## **Laboratory Manual**

### **General Chemistry II Honors**

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### Preface: Calculations in the Chemistry Laboratory

### **The Unit Basis Method**

Experience with numerical problems is extremely important in your study of chemistry, for it gives you practice in thinking problems through and deepens your understanding of the principles involved. However, if you just go through the mechanical steps of solution, giving no thought to the meaning of what you are doing, you may actually impair your understanding of the principles. In the following pages, you will learn a simple method of approaching problems—the "Unit Basis Method"—that is founded on clear thinking and is applicable not only to chemistry but also to other sciences and to "everyday problems." In fact, you have undoubtedly used this method many times already, for example when shopping. If you diligently use the "Unit Basis" in your chemistry problems, you will be thoroughly satisfied with the results.

One important principle underlies the discussion that follows. In the types of problems considered, numbers are meaningless in themselves; **only numbers associated with units have a meaning. Therefore, only numbers associated with units are used in calculations.** 

### The "Unit Basis Method" in Everyday Problems

First, we shall apply the Unit Basis Method to some very simple everyday problems. The advantages of doing this are the following: (1) all the terms are familiar, and (2) the problems are so simple that you can focus your attention on **the method alone.** 

Consider the following example:

#### **Problem 1**

The cost of 8 pencils is 40 cents. How many cents do 10 pencils cost?

#### Solution

• **Step 1:** Start by setting up the question (Q) in a form that clearly shows what is given in the problem (the "data") and what you must find out (the "unknown"). It is helpful to use the same format each time; a convenient one is shown below; Question:



• **Step 2:** To answer the Q shown in step 1. You need to find a link between the data and the unknown. In this example the link is given in the text of the problem itself:

#### Link:

40  cents = 8  pencils	
------------------------	--

• Step 3: The link may be rewritten in the form of two Unit Basis

40 cents	5.0 cents
8 pencils	1 pencil
$\frac{8}{2}$ pencils =	$=\frac{0.2 \text{ pencils}}{0.2 \text{ pencils}}$ = number of pencils purchased with 1 cent
40 cents	1 cent

Note that the meaning of a Unit Basis Fraction (UBF) is the same whether the division has been carried out or not:

 $\frac{40 \text{ cents}}{8 \text{ pencils}} = \frac{5.0 \text{ cents}}{1 \text{ pencil}} = \text{number of cents necessary to buy 1 pencil}$ 

- each of those Unit Base Fractions tell you the cost of 1 pencil.

Step 4. Next examine the meanings of the two UBFs and select the UBF needed to answer the question Q. In this example, you want to find the number of cents necessary to buy 10 pencils. So you need the UBF that gives "the number of cents necessary to buy one pencil" Once you know how many cents are needed to buy 1 pencil, it is easy to find how many are needed to buy 10 pencils—or 25 or 54, or any desired number of pencils:

The answer is expressed in "cents." This is the correct unit as shown in the setup of the question in Step 1.

#### Solve and Check Units

10 nepeits v	5.0 cents	- 50	cents
10 penciis x	1 pencil	- 50	cents

Note: In solving problems of this type, the units are treated as arithmetical quantities and can be cancelled.

Thinking out a problem using the Unit Basis Method will take a little extra time until you get accustomed to it. However, it is really the best way to solve the problem. Incidentally, you may be interested to know that this method is the way scientists solve their own problems. A major advantage of the Unit Basis is that you can check your final answer for the correct units. Remember units are a wonderful built-in check of your reasoning.

### Summary Outline of the Unit Basis Method

- **Step 1:** Rewrite the question to show the data and the unknown.
- Step 2: Find the link
- Step 3: From the link work out the desired Unit Basis Fraction
- **Step 4:** Use the Unit Basis Fraction to solve the problem. Check that the answer thus obtained has the desired units.

The same Unit Basis Method employed in the previous "money- pencils" problem can be used for problems involving systems of measurements. We will begin with some very simple examples:

### Problem 2

The length of a football field is 100 yards. What is the length of the field, expressed in feet?

• Step 1: Question

100 yards = ? feet

• Step 2: Link: 1 yard = 3 feet

Note: In this example the link is not given in the problem itself you are supposed to know it or to find it in a conversion table.

#### Step 3: UBFs:

$$\frac{1 \text{ yd}}{3 \text{ ft}} = \frac{0.33 \text{ yd}}{1 \text{ ft}} = \text{number of yards in 1 foot}$$
$$\frac{3 \text{ ft}}{1 \text{ yd}} = \text{number of feet in 1 yard}$$

**Step 4:** Solve and check: You want to find how many feet are in 100 yd. Thus, the UBF you need is the one that gives the "no. of ft in one yd", that is  $\frac{3 \text{ ft}}{1 \text{ yd}}$ .

$$100 \text{ yd} \times \frac{3 \text{ ft}}{1 \text{ yd}} = 300 \text{ ft} \text{ (no. of ft in 100 yd)}$$

• No. of yd.

- Check answer: the unit is ft, as indicated in the question.
  - UBF giving no. of ft in 1 yd.

In problems 1 and 2, the solutions required **only one link** and therefore only one Unit Basis Fraction. You can further develop this approach by solving problems that involve **two** (or more) links and therefore two (or more) Unit Basis Fractions. This extension of the Unit Basis Method is illustrated by some relatively simple problems involving everyday conversions of lengths.

#### Problem 3

Calculate the length in inches of standard football field, which is 100 yds.

• **Step 1:** 100 yds = ? inches

To solve this problem, you can first convert yards to feet, and then convert feet to inches.

To convert yards to feet, you need a link between these units (1yd = 3 ft) and to convert feet to inches, you need a link between these units (1ft = 12 in). So, you really have two Q's.

• QI:100 yds = ? feet

You have already answered this question in problem 2. The answer was 300 feet.

- QII: 300 ft = ? inches
- **Step 2:** Link for QII: 1 ft = 12 in

• Step 3: UBF's from above link:

 $\frac{12 \text{ in}}{1 \text{ ft}}$  no of inches in 1 ft. and  $\frac{1 \text{ ft}}{12 \text{ in}} = \frac{0.833 \text{ ft}}{\text{in}} = \text{no. of ft in 1 inch}$ 

• **Step 4:** Solve and check units:

$$300 \text{ ft} \times \frac{12 \text{ in}}{1 \text{ ft}} = 3600 \text{ in}$$

Once you understand the method, you can solve the problem more rapidly by combining the successive conversions in one step. Also, after a little practice you will quickly recognize the UBF required to solve the problem, so you need not right the other one. Here is a streamlined solution of problem 3:

- **Step 1:** Q: 100 yd = ? in
- Step 2: Links: 100 yd = ? ft = ? in

1 yd = 3 ft and 1 ft = 12 in

• Step 3: UBFs:

3 ft 12 in
$\frac{1}{1 \text{ yd}}$ $\frac{1}{1 \text{ ft}}$

• Step 4: Solve and check:

$$100 \text{ yd} \times \frac{3 \text{ ft}}{1 \text{ yd}} \times \frac{12 \text{ in}}{1 \text{ ft}} = 3600 \text{ in}$$

The preceding examples illustrate that the key to the unit method is to have the necessary link or links. Where do you obtain the relationship that lets you write the desired link?

