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Experiment 1
Synthesis of "Copper Carbonate": What is it?

Chemistry 132
Spring 2013

Background

A careful reading of an advanced inorganic chemistry text will convince you that $CuCO_3$ can only be made under a high pressure of CO_2. What we call copper carbonate is a complex salt referred to as a basic copper carbonate, basic because the material has hydroxide groups as well as carbonate groups. There is more than one basic copper carbonate and several have been known since ancient times. These include the following:

name	color	formula
malachite	green	$Cu(OH)_2Cu(CO_3)$
azurite	blue	$Cu(OH)_2Cu_2(CO_3)_2$

Synthesis

Your synthesis is on the surface a straightforward metathesis (double displacement) reaction:

$$CuSO_4(aq) + Na_2CO_3(aq) \rightarrow CuCO_3(s) + Na_2SO_4(aq)$$

The isolation step succeeds because only the desired product "$CuCO_3$" is insoluble and can be isolated by filtration.

Chemical Analysis

As you will discover when you complete the *Preparation* page for this experiment, "$CuCO_3$", malachite and azurite all have different mass percent Cu. This difference is the basis for your analysis of the material you make in the lab. Copper carbonates, hydroxides, and oxides can all be converted to metallic copper by treatment with $H_2(g)$ or $CH_4(g)$ at elevated temperatures. The laboratory gas tap serves as a convenient source of natural gas, which is chiefly CH_4. You will construct an apparatus for the reduction of copper salts. By weighing the material put in the reactor and weighing the copper present after reduction, you will be able to determine the mass percent copper of the original sample.

Statistical Analysis

How accurate is the value you obtain for % Cu? This is a difficult question to answer with just your data alone, but statistical treatment of the class data will allow you to make a good estimate of the accuracy of the average of the class data.

References

C. Zidick, T. Weismann: *"The Reduction of CuO with Burner Gas and without a Fume Hood." J. Chem. Ed.* **1973**, *50*, 717-718.

A. B. Hoffman, A. J. Hoffman: *"Reduction of Copper(II) Oxide by Alkanes of Low Molecular Weight." J. Chem. Ed.* **1974**, *51*, 418-420.

D. Sheeran: *"Copper Content in Synthetic Copper Carbonate: A Statistical Comparison of Experimental and Expected Results." J. Chem. Ed.* **1998**, *75*, 453-456.

Experimental Procedure

Weigh all reactants and products accurately on the analytical balances! Always record all the digits of the mass reading from the balance.

Synthesis of "$CuCO_3$." Prepare a solution of copper sulfate by adding 3.8 g (0.015 mol) $CuSO_4 \cdot 5H_2O$ to 40 mL of H_2O. Prepare a solution of Na_2CO_3 (2.0 g, 0.019 mol) in 60 mL H_2O. Slowly add the copper solution to the sodium carbonate solution. Note any observations.

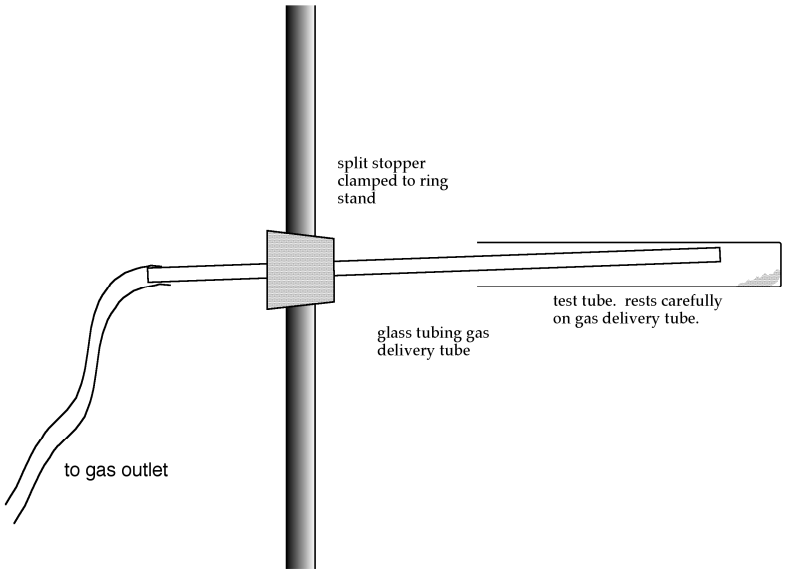
Warm the resulting mixture to 55-60 °C on a hot plate. Stir gently. Note any color change or other observations. Cool the reaction mixture by immersing the beaker in a room temperature water bath.

