Laboratory Manual

General Chemistry I Honors

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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:

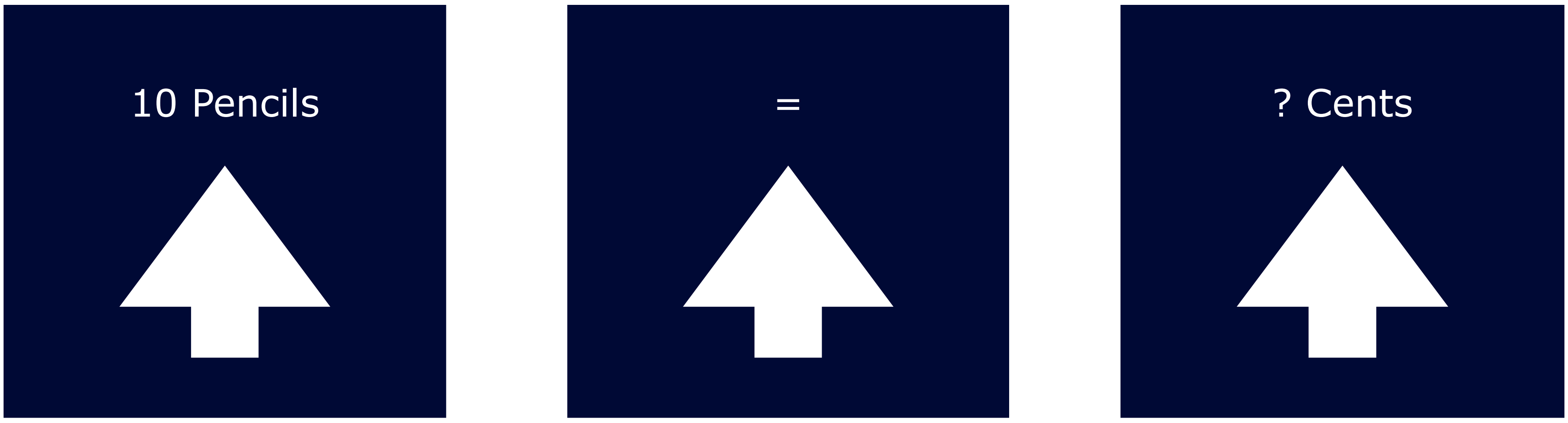
Problem 1

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:

(Q):



This query is given in the problem (number with units).

In these expressions, the "=" stands for "equivalent to".

The "?" stands for the number (+units) to be found.

- **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:

$$40 \text{ cents} = 8 \text{ pencils}$$

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

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

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

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 set-up of the question in Step 1.

Solve and Check Units

$$10\ \cancel{\text{pencils}} \times \frac{5.0\ \text{cents}}{1\ \cancel{\text{pencil}}} = 50\ \text{cents}$$

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 that 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 $\frac{3 \text{ ft}}{1 \text{ yd}}$.

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

• 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

• **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: $1 \text{ ft} = 12 \text{ in}$

- **Step 3:** UBF's from above link:

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

- **Step 4:** Solve and check units:

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

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: $100 \text{ yd} = ? \text{ in}$

- **Step 2:** Links: $100 \text{ yd} = ? \text{ ft} = ? \text{ in}$

$$1 \text{ yd} = 3 \text{ ft and } 1 \text{ ft} = 12 \text{ in}$$

- **Step 3:** UBFs:

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

- **Step 4:** Solve and check:

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

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 “right most” non-significant digit is counted as “zero,” and (2) non-significant figures amounting to five or more at the right most 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:

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

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: $\frac{-20.612\text{ g}}{?} = 1.224$ 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: $\frac{-20.6\text{ g}}{?} = 1.236$ 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 0.000000006 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: $\left(10^2 = 10^{+2}\right.$ and $\left.10^3 = 10^{+3}\right)$.

Table 1: Some Positive Powers of 10

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	$1000000 = 1 \times 10^6$

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.**

Table 2: Some Negative Powers of 10

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	$0.000001 = 1 \times 10^{-6}$

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. 260000000

Solution

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

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

C. $260000000 = (260000000.) = 2.600 \times 10^8$

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. 5×10^{10}

B. 4.58×10^{-4}

C. 1.65×10^0

D. 6.02×10^{23}

Solution

A. 50,000,000,000

B. 0.000458

C. 1.65

D. 602,000,000,000,000,000,000,000

Problem 7

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

A. 42.6×10^3

B. 259×10^0

C. 62.7×10^{-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:

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

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

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

$= 4.26 \times 10^4$

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

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

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.
$$\begin{array}{r} 6.02 \times 10^{23} \\ +3.01 \times 10^{23} \\ \hline =9.03 \times 10^{23} \end{array}$$

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

B.
$$\begin{array}{r} 6.02 \times 10^{23} \\ +\ 5.92 \times 10^{23} \\ \hline =11.94 \times 10^{23} \end{array}$$

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.

$$\begin{array}{r} 0.013 \times 10^{-1} \\ +\ 6.35 \times 10^{-1} \\ \hline =6.453 \times 10^{-1} \end{array}$$

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.

$$\left(6.3 \times 10^{3}\right) \times \left(3.4 \times 10^{7}\right) = ?$$

- **Step 1:** Collect all digit factors together and all exponential factors together:

$$\left(6.3 \times 3.4\right) \times \left(10^{3} \times 10^{7}\right) = ?$$

- **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:

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

Here is another example:

$$\left(1.3 \times 10^{-5}\right) \times \left(9.45 \times 10^{4}\right) = ?$$

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

$$12.285 \times 10^{-1} = 1.2 \times 10^{0} = 1.2$$

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:

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

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

$$\left(2.3 \times 10^3\right)^2 = ?$$

$$\left(2.3\right)^2 \times \left(10^3\right)^2 = \left(2.3\right) \times \left(2.3\right) \times 10^{(3\times2)} = 5.29 \times 10^6 = 5.3 \times 10^6$$

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

Negative Powers

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

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

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

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

Experiment One: Which Antacid Is Most Effective?

Pre-Lab Questions, Experiment Data Tables, and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment One Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

Pre-Lab Questions

1. To what class of compounds do antacids belong? Why is NaCl not a good antacid?

2. If 13.0 g of antacid A have neutralized 40 mL of 0.56 M HCl, while 6.00 g of antacid B have neutralized 25 mL of the same acid solution, then which of the two acids has the greater neutralizing power? Justify answer with calculations.

