Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #1**

## ACID/BASE PROPERTIES OF ANTACIDS

## INTRODUCTION/OBJECTIVE

You will determine the "acid-absorbing" properties of three common antacid products. You can use the same titration program that you previously used. You will compare three antacids, and you will be required to do duplicate determinations of the antacid strength on each one. These duplicate runs must be in agreement with each other; if you have two runs which are very different, you will need to do a third one. This is common practice in the laboratory world; two runs are done as a check, and if they don't agree, one has to do a third.

## **BACKGROUND**

Before you begin this project, it is important that you have a good understanding of acids, bases, and titrations. Only some basics will be covered in this handout. Consult your text for the chapter on acids, bases and titrations.

Commercial antacid tablets contain bases. People self-prescribe antacid tablets to relieve "heartburn" and "acid indigestion" which they feel is caused by the stomach excreting excess stomach acid (HCl). In principle, the base in the antacid neutralizes the excess stomach acid.

There are standard pharmaceutical testing techniques for evaluating antacid tablets. These techniques not only test for acid neutralizing capability, but account for gastric residence time, body temperature, the rate at which the antacid reacts, and the actual pH rise in the stomach. All of these factors are important physiologically. For example, if the antacid causes the pH of the stomach to rise above 3 (the stomach is normally around pH 1), the digestive enzyme pepsin begins to deactivate, causing digestion problems. In this project, you will only be concerned with determining the acid-absorbing capability of antacids.

Titration is one method which can be used to find the acid-absorbing capability of an antacid tablet. Titration techniques are commonly used to determine unknown concentrations of acids and bases. If the base concentration is unknown, a known amount of the base is titrated with a measured amount of a known concentration of acid. If the acid concentration is unknown, the unknown is then titrated with a measured amount of a known concentration of base.

When monitoring the change in pH as base is added, the resulting solution will experience a rapid rise in pH at the equivalence point. At the equivalence point, moles of acid present = moles of base added

The middle of the steep rise (where the slope is the steepest) is the <u>equivalence point</u>. Refer to your previous experiments on best how to determine this value precisely. You will be expected to calculate the number

of moles of acid and base from the titration data.

Since antacids are designed to dissolve in the stomach, you will need to determine the best way to get the tablet dissolved. The information below gives you a hint on how to proceed.

	moles of HCI		moles of HCI		moles of HCI
(1)	added to	=	neutralized	+	neutralized
	an antacid tablet		by antacid		by NaOH

#### EXPERIMENTAL PROCEDURES

## **Preparation of the Antacid Sample**

You will analyze three different brands of antacid tablets, at least two tablets for each one. Check ingredients on the ones you choose; one of them **must contain aluminum**, and one must be **aluminum-free**. You and your partner will need to work out a procedure for your experiment and submit it to your instructor for approval. Be sure you calibrate you pH probe as indicated in the Measurement Manual or your previous titration experiments.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You will need to find the equivalence point of each titration.
- 3. You will use the titration results to perform all calculations necessary to find the grams of active ingredient in each antacid tablet and the weight percent of inert ingredients.
- 4. You may be asked to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during a later lab period.

## INSTRUCTOR RESOURCES

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Instructor resources will be listed ion order for each experiment.

## **INDIVIDUAL PROJECT #1**

## **ACID/BASE PROPERTIES OF ANTACIDS**

#### INTRODUCTION/OBJECTIVE

Students will determine the "acid-absorbing" properties of three common antacid products. They can use the same titration program that they previously used, or try a new approach. They will compare three antacids, and should titrate duplicate determinations of the antacid strength on each one. These duplicate runs should be in agreement with each other; if they have two runs which are very different, they will need to do a third one. This is common practice in the laboratory world; two runs are done as a check, and if they don't agree, one has to do a third.

#### Background

See student hand out for background information.

It would seem that in order to find the acid neutralizing capability of an antacid, one could dissolve the tablet in water and titrate the solution with a known concentration of acid, such as HCI. However, since antacids are designed to dissolve in the stomach, many are not very soluble in water. Therefore, the student will need to be guided to realize that he will need to dissolve the antacids in a known, excess amount of HCI. They will then titrate this resulting acidic solution with a known concentration of NaOH. Thus, at the equivalence point:

	moles of HCI		moles of HCI		moles of HCI
(1)	added to	=	neutralized	+	neutralized
	an antacid tablet		by antacid		bv NaOH

This hint is given the students in their handout.

Preparation of the Antacid Sample (Students should be guided to these procedures and results.)

Students will analyze three different brands of antacid tablets, at least two tablets for each one. Check ingredients on the ones you choose; one of them **must contain aluminum**, and one must be **aluminum-free**. Students should be guided towards the following procedure.

- 1. Weigh an antacid tablet. Grind up the antacid tablet in a mortar and transfer the powdered sample to a clean 250 ml beaker. Rinse the mortar with a few milliliters of water to capture the remaining powder and add this water to the beaker.
- 2. Using a pipette or buret, put 50.0 ml of 0.3 M HCI into the beaker with the tablet (*note the exact concentration of the HCI on the bottle*). Heat the beaker to boiling with a hot plate, gently stirring the solution. Let the solution

boil for 5 minutes, then let it cool to room temperature. **SAFETY NOTE:** Use extreme caution around the boiling acid! They might place the beaker in an ice/water bath to speed up the cooling (don't let water get into the beaker!).

## **Titrating Procedure**

While the beaker is cooling, prepare to titrate the sample with standard base. Have the students follow the preparations for any previous titrations they have done. There are explicit instructions on the *MicroLAB* CD in the section entitled **Useful Titration Operations** for a variety of ways of doing titrations. Help them select one if you have the CD, or give then your own instructions.

Students will need to calibrate their pH electrode at three buffer concentrations, 4, 7 and 10 pH, and check afterward to see that the electrode gives the correct reading in the pH 7 buffer.

When their antacid solution has cooled to room temperature, insert the pH electrode in the beaker, load the program for the titration, and carry it out. Be sure that they continue the titration until they reach a pH of at least 10 or 11.

They will need to decide exactly how they want to present their results in verbal and/or written reports. One thing they will want to look at is the difference between the antacids which contain aluminum and those which do not. Help them understand and remember as they do their calculations that the base they add during the titration is reacting with the **excess** acid - that is, with the acid that the antacid tablet <u>did not neutralize</u>. In each of their titrations they will calculate how much acid was added before the mixture was boiled to dissolve the antacid, how much acid they titrated with base, and then subtract "titrated" from "total" to find how much the antacid consumed. Some of the results will probably surprise you and them!

## MINIMUM REQUIREMENTS

- 1. See student handout for this information.
- 2.

#### REFERENCES

Hem, S.L. "Physicochemical Properties of Antacids," J. Chem. Educ., 52 (6), 1975, 383-385.

Batson, W.B. and Laswick, P.H. "Pepsin and Antacid Therapy: A Dilemma," J. Chem. Educ., 56 (7), 1979, 484-486.

## **Reactions of Antacid Tablets**

```
+ HC1
                                        ---> NaCl
                                                           + H_2O
                                                                        + CO_{2}
NaHCO<sub>3</sub>
CaCO<sub>3</sub>
                    + 2 HCl
                                        ---> CaCl_2
                                                           + H,O
                                                                        + CO_{2}
MgCO<sub>3</sub>
                                         ---> MgCl_2
                                                           + H_2O
                                                                         + CO_{2}
                    + 2 HC1
Mg(OH),
                    + 2 HC1
                                         ---> MgCl_2
                                                           + H_2O
AI(OH)_2NaCO_3 + 4 HC1
                                         \rightarrow AlCl<sub>2</sub>
                                                           + NaCl
                                                                        + 3 H2O
                                                                                      + CO_{2}
```

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## **INDIVIDUAL PROJECT #2**

## ACID/BASE PROPERTIES OF BUFFERED ASPIRIN

## INTRODUCTION/OBJECTIVE

You will determine the buffering properties of three kinds of buffered aspirin by titrating them first with acid and then with base. Each titration must be done in duplicate. This is common practice in all laboratories, in order to check the accuracy of results.

## **BACKGROUND**

## **Buffering Capacity of Aspirin**

You have all seen commercials touting the advantages of using one brand of aspirin over another. Some manufacturers say that buffered aspirin is better for you because it prevents stomach upset. If an aspirin is effectively buffered, the pH will not vary much upon the addition of moderate quantities of acid and base.

Aspirin is itself an acid. This is probably why it can so easily irritate the digestive tract, and why putting in a buffer makes it easier for some people to tolerate using it. Buffering a system means that you add some chemicals to it that react with an acid and with a base, so that if you add either one, it is absorbed and does not change the pH very much. This means that if you titrate a buffered system with either an acid or a base, initially the pH should change slowly. Eventually you will reach a point where a big change occurs as the buffering capacity is exceeded. In this project you will titrate two different brands of buffered aspirin and one of an unbuffered brand, and compare the results. Standard solutions of HCI and NaOH are provided in the laboratory for your use.

#### EXPERIMENTAL PROCEDURES

Many types of medicinal tablets are processed with a "binder" to hold the ingredients together in tablet form. Some of these "binders" may not be soluble, so you will have to determine the best way to get the aspirin tablet in solution, and whether not all of it will go into solution.

You and your partner will need to work out a procedure for your experiment and submit it to your instructor for approval. Be sure you calibrate your pH probe as indicated in the Measurement Manual or your previous titration experiments.

Weigh one of your aspirin tablets, grind it up in a mortar, and dissolve it in 100 ml of water in a beaker. Some tablets may not dissolve completely; it depends on what binders and other components have been used in the formulation. Just be sure that if anything is still undissolved after 5 minutes it is fine powder at the bottom of the beaker.

While the aspirin is dissolving, prepare to titrate the sample with standard base. Rinse your buret three times with the standard base provided; be sure and record the concentration in your lab notes. Fill the buret after rinsing and clamp the buret upright on a ring stand at your work station. If you are using a drop counting experiment, set and measure the flow rate. You will have to determine how to stop the flow without changing the dropping rate.

Calibrate your glass electrode in a small amount of buffer, and check afterward to see that the electrode gives the correct reading in the buffer. Insert the glass electrode in the aspirin solution, load the program for the titration, and carry it out. Be sure that you continue until you reach a pH of at least 10 or 11.

Repeat this process with the base as titrant for each of your three aspirin samples. Then rinse out the buret with de-ionized water, repeat the rinsing process with the standard HCI solution, and do three more titrations of your samples with acid. In this case, be sure that you carry the titration to a pH below 2.

