

## Supporting Information

### **The Synthesis of Ruthenocene- A Methodology Appropriate for the Inorganic Undergraduate Curriculum**

Shane Harrypersad, John P. Canal\*

Department of Chemistry, Simon Fraser University, Burnaby, British Columbia, Canada,  
V5A 1S6

\*Contact Email: [jcanal@sfu.ca](mailto:jcanal@sfu.ca)

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## Material:

The supplies required for this experiment are contained in the standard equipment found in most inorganic chemistry laboratories (glassware, analytical balances, hotplate/stirrers, etc.) Ruthenium(III) chloride was obtained from ChemScene, reagent grade acetone and 95% ethanol from Commercial Alcohols, Ultra High Purity grade nitrogen gas from Praxair and the zinc dust was obtained from Sigma-Aldrich. Cyclopentadiene was freshly prepared by the laboratory technician, through thermally cracking dicyclopentadiene.

## Equipment:

- Round bottom flask (50 mL)
- Inlet tube
- Hot plate/stirrer
- Stir bar
- Pasteur pipette & bulb
- Rotary evaporator
- Petri dish cover/bottom
- Beaker (250 mL or greater)
- Thermometer
- Spatula
- Product vials
- N<sub>2</sub> tank regulator
- Tubing for N<sub>2</sub> gas
- Weight boats

## Chemicals:

- RuCl<sub>3</sub>·xH<sub>2</sub>O (CAS: 14898-67-0)
- Zn dust (CAS: 7440-66-6)
- 95% Ethanol (CAS: 64-17-5)
- Cyclopentadiene (CAS: 542-92-7)
- N<sub>2</sub> (Ultra high purity) gas (CAS: 7727-37-9)
- Acetone (Reagent grade) (CAS: 67-64-1)

## Hazards:

Eye protection, lab coat and gloves should always be worn. All laboratory work should be done in a fume hood. All materials should be handled and disposed of based on the information provided in their safety data sheets. The hazard for each material includes acetone (flammable, irritant)<sup>1</sup>, cyclopentadiene (flammable, irritant, environmental hazard)<sup>2</sup>, ethanol (flammable)<sup>3</sup>, nitrogen (compressed gas)<sup>4</sup>, ruthenium (III) chloride (irritant,

corrosive)<sup>5</sup>, zinc dust (flammable, environmental hazard)<sup>6</sup>. Nitrogen gas should only be used by an individual familiar with the proper use of compressed gas and compressed gas cylinders.<sup>7</sup>

#### References:

1. Compound Summary Acetone. <https://pubchem.ncbi.nlm.nih.gov/compound/180> (accessed Aug 2022).
2. Compound Summary Cyclopentadiene. <https://pubchem.ncbi.nlm.nih.gov/compound/Cyclopentadiene> (accessed Aug 2022).
3. Compound Summary Ethanol. <https://pubchem.ncbi.nlm.nih.gov/compound/702> (accessed Aug 2022).
4. Compound Summary Nitrogen. <https://pubchem.ncbi.nlm.nih.gov/compound/947> (accessed Aug 2022).
5. Compound Summary Ruthenium chloride (RuCl<sub>3</sub>). <https://pubchem.ncbi.nlm.nih.gov/compound/82323> (accessed Aug 2022)
6. Compound Summary Zinc. <https://pubchem.ncbi.nlm.nih.gov/compound/23994> (accessed Aug 2022).
7. How do I work safely with – Compressed gases. [https://www.ccohs.ca/oshanswers/prevention/comp\\_gas.html](https://www.ccohs.ca/oshanswers/prevention/comp_gas.html) (accessed Aug 2022).

Table S1: Cost Analysis: Ferrocene vs. Ruthenocene (July 2022)

Ferrocene:								
Reaction taken from: L. F. Fieser, K. L. W. Williamson, Organic Experiments 7th ed, 1992, D. C. Heath and Company, Toronto, p. 297.								
Compound	amount g/mL	mmoles	Supplier	amount g/mL	cost (CDN)	\$ per 1 g_mL	\$ used (CDN)	website
FeCl <sub>3</sub>	7	55.23	Millipore Sigma	25	\$139.00	\$5.56	\$38.92	<a href="https://www.sigmaaldrich.com/CA/en/product/aldrich/372870">https://www.sigmaaldrich.com/CA/en/product/aldrich/372870</a>
	7			250	\$835.00	\$3.34	\$23.38	
DMSO	25		Millipore Sigma	100	\$144.00	\$1.44	\$36.00	<a href="https://www.sigmaaldrich.com/CA/en/product/sial/276855">https://www.sigmaaldrich.com/CA/en/product/sial/276855</a>
	25			2000	\$631.00	\$0.32	\$7.89	
dimethoxyethane mL	60		Millipore Sigma	100	\$94.80	\$0.95	\$56.88	<a href="https://www.sigmaaldrich.com/CA/en/substance/12dimethoxyethane9012110714">https://www.sigmaaldrich.com/CA/en/substance/12dimethoxyethane9012110714</a>
	60			4000	\$581.00	\$0.15	\$8.72	
6M HCl	90		Millipore Sigma	500	\$99.80	\$0.20	\$17.96	<a href="https://www.sigmaaldrich.com/CA/en/product/sigald/320331">https://www.sigmaaldrich.com/CA/en/product/sigald/320331</a>
	90			2500	\$200.00	\$0.08	\$7.20	
KOH	25		Millipore Sigma	25	\$34.30	\$1.37	\$34.30	<a href="https://www.sigmaaldrich.com/CA/en/product/sigald/221473">https://www.sigmaaldrich.com/CA/en/product/sigald/221473</a>
	25			1000	\$123.00	\$0.12	\$3.08	
				(Cost per student)		Total	\$184.06	(Max)
						Total	\$50.26	(Min)
				Cost per student per 0.964 mmol FeCl <sub>2</sub> (max)			\$3.21	(Max: CDN \$)
				Cost per student per 0.964 mmol FeCl <sub>2</sub> (min)			\$0.87	(Min: CDN \$)

Table S2: Cost Analysis: Ferrocene vs. Ruthenocene (July 2022)