3. After taking an antacid, the pH of your stomach solution will:

   E. Increase

   F. Decrease

   G. Stay the same

4. Suppose an antacid has the formula $B(OH)_2$ where B is a dipositive metal ion. If your stomach acid is HCl, write a chemical equation that describes what happens after the antacid reaches the stomach.

5. What is the difference between acid neutralizers and acid blockers?

Introduction

There are different antacids marketed today, and they range in price from cheap to semi- expensive. The efficacy of each one is not dependent on price but rather on the active ingredients as well as each person's bodily response to the antacid. Independent of these two factors and to answer the effectiveness question, we can determine the acid neutralizing power of different antacids and rank each accordingly.

Background

Antacids are among the most heavily advertised pharmaceutical products: Brand A "consumes forty-five times its own weight in stomach acid." Brand B "coats your stomach with a pink, soothing lining." Brand C "spells R-E-L-I-E-F." In drugstores, the antacids' section often takes up a complete set of shelves. There you can see bottles with liquids, jars with powders, and boxes with tablets. Some preparations are pink, some green, and some white. Some are plain, and some are mint or orange flavored. If you needed to buy an antacid, which one would you choose? Which of the advertising claims would you most believe?

This is one case where you can, with little effort, really "check the claim." At least, you can do so regarding the neutralizing power of the antacid. First, however, you need to think about what an antacid really does.

The gastric juices present in your stomach are acidic and contain a carefully controlled amount of hydrochloric acid, HCl. Sometimes, likely due to poor eating or stress, our body reacts. Typically, this reaction causes more than the usual amount of acid to be produced in the stomach and hyperacidity results. (Hyperacidity means "too much acidity.") This is what some people call "acid indigestion."

What is the most immediate way to counteract hyperacidity? Just add enough of an appropriate base to neutralize the excess stomach acid. This is exactly what antacids are intended to do. How well they do it is what we need to find out.

Purpose

The purpose of this work is to determine the neutralizing capacity of various commercially available antacids, and in doing so help people in making informed decisions as to which antacid to use.

Materials

Rolaids, Maalox, Mylanta, Alka-Seltzer, Baking Soda, CVS-Brand Antacid, Burets, Buret Holder, Ring Stand, Erlenmeyer Flask, Stirring Rod, Medicinal Dropper, Phenolphthalein Indicator, 0.52 M Sodium Hydroxide solution, and 0.44 M Hydrochloric Acid solution

Procedure

1. Add approximately 0.5 grams of powdered antacid to a previously weighed Erlenmeyer flask.

2. To this flask, add 25.0 mL of the 0.44 M hydrochloric acid, and stir the mixture well until all the antacid has dissolved; any undissolved antacid additives will not have any adverse effect on the experimental results.

3. To this clear solution or suspension, add 2-3 drops of phenolphthalein indicator and titrate (the colorless or colored solution depending on the antacid) with the 0.52 M sodium hydroxide.

4. Record the amount of the base (sodium hydroxide) needed to reach the end point (where the color changes from colorless to light purple).

Data Table One

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Antacids	Mass 1 (g)	Mass 2 (g)	Average Mass (g)	Neutralizing Power
Rolaids				
Mylanta				
CVS Brand Antacid				
Baking Soda				
Maalox				
Alka-Seltzer				

Data Table Two

Antacids	Initial Buret Reading (ml)	Final Buret Reading (ml)	Volume of Base used (ml)	Mmol of Base used = mmol of Acid neutralized
Rolaids				
Mylanta				
CVS Brand Antacid				
Baking Soda				
Maalox				
Alka-Seltzer				

Post-Lab Analysis

Calculate the mmol of the initial HCl amount present (mmol = molarity x volume in mL), and then calculate the mmol of NaOH used (volume of based used in titration x molarity of base); the difference between the two mmols is the amount of acid neutralized by the antacid. Use this amount and divide by the average mass of the antacid used to determine the acid neutralizing power per gram of antacid. Additionally, report on the active ingredients of each antacid and argue if one component of the antacid affects the neutralizing potential.

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Experiment Two: Extraction of Fat and Determination of Fat Content of Candy Bars

Pre-Lab Questions, Experiment Data Tables, and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Two Word Document Download.

Pre-Lab Questions

1. Why is hexane/pentane the proposed solvent for this experiment? Why wouldn't water or any polar solvent work for the extraction?
2. When 2.788 g of Candy A was extracted with hexane, 0.884 g of total fat was obtained. Calculate the percentage of fat in Candy A.
3. Is the use of pentane or hexane expected to produce the same or different results? Explain.
4. How close do you suppose your results will be to the reported value? Explain.
5. Based on anticipated results, would you advise anyone to consume as much or as little candy as they could daily? Explain.

Introduction

Until recently, chocolate manufacturers were never held liable for revealing the fat content of their products. Contrary to popular belief and to the surprise of most consumers, the sugar content in each candy bar is not responsible for the taste; it is the fat. In fact, the more fat these manufacturers can add into their products, the better they taste. How much fat is really in each candy bar? We will extract the fat from Symphony, Hershey's, Nestle Crunch, and Dove candy bars to compute the total fat content and compare our results to their manufacturer's reported numbers.

Background

Our body needs some fats to conduct some of its functions. For instance, we need fat for healthy hair and skin, body insulation, and filler for our fat cells. Fats such as linoleic and linolenic acids are "essential fats" needed for brain development, inflammation control, and blood clotting. These fats cannot be made in the body, so we must rely on external sources to obtain them.

During exercise, our body uses calories we derive from carbohydrate sources, but that is only good enough for 15-20 minutes of exercise. Longer exercise time means that other calorie sources must be present and what better source than good old reliable fat. One gram of fat stores more calories than a gram of protein and carbohydrate combined.

Fat can be saturated or unsaturated. Saturated fat is bad for us because it raises our LDL (Low Density Lipids-bad cholesterol) level, which elevates the risk for stroke, heart attack, and other health problems. Unsaturated (mono and poly) fat, on the other hand, lowers our LDL levels.

Trans fatty acids (also called hydrogenated fats) are also unhealthy. We see them in most products because they are used to keep products fresh. Trans fats lower HDL (High Density Lipids-good cholesterol) levels in the system.

Purpose

The purpose of this work is to determine the fat content in each candy product, thus checking manufacturers' claims. In doing so, the goal is to help people make informed decisions as to which candy to consume and how much.

Materials

Dove, Nestle Crunch, Hershey's, Symphony, magnetic stir plates, magnetic stirrers, mortar and pestle, hexane/pentane, medicinal droppers, beakers, and balance scale.