You can plot and work with these results on the spreadsheet, but you will need to decide exactly how you want to present your results in your reports. For each sample you should have two titrations, one with acid and one with base. For each type of aspirin they should start at about the same pH and go opposite directions. Look at how much acid/base it takes to change the pH by one unit in either direction, and also two units. Make a table of these results for your report. Are your results symmetrical, with respect to acid and base? Would you expect them to be?

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, aks your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must titrate three different kinds of aspirin with standard acid and base, continuing your titration out to near the limits of the pH scale.
- 3. You will need to find the equivalence point of each titration for each of the three brands of aspirin tablets.
- 4. You must construct a table of volumes of acid and base necessary to change the pH from its initial value by one pH unit and by two pH units in either direction.
- 5. Your instructor may ask you to a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

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#### **INDIVIDUAL PROJECT #2**

## ACID/BASE PROPERTIES OF BUFFERED ASPIRIN

## INTRODUCTION/OBJECTIVE

To determine the buffering properties of three kinds of buffered aspirin by titrating them first with acid and then with base. Each titration must be done in duplicate

#### BACKGROUND

## **Buffering Capacity of Aspirin**

See Student hand out page

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

Students will need to work out a procedure for their experiment and submit it to their instructor for approval. Be sure they calibrate their pH probe as indicated in the Measurement Manual or their previous titration experiments.

Weigh one aspirin tablet, grind it up in a mortar, and dissolve it in 100 ml of water in a beaker. Some tablets may not dissolve completely; it depends on what binders and other components have been used in the formulation. Just be sure that if anything is still undissolved after 5 minutes it is fine powder at the bottom of the beaker.

While the aspirin is dissolving, they prepare to titrate the sample with standard base. Follow any of the procedures listed in the **Useful Titration Operations** section on the CD, or those provided by yourself.

Students should calibrate the pH electrode in pH 4, 7 and 10 buffers, and check afterward to see that the electrode gives the correct reading in the buffer. Insert the pH electrode in the aspirin solution, load the program for the titration, and carry out the titration. Be sure that you continue until you reach a pH of at least 10 or 11.

Repeat this process with the base as titrant for each of their three aspirin samples. Then rinse out the buret with de-ionized water, repeat the rinsing process with the standard HCI solution, and do three more titrations of their samples with acid. In this case, be sure that they carry the titration to a pH below 2.

They will need to decide exactly how to present the results in their reports. For each sample they should

have two titrations, one with acid and one with base. For each type of aspirin they should start at about the same pH and go opposite directions. They should look at how much acid/base it takes to change the pH by one unit in either direction, and also two units. Make a table of these results for their report. Are the results symmetrical, with respect to acid and base? Would you expect them to be?

## MINIMUM REQUIREMENTS

1. See student handout for this information.

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## **INDIVIDUAL PROJECT #3**

#### ACID/BASE PROPERTIES OF COMMON HOUSEHOLD PRODUCTS AND FOOD

#### INTRODUCTION/OBJECTIVE

You will be determining the acid/base properties on many common household and food products. You will also be using acid/base titration techniques to determine the percentage of hydrochloric acid in a common liquid toilet bowl cleaner.

## **BACKGROUND**

Before you begin this project, it is important that you have a good understanding of acids, bases, pH, and titrations. Only some basics will be covered in this handout. You should understand the Chapter on Acids and Bases in your textbook.

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is very often the acid/base properties of a food product which give it a characteristic taste. ((CAUTION! DO NOT TASTE ANY OF THE CHEMICALS USED IN THIS LAB!) It is also the acid/base properties of many household products which cause them to work.

In this project, you will measure the pH of many food and household products. You will determine what ingredient is responsible for the pH and what (if any) function this ingredient has in the product. In addition, you will quantitatively measure the percentage of hydrochloric acid in a popular liquid toilet bowl cleaner.

## **EXPERIMENTAL PROCEDURES**

# CAUTION! DO <u>NOT</u> MIX ANY OF THE HOUSEHOLD PRODUCTS TOGETHER! VERY TOXIC GASES MAY RESULT!

## Part I. pH of Food and Household Products.

To better organize your data, set up a data table which includes <u>at least</u> the following: product/name brand, diluted/straight, pH, color, active ingredient, and function. For some of the household products, your instructor may want you to include chemical equations which describe how the active ingredient works (this may require some outside research). Write a simple pH program to make these tests.

You will need to decide how you will sample each of the different types of foods to get the desired results.

## Part II. Quantitative Measurement of HCl in a Liquid Toilet bowl Cleaner

In this part of your project, you will find the percent hydrochloric acid in Lysol Liquid Toilet Bowl Cleaner (**CAUTION! AVOID CONTACT WITH SKIN!** If contact occurs, rinse promptly in the sink and get your lab instructor!) Hydrochloric acid (HCl) is the active ingredient in many liquid toilet bowl cleaners. HCl will react with hard water scale (calcium carbonate) in an acid/base reaction which dissolves the scale. Because of the concentrated acidic nature of the bowl cleaner, it also acts as a disinfectant. Other ingredients are often added to enhance the germicidal properties and flow characteristics.

You should already have performed a titration, should know the relationship of moles of acid to moles of base at the equivalence point know how to determine the equivalence point using derivatives.

You will need to determine how much to dilute and how to titrate the bowl cleaner. You will need to calibrate the pH electrode in pH 4, 7 and 10 buffers before use.

You will need to determine which titration procedure you will use in consultation with your instructor.

You will need to do each titration at least three times, and in your report give the individual results, the average and the standard deviation.

You will need graphs of all three titrations and have determined the equivalence points from these graphs, and from this data calculate:

at the equivalence point of each titration
moles of base added = (liters of base added)x(molarity of base) OR
mmoles of base added = (ml of base added)X(molarity of base)
Moles NaOH consumed
Moles HCl in sample (= Moles NaOH consumed)
Grams HCl in sample
% HCl in sample

You will need to report your final results in terms of the percentage of HCl in the sample. The percentages should agree with each other. All of the other numbers may differ, depending on the details of your experiments. Compare your result with the stated value from the product's label.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must turn in a table of your results from Part 1.
- 3. You must turn in a plot of pH versus ml of NaOH added for the liquid bowl cleaner. You will use the plot to find the equivalence point for each of your 3 titrations.
- 4. You will use the titration results to perform all calculations necessary to find the mass percent of hydrochloric acid in the liquid bowl cleaner.
- 5. Your instructor may ask you to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

## Dale A. Hammond, PhD, Brigham Young University Hawaii

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#### **INDIVIDUAL PROJECT #3**

#### ACID/BASE PROPERTIES OF COMMON HOUSEHOLD PRODUCTS AND FOOD

#### INTRODUCTION/OBJECTIVE

Students will be determining the acid/base properties on many common household and food products. They will also be using acid/base titration techniques to determine the percentage of hydrochloric acid in a common liquid toilet bowl cleaner.

#### BACKGROUND

See student hand out for background information.

## ((CAUTION! STUDENTS SHOULD NOT TASTE ANY OF THE CHEMICALS USED IN THIS LAB!)

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

# CAUTION! STUDENTS SHOULD <u>NOT MIX ANY OF THE HOUSEHOLD PRODUCTS TOGETHER! VERY TOXIC GASES MAY RESULT!</u>

## Part I. pH of Food and Household Products.

You will need to help the students determine how to organize their data to include such factors as product/name brand, diluted/straight, pH, color, active ingredient, and function. You should include chemical equations which describe how the active ingredient works (this may require some outside research by the students). They should write a simple pH program to make these tests.

You will also need to help them determine the best way to measure the pH of each substance.

- 1. For all food (consumable) products, they may take the pH of the straight product (e.g., orange juice, coke, etc.).
- 2. For all liquid household products, dilute 1 volume of product to 5 volumes of distilled water. This dilution will make the products safer to handle.
- 3. For powdered household products, add one gram of solid per 50 ml of water (e.g., detergents, crystal bowl cleaners).
- 4. For solid household products, add one gram of solid per 50 ml of water and mix well in a blender (e.g., fruits, vegetables).
- 5. Measure the pH of all of the available products using the *MicroLAB* interface pH program. They may test products of their own, with your permission.

#### Part II. Quantitative Measurement of HCI in a Liquid Toilet bowl Cleaner

Students will determine the percent hydrochloric acid in Lysol Liquid Toilet Bowl Cleaner

(CAUTION! AVOID CONTACT WITH SKIN! If contact occurs, rinse promptly in the sink and get your lab

instructor!) Hydrochloric acid (HCI) is the active ingredient in many liquid toilet bowl cleaners. HCI will react with hard water scale (calcium carbonate) in an acid/base reaction which dissolves the scale. Because of the concentrated acidic nature of the bowl cleaner, it also acts as a disinfectant. Other ingredients are often added to enhance the germicidal properties and flow characteristics.

You will need to help students determine the best method of measuring the amount of acid in the toilet cleaner, as follows:

- 1. To prepare the bowl cleaner for titration, add 5.0 ml of liquid bowl cleaner (using a pipette or graduated cylinder) to a dry, clean, previously weighed 250 ml beaker. Weigh the beaker and bowl cleaner to obtain the mass of the bowl cleaner. After you've determined the mass of the bowl cleaner, add about 40 ml of distilled water.
- 2. Calibrate their pH electrode. Instructions are given in your previous experiment if you have forgotten how to do this.
- 3. Prepare their buret to do another titration like the one they did in a previous experiment. Rinse the buret 3 times with the standard NaOH provided for this experiment, then fill the buret, insert the constant flow tube, and clamp buret securely upright on a ring stand at your work station. They will use the same program they used in your previous experiment, or they may try a different technique for experience.
- 4. Place their sample beaker of toilet bowl cleaner under the buret and insert the pH electrode in the solution. They should check to insure that the pH electrode is reading properly, then continue the titration until they reach a pH of at least 10.
- 5. In any case where one needs reliable data from an experiment, one repeats it several times. The students should repeat this titration at least twice more, and in their report give the individual results, the average and the standard deviation.
- 6. Graph the results from all three titrations, using the *MicroLAB* spreadsheet. Determine the equivalence points from these graphs, and from this data calculate:

```
at the equivalence point of each titration
moles of base added = (liters of base added)x(molarity of base) OR
mmoles of base added = (ml of base added)X(molarity of base)
Moles NaOH consumed
Moles HCI in sample (= Moles NaOH consumed)
Grams HCI in sample
% HCI in sample
```

The percents should agree with each other. All of the other numbers may differ, depending on the details of your experiments. Compare their result with the stated value from the product's label.

