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ABSTRACT

This resource guide provides information for 13 laboratory experiments designed to be conducted in small schools with limited equipment and materials. For each experiment, the document outlines necessary equipment and materials, experiment procedures, and questions to be answered. The experiments are: (1) studying the properties of water; (2) floaters and sinkers; (3) drop height; (4) Boyle's Law; (5) hydrogen and oxygen generation; (6) the Molar volume of a gas; (7) sodium hydrogen carbonate stoichiometry; (8) ammonia fountain; (9) ionic reactions; (10) chromate-dichromate equilibrium; (11) introduction to chemical thermodynamics; (12) rates of chemical reactions; and (13) finding the detection limit for calcium with ethylenediamine tetraacetic acid (EDTA). A list of resource persons is included. (ALL)

MICRO LABS FOR HIGH SCHOOL CHEMISTRY

RUSSELL THIEL

SMALL SCHOOL CONFERENCE KSU

MICRO LAB INDEX

Studying the Properties of Water	1
Floaters and Sinkers	2
Drop Height	3
Boyle's Law	4
Hydrogen and Oxygen Generation	5
The Molar Volume of a Gas	6
Sodium Hydrogen Carbonate Stoichiometry	7
Ammonia Fountain	8
Ionic Reactions	9
Chromate-Dichromate Equilibrium	10
Introduction to Chemical Thermodynamics	11
Rates of Chemical Reactions	12
Finding the Detection limit for EDTA	15-26

STUDYING THE PROPERTIES OF WATER
Developed by D. Mosher & K. McElvein

EQUIPMENT:

PART A
- Unknown liquid
- Toothpick
- Special toothpick
- Beral pipette
- Wax paper

PART B
- Penny
- Dropper
- Water
- Liquid soap

PROCEDURE:

PART A - ONE DROP

1) Place a drop of water about the size of the tip of your little finger on the wax paper.
2) Use the toothpick as a stirring rod to examine the droplet; move and touch the drop. Describe the shape of the droplet. Draw a side view and a bird's eye view (from above) of the droplet.
3) Gently touch the top of the droplet with your finger and then raise your finger slowly. What happens?
4) Can you divide the drop into 2 or 3 smaller droplets? Is there a difference in the behavior of the little drops?
5) Join all the droplets together again.
6) Use the Special toothpick and touch the droplet. What happens? (The special toothpick is made by soaking as many tooth picks as needed in a strong solution of soap over night, and then allowing them to dry.)

PART B - HOW MANY DROPS?

1) Guess how many drops of water you think you can put on the "head" side of a penny.
2) Carefully add drops of water to your penny. (You are not a bombardier!) Add the drops to the big drop that forms on the penny until the water spills off. Keep careful count of the number of drops you use.
3) How well did you guess in your hypothesis?
4) Repeat the above procedure for the "tails" side. Is the number of drops that you can get on the tails side the same as the number you got on the heads side? Did you make a better guess?
5) Add a drop of soap to your water and carefully put drops of water on the head of the penny again. How does the number of drops campare to your answer in step #2?

FLOATERS AND SINKERS
Developed by D. Mosher & K. McElvein

EQUIPMENT:

Bucket or large tray	Water
Pencils	Pencil sharpeners
Coins	Egg shells
Styrofoam	Paperclips
Aluminum foil	Clay
Bottle caps	ect.

PROCEDURE:

1) (For the Teacher to do) Ask the class to look at their objects in a "different way." Fill a bowl with water and show each object in turn. Ask the students if they think the object will sink or float when dropped in the water. Encourage preductions.

2) Globs of clay will sink in water. Can clay be molded into a shape so that it will float?
3) A piece of aluminum foil will float if you set it on top of the water. If you push it under the surface, it will sink. What happens if you crumple the foil?
4) Objects float better in salt water and hot water. Use the same objects that you used before with salt water and hot water. Note and record any differences.
5) Liquids float and sink too. Experiments with vegetable oil, alcohol, and heavily sugared coffee or colored water. Observe the layers that form.
6) Fill an empty plastic soda bottle with hot water and set it in a bucket of cold water. Will it sink or float?
7) Try floating full soda cans. Use "regular" sugared sodas and diet sodas. Are there differences?
8) Try stacking varying concentrations of salt solutions on each other in a soda straw.
9) (To the teacher) Use you imagination here and try many different things. Allow students to bring objects to try. Try reshaping objects.

DROP HEIGHT

Developed by D. Mosher & K. McElvein

QUESTION:

What is the effect of the height from which a drop of water falls on the size of the drop print?

EQUIPMENT:

Eye dropper	Small plastic cup
Food coloring	Index card
Water	Metric ruler

PROCEDURE:

1) Formulate and record a hypothesis to answer the question: What will happen to the size of a drop print as you raise the droppr?
2) Add 2 drops of food coloring to the plastic cup, and fill it half full of water.
3) Construct a data table including columns to record the height of the dropper and the width of the drop print. Go from a dropper height of 2 cm to 20 cm by increments of 2 cm.
4) Partially fill the eye dropper with the colored water. Hold the metric ruler vertically on the index card.
5) Place the tip of the dropper at the 2 cm mark. Slowly squeeze the bulb and allow one drop to drip onto the card.
6) Immediately measure and record the width of the drop print.
7) Repeat steps 4 and 5 for each height listed in the data table.
8) Make a line graph of the results. Place the drop height on the horizontal axis and the drop print size on the vertical axis.

QUESTIONS:

1) What was the drop print size when the dropper was held at the 12 cm mark?
2) Why was the food coloring used?
3) How does your hypothesis compare with the results of the activity?
4) What variable were controlled?
5) What is the effect of drop height on the size of the drop prints?
6) Why did you have to measure the drop print size immediately?