- 1. Sometimes you may recall the relationship from your general knowledge
- 2. Sometimes you may need to look it up in a table of conversions for other reference sources

3. Sometimes you have to work it out for yourself

You will soon find that searching for the links is an effective way to review what you are learning about chemistry.

### **Measurement and Scientific Notation**

To measure means to determine the size (magnitude) of anything by comparing it to some accepted reference standard. Thus, **all measurements are relative**, and they all depend on the standard used as the reference unit. For this reason, **a measured value must always be expressed by a number accompanied by the appropriate unit or units.** 

There are three important aspects involved with measurements:

- 1. A suitable measuring instrument
- 2. An accepted standard of reference that can serve as the basis for the "marking off" (calibration) of the measuring instrument.
- 3. A conventionally accepted and uniform way to report a measured value so that it will convey the necessary information clearly and do so in a convenient form.

### **Significant Figures**

**How "good" is a measurement?** Almost all numbers used in a science come directly or indirectly from some measurement, so "good science" depends to a very large extent on "good measurement." There are two somewhat distinct aspects to how good a measured value is.

One aspect is called **precision** and refers to how closely repeated measurements of the same quantity agree with one another when carried out under very much the same conditions. Precision is, in a sense, the reproducibility of the measured values.

The other aspect is called **accuracy** and refers to how close a certain measured value comes to the "true value"— that is, to the officially accepted value. (Of course, the "true value" itself is never really known, except within certain limits.) The accuracy of a measurements depends to a large extent on the sensitivity of the instrument used, the **sensitivity** being the ability of the instrument to detect and measure even very minute differences of the quantity being measured.

Scientific measurements are never performed only once; they are always repeated—often many times. The various results are then compared and averaged, discarding those values that happened to be too "far off" from the average. At the end, the final result is reported in such a way as to give some indication of its precision. In simple chemical problems, the precision of a measured value is often indicated by the number of significant figures used in expressing the value.

#### What Are Significant Figures?

First let us review briefly the official meaning of some common terms:

- A number is an expression of quantity.
- A digit or figure is any one of the characters (0, 1, 2, ...9) used to write numbers
- A **significant figure** is a meaningful digit in a number. It stands for its value in its specific place: for example, in the number 243, the 2 stands for "2 hundredths" and 4 stands for "4 tens" and the 3 stands for "3 ones". The three digits in the number 243 are all significant—that is, the number 243 has three significant figures.
- The general rule about reporting numerical values obtained from measurements or calculations is:
- Give as many significant figures as to include only one uncertain digit the last one at the right.

This means that all the digits must be "sure" except the last one at the right which is a "best guess." When you need to round off a number to the correct significant figures follow this rule: (1) non-significant figures amounting to less than five at the "rightmost" non-significant digit is counted as "zero," and (2) non-significant figures amounting to five or more at the rightmost non-significant digit are counted as a "one" to be added to the last significant digit.

#### **Problem 4**

Round off the following numbers so that each has only three significant figures:

- A. 1.0632
- B. 0.42468

#### Solution

A. 1.0632 = 1.06 (right most dropped digit is less than 5)

B. 0.42468 = 4.425 (right most dropped digit is more than 5)

Digits to be dropped are counted as "zero" digits to be dropped are counted as "1" to be added to last significant digit

The examples in Problem 4 illustrate that the digits "0" can be used in two different ways. At times it represents a significant figure: for example, the mass of sample may be reported at 16.50 grams, meaning that it is closer to 16.50 than to 16.49 or 16.51. In this case, "0" in the number 16.50 is significant, and the number therefore has four significant figures. Similarly, the number 10.50 also has four significant figures, two of which are zeros. At other times the digit "0" may be used as a decimal place marker. For example, the mass of a sample may be reported as 0.068 gram. Here neither of the two digits "0" is significant; each "0" merely serves to indicate that the 6 means "6 hundredths" and that the 8 means "8 thousandths" of a gram. The value 0.068 gram, therefore, has only two significant figures—the 6 and the 8. (Often people drop the digit "0" before the decimal. For example, the value 0.068 gram may be reported as ".068" gram; this practice is not recommended in chemistry).

**Common-Sense Rules About the Use of Significant Figures**. Assume that you have just carried out three independent measurements of the thickness of a bone sliver. The three measured values, as read on the instrument used, are as follows: 0.253 cm, 0.255 cm, 0.252 cm. The average thickness is then calculated as:

average value  $\frac{(0.253 + 0.255 + 0.252)}{3}$  cm =  $\frac{0.760}{3}$  cm = 0.253333...cm

Notice, that the number 3 in the denominator of the above fraction is an exact number (three measurements, no more or less) so you can think of it as 3.00... with as many zeros as you wish. For simplicity, an exact number is written without any decimals, keeping in mind that the other number(s) in the calculation are those which determine the number of significant figures in the answer. How many significant figures does the answer have in this example? From the division you can get just about as many digits as you wish, since 0.760 is not exactly divisible by 3. Where should you stop? Clearly the final answer cannot be any better than the initial measured values, each of which had only three significant figures. In this example therefore, you should express your answer with three significant figures—that is, 0.253 cm. (In this number, the "0" plays the role of a place marker, the 2 and the 5 are sure figures, and the last digit, 3, is a best guess.)

As a useful general guideline, the result of a multiplication or division should not be reported with more significant figures than any of the values entered in the calculation.

How about adding or subtracting values? In this case use the following example as a guideline:

#### Experiment 1

- Mass of beaker + sample: 21.836g
- Mass of empty beaker:  $\frac{-20.612 \text{ g}}{?} = 1.224 \text{ g}$  (both measured on a sensitive analytical balance)

## Both starting values have the same number of digits (three) after the decimal point. The answer also has the same number of digits (three) after the decimal point.

### Experiment 2

- Mass of beaker + sample: 21.836 g (measured on a sensitive analytical balance)
- Mass of empty beaker:  $\frac{-20.6 \text{ g}}{?} = 1.236 \text{ g}$  (measured on a technical balance)
- Mass of sample: =1.2g (correct no. of significant figures)

### **Numbers in Exponential Form**

Nature offers many examples of extremely tiny, and many examples of extremely huge, and countless examples of the in-between. The diameter of a polio virus sphere is about 0.000002 of an inch or 0.00000006 m; an average person is about 5 ½ to 6 feet tall- a little less than 2 m; the distance from the earth and the moon is 240,000 miles or 390,000,000 m. Length is not the only property that shows such a spectacular range of values. The electrical driving force that triggers lightning during the storm is a million times greater than that require to operate a lightbulb, and this in turn is a million times greater than the nerve impulse that causes the blink of an eye.

To handle the fantastic range of values that nature presents, we have developed some special expressions. In common language, we refer to the "order of magnitude" of things, where "one order of magnitude" indicates a ten-fold increase or decrease in size.