Once the mixture has reached room temperature, collect the product by filtration with a Büchner funnel. When the product looks dry, carefully disconnect the vacuum hose. The product has the consistency of modeling clay at this point and is difficult to get off of the filter paper. Slowly, pour 20 mL acetone onto the precipitate, disturbing the precipitate as little as possible. Wait about 30 seconds, then reconnect the aspirator hose and allow all the acetone to be pulled through the product. Repeat with another 20 mL portion of acetone. Continue to apply suction. The layer of product should begin to crack irregularly, like a dry lake bed. After a minute or so, disconnect vacuum and collect your product. Pulverize any clumps in your product and dry under a heat lamp for 5 minutes. Weigh your product. Assuming the product to be $CuCO_3$, what is your percent yield?

Caution: Acetone is highly flammable. Keep it well away from open flames.

Analysis of "$CuCO_3$." Construct an apparatus for reduction of the product similar to the diagram below. Determine the weight of a clean dry 18 × 150 mm test tube. To this tube, add approximately 0.4 g of product, weighed with milligram precision. Take care to concentrate the material at the bottom of the tube—keep the walls of the tube clean! Weigh the tube and copper carbonate together to determine the exact mass of copper carbonate. Once the copper carbonate

has been reduced to metallic copper some of the copper will adhere to the test tube walls, which makes it necessary to weigh by difference.



Once the test tube is loaded with material, reassemble the reduction apparatus. Begin a gentle flow of gas through the test tube, being careful not to disturb the solid with the flow of gas. Attempt to light the gas exiting the test tube. If it does not light, slowly increase the gas flow. When it does light, adjust the gas flow so that the flame height is 5–7 cm tall. This flame serves to burn off the excess methane not used to reduce the copper carbonate and will also oxidize any carbon monoxide formed in the reduction to carbon dioxide.

Now, light a Bunsen burner and, using the hottest part of the flame, heat the area of the test tube that contains the copper carbonate. Heat for at least 5 minutes and be sure to note any color changes or other changes that occur during the reduction. After reduction is complete remove the Bunsen burner but allow the flame to burn from the mouth of the test tube for 5 minutes or until the copper in the test tube is cool. Next, turn off the burner gas to extinguish the flame and, once the entire test tube is at room temperature, disassemble the apparatus. Weigh the tube to determine how much copper it contains. Tap the tube and scrape the inside with a spatula to remove some copper. Does it have metallic properties?

Chemical Analysis

Report your results as directed by the lab instructor. You might write them on an erasable marker board or enter them into a spreadsheet program on a computer in the laboratory. The instructor will inform you how to retrieve the class data.

From the class data, obtain an average, a standard deviation (SD), and a 95 % confidence interval. Use the Q-test to discard outliers (see Appendix).

Use Student's *t*-test (Appendix) to decide, at the 95 % confidence level, whether or not the formulation $CuCO_3$ can be ruled out. Can one of the formulas for basic copper carbonate also be ruled out?

Laboratory Report Instructions

1. Write your name and Slayter box number at the top of the first page of your report.
2. Write a balanced equation for the reaction of $CuSO_4$ and Na_2CO_3 based upon the knowledge you gained from this experiment. Your equation should have water as a reactant (both reactants are aqueous after all!).
3. (a) Write a balanced equation for the reaction of basic copper carbonate to give copper oxide (CuO). (b) Write a balanced equation for the reaction of CuO with methane to give Cu. Assume for reactions (a) and (b) that the only carbon containing product formed is CO_2.
4. Do you have any experimental observations to suggest that the reduction you performed occurred in two steps as described in question (2)? If so, state them.
5. Report your percent yield for the synthesis of "$CuCO_3$" (assume product is malachite or azurite based on your % Cu data)
6. Your observed mass % Cu in basic copper carbonate and the class average mass % Cu in basic copper carbonate (with standard deviation and 95% CI)
7. Apply Student's *t*-test to the class data and judge which (if any) of the formulae for (basic) copper carbonate can be ruled out based on the *t*-value and the 95% confidence level.