<b>Ruthenocene:</b>									
Compound	amount g/mL	mmoles	Supplier	Amount g/mL	cost (CDN)	\$ per 1 g_mL	\$ used (CDN)	website	
RuCl3	0.2	0.964	Chemscene	5	\$140.81	\$28.16	\$5.63	<a href="https://www.chemscene.com/CS-W020395.html">https://www.chemscene.com/CS-W020395.html</a>	
	0.2			25	\$656.26	\$26.25	\$5.25		
				5 --> 112 us					
				25 --> 522 us					
ethanol	10		millipore	1000	\$184.81	\$0.18	\$1.85	<a href="https://www.sigmaaldrich.com/US/en/product/sial/493511">https://www.sigmaaldrich.com/US/en/product/sial/493511</a>	
	10			4000	\$516.71	\$0.13	\$1.29		
				1L --> 147 US					
				4 L --> 411					
acetone	0.5		millipore	500	52.5	\$0.11	\$0.05	<a href="https://www.sigmaaldrich.com/CA/en/substance/acetone580867641">https://www.sigmaaldrich.com/CA/en/substance/acetone580867641</a>	
	0.5			4000	235	\$0.06	\$0.03		
Zn dust	0.225		millipore	100	70.3	\$0.70	\$0.16	<a href="https://www.sigmaaldrich.com/CA/en/product/aldrich/209988">https://www.sigmaaldrich.com/CA/en/product/aldrich/209988</a>	
	0.225			1000	146	\$0.15	\$0.03		
				(Cost per student)	Total	\$7.69	(Max: CDN \$)		
				per 0.964 mmol RuCl3	Total	\$6.60	(Min: CDN \$)		
<b>Summary</b>	Max \$: Fe:Ru	2.4 times price different per student							
	Min \$: Fe:Ru	7.5 times price difference per student							

## Instructor tips:

1. Zinc dust is required for the reaction to occur. If zinc granules are used instead of zinc dust the reaction does not proceed (or proceeds at a rate too slow to obtain a product within the laboratory period).
2. The stock  $\text{RuCl}_3$  most likely will be “wet”, thus the weight of the material can not be directly used as the number of associated water molecules is not known. The percent yield can instead be calculated by the percent Ru found in the stock sample. This value is provided by the supplier through their analysis of the material and should be provided to the students. When not in use tightly seal the  $\text{RuCl}_3$  container to limit water exposure.
3. The use of the roto-evaporator is a short process. The solvent should be removed in about 5 mins, per students. If a roto-evaporator is not available, the solvent can also be removed in the fume hood by flowing air over the solvent and placing the reaction flask in a room temperature water bath.
4. During sublimation, students need to be careful when removing the Petri dish cover containing the pure ruthenocene. Most ruthenocene will collect as a film on the glass surface but a fair amount will form feather like crystals. These can easily be lost (blown away) if the cover is moved quickly.
5. During sublimation, some ruthenocene may sublime to side of the Petri dish bottom (see photo journal below). This needs to be scraped back into the centre of the Petri dish bottom and the sublimation allowed to continue so the ruthenocene can collect on the Petri dish cover.
6. Students need to gradually increase the temperature of the hotplate until sublimation starts. If students raise the temperature too quickly, such as setting the hotplate on high at the start of the process, the hot plate surface will quickly exceed the sublimation temperature and ruthenocene will sublime out of the sublimation set-up. Sublimation product will initially collect on the cooled petri dish cover before re-subliming out.
7. During the melting point determination some students reported that the product was “disappearing” without melting. The melting point of ruthenocene is 199-200 °C while sublimation slowly occurs at 70 °C, thus some product in the melting point

tube will sublime before reaching the melting point temperature. Even with a degree of sublimation, all students were able to correctly determine the melting point.

8. As ruthenocene is stable, this experiment can be performed in two laboratory periods, with the sublimation and analysis conducted separate from the synthesis.
9. Several errors can affect the student yield. The lowest student yield reported was 20%, which still provided enough material for the analysis (IR and melting point). We asked that our students to run an IR and melting point, while we provide the NMR and MS spectra. With the average yield, students would also have material to run the additional analysis.

Sources of error include:

- a. The N<sub>2</sub> flush of ethanol and maintaining of the N<sub>2</sub> reaction environment is important. Failure to properly flush ethanol with N<sub>2</sub> or careless maintaining the N<sub>2</sub> atmosphere will result in a low yield.
- b. Students heated the sublimation set-up too quickly, and the product sublimed out of the set-up.
- c. The transfer of the crude product from the round bottom flask to the Petri dish bottom is another step that can affect the yield if not done correctly.
- d. Some students did not completely sublime the product or removed the Petri dish cover too quickly and lost product.
- e. Fresh cyclopentadiene should be used. Although it can be stored in the freezer for a month or two it does slowly decompose. The initial group that conducted this experiment used a sample of cyclopentadiene that was a week old with the second group completing the experiment a week later. There was no difference in the report yields from the two groups.



## Photos Journal:



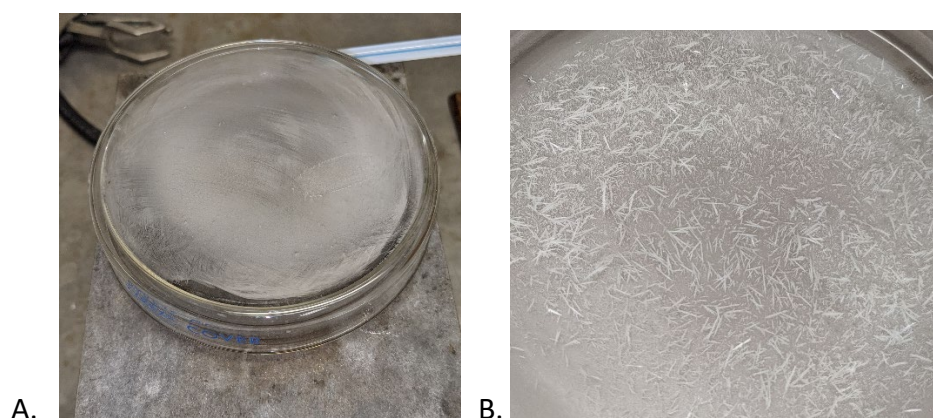
Figure S1: The reaction during the 1.5 hour of stirring under the N<sub>2</sub> atmosphere.



Figure S2: Crude product collected on the Petri dish bottom before sublimation.