In scientific work, very large and very small numbers are expressed in a conventional shorthand way, called exponential form, which is based on the use of powers of ten.

#### **Positive Powers of 10**

Table 1 list some examples of positive powers of 10. As you see, is a shorthand way to write 100 and is a shorthand way to write 1,000. A number such as and is called a positive power of 10. In such a number, 10 is called the base and the superscript (2 or 3 in our examples) is called the exponent. In every power, when the exponent has no sign, it is understood to have a (+) sign:  $(10^2 = 10^{+2} \text{ and } 10^3 = 10^{+3}).$ 

Number	Positive Powers of 10
One	$1 = 1 \times 10^0$
Ten	$10 = 1 \times 10^1$
One-hundred	$100 = 1 \times 10^2$
One-thousand	$1000 = 1 \times 10^3$
Ten-thousand	$10000 = 1 \times 10^4$
One-hundred-thousand	$100000 = 1 \times 10^5$
One-million	$1000000 = 1 \times 10^6$

#### **Table 1: Some Positive Powers of 10**

A change from a power of ten to the next higher power means a ten-fold increase in value, that is, an increase "by one order of magnitude"

The exponent of a **positive** power of 10 tells you how many zeros you must add to the **right** of the digit 1 to express the number in the usual "longhand" form, so is a 1 followed by 9 zeros, that is 1,000,000,000. In order to convert a "longhand" number, which is a multiple of 10 such as 100,000, to an exponential form, you may simply count the zeros that follow the digit 1. In 100,000 there are 5 zeros so 5 will be the exponent:  $100,000 = 10^5$ .

You can also calculate this conversion another way. Look again at 100,000 as your example. Where is the decimal point in this number? It is not written out explicitly, but it is understood to be at the right of the last digit. So, 100,000 is

really 100,000, isn't it? To go from 100,000 to 1, you must move the decimal point to the **left**, (100000.0). More specifically, you must move the decimal point 5 places to the left, and this tells you that +5 is the exponent in the power of 10. That is:  $100000 = 10^{+5} = 10^{5}$ .

This illustration may look like a lot of unnecessary work; you had arrived at the same answer more quickly by counting the zeros! True, for this very simple example, but there are cases where "counting the zeros" does not work, whereas the "move- the- decimal- point" method works for every number.

#### **Negative Powers of 10**

Table 2 lists some negative powers of ten. As you see, is a shorthand way of writing 0.01; a number such as is called a negative power of 10. Notice that a negative power of 10 is equal to 1 divided by the corresponding positive power of  $10^{-7}$ . For example: equals  $\frac{1}{10^{-2}}$ 

This illustrates a general rule: When a power of 10 is transferred from the numerator to the denominator, or vice versa, the sign of the exponent is changed.

Number	Negative Powers of 10
One-tenth	$0.1 = 1 \times 10^{-1}$
One-hundredth	$0.01 = 1 \times 10^{-2}$
One-thousandth	$0.001 = 1 \times 10^{-3}$
One-ten-thousandth	$0.0001 = 1 \times 10^{-4}$
One-hundredth-thousandth	$0.00001 = 1 \times 10^{-5}$
One-millionth	$0.000001 = 1 \times 10^{-6}$

#### Table 2: Some Negative Powers of 10

How can you quickly express a small number, for example 0.0000001, as a (negative) power of 10? To go from 0.0000001 to 1 you must move the decimal point **to the right** (0.0000001). More specifically, you must move the decimal point 7 places to the **right**, so -7 (minus 7) is the exponent. That is 0.0000001 equals  $10^{-7}$ .

Writing Any Number in Exponential Form. To express any number in a standard exponential form, you need to write it as a product of two factors. One is a digit factor, consisting of a number with just one digit at the left of the decimal point. The other is an exponential factor which is an appropriate power of 10.

Here is what you do for the example of a large number such as 2460000. (Assume this number to have four significant figures.) First, write the decimal point where it is understood to be (2460000.). Then move the decimal point to the left, until only **one digit**, 2, remains at the left of the decimal point (2460000.). This gives you 2.460 (a number between 1 and 10) as the digit factor. (Four digits have been retained since the given number has four significant figures; the other zeros were "place markers" and were dropped.) The digit factor must now be multiplied by an appropriate power of 10. What is this power? You have moved the decimal point six places to the **left**. Therefore, the exponent of the power of 10 is (+)6, that is, simply 6. Thus,  $10^{+6}$  or  $10^{6}$  is the desired exponential factor.

Here is a quick way to summarize the entire process:

 $2460000 = (2460000.) = 2.46 \times 10^{6}$ 

2.46 is the digit factor and  $10^6$  is the exponential factor

Similarly, you can express a very small number, such as, 0.000000729, as the product of a "digit factor" and of an "exponential factor." In this case, the exponential factor is a **negative** power of 10.

 $0.000000729 = (0.000000729) = 7.29 \times 10^{-7}$ 

7.29 is the digit factor and  $10^{-7}$  is the exponential factor

In general: Any number can be expressed in **standard exponential form** as the product of a digit factor (a number with just one digit to the left of the decimal point) and an **exponential factor** (a power of 10 having a whole number exponent, either positive or negative.)

### Problem 5

Write the following ordinary numbers in the standard exponential form. Assume each number to have four significant figures:

A. 4256.

B. 0.00005291

C. 26000000

#### Solution

A.  $4256 = (4256.) = 4.256 \times 10^3$ 

B.  $0.00005291 = (0.00005291) = 5.291 \times 10^{-5}$ 

C.  $26000000 = (26000000.) = 2.600 \times 10^8$ 

#### **Problem 6**

Convert the following numbers from standard exponential form to ordinary "longhand" form. (Keep in mind that large "longhand" numbers are not written in scientific form and usually do not have the correct number of significant figures.)

- A.  $5 \times 10^{10}$
- **B.**  $4.58 \times 10^{-4}$
- **C.**  $1.65 \times 10^{0}$
- D.  $6.02 \times 10^{23}$

#### Solution

- A. 50,000,000,000
- B. 0.000458
- C. 1.65
- D. 602,000,000,000,000,000,000

#### Problem 7

Convert the following non-standard exponential numbers to the standard exponential form:

- A.  $42.6 \times 10^3$
- **B.**  $259 \times 10^{0}$
- **C.**  $62.7 \times 10^{-4}$

#### Solution

A. In this and similar problems, first rewrite the given digit factor so that it is expressed in standard exponential form with just one digit before the decimal point.