EVALUATION OF EXPERIMENTAL UNCERTAINTY

A. Repeated Measurements Do Not Lead to Identical Observations

Experimental chemists are continually faced with the fact that repeated measurements do not lead to a set of identical observations. No chemistry student would be surprised to obtain the following data in Table 1:

Table 1. Three consecutive measurements of a Ni sample using a top loading balance.

Trial	Mass of Ni Sample
1	5.018 g
2	5.014 g
3	5.019 g

Why is a different result obtained upon each weighing of the Ni sample?
What **is** the mass of the Ni sample?
How do we talk about and report these experimental results?

B. Random and Systematic Error (why different results are obtained)

These results show that every experimental measurement is subject to a certain amount of *error*. This error can be systematic and/or random in nature.

Systematic error results from a consistent offset in a measurement device or a consistent mistake in procedure. For example, in making the measurement described above, systematic error can be introduced if the balance zero shifts in between measurements. Different results will be obtained if the Ni sample is handled with wet fingers in between subsequent measurements. In theory, systematic error can (and should!) be avoided.

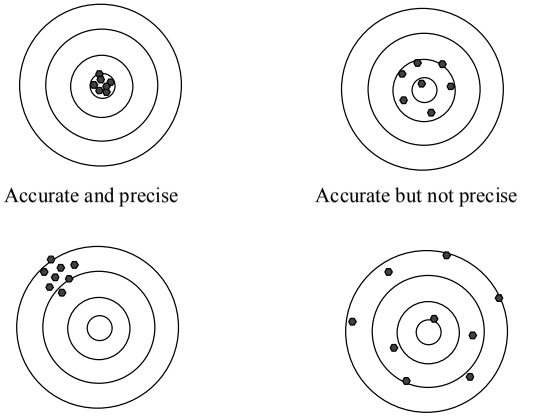
Random error arises from completely random fluctuations in the measurement process. This type of error is *un*avoidable: All measurements, no matter how "good" are subject to some random error. This is why, even with a device such as the electronic analytical balance and even having eliminated all sources of systematic error, we will obtain *different* (albeit only slightly) results upon reweighing the Ni sample. Fortunately this type of random fluctuation is, in general, predictable, and can be described mathematically. Therefore, we can arrive at a single "best" value which represents the mass of the Ni sample. We are *equally* concerned with how "good" that value is and random error theory allows us to determine this as well.

C. Precision and Accuracy (the true mass of the Ni sample)

The amount of random error is expressed as the *precision* of the measurement. *PRECISION* is the quality of agreement among the members of a set of measurements.

It is important to realize that precision is not the same as *accuracy*. We can (and often do!) obtain a precise but inaccurate result. The "closer" the values, the more precise the data. *ACCURACY* is the quality of agreement between the experimental result and the true value.

The common sense meanings of these terms are shown in the following illustration:



D. Reporting Measurements (how we talk about our experimental results)

We use the following concepts to report our experimental results when we have multiple measurements of the same quantity. Each concept will be discussed in detail in the remainder of this Appendix.

The **Average** of the measurements is the best way to simply report your data.

When we want to provide information on the variability of our data, we use the **Standard Deviation** of our measurements. We also calculate the **Confidence Interval** to indicate the variability we expect if we measured the quantity again.

We use the **Q Test** to determine if we have an erroneous result and the **t test** to determine if our measured value if statistically different than the true value.