Figure S3: Side view of the sublimation process (Ice filled beaker on top of a Petri dish cover, which is on the Petri dish bottom). Notice the crystallization occurring on the side of the Petri dish bottom. These crystals need to be scrapped back into the middle of the dish.



A. B. Figure S4: (A) Top view of the ruthenocene collected on the Petri dish cover. (B) Feather like crystals of ruthenocene collected on the Petri dish cover. (Both photos were taken before the sublimation had finished)

Figure S5: Spectra (Assignment of peaks is provided in the Answer Key below):

NMR Spectrum:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )

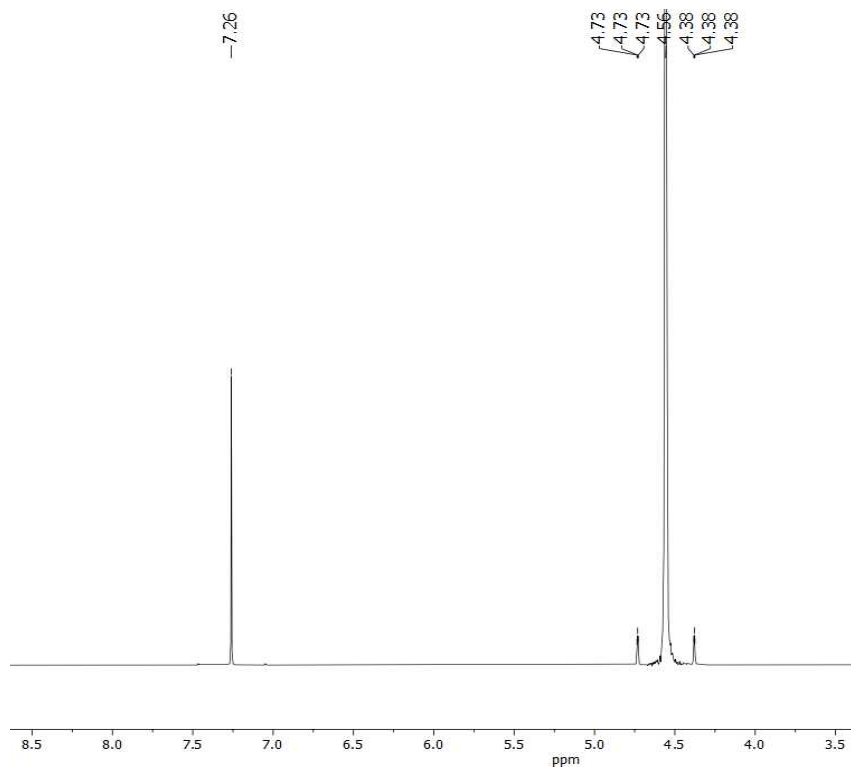


Figure S6: NMR Spectrum:  $^{13}\text{C}\{^1\text{H}\}$  NMR ( $\text{CDCl}_3$ )

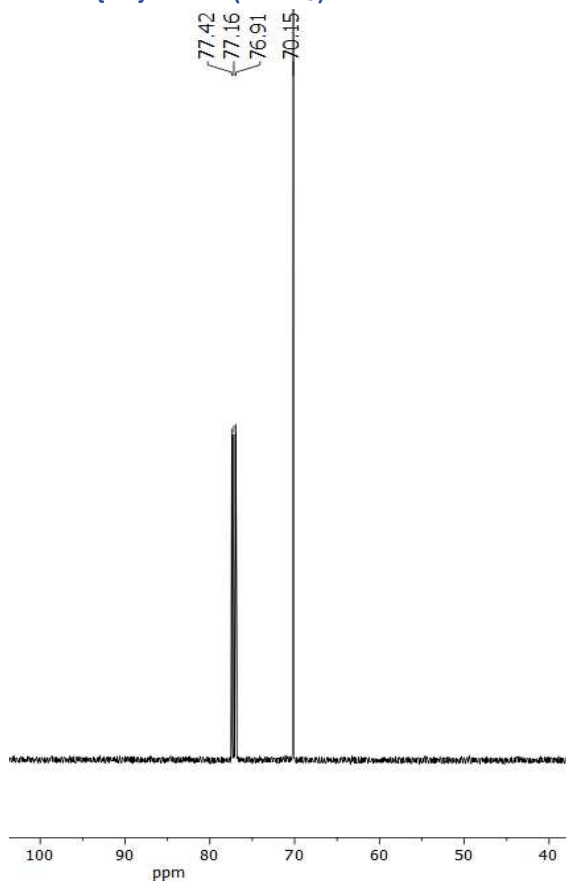
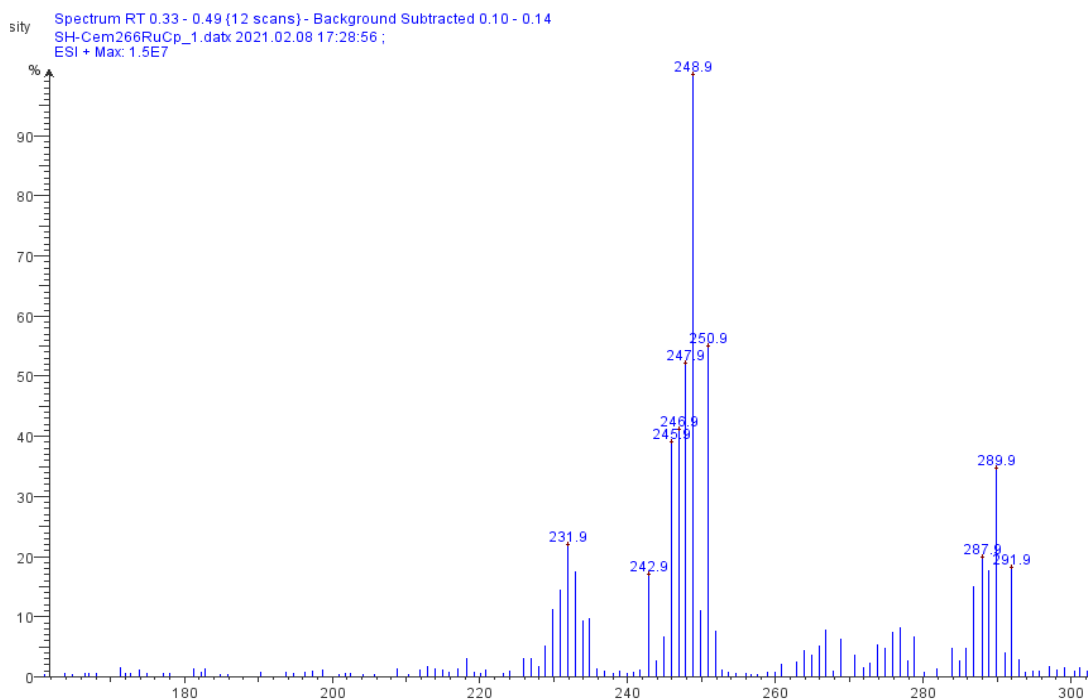
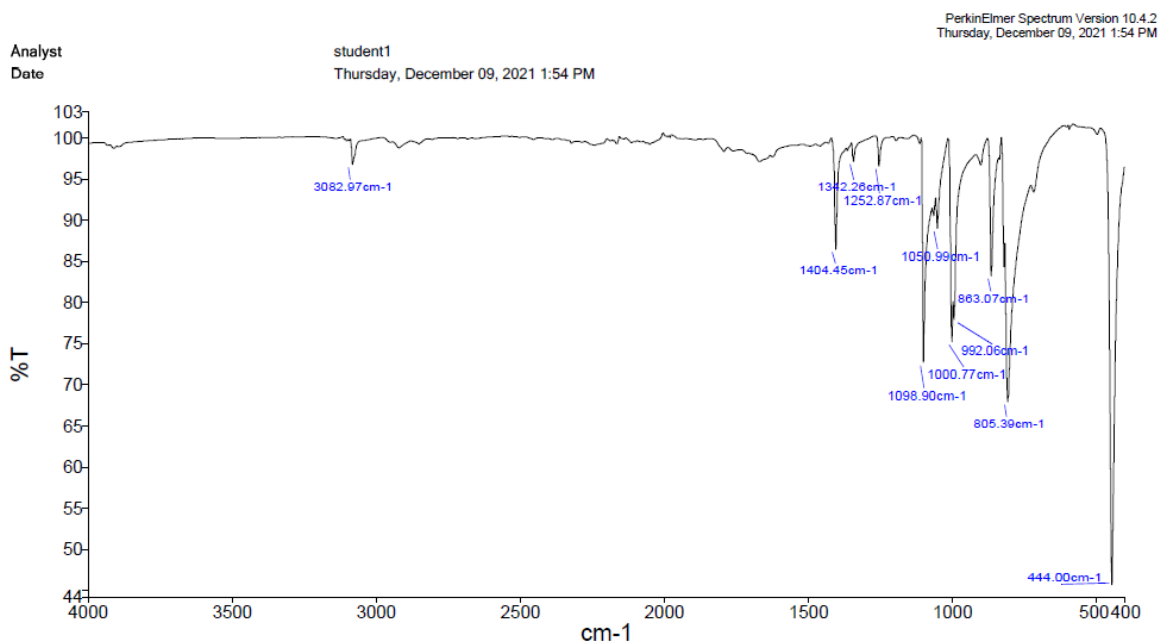