 $42.6 = 4.26 \times 10^{1}$ 

The resulting number is then multiplied by the original exponential factor to give the entire number. Thus:

 $42.6 \times 10^3 = (4.26 \times 10^1) \times 10^3$  (collect the exponential factors)

 $= 4.26 \times (10^1 \times 10^3)$ 

 $= 4.26 \times 10^{(1+3)}$  (to multiply the exponential factors, add the exponents (each with its sign))

 $= 4.26 \times 10^4$ 

B.  $259 \times 10^{0} = (2.59 \times 10^{2}) \times 10^{0} = 2.59 \times 10^{(2+0)} = 2.59 \times 10^{2}$ 

C.  $62.7 \times 10^{-4} = (6.27 \times 10^{1}) \times 10^{-4} = 6.27 \times 10^{(1-4)} = 6.27 \times 10^{-3}$ 

**Scientific Notation.** A measured value, when expressed in a standard exponential form and with the correct number of significant figures, is commonly said to be in "scientific notation." Scientific notation is the conventional way to express numbers in science. It is useful because it lets you see at a glance which figures in the number you can "trust," and it is convenient because the exponential form makes both large and small numbers easy to handle.

### **Doing Arithmetic With Numbers in Scientific Notation**

#### **Addition and Subtraction**

Numbers with the same exponential factor may be added and subtracted directly:

#### Examples

A.  $\frac{6.02 \times 10^{23}}{+3.01 \times 10^{23}}$  $=9.03 \times 10^{23}$ 

The digit factors are added. The exponential factor is unchanged. The answer is in standard exponential form, so no further work is needed.

B.  $\frac{6.02 \times 10^{23}}{+ 5.92 \times 10^{23}}$  $= 11.94 \times 10^{23}$ 

The first answer must be put in standard exponential form.

 $= 1.19 \times 10^{24}$ 

Numbers that have different—but not too different—exponential factors must be converted to the same exponential form before adding or subtracting. If the exponential factors of the numbers are very different, the smaller number(s) are ignored. As a general rule, in adding and subtracting, neglect all numbers whose exponent is smaller than the largest exponent by 3 units or more.

#### Examples

exponents differ by one unit

A.  $1.03 \times 10^{-2} + 6.35 \times 10^{-1} = ?$ 

the smaller number is converted and then added.

$$0.013 \times 10^{-1} \\ + 6.35 \times 10^{-1} \\ = 6.453 \times 10^{-1}$$

this first answer is rounded off to two decimal figures because one of the numbers added only had two decimal figures.

 $= 6.45 \times 10^{-1}$ 

Exponents differ by 12 units

B.  $8.95 \times 10^{-3} + 6.20 \times 10^{-15} = 8.95 \times 10^{-3}$ 

C.  $8.95 \times 10^{-3} + 6.20 \times 10^{-15}$ 

The smaller number is neglected.

Exponents differ by 4 units

D.  $1.5 \times 10^2 - 3.7 \times 10^{-2} = 1.5 \times 10^2$ 

The smaller number is neglected.

#### Multiplication

To multiply numbers written in standard exponential form, follow a stepwise procedure as illustrated by the following example.

 $(6.3 \times 10^3) \times (3.4 \times 10^7) = ?$ 

• Step 1: Collect all digit factors together and all exponential factors together:

 $(6.3 \times 3.4) \times (10^3 \times 10^7) = ?$ 

• **Step 2:** Multiply the digit factors as usual; multiply the exponential factors by adding the exponents, if necessary; rearrange the number to standard a scientific notation:

 $(21.42) \times (10^{(3+7)}) = 21.42 \times 10^{10} = 2.1 \times 10^{11}$ 

Here is another example:

$$(1.3 \times 10^{-5}) \times (9.45 \times 10^4) = ?$$
  
 $(1.3 \times 10^{-5}) \times (9.45 \times 10^4) = [12.285] \times [10^{(-5+4)}] = ?$   
 $12.285 \times 10^{-1} = 1.2 \times 10^0 = 1.2$ 

#### Division

The procedure is outlined in the following example.

• **Step 1:** Collect digit factors and exponential factors separately:

$$\frac{4.2 \times 10^8}{2.1 \times 10^5} = ?$$

• **Step 2:** Carry out the indicated operations:

 $2.0 \times \overline{10^{(8-5)}} = 2.0 \times 10^3$ 

To divide an exponential factor by another, subtract the exponents

Answer is already in standard form

Here is another example:

$$\frac{3.2 \times 10^{-6}}{4.6 \times 10^2} = \frac{3.2}{4.6} \times \frac{10^{-6}}{10^2} = ?$$
$$= 0.6956... \times 10^{(-6-2)} = 0.6956... \times 10^{-8} = 7.0 \times 10^{-9}$$

#### **Powers**

To raise a number written in exponential form to a power, raise both the digit factor in the exponential factor to the desired power independently; here are two worked-out examples:

#### **Positive Powers**

$$(2.3 \times 10^3)^2 = ?$$
  
 $(2.3)^2 \times (10^3)^2 = (2.3) \times (2.3) \times 10^{(3 \times 2)} = 5.29 \times 10^6 = 5.3 \times 10^6$ 

To raise an exponential factor to a power, multiply the exponent by the power.

#### **Negative Powers**

$$(2.3 \times 10^3)^{-2} = ?$$

$$(2.3)^{-2} \times (10^3)^{-2} = \frac{1}{(2.3)^2} \times 10^{-6} = \frac{1}{(2.3) \times (2.3)} \times 10^{-6} = ?$$

$$= \frac{1}{(2.3)^{-2} \times 10^{-6}}$$

$$= \frac{1}{5.29} \times 10^{-6} = 0.189 \times 10^{-6} = 1.9 \times 10^{-7}$$

### **Experiment One: Chemical Spectroscopy Lab**

Data Analysis/Interpretation and Post-Lab Questions are available for Word Document download via <u>PA-ADOPT: Experiment One Word Document Download</u>. Download the word file and edit directly on your computer or upload it later to a Google Drive account for online editing.

Purpose: Obtain and analyze the spectra of unknown organic substance using spectra methods.

From the percent composition, the empirical formula can be determined. If the molecular mass is known, the molecular formula of the compound can then be obtained. The mass spectrometry offers molecular weight information (as well as other information) quite easily. Other spectroscopic techniques—infrared and nuclear magnetic resonance spectroscopies—can be used in conjunction to provide structural (bonding) details, leading to the structural formula of the compound.

### **Mass Spectrometry**

As a chemical instrument, the mass spectrometer uses high energy electron beams to bombard molecules, thus converting them into ions. These ions are further subjected to an electric field, which separates them based on their mass to charge ratios. Remember that ionization involves the formation of ions, usually from a gaseous molecule or atom. The energy required to do this is called the ionization energy or potential.

#### $X \rightarrow X^+ + e^-$

The mass to charge (m/e) stated above is given as:

 $m/e = Hr^2/2V$  where H is the magnetic field strength, r is the radius of curvature (of the flight path of the particle), and V is the potential difference of the ion-accelerating plates.

In a mass spectrum, the tallest peak is called the base peak, to which the abundance of other peaks in the spectrum are relative. When an electron is removed from a molecule during ionization, an ion is produced that is close in weight to the original molecule. This ion is known as the molecular ion peak (M<sup>+</sup>). In order words, the m/e of the molecular ion peak is the molecular weight of the compound. Lastly, most elements exist in two or more isotopic forms.



