
Experiment #15: Identification and Characterization of Compounds X and Y

In this multi-week lab, each student will carry out an organic synthesis where an unknown compound X is converted into compound Y. A series of subsequent tests and characterizations will then be performed in order to deduce the structural formulas of both unknown compounds. At the end of the process, you will submit a formal laboratory report outlining the experiment, its results, and a discussion of your conclusions along with structural representations of both compounds (use of ChemSketch is optional). A rubric outlining the guidelines and expectations for the formal report is found at the end of the experimental procedure described herein.

Overview:

Part I – In this step, you will boil a white powder (unknown compound X) with hydrochloric acid to produce another white powder (unknown compound Y).

Part II – By conducting a simple flame test, the compound will be tested for the presence of chlorine.

Part III – A melting point test will be performed on compound X and two known substances.

Part IV – The solubility and pH properties of X and Y will be examined.

Part V – Using provided data from combustion analyses, the empirical formula of both compounds will be determined.

Part VI – You will titrate both compounds to determine their gram equivalent weights, or how many grams per mole of molecule are “attached” to each acidic hydrogen.

Part VII – In order to determine the molecular weight, the colligative property of freezing point depression for compounds X and Y will be examined.

Part VIII – Finally, the mass spectra (MS) and Infrared spectra (IR) of both compounds will be examined and analyzed for further structural elucidation.

PART I

1. Weigh and record the mass of a clean dry 100 mL beaker. Add about 3 grams (record the exact mass) of compound X to this beaker. Then add about 5 mL of distilled water to compound X to dissolve it completely (warm up if necessary).

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2. Use the reagent pump to add 7.0 mL of 12 M (conc) HCl to the solution and cover with a small watch glass.
3. Place the beaker inside a 250 mL beaker which is about one-third full of DI water. Heat this water bath to a GENTLE boil for about 5 minutes or until a solid material forms in the smaller beaker. Keep the small watch glass on the inside beaker while boiling.
4. Cool the solution by placing the smaller beaker in a cold water bath.
5. Weigh and record the mass of a piece of filter paper. Pour your newly synthesized compound Y mixture through this filter paper so that the liquid drains into a 250 mL Erlenmeyer flask. Wash any remaining solid into the filter paper by rinsing the beaker with small amounts of cold DI water from a wash bottle. Allow the crystals to drain. Wash the precipitate 3 or 4 times with 5 mL portions of cold distilled water.
6. Transfer the crystals and filter paper to a previously weighed large watch glass. Dry the crystals by using one of the following methods: (1) place the watch glass over an appropriate sized beaker about one-third full of boiling water; or (2) place in a drying oven.
7. Once the sample is dry, weigh and record the mass of the watch glass, filter paper, and crystals. Label the crystals as compound Y and keep them in a stoppered test tube in your locker for further analysis. Do not leave the crystals in an open container in your locker, as this may damage the locking mechanism.

Mass of Compound Y: _____

PART II

1. In the fume hood, take a piece of thick copper wire and heat it in a Bunsen burner flame until its glowing. Note the flame color. Let the wire cool for 20 seconds, and dip the wire into a container with Parlon. Cover the tip in the powder, place the compound in the flame and burn it. Observe the color. Parlon contains chlorine, which burns with a distinctive color in the presence of copper.
2. Follow the same procedure using another wire to test a sample of Chemical X. This should not have any unusual color, only burning with the orange color of a typical hydrocarbon fuelled fire.
3. Follow the same procedure using another wire to test a sample of Chemical Y. Does it contain chlorine?

Parlon Flame Color: _____ Chemical X Flame Color: _____

Chemical Y Flame Color: _____

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PART III

Use the mel-temp apparatus for melting point determination. To determine the melting point, place a small amount of crystals into a thin-walled capillary tube (about 0.7–1.0 cm). Place the tube into the mel-temp apparatus, turn it on, and record the temperature range at which the sample melts. You can heat quickly to start, but when you get near the melting point (10 – 20 °C), lower the rate of heating so you can get a more accurate reading. If you don't know the melting point, you can do one fast run to get an approximate reading, then do a more careful and gradual second run. Please note that you cannot rerun a sample once it has melted.