## MINIMUM REQUIREMENTS

1. See student handout for this information.

Dale A. Hammond, PhD, Brigham Young University Hawaii

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## **INDIVIDUAL PROJECT #4**

## THERMOMETRIC DETERMINATION OF AMMONIA IN HOUSEHOLD CLEANER

#### INTRODUCTION/OBJECTIVE

You will be using Thermometric and potentiometric acid/base titration techniques to determine the percentage of ammonia in two common liquid household cleaners.

## **BACKGROUND**

Before you begin this project, it is important that you have a good understanding of acids, bases, pH, and titrations. Be sure to review the chapter on acids and bases in your text.

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is the acid/base properties of many household products which cause them to work. For example, in Parsons Clear Ammonia household cleaner, the major active ingredient is *aqueous ammonia*. Ammonia acts as a surfactant and makes non-polar substances, such as grease, at least partially soluble in water. Aqueous ammonia is often the ingredient of choice in products that claim to be "grease cutters." Aqueous ammonia is actually a mixture of  $NH_{3(aq)}$  and water. The resulting solution is basic because the  $NH_3$  acts as a base, accepting protons from water:

(1) 
$$NH_3(aq) + H_2O(I) ---> NH_4^+(aq) + OH^-(aq)$$
  $K_b = 1.8 \times 10^{-5}$ 

To determine the mass percent of ammonia in a household cleaner, you can titrate a known mass of the cleaner with a strong acid, such as HCl:

(2) 
$$NH_3(aq) + HCl(aq) ---> NH_4^+(aq) + Cl^-(aq)$$

The pH at the equivalence point will be less than seven for such a titration because of hydrolysis:

(3) 
$$NH_4^+(aq) + H_2O(I) ---> NH_3(aq) + H_3O^+(aq)$$

The equivalence point can be determined potentiometrically, as you did in a previous experiment for acetic acid. However, the equivalence point may also be found *thermometrically*. Reaction (2) is exothermic, so heat is evolved during the titration. As heat is evolved, the temperature of the reaction mixture will rise. When the equivalence point is reached, reaction (2) is complete and heat is no longer evolved. The temperature of the mixture will stabilize, rising at a significantly slower rate or possibly dropping a bit. The point at which the temperature curve stabilizes or significantly changes slope is the equivalence point. It is important to note that the temperature rise associated with the amounts of solution you will use is relatively small, but ideally suited for measurement by a temperature probe and the *MicroLAB* interface.

In this project, you will quantitatively determine the percentage of ammonia in two popular liquid household cleaners. You will determine the equivalence points both potentiometrically and thermometrically.

## **EXPERIMENTAL PROCEDURES**

## Lab Interface Preliminaries

1. You should calibrate the temperature probe using ice water and hot tap water at a minimum of three different temperatures over the range of 0 to 50 °C. You must also calibrate the pH electrode using pH 4, 7 and 10 buffers before using.

## **Procedures**

You will need to determine how to best conserve the heat from the reaction during the thermometric titration and how fast the titration must be carried out in order to meet the conflicting requirements of speed to avoid heat loss but ensure good mixing and stirring speed.

You will also need to determine how large a sample you must take to get a good equivalence volume, and you will need to know the exact concentration of the acid used for the titration.

You will need to determine which titration method will best satisfy the conditions for the thermometric and the potientiometric titrations. As your instructor for a copy of **Useful Titration Operations**.

You must titrate samples of two household cleaners *at least* three times each, averaging the thermometric results and the potentiometric results to obtain a value for the mass percent of ammonia in each cleaner.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must obtain graphs to determine the equivalence points for your titrations. At a minimum, you must include the following graphs for each titration:
  - a. pH versus volume of HCl and the derivative of the pH vs. volume curve plotted on the same graph.
  - b. pH and temperature versus volume plotted on the same graph.
  - c. Temperature versus volume of HCl and the derivative of the pH vs. volume curve plotted on the same graph.
- 3. Include a table of results from all your titrations.
- 4. Calculate the mass percent of ammonia in both household cleaners using the potentiometric *and* thermometric equivalence points. Compare these results.
- 5. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

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#### **INDIVIDUAL PROJECT #4**

## THERMOMETRIC DETERMINATION OF AMMONIA IN HOUSEHOLD CLEANER

## INTRODUCTION/OBJECTIVE

Students will be using thermometric and potentiometric acid/base titration techniques to determine the percentage of ammonia in two common liquid household cleaners.

#### BACKGROUND

## See student handout for this information

In this project, students will quantitatively determine the percentage of ammonia in two popular liquid household cleaners. They will determine the equivalence points both potentiometrically and thermometrically.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

#### Lab Interface Preliminaries

Students should calibrate the temperature probe at a minimum of three temperatures, using ice water and hot tap water. They must also calibrate the pH electrode using pH 4, 7 and 10 buffers before using.

#### **Procedures**

- 1. To obtain good temperature data, students should perform the titration in two nested Styrofoam cups. They will be using 0.1 M HCl as the titrant. *Note the exact concentration of HCl on the bottle!*
- 2. They must determine how large of a sample (from a mass standpoint) of the household cleaner to use in order to get a "good" equivalence point. This will involve some thinking and/or "trial and error" on their part. Do <u>not</u> use straight cleaner! They must dilute however much they use by at least a factor of 5 with distilled water! The nested Styrofoam cup must contain at least 30 ml of diluted cleaner to ensure that the pH electrode is fully submerged.
- 3. Titrate the above solution with 0.1 M HCl. *Note the exact concentration of the HCl on the bottle!* The titration set-up is the same as was done in a previous experiment.
- 4. For the thermometric titration, they should perform a timed titration by letting the acid drop into the

analyte solution at a rate of about 1-2 drops per second with good stirring. Remember, they are measuring pH *and* temperature in this project. If they are using a Temp(IC) probe, then this must be covered with saran wrap to insulate the ground so as not to short out the pH probe. When the experiment is complete, they can convert time into volume, using a measured flow rate. This technique may take a little practice! It is important that the solution is well stirred throughout the titration.

5. They must titrate samples of two household cleaners *at least* three times each, averaging the thermometric results and the potentiometric results to obtain a value for the mass percent of ammonia in each cleaner.

## MINIMUM REQUIREMENTS

See student handout for this information.

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #5**

#### PHOSPHORIC ACID CONCENTRATIONS IN COLAS

## INTRODUCTION/OBJECTIVE

You will determine the phosphoric acid concentration in two cola products using acid/base titration techniques.

#### BACKGROUND

Before you begin this project, it is important that you have a good understanding of acids, bases, pH, and titrations. Be sure to review the chapter on acids and bases in your text.

Acids and bases are chemical compounds which we encounter constantly in our everyday lives. It is the acid content of many foods and beverages which contribute significantly to the taste. Soft drinks often contain varying quantities of several acids; in cola products, these acids are predominantly carbonic acid (from the carbonated water) and phosphoric acid. By de-carbonating the cola, you can determine the phosphoric acid concentration through titrimetric analysis. The phosphoric acid content is usually quite low, so good technique is critical.

## **EXPERIMENTAL PROCEDURES**

## **Lab Interface Preliminaries**

You must calibrate the pH electrode with pH 4, 7 and 10 buffers before using it.

## **Procedures**

You must determine the best way to de-carbonate the colas, and then how to obtain an exact volume to carry out your titration on.

You must determine the best concentration of base to use in the titration in order to obtain a good equivalence point.

You will need to determine which titration method to best satisfy the conditions for the thermometric and the potientiometric titrations. As your instructor for a copy of **Useful Titration Operations**. Be sure that you continue until you reach a pH of at least 10.

You should be able to see two equivalence points, and you should report the equations for each reaction represented by each equivalence point.

Using the data from the first equivalence point, calculate the concentration of phosphoric acid in the cola. You must titrate each of two colas *at least* three times, averaging the results and determine the standard deviation.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must titrate two different samples of colas 3 times each, and report your raw values, their average and the standard deviation in a table.
- 3. You must obtain graphs to determine the equivalence points for your titrations. At a minimum, you must include a graph of pH versus volume of NaOH and the derivative of the pH vs. volume curve plotted on the same graph. You must include these graphs for each titration.
- 4. Using the first equivalence point, calculate the concentration of phosphoric acid in each of the two colas you chose.
- 5. You may be asked to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #5**

## PHOSPHORIC ACID CONCENTRATIONS IN COLAS

## INTRODUCTION/OBJECTIVE

Students will determine the phosphoric acid concentration in two cola products using acid/base titration techniques.

#### BACKGROUND

See student handout for this information

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

## Lab Interface Preliminaries

Students will need to calibrate the electrode with pH 4, 7 and 10 buffers before using it.

## **Procedures**

- 1. Pour about 100 ml of cola into a clean, dry 250-mL beaker. Measure the pH of the cola at room temperature. To prepare the cola for titration, it must first be de-carbonated. Cover the beaker containing the cola with a watch glass and boil for about 20 minutes. This will remove the carbon dioxide from the cola, which would interfere with the phosphoric acid titration. Measure the pH of the de-carbonated cola after it has cooled to room temperature.
- 2. Using a 25-mL volumetric pipette, place 25.0 ml of room temperature, de-carbonated cola into a 150-mL beaker. Place a *calibrated* pH electrode and a stirring bar into the beaker with the cola.
- 3. The students should be helped to discover that the cola needs to be titrated with **0.01 M NaOH**. Note the exact concentration of the NaOH on the bottle! Prepare to titrate the sample with standard 0.01 M base. Help the students understand the need to rinse their buret 3 times with the standard base provided; be sure and record the concentration in your lab notes. **Be careful that they take the right standard base solution!!** Several other projects also use standard base, but no other uses a solution as weak as this. They may use the same titration technique as used in a previous experiment, or try another one as given in the **Useful Titration Techniques**.

When the cola sample is ready, insert the pH electrode in the beaker, set up the program for the titration, and proceed. Be sure that you continue until you reach a pH of at least 10.

Graph and work with these results on the spreadsheet, but they will need to decide exactly how they want to present their results in their reports.