E. Average

Given that we get different experimental values when we repeat a measurement, as illustrated in Table 1, what is the best way to report our results? In general, the "best" value is simply the *average* of a set of data. For the data in Table 1, the average mass of the Ni sample is

$$\frac{5.018\ g + 5.014\ g + 5.019\ g}{3} = 5.017\ g$$

F. Standard Deviation

An indication of how "good" or reproducible or precise the experimental result is can be obtained by finding the *standard deviation*, *s*, of the data set. The standard deviation is a measure of precision and is typically used to show how closely data fall around the average value of a measurement. The following formula is used to calculate the standard deviation of small sets of data, like those we ordinarily deal with in this laboratory:

$$s = \sqrt{\frac{\sum_i (x_i - \bar{x})^2}{n - 1}}$$

in this equation, x_i is each individual data point, $\bar{x}$ is the average of the data set, Σ is the summation symbol, and n is the number of individual measurements made. For relatively small data sets (10 or fewer measurements), this calculation can be performed using an ordinary calculator. Organizing the calculations in a table can help avoid confusion (and mistakes!):

x_i	$\bar{x}$	$(x_i - \bar{x})$	$(x_i - \bar{x})^2$
5.018 g	5.017 g	0.001 g	1 x 10^{-6} g^2
5.014 g	5.017 g	-0.003 g	9 x 10^{-6} g^2
5.019 g	5.017 g	0.002 g	4 x 10^{-6} g^2
		$\sum_i (x_i - \bar{x})^2$	14. x 10^{-6} g^2

So $s = \sqrt{\frac{14 \times 10^{-6}\ g^2}{2}}$ $s = 2.6$ x 10^{-3}g.

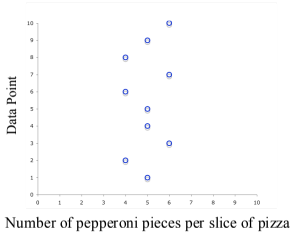
Rounding off gives a final value of 3 x 10^{-3}g.

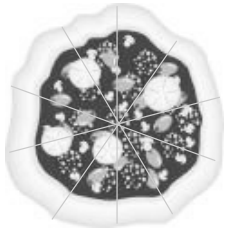
Many calculators are preprogrammed to perform this calculation. Check yours to see if this is the case: It can save you a little number crunching. Most computer spreadsheets can be used to find the standard deviation of a data set as well. We will show you how to use **Excel**, but you are welcome to use any spreadsheet you have available.

Additional illustration of average and standard deviation.

An analysis was carried out to determine the number of pieces of pepperoni on a typical slice of pizza from two different pizza delivery places (A and B). Two pizzas, having ten slices each, were examined and the data are shown below.

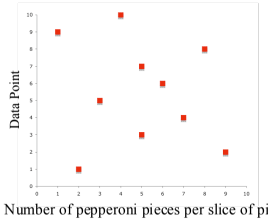
Pieces per slice Pizza A	
5	4
4	6
6	4
5	5
5	6
Average = 5	
Std Dev = 0.8	





All slices have between 0 and 10 pieces of pepperoni

Pieces per slice Pizza B	
2	6
9	5
5	8
7	1
3	4
Average = 5	
Std Dev = 2.5	



Remember in general, the best way to report data containing measurements repeated multiple times is to report the *average*, or mean of a set of data. The average denotes a value that we regard as typical for the measurement. In this analysis, we discover that the average number of pepperoni pieces per slice, five, is the same for both pizzas. Both delivery places would seem like a good place to order from.

Now let's ask a question. If you were to share a pizza with your friends and wanted to ensure that everyone gets their fair share of pepperoni, which pizza would you feel better about ordering, pizza A or B? You would probably choose A, but why? Well, if we look a bit closer at the graphs, we see that the data for pizza A are more closely clustered around the average value. This means the slices in pizza A are more likely to be similar than the slices in pizza B. The distribution of pepperoni on the graphs above illustrates a term we call the standard deviation.

The *standard deviation* of a measurement indicates the reproducibility of a result.

It is easy to see that the standard deviation for pizza A is much smaller than that for pizza B. The smaller the deviation of points about the average value, the more certain we tend to be about the value of our measurement. For pizza B, the deviation is large (compared to A) which leads to a degree of *uncertainty* about the pizza. If you share pizza B, you may or may not get an equal share of pepperoni.