Figure S7: Electrospray Ionization (ESI) Mass Spectrum



The peaks at 231.9 is due to ruthenocene. The instrument uses ammonium acetate and acetonitrile, which often produce adducts, such as the two seen in this spectrum (248.9: ruthenocene-NH<sub>3</sub>; 289.9: ruthenocene-NH<sub>3</sub>-CH<sub>3</sub>CN)

Figure S8: Infrared Spectrum (ATR\*)



\* ATR: Attenuated Total Reflection IR. Solid state.

Figure S9: NMR Spectrum:  $^1\text{H}$  NMR (GaussView5/Gaussian)

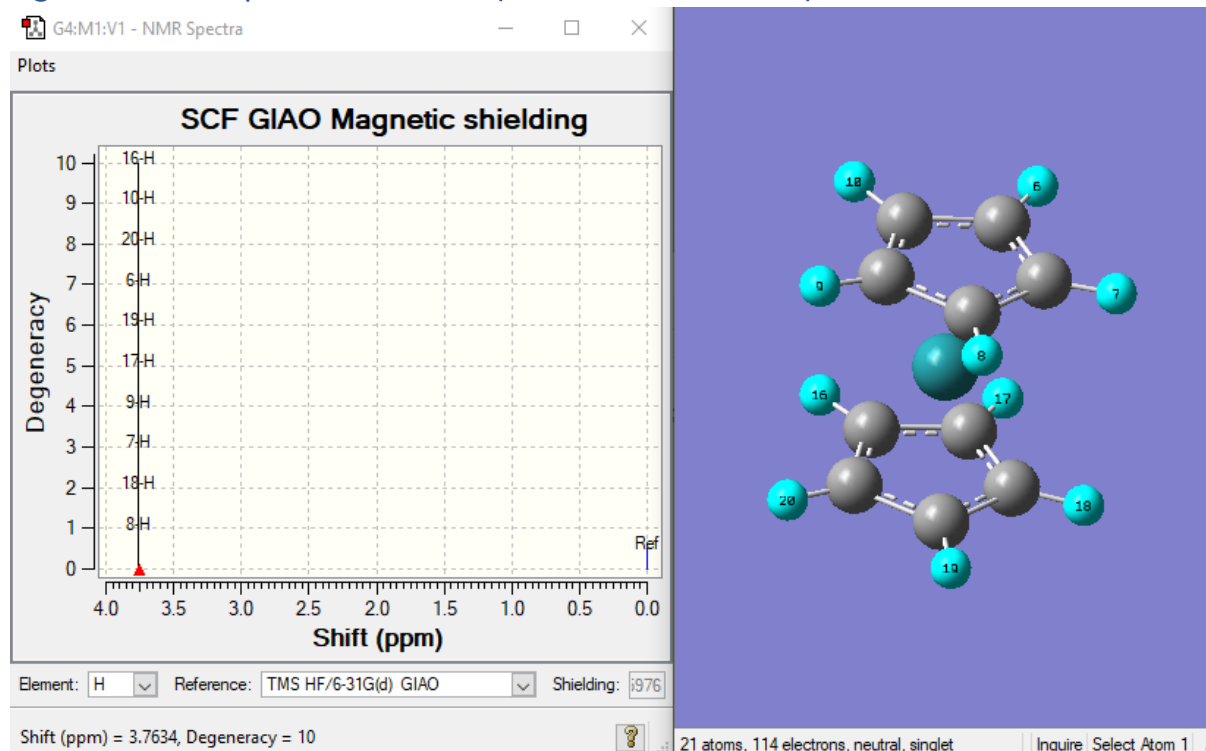
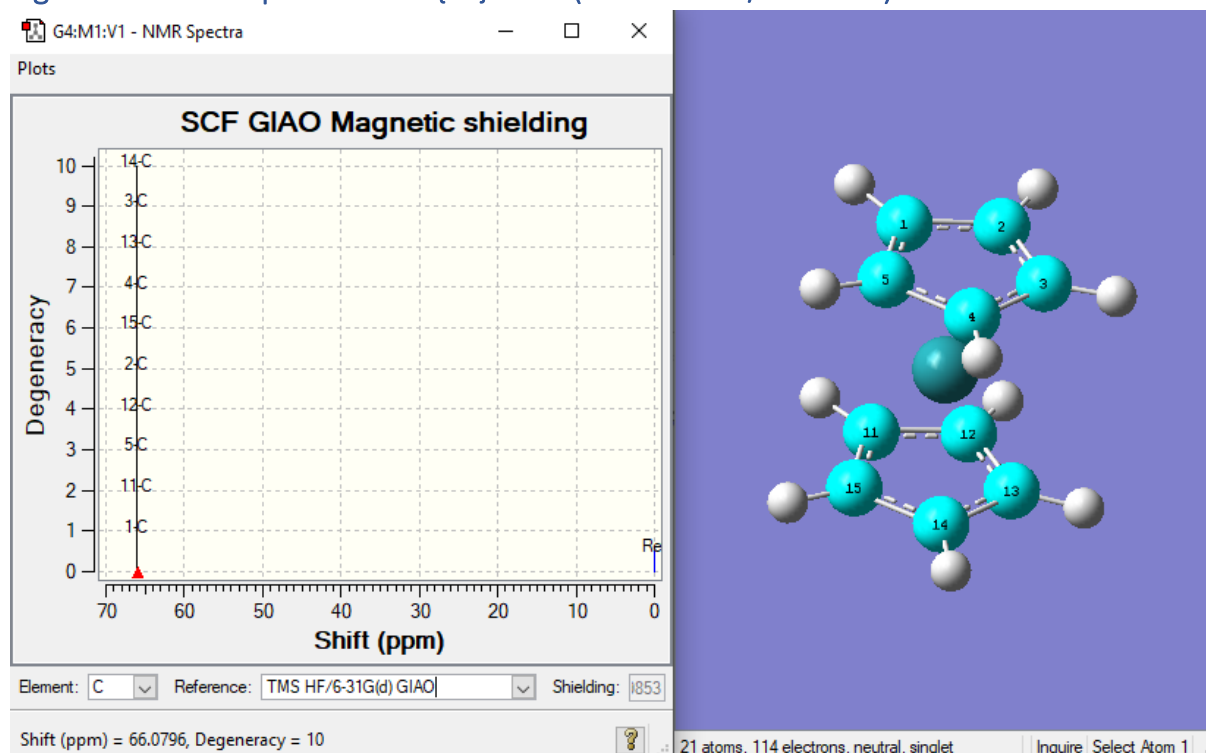
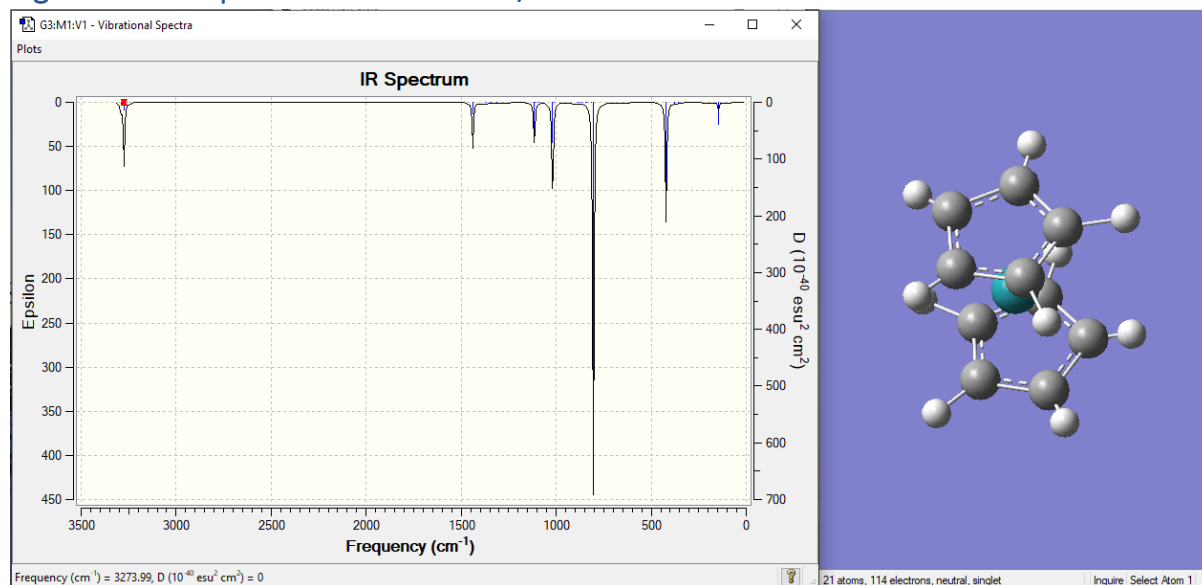


Figure S10: NMR Spectrum:  $^{13}\text{C}\{^1\text{H}\}$  NMR (GaussView5/Gaussian)



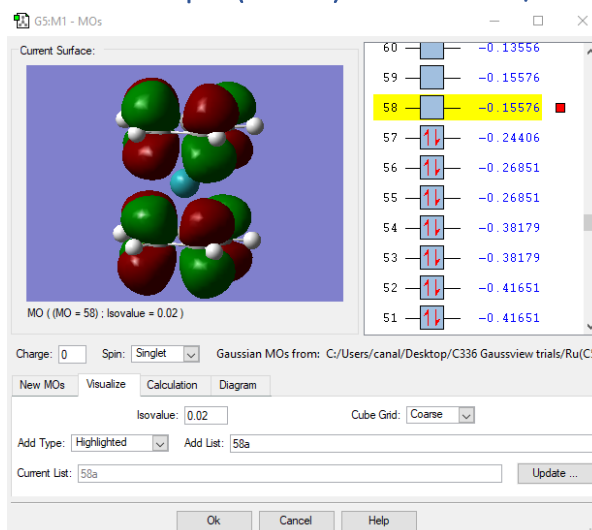
The predict  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectrum based on the Gaussian calculation on ruthenocene, as well as the structure showing the degenerate H and C atoms.