Mass Spectrum of Ethyl Bromide (by author)

Hydrogen has a heavier isotope <sup>2</sup>H (deuterium), and carbon also exists as <sup>13</sup>C in addition to the naturally occurring <sup>12</sup>C. As a result of this, peaks such as M+1 and M+2 often occur in a mass spectrum. For the 1-Bromoethane spectrum shown above, the molecular ion (M+) peak is at a m/e of 108, and the M + 2 peak is at a m/e of 110. This observed difference is the <sup>79</sup>Br and 81Br isotopes. Other peaks on the spectrum correspond to different fragments formed that hit the detector. For instance, a m/e peak at 29 most often is the ethyl ion,  $CH_3CH_2$ + peak, and the one at a m/e of 15 is the methyl ion,  $CH_3$ + peak.

### Infrared (IR) Spectroscopy

The infrared (IR) region of the electromagnetic spectrum ranges from 2.5–15  $\mu$ m. This region lies between the longer wavelength of the radio waves and the shorter wavelength of the visible light. The region between 2.5 and 15  $\mu$ m specified above is the vibrational portion of the IR region. The IR region provides very important information about molecular structure. The unit associated with this vibrational portion is wavenumber ( $\tilde{v}$ ), which is simply the inverse of centimeters (cm<sup>-1</sup>).

$$\widetilde{v}$$
 (cm<sup>-1</sup>) =  $\frac{1}{\lambda \text{ (cm)}}$ ; cm<sup>-1</sup> =  $\left(\frac{1}{\mu m}\right) \times 10000$ ;  $\mu m = \left\{\frac{1}{(cm^{-1})}\right\} \times 10000$ 

A higher wavenumber means higher energy. Also, the vibrational IR region extends from 4000 to 400 (cm<sup>-1</sup>), again, a range corresponding to 2.5–15  $\mu$ m.

In infrared spectroscopy, molecules become excited (transition to a higher energy level) upon absorption of this energy. The radiation absorbed corresponds to energy changes (8–40 kJ/mole), which embeds stretching and bending vibrational frequencies of most covalent bonds. Finally, only bonds with dipole moment absorb infrared radiation. For example, F<sub>2</sub>, Cl<sub>2</sub>, and H<sub>2</sub> do not absorb infrared radiation because they each have a zero-dipole moment.

Lastly, the IR spectroscopy provides structural information about a molecule by providing absorption characteristics of different bond types—C-H, S-H, O-H, C-C, C-O, C=C, and C=O, to name a few. A comprehensive list of stretching vibrations and the region of occurrence can be found in different spectroscopy books and/or online. For instance, you will find N-H or O-H between 4000–3500 cm<sup>-1</sup>; C=O between 1800–1650 cm<sup>-1</sup>; and C=C and C=N between 1650–1550 cm-1.

Below is the infrared spectrum of 1-Bromoethane, or ethyl bromide:



Predicted Infrared Spectrum of Ethyl Bromide (by author)

In the IR spectrum of 1-Bromoethane shown above, the peak near 3000 cm<sup>-1</sup> is the C-H (vib), the C-C (vib) is around 1250 cm<sup>-1</sup>, and the C-Br (vib) is the medium peak near 850 cm<sup>-1</sup> and the strong one near 550 cm<sup>-1</sup>. The infrared spectrum as a tool for structural elucidation is more important if the O-H group (broad, near 3500 cm<sup>-1</sup>) is present or absent. Also, the carbonyl group (C=O;

sharp, between 1650–1850 cm<sup>-1</sup>) for different classes of compounds (aldehyde, ketone, carboxylic acid, ester) can easily be spotted. The absence of the carbonyl group is also highly informative.

### **Nuclear Magnetic Resonance (NMR) Spectroscopy**

This method of chemical (molecular) identification is more important to organic chemists than the previous two discussed. Organic Chemistry is often regarded as the chemistry of carbon. Alongside carbon are hydrogen, nitrogen, and a few other elements. It is important, therefore, to have a technique readily available to identify these compounds, and the NMR offers just that.

Proton (<sup>1</sup>H) and carbon (<sup>13</sup>C) NMR spectroscopy generate signals that reflect the unique magnetic environment of each hydrogen and carbon atom in a molecule. These signals arise due to variations in the local electron density, chemical bonding, and proximity to electronegative groups, providing detailed information about the molecular structure, functional groups, and connectivity. For instance, the NMR spectrum of 1-bromopropane—CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>Br—will be expected to show at least three signals because there are three different hydrogens shown in the compound. The first is the hydrogen of the methyl group (CH<sub>3</sub>). Then, there is the hydrogen of the methyl group, and the other is to the farthest right with Br attached to it. The positioning of each, as I have described, constitutes different magnetic environments; as such, each will give a signal at different chemical shifts, defined below. The signal produced occurs when the certain nuclei absorb energy, and the absorbed energy leads to a change in spin relative to the applied magnetic field.

The position or place of appearance of each proton is called a chemical shift  $(\partial)$ :

$$\partial = \frac{(\text{shift in Hz})}{(\text{spectrometer frequency in MHz})}$$

For instance, CH3Br produces a shift of 243 Hz from the reference solvent (TMS) in a 90 MHz NMR(?); the chemical shift is as follows:

$$\partial = \frac{243 \text{Hz}}{90 \text{MHz}} = 2.7 \text{ppm}$$

The approximate chemical shifts for some protons are as follows:

Methyl (CH<sub>3</sub>: 0.7–1.3), methylene (CH<sub>2</sub>: 1.2–1.4), and methine (CH: 1.4–1.7). These hydrogens may move to a higher chemical shift (downfield) or a lower chemical shift (upfield) from where they are typically found depending on other groups (electron donating or electron withdrawing) that are either directly attached or next to them. Hydrogens of the phenyl group (Ph-H: 6.5–8.0); hydrogens attached to carbonyl (HC=O; aldehyde: 9.0–10.0; HO-C=O; carboxylic acid: 11.0–12.0); and hydrogens of alkenes and alkyne, as well as other functional groups (phenol, amine, amide, alcohol), have their respective chemical shifts.



<sup>1</sup>H NMR Spectrum of Ethyl Bromide (by author)

The NMR spectrum of 1-Bromoethane shown above has two distinct signals. The signal for the methyl group (CH<sub>3</sub>) is a triplet due to the two hydrogens of the CH<sub>2</sub> group (n + 1 rule) and the farthest upfield (low  $\partial$ ). The signal due to the methylene group (CH<sub>2</sub>) is a quartet and the farthest downfield (high  $\partial$ ) because of the attached Br group, which is an electron withdrawing specie.

### **Lab Activity**

- Obtain from your instructor the IR, NMR, and Mass spectra for your assigned unknown. The compounds to be assigned are as follows:
  - CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>Br: 1-Bromopropane
  - CH<sub>3</sub>CH<sub>2</sub>CH2NO<sub>2</sub>: 1-Nitropropane

- CH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>NH<sub>2</sub>: 1-Amino propane
- BrCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Br: 1,3-Dibromo propane
- This is a group assignment; each group must have at least four students.
- Conduct the spectra analysis and identify which of the 4 compounds you were assigned.