The melting point of compound Y is close to 300 °C. Do NOT attempt to take its melting point as the electronic thermometers will fail at such a high temperature.

While you are testing compound X, also test 4-aminobenzoic acid and phenacetin to calibrate your results. The Mel-temp will run three samples at the same time. Perform two separate trials for each substance. These two values should be within 3° of each other.

	<u>Trial 1</u>	<u>Trial 2</u>	<u>CRC Handbook</u>
4-Aminobenzoic Acid	_____	_____	_____
Phenacetin	_____	_____	_____
Compound X	_____	_____	_____
Compound Y	<i>(not experimentally measured)</i>		<u>~300 °C</u>

PART IV

Dissolve about 0.2 gram of compounds X and Y separately in a 100 mL beaker containing 40 mL DI water. Use these solutions for all tests in this section.

Compare the solubility of X and Y in water.

X:

Y:

Use pH paper to determine the pH of both solutions: X: _____ and Y: _____

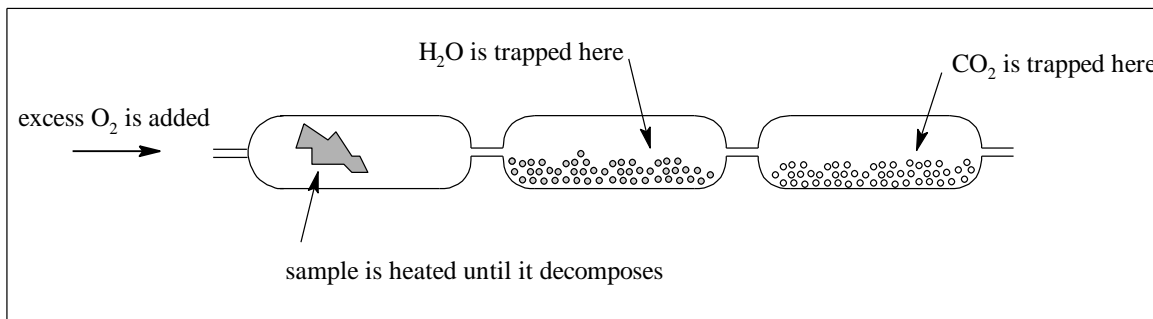
To each solution add a small quantity of Na₂CO₃. What happened? What class of compounds causes sodium carbonate to react this way?

X:

Y:

PART V

The empirical formula of an organic compound can be determined via combustion analysis. A typical combustion apparatus is shown below:



A sample of the chemical is burned in oxygen in a tube packed with copper oxide to ensure complete combustion. The carbon in the compound is converted to CO_2 , which is absorbed in a tube packed with ascarite. The hydrogen is converted to water vapor and is absorbed in a tube packed with drierite. The increase in weight of the absorber tubes corresponds to the CO_2 and H_2O produced during combustion. Compounds X and Y do NOT contain any nitrogen, sulfur, or halogen atoms; both compounds do contain oxygen.

A 1.0542 g sample of X was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	75.2382 g
Weight of ascarite tube after combustion	76.8377 g
Weight of drierite tube before combustion	81.4128 g
Weight of drierite tube after combustion	81.7418 g

Given this data, calculate the empirical formula of compound X: _____

A 1.4745 g sample of Y was burned in a combustion apparatus. The results were:

Weight of ascarite tube before combustion	80.7821 g
Weight of ascarite tube after combustion	83.0196 g
Weight of drierite tube before combustion	78.2988 g
Weight of drierite tube after combustion	78.7560 g

Given this data, calculate the empirical formula of compound Y: _____

PART VI

Before you can titrate your unknowns, you need to first determine the exact molarity of the NaOH solution you are going to use, a process known as standardization. Sodium hydroxide solutions often change concentration if they are exposed to air for long periods, and they are usually checked when first made and after a few days of use.