4. They should be able to see two equivalence point regions and report the equations for these two, one for each of the following reactions:

$$H_{3}PO_{4}(aq) + OH^{\text{-}}(aq) ---> H_{2}PO_{4}^{\text{-}}(aq) + H_{2}O(l)$$

$$H_2PO_4(aq) + OH(aq) ---> HPO_4(aq) + H_2O(1)$$

5. Using the data from the first equivalence point, they will calculate the concentration of phosphoric acid in the cola. They must titrate each of two colas *at least* three times, averaging the results.

## MINIMUM REQUIREMENTS

See student handout for this information

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #6**

#### MEASURING THE CALORIC CONTENT OF SNACK FOODS

## INTRODUCTION/OBJECTIVES

You are to determine the caloric (heat) content of a number of snack food products by burning them in a special calorimeter which you will need to construct. You will also need to calibrate the calorimeter by burning a pure substance for which you can calculate the  $\Delta H_{combustion}$ .

## **BACKGROUND**

Food provides two things for us -- molecules and atoms which act as building blocks for new and replacement cells, and energy to keep our metabolism going. The amount of energy that can be produced by metabolism of a relatively small amount of food is sometimes quite surprising. The problem involved in this project is to determine the amount of heat that is produced as the food is burned. While chemical reactions in our bodies consume the food in a little different manner, the overall effect is the same: relatively complex molecules are broken into simpler molecules with a concurrent liberation of heat. Your experimental problem for this project is to determine the number of calories of heat that are produced by combustion per gram of each snack food. For this project you will use calorimetric measurements to compare several snack food products. You will also analyze sources of error in your measurements and devise a method to "correct" your measurements for some kinds of error.

## **EXPERIMENTAL PROCEDURES**

You will burn various snack foods in the laboratory and measure the heat released as a change in the temperature of a weighed water sample. The heat produced by this burning can be captured by a small metal can containing water suspended above the burning food. A Vienna Sausage can makes a good container. It is light, and the specific heat of the metal is about 0.1 cal/g-degree C. It also fits easily into the top of a soda or juice can which can be used as a support and a wind and heat screen. You will need to devise a way to hold the burning food close enough to the bottom of the calorimeter can to maximize the amount of heat energy captured and so that it does not fall off as it burns. You also might want to consider how to minimize heat loss from the calorimeter. If there is any unburned material remaining afterwards, you will need to determine how to compensate for this.

You should identify possible sources of heat loss and other experimental errors, and set up your experiment to minimize these. How do you compensate for the mass of the metal cup, which has a different specific heat than water?

Calibration is one of the most important aspects of making quantitative measurements. You must determine some manner of calibrating your calorimeter system in order to relate the temperature changes you will be measuring back to the number of calories or joules of energy released by the burning process. This

is usually done by choosing some substance for which the enthalpy of formation is accurately known, and from which you can calculate the enthalpy of combustion, then calibrate the system by burning a known mass of it in the calorimeter.

## **Data Collection:**

Devise a computer program that will allow you to monitor the water temperature as the snack food burns. Besides the change in temperature and the mass of the snack food, what other information will you need to compute the calorie production per gram of snack food burned? Does all of the snack food burn, or will you have to take into account any unburned material when you do your calorie calculation? Where does your experimental set-up have opportunity for loss of energy? Repeat each snack food two or three times to see if the calorie production per gram is relatively constant.

## MINIMUM REQUIREMENTS

The following are minimum requirements for completion of this project:

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, see your copy of **Independent Inquiry Experiment Introduction**, and obtain from your instructor copies of the following: **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. Perform calorimetric measurements on several samples of the same snack food and analyze your results to determine the effects of random errors in your measurement techniques.
- 3. Perform calorimetric measurements on three or four different snack foods using three trials for each food product. Compare the averaged experimental results, in calories per gram, for each product to the nutritional information on the product label. Using the label information as known standards, analyze your experimental measurements for systematic errors.
- 4. After considering possible sources of systematic errors in your experimental techniques and the information obtained from the calibration procedure, calculate a "correction factor" for systematic errors that can be applied uniformly to your experimental results to give better agreement with the calorie information on the product labels.
- 5. If time permits, test your correction factor by performing measurements on one or two additional products, correcting your results for systematic errors and comparing your adjusted results to the product labels.
- 6. Your report should include sample calculations for your calibration procedure, along with sample calculations converting measured temperatures and masses to calories per gram of food product, tables of measured and calculated values for various snack foods and discussion of errors and "corrections" for systematic errors.
- 7. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

Some useful conversion factors:

1 ounce = 28.35 grams

1 food calorie = 1000 heat calories

## INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #6**

## MEASURING CALORIC CONTENT OF SNACK FOODS

## INTRODUCTION/OBJECTIVES

Students are to determine the caloric heat content of a number of snack food products by burning them in a special calorimeter which they will need to construct. They will also need to calibrate the calorimeter by burning a pure substance for which they can calculate the  $\Delta H_{combustion}$ .

## **BACKGROUND**

See student handout for this information

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

Students will burn various snack foods in the laboratory and measure the heat released as a change in the temperature of a weighed water sample. The heat produced by this burning can be captured by a small metal can containing water suspended above the burning food. A Vienna sausage can makes a good container. It is light, and the specific heat of the metal is about 0.1 cal/g-degree C. It also fits easily into the top of a soda or juice can which can be used as a support and a wind and heat screen. They will need to devise a way to hold the burning food close enough to the bottom of the calorimeter can to maximize the amount of heat energy captured and so that it does not fall off as it burns. They also might want to consider how to minimize heat loss from the calorimeter. If there is any unburned material remaining afterwards, they will need to determine how to compensate for this.

They should identify possible sources of heat loss and other experimental error, and set up the experiment to minimize these. Do they compensate for the mass of the metal cup, which has a different specific heat than water?

Calibration is one of the most important aspects of making quantitative measurements. They must determine some manner of calibrating your calorimeter system in order to relate the temperature changes they will be measuring back to the number of calories or joules of energy released by the burning process. This is usually done by choosing some substance for which the enthalpy of formation is accurately known, and from which you can calculate the enthalpy of combustion, then calibrate the system by burning a known mass of it in the calorimeter. Methanol or ethanol are good choices for this.

## **Data Collection:**

Students will devise a computer program that will allow you to monitor the water temperature as the snack food burns. Besides the change in temperature and the mass of the snack food, what other information will you need to compute the calorie production per gram of snack food burned? Does all of the snack food burn, or will they have to take into account any unburned material when they do your calorie calculation? Where does your experimental set-up have opportunity for loss of energy? Repeat each snack food two or three times to see if the calorie production per gram is relatively constant.

## MINIMUM REQUIREMENTS

See student handout for these.

Some useful conversion factors:

1 ounce = 28.35 grams

1 food calorie = 1000 heat calories

## Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### INDIVIDUAL PROJECT #7

#### MOLAR ENTHALPYS OF ACID/BASE REACTIONS

## INTRODUCTION/OBJECTIVES

When acids and bases react, there is a change of enthalpy. You are to determine the sign and magnitude of these changes on a molar basis for several different acid base reactions.

## BACKGROUND

You should have previously been introduced to the concept of determining enthalpy changes. You should review these sections of your text before proceeding further. Using the temperature probe and coffee cup calorimeter, you can determine the molar enthalpy (or heat) of reaction of several acid/base reactions. For each reaction, determine the heat produced by reaction of 1 mole of aqueous acid with 1 mole of aqueous base (note that you do <u>not</u> need to use a mole of each in the reactions that you do!). The reactions you will perform are:

hydrochloric acid + sodium hydroxide

nitric acid + sodium hydroxide

acetic acid + sodium hydroxide

You will also react these acids with the base potassium hydroxide to determine the heat of these reactions.

## **EXPERIMENTAL PROCEDURES**

You will need to write or utilize a computer program for the *MicroLAB* interface to measure the temperature change during the reaction of measured volumes of the acid and base solutions.

You will need to calculate the volumes of each solution to use to determine a useful temperature change.

You will need to calculate the moles of reactants used, since you know how much solution you used and the solution concentration (about 1 M, but check the bottles to get the exact concentrations).

You will need to do three determinations of the enthalpy of reaction for each acid/base combination.

From the temperature change, you can determine the heat produced from your reaction and calculate the enthalpy change per mole.

You should also calculate the molar enthalpy of reaction for each reaction using the enthalpys of formation

in your textbook or another source. Compare your theoretical enthalpy of reactions to those that you determined in the experiment.

Your report will include your experimental determination of the enthalpys of reaction, your theoretical enthalpys of reaction determined from the textbook enthalpys of formation, and an explanation of any discrepancies between these values.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must react two different samples of each, and report your raw values and their averages in a table.
- 3. You must obtain thermometric graphs to determine the enthalpys for your titrations.
- 4. Using these graphs to determine the temperature change, calculate the enthalpy of neutralization for each of the acids titrated.
- 5. You should also calculate the molar enthalpy of reaction for each reaction using the enthalpys of formation in your textbook or another source.
- 6. You will need to compare your theoretical enthalpy of reactions to those that you determined in the experiment.
- 7. You may be asked to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #7**

## MOLAR ENTHALPIES OF ACID/BASE REACTIONS

## INTRODUCTION/OBJECTIVES

When acids and bases react, there is a change of enthalpy. Students are to determine the sign and magnitude of these changes on a molar basis for several different acid base reactions.

#### BACKGROUND

See student hand out for these.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

Students will need to write or utilize a computer program to measure the temperature change during the reaction of measured amounts of the acid and base solutions. They will need to calculate the moles of reactants used, since they know how much solution they used and the solution concentration (about 1 M, but check the bottles to get the exact concentrations). From the temperature change, they can determine the heat produced from your reaction and calculate the enthalpy change per mole. They should also calculate the molar enthalpy of reaction for each reaction using the enthalpys of formation in their textbook or another source. Compare their theoretical enthalpy of reactions to those that they determined in the experiment. Do three determinations of the enthalpy of reaction for each acid/base combination.

Their report will include their experimental determinations of the enthalpys of reaction, their theoretical enthalpys of reaction determined from the textbook enthalpys of formation, and an explanation of any discrepancies between these values.

## MINIMUM REQUIREMENTS

See student hand out for these.

## Dale A. Hammond, PhD, Brigham Young University Hawaii

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#### INDIVIDUAL PROJECT #8

#### ENTHALPYS OF SOLUTION

## INTRODUCTION/OBJECTIVES

When solutes dissolve in a solvent to form a solution, there is usually an energy exchange. In this experiment, you will determine the sign and magnitude of this energy change for several salts.