Procedure

1. Each candy bar must be frozen in a -80°C degree freezer overnight.
2. Weigh the whole candy bar initially for its total mass, then place some pieces of the candy bar in a mortar and pestle and grind to powder.
3. Weigh about 1.0-1.2 g of the resulting powder and transfer it into a 250 ml beaker.
4. Add two hundred milliliters (200 ml) of hexane/pentane to sequentially extract the fat.
5. Repeat the pentane extraction about two more times, combine the extracts, and leave under a fume hood to dry (it may take up to five days).
6. Weigh the extracted fat and complete the table of results below.

Data and Results

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Candy Bars	Mass 1(g)	Mass 2 (g)	Mass 3 (g)	Mass of Fat Extracted (g)	Mass of Fat Extracted (g)	Mass of Fat Extracted (g)	Average Fat Extracted (g)
Nestle Crunch							
Dove							
Symphony							
Hershey's							

Candy Bars	Amt. of Fat/g	Amt. of Fat/g	Amt. of Fat/g	% Fat/ g	% Fat/g	% Fat/g	Average Fat %/ g	Mass of Fat (total) Reported (g)
Nestle Crunch								
Dove								
Symphony								
Hershey's								

Post-Lab Analysis

After one week of drying (to evaporate the solvent), weigh the extracted fat. Calculate the amount of fat per gram of candy. Report the percentage (%) of fat extracted from each candy bar, based on the initial mass of the candy bar.

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Experiment Three: Identifying Some Constituents of Bones

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Pre-Lab Questions

1. What test, if any, would you carry out to test the collagen obtained?

2. List three characteristics and/or functions of collagen.

3. A solution containing inorganic ions is known to have a pH of 5.00. How many moles of H^+ are present in 400 ml of this solution?

Introduction and Background

Bones are one of nature's many "composite materials" and have unique characteristics such as mechanical strength, hardness, and relatively low density. Bones consist of a tough, somewhat flexible matrix made of collagen (a structural protein) and fibers. This matrix acts as a support network for the inorganic salts that consist mainly of calcium phosphate and other minor constituent ions, such as Mg^{2+}, Fe^{3+}, Na^+, F-, OH^-, CO_3^{2}. Inorganic salts make up almost 65% of the mass of adult human bones and are responsible for their hardness.

It is possible, through the relatively simple sequence of chemical operations outlined in the flow-chart on the next page, to break apart and separate the major organic and inorganic constituents of bones. Once separated, each component can be individually identified by appropriate analytical testing.

The chemical procedure outlined in the flowchart is an example of a "qualitative analysis scheme." Its success depends on knowledge of chemical facts, thoughtful planning, and the careful execution of each step.

Purpose

To provide the student with experience in the qualitative analysis scheme, illustrate some reactions of biologically important ions, and acquaint the student with chemical composition of bones.

Materials

Small piece of chicken bone, 250 ml beakers, glasses, 3 M hydrochloric acid, distilled water, wash bottle, hot plate, funnel, funnel support, three filter paper circles, pH paper, glass rods, spatulas, test tubes, test tube rack, 3 M sodium hydroxide, NaOH, 6 M ammonia, NH_4OH, ammonium oxalate, $(NH_4)_2C_2O_4$, Conc. nitric acid, HNO_3, potassium hexacyanoferrate(II), $K_4[Fe(CN)_6]$, potassium thiocyanate, KSCN, 0.1% copper(II) sulfate, $CuSO_4$, and ammonium molybdate reagent

Procedure

Obtain a piece of bone (break into small pieces, if possible) and scrape off any residue of meat, fat, and marrow (if present). Rinse the bone well with water and place it in a small beaker. Add about 20 mL (just enough to cover the bone pieces) of 3 M hydrochloric acid. Cover the beaker with a watch glass and place it in under the hood for one week.

1. After soaking for one week in HCl, the inorganic part of the bone should have dissolved, leaving behind a soft mass consisting of the undissolved collagen structure.

2. Pour the solution into a clean beaker for later use (the solution may be cloudy, with some floating matter); leave the collagen mass in the original beaker.

3. Wash the collagen mass by squirting enough distilled water over it, swishing it around gently, and pouring the water off into a waste collection flask. Repeat this step several times, until the wash water appears neutral when a drop of it is touched to a strip of pH paper.

4. Discard the washing liquid. By doing so, you will have separated the inorganic material (solution) from the organic material (undissolved soft mass).

5. Cover the well-washed, undissolved organic material with distilled water and place the beaker on a hot plate. Allow the water to boil gently for about 30 minutes, making sure that too much water does not evaporate. The heating causes some of the organic material to dissolve in the water.

6. Remove the beaker from the hot plate and set it down to cool.

7. Prepare a fluted filter paper in a clean funnel, wet the filter paper with distilled water, and place the funnel on a ring stand. Filter the cloudy solution from step 1, collecting the liquid in a clean beaker. After all liquid has passed down, discard the paper filter and its contents.

8. Check the acidity of the clear filtered solution by placing a drop of it on a piece of pH paper. It should cause a red (acidic) stain.

9. Gradually add a 6 M ammonia (NH_4OH) solution dropwise while stirring. After the first few drops have been added, check the pH after each addition of ammonia. As soon as a drop of solution touched on a strip of pH paper produces a blue color (basic), stop adding the ammonia.

10. Add acetic acid dropwise while stirring, until a drop of solution produces a red color on the pH paper.

11. In this step, the successive additions of ammonia (weak base) and acetic acid (weak acid) serves to raise the pH and produce a slightly acidic buffered solution. Under these conditions, Fe(III) phosphate, $FePO_4$, is insoluble and precipitates, whereas calcium phosphate remains dissolved.

12. Prepare another fluted filter paper (in a clean funnel) and filter the mixture obtained in Steps 9 and 10. Collect the clear filtered solution in a clean beaker.

13. Wash the solid collected on the filter by filling the funnel with water and allowing the water to drain into a waste-collection flask. Repeat this washing procedure three times.

14. Discard the washing liquid.

15. To the clear filtered solution from Step 12, add 5 mL of ammonium oxalate solution. A white precipitate of insoluble calcium oxalate, CaC_2O_4, should form, indicating the presence of calcium ions in the bone structure.

16. Scrape off the washed precipitate collected on the filter paper in Step 12. Place it into a clean container and add about 10 mL of 3 M HCl. Warm the mixture gently on a hot plate to promote dissolution.