1. Pour about 150 mL of the approximate 0.2 M NaOH solution into a clean, dry 250 mL beaker which is labeled NaOH. Fill a clean buret with this solution.
2. Into a clean 125 mL Erlenmeyer, place about 0.5 g of KHP acid (KHP = Potassium Hydrogen Phthalate = $\text{KHC}_8\text{H}_4\text{O}_4$; MW of KHP = 204.2 g/mol). Record the exact amount of KHP in the flask.
3. Dissolve the KHP acid in your flask in about 30 mL of DI water. Add about 3 drops of phenolphthalein indicator to the flask.
4. Take the initial buret reading of the level of NaOH, and slowly add NaOH to the flask from the buret. Swirl flask continually. The solution will change from clear to light pink in exactly one drop, so watch closely. If the color disappears with swirling, then you have not reached the endpoint. A dark pink color indicates you have passed the endpoint. When you think you are close to the endpoint, begin to add the NaOH one drop at a time. Record the exact volume of NaOH solution added.
5. Repeat the titration until the molarity is consistent within 0.004 M.

Data for Part VI Standardization:

Recall that at the endpoint, moles OH^- = moles H^+ . Because each mole of KHP contains one acid group, the moles of H^+ = moles KHP.

Mass KHP acid	_____	_____	_____
Moles KHP acid	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Molarity NaOH	_____	_____	_____
Average Molarity (Use values within limit)	_____	_____	_____

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Now knowing that both compounds X and Y are acidic, you can titrate each using your previously standardized NaOH solution. Moreover, if you could determine the molecular weight using another technique (Part VII), you can ultimately determine how much mass of the compound is “attached” to each acidic proton. This is known as the “gram equivalent weight”.

1. To an empty 250 mL Erlenmeyer flask, add about 0.20 grams of X to the flask (record its exact mass).
2. To the flask, add about 30 mL of DI water and 3 drops of phenolphthalein indicator. Titrate the sample with standardized NaOH solution. The endpoint will be a faint pink color that persists for more than 30 seconds.
3. Repeat the titration until you get two consistent results (within 2%), and compare the mass X / mole H^+ values.

Data for Part VI Titration of Compound X

Mass X	_____	_____	_____
Volume of NaOH added	_____	_____	_____
Moles OH^-	_____	_____	_____
Moles H^+	_____	_____	_____
Grams X per mole H^+	_____	_____	_____
Average grams X per mole H^+ (Use values within limit)		_____	
Mole H^+ per mole X (Must know molecular weight from Part VII)		_____	

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Repeat the titration using compound Y. Compound Y titrates slowly, and it may take longer for the pink color to persist as Y will still be dissolving during the titration. You may wish to start the titration before Y has completely dissolved. It should dissolve before you are halfway through adding the NaOH solution.

Data for Part VI Titration of Compound Y

Mass Y _____

Volume of NaOH added _____

Moles OH⁻ _____

Moles H⁺ _____

Grams Y per mole H⁺ _____

Average grams Y per mole H⁺
(Use values within limit) _____

Mole H⁺ per mole Y
(Must know molecular weight from Part VII) _____

PART VII

The freezing point of a solution is lower than that of the pure solvent. The extent of this freezing point depression depends on multiple factors including the concentration of the solution. This is the phenomenon involved when salt is spread on ice to cause it to melt. Raoult found that the depression of the freezing point of a solution is directly proportional to the molal concentration of the solution. That is,

$$\Delta T_f = i K_f m = i K_f \left(\frac{\text{mol}_{\text{solute}}}{\text{kg}_{\text{solvent}}} \right) = \left(\frac{i K_f \text{ grams}_{\text{solute}}}{\text{kg}_{\text{solvent}} \text{ MW}_{\text{solute}}} \right)$$

where ΔT_f = freezing point depression of a solvent, and $i = 1$ (*van't Hoff factor*) for compounds X and Y. K_f is the freezing point constant for the solvent. It is a characteristic only of the solvent and is independent of the solute.

$$K_f = \frac{1.86 \text{ }^\circ\text{C}}{m} = \frac{1.86 \text{ }^\circ\text{C} \cdot \text{kg}_{\text{solvent}}}{\text{mol}_{\text{solute}}}$$

Once you know the freezing point depression (ΔT_f) and the weight of solute and solvent in the solution, you can determine the molecular weight of solute.