Figure S11: IR Spectrum: GaussView5/Gaussian



The IR spectrum generated by the Gaussian calculation and a snapshot of the animation showing the vibration (i.e., C-H bond vibration) that generates the signal at 3274 cm<sup>-1</sup>.

Figure S12: Molecular Orbital Example (LUMO): GaussView5/Gaussian



## Formal Laboratory Report:

### Format:

The students summarize their experimental finding in a formal laboratory report. Some of the items (questions) that need to be address in the formal laboratory report are provided to the students (see below), as well as a template (see below). Students decide how to organize the material, present the data and the level of explanation.

### Formal Report Template:

Student Name/Station number  
Course information  
Date  
(Times New Roman, 18, Align Text Right)

Title of The Experiment (Times New  
Roman, 26, Center text)

## **Introduction (Times New Roman, 14, bold for title of section, justify text, Line spacing 1.5, Spacing after 6 pt)**

Introduction should contain at least two paragraphs. In the first paragraph you will introduce the topic, and in the second paragraph you will correlate the experiment with the topic. What are the techniques used and what do they tell you about the compound you synthesized? Future tense, references needed (references should be numbered according to the order of appearance, and if used again should have the same number given the first time you used it). (Times New Roman, 12, Justify, Line spacing 1.5, Spacing after 6 pt).

## **Experimental**

What were the steps for the synthesis and characterization of your compound? Information on equipment used, observations and details on the experiment. It should be written as paragraphs, in the past tense, passive voice.

## **Data**

It should contain all the data and calculations obtained. When possible, organize your data in tables.

**Table 1:** Yield and melting point of AB<sub>2</sub> and AB<sub>4</sub> (Tables should have border only Top, bottom for the whole table, and bottom for the first row)

<b>Compound</b>	<b>Yield (%)</b>	<b>Melting point (°C)</b>	<b>Literature melting point (°C)</b>
AB <sub>2</sub>	45	Decomposed	320 <sup>1</sup>
AB <sub>4</sub>	32	142-144	144 <sup>2</sup>

Include chemical reaction, but do not forget to specify what are the phases for your reagents and products.

For calculations, in document elements section of word, select Equation (top right corner) and insert a new equation. Remember to show every calculation and explain the reasoning behind it. Which reagent is the limiting reagent? Show how you got to that conclusion.

$$Yield(\%) = \frac{(\text{experimental mass of product})}{(\text{theoretical mass of product})} \times 100\%$$



## **Discussion**

The discussion is where you use evidence to answer the objective of the lab exercise. Remember to answer all the questions below about the analyses done for the experiment:

- What does the data tell me about my compound?
- What evidence supports my conclusion?
- What evidence rules out other conclusions?
- What are the reaction conditions that led to this outcome?

References are needed in this section as well.

## **Conclusion**

The conclusion should be a concise wrap up of your results and should answer the objective of the laboratory. It should contain specifics: e.g., yield, melting point, structures, etc. – but not introduce new information.

## **References**

References should be in a numbered list in the order that they appear in the write up. Follow ACS style for referencing. The same reference should not receive different numbering.

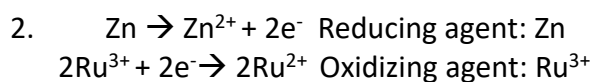
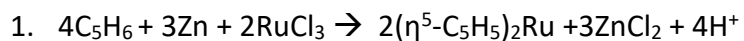
### Formal Report Questions/Items to include:

1. Give the balanced chemical equation for the synthesis of ruthenocene.
2. This is an oxidation-reduction reaction. Write out the half reactions and identify the oxidizing agent and the reducing agent.
3. Draw a structural diagram of ruthenocene.
4. Calculate the percent yield of ruthenocene. Show all steps of your calculation. List the limiting reagent. (Assumption: The sample of  $\text{RuCl}_3$  is dry and contains 37.6% Ru)

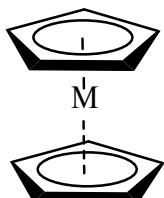
Mass of ruthenocene: \_\_\_\_\_

5. Observed Melting Point: \_\_\_\_\_ Literature Melting Point \_\_\_\_\_
6. LUMO of ruthenocene based on the Gaussian calculations.
7. Ruthenocene is stable in air. Why was a nitrogen atmosphere required for the synthesis? Be specific and use equations to illustrate your answer.
8. Assign the bands observed in the infrared spectrum (Experimental and Gaussian calculate values).
9. Assign the observed peaks in the NMR spectra (Experimental and Gaussian calculate values).
10. In the discussion of the IR spectrum, based on Gaussian calculations results, describe the motion within the complex to give the "large" IR signal at  $\sim 1115 \text{ cm}^{-1}$ .
11. Discuss the results of each of the spectroscopic data (IR, NMR and Mass Spec), indicating how the results support the structure of ruthenocene.
12. Analyse this experiment from the perspective of Green Chemistry.
  - a. Define green chemistry and why it is important.
  - b. Include the 12 Principles of Green Chemistry in a table format, with a 1-2 sentence explanation of each principle.
  - c. Compare this experimental method to the previously published method by Kundig and Monnier (2004) and discuss which one is a "greener" experiment and why by mentioning which of the 12 principles of green chemistry was adopted.