### **Post Lab Questions**

- 1. Calculate the percent composition for your "now" known compound.
- Calculate the Index of Hydrogen Deficiency (IHD) for your "now" known compound.
- 3. Calculate the wavenumbers for the:
  - a) C-O
  - b) C=O
  - c) C=N
  - d) N=N
- 4. The IR spectrum of your unknown compound has no peak at 1650 cm-1. Why?
- 5. Sketch the NMR spectrum of 2-Bromo butane.

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# Experiment Two: Total Organic Carbon (TOC) Analysis of Different Bottled-Water

Data Analysis/Interpretation and Post-Lab Questions are available for Word Document download via <u>PA-ADOPT: Experiment Two Word Document Download</u>. Download the word file and edit directly on your computer or upload it later to a Google Drive account for online editing.

The Total Organic Carbon (TOC) analyzer is an instrument (non-destructive to sample) designed to measure the quantity of organic compounds present in a water sample. It is a fast, nondestructive technique used in various industries, such as pharmaceuticals, wastewater treatment plants, and others. Untreated water will have inorganic and organic contaminants, some of which if left untreated can cause significant health risks to humans. The Environmental Protection Agency (EPA) is the federal agency responsible for setting the standard for acceptable levels of different chemicals found in our water.



TOC instrument located at Cheyney University of Pennsylvania's Science Center. Image by Author.

There are four steps involved in sample analysis: 1) Sample Introduction—the water sample is automatically picked up by an injection port; for multiple samples, the instrument comes with an autosampler, which allows the analysis of up to fifty samples; 2) Oxidation—this could be via chemical or combustion; in the equipment pictured above, the organic carbon is oxidized to carbon dioxide in a high-temperature chamber; 3) Detection—this is achieved via solid-state or

non-dispersive infrared detector (NDIR). The IR beam goes through the CO2 sample; 4) Readout/Display—measured quantity is displayed on the screen.

### Purpose

The purpose of this lab activity is to determine (measure) the TOC contents of different bottled and tap water from different regions/counties in Pennsylvania (PA) using the Total Organic Carbon (TOC) Analyzer.

### Materials

Total Organic Carbon (TOC) Analyzer, sample tubes, filtering flasks (small pores), ultrapure air, tap water samples, bottled water samples, and centrifuge.

### Procedure

Collect samples of tap water from different locations and collect different sold bottled water (Fiji, ACME, Aquafina, Glaceau Smart, Nice, Dasani, Waterguy, Delish, Poland Spring, Acadia, and Deer Park); refrigerate all water samples as soon as collected to halt any bacteria growth. Then filter each water sample through a small pore filtering flask and quickly centrifuge the water sample to remove any microscopic sediment. Instantly transfer the supernatant into a sample tube and record the pH. Turn on the instrument and allow it to undergo the usual initialization. Set the instrument parameters provided by your instructor to analyze for Total Organic Carbon (TOC) and not Total Nitrogen analysis, since the instrument can do both. Collect and analyze data.

### **Data Analysis/Interpretation**

- Compare the TOC values for the different bottled water used. What conclusion (if any) do the obtained results tell you?
- Compare the TOC values for the different tap water used. What conclusion (if any) do the obtained results tell you?
- Compare the TOC values for the bottled water to those of the tap water samples. What conclusion (if any) do the obtained results tell you?

### **Post Lab Questions**

- 1. How would you design an experiment to measure the sugar content of a sample using the TOC analyzer?
- 2. The fluoride content of a sample (water) is reported as 0.28 mg/ L. Is this above or below the recommended FDA standard?
- 3. List three Volatile Organic Chemicals (VOCs) identified by the FDA as water/ environmental contaminants.
- 4. Name/list two methods by which pathogens present in water can be eliminated.
- 5. The nitrate ion (NO<sub>3</sub>-) concentration in a certain water sample is 45–88 ppm. This corresponds to Nitrate-Nitrogen (NO<sub>3</sub>--N) concentration of 11–20 ppm. What is the nitrate ion concentration for a (NO<sub>3</sub>--N) equivalence of 8 ppm?

### **Bibliography/Resources**

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The hazards of chemical contaminants in unfiltered drinking water. (2014, December 1). Water Technology Online. <u>https://www.watertechonline.com/</u> water-reuse/article/15541106/the-hazards-of-chemical-contaminants-inunfiltered-drinking-water

### Experiment Three: Total Organic Carbon (TOC) Analysis of Sugar in Carbonated and Non-Carbonated Drinks

Data Analysis/Interpretation and Post-Lab Questions are available for Word Document download via <u>PA-ADOPT: Experiment Three Word Document</u> <u>Download</u>. Download the word file and edit directly on your computer or upload it later to a Google Drive account for online editing.

The Total Organic Carbon (TOC) analyzer is an instrument (non-destructive to sample) designed to measure the quantity of organic compounds present in a water-sample. It is a fast, non-destructive technique used in various industries, such as pharmaceuticals, wastewater treatment plants, and others. Untreated water will have inorganic and organic contaminants, some of which if left untreated can cause significant health risks to humans. The Environmental Protection Agency (EPA) is the federal agency responsible for setting the standard for acceptable levels of different chemicals found in our water.



TOC instrument located at Cheyney University of Pennsylvania's Science Center. Image by Author.

Spectroscopic techniques in general have a wide range of applications in different industries and areas. These areas include food, pharmaceuticals, beverages, material science, petrochemical, cosmetics, and biotechnology. The TOC analyzer requires a very small sample for analysis, and it is usually non-destructive. There are four steps involved in sample analysis: 1) Sample Introduction- the water sample is automatically picked up by an injection port; for multiple samples, the instrument comes with an autosampler, which allows the analyses of up to fifty samples; 2) Oxidation- this could be via chemical or combustion; in the equipment pictured above, the organic carbon is oxidized to carbon dioxide in a high temperature chamber; 3) Detection- this is achieved via solid-state or non-dispersive infrared detector (NDIR). The IR beam goes through the CO2 sample; 4) Readout/Display- measured quantity is displayed on the screen.

### Purpose

The purpose of this lab activity is to determine (measure) the sugar content of various drinks and check manufacturer's claim using the Total Organic Carbon (TOC) Analyzer

### Materials

Total Organic Carbon (TOC) analyzer, sample tubes, filtering flasks (small pores), ultra-pure air, Coca Cola, Sprite, Ginger Ale, Kool-Aid, Capri sun, Fanta, Seven-Up, Apple Juice, Cranberry Juice, Lemonade, Gatorade, Pedialyte, Prime, Grape Juice, Mountain Dew, Red Bull, and Pepsi Cola.

### Procedure

Prepare five standard sugar solutions of 0.1, 0.5, 1.0, 2.0, and 4.0 mg/mL; seek your professor's assistance with making the solutions should you need help. Set the instrument parameters provided by your instructor to analyze for Total Organic Carbon and record the corresponding TOC (in ppm) for each of the known solutions. Using the instrument parameters, obtain the plot of TOC (in ppm) content versus the Sugar Conc (in mg/mL).