    Use part of the solution obtained in Step 16 to carry out the following test for phosphate ions PO_4^{3-}. Place 2 mL of solution into a clean test tube. Add a few drops of concentrated nitric acid, HNO_3. ***CAUTION:*** Concentrated nitric acid is a highly corrosive material, so warm the mixture gently; do not boil. To the hot solution, add a few drops of ammonium molybdate reagent; a bright yellow precipitate is a positive test for phosphate ions.

17. Use part of the clear solution obtained in Step 16 to carry out the following tests for iron. Place 2 mL of solution in a clean test tube. Add a few drops of solution of potassium hexacyanoferrate (II), $K_4[Fe(CN)_6]$. A blue color indicates the presence of Fe^{3+} ions.

18. Place 2 mL of solution in another clean test tube, Add a few drops of solution of potassium thiocyanate, KSCN. A red color confirms the presence of Fe^{3+}ions.

Post-Lab Analysis

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Correctly list all the ions present in your assigned bone.

Bibliography/Resources

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Experiment Four: Synthesis of Tylenol

Pre-Lab Questions and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Four Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

Pre-Lab Questions

1. If you start with 0.75 g of p-aminophenol and 2.0 ml acetic anhydride (assume this is in excess), calculate the amount of Tylenol expected.
2. Why is it important to acidify the p-aminophenol prior to its reaction with acetic anhydride? Explain.
3. How is the synthesis (making) of Tylenol similar to that of Aspirin? Which starting material is the same or different?
4. How would you estimate the dollar amount involved in the making of one tablet of Tylenol, using the same starting material proposed in this experiment?
5. State some properties of Tylenol.

Introduction and Background

Generically known as acetaminophen, Tylenol is an analgesic used in the treatment of minor aches and in fever reduction. It is the most popular painkiller in the United States, and Americans take over eight billion pills (tablets or capsules) annually. Used at recommended dosage, Tylenol possesses very minor risk but when taken too often, either intentionally or unintentionally for better and faster results, the consequences can be dire. Tylenol, when used as directed, it is safe and effective. The makers of Tylenol say this: "It's important for people to know that it's not the recommended dosage of acetaminophen that poses the risk. Rather, it is when people take more than the recommended dose".

As with any medicine, paying attention to the dose is important. Here's what the FDA says about this: *"Acetaminophen can cause serious liver damage if you take too much. It is very important to follow your doctor's directions and the directions on the medicine label."*

Tylenol is also a highly effective, fever reduction (anti-pyretic) agent. In fact, the use of Tylenol, instead of aspirin, to treat fevers in children has reduced the occurrence of Reye's syndrome, an often-fatal form of liver failure. Some symptoms of liver failure because of excessive consumption of Tylenol include vomiting, fatigue, loss of appetite, dark urine and stool and abdominal pain.

Purpose

Synthesis and identification of Tylenol from p-aminophenol and acetic anhydride.

Materials

p-aminophenol, acetic anhydride, Erlenmeyer flask, Buchner funnel, decolorizing charcoal (Norite), water, water aspirator or vacuum, spatula, concentrated hydrochloric acid, filter paper, and glass rod

Procedure

1. Weigh 2.1 g of p-aminophenol into a 125 mL Erlenmeyer flask.

2. Add 35 mL of water followed by 1.5 mL of concentrated hydrochloric acid.

3. Swirl the flask to dissolve the amine hydrochloride formed.

4. Add a few more drops of concentrated acid, if necessary, to dissolve the amine completely as the hydrochloride (it will be difficult to determine since the solution is very dark).

5. Add 0.3-0.4 g of decolorizing charcoal (Norite) to the solution (this is much more than usual but necessary because the crude p-aminophenol contains a lot of polymeric material), swirl the solution on a steam bath for 4-8 minutes, and periodically check to see if the solution is decolorizing (it will be difficult to determine since the solution is dark).

6. Remove the charcoal by gravity filtration into another 125 mL Erlenmeyer flask, using fluted filter paper while the solution is warm.

7. Rinse the filter paper with 1 mL of water.

8. If the charcoal comes through the filter paper, it may be necessary to refilter or to use a filter aid, Celite.

9. The filtrate may be clear or, more likely, a tea color. If the solution is deep brown, add 0.1 g of Norite, heat on the steam bath for a few minutes, and filter. ***The filtrate will darken with time!***

10. While decolorizing the p-aminophenol, prepare a buffer solution by dissolving 2.5 g of sodium acetate trihydrate in 7.5 mL of water, which will give 8.8 mL of solution. Clarify the solution by gravity filtration, if necessary.

11. Warm the filtered aqueous p-aminophenol hydrochloride solution on a steam bath, then add the buffer solution in one portion, with swirling.

12. Immediately add 2.0 mL of acetic anhydride while continuing to swirl the solution. Continue heating on the steam bath, while swirling vigorously for 10 minutes.

13. Cool the solution in an ice-water bath, stirring with a glass rod until the crude acetaminophen begins to crystallize. A little bit of rubbing/scratching with a glass rod near the surface often stimulates the crystallization.

14. After crystallization begins, allow the solution to sit in the ice bath for an hour. Filter your product using a Buchner funnel and the water aspirator or house vacuum line. Wash (rinse) the crystals once with small amount of cold water (a few mL should suffice). Allow the crystals to air dry under vacuum.

15. Collect the crude crystalline product and weigh to the nearest tenth of a gram. Record the weight.

Recrystallization Procedure

1. To all but 100 mg of your crude Tylenol, dissolve the solid using the minimum amount of hot (boiling) water. Do this carefully and avoid using too much hot water.

2. Keep the solution hot and add another 2 mL of hot water. If there are no insoluble particles in the solution, you can allow it to cool slowly without having to first filter. If not, decant the hot solution or try to remove the particles with a spatula or Pasteur pipette, while keeping the solution warm.

3. After crystallization begins, cool the solution more rapidly using an ice bath. When crystallization completes (15 minutes), collect the crystals, rinsing once with a few mL of cold water, and air dry.

4. Record the weight of the dry, recrystallized Tylenol.

Post-Lab Analysis

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Calculate the percent recovery from recrystallization (for instance, if you obtain 0.62 g of recrystallized product after starting with 1.0 g crude product, you have 0.62/1.0 x 100 = 62% recovery). Calculate the theoretical yield of dry recrystallized product from the initial amount of p-aminophenol used. Use the actual recrystallized amount obtained and calculate the percent yield of Tylenol. Using the melting point apparatus, obtain the melting point of your Tylenol (Lit MP is 169-170.5°C).