Boyle's Law

Introduction:

Gases are compressible. The quantitative relationship between pressure and volume was studied by Robert Boyle. In this experiment, you will try to discover a relationship between the volume and pressure of a sample of gas trapped in a Beral pipet.

Procedure:

Prepare a Boyle's Law Beral pipet by filling the pipet with a colored water solution, food coloring does well. Do not fill the stem with the solution. Seal the end of the stem by heating and pressing closed with a pair of pliers.

Measure the length of the gas (air) in the stem of the pipet in millimeters, and record in the data table. Place a book on the bulb of the pipet, and again measure and record the length of the gas in the stem. Continue by adding more of the same kind of books, one at a time, measuring and recording the length of the gas in the stem until at least 5 data points have been collected. The same kind of book should be added each time to keep the pressure constant at one *book*.

The volume of the air can be calculated by measuring the diameter of the stem, but it can be shown that the volume is directly proportional to the length of the stem. as long as the diameter of the stem is a constant. Therefore, the pressure (in *books*) versus the volume (in millimeters of stem) can be graphed and the correlation is a graphical representation of Boyle's Law.

The microscale version of this lab was adapted from a lab from:

MicroChemistry
by Tom Russo
Kimtec Educational Corporation
P. O. Box 57
Kensington, Maryland 20895

This lab manual for high school general chemistry contains some 25 microscale labs.

Hydrogen and Oxygen Generation

Introduction:

This lab will demonstrate the controlled generation of hydrogen and oxygen in small quantities. The properties of these gases, alone and combined in various proportions, will be explored.

Procedure:

Hydrogen Generation

Fill a 13x100 test tube two thirds full of mossy zinc. Insert a beral pipet through the top of a size 00-two holed stopper. Push it through until the bulb of the pipet rests against the stopper. Cut the stem of the pipet off approximately 5 mm below the stopper. Place the cut portion of the stem in the second hole of the stopper until approximately 5mm extends below the bottom of the stopper. Remove the pipet with bulb from the stopper and fill it (one squeeze full) with 3 M HCl Return the pipet to the stopper and the stopper to the test tube.

Fill the bulbs of six beral pipets which have had the stems removed and the openings slightly enlarged, with water. Squeeze a few drops of HCl onto the zinc. Gas bubbles will be apparent. After 20 seconds, collect six bulbs full of the gas by placing a water filled beral pipet bulb over the end of the stem protruding from the stopper. If the gas generation slows, squeeze more HCl onto the zinc. Test the gas by placing the opening of a bulb of gas near a candle flame and squeezing gently. Repeat until a clear observation is made. Set the hydrogen generator aside for later use.

Oxygen Generation.

Fill another 13x100 test tube one third full with 3% hydrogen peroxide. Insert a complete beral pipet through the top of one of the holes in a size 00 two holed stopper. Push it through until the bottom edge of the stem would be just above the level of hydrogen peroxide in the test tube. Place a 7 cm portion of pipet stem in the other hole of the stopper. Push it through until 5 mm of stem protrudes below the bottom of the stopper. Take the stopper arrangement and coat the bottom 1 cm of the stem of the complete beral pipet with petroleum jelly. Roll the coated stem in fresh baker's yeast. Place the stopper in the test tube. Be sure the coated stem does not touch the hydrogen peroxide.

Gently push the coated pipet into the hydrogen peroxide. Bubbles of oxygen gas will immediately be formed. By adjusting the depth the coated pipet is submerged, the amount of oxygen produced can be controlled. After 20 seconds of evolution, collect six bulbs of gas. Test the gas by lighting the end of a toothpick in a candle flame and blowing the flaming toothpick out. The toothpick is now a glowing splint. Rapidly thrust the glowing end of the toothpick into the opening in a gas filled pipet bulb. Repeat until clear observations are made. Record observations.

Combining Hydrogen and Oxygen

Refill the six pipet bulbs with water. Refresh the hydrogen generator by squeezing more HCl on the zinc. Refresh the oxygen generator by submerging more of the coated stem in the hydrogen peroxide. After 20 seconds, collect a bulb three fourths full of hydrogen gas. Take the partially filled bulb to the oxygen generator. Complete filling it with oxygen. Use the candle flame to flame test this mixture of gases. Repeat steps 5 and 6 collecting one half a test tube of hydrogen, then repeat steps 5 and 6 collecting one fourth a test tube of hydrogen.

Reference:

The microscale version of this lesson was developed by:

Kin Mack
Charlotte Country Day School
1440 Carmel Rd.
Charlotte, N. C.

The Molar Volume of a Gas

Background:

The molar volume is the volume occupied by one mole of gas. This volume is a function of temperature and pressure.

In this experiment you will react a known mass of magnesium with an excess of hydrochloric acid (HCl) to produce hydrogen gas as shown.

$$Mg(s) + 2HCl(aq) \rightarrow MgCl_2(aq) + H_2(g)$$

The hydrogen gas produced will be collected by the displacement of water. You will then compute the volume at standard conditions using the gas model and equations. The STP volume will be used to calculate the molar volume of hydrogen gas.