Answers to Formal Report Questions/Items to include:



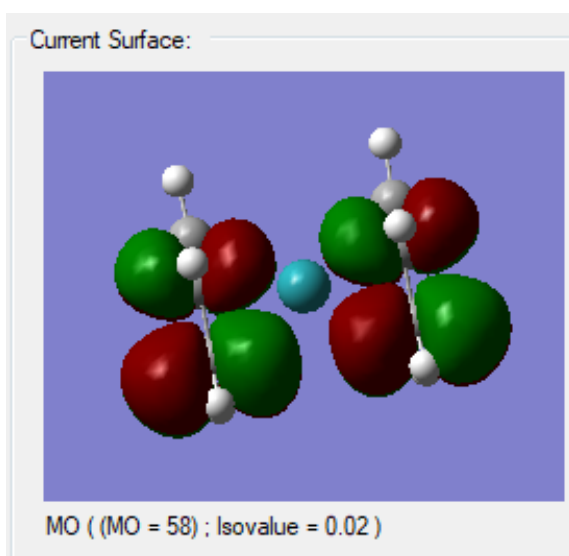
3.



4. Values depends on the amount of starting material.

5. Observed Melting Point: \_\_\_\_\_ Literature Melting Point 195-200 °C

6. LUMO of ruthenocene



7. To prevent oxidation of  $Ru^{2+}$  to  $Ru^{3+}$  and to prevent decomposition of the cyclopentadienide anion ( $C_p$ )

8. Table S3: Assign the bands observed in the infrared spectrum:

Observed Band (list highest to lowest wavelength)	Assignment	Gaussian Calculations Values
3083	$\nu(\text{CH})$	3276.34
1404	$\nu(\text{C}=\text{C})$	1438.67
1098	$\nu(\text{C}=\text{C})$	1114.61
1001	$\delta(\text{CH})$	1018.73
863	$\pi(\text{CH})$	-
805	$\pi(\text{CH})$	804.31
443	Ring tilt	421.62

9. Table S4: Assign the observed peaks in the NMRs (provide a labelled structural diagram).

Peak	Assign	Gaussian Calculations Values
$^{13}\text{C}$		
77.16 (triplet)	$\text{CDCl}_3$	
70.15 (singlet)	Cp C's	66.09
$^1\text{H}$		
7.26 (triplet)	$\text{CDCl}_3$	
4.56 (singlet)	Cp H's	3.76
4.38, 4.73 satellite pks	$^{13}\text{C} \sim 1.01\%$ , $I=1/2$	
	$^{13}\text{C} I=1/2$	

10. The Cp rings expand and contract. The rings do the opposition motion (i.e., one expands while one contracts, and then reverse)

11. IR: IR used to show that Cp is present in the complex based on the IR stretches.

**NMR:** Both  $^1\text{H}$  and  $^{13}\text{C}$  NMR support an ( $\eta^5\text{-C}_5\text{H}_5$ ) bonding arrangement as any other bonding mode would give more NMR peaks. There is only one peak since the ligand is spinning rapidly, too fast for NMR to distinguish the individual atoms.

**Mass Spec:** Confirms two Cp ligands ( $M^+$  peaks matches ( $\eta^5\text{-C}_5\text{H}_5$ )<sub>2</sub>Ru) and shows the Ru isotope pattern.

## 12. Green Chemistry:

- a. Green chemistry is the development of environmentally benign products and processes, that makes use of renewable resources. It is important to reduce our environmental foot point in the field of chemistry.
- b.

1. Prevention:	It is better to prevent waste than to treat or clean up waste after it has been created.
2. Atom Economy:	Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product.
3. Less Hazardous Chemical Syntheses:	Wherever practicable, synthetic methods should be designed to use and generate substances that possess little or no toxic-ity to human health and the environment.
4. Designing Safer Chemicals:	Chemical products should be designed to affect their desired function while minimizing their toxicity.
5. Safer Solvents and Auxiliaries:	The use of auxiliary sub-stances (e.g., solvents, separation agents) should be made unnecessary wherever possible and innocuous when used.
6. Design for Energy Efficiency:	Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.
7. Use of Renewable Feed stocks:	A raw material or feedstock should be renewable rather than depleting when-ever technically and economically practicable.
8. Reduce Derivatives:	Unnecessary derivatization (use of blocking groups, protection/deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.
9. Catalysis:	Catalytic reagents (as selective as possible) are superior to stoichiometric reagents.
10. Design for Degradation:	Chemical products should be designed so that at the end of their function they breakdown into innocuous degradation products and do not persist in the environment.
11. Real-time Analysis for Pollution Prevention:	Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.
12. Inherently Safer Chemistry for Accident Prevention:	Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.
<sup>1</sup> Taken from: <a href="https://pubs.acs.org/doi/pdf/10.1021/ed081p172">https://pubs.acs.org/doi/pdf/10.1021/ed081p172</a>	

- c. The answers to this question varied by students, with students selecting different principles to make their argument. This was expected as the questions were meant for students to think of reactions in terms of green chemistry.

All students felt that the new experiment was an improvement over the published methods (50 students over two semesters). From the list of the 12 Principles of Green Chemistry, the two most common reasons were: (1) Prevention and (5) Safer Solvents and Auxiliaries.

Sample students' comments include:

1. (1) Prevention:
  - a. The old procedure uses large amounts of toluene and ethanol (500 ml and 80 ml), while the new method only uses roughly 10ml ethanol and 5 ml acetone. This is a vast difference in chemical waste and green chemistry heavily favors the new method.
  - b. The old method uses a lot of toluene as a solvent and silica gel to extract the ruthenocene. The new method only uses a little bit of acetone in that step.
  
2. (5) Safer Solvents and Auxiliaries:
  - a. Both procedures use ethanol so that can be ignored, however the old procedure uses large amounts of toluene which is toxic. The new procedure uses acetone which is also toxic but is used in minimal quantities.
  - b. Toluene is more dangerous to use than 95% ethanol and acetone, which we use in our daily lives.

A summary of the frequency of each principle used as the reason why this experiment is "greener" is provided in the Table S6 below.