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Experiment Five: Separation and Identification of Components of Combination Analgesics

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Pre-Lab Questions

1. Which component of the combination analgesic would come out first? Which solvent would you use in its extraction and why?

2. Cheysuprin, by its name contains sucrose, aspirin and one other analgesic. Can you use an organic solvent to differentiate the two analgesics?

3. Compare the structures of phenacetin and acetaminophen. What is the same about them and what is different?

4. Name all the functional groups present in acetaminophen.

5. Is phenacetin water soluble? Explain.

6. How is the synthesis (making) of Tylenol similar to that of aspirin? Which starting material is the same or different?

Introduction and Background

Among the over-the-counter pharmaceuticals with the greatest sales are the common analgesic, led by aspirin. Aspirin, or acetylsalicylic acid (ASA), was once the only choice for the relief of pain (analgesic properties) for reducing fevers (antipyretic properties) and reducing swelling, particularly the swelling associated with rheumatoid arthritis (anti-inflammatory properties). Even today, over twenty million pounds of aspirin are sold in the US annually. Aspirin is prepared from salicylic acid, which was originally derived from salicin, a compound first isolated from the bark of the white willow tree (Salix alba). The use of aqueous extracts of willow bark to relieve fever and pain was first reported in the late 1700's in England but goes much further back in history. In the middle 1800's, salicylic acid was obtained from willow bark, from the meadowsweet plant (Spirea Ulmaria), and from oil of wintergreen (from Gaultheria procumbens). It was found to be effective as an analgesic and antipyretic compound. However, physicians also found that salicylic acid was so acidic that it can cause irritation and bleeding in the mucous membranes of the digestive tract. In 1893, Felix Hofmann, a chemist working for the German dye and pharmaceutical company, Bayer, found a way to prepare the acetyl derivative of salicylic acid (ASA) in a large scale. This derivative proved to be as effective as salicylic acid in the relief of pain and fever with less irritability. Bayer marketed this drug under the trade name aspirin, and for many years, controlled a lucrative market in over-the-counter sales of this pharmaceutical.

The advent of aspirin did not stop pharmaceutical firms from investigating other potentially analgesic drugs. There are people who are allergic to aspirin or who cannot take aspirin due to aspirin's abrasive side effects of the digestive tract. Once again, Bayer was first with the next effective analgesic, phenacetin, which was prepared from p-aminophenol, a by-product of the manufacture of another company made by Bayer. For a number of years, phenacetin, which had exceptionally good analgesic and antipyretic properties, was used as a component of APC tablets, an analgesic preparation that contains aspirin, phenacetin, and caffeine. However, phenacetin has now been supplanted by acetaminophen, which is easier to produce. Acetaminophen is the sole analgesic component in over-the-counter products such as Tylenol and Datril. However, acetaminophen and phenacetin also cause a type of anemia called methemoglobinemia. While it does not seem to be a major problem, certain people are more susceptible to this anemia and should avoid using products containing acetaminophen.