1. Calibrate the thermometer. Place one Styrofoam cup inside another. Fill the inner cup with ice and water to fill the cup and cover with a lid. Rinse a thermometer thoroughly with DI water, and insert it into the cup through the lid. Stir the ice water and record the temperature to the nearest 0.01 °C when it becomes constant.
2. Assemble your freezing point apparatus by placing 2 – 3 scoops of rock salt in a 400 mL beaker and add about 150 mL of water. Stir this mixture with a glass stirring rod to saturate the solution and then fill the beaker with ice. Keep the glass stirring rod in this beaker and stir the ice water / salt mixture every few minutes during the experiment.
3. Weigh and record the mass of a clean, dry 200-mm test tube. Add about 1 gram of compound X (record the exact amount). Pour approximately 10 mL of DI water into the test tube, completely dissolve compound X (warm slightly if necessary), reweigh, and record the total mass. Support the test tube in a plastic beaker during these weighings.
4. Clamp the test tube to a ring stand using a utility clamp. Place a clean, dry plastic stirrer into the test tube and lower the test tube into the ice water/salt mixture. Carefully lower the thermometer into the test tube, and make certain the thermometer bulb does not touch the glass. CONSTANTLY move the plastic stirrer in a rapid manner. Continue until you observe the formation of a solid; then gently stir and read and record the freezing temperature of the compound X solution to the nearest 0.01 °C.
5. Remove the test tube and allow the solution to return to room temperature, then repeat the procedure with the same solution once more. If your molecular weight values are not within 10% of each other, do a third trial. Clean and return all equipment when done.

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Data for Part VII

Mass of compound X _____

Mass of H₂O _____Trial 1Trial 2

Freezing temperature of pure water _____ = _____

Freezing temperature of solution _____

 ΔT_f _____

Molecular weight of compound X _____

Average Molecular Weight (values must be within 10%): _____

PART VIII

Chemists generally turn to instrumentation first in most research and industrial settings. In this section, you are going to use two techniques to investigate your compounds.

Mass Spectrometry (MS) is a powerful technique that gives you the molecular weight of most compounds quickly and accurately. It is easy enough to employ that it is used for multiple purposes, for example, to scan luggage for explosive residues in airports. Analysis of the small fragments and exact patterns of the mass spectra is difficult, but finding the molecular weight of the compound is usually trivial. A mass spectrum features various mass-to-charge ratios of charged particles, where unknown compounds are identified by their fragmentation patterns. For this lab, you are looking for the *molecular (or parent) ion* which corresponds to the unknown compound's molecular mass. It is important to note (as you will see) that the molecular ion can be weak with a small height on the actual spectrum; don't assume that the largest peak present in your spectrum necessarily corresponds to the molecular ion peak. Consider the following: does your average molecular weight value from Part VII correlate with any peaks in your mass spectra in Part VIII?

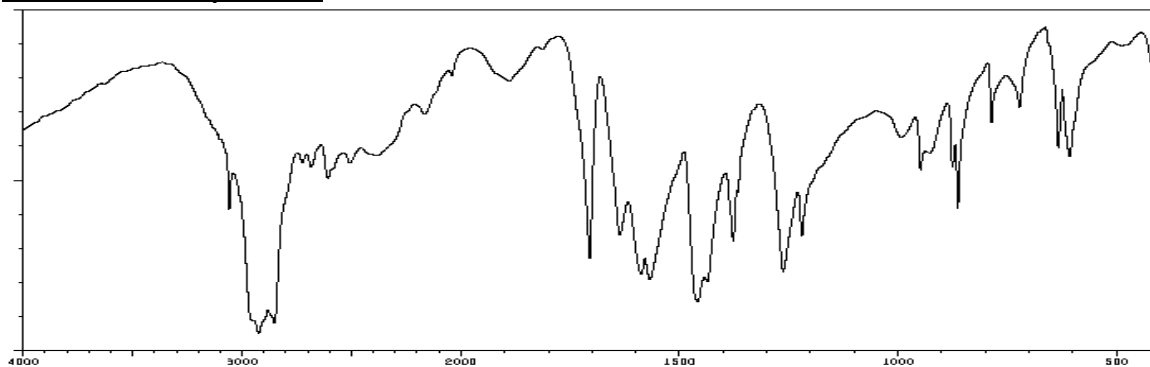
Infrared Spectroscopy (IR), which is more difficult to interpret, gives you information about which type of functional groups are present in a molecule. By comparing peaks to known values, you can determine if your molecule has those types of functional groups.