## **BACKGROUND**

You should have previously been introduced to the concept of determining enthalpy changes. You should review these sections of your text before proceeding further. Using a temperature probe and coffee cup calorimeter, you can determine the molar enthalpy (or heat) of solution of several salts. For each salt, determine the heat produced by reaction of an accurately weighted amount of the solid salt with a quantitatively measured amount of water. The salts you will study are:

Ammonium Nitrate NH4NO3 Sodium Chloride NaCl Sodium Hydroxide NaOH

## **EXPERIMENTAL PROCEDURES**

You will need to write or utilize a computer program to measure the temperature change during the dissolution of measured amounts of each of the salts.

You will need to decide the best experimental set up to get accurate heat measurements, minimize heat loss, and proper stirring of the solutions.

You will know the moles of reactants used, since you know the number of grams of salt you used. From the temperature change, you can determine the heat produced from your reaction and calculate the enthalpy change per mole.

You will need to do three determinations of the enthalpy of solution for each salt and average their results.

Your report will include graphs of each of your experimental determinations of the enthalpys of solution, sample calculations for determining the enthalpy of solution and an explanation of any differences between the values for each salt.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must measure the heat loss of at least two different samples of each salt, and report your raw values and their averages in a table. If there is more than 2% difference in the duplicates, you must do at least one more measurement on that system.
- 3. You must use the *MicroLAB* to obtain graphs to determine the change in temperature of the system. You must include these graphs for each salt sample.
- 4. Using the temperature change, calculate the enthalpy of solution for each salt, along with the % error in the measurements.
- 5. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

## **INDIVIDUAL PROJECT #8**

## ENTHALPYS OF SOLUTION

## INTRODUCTION/OBJECTIVES

When solutes dissolve in a solvent to form a solution, there is usually an energy exchange. In this experiment, students will determine the sign and magnitude of this energy change for several salts.

## **BACKGROUND**

See student hand out for this

EXPERIMENTAL PROCEDURES (Students should be guided to these procedures and results.)

They will need to write or utilize a computer program to measure the temperature change during the dissolution of measured amounts of each of the salts. They will calculate the moles of reactants used, since they know the number of grams of salt they used. From the temperature change, they can determine the heat produced from their reaction and calculate the enthalpy change per mole. Do three determinations of the enthalpy of solution for each salt.

Their report will include graphs of each of their experimental determinations of the enthalpys of solution, sample calculations for determining the enthalpy of solution and an explanation of any differences between the values for each salt.

## MINIMUM REQUIREMENTS

See student hand out for these.

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### INDIVIDUAL PROJECT #9

#### DETERMINATION OF A MIXTURE OF COPPER AND COBALT BY COLORIMETRY

## INTRODUCTION/OBJECTIVES

Colorimetry is a commonly used technique for finding the concentrations of compounds in materials. In this project you will (1) plot the absorption curves of a copper and a cobalt salt solution, (2) determine the best wavelength to carry out a quantitative analysis of an unknown of each salt, and (3) determine the concentration of an unknown of each salt.

#### BACKGROUND

You may have used colorimetry in an earlier experiment to find the concentration of an unknown solution. You should review the information in that experiment at this time to refresh your memory. This time we will use *MicroLAB* Colorimeter Experiment to determine the concentrations of copper (II) and cobalt (II) in unknown solutions.

## EXPERIMENTAL PROCEDURE

Your first goal is to obtain a spectrum for copper and for cobalt, by measuring absorbance versus wavelength. After scanning the blank in the *MicroLAB* colorimeter, scan the full strength copper and cobalt solutions to obtain their spectral displays. Convert the spectral display from percent transmission to absorbance by clicking on absorbance in the top bar. Examine each spectrum carefully and determine the best wavelength for your analysis (the wavelength of greatest absorbance) for each species.

You will now need to dilute the standard solutions to make a calibration curve for copper and for cobalt. Make 5 different concentrations of copper (II) solution ranging from 0.20 M to 1.00 M, and 5 different concentrations of cobalt (II) solution ranging from 0.20 to 1.00 M. Measure the transmittance of copper (II) at your chosen wavelength for all five solutions. Use the same cuvette for all of the measurements, each time rinsing it with the solution to be measured. Convert the transmittance of your standards to absorbance and do a Linear Curve fit to the data. This gives you a calibration curve for copper (II). Repeat this process for the cobalt (II) solution. Measure the absorbance of the unknown mixture of copper (II) and cobalt (II) at the wavelength at which you ran the copper (II) calibration curve, then measure the absorbance of the unknown mixture at the wavelength at which you ran the cobalt (II) calibration curve.

Now use these absorbencies to find the concentration of the copper (II) in the unknown mixture of copper (II) and cobalt (II) using the copper (II) calibration curve and the concentration of the cobalt (II) using the cobalt (II) calibration curve.

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. Include your spectra for copper (II) and cobalt (II), your calibration curves for each with Linear Curve fits, the concentrations of the copper (II) and cobalt (II) in your mixed unknown, and the spectrum of your mixed unknown.
- 3. a discussion of any errors in these unknown concentrations due to interferences of the absorptions of one ion and the wavelength analysis of the other ion in your report.
- 4. You may by asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

## INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

## **INDIVIDUAL PROJECT #9**

#### DETERMINATION OF A MIXTURE OF COPPER AND COBALT BY COLORIMETRY

## INTRODUCTION/OBJECTIVES

Colorimetry is a commonly used technique for finding the concentrations of compounds in materials. In this project students will (1) plot the absorption curves of a copper and a cobalt salt solution, (2) determine the best wavelength to carry out a quantitative analysis of and unknown of each salt, and (3) determine the concentration of an unknown of each salt.

## **BACKGROUND**

They may have used colorimetry in an earlier experiment to find the concentration of an unknown solution. They should review the information in that experiment at this time to refresh their memory. They will use *MicroLAB* Colorimeter Experiment to determine the concentrations of copper (II) and cobalt (II) in unknown solutions.

**EXPERIMENTAL PROCEDURE** (Students should be guided to these procedures and results.)

NOTE: Both copper and cobalt are considered toxic to the environment! You can substitute blue food coloring and red food coloring with exactly the same results, and the waste can be flushed down the drain by the instructor after the labs are finished. It is recommended that they be labeled and treated as though they were copper and cobalt solutions for the training of the students in using environmentally toxic materials.

**NOTE:** solutions should be made up so that they give an absorbance of about 1.2 absorbance units in the *MicroLAB* colorimeter and labeled as 1.0 M solutions, then the diluted standards will all fall into the acceptable absorbance range.

Their first goal is to obtain a spectrum for copper and for cobalt, by measuring absorbance versus wavelength using the *MicroLAB* colorimeter. Follow the instructions in the **Measurement Manual**, or from a previous experiment. Repeat this with the cobalt solution Change transmittance to absorbance by clicking on the **Absorbance** tab in the upper task bar. This performs the following operation:

absorbance =  $-\log (\% T/100)$ 

Students should determine the best wavelength for their analysis (the wavelength of greatest absorbance) for each species.

They will need to dilute the standard solutions to make a calibration curve for copper and for cobalt. Make 5 different concentrations of copper (II) solution ranging from 0.20 M to 1.00 M, and 5 different concentrations of cobalt (II) solution ranging from 0.20 to 1.00 M. Measure the transmittance of copper (II) at their chosen wavelength for all five solutions, plus the transmittance of the unknown. Use the same cuvette for all of the measurements, each time rinsing it with the solution to be measured. Convert the transmittance of their standards to absorbance, to obtain the graph of absorbance versus concentration of copper (II). This gives them a calibration curve for copper (II). Repeat this process for the cobalt (II) standard solutions. Measure the absorbance of the unknown mixture of copper (II) and cobalt (II) at the wavelength at which you ran the cobalt (II) calibration curve.

Now use these absorbencies to find the concentration of the copper (II) in the unknown mixture of copper (II) and cobalt (II) using the copper (II) calibration curve and the concentration of the cobalt (II) using the cobalt (II) calibration curve.

If you have a particularly good set of students doing this experiment, you might have them do a confirmation experiment by obtaining the spectrum of the 1.00 M sample of copper and cobalt in the **Color Mixer Experiment** program, along with their unknown, then have them electronically mix the copper and cobalt solutions, and using the percentage of mixture slider, match the digitally mixed spectrum with the unknown spectrum to obtain the percentages of copper and cobalt in the unknown. Fine adjustments for exact matching can be made on the slider by using the up and down arrows on the keyboard. Use these percentages to calculate the concentration of copper and cobalt in the unknown by multiplying the fraction by the initial concentration of the standard solutions.

## MINIMUM REQUIREMENTS

See student hand out for these.

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #10**

#### IDENTITY OF GROUP 1 CARBONATE SAMPLES

#### INTRODUCTION/OBJECTIVES

This is an experiment in using masses and changes in mass to determine the identity of group 1 elements in unknown carbonate compounds. Your task will be to identify three unknown carbonate salts by weight measurements alone.

#### BACKGROUND

Reacting a metal carbonate salt, using "M" as a generic symbol for a group 1 metal with a solution of hydrochloric acid (HCl), carbon dioxide gas is released from the reaction:

$$M_2CO_3 - 2 HC1 \longrightarrow 2MC1 + H_2O + CO_2(g)$$

If you weigh (a) the starting metal carbonate sample, (b) the starting mass of 1 M HCl and  $\mathbb{C}$ ) the mass of the final solution, you can calculate:

- 1. the mass of CO<sub>2</sub> produced and hence the moles of CO<sub>2</sub>
- 2. the moles of metal carbonate and hence the moles of metal M you began with
- 3. the molar mass and thus the identity of metal M.

This experiment will require careful experimental technique. You will make calculations involving mass and moles. As usual in quantitative work, you will need to perform three determinations for each unknown and will average the molar masses found to get a final result for each unknown and the standard deviation. You will analyze three different unknowns (3 times each). The group 1 metals in the three unknown carbonates are lithium, sodium and potassium.