Procedure:

1. Fill a 600-mL beaker about 3/4 full with water.
2. Obtain a short piece of magnesium ribbon from your instructor. Record its length to the nearest mm in your data table. Also record the mass of 1 meter of magnesium ribbon. This data will be provided by your instructor.
3. Obtain a piece of cotton thread about 10 cm long. Tie one end around the piece of magnesium ribbon, leaving about 7-8 cm of thread free. Gently bend the piece of magnesium ribbon so it will fit into a 10-mL graduated cylinder.
4. Obtain 3 mL of 3 M hydrochloric acid (HCl) in your 10-mL graduated cylinder, taking care to keep it off your skin.
5. Using a plastic squeeze bottle or a dropper, and using care to mix the acid and water gently, fill the graduated cylinder to the top with distilled water.
6. Lower the piece of magnesium ribbon into the graduated cylinder, coiling 1-2 cm extra thread into the top. Drape the remaining thread over the edge of the graduated cylinder and firmly insert a one-hole cork stopper. If there is an air space, add water through the hole in the cork.
7. Place your finger over the hole in the cork and invert the graduated cylinder. Lower the stoppered end of the graduated cylinder into the beaker of water. Note any evidence of a chemical reaction.
8. Allow the apparatus to stand for 5 minutes after the magnesium has completely reacted. Then, tap the sides gently to dislodge any gas bubbles that may have become attached to the sides of the graduated cylinder.
9. Move the graduated cylinder vertically (keeping the open end submerged) until the water level inside the cylinder is the same as the water level in the beaker. This is done to equalize the pressure of the gas trapped inside the cylinder with atmospheric pressure. Read the volume of gas in the cylinder. (Remember, the scale is upside down.)
10. Record the temperature of the water and barometric pressure.
11. Repeat the experiment.

Reference:

The Woodrow Wilson microscale version of this lesson was developed by:

Ginger Tannenbaum
Fairfield Senior High
1941 S. Staunton Drive
Fairfield, OH 45014

Sodium Hydrogen Carbonate Stoichiometry

Introduction:

The reaction of sodium hydrogen carbonate with hydrochloric acid and the Ideal Gas Law are used to check the validity of the stoichiometry.

Safety:

Goggles and aprons should be worn at all times in the Chemistry laboratory. The small "pops" produced in this lab should not be harmful but students should be aware that a noise is produced to avoid possible accidents. The 3 M HCl is corrosive and spills should be quickly cleaned up. If HCl is spilled on the skin, it should be washed off with tap water.

Procedure:

Clean and dry a 17 x 150 mm test tube in a Bunsen burner. Cool, mass, and record the mass. Mass 0.40 - 0.50 g of $NaHCO_3$ in the test tube.

Slip a Beral pipet through one of the holes in a size 0-two-hole stopper. Holding the bulb of the pipet next to the stopper, cut the pipet so that 5 mm of the stem protrudes below the stopper. Place the remaining portion of the stem in the other hole of the stopper so that about 5 mm protrudes below the stopper.

Remove the pipet portion containing the bulb, and take it to your teacher to obtain your sample of HCl. The 6-M HCl you are using should be handled with all precautions pertaining to strong acids. Return the pipet to the hole in the stopper. Be careful not to lose any of the acid. The pipet and stopper arrangement may be held upside down to prevent spillage. Seal the two portions of the pipet into place. Silicon glue works well. Place the stopper firmly in the test tube. Do not squeeze the pipet bulb. Place the entire apparatus under water in a sink or pneumatic trough.

Fill a 125 mL Ehrlenmeyher flask with water and invert it over the open pipet stem of your apparatus. Quickly and firmly in one motion, squeeze and release the pipet bulb. Carefully collect the gas in the 125 mL flask. When the reaction is complete, remove the test tube from the sink and set it upright on the lab bench. Rinse the flask until the water levels inside the sink are even. Cover the mouth of the flask and remove it from the sink, being careful not to lose any of the water. Measure the volume of water in the flask.

Refill the flask and measure the total volume. By subtracting you can calculate the volume of CO_2 produced. Compare this with the amount predicted by stoichiometry.

Carefully evaporate the water from the test tube. Heating too rapidly may cause the solution to splatter. A hot sand bath can be prepared by placing 1 to 2 inches of sand in a metal pneumatic though and heating on a hot plate. When the test tube is dry, let it cool and mass. By subtracting the original mass of the test tube, determine the mass of NaCl produced. Compare this with the amount predicted from stoichiometry. Calculate percent yields for both products.

Reference:

The microscale version of this lesson was developed by:

Kin Mack
Charlotte Country Day School
1440 Carmel Rd.
Charlotte, N. C.

AMMONIA FOUNTAIN
Adapted by D. Epp (DMT)

EQUIPMENT:

Beral pipet with 3-4 cm drawn out stem	
Beral pipet with 1 cm stem	100 ml beaker
250 ml beaker	Hot plate or burner
Concentrated ammonia solution	Phenolphthalein indicator solution

PROCEDURE:

1) Fill the 250 ml beaker about 1/3 full with water and heat to between 80 to 90 °C. Do not boil the water.
2) Fill the 100 ml beaker about 2/3 full of cold water, and add several drops of phenolphthalein indicator and set aside.
3) Take the longer pipet to the hood and fill it about 1/2 full with concentrated ammonia.
4) Return to your lab station and squeeze the shorter pipet, forcing all the air out. Fit it snugly onto the tip of the longer pipet. Continue to squeeze the shorter pipet.
5) Put the longer pipet into the hot water in the 250 ml beaker. As the ammonia solution warms, ammonia gas will bubble off and will enter the smaller pipet. Slowly release the pressure on the smaller pipet as it fills with ammonia.
6) When the smaller pipet is filled with ammonia gas, detach it from the longer pipet and place its tip into the cold water in the 100 ml beaker. Wait and observe. Do not squeeze the pipet while waiting.
7) Empty the pipets into the sink and reao steps 3 - 6.