On the next page are the MS and IR for both compounds. Your instructor will give you guidance about the level of interpretation that is expected. You can find databases of spectra from the SDBS (spectral data base system from Japan's AIST) online to compare these spectra to.

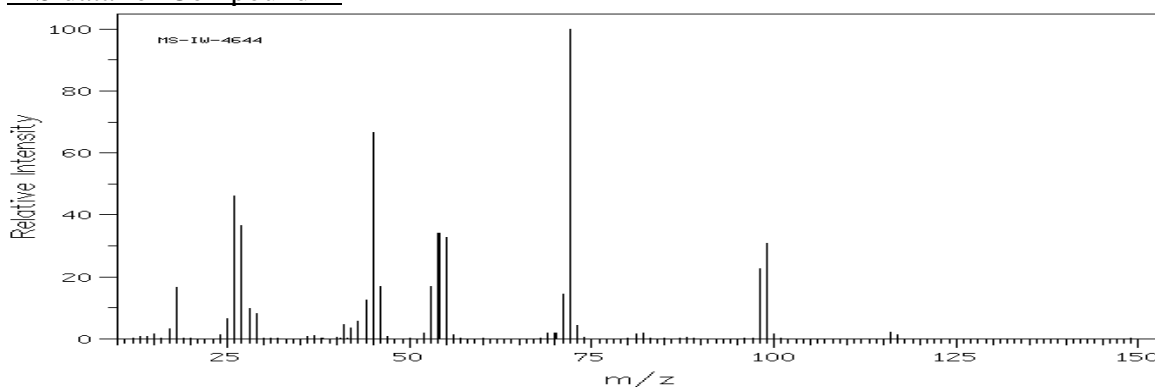
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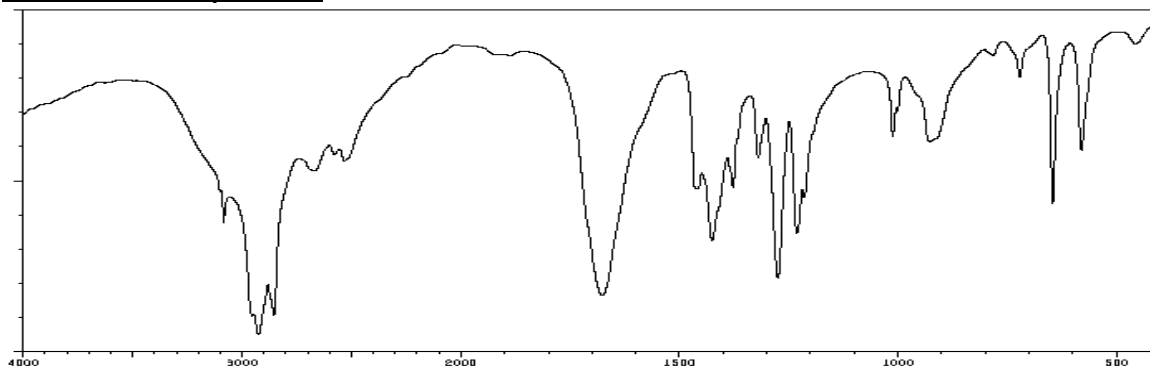
IR data for Compound X