#### EXPERIMENTAL PROCEDURES

One important decision is left to you. How big of a sample of your unknowns do you need to use and how much HCl should you add? This can be decided in a lot of ways, but it basically boils down to needing to produce enough  $CO_2$  to get a measurable increase in pressure from the reaction. However, using more of any chemical than necessary is wasteful and environmentally irresponsible. Since your results will be based on differences in mass, you will need to determine the balance sensitivity needed to minimize the amounts of chemicals needed. In order to identify the three metal carbonates you will need experimental precision to at least two significant figures. If you use enough metal carbonate to produce from 300 to 400 torr increase of pressure by the released  $CO_2$ , you will get three significant figures with some error in the third figure. Before

doing the experiment you should calculate the amounts of metal carbonate needed to produce this pressure change from Li<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub>. From these calculations you should be able to determine an amount of unknown metal carbonate that would produce that pressure change from any of the three possible unknowns.

Having determined an appropriate sample size for the metal carbonate, you can calculate the amount of 1.0 M HCl needed to react with that amount of each of the three possible carbonates. You may ask your lab instructor to check your calculations before beginning the experiments. You should perform the analysis of each salt in duplicate, and if they don't agree, then perform a third analysis.

#### MINIMUM REQUIREMENTS

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. Your report on this project must include a table of all of the raw data, measured and calculated results for each salt,
- 3. a description of the calculations performed and your rationale for choice of sample sizes, as well as your identification of the three unknowns.
- 4. You must also discuss possible sources of error in your measurements.
- 5. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

# INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #10**

#### **IDENTITY OF GROUP 1 CARBONATE SAMPLES**

#### INTRODUCTION/OBJECTIVES

This is an experiment in using masses and changes in mass to determine the identity various unknown carbonate compounds of group 1 elements. The student's task will be to identify three unknown carbonate salts by weight measurements alone.

#### BACKGROUND

See student hand out for these.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

Students will need to determine how big of a sample of the unknowns do they need to use and how much HCl should be added? This can be decided a lot of ways, but it basically boils down to needing to produce enough CO<sub>2</sub> to get a good pressure change from the reaction. However, using more of any chemical than necessary is wasteful, environmentally irresponsible and could cause dangerous exploding problems from the pressure. Since their results will be based on differences in mass, they can minimize the amounts of chemicals needed by using the most sensitive balance available in the laboratory --- one that weighs to 0.001 grams. In order to identify the three metal carbonates they will need experimental precision to at least two significant figures. If they use enough metal carbonate to produce a change of 300 - 400 torr from the CO<sub>2</sub>, they will get three significant figures with some error in the third figure. Before doing the experiment you should calculate the amounts of metal carbonate needed to produce this change in pressure from Li<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>CO<sub>3</sub> and K<sub>2</sub>CO<sub>3</sub>. From these calculations they should be able to determine an amount of metal carbonate that would produce the same pressure from any of the three possible unknowns.

Having determined an appropriate sample size for the metal carbonate, they can then calculate the amount of 1.0 M HCl needed to react with that amount of each of the three possible carbonates. Since the reaction requires an excess of HCl to completely react with the carbonate, they should use about twice the calculated amount of HCl needed for the metal carbonate that requires the most HCl. They may ask their lab instructor to check their calculations before beginning the experiments. They should perform the analysis of each salt in duplicate, and if they don't agree, then perform a third analysis.

## **MINIMUM REQUIREMENTS:**

See student hand out for these.

A variation on this project would be to use the MicroLAB pressure sensor to measure the increase in pressure of the released  $CO_2$ , which is proportional to the number of moles of  $CO_2$  produced. The most  $CO_2$  will be produced by the compound with the lowest formula weight. If the students take the same mass for each of the three unknown salts, then the pressure increase will be inversely proportional to the formula mass of the salt, i.e., the potassium salt will produce the least increase in pressure and the lithium salt will produce the greatest increase in pressure.

Here again, the students will have to calculate a safe increase in pressure and the corresponding mass of salt and acid.

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #11**

#### FACTORS AFFECTING FREEZING POINT DEPRESSION IN WATER

#### INTRODUCTION/OBJECTIVE

Any one who has ever made fudge or other candy is familiar with the fact that the longer one boils the mixture, the higher the temperature becomes. However, when one boils pure water, the temperature remains constant until all of the water has been boiled away. Why is this so? Likewise, everyone who has owned a car is aware that if the cooling system is not working correctly, the car overheats in the summer, or the radiator freezes and bursts in the winter. One way of reducing both of these likelihoods is to add "antifreeze" to the radiator, which helps on both ends of the temperature range. How does "antifreeze" help solve this problem? To help understand these observations, you will study solutions of one molecular compound and two ionic compounds. For the two ionic compounds, you must choose one that will furnish two ions in solution, and another that furnish four ions in solution.

#### **BACKGROUND**

You are encouraged to review the chapter in your text dealing with colligative properties of solutions before beginning this investigation. You should also review the chapter on concentration of solutions so you thoroughly understand the definitions. In addition, review the sections discussing molecular versus ionic compounds, and what happens to them upon the dissolution process.

#### **EXPERIMENTAL**

#### **Lab Interface Preliminaries**

You will be using a temperature probe to measure temperature. You should carefully calibrate the temperature probe at a minimum of three points between -5  $^{\circ}$ C (using a salt - ice slush) and about 50  $^{\circ}$ C to get the best linear data.

#### **Procedures**

You will need to determine the best way to obtain the necessary data while minimizing the loss of heat to the outside.

In consultation with your instructor, you will first determine the one molecular and three ionic compounds that you will investigate. You and your partner should review the appropriate chapters in your text, then decide which compounds will accomplish the desired results. You should choose ionic compounds that will furnish two and four ions in solution

You and your partner will need to determine how large a sample (from a mass standpoint) must be taken of

the compounds to be studied in order to get a "good" temperature change. This will involve some thinking or "trial and error" on your part. Your nested test tubes must contain about 5 ml of solution in order to obtain a good freezing point. You are not limited to making 5 ml of solution, but you should not be wasteful of the compounds because they are expensive to buy, even more expensive to dispose of, and pollute the environment if poured down the drain.

You will need to determine the procedure that will give you the best results. For example, how vigorously should the solution be stirred? How will you compensate for supercooling? Will you need to compensate for heat gain by the system by back extrapolation? Etc.

You should do at least duplicates of each solution, and you should do at least 3 different concentrations of each compound in order to really understand what is going on.

## MINIMUM REQUIREMENTS

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must include a table of results for all of your solutions in your write up, including compound, concentration and freezing point.
- 3. You must submit graphs of your freezing point experiments, and use these for determining the freezing points of the solutions.
- 4. You should try to explain the differences in the freezing points of the three compounds studied.
- 5. You may be asked by your instructor to give a short presentation (~ 5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

# INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #11**

#### FACTORS AFFECTING FREEZING POINT DEPRESSION IN WATER

#### INTRODUCTION/OBJECTIVE

Any one who has ever made fudge or other candies is familiar with the fact that the longer one boils the mixture, the higher the temperature becomes. However, when one boils pure water, the temperature remains constant until all of the water has been boiled away. Why is this so? Likewise, everyone who has owned a car is aware that if the cooling system is not working correctly, the car overheats in the summer, or the radiator freezes and bursts in the winter. One way of reducing both of these likelihoods is to add "antifreeze" to the radiator, which helps on both ends of the temperature range. How does "antifreeze" help solve this problem? To help understand these observations, you will study solutions of one molecular compound and two ionic compounds. For the two ionic compounds, you must choose one that will furnish two ions in solution, and another that furnish four ions in solution.

#### **BACKGROUND**

See student hand out for this.

**EXPERIMENTAL** (Students should be guided to these procedures and results.)

#### Lab Interface Preliminaries

Students will be using a temperature probe to measure temperature. They should carefully calibrate the temperature probe at a minimum of three temperatures between -5 °C (using a salt - ice slush) and about 50 °C to get the best linear data.

#### **Procedures**

- 1. To obtain good temperature data, students should perform the determination in two nested test tubes immersed into the ice bath, with the temperature probe immersed into the internal solution.
- 2. In consultation with their instructor, they will first determine the molecular and three ionic compounds that they will investigate. They should review the appropriate chapters in their text, then decide which compounds will accomplish the desired results.
- 3. They must then determine how large a sample (from a mass standpoint) must be taken of the compounds to be studied in order to get a "good" temperature change. This will involve some thinking or "trial and error" on their part. The nested test tubes must contain about 5 ml of solution in order to obtain a good

freezing point.

- 4. Once they have determined the correct amounts of solute and solvent to use, they will need to determine the procedure that will give them the best results. For example, how vigorously should the solution be stirred? How will they compensate for supercooling? Will they need to compensate for heat gain by the system by back extrapolation? Etc.
- 5. They should do at least duplicates of each solution, and they should do at least 3 different concentrations of each compound in order to really understand what is going on.

## MINIMUM REQUIREMENTS

See student hand out for these.

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### INDIVIDUAL PROJECT #12

#### PENETRATING ABILITY OF BETA AND GAMMA RADIATION

#### INTRODUCTION/OBJECTIVE

You are to explore the penetrating ability of beta and gamma radiation in terms of the (1) the areal density of the material penetrated, i.e., the mg of matter per square centimeter, and the change in radiation flux with (2) distance and (3) time.

#### BACKGROUND

Before you begin this project, it is important that you have a good understanding of the nature of beta and gamma radiation. Only some basics will be covered in this handout. Consult your text for the chapter on radiochemistry or nuclear energy.

Nuclear radiation is detected by observing its interaction with matter. Since the energy of nuclear radiation is so high -- several hundred thousand times as much as the energy produced by a flashlight battery -- the passage of alpha, beta, or gamma radiation through matter knocks many electrons from the atoms near the path of the radiation. Detection of radiation can be accomplished by simply collecting and counting these electrons.

The Geiger-Muller tube is one of the simplest and most common detectors of nuclear radiation. The counter you will use in this experiment uses this kind of detector. A G-M tube consists of a cylindrical glass or metal envelope with a wire stretched down the center. This wire is connected to a very high voltage -- usually on the order of 900 volts. The return lead from the battery is connected to the metal coating on the inside of the tube. The tube is filled with a gas such as argon.

Each time nuclear radiation passes through the tube, atoms are ionized and free electrons are produced. These electrons are collected at the central wire electrode and passed through the counting circuits to the battery. An equal number of electrons are pushed out the negative side of the battery to the case of the G-M tube, where they recombine with the positive ions which were formed in the tube.

In a Geiger counter, the counting circuits count individual groups of electrons collected with the passage of each beta particle or gamma ray (alpha particles cannot penetrate the thin window of the Geiger tube.)