QUESTIONS:

1) Does ammonia gas appear to be very soluble in water?
2) What color does the phenolphthlein turn when the cool water is sucked up into the pipet?
3) From your answer to question #2, is an ammonia solution acidic or basic? How do you know?
4) As an extension to this exercise use the same procedure with concentrated HCl instead of concentrated ammonia and substitute methyl red for phenolphthlein.

IONIC REACTIONS
Adapted by D. EPP (DMT)

EQUIPMENT:

Part A:

6 Solutions in dropper bottles so precipitates will form when 2 solutions are mixed.
Sheet of plastic
Toothpick
Notebook paper

Part B:

0.10 M solutions of
- $AgNO_3$
- KCl
- K_2CrO_4
- KI

Toothpick
Plastic sheet

PROCEDURE:

PART A

1) Set up a 6 X 6 grid on the notebook paper labeling the column and rows A - F.
2) Place the plastic sheet over the grid.
3) Make all possible combinations of solutions as specified by the grid.
4) Clean up your area.

PART B:

5) Put 2 drops of $AgNO_3$ on the plastic sheet.
6) Add 2 drops of KCl to the $AgNO_3$.
7) Look at a solution of KNO_3. Is there a precipitate in it?
8) To the same sample add several drops of KI solution and stir with a toothpick.
9) To a fresh area on the sheet mix 2 drops of the $AgNO_3$ and 2 drops of K_2CrO_4
10) Add KI dropwise and stir with a clean toothpick.
11) To a fresh area on the plastic mix 2 drops of $AgNO_3$ and 2 drops of KI.
12) Add K_2CrO_4 dropwise with stirring until you have added 10 drops.

QUESTIONS:

Part A

1) For each combination of solutions that shows a reaction, list all possible combinations of ions.
2) Eliminate the combinations that are starting solutions or do not show reactions when mixed
3) Write the molecular and net ionic equations for each combination that shows a reaction.

Part B

4) What is the precipitate when KCl is added to $AgNO_3$?

5) What happens to the precipitate of KCl and $AgNO_3$ when KI is added? What is the new precipitate?
6) Which precipitate is more soluble, the one formed with $AgNO_3$ and KCl or the one formed with $AgNO_3$ and KI? What evidence do you have for your answer?
7) What happens when you add KI to the $AgNO_3$ - K_2CrO_4 mixture?
8) What happens to the color of the solution and the precipitate when K_2CrO_4 is added to the mixture of $AgNO_3$ and KI? Remember that a solution of the $CrO_4^=$ is characteristically yellow in color.
9) Does the $CrO_4^=$ ion replace the I^- ion in the precipitate formed in the $AgNO_3$ - KI mixture?
10) Which precipitate, Ag_2CrO_4 or AgI, is more soluble?
11) Predict what would happen if you started out with a $AgNO_3$ solution and added KCl, KI and K_2CrO_4 in succession. Try it.
12) Look up the K_{sp} values of AgCl, AgI, and Ag_2CrO_4. Using those values explain why the precipitates changed as they did.

CHROMATE-DICHROMATE EQUILIBRIUM

Adapted K. Skelly (DMT) & S. Zoltewicz (DMT)

EQUIPMENT:

1.0 M solutions of:
- KNO_3
- $BaCl_2$
- K_2CrO_4
- $K_2Cr_2O_7$
- Na_2CrO_4
- $Na_2Cr_2O_7$
- NaOH
- HCl

13 X 100 test tubes or spot plates
Beral pipets for each solution for each group

PROCEDURE:

1) Examine solutions of KNO_3, K_2CrO_4, $K_2Cr_2O_7$, Na_2CrO_4, and $Na_2Cr_2O_7$.
2) Add 10 drops of K_2CrO_4 to a depression in your spot plate.
3) Add HCl to that same depression until a change is observed. Record your observations and save the solution.
4) Add 10 drops of $K_2Cr_2O_7$ to another depression of your spot plate.
5) Add NaOH to that depression until a change is observed. Record your observations and save the solution.
6) To a third depression of your spot plate, add 5 drops of K_2CrO_4.
7) Add 2 drops of $BaCl_2$ to that third depression and observe.
8) To a fourth depression, add 5 drops of $K_2Cr_2O_7$ and 2 drops of $BaCl_2$. Observe.
9) Add several drops of $BaCl_2$ to the solutions from steps 3 and 5. Observe.
10) Clean up your work area.

QUESTIONS:

1) From step #1, what can you conclude about the color of the $CrO_4^{=}$, and the $Cr_2O_7^{=}$ ions?
2) From step #7, what can you conclude about the solubility of $BaCrO_4$?
3) From step #8, what can you conclude about the solubility of $BaCr_2O_7$?
4) When you added $BaCl_2$ to the solutions from steps #3 and 5, what can you conclude about the concentrations of the $CrO_4^{=}$, and the $Cr_2O_7^{=}$ ions?

EXTENSION:

Other colored reversible systems include:

A) $Fe^{3+} + SCN^- \longleftrightarrow FeSCN^{2+}$

B) $Co^{2+} + 4Cl^- \longleftrightarrow CoCl_4^{2-}$

C) $Cu^{2+} + 4NH_3 \longleftrightarrow Cu(NH_3)_4^{2+}$

AN INTRODUCTION TO CHEMICAL THERMODYNAMICS
Adapted by D. Mosher & K. McElvain

PURPOSE:
To observe an exothermic and endothermic dissolving process, and to observe an endothermic chemical reaction.