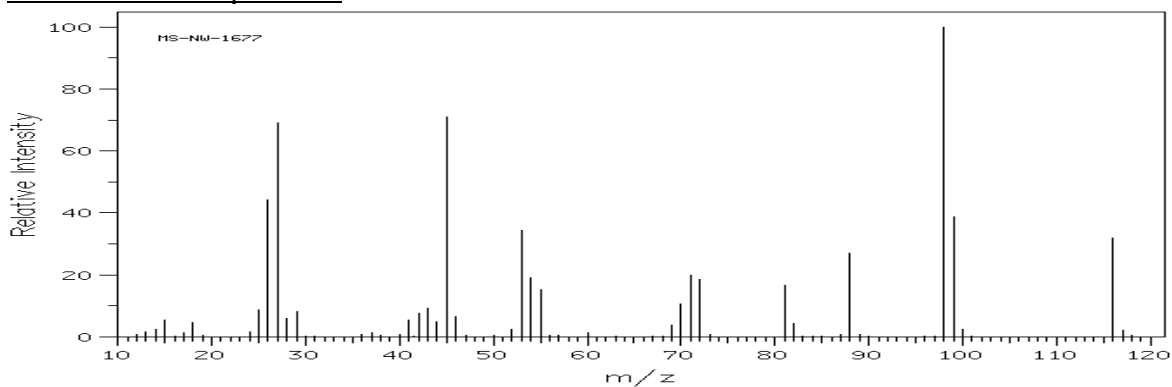
MS data for Compound X



IR data for Compound Y

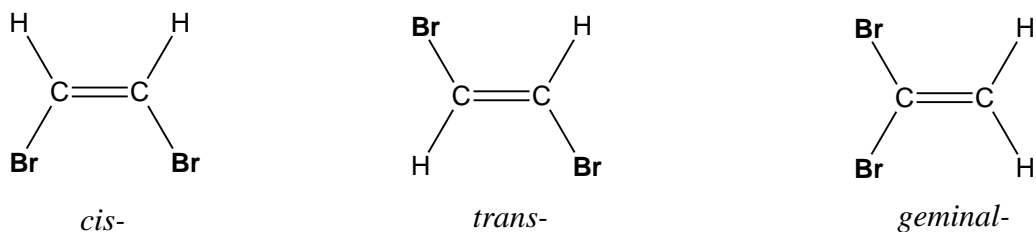


MS data for Compound Y



ANALYSIS:

Are the molecular weights of X and Y the same? Are the empirical formulas of X and Y the same? How can two compounds have the same formula but be different? A clue to this problem can come from looking at similar compounds called geometric isomers, or compounds that have the same chemical formula and basic “layout” but possess different shapes. For example, dibromoethene comes in one of three isomers: *cis*-, *trans*-, and *geminal*- as shown below:



The physical properties of all three isomers are different. Therefore, use your data from the various portions of this experiment to deduce structural representations for both compounds X and Y. Was the compound acidic? How can molecules made up of C, H and O become acidic? Recall that most organic acids have the $-\text{CO}_2\text{H}$ group present. Moreover, as another point of consideration, examine the data acquired in Part VI. The gram equivalent weights of X and Y tell you the weight of atoms (in grams/mole) that are associated with each acidic hydrogen. Using the compound's molecular weight, its empirical formula, and knowing what functional groups are present, you should be able to deduce a few plausible choices.

To determine the EXACT nature of X and Y, you need to consult a reference text (such as the *CRC Handbook* or *Merck Index*) and/or a reputable website such as the Aldrich Chemical site or Wikipedia. Compare the properties and melting points of the various compounds you find, and try to match X and Y to their real identities. Don't forget to write down specific information as to where you obtained any literature information. This should include (but is not limited to) author or editor, title of source, edition where applicable, year, and page numbers.

Your final report should contain the technical and common names along with complete structural representations (including all bonds, respective angles, hybrid orbitals invoked by all central atoms, and total number of σ and π molecular orbitals present) of compounds X and Y. This formal, typed report must adhere to the guidelines (rubric) found on the next two pages. You can neatly draw and label the molecules “by hand”; in addition, you can download ACD/ChemSketch 11.0 Freeware onto a personal computer. This free software is a chemically intelligent drawing tool that works well with Microsoft Word[®] and can be readily downloaded at: <http://www.acdlabs.com/>.