Example: Using the data for pizza B

Average: $\dfrac{2+9+5+7+3+6+5+8+1+4}{10} = 5$

Standard Deviation: $s = \sqrt{\dfrac{\sum_i (x_i - \bar{x})^2}{n-1}}$

x_i	$\bar{x}$	$(x_i - \bar{x})$	$(x_i - \bar{x})^2$
2	5	-3	9
9	5	4	16
5	5	0	0
…	…	…	…
4	5	-1	1
		$\sum_i (x_i - \bar{x})^2$	

$$s = \sqrt{\frac{(9+16+0+\cdots 1)}{10-1}}$$

$$s = 2.58$$

Review:
The **average** of a measurement provides us the "best" or most typical value.
The **standard deviation** tells us how close multiple measurements fit that average value.

G. Confidence Intervals

While the standard deviation provides a measure of the variability in the individual measurements and thus the precision of the experiment, an even more useful quantity is the *confidence interval.* The confidence interval (C.I.) provides a measure of how much variability we would expect in measuring the average value of the same quantity again. It is related to *s* but is **not** equal to *s*. The mathematical formula is

$$C.I. = \frac{t \cdot s}{\sqrt{n}}$$

In this equation, *s* is the standard deviation, *n* is the number of independent measurements used to calculate the average and t is the appropriate choice from a reference table (see the next page). Be careful that the table you use is written in terms of *n* the number of measurements and not the variable known as degrees of freedom.

So, for the Ni example, t (from the table) = 4.303 and

$$C.I. = \frac{4.303(0.003)}{\sqrt{3}} = 0.00745$$

∞

Significant Figures and the Confidence Interval

To express the number of significant figures in the average correctly, the confidence interval should always be rounded to one significant digit and then average value rounded to agree with the confidence limit. (One exception is when the significant digit in the C.I. is one, then a second digit may be retained and the average rounded accordingly.) For our example the appropriate way to report the experimental weight of a nickel is:

$$5.017 \pm 0.007\ g$$

Note a couple of things about the size of the C.I.

a. As *s* decreases, so does C.I.

b. As *n* increases, t decreases, yielding a smaller C.I. The denominator increases with *n* so C.I. gets smaller due to this too.

A smaller value for the C.I. indicates more precision in the data.

For the nickel data you would express the experimental result as 5.017 ± 0.007 g. This tells us that the *actual* weight of this particular nickel sample has a 95% probability of being in the interval from 5.010 g to 5.024 g. Notice two things: First, there is still a 5% chance that the actual weight of the sample is *outside* this range. This level of uncertainty is considered acceptable for most scientific work. Second, the possibility of *systematic* error has not been accounted for.

Table 2. Values of Student's t at various confidence levels for *n* (number of measurements)

	Confidence Level			
n	t (50%)	t (90%)	t (95%)	t (99%)
2	1.000	6.314	12.706	63.657
3	0.816	2.920	4.303	9.925
4	0.765	2.353	3.182	5.841
5	0.741	2.132	2.776	4.604
6	0.727	2.015	2.571	4.032
7	0.718	1.943	2.447	3.707
8	0.711	1.895	2.365	3.500
9	0.706	1.860	2.306	3.355
10	0.703	1.833	2.262	3.250
11	0.700	1.812	2.228	3.169
12	0.697	1.796	2.201	3.106
∞	0.674	1.645	1.960	2.576

The pizza example also illustrates confidence intervals

Using again the data for our two pizzas, we know that the average number of pepperoni pieces per slice of pizza is 5. But is that the real or *true* value for all of the pepperoni pizzas? Are there really 5-pieces of pepperoni per slice? The average provides the typical or best value that we can determine using our method of measurement. The only way to know the true or exact number of pepperoni per slice would be to measure each and every piece of pepperoni, on each and every slice, for every pizza that is made. Not even college students can eat that much pizza!