Salicin

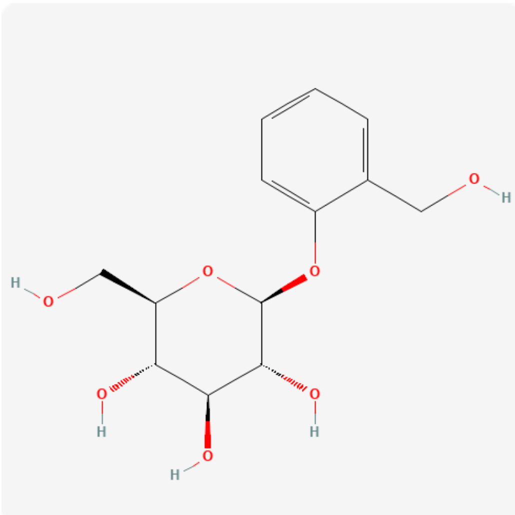


Figure 1

Salicylic Acid

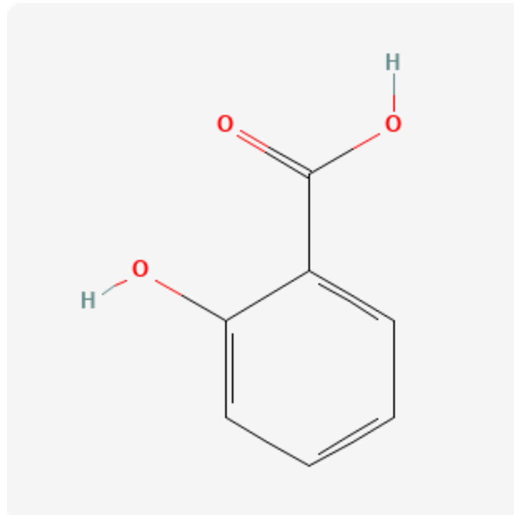


Figure 2

Acetylsalicylic Acid

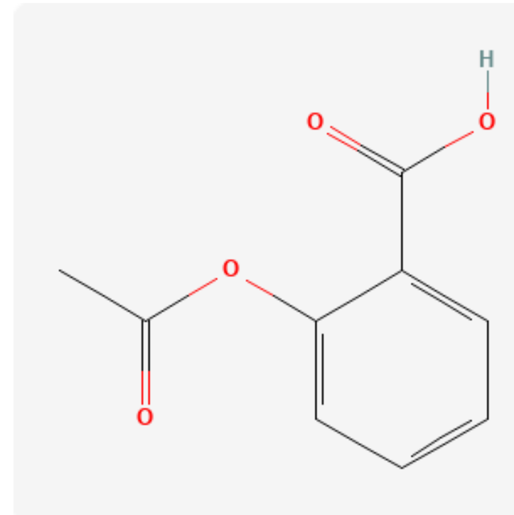


Figure 3

Phenacetin

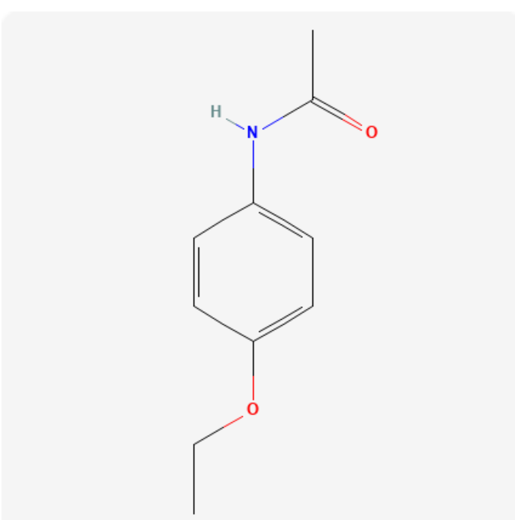


Figure 4

Acetaminophen

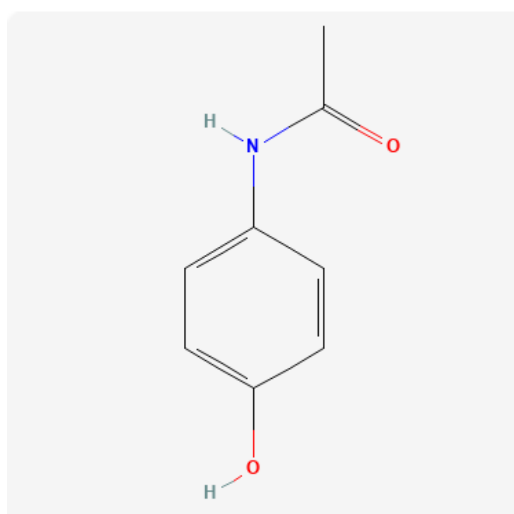


Figure 5

Caffeine

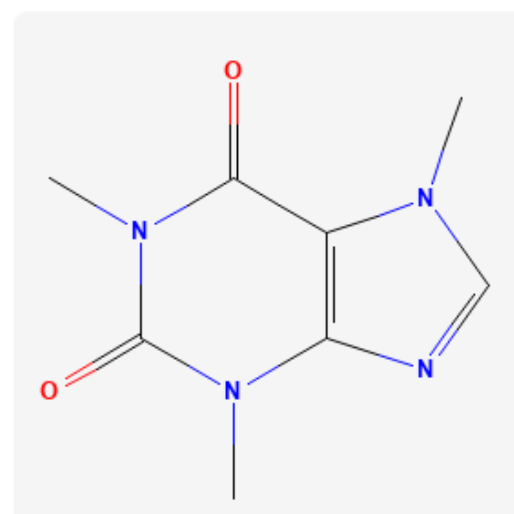


Figure 6

P-Aminophenol

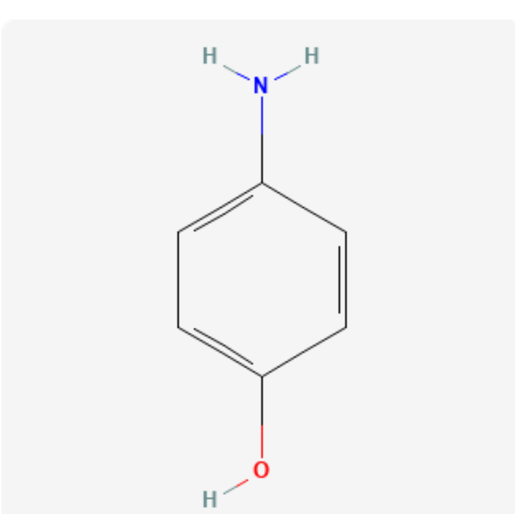


Figure 7

More recently, another compound has become popular as an over-the-counter product. This is the compound Ibuprofen, which is principally an anti-inflammatory compound. It also has analgesic and antipyretic properties and was first marketed under the trade name Motrin. It is more effective than aspirin as an analgesic and requires lower doses to achieve the same pain relief. It also has effectiveness over a wider dose range than aspirin. Like the other analgesics, Ibuprofen does have some side effects, and people who are allergic to aspirin often take ibuprofen. People with kidney disease, ulcers, hypertension, and heart disease are also advised not to take ibuprofen.

Ibuprofen

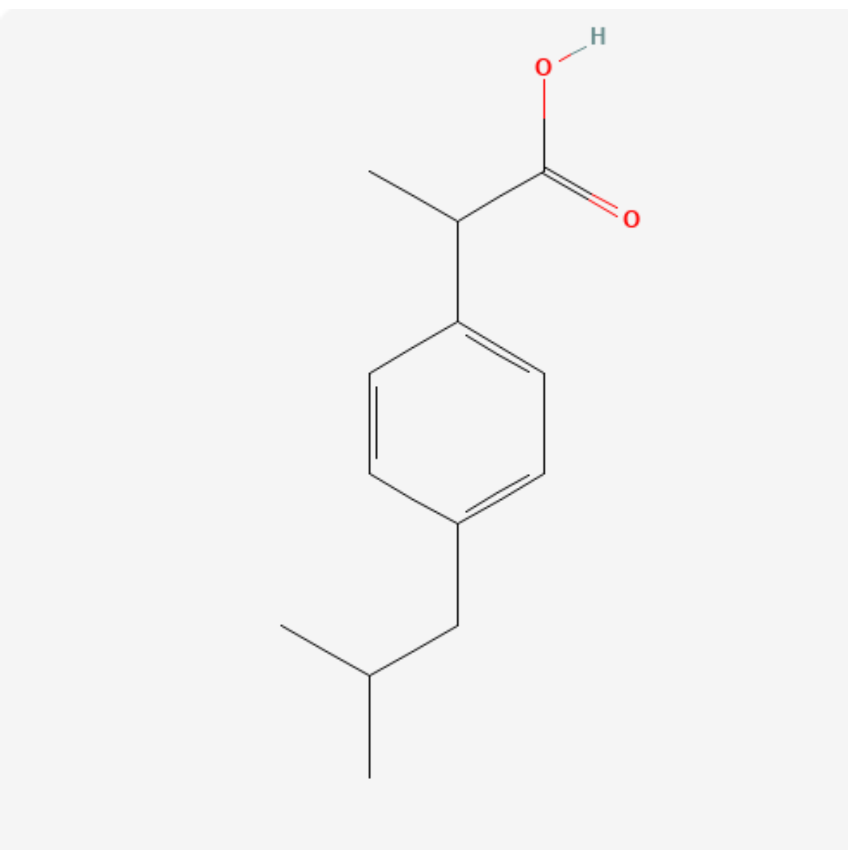


Figure 8

Each tablet or capsule of a commercial analgesic product contains only a small amount of the actual drug. For example, each 5-gram Bayer Aspirin tablet contains only 325 mg of aspirin. Each Extra-Strength Excedrin tablet contains 250 mg of acetaminophen, 250 mg of aspirin, and 65 mg of caffeine. The remainder of the tablet is made of inert filters, such as starch and binders to hold everything together. This is typical of many over-the-counter and prescription pharmaceuticals. Consequently, if one were to analyze a pharmaceutical preparation, it is often necessary to separate the individual components from one other. One way to accomplish this is to take advantage of the different solubilities of the individual components. For example, many of the fillers and binders are soluble in water but not in organic solvents. On the other hand, the active ingredients are often more soluble in organic solvents than in water. Alternatively, different functional groups within the mixture of organic compounds will undergo characteristic reactions. These differences can be used to achieve a separation.