Moorpark College Chemistry Department Laboratory Report Rubric Total: _____

CATEGORY	4 – Accomplished	3 – Good	2 – Developing	1 – Beginning	0 – Substandard	Score
Abstract	Clear, concise (~½ page), and thorough summary of results with appropriate literature references.	Refers to most of the major results; some minor details are missing or not clearly stated.	Misses one or more major aspects of the results.	Missing several major aspects of the results and merely repeats information from the	None, unrelated, or plagiarized.	_____ ×2
Introduction	A cohesive, well-written summary (including relevant reaction chemistry) of the background material pertinent to the experiment with appropriate literature references (at least one scientific reference if required by your instructor) and a statement of purpose.	Introduction is nearly complete but does not provide context for minor points. Contains relevant information but fails to provide background for one part of the experiment, or certain information is not cohesive.	Certain major introductory points are missing (e.g., background, theory, reaction chemistry), or explanations are unclear and confusing. References are not scholarly.	Very little background information is provided, and information is incorrect. No references are provided.	None, unrelated, or plagiarized.	_____ ×2
Methods & Materials	Contains a complete listing of safety information, a narrative of experimental procedures followed, and materials used. Omits information that can be assumed by peers. Includes observations when appropriate and only important experimental details.	Narrative includes most important experimental details. Missing one or more relevant pieces of safety information or experimental procedure.	Narrative is missing several experimental details and safety information or includes insignificant procedural details.	Several important experimental details and safety information are missing. Procedural steps are incorrect, illogical, or occasionally copied directly from the laboratory manual.	None, unrelated, or plagiarized (including completely copied from the laboratory manual).	_____ _____
Results & Calculations	All figures, graphs, and tables are numbered with appropriate titles and captions. Sample calculations are shown and correctly solved. All data is explicitly mentioned in the text.	All figures, graphs, and tables are correctly drawn, but some have minor problems or could still be improved. All data and sample calculations are mentioned in the text.	Most figures, graphs, and tables are included, but some important or required features are missing. Certain data and sample calculations are not explained in the text and/or solved incorrectly.	Figures, graphs, and tables are poorly constructed, have missing titles, captions or numbers. Certain data and sample calculations are not referenced in the text and solved incorrectly.	None, unrelated, or plagiarized.	_____ ×2

CATEGORY	4 – Accomplished	3 – Good	2 – Developing	1 – Beginning	0 – Substandard	Score
Discussion & Conclusion	Demonstrates a logical, coherent working knowledge and understanding of important experimental concepts, forms appropriate conclusions based on interpretations of results and/or spectrum (spectra) analysis, addresses any post-lab questions in paragraph format, includes applications of and improvements in the experiment, refers to the literature when appropriate, and demonstrates accountability by providing	Demonstrates an understanding of the majority of important experimental concepts, forms conclusions based on results and/or spectra (spectrum) analysis but either lacks proper interpretation, does not answer post-lab questions in paragraph format, suggests inappropriate improvements in the experiment, refers to the literature insufficiently, or lacks overall	While some of the results have been correctly interpreted and discussed, partial understanding of results is still evident. Student fails to make one or two connections to underlying theory.	Does not demonstrate an understanding of the important experimental concepts, forms inaccurate conclusions, does not answer post-lab questions in paragraph format, suggests inappropriate improvements in the experiment, refers to the literature insufficiently, and lacks overall	None, unrelated, insignificant error analysis and incorrect explanation, or plagiarized.	_____ ×2
References* (see example)	All sources (information and graphics) are accurately documented in ACS format. At least one reference is taken from primary scientific literature relevant to the report if required by instructor.	All sources are accurately documented, but a few are not in ACS format. Some sources are not accurately documented.	All sources are accurately documented, but many are not in ACS format. Most sources are not directly cited in the text.	All sources are accurately documented but not directly cited in the text.	Sources are not documented nor directly cited in the text.	_____ ×2
Miscellaneous (check all that apply) <input type="checkbox"/> Mechanics, grammar, & appearance <input type="checkbox"/> Appendix <input type="checkbox"/> Lab Notebook <input type="checkbox"/> VSEPR & Valence Bond drawings	Grammar and spelling are correct. All required components are included, complete, and/or illustrated correctly. Paper is not written in first person. Includes ChemSketch image(s) if required by instructor. See lab manual for more details.	Less than three grammatical and spelling errors are present. Missing one required component or features an improperly labeled molecular representation.	More than three grammatical and spelling errors are present or paper is written in first person. Features multiple errors with labeled molecular representation.	Frequent grammatical and spelling errors, and writing style lacks cohesion and fluidity. Paper is written in first person. Labeled molecule contains multiple errors.	None, unrelated, or plagiarized.	_____ ×2

*Journal citations include author or editor, *title (in italics)* followed by a period, **year (boldface)**, *volume (in italics)*, and page numbers. Example: Schrauzer, G.N.; Windgassen, R.J. *J. Am. Chem. Soc.* **1966**, *99*, 3738–3743. For more examples, see summary of ACS Style Guide (found online)