The main sources of error when using these spectrometers come from poor technique, and you can avoid these by following these guidelines every time you measure with the spectrometer. A representative range of a solution of the Al3+-aluminon complex is shown in Figure 1. The spectrum should show a wide peak close to 525 nm. If the shape of the spectrum looks dramatically different than in Figure 1, contact the teacher. Measure the absorbance at 525 nm (More info) for solutions 1 to 5. Graph the absorbance at 525 nm as a function of [Al3+] in Excel (Help Me) and perform a linear regression of the data by inserting a trendline (Help me) into the chart. View your teacher's graph. he or she has approved it, you can proceed to the next section. Figure 1. Representative absorbance spectrum for a diluted solution of the Al3+/aluminone complex in acetate buffer. Determination of aluminum in Alum Accurate (to three decimal places) weighs out about 0.2 g of alum. Quantitative transfer, as the teacher will demonstrate, to a small beaker and add distilled water to bring the volume to about 15 ml. Place the cup on a hob in the hood, cover with a bell glass and heat to boil, stirring occasionally with the glass tube rod. After stirring, rinse the glass rod into the beaker with a small amount of distilled water from the washing bottle. While the mixture is heating, clean and dry (externally only) volumetric bottles. Also prepare for a gravity filtration directly into the 100 ml volumetric flask using a long-stemmed glass funnel. Remove the beaker from the hob just when the solution starts to boil. Caution! The cup, the watch glass and the hob top are all warm. The teacher shows the safe way to remove the beaker from the hob. Immediately pour the warm solution into the funnel. As the solution is filtered, rinse the cup, bottom of the bell glass and the battered rod each with several small sinks of distilled water into the funnel. Once the solution is completely filtered, remove the funnel from the volumetric and rinse the volumetric neck with several small portions of distilled water. Volumetric should now be cool to the touch, but if it isn't, wait until it is. Dilute the solution in the flask of the mark. Transfer 3.00 ml of this solution to 25 ml of volumetric flask and dilute as before. Beeping 3.00 ml of the alum solution that you just prepared for a 50 ml volumetric. Add 20 ml of acetate buffer and 5 ml of aluminone solution using graduated cylinders and then dilute to the marked with distilled water. Wait 20 min and measure the absorbance at 525 nm, as you did for the other solutions. Register this value in your notebook. Results and analysis week 1 From the amounts of reactants you actually used, calculate the theoretical return of the alum (Help me). Week 2 Calculate the percentage yield of the alum from the theoretical return you determined last week, and the amount of alum you actually got. Share your percentage return with your classmates. Determine the percentage water by weight in the alum and the number of water of hydration in the alum. Share these numbers with the rest of the class, as instructed. Perform a Q-test (Help me) on the class data and discard the deviant datum, if there is one. From your class data, calculate the average percentage of water by weight in the alum, the standard deviation (Help me) for the data, and determine the confidence limits (Help me) with 95% confidence. Based on the familiar formula of alum, the expected value of the percentage water by weight in the sample. Calculate a percentage error for the class average and the result. Record all data and calculated results in your notebook. You can do the calculations in Excel, and if you do, you need to paste copies of the output into the portable lab notebook. Week 3 Calculate the concentration of the aluminium storage solution (the solution you had after the second dilution) and the concentration of aluminium in each of the solutions you prepared from the storage solution. In the Results section of the notebook, type these values in the table (Table 1 above). In the calculations assume that the volumes of bottles and pipettes are as shown in Table 2. Volumetric Volume (ml)1 ml pipet1.002 ml pipet2.003 ml pipet3.005 ml pipet5.0025 ml pipet25.0050 ml pipet50.00100-ml pipet100.00table 2. Nominal volumes of volumetric glass used in this exercise. From the equation for best-fit line for absorbance of 525 nm as a function of [Al3+] determine percent aluminum by weight in alum (Help me) and share the results with the class. Perform a Q test (Help me) on your class data, then calculate the al by weight by weight in alum, the standard deviation (Help me) of the data, and finally find the confidence limits (Help me) to 95% trust. Determine what the true percentage