Table S6: 12 Principles of Green Chemistry	Number of times used
1. Prevention:	47
2. Atom Economy:	24
3. Less Hazardous Chemical Syntheses:	24
4. Designing Safer Chemicals:	10
5. Safer Solvents and Auxiliaries:	42
6. Design for Energy Efficiency:	29
7. Use of Renewable Feed stocks:	14
8. Reduce Derivatives:	10
9. Catalysis:	3
10. Design for Degradation:	7
11. Real-time Analysis for Pollution Prevention:	14
12. Inherently Safer Chemistry for Accident Prevention:	29

Table S7: Grading Rubric:

Writing Criteria	√	X	Marks	Comments
<b>1. Title page</b> <ul style="list-style-type: none"> <li>• Title</li> <li>• Name/Station number</li> <li>• Date</li> <li>• Course information</li> </ul>			<b>2</b>	
<b>2. Introduction (Writing)</b> <ul style="list-style-type: none"> <li>• What is known or has been done</li> <li>• What , why and how it will be done</li> <li>• Clear statement of objective</li> <li>• Paragraph/sentence style with references</li> </ul>			<b>2</b>	
<b>3. Experimental</b> <ul style="list-style-type: none"> <li>• Paragraph / sentence style</li> <li>• Past tense, passive voice</li> <li>• Procedure is reproducible (include observations)</li> </ul>			<b>2</b>	
<b>4. Presentation of Data/Results</b> <ul style="list-style-type: none"> <li>• Tables</li> <li>• Spectra included and properly labeled</li> <li>• Diagrams</li> <li>• Equations</li> <li>• Calculations</li> <li>• Literature values / references</li> </ul>			<b>2</b>	
<b>5. Discussion</b> <ul style="list-style-type: none"> <li>• Clear with logical connections</li> <li>• Grammar, paragraph, and sentence style</li> <li>• Communicates an understanding of the chemistry</li> <li>• Use of relevant references to support discussion</li> </ul>			<b>2</b>	
<b>Subtotal:</b>			<b>10</b>	

*General Comments*

Chemistry Criteria	√	X	Marks	Comments
<b>6. Presentation of Data/Results</b> <ul style="list-style-type: none"> <li>Balanced equation: <ul style="list-style-type: none"> <li>Ruthenocene 2</li> <li>Redox reaction 3</li> </ul> </li> <li>Present yield 3</li> <li>Melting point 2</li> </ul>				
<b>7. Assigning Spectra (IR/NMR)/MO diagram</b> <ul style="list-style-type: none"> <li>IR Tables 6</li> <li>NMR Tables 5</li> <li>MO Diagram 2</li> </ul>				
<b>8. Discussion</b> <ul style="list-style-type: none"> <li>What is the structure of ruthenocene? 2</li> <li>Why rxn. done under N<sub>2</sub>? 2</li> <li>IR motion of peak ~1115 cm<sup>-1</sup>? 2</li> </ul> <p>Interpretation of Data:</p> <ul style="list-style-type: none"> <li>IR 1</li> <li>NMR 2</li> <li>Mass spec. 2</li> </ul>				
<b>9. Green Chemistry Discussion</b> <ul style="list-style-type: none"> <li>Definition and importance 2</li> <li>12 Principles of Green Chemistry 6</li> <li>Experiment evaluation 4</li> </ul>				
<b>10. Conclusion</b> <ul style="list-style-type: none"> <li>Reflects the objective + summarizes results</li> <li>Short and concise 2</li> </ul>				
<b>11. References</b> <ul style="list-style-type: none"> <li>Information referenced completely</li> <li>All references cited</li> </ul>			2	
<b>Subtotal:</b>			50	
<b>Total:</b>			60	



## Student Comments:

Questions: What specific aspects of the Ruthenocene experiment did you like?

Student Comments:

- I liked completing the sublimation portion. I have never completed sublimation in this way, so it was very interesting to see this technique.
- The procedure of the experiment was very interesting since a nitrogen line, rotary evaporator, and sublimation was involved. I have never used any of these techniques before in any other chemistry lab, so it was nice to learn how each of the methods work and to get to do it.
- The experimental part was fun, personally I learned lots of new techniques.
- Sublimation is cool. One of my favourite chemicals to work with is iodine for this exact reason.
- The synthesis was nice, Observing the product. The ideas applying to organometallic chemistry, point groups and effects of structure on properties were interesting
- I had never seen a sandwich structure before this experiment. I also enjoyed the techniques employed in the synthesis portion; it was my first time sublimating a crude product to obtain the final product.
- The Gaussview spectra comparison was interesting but since the real spectrum is much more complicated, it's difficult to decide how much to trust the theoretical assignments. I like that the product was repurposed (further research), rather than thrown out. The Rodovap and sublimation parts were cool; I'd never used a rodovap before, nor done a sublimation like that. The final product was floofy and fun, compared to the mud mess it was sublimed from - cool transformation (but maybe not as fun as the color changing ones).
- I liked the integration of the Gaussian/GaussView calculations into the experiment, it gives a good insight into the interpretation of the IR spectrum.
- The idea of our products being useful and utilized is more fulfilling than having them simply be thrown out at the end of the lab. The metallocenes in general were an interesting topic and were introduced in an engaging way.
- Sublimation part
- Using rotary evaporator and interesting sublimation method
- I think it was my first time working with ruthenium (most experiments use first row transition metals) and also my first-time subliming something.
- Sublimation (it looks like that the black solid product created white gas. then it turned to white crystalline solid)
- The nature of M-L binding was fun to research got very nice crystals bummer they weren't colorful
- Rotary evaporator operation (first seen in CHEM 380, this is the second time to use). Sublimation (totally new compared to other chemistry courses)
- I liked that that I was exposed to some new techniques I never had a chance to use in the lab before such as sublimation and the rotary evaporator. It was also cool to learn more about metallocenes, which is an interesting chemistry topic.
- The gaussview assignment

- It was the first time I had done a sublimation experiment, so I enjoyed learning and experiencing that. The product was also very interesting visually, while it was not colourful, it looked like wispy crystals.
- I particularly enjoyed the method of direct product purification through sublimation. This has never been used in my experience and worked extremely well.
- I very much enjoyed using the sublimation of ruthenocene to isolate it as that was a new technique to me.
- Analyzing NMR and IR
- The sublimation process was new and interesting
- Personally, it was my first lab application of sublimation, and it was mildly interesting to witness.
- Sublimation
- Using GaussView in the report
- I found the sublimation process interesting since it's a new method that I've never done before and it was quite hands-on. I also liked using the rotary evaporator since I've never used that before either.
- The sublimation step because it was a technique I've never done before
- I liked that we were using techniques that were not used in previous labs to synthesize ruthenocene.