Another factor you will need to consider is the natural radioactive background. There are many naturally radioactive elements in our surroundings contributing to background radiation we are all exposed to. Some of these are <sup>14</sup>C, <sup>40</sup>K, in our bodies, and Uranium and its daughter products in the soil and rocks. Background radiation is usually measured by taking a long count time, such as 10 or 100 minutes, then dividing the total counts by the count time to get an average counts per minute (cpm).

#### EXPERIMENTAL PROCEDURES

Your instructor will provide a G-M counter system and the appropriate radioactive sources and teach you how to use it. Some of the variables that you might explore are:

- 1. Determine the background cpm for your local environment.
- 2. Chose three different materials of widely differing areal densities (i.e., mg material per cm<sup>2</sup>) and determine the penetrating ability of both beta and gamma radiation.
- 3. Explore the effect of distance of the source from the counter on the counts measured.
- 4. Explore the effect of time, i.e., how does the radiation from a given sample change with time.
- 5. Explore the radioactivity of various objects in our environment.

#### MINIMUM REQUIREMENTS

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. Graphs of counts vs. areal density and the natural logarithm of counts vs. areal density for each of the materials used in #2 above.
- 3. Graphs of the effect of distance vs. counts, and time vs. counts for #s 3 and 4 above.
- 4. The radioactivity observed for some items in our surroundings.
- 5. You may be asked by your instructor to give a short presentation (~ 5 minutes) about your project to your lab instructor and the rest of the class during the last lab period.

# INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDIVIDUAL PROJECT #12**

#### PENETRATING ABILITY OF BETA AND GAMMA RADIATION

#### INTRODUCTION/OBJECTIVE

Students are to explore the penetrating ability of beta and gamma radiation in terms of the (1) the areal density of the material penetrated, i.e., the mg of matter per square centimeter, and the change in radiation flux with (2) distance and (3) time.

#### **BACKGROUND**

See student hand out for this.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

The instructor will provide a G-M counter system and the appropriate radioactive sources and teach them how to use it. Some of the variables that they might explore are:

- 1. Determine the background cpm for your local environment.
- 2. Chose three different materials of widely differing areal densities (i.e., mg material per cm²) and determine the penetrating ability of both beta and gamma radiation.
- 3. Explore the effect of distance of the source from the counter on the counts measured.
- 4. Explore the effect of time, i.e., how does the radiation from a given sample change with time.
- 5. Explore the radioactivity of various objects in our environment and determine their emission type, e.g., whether they are beta, gamma or beta and gamma emitters.

# MINIMUM REQUIREMENTS

See student hand out for these.

Dale A. Hammond, PhD, Brigham Young University Hawaii

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#### **INDEPENDENT INVESTIGATION # 13**

#### COLORIMETRIC DETERMINATION OF PHOSPHOROUS IN PLANT FOOD

#### INTRODUCTION/OBJECTIVE

Students will determine the phosphorus content in three different brands of plant food. For each brand, two trials must be run and if the results do not agree, you may need to do third trial. The chemical reagents that will be supplied in the laboratory for this project are commercial analysis packets that are commonly used for such analyses, even by gardeners.

#### BACKGROUND

Plants require essential nutrients supplied by fertilizer in a form that can be utilized by the plant. The three important nutrients commonly supplied in plant foods are nitrogen, phosphorus and potassium. Labels display three numbers that indicate their percentages: nitrogen as % N; phosphorus as %  $P_2O_5$ ; potassium as %  $K_2O$ . (A label that reads 15-30-15 indicates 15% N, 30%  $P_2O_5$  and 15%  $K_2O$ .)

#### EXPERIMENTAL PROCEDURE

The experimental procedure used to determine phosphorus content is as follows:

For each different plant food, you will need to do the following:

- 1. Determine the amount of plant food for each type that you will need to dissolve quantitatively in 100 ml of de-ionized water to perform these analyses.
- 2. Determine the amount of Na<sub>3</sub>PO<sub>4</sub> required to make a standard solution 20% more concentrated than needed in step 1.
- 3. Obtain five standard solutions ranging up to the concentration of the solution in step 2 by dilution of the original concentration.
- 4. Follow the instructions on the commercial phosphate analysis kit to measure the absorbance of each of the standards produced in step 3 to obtain a Beer's Law plot.
- 5. For each different brand of plant food, make three solutions using the mass and volume calculated in step 1 and measure each of these against the Beer's law plot from step 4.
- 6. Use the concentration of the  $PO_4^{3-}$  to calculate the number of grams of phosphorus per gram of each plant food sample, the grams of  $P_2O_5$ , the  $P_2O_5$  percentage for each plant food sample and the difference between the measured and label vales of  $P_2O_5$ .

## MINIMUM REQUIREMENTS

1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, aks your instructor for copies of **Independent** 

# Inquiry Experiment Introduction, Making Oral and Written Presentations and Organizing the Research and Its Report.)

- 2. Submit *MicroLAB* Colorimeter Experiment Main Screen print outs for your phosphorus standards, tabulate the concentration of  $PO_4^{3-}$  for each trial.
- 3. Your report on this project must include all of the calculations for one of your trials and a summary table in the discussion section of each type of plant food including the sample weight, concentration of  $PO_4^{3-}$ , grams of phosphorous, grams of  $P_2O_5$ , the percent  $P_2O_5$  for each trial of each plant food
- 4. You must also compare your averaged results for each brand of plant food to the P<sub>2</sub>O<sub>5</sub> percentages indicated on the package labels, calculate the percent difference between them and discuss possible sources of error in your measurements.
- 5. You are to evaluate which of the plant foods you tested would be best for plants based solely on the percent P<sub>2</sub>O<sub>5</sub> contained therein.
- 6. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period

# INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDEPENDENT INVESTIGATION # 13**

#### COLORIMETRIC DETERMINATION OF PHOSPHOROUS IN PLANT FOOD

Students will determine the phosphorus content in three different brands of plant food. For each brand, two trials must be run and if the results do not agree, they may need to do third trial. The chemical reagents that will be supplied in the laboratory for this project are commercial analysis packets that are commonly used for such analyses, even by gardeners.

#### BACKGROUND

See student hand out for this.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

The experimental procedure used to determine phosphorus content is as follows:

For each different plant food, you will need to do the following:

- 1. From the % P<sub>2</sub>O<sub>5</sub> listed on the plant food label with the highest percentage, you will need to calculate the concentration of PO<sub>4</sub><sup>3-</sup> this will produce when a precisely known mass is dissolved in water and diluted to 100 ml in a volumetric flask.
- 2. Make a standard solution that is about 20 percent more concentrated in Na<sub>3</sub>PO<sub>4</sub> than that calculated in step 1.
- 3. From the standard mixed in step 2, make 4 additional standards by dilution such that you have 0.2, 0.4, 0.6 and 0.8 of the original solution.
- 4. Follow the instructions on the commercial phosphate analysis kit to measure the absorbance of each of the standards produced in step 3 to obtain a Beer's Law plot.
- 5. For each different brand of plant food, make three solutions using the mass and volume calculated in step 1. and measure each of these against the Beer's law plot from step 4.
- 6. Use the atomic weight of phosphorus, the molecular weight of PO<sub>4</sub> and the concentration of the PO<sub>4</sub> to calculate the number of grams of phosphorus per gram of each plant food sample.
- 7. Use the atomic weight of phosphorus, and the molecular weight of  $P_2O_5$ , calculate the  $P_2O_5$  content (in grams) of the plant food sample.
- 8. Calculate the  $P_2O_5$  percentage for each plant food sample.

#### **MINIMUM REQUIREMENTS:**

See student hand out for this.

Dale A. Hammond, PhD, Brigham Young University Hawaii

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#### **INDEPENDENT PROJECT #13**

#### QUANTITATIVE DETERMINATION OF VITAMIN C IN VARIOUS FRUITS

Vitamin C, a controversial vitamin, apparently has several functions in the body. One of its functions is that of a coenzyme in certain oxidation-reduction reactions in the body.

Vitamin C or ascorbic acid is abundant in many foods. The current recommended daily allowance (RDA) published by National Academy of Sciences is 60 mg. Many scientists including the famed Linus Pauling believe that we should ingest gram quantities daily for optimum health.

Pure vitamin C is a colorless, water soluble solid. It is somewhat stable in acidic solutions. In neutral and alkaline solutions it is rapidly oxidized by oxygen in the air. This tendency toward air oxidation in foods leads to decreased vitamin C content in the meals that we eat.

The fact that vitamin C does oxidize readily is the basis of determinations of its content in foods. N-bromosuccinimide (NBS), a mild oxidizing agent, is used to titrate the Vitamin C in food samples.

#### **SOLUTIONS:**

NBS reacts with vitamin C (ascorbic acid) on a one to one ratio. Solution: Dissolve 0.22 g of NBS in 8 ml of acetone, dilute to 1.000 L in a volumetric flask and mix well. This must be mixed fresh before the lab and refrigerated for preservation.

Vitamin C Standard Solution: Dissolve 0.125 mg Ascorbic acid and 3.5 g of oxalic acid in 100 ml of water in a 250 ml volumetric flask, then dilute to the mark with water and mix well. This must be mixed fresh before the lab and refrigerated for preservation. Since the NBS is unstable, 10 ml of the vitamin C solution is used to standardize the NBS each time before the titrations begin. The standardization must be done in at least duplicate analyses.

4 % KI (w/v) solution to serve as the indicator.

1 % oxalic acid (w/v) solution to preserve the vitamin C in the fruits from air oxidation.

10 % HOAc solution. Add 2 ml to each titration to stabilize the system.

Starch solution (refrigerate): Spray starch from a spray can for several seconds into 50 ml of water in a 250 ml beaker, then stir well. This will last about 2 weeks if refrigerated.

#### STANDARDIZING OF NBS SOLUTION

The N-bromosuccinimide is somewhat unstable. Therefore, the volume of NBS required to react with 1 mg of Vitamin C is determined at the beginning of the day's determinations. This is done by titrating 10 ml of 0.50 mg/ml solution of vitamin C with the NBS solution. (Total of 5 mg Vitamin C) You will need to work out the relationships involved.