For the carbonated drinks, degas about 50 mL of each sample by pouring it back and forth in two different Erlenmeyer flasks. Once you no longer see any bubbles, the sample is ready for loading. Read the TOC (in ppm) content for each carbonated drink.

For non-carbonated drinks, simply load each directly into sample containers on the autosampler. Activate the instrument to obtain the TOC (in ppm) reading for each sample.

### **Data Analysis/Interpretation**

- Obtain the standard curve by plotting TOC (in ppm) on the y-axis versus Sugar Concentration (in mg/mL) on the x-axis. Obtain the slope, and the equation for the best-fit line obtained.
- Using the equation obtained for the standard solutions, obtain (calculate) the sugar contents (in mg/mL) for all the carbonated drinks sampled.
- Using the equation obtained for the standard solutions, obtain (calculate) the sugar contents (in mg/mL) for all the non-carbonated drinks sampled.
- Create a table with four columns with the following headings: Obtained Results (report the data obtained for each drink); Reported Results (report the manufacturer's sugar content for each drink); Deviation (report the difference between the number you obtained and the number the manufacturer reported. Some conversions may need to be done here; ask your professor for guidance); and percentage error (assuming the manufacturer of each drink was correct).

**Obtained Results** 

**Reported Results** 

Deviation

**Percentage Error** 

### **Post Lab Questions**

- 1. Based on the equation you got, calculate the sugar content (in mg/mL) for an unknown drink with TOC (in ppm) reading of 587 ppm.
- 2. How would you determine "the deviation from linearity" for your standard solutions?
- 3. How accurate is this method for determining sugar concentration in carbonated and non-carbonated drinks? Discuss.
- Is the TOC method accurate for determining sugar concentration in carbonated or non-carbonated drinks? Discuss any agreement or disagreement between the two sets of results.
- 5. What will you do to improve the accuracy of results? What will you change in the experimental procedure, if any?

### **Bibliography/Resources**

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### Experiment Four: Saponification Reaction -Making Solid and Liquid Soap

Post-Lab Questions are available for Word Document download via <u>PA-ADOPT</u>: <u>Experiment Four Word Document Download</u>. Download the word file and edit directly on your computer or upload it later to a Google Drive account for online editing.

### **Introduction and Background**

From a previous experiment, where we separated fat from different candy bars, you were asked to find a suitable use for the extracted fat. As it turns out, we can react the extracted fat with a strong base such as sodium or potassium hydroxide, and a soap is formed. This simple reaction is referred to as a saponification reaction. In a saponification process, a triglyceride (fat or oil) undergoes alkaline hydrolysis with a strong base, leading to the formation of a fatty acid salt (soap) and glycerol. Basically, the hydroxide ions in solution breaks the ester bonds of the fatty acid and glycerol of the triglyceride, creating free soluble fatty acids and glycerol.

### Reaction



Cropped from "Saponification reaction" by <u>Ttzavaras</u> at <u>Greek Wikipedia</u>, licensed under <u>Creative Commons</u> <u>Attribution-Share-Alike 3.0 Unported license (CC BY-SA 3.0)</u>.

Saponification can be a one-step process or two-step process. The one-step, a faster and most often used method for making soap, does hydrolysis in a single step. In the two-step method, the triglyceride is hydrolyzed to carboxylic acid, followed by alkali neutralization of the fatty acid. As you might guess, the two-step, although slower, enhances the yield and purity of the product—in this case, soap.

### Purpose

Synthesis of solid and liquid, fragrance and non-fragrance soaps through saponification reaction.

### Materials

Choice of oil, sodium or potassium hydroxide (9.0 M) solution, hot plate or oil bath, stearic acid, food coloring, stirring rod, beaker (250 mL), mortar and pestle, pipets, filtering funnel, sodium chloride.

### Procedure (Solid Soap)

Heat oil (or fat obtained from candy bar extraction) on a hot plate to between 35 and 40°C, pour about 10 mL of the warm oil into a 250 mL beaker, gently add 2-4 drops of desired fragrance using a pipet, slowly stir in 3 mL of the 9.0 M of sodium hydroxide solution to the beaker containing the scented oil, and continue the stirring for another 30 minutes. As the mixture starts to thicken, add 3-4 drops of food coloring, stirring continuously. Pour about half of the mixture into a mortar, then add some stearic acid to further harden the soap. Test the pH of the soap at this point with a pH paper or a pH sensor. Lastly, add between 10 and 15 g of sodium chloride, and isolate, using vacuum filtration, the soap product. Leave the soap for at least 24 hours in the mortar (this will harden the soap).

### **Vacuum Filtration Procedure**

- 1. Position the filter paper over the funnel.
- 2. Place a rubber plug on the flask's mouth.
- 3. Fit the filtration funnel to the rubber plug.
- 4. Connect the vacuum pump firmly to the vacuum flask through side nozzle.
- 5. Pour the mixture into the filter paper in the filtering funnel and turn on the vacuum pump
- 6. Then extract the solid from the filtrate.

### **Procedure (Liquid Soap)**

Obtain some olive oil, vegetable oil or castor oil (you can also mix the three in equal amounts. Add some sugar while stirring, add some sodium lactate and heat the mixture to between 200-215°F. Obtain some potassium hydroxide pellets from your instructor and add about one tenth of the potassium hydroxide quantity in sodium hydroxide pellets. Slowly add the mixed bases to the hot oil and wait for the saponification process to begin (approx. 30-45 mins). Use a mixer to mix everything together (avoid any kind of overspill) The mixture may appear pasty at this point. Leave the product for some time, test the pH of the soap (about 10-10.5 is fine). Add some hot water while mixing or stirring. At this point, you may add a preservative, some fragrance or color, and leave the product overnight.

### **Post Lab-Questions**

- 1. In the reaction performed, what is/are the reactant (s) and what is/are the product(s)?
- 2. Do you think the type of fat used will make a difference in the product? Why or why not?
- 3. List other products that can be made via saponification. Explain the difference when compared to the reaction carried out in this laboratory exercise.
- 4. What is the difference between esterification and a saponification reaction?
- 5. What major ingredient changes between making liquid and solid soap?

### **Bibliography/Resources**

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### Experiment Five: Synthesis and Analysis of Potassium Aluminum Sulfate Dodecahydrate, KAI(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O

Data Analysis and Experiment Data Tables are available for Word Document download via <u>PA-ADOPT: Experiment Five Word Document Download</u>. Download the word file and edit directly on your computer or upload it later to a Google Drive account for online editing.