EQUIPMENT:

3 empty flat bottom vials	Small piece of wood
Calcium chloride ($CaCl_2$)	Ammonium Nitrate (NH_4NO_3)
Barium Hydroxide ($Ba(OH)_2$)	Ammonium Thiocyanate (NH_4SCN)

PROCEDURE:
Part A)
1) Fill a vial to a depth of 1 cm with $CaCl_2$. Place the same amount of NH_4NO_3 in another vial.
2) Add enough water to fill each vial to a depth of 2 cm.
3) Feel the vials and record your observations.

Part B)
4) To the third vial add equal amounts of $Ba(OH)_2$ and NH_4SCN.
5) Place the top of the vial and shake gently. Observe.
6) Place the vial on a wet piece of wood.
7) Allow the vial to stand on the wood for about 1 minute. Then try to pick up the vial.
8) Open the vial and carefully smell the mixture.

QUESTIONS:
1) What did you observe when you mixed the calcium chloride and water?

2) What were your observations when you mixed the ammonium nitrate and water?

3) Did any physical state changes take place in vial #1 and 2?

4) What changes in physical states took place in the third vial?

5) Thinking at the molecular level, what do you think could be happening to cause these energy changes?

6) Which of the physical states do you feel has the greatest degree of order to their structures? (Solids, solutions, liquids, or gases) Which physical state do you feel is the least ordered?

7) Were all of these changes that took place spontaneous changes? What evidence do you have for your answer?

8) Do you feel that order changes have anything to do with causing chemical reactions? What evidence do you have for your hypothesis?

9) Do you feel that energy changes have anything to do with causing chemical reactions to occur? What evidence do you have for your hypothesis?

RATES OF CHEMICAL REACTIONS
Developed by D. Mosher & K. McEvein

EQUIPMENT:

25 ml beakers (3)
Alka-Seltzer tablets (3)
Celsius thermometer
Ice water
Room temperature water
Warm water
Stop watch or watch with a sweep second hand
Masking tape
100 ml graduated cylinder

PROCEDURE:

1) Copy the data table.
2) Use the masking tape to label the beakers A, B, and C.
3) Write a hypothesis stating the effect of temperature on the decomposition of an Alka-Seltzer tablet.
4) Pour 150 ml of ice water into beaker A. Measure and record the temperature in the data table.
5) Drop the Alka-Seltzer tablet into the ice water. Start the stop watch as soon as the tablet enters the water. Stop it when the tablet has completely decomposed.
6) Pour 150 ml of room temperature water into beaker B. Measure and record the temperature of the water.
7) Repeat step #5 with the room temperature water.
8) Pour 150 ml of warm water into beaker C. The temperature should be about 50 ^{o}C. Measure and record the temperature of the water.
9) Repeat step #5 using the warm water.

DATA TABLE:

BEAKER	TEMPERATURE (^{o}C)	REACTION TIME (SEC)
A		
B		
C		

QUESTIONS AND CONCLUSIONS:

1) In which beaker did the reaction occur most rapidly?
2) In which beaker was the reaction the slowest?
3) What evidence of chemical change did you observe when the Alka-Seltzer tablet placed in water?
4) What is the type of reaction you observed?
5) What is the effect of temperature on the reaction time of an Alka-Seltzer tablet?
6) Why did each beaker contain 150 ml of water?
7) How does your hypothesis compare with the results of your experiment?

Finding the Detection Limit for Calcium with EDTA and Determining the Calcium Ion Concentration of Tap Water and Various Mineral Waters

Background Information

Mineral content in water determines if it will be hard or soft. The two major minerals in water are calcium and magnesium (in the form of Ca^{2+} and Mg^{2+} ions); ions such as iron, lead and others also contribute to hard water. Rain is soft as none of these minerals are present. As the water travels through the soil and rocks, minerals are dissolved causing water to be "hard". The presence of Ca^{2+} and Mg^{2+} ions in water reduces the effectiveness of soaps to suds and clean, leaving a dingy gray residue on clothes, and spots on dishes. This water is more abrasive than soft water. The tiny mineral particles combine with soap curd or detergents to become like little pieces of rock pounding away at clothing fibers.

Skin and hair are affected by hard water. A greater amount of shampoo and soap is needed to clean, and hard water doesn't rinse as well as soft water. That means soap residues remain, leaving skin susceptible to blemishes and hair less shiny.

Hard water also causes scale to build up in plumbing, water heaters and pipes, limiting the water flow and reducing the life of the product. The white scale also deposits on water faucets and shower heads as a result of water evaporation with the minerals remaining behind. This scale can be removed by using vinegar, which is a weak acid and dissolves the deposits. By placing vinegar in a plastic bag and the bag and vinegar secured around the shower head, the white deposits will be dissolved in time. Use caution and goggles when doing this, as it could spill out in your eyes. Use running water to dilute the vinegar if spilled on your skin.

Soft water tanks contain resin beads that are coated with Na^{1+} or K^{1+} ions. Water softeners function to remove the Ca^{2+} and Mg^{2+} ions by exchanging these ions for sodium or sometimes potassium ions. As the water flows through the softener the Ca^{2+} and Mg^{2+} ions are attracted to the resin beads freeing the Na^{1+} or K^{1+} ions which then flow in your water in the house. When all the resin beads are coated

with the mineral deposits of salt water it becomes necessary to renew the resin beads. Salt (Na^{1+} and Cl^{1-}) is pumped through the tank replacing the Na^{1+} ions and releasing the hard water ions of Ca^{2+} and Mg^{2+}. Hardness is commonly expressed in units of grain per gallon (gpg) which is equivalent to 17.12 mg/L. (1 gpg = 17.12 mg $CaCO_3$/L).