Objective

In this experiment, you will separate the individual components of a special Cheyney-made analgesic product called Cheysuprin. There are two versions of the analgesic mixtures: Cheysuprin-A and Cheysuprin-B. Both products contain aspirin as one analgesic component and sucrose as a filler. One contains phenacetin as the second analgesic component, while the other contains acetaminophen. You will separate the individual components of one of the Cheysuprins, as assigned by your instructor, based upon the different solubilities and reactivities of each component, then determine the amounts of each component in the Cheysuprin.

Sucrose

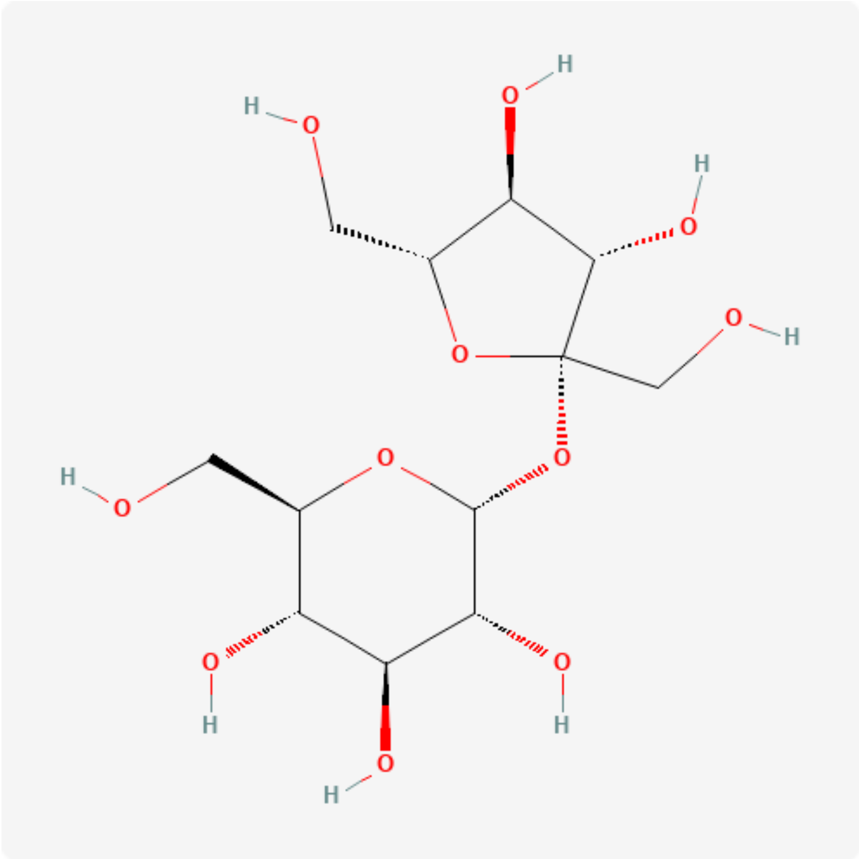
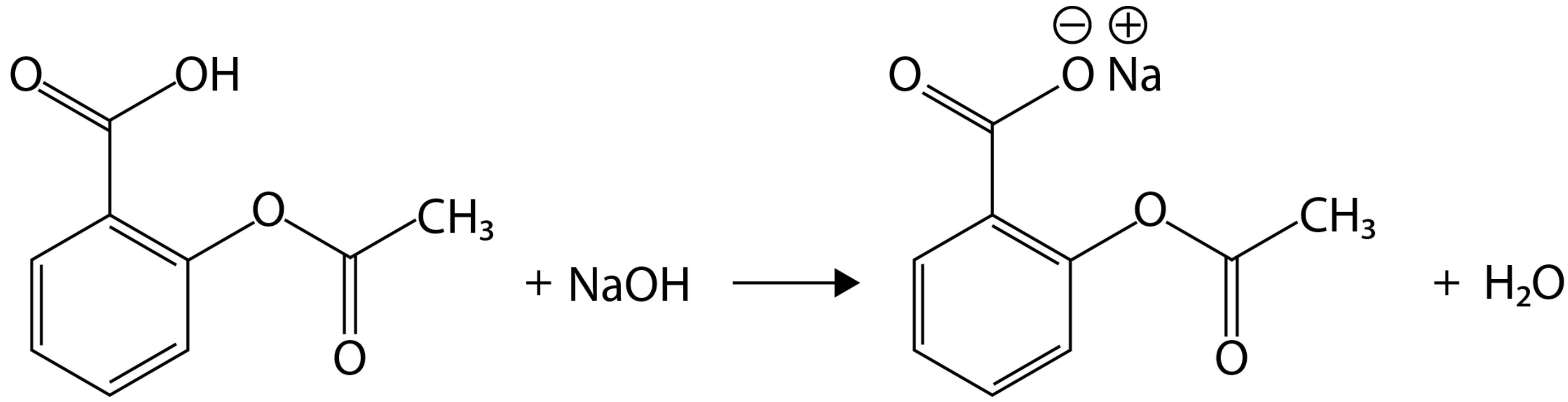