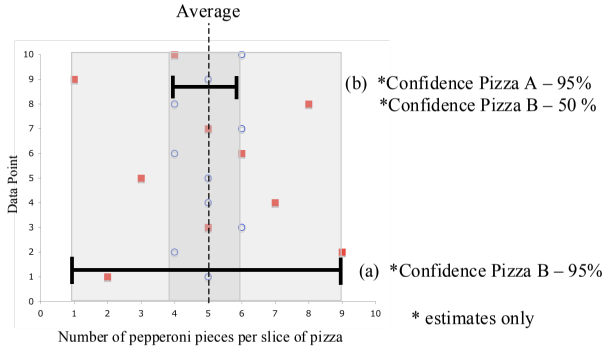
In reality, the best we can do is to provide an estimate based on the measurements we obtain. The trick is, how do we convince others that our estimate, our average value, is representative of all pizzas? We had decided that we were fairly confident that if you ate a slice from pizza A, there was a good chance you were going to have 5 pieces of pepperoni. Why? Because, the standard deviation of the data for pizza A is small and all data fall closely about the average number of 5. On the other hand, our confidence for the same measurement for pizza B is pretty low because of the large deviation. For pizza A you would state that you are confident that the *true value* (absolute real number of pieces per slice for all pizzas) is close to the *average value* of 5.

Confidence Interval: Provides a measure of how much variability we expect in the average value when measuring a quantity multiple times.

While we may be confident in our measurements for pizza A and not so confident for pizza B, how confident are we? How close is our measured average to the true (and unknown) value? Let's combine the graphical data for pizzas A and B and use this for an example.

For pizza B, in units of percent (%), how confident are you that the number of pepperoni pieces per any "measured" slice falls between 1 and 9? (bar (a)) Your estimate might be close to 90 - 95% because the data show that all our slices have a number in this range. (We can never be 100% sure!)

Figure 1
Combined Data
Pizza A ○
Pizza B ■



Another way of asking the above question is how confident are you that the real or *true* number of pieces per slice falls in the range:

5 ± 4 pieces
Average → ← Interval

Again, if your estimate is around 95%, this implies that you are *95% confident that the true value falls within +4 and -4 units of the average value of 5 pieces per slice.*

Compare this with bar (b) on the graph. For pizza B, how certain are you that the true value falls in the range of 4 to 6 (5 ± 1)? Your estimate should now be much lower, 40% maybe 50%? What you should notice is that by decreasing the interval, the certainty that the true value falls in that range of values becomes less. Your confidence in the measurement is smaller.

What if bar (b) was applied to the data for pizza A? For pizza A, how certain are you that the true number of pepperoni pieces falls in the interval between 4 and 6? Your estimate in this instance might be as high as 90 or 95%. Much higher than the estimate for pizza B. (* see Figure 1)

But why is this so?
What information do we have that makes us more confident the true value falls in the range of 5 ± 1, for pizza A, but not so confident it falls in this range for pizza B?
Here is where standard deviation and confidence intervals come into play.

Let's go back to our formula for the confidence interval (C.I.):

$$C.I. = \frac{t \cdot s}{\sqrt{n}}$$ t = t value, s = standard deviation, and n = number of measurements

If we look at the equation we see that the confidence interval is directly proportional to the standard deviation. This implies that *as the standard deviation of a measurement becomes smaller, the confidence interval also becomes smaller*. Using our estimates in Figure 1, we see that we are 95% confident that there will be 5 ± 4 pieces of pepperoni per slice on pizza B. But for pizza A, we are 95% confident that there will be 5 ± 1 pieces of pepperoni per slice. This is so because the deviation of points is so much smaller for pizza A. With a smaller standard deviation, we can report, *with the same confidence*, that the true value will be actually *closer* to the average value for pizza A than for pizza B.