For over fifty years, a polyaminocarboxylic acid, ethylenediamine tetraacetic acid (EDTA), has been known to form stable complexes with polyvalent metal ions. After World War II, this compound became the preferred reagent for the determination of the hardness of water. The general method of analysis is to buffer the sample to a pH of 10, use a colored indicator, and titrate the water sample with a standard solution of EDTA. The indicator used is calmagite; this indicator will be red in an excess of calcium ion and blue in an excess of EDTA. In order to determine the amount of calcium ions in solution, we must look for a color just between red and blue.

Purpose

In this experiment the student will determine the lowest concentration of calcium ion that can be detected by titrating a sample with EDTA. The student will also have the opportunity to determine the calcium ion concentration in tap water and in various mineral waters.

Materials (per lab group)

Chemicals:

- 100 mL 0.01 M EDTA (per class)
- 5 mL 0.00005 M EDTA/buffer solution
- 5 mL 0.10 M calcium chloride
- 1 mL 1% calmagite solution
- distilled water

Equipment:

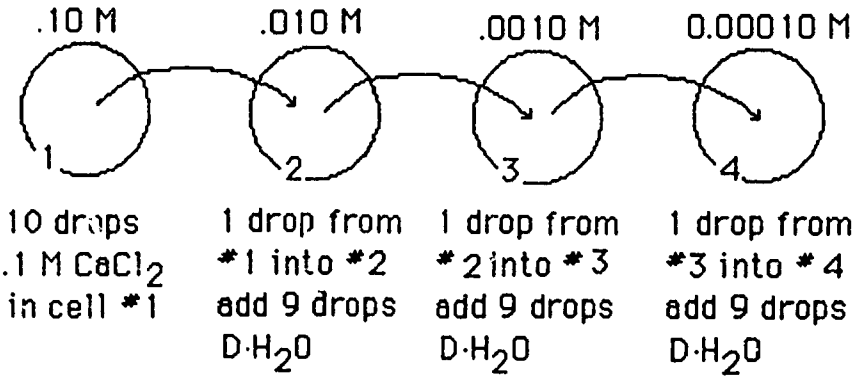
- 5 thin-stem Beral pipets or 5 eye droppers
- 1 24-well plate
- 1 100-mL graduated cylinder
- 1 10-mL graduated cylinder
- 1 50-mL beaker

Precautions/HazardsSafety goggles should be worn throughout the course of the lab. All chemicals should be handled with care, and hands and work areas should be cleaned thoroughly in case of a spill.

Procedure

Part I: Determine the limit of EDTA solution with Ca^{2+}.

1. Add 10 drops of the 0.10 M Ca Cl_2 solution to one well and follow the sequence in the diagram below for wells 2 - 4.



Fill in the color of each concentration above.

2. Add 1 drop of indicator solution to wells 1 - 4.

3. Add 4 drops of the EDTA - buffer solution to each well and observe the color.

4. Identify the last well in which the solution shows a **red** color. Make up a solution which has about 60 drops of that solution in a beaker. (If you have identified that .1 M Ca Cl_2 solution is the last red solution (the first cell) then no mixing is required - use the stock .1 M $CaCl_2$ solution.) To do this calculate the number drops of .1 M $CaCl_2$ and water necessary to make that solution. Use a 50-mL beaker to prepare the solution.

NOTE: The solution you just made in #4 above will be used in steps 5 - 10 to further make a series of 10 dilutions. If we look back at the diagram on page two, visualize 10 new wells between well #1 and #2, this will be the 10 dilutions described in steps 5 - 10 (eg. well

#1's concentration is 0.10 M and well #2 has 0.010 M concentration. The first well you make will be the same as well #1 [0.10 M] and #10 will be just like well #2 [0.010 M] in your first trial. Each well between #1 and #10 will be 0.010 M different from the one next to it.

5. Using the solution just made in #4 above, add 10 drops to the first well in row 2 of the plate.

6. Into the second well add 9 drops of this solution plus 1 drop of D·H_2O.

7. Into the third well take 8 drops of this solution plus 2 drops of D·H_2O.

8. Continue this progression until there is only 1 drop of this solution and 9 drops of D·H_2O.

9. Add one drop of indicator to each well.

10. Add 4 drops of EDTA-buffer solution to each well and observe the color.

Part II: Determine the hardness of water and mineral water.

Add 9 drops of tap water and add 1 drop of indicator and 4 drops of EDTA-buffer solution. Compare the color produced by the tap water with that of the standard serial dilutions you made in Part I. The same color would indicate the same concentration of Ca^{2+} ions in solution. If the water is very hard, and the color of the water after treatment is red you will have do do a serial dilution on it the same as with the standard stock solution (steps 5 - 10) and then calculate the amount of Ca^{2+} ions in solution.

Add 9 drops of mineral water to one well and follow the same procedure as with tap water above. It is quite possible that the mineral water will have to be diluted, as the calcium ion concentration is much higher than in tap water. If a dilution is required remember to multiply the dilution rate times the concentration that you matched with the known 0.1M Ca^{2+} ion solution. Using an example of a dilution of 1/2 of the mineral water and the concentration color matched with the known 0.1M Ca^{2+} ion

solution it will be necessary to multiply by 2 times that concentration, or in this case 0.2 M Ca^{2+} ion.