Al by weight is for the alum, and then calculate a percentage error for the class average and the result. Record the calculations in your notebook, as you did for the Week 2 metrics, and include any worksheet output. Conclusions The first week of this exercise was a synthesis. Therefore, your Discussion of conclusions section for this week should follow synthesis outline. Note that you will not be able to discuss the results for week 1 until after you have received the mass of the product and done qualitative tests on it. It is recommended to reserve two or three pages in the notebook for week 1 Discussion of conclusions and error analysis as you prepare for Week 2. An important question that you need to address in the discussion of the conclusions section for week 1 is why is your percentage return on alum less 100% with specific references to what you did and observed. Weeks 2 and 3 are both measuring exercises. In week 2 Discussion of conclusions and error analysis you should include a brief discussion of qualitative test results. In both Week 2 and Week 3, you collect proof of the identity and purity of your alum. So you need to include a brief discussion about whether your quantitative results support your alleged synthesis of alum. Although a propagation of misanalysis is possible, we will not perform one here. However, you should be able to identify where your main sources of uncertainty are and qualitatively discuss how they your results. Summary of results Week 1 Use Table 3 to report the results for Week 1. Mass of Al Used (g)Theoretical Yield of Alum (g)Mass of Alum Achieved (g)% Yield of Alum Table 3. Summary table for the first week. Week 2 Summarizes the results for Week 2 using Tables 4 and 5. In the second column of Table 4, type either positive or negative, as needed. Don't forget to report the confidence interval of the class data in Table 5. Potassium test: Sulfate test: Table 4. Summary table for qualitative tests. First Mass Alum (g) Mass of Anhydrous Alum (g)% Water of Mass in Alum% Error in % Water of MassNumber of Water by Hydration Our Results Class Results----- Table 5. Summary table for quantitative determination of water in alum. Week 3 Table 6 should be used to summarize the results for the third week's work. Remember to include the confidence interval at class average % Al by mass in alum. Mass of Alum Used (g)Inclination of Calibration Curve (AU· M-1)Calibration Curve Interception (AU)Absorbance by Alum Solution (AU)% Al by Mass in Alum% Error in % Al of Mass Our Results Class Results----- Table 6. Summary table for Week 3. References 1. Click here to download this file in PDF format. Note that hyperlinks are not active in the pdf version. 2. Smith, W. H.; Sager, E. E. and Sievers, I. J. Anal. Chem. 1949, 21, 1334-1338. 3. Marczenko, Z. Spectrophotometric determination of elements; Ellis Horwood Ltd.: Chichester, England, 1976, p. 116-117. The absorbance has no devices (although sometimes absorbent devices are used, abbreviated AU). The unit of concentration is molar, M, and the unit of the orbit is usually cm. Therefore, the device of molar absorptivity is M-1 cm-1. You will notice that in the synthesis section of this exercise (week 1) we measure volumes with graduated cylinders and cups, but in week 3 we use volumetric pipettes and bottles. This is no coincidence! In the first week we are less concerned with precision than we are in the third week. This is due to the inherent uncertainty of most synthetic procedures caused by side effects and other uncontrollable factors. In the quantitative measurement in week 3 we also perform a chemical reaction, but one that we know gives a specific answer. And then we can be much more precise. Since our results, and the conclusions we draw from it, critically depend on how well we did our measurement, we use the more precise volumetric bottles and pipettes. Under very acidic conditions, sulfate exists mainly as HSO4, hydrogen sulphation, which is not combined with Al3+ to form an insoluble compound. Your vacuum filtration device should look like the one shown above. Note the clamp that holds the filtration flask to the ring rack that prevents the filter flask Overturned. The spectrometer cannot read exactly 525.0 nm. Any wavelength within a few tenths of a nanometer will work just as well. Make sure you use the same wavelength for all solutions, and that you notice the exact wavelength in your notebook. This lab has a number of safety hazards associated with it, which is indicated in the text with the word CAUTION!. These dangers are generally associated with heating things (eg, dixics and solutions). The solutions are generally benign, with the exception of 9 M H2SO4, which is quite corrosive. Corrosive.

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