Figure 9

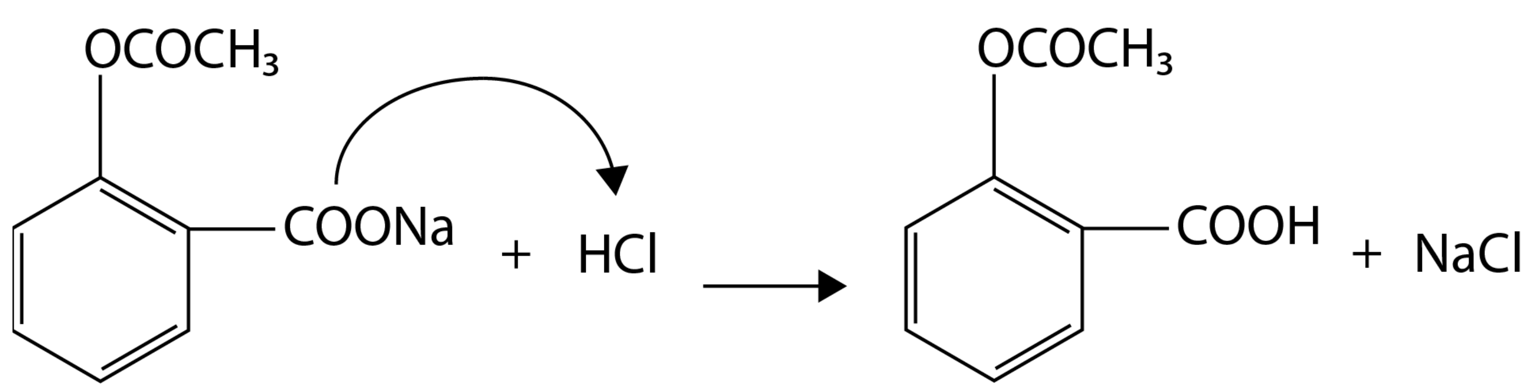
The first step of the separation will be based on different solubilities. Sucrose, or table sugar, is very water soluble, but acetylsalicylic acid, acetaminophen, and phenacetin are not particularly soluble in water. On the other hand, acetylsalicylic acid, acetaminophen, and phenacetin are all soluble in dichloromethane, while sucrose is not. By first mixing the Cheysuprin with dichloromethane, the sucrose can be removed by simple filtration. This is an example of a separation based on solubility. Next, the dichloromethane is extracted with a sodium hydroxide solution. The acetylsalicylic acid, as the name implies, is an acid and sodium hydroxide is a base. They react, as shown below, to form a salt that is now more soluble in water than dichloromethane. The acetaminophen or phenacetin is not affected by treatment with the base unless heat is applied, so the third component remains dissolved in the dichloromethane. Since water and dichloromethane are not miscible, the layers can be separated, thus separating the two analgesic components. The acetylsalicylic acid can then be recovered by converting the salt back to the acid with concentrated hydrochloric acid.



Acetylsalicylic Acid + NaOH conversion to Sodium Acetylsalicylate + H_2O

Chemical Hazards/Caution

- **Acetylsalicylic Acid:** poison, irritant
- **Acetanilide:** toxic, irritant
- **Phenacetin:** cancer suspect agent, mutagen
- **Dichloromethane:** carcinogen, toxic, irritant
- **Sodium Hydroxide:** toxic, corrosive agent
- **Hydrochloric Acid:** poison, toxic by inhalation, corrosive irritant



Sodium Acetylsalicylate + HCl conversion to Aspirin + NaCl

Materials and Supplies

Cheysuprin-A, Cheysuprin-B, 125 mL Erlenmeyer flasks, dichloromethane, spatula, weighing paper, balance, fluted filter paper, funnel, ring stand, O-rings, distilled water, separatory funnel, 1 M sodium hydroxide, NaOH, ice bath, conc. hydrochloric acid, HCl, glass rod, and pH paper

Procedure

1. Weigh approximately 2.5 g of the assigned Cheysuprin (unknown analgesic) sample into a 125 mL Erlenmeyer flask.
2. Add 50 mL of dichloromethane and vigorously swirl the mixture until no more of the solid appears to dissolve.
3. Make sure that any clumps of the solid are broken up during the stirring. Weigh a fluted piece of filter paper.
4. Filter the mixture through the pre-weighed filter paper by gravity filtration.
5. Wash the collected solid (the sucrose) with 5 mL of dichloromethane.
6. Place the filter paper containing the collected sucrose on a watch glass and allow it to dry completely (it may take a couple of days).
7. When the filter paper and sucrose are completely dry, weigh the filter paper plus the solid, and calculate the amount of sucrose recovered.
8. Report the results as a weight and percent of the total weight of the Cheysuprin sample (percent composition).
9. Add the dichloromethane solution to a separatory funnel and extract it with two 25 mL portions of 1M sodium hydroxide solution.
10. Add one portion of the sodium hydroxide solution to the separatory funnel containing the dichloromethane solution, stopper the funnel, and place a finger over the stopper to hold it in place.
11. Shake the funnel back and forth gently for 1-2 minutes (release the stopper every 30 seconds to release fumes. Point away from your face and others!). Keep shaking after venting.
12. When the extraction period is finished, release the pressure one final time, close the stopcock, and place the funnel on a ring stand/clamp.
13. Remove the stopper and drain the lower dichloromethane layer into a flask.
14. When the layer is removed, close the stopcock, and pour the aqueous layer from the funnel into a different Erlenmeyer flask.

15. Return the dichloromethane layer into the funnel and repeat the separation procedure with a second portion of sodium hydroxide.
16. Combine the aqueous layer in the same flask and save the dichloromethane layer in a separate flask for the last step.
17. Cool the combined aqueous layers in an ice bath, and slowly add 10 mL of concentrated hydrochloric acid, with stirring.
18. When the addition is complete, test the pH of the solution by inserting a glass rod, then touching the solution at the tip of the rod on pH paper. (If pH is 2 or lower, add additional HCl until the pH is 2 or below.)
19. Continue to cool the aqueous solution in the ice bath until all the solid acetylsalicylic acid forms.
20. Collect the solid by gravity filtration through a piece of pre-weighed filter paper and wash the solid with 5 mL of cold water.
21. Allow the water to pass through the filter paper in the funnel.
22. Open the filter paper on a watch glass and place under the hood for the collected solid to dry (it may take a couple of days).

Post-Lab Analysis

Pre-Lab Questions and Post-Lab Analysis are available for Word Document download via Google Drive: Experiment Five Word Document Download. Make a copy of the document in a personal Google Drive account or download the document in order to edit.

List the components of Cheysuprin-A or Cheysuprin-B. Calculate the amount of each component, relative to the initial mass of your assigned analgesic.

Bibliography/Resources

Sneden, A. T. (1995) *Organic Chemistry Laboratory Manual*, Virginia Commonwealth University.

Figure References (Return to Experiment 5)

Figure 1: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 439503, Salicin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Salicin.

Figure 2: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 338, Salicylic Acid. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Salicylic-Acid.

Figure 3: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 2244, Aspirin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Aspirin.

Figure 4: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 4754, Phenacetin. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Phenacetin.

Figure 5: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 1983, Acetaminophen. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Acetaminophen.

Figure 6: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 2519, Caffeine. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Caffeine.

Figure 7: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 403, 4-Aminophenol. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/4-Aminophenol.

Figure 8: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 3672, Ibuprofen. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Ibuprofen.

Figure 9: National Center for Biotechnology Information (2023). PubChem Compound Summary for CID 5988, Sucrose. Retrieved January 19, 2023 from https://pubchem.ncbi.nlm.nih.gov/compound/Sucrose.