Suggested Questions (Prelab)

1. Write the equation for the dissociation of $CaCl_2$.

2. For each mole of $CaCl_2$ how many moles of Ca^{2+} ions are formed?

3. How many grams of $CaCl_2 \cdot 2H_2O$ are required to make a 0.10 M solution of Ca^{2+} ions in 100 mL of solution?

4. If 1.0 mL of O.1O M Ca^{2+} ions is added to 9 mL of water, what is the molarity of the new solution?

5. What is meant by the term chelate?

6. What is the purpose of adding a buffer to the sample solution?

7. Write the step-wise equations for the dissociation of EDTA from H_4EDTA to $EDTA^{4+}$.

Suggested Questions (Postlab):

1. What is the color of calmagite solution that has an excess of Ca^{2+} ions? of EDTA ions?

2. What is the concentration of your EDTA test solution?

3. What is the concentration of your most dilute Ca^{2+} solution that showed a color change?

4. What is the concentration of the first solution of Ca^{2+} ions that showed no color change with EDTA?

5. What is the concentration of Ca^{2+} ions in tap water?

6. Why would you have to dilute your water sample if the color was red?

7. Outline your procedure if your water had to be diluted, showing your calculations of how you made your diluted water sample.

8. What is meant by the term water hardness?

9. What is meant by the term complex ion?

10. Magnesium ion complexes with EDTA like Ca^{2+} ion. When the hardness of water is being measured, then, is Ca^{2+} the only ion being determined? Explain.

Disposal All chemicals used in this experiment are nontoxic. They may be flushed down the drain with copious amounts of water.

References

EDTA Titrations, an Introduction to Theory and Practice, Pergamon Press; 1964.

Standard Methods of Calcium Determination as presented in the Methods for Chemical Analysis of Water and Wastes, United States Environmental Protection Agency.

"Hardness"; Standard Methods for the Examination of Water and Wastewater; American Public Health Association; pages 194-199; 1981.

Submitted by Baird S. Bell and Russel E Thiel (Dreyfus '89), some revisions by Ronald Ulrich (Dreyfus '89 and Chem Team 10).

Teacher's Guide

Finding the Detection Limit for Calcium with EDTA and Determining the Calcium Ion Concentration of Tap Water and Various Mineral Waters

Description

This exercise will give the student an idea that analytical methods must be matched to the species under consideration. This method can be used as a springboard for the discussion of the metal ions in water that cause hardness and how one, calcium, can be removed. Through the course of this exercise the student will determine the lowest concentration of calcium ion that can be detected by titrating a sample with EDTA. The student will also have the opportunity to determine the calcium ion concentration in tap water and various mineral waters.

This experiment is intended to be used early in the year for a regular track chemistry course in the discussion of concentration or during the study of measurement techniques. A 0.00005 M solution of EDTA is used to titrate serial dilutions of a solution of Ca^{2+}, calmagite is used as the indicator. The student practices making dilutions and transfering solutions. A point is reached where the change in color of the calmagite cannot be seen.

Time:
General chemistry: 45 - 50 minutes
Advanced courses: 90- - 100 minutes

Materials

Chemicals:

EDTA standard stock solution (0.010M):
0.373 g of disodium ethylenediaminetertraacetate dihydrate (also called ethylenedimitrllo-tetraacetic acid disodium salt) dissolved in 100 mL of distilled water.

EDTA solution to use in lab trials (5.0×10^{-5} M):
Mix 1.0 mL of standard stock solution + 9 ml of distilled water (This is now 0.001 M EDTA). Take this 10 mL of solution add 190 mL of buffer solution (This is now 5.0×10^{-5} M).

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Buffer Solution: (pH=10)

Use commercially prepared buffer or dissolve 16.7 g of ammonium chloride (NH_4Cl) in 143 mL of concentrated ammonia solution. ($NH_3 + H_2O$). Dilute to a total of 100 mL. (Store in glass only.)

Calcium Chloride (0.10 M):

Dissolve 1.47 g of calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$) in 100 mL of water.

Calmagite solution .1%:

1-(1-hydroxy-4methyl-2-phenylazo)-2-naphthol-4-sulfonic acid. Dissolve 0.10 g of calmagite in 100 mL of water.

Equipment: See student version of the lab for the equipment list.

Modifications/Substitutions/Extensions:

Grade School/Middle School Science: This procedure is quite open-ended, therefore it can be applied to all levels of chemistry. For grade school and middle school classes, food color and tap water can be used. Place a drop of food color in the first well and observe the color. To the next well, add one drop of food color and 4 drops of water. Observe the color. Transfer one drop from the second well and 4 more drops of water; observe again. Continue this process until the color cannot be detected. More than one food color could be used to see if color is a factor that needs to be considered in detection limits. A discussion could then be held on how much the food color can be diluted, and if the color makes any difference.

High School/AP Chemistry: This lab gives the students practice preparing solutions volumetrically, and practice calculating the concentrations of solutions by using dilution factors. A known molarity of EDTA can be used and total hardness of tap water can be determined. Unknown solutions could be added easily by asking the students to find the hardness of local water supplies. Visual analysis could be replaced by colorimetric methods in a Spectronic 20. The

limit of detection by this method could be compared to other methods. Class discussion could then center on the idea that the methods of analysis depend largely on the solutions to be analyzed.

Discussion

For over fifty years a polyaminocarboxylic acid, ethylenediamine tetraacetic acid (EDTA), has been known to form stable complexes with polyvalent metal ions. After World War II, this compound became the preferred reagent for the determination of the hardness of water. The general method of analysis is to buffer the sample to a pH of 10, use a colored indicator, and titrate the hard water sample with a standard solution of EDTA. The chemistry of this determination is a good study of the concepts of the complex ion formation and equilibrium.

EDTA forms a stable complex with a metal ion because of its unique ability to flex around the metal ion and enclose it in a cage structure. The large number of negative charges on the ends of the "arms" of the EDTA molecule act as a set of hooks or claws to be attracted to the metal ion. At a pH of 10 all four hydrogen ions are stripped away from the EDTA molecule leaving an anion with a 4- charge. The molecule is said to be a polydentate (many clawed) species because of these multiple charges. These hooks are attracted to the coordination sites of the metal ion. When this happens, the metal ion is literally surrounded by the EDTA ion. Because of the high charge on both the EDTA ion and the metal ion, the structure that develops the resulting complex is quite stable.