Alum is a crystalline white or off-white substance (di-cation) composed of positive one (+1) and potassium and positive three (+3), aluminum species. In today's experiment, you will help the recycling efforts for aluminum soda cans by first reacting it (soda can) with potassium hydroxide (a strong base), and then with sulfuric acid (a strong acid), resulting in the final product (alum). The environmental significance (recycle), as well as the economic impact (cost effectiveness) of turning aluminum metal into alum (a known vaccine adjuvant for almost seventy years) are enormous. The process to convert aluminum to alum involves the four steps below:

**Step 1:** Aluminum reacts with potassium hydroxide, KOH

 $2AI_{(s)} + 2KOH_{(aq)} + 6H_2O_{(l)} \rightarrow 2K_{(aq)} + 2AI(OH)_{4(aq)} + 3H_{2(g)}$ 

Step 2: Initial reaction with sulfuric acid, H2SO4

 $2AI(OH)^{-}_{4 (aq)} + H_2SO_{4(aq)} \rightarrow 2 AI^{3+}_{(aq)} + 3 SO_{4^{2-}(aq)} + 6H_2O_{(I)}$ 

Step 3: Subsequent reaction with sulfuric acid, H2SO4

 $2AI(OH)_{3 (s)} + 3H_2SO_{4(aq)} \rightarrow 2 AI^{3+}_{(aq)} + 3SO_{4^{2-}(aq)} + 6H_2O_{(I)}$ 

**Step 4:** Precipitation reaction

 $K^{+}_{(aq)} + AI^{3+}_{(aq)} + 2SO_{4^{2-}(aq)} + 12H_2O_{(I)} \rightarrow KAI(SO_4)2.12H_2O_{(s)}$ 

The protected thin film of aluminum oxide decreases its reactivity with acids. But in the presence of an excessively strong base such as potassium hydroxide, tetrahydroxoaluminate ion is formed, as well as aluminum hydroxide, which falls out of the solution. Further acidification results in aluminum hexahydrate and the eventual precipitation of alum.

### Purpose

The purpose of this lab activity is to synthesize potassium aluminum sulfate dodecahydrate (alum), and to determine the percent yield of the product.

### Materials

Aluminum foil or aluminum soda can, 3M sulfuric acid, H<sub>2</sub>SO<sub>4</sub> solution, 3M potassium hydroxide, KOH solution, ice bath, baking soda, NaHCO<sub>3</sub>, dilute acetic acid, CH<sub>3</sub>COOH solution (vinegar), 50% aqueous ethanol, CH<sub>3</sub>CH<sub>2</sub>OH solution, glass rod, 250 mL beaker, 50 mL graduated cylinder, hot plate, filtering flask, watch glass, distilled water, and weighing balance.

### Procedure

Weigh approximately 1.2 g of chopped up aluminum soda can or pieces of aluminum foil into a 250 mL beaker. Slowly add 25 mL of the 3M KOH solution (reaction is exothermic), stirring and allowing all the aluminum pieces to dissolve. Filter the resulting mixture to retain any undissolved substance on the filter paper and rinse the filter paper with 5 mL distilled water. Allow the mixture to cool to room temperature. Once cooled, slowly add 30 mL of the 3M sulfuric acid, H<sub>2</sub>SO<sub>4</sub> solution with stirring (aluminum hydroxide will precipitate initially but more acid will send it back into solution). Place the mixture on a hot plate to concentrate the mixture to about 50 mL. DO NOT HEAT AGGRESSIVELY!

Allow the beaker to cool overnight. The slow overnight cooling also allows the precipitation of alum to complete. If time permits, however, prepare an ice bath, and allow the beaker to sit in an ice bath for fifteen to twenty minutes. Collect the alum crystals through vacuum filtration, ensuring that all the alum crystals are collected. Wash the alum crystals with 10 mL cold distilled water, and then with another 25 mL portion of the 50% aqueous ethanol solution. Allow the alum crystals to air dry; then weigh the obtained product.

### **Data Analysis/Interpretation**

- Assuming that the aluminum can, or foil is pure aluminum, calculate the theoretical yield of alum (MM = 474.39 g/mol)
- Calculate the percent yield of alum

• What factors affect the yield of alum if your yield is less than or greater than 100%?

### **Analysis of Alum**

In this lab exercise, we will explore some of the physical properties of alum (melting point and density) to confirm the alum you made.

### **Melting Point Determination**

#### **Materials Needed**

Mel-Temp apparatus and capillary tubes, watch glass, spatula.

#### Background

The melting point (mp) of a solid is the temperature at which it melts to a liquid. The mp is an important physical property of a substance because it is easily measured and helpful in identifying the substance. When properly determined, the experimental mp of a solid also reveals whether it is pure or not. To measure a mp, one generally heats a small sample of the substance while monitoring the applied temperature with a thermometer. This procedure gives a range of temperatures as the mp. The temperature at which liquid is first seen is the lower end of the melting point range, and the temperature at which no solid remains is the upper end of the melting point range. A pure substance normally has a melting point range no larger than 1-2°C.

#### Loading the Capillary Tube

Place 20-30 mg of the solid on a watch glass and use a spatula to grind it to a powder; then use the spatula to gather the powder into a small pile. Stick the open end of a capillary tube into the pile and then invert the capillary; tap the sealed end on the tabletop to force the solid to drop to the bottom. (The instructor will also demonstrate some alternative methods for getting the sample to the bottom of the tube.) The height of solid at the bottom of the capillary should be about 2-3 mm.

#### **Using the Mel-Temp Apparatus**

Place the loaded capillary into one of the 3 slots on the Mel-Temp. Turn the Mel Temp power switch on and choose a voltage setting. The instructor will supply a

heating curve for the Mel-Temp that assists with this. The most important guideline in setting the voltage is that the temperature does not rise faster than 2-3 degrees per minute when the sample is melting. While the sample is heating, watch it while frequently checking the temperature. Note the temperature at which liquid starts to pool up in the bottom of the capillary. This is the start of your mp range. Continue to heat and observe, noting the temperature at which the sample appears to be a completely transparent liquid; this is the final temperature of the range (Cloudiness indicates solid particles suspended in the liquid, so if it is cloudy, it is not completely melted.) Dispose of used capillaries in the broken-glass waste container.

#### **Density Determination**

How to determine the density of your alum

#### **Materials Needed**

10 mL graduated cylinder, 50 mL Erlenmeyer flask, ethyl alcohol, alum, spatula

#### Procedure

- Determine the mass of the solid. This can be done by weighing out between
   1.0 and 1.5 g of the alum. Record the exact mass.
- 2. Determine the volume. Fill the 10 mL graduated cylinder halfway (5 mL) with ethyl alcohol. Record the volume.
- 3. Add the alum you have weighed into the ethyl alcohol, be careful not to spill the solid or allow any to cling to the wall of the cylinder. You want all the solid inside the liquid.
- 4. Record the new volume of the cylinder; the volume of the liquid displaced equals the volume of the object.
- 5. Determine the density of alum by dividing the mass by the volume.
- 6. Compare the density to the known density of alum (2.71 g / mL)

#### Data

Category (unit)	Amount
Mass of Alum (g)	
Volume of ethyl alcohol (mL)	
Volume of ethyl alcohol + alum (mL)	
Volume of alum (mL)	
Density of alum (g/mL)	

- Density Calculation
- Percent Purity Calculation

### **Bibliography/Resources**

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