## **Experimental Procedure:**

- 1. Obtain approximately 150 ml of the NBS solution by pouring into a clean, dry 250 ml beaker. This solution will be used for all parts of the experiment. Keep the remainder of the NBS solution in the refrigerator.
- 2. You will need to determine what volume of the vitamin C solution you will titrate, and how to measure that volume exactly. **Remember:** Oxalic acid, which is used as a preservative in the vitamin C solution, is toxic.
- 3. Add 5 ml of 4% KI solution, 2 ml of 10% acetic acid solution, 10 drops of 4% starch and approximately 30 ml of distilled water to the flask with the Vitamin C solution. How precisely do these volumes need to be measured?
- 4. You will need to decide what type of titration you will perform. See **Useful Titration Operations**, which can be obtained from your instructor.
- 5. Titrate the Vitamin C solution with the NBS. The end point is reached when one drop of NBS solution causes a permanent dark blue color. What color of background should you have for this titration.
- 6. Record the data and perform the appropriate calculations to determine the amount of vitamin C per 8 oz. serving in each food substance you analyzed.

#### FRUIT JUICE AND DRINK ANALYSIS

Determine and compare the vitamin C content of any four of the following juices and drinks:

- fresh orange juice
- orange juice at room temperature for 2-3 hours
- freshly mixed Tang
- mixed Tang at room temperature for 2-3 hours
- freshly reconstituted frozen orange juice
- freshly mixed orange Kool Aid
- squeezed orange juice which has been refrigerated
- mixed Tang which has been refrigerated

#### FRUIT & VEGETABLE ANALYSIS

Determine and compare the vitamin C content of any two of the following vegetables:

- fresh green pepper
- raw cabbage
- cooked cabbage
- fresh apple (peel when determination is made)
- peeled apple (approximately ½ to 1 ½ hours)

- fresh red pepper

#### **Experimental Procedure:**

- 1. Determine how much of the fruit or vegetable you will need to weigh out. (If cooked cabbage, gently boil in 30 ml distilled water in beaker after weighing.)
- 2. You will need to determine how you will obtain the vitamin C out of the fruit or vegetable, and how to make sure that you extracted most of the vitamin C
- 4. Transfer the approximately 30 ml of filtrate to a 125 ml Erlenmeyer flask.
- 5. Add 5 ml of 4% KI, 2 ml of 10% acetic acid and 10 drops at 4% starch to the flask.
- 6. Titrate with NBS until dark blue.
- 7. Record the volumes of NBS needed for each titration in the report sheet and calculate the mg of vitamin C per 3 oz. serving.

# **MINIMUM REQUIREMENTS:**

- 1. You must report your experimental design, the variables involved in the measurement, how you controlled those variables, etc. (For assistance in this, ask your instructor for copies of **Independent Inquiry Experiment Introduction**, **Making Oral and Written Presentations** and **Organizing the Research and Its Report**.)
- 2. You must describe the procedures you used for extraction and titration of the vitamin C
- 3. You must provide a table showing the raw amount, the amount of titrant, the calculated amount of vitamin C for the raw amount of food taken, and the calculated amount of vitamin C for either the 8 oz serving of juice or 3 oz serving of food.
- 4. You may be asked by your instructor to give a short presentation (~5 minutes) about your project to your lab instructor and the rest of the class during the last lab period

# INSTRUCTOR RESOURCES

Dale A. Hammond, PhD, Brigham Young University Hawaii

John R. Amend, PhD, Montana State University

#### **INDEPENDENT PROJECT #14**

#### QUANTITATIVE DETERMINATION OF VITAMIN C IN VARIOUS FRUITS

#### **BACKGROUND**

See student hand out for this.

**EXPERIMENTAL PROCEDURES** (Students should be guided to these procedures and results.)

The procedures suggested here uses a KI/starch combination to indicate the end point of the titration. This is difficult to see in many of the fruit and vegetable mixtures. However, If you have a good pair of students doing this experiment, these analyses can also be carried out potentiometrically with an ORP electrode, with very good results. The titration procedure is basically the same as a pH titration, discussed in the **Measurement Manual** or in previous titration experiments.

#### **SOLUTIONS:**

N-bromosuccinimide (NBS) Solution: Dissolve 0.220 g of NBS in 8 ml of acetone, dilute to 1.000 L in a volumetric flask and mix well. This must be mixed fresh before the lab and refrigerated for preservation.

Vitamin C Standard Solution: Dissolve 0.125 mg Ascorbic acid and 3.5 g of oxalic acid in 100 ml of water in a 250 ml volumetric flask, then dilute to the mark with water and mix well. This must be mixed fresh before the lab and refrigerated for preservation. Since the NBS is unstable, 10 ml of this solution is used to standardize the NBS before the titrations begin. The standardization must be done in at least duplicate analyses.

4 % KI (w/v) solution to serve as the indicator.

1 % oxalic acid (w/v) solution to preserve the vitamin C in the fruits from air oxidation.

10 % HOAc solution. Add 2 ml to each titration to stabilize the system.

Starch solution (refrigerate): Spray starch from a spray can for several seconds into 50 ml of water in a 250 ml beaker, then stir well. This will last about 2 weeks if refrigerated.

#### STANDARDIZING OF NBS SOLUTION

The N-bromosuccinimide is somewhat unstable. Therefore, the volume of NBS required to react with 1 mg of Vitamin C is determined at the beginning of the day's determinations. This is done by titrating 10 ml of 0.50 mg/ml solution of vitamin C with the NBS solution. (Total of 5 mg Vitamin C)

 $\frac{5 \text{ mg Vitamin C}}{\text{Volume NBS used}} = \frac{\# \text{ mg Vitamin C}}{1.00 \text{ ml NBS}}$ 

Example:  $\frac{5 \text{ mg Vitamin C}}{20 \text{ ml NBS}} = \frac{0.25 \text{ mg Vitamin C}}{1.00 \text{ ml NBS}}$ 

**Titration Procedure:** (Students should be guided to these procedures and results.)

It is recommended that the *MicroLAB* drop counter buret, described in the **Useful Titration Operations** on the CD, be utilized in these titrations for better accuracy and for ease of titration.

- 1. Obtain approximately 150 ml of the NBS solution by pouring into a clean, dry 250 ml beaker. This solution will be used for all parts of the experiment.
- 2. Pipet 10.00 ml of the 0.50 mg/ml Vitamin C solution into a clean, dry 125 ml flask. (Remember to use a pipet bulb. Oxalic acid, which is used as a preservative in the vitamin C solution, is toxic. Your instructor will demonstrate how to properly use a pipet.)
- 3. Add 5 ml of 4% KI solution, 2 ml of 10% acetic acid solution, 10 drops of 4% starch and approximately 30 ml of distilled water to the flask with the Vitamin C solution. (Use your graduated cylinder for these measurements.)
- 4. Rinse a 50-ml buret with about 5 ml of the NBS solution. Then, fill the buret. (Your instructor will show you how this is done if you need help.)
- 5. Titrate the Vitamin C solution in the 125 ml flask with the NBS. The end point is reached when one drop of NBS solution causes a permanent dark blue color. (Have a white background behind the flask so that the blue coloration can be detected easily.)
- 6. Record the buret reading and fill in the report sheet.

#### FRUIT JUICE AND DRINK ANALYSIS

Determine and compare the vitamin C content of any four of the following juices and drinks: (Calculate the mg of vitamin C per 8 oz. serving.)

- fresh orange juice
- orange juice at room temperature for 2-3 hours
- freshly mixed Tang
- mixed Tang at room temperature for 2-3 hours
- freshly reconstituted frozen orange juice
- freshly mixed orange Kool Aid
- squeezed orange juice which has been refrigerated
- mixed Tang which has been refrigerated

#### **Experimental Procedure:**

- 1. Pipet 10 ml of the juice being analyzed into a 125 ml Erlenmeyer flask.
- 2. Add approximately 30 ml of 1% oxalic acid solution, 5 ml of 4% KI, 2 ml of 10% acetic acid solution and 10 drops at 4% starch.
- 3. Titrate the sample with the NBS solution. The blue end point will be somewhat masked by the orange color of the juice and is often difficult to detect. Watch for a discoloration of the original orange to yellow color.
- 4. Record the volumes of NBS used for each titration on the report sheet and calculate the mg of vitamin C per 8 oz. serving.

## FRUIT & VEGETABLE ANALYSIS (Calculate the mg of vitamin C per 3 oz. serving.)

Determine and compare the vitamin C content of any two of the following vegetables:

- fresh green pepper
- raw cabbage
- cooked cabbage
- fresh apple (peel when determination is made)
- peeled apple (approximately 1/2 to 1 1/2 hours)
- fresh red pepper

#### **Experimental Procedure:**

- 1. Accurately weigh 5 g of the fruit or vegetable. (If cooked cabbage, gently boil 30 ml distilled water in beaker after weighing.)
- 2. Place the fruit or vegetable and 15 ml of 1% oxalic acid solution in a mortar and grind thoroughly.
- 3. Filter the mixture through 2-3 layers of cheesecloth and collect the filtrate in a beaker. (Rinse the mortar with an additional 15 ml of 1% oxalic acid and pour through the same cheesecloth.)
- 4. Transfer the approximately 30 ml of filtrate to a 125 ml Erlenmeyer flask.
- 5. Add 5 ml of 4% KI, 2 ml of 10% acetic acid and 10 drops at 4% starch to the flask.
- 6. Titrate with NBS until dark blue.
- 7. Record the volumes of NBS needed for each titration in the report sheet and calculate the mg of vitamin C per 3 oz. serving.

Sample Report form that students can be guided to constrict.	
Name	
Vitamin C Rep	port Sheet
Standardizing the NBS Solution	
ml NBS to titrate 10 ml of standard vitamin	C. solution
(10 ml vitamin C solution contains 5 mg vitamin C	
$\frac{5 \text{ mg vitamin C}}{\text{Vol. NBS}} = \frac{\text{\# mg vitamin C}}{1.00 \text{ml NBS}}$	
Analysis of Fruit Juice and Fruit Drink	
Identity of Juice or Drink	per 8 oz. juice or drink
ml NBS x K x 23.6 =	mg vitamin C
ml NBS x K x 23.6 =	mg vitamin C
ml NBS x K x 23.6 =	mg vitamin C
ml NBS x K x 23.6 =	mg vitamin C
2.35 ml/8 oz., therefore, 23.6, 1	0 ml samples in 8 oz.
Analysis of Solid Fruits and/or Vegetables	
Identity of Fruit or Vegetable	per 3 oz. of fruit or vegetable
ml NBS x K x 17 =	mg vitamin C
ml NBS x K x 17 =	mg vitamin C

85 grams/3 oz., therefore, 17, 5 gram samples in 3 oz.