The second important factor in determining confidence interval is the number of individual measurements made (n). It is logical to think that the more times we make a measurement, the better our reported average will be, and the more confident we will be in our measured value. From the equation we observe that the confidence interval is inversely proportional to the number of measurements. *As n increases, the C.I. becomes smaller and the certainty that the true value is close to the average increases.*

The third variable is called the t value. The t value is a constant found in a table of values and helps us define the range of the interval. Notice that the t value depends on the number of measurements made. Let's examine pizza B again, use the t values in Table 2, and *calculate the actual confidence intervals.*

Data for pizza B: $n = 10$ $s = 2.5$ pieces average = 5 pieces

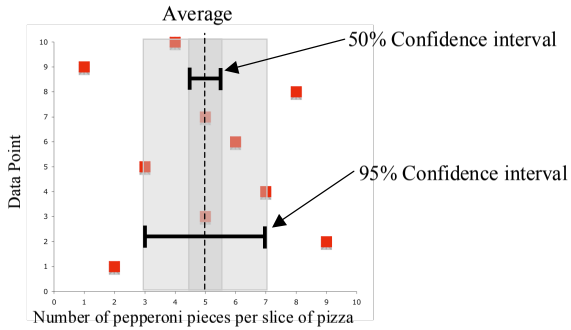
For n = 10 at 50% confidence: $C.I. = \frac{(0.70)\cdot(2.5)}{\sqrt{10}} \approx 0.55$

5.0 ± 0.6 pieces at the 50% C.I.

For n = 10 at 95% confidence: $C.I. = \frac{(2.28)\cdot(2.5)}{\sqrt{10}} \approx 1.8$

5.0 ± 1.8 pieces at the 95% C.I.

Figure 2 Actual Confidence Intervals for Pizza B



What do the above results tell us and do they make sense? The results say that we are 50% confident that the true value falls within ± 0.6 pieces of the average value of 5.0, and 95% confident the true value falls within ± 1.8 pieces of the average. As before, if we have a larger range of values, our certainty (C.I.) becomes larger (hence a larger t value). This is exactly what we should expect.

The calculation follows the same trend as our earlier estimate of the 95% and 50% confidence intervals for pizza B. In our estimate, we were 95% confident that the number of pieces of pepperoni on another slice of pizza from delivery place B would be in the range of 5 ± 4 pieces. However, for the exact same data, our estimate was that we were only 50% confident that the number of pieces of pepperoni would be in the range of 5 ± 1 pieces. For these data the value of *n* and *s* are constant and have not changed, but the range of number of pieces of pepperoni has changed. From the equation we can see that if *s* and *n* are constant, then the t value must be different for different confidence intervals.

H. Presenting Your Experimental Results

You should routinely present your experimental data in terms of the average ± C.I.

EXPERIMENTAL RESULT = AVERAGE ± CONFIDENCE INTERVAL

Remember: The confidence interval should always be rounded to one significant digit (unless the significant digit in the C.I. is one, then keep the second digit as well) and the average value should be rounded to agree with the confidence limit. In other words, the precision of your average value and C.I. must be the same (reported to the same number of decimal places).

For the Ni data in Table 1, you would express the experimental result as 5.017 ± 0.007 g. For the pizza B data, you would express the experimental result as 5.0 ± 1.8 pieces.

I. Using Confidence Intervals

You will most often use confidence intervals to determine if your experimental results agree with the expected or "true" result. Your value for the average will rarely be identically the same as the "true" result. However, we **do** expect the confidence interval to include the "true" result. If this is so we say that the two "agree." Conversely, if the "true" result lies **outside** the confidence interval, you should conclude that the experimental and "true" results are significantly **different**. In the context of our laboratory, this usually indicates a **systematic** error in measurement device or procedure. In the larger context of scientific experimentation, it can mean that your hypothesis needs some tinkering or even a major overhaul.

J. The Single Experimental Result

There is an intuitive notion, supported by statistical theory, that the quality of an experimental result will improve as the number of experimental trials is increased. Unfortunately, it is not always practical to make repeat observations. A measurement may take too long to be repeated, or the observed parameter may be changing with time. Consequently, many Chem 122 laboratory computations will be based on single trial observations.