This equation for the formation of the complex between the calcium ion and EDTA is : $Ca^{2+} + EDTA^{4-} \rightarrow CaEDTA^{2-}$. The equilibrium constant expression for this reaction was published in 1956 by Schwarzenkach. Its value, $K_{eq} = [CaEDTA^{2-}] / [Ca^{2+}][EDTA^{4-}]$, is equal to 5.0×10^{10}. The very large value for the K_{eq} is an indicator of the stability of the complex.

There are several important factors to consider when an indicator is selected. Some of the most important factors include: A) the indicator must be a different color when there is an excess of metal ion than when there is an excess of EDTA, B) the indicator must form a complex with EDTA that is less stable than the calcium-EDTA complex, and C) the indicator should not be affected adversely by

other ions in the solution. Calmagite, the indicator in this procedure, fulfills these requirements. It is red in an excess of calcium ion and blue in an excess of EDTA. The end point of the titration can be enhanced by the addition of a small amount of magnsium ion to the EDTA solution. If the magnesium ion is desired, 0.31 g of $MgSO_4 \cdot 7H_2O$ can be added to each 100 mL of EDTA solution.

Magnesium also forms a stable complex with the EDTA ion. Magnesium is another major cause of water hardness. Generally, then, a titration of hard water with EDTA will show the total hardness of the water sample instead of just the hardness supplied by the calcium ion. The magnesium complex is not as stable as the calcium complex. Because of that fact, methods have been developed to differentiate between the calcium ion and the magnesium ion in hard water with EDTA. Those methods are beyond the scope of this exercise.

If this exercise is to be performed by advanced students where accuracy is a consideration, the samples and standards should be stored in plastic or hard glass containers. Solutions can be contaminated by the leaching of metal ions from a soft glass bottle when the solutions are stored in these containers for long periods of time.

Answers to Student Questions (Prelab)

1. Write the equation for the dissociation of $CaCl_2$.
   $CaCl_2(s)$ --- -> $Ca^{2+}(aq)$ + 2 $Cl^-(aq)$

2. For each mole of $CaCl_2$ how many moles of Ca^{2+} ions are formed?
   One

3. How many grams of $CaCl_2 \cdot 2H_2O$ are required to make a 0.10 M solution of Ca^{2+} ions in 100 ml of solution?
   0.10 M x 0.10 L x 147 g/mole = 1.47 g

4. If 1.0 ml of 0.10 M Ca^{2+} ions is added to 9 ml of water, what is the molarity of the new solution?
   0.10 M X 1.0 ml/10.0 ml = 0.0010 M

5. What is meant by the term chelate?
   A chelate is a chemical species that usually has a negative charge that is attracted to and holds a metal ion in solution.

6. What is the purpose of adding a buffer to the sample solution?
   The buffer adjusts the pH of the solution to a specific level. The pH needs to be held constant to insure that the indicator responds to the changes in the metal ion. *Teacher's note* - The sample solution needs to be buffered to a pH of 10 to insure that the calmagite will show a different color when it is in an excess of calcium ion or an excess of EDTA.

7. Write the step-wise equations for the dissociation of EDTA from H_4EDTA to $EDTA^{4-}$.
   a. $H_4EDTA + H_2O \text{ ----> } H_3EDTA^{1-} + H_3O^{1+}$
   b. $H_3EDTA^{1-} + H_2O \text{ ----> } H_2EDTA^{2-} + H_3O^{1+}$
   c. $H_2EDTA^{2-} + H_2O \text{ ----> } HEDTA^{3-} + H_3O^{1+}$
   d. $HEDTA^{3-} + H_2O \text{ ----> } EDTA^{4-} + H_3O^{1+}$

Answers to Student Questions (Postlab):

1. What is the color of calmagite solution that has an excess of Ca^{2+} ions? of EDTA ions?
   red

2. What is the concentration of your EDTA test solution?
   0.000050 M

3. What is the concentration of your most dilute Ca^{2+} solution that showed a color change?
   0.00010 M

4. What is the concentration of the first solution of Ca^{2+} ions that showed no color change with EDTA?
   0.000010 M

5. What is the concentration of Ca^{2+} ions in tap water?
   0.0050 M (will vary with different water samples)

6. Why would you have to dilute your water sample if the color was red?

   The water contains a high concentration of calcium ions and will need to be diluted in order to find the detection limit of the EDTA.

7. Outline your procedure if your water sample had to be diluted, showing your calculations of how you made your diluted water sample.

   Answers will vary depending on what kind of dilution was carried out.

6. What is meant by the term water hardness?

   Hardness is a term that refers to the total concentration of the calcium and magnesium ions in solution.

7. What is meant by the term complex ion?

   A complex ion is the combination of positive and negative ions that will hold a metal ion in solution.

8. Magnesium ion complexes with EDTA like Ca^{2+} ion. When the hardness of water is being measured, then, is Ca^{2+} the only ion being determined? Explain.

   No. Since EDTA forms a stable complex with both of these metal ions, in a titration enough of the EDTA must be added to tie up both kinds of ions. Therefore, when EDTA is used to determine hardness of water, it shows the total hardness of the water not just the calcium ion concentration.

Submitted by Baird S. Bell and Russel E Thiel (Dreyfus '89), some revisions by Ronald Ulrich (Dreyfus '89 and Chem Team 10).