The single observed value is clearly the "best" value for a single trial observation (The result should be expressed as this value ± estimated error). The **estimated** error must be based on the experimentalist's subjective "guesstimation" of the uncertainty in the measurement. The estimated error can be no smaller than the **least count** of the measuring device (1/10 the smallest readable scale division), but it may be larger if there is a fluctuation in the observed value or if the measuring device is difficult to read. It should be obvious that *no statistical inference can be drawn from a single trial experimental result or from any computation based on that result.*

K. The Q Test for Rejecting a Suspect Observation

Suppose we attempted to improve the Table 1 result by adding a fourth measurement, obtaining values of 5.018, 5.014, 5.019 and 5.045 g for the mass of our nickel. Our intuition leads us to suspect the 5.045 g value. Perhaps the balance zero shifted. Perhaps a reading error was made. Perhaps the nickel was handled with wet fingers.

Clear evidence of a non-random error is sufficient cause to eliminate a suspect value. Since the other values are so close to one another, it is tempting simply to throw out the 5.045 g result. However, the high value could be the result of an improbable, but not impossible, combination of random errors. If this is the case, the measurement is valid and should be incorporated in the experimental result. A statistical test called the **Q TEST** is often used to establish a criterion for distinguishing between an erroneous result and an improbable combination of random errors.

The Q FACTOR is computed as follows:

$$Q = \frac{(suspect\ value) - (nearest\ neighbor)}{(high\ value) - (low\ value)} = \frac{gap}{range}$$

The **gap** is the difference between the suspect value and the nearest value. The **range** is the difference between the highest and lowest values <u>including</u> the suspect result. In our four trial experiment,

$$Q = \frac{5.045 - 5.019}{5.045 - 5.014} = 0.84$$

The computed Q factor is now compared with the 90% rejection quotient in Table 3. We conclude that the 5.045 g result is not valid, since its range quotient of 0.84 is GREATER than the 0.76 rejection quotient for a four trial experiment. Since we are dealing with a 90% rejection quotient, we say that there is a 90% probability that the 5.045 g trial would <u>not</u> result from any combination of **random** errors. Therefore, we can conclude that a **systematic** error was inadvertently introduced and we remove the 5.045 g value from the data set <u>before</u> the average is computed.

THE Q TEST MAY BE APPLIED ONLY ONCE TO A GIVEN SET OF DATA

Table 3. Values of Q for 90% Rejection Quotient

n	Q (Rejection Quotient, 90%)
2	----
3	0.94
4	0.76
5	0.64
6	0.56
7	0.51
8	0.47
9	0.44
10	0.41
11	0.39
12	0.38
∞	----

L. The t Test

The t test is used to determine whether an experimentally determined value is *statistically different* than the known true value.

Suppose you are given a sample of manganese-containing steel. The label reads 11.00 % Mn. Being an experimental chemist, you choose to determine the % Mn for yourself. You take 5 samples and determine % Mn of each sample. Your results are summarized below:

experiment #	**% Mn**
1	9.95
2	10.17
3	10.22
4	10.48
5	10.31

Intuitively, it sure looks like this bottle is mislabeled. How sure are you? The t test allows you to potentially rule out this bottle as the 11.00 % Mn sample. You'll seek to reject the following hypothesis:

This bottle contains steel with 11.00 % Mn. The difference between this true value and the experimental result is purely due to random error.

First, get the statistical information already described in this appendix. You should find:

Average ($\bar{x}$)	Standard deviation (s)	95% C.I.
10.23	0.194	0.241

According to the rules for significant figures, the result is reported % Mn = 10.2 ± 0.2 %. Now, you test the hypothesis by calculating the t statistic (t_{calc}) and comparing it to the critical value of t (t_{crit}) in Table 2 (n = 5, 95%, t = 2.776). **If $t_{calc} > t_{crit}$ then the hypothesis is rejected.** Step by step:

1. Calculate t:

$$t = \frac{\bar{x} - \mu}{s/\sqrt{n}} = \frac{10.23 - 11.00}{0.194/\sqrt{5}}$$

   where $\bar{x}$ = average % Mn = 10.23

   μ = true value of % Mn = 11.00 (assumed for this test)

   s = standard deviation = 0.194

2. Compare t_{calc} with t_{crit}. Here $t_{calc}(8.87) > t_{crit}(2.776)$.
3. Conclude that the hypothesis can be rejected with at least 95% certainty.