CEM 355 US 2018
Grading Rubric

Melting Point Calibration	Points
Experimental	**20**
• 3rd person past tense in paragraph form with passive voice	2
• Description of use of Melting Point Apparatus	18
• Copied from laboratory handout	-5
Results and Discussion	**40**
• Data	
○ Calibration Curve	5
• Answers to questions	
1. Why might the melting point of a compound vary from its known literature value? What causes this change? There may be an impurity	5
2. How might the heating rate effect the observed melting point? Why? Too high of a heating rate may result in a higher observed melting range than the literature value.	10
3. How much sample is ideal for a melting point determination? What might happen if too much sample is used? One crystal or granule (2-3 mm, not packed), a broad melting point range will result from too much sample.	5
4. What is the optimal method for determining melting point using the SMP-30s? Students should include concise directions for operating the SMP-30, including preparation of sample.	10
• Waste Tag	5
Format – Deduct after finishing grading everything else	
• Unlabeled axis on graph	-2
○ Numbers without units	-2
• **Total Points**	**60**

CEM 355 US 2018
Grading Rubric

Distillation	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• Briefly explain the experiment and relevant background	4
• References where appropriate	1
Experimental	**5**
• Mass/volume, moles for all reagents included	5
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Temperature vs. Volume graph – Distillations 1-3	10
• GC data – Distillation 1	5
• GC data – Distillation 2	5
• GC data – Distillation 3	5
• GC data – Distillation 4/5	5
• Discussion of data presented in report	5
• Answers to questions:	
1. Calculate 'n' for each distillation using the Fenske equation. Full calculations do not need to be shown, but these results should be summarized in a data table.	10
2. Which distillation gives the purest product? Why? What data supports this? Higher purity is indicated by larger 'n'. Should be evidenced by Fenske eqn. based on GC data.	10
3. Does the data support or vary from theory? What does this suggest? If data varies from theory, the system may have been heated too quickly leading to too quick of a distillation	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? (Think in terms of experimental steps, i.e., set up, heating parameters, etc.) A reasonable answer should be given.	10
• Waste Tag	5
Conclusion	**5**
• Data-based conclusion	5
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
• References don't use ACS format	-5
Total Points	**100**

CEM 355 US 2018
Grading Rubric

<table>
<tr><th>Cyclohexene</th><th>Points</th></tr>
<tr><td>Pre-Lab
• Turned in at beginning of lab
• Copied from laboratory handout</td><td>5
5
-5</td></tr>
<tr><td>Introduction
• 3rd person past tense in paragraph form with passive voice
• Briefly explain the experiment and relevant background
• References where appropriate</td><td>5
2
3
-1</td></tr>
<tr><td>Experimental
• 3rd person past tense in paragraph form with passive voice
• Mass/volume, moles for all reagents included
• Copied from laboratory handout</td><td>5
2
3
-5</td></tr>
<tr><td>Results and Discussion
• Data: Half Refractive Index/Half Volume
<table>
<tr><th>Refractive Index</th><th>Points</th><th>Volume (mL)</th></tr>
<tr><td>1.4460 – 1.4470</td><td>20</td><td>≥ 11.0</td></tr>
<tr><td>1.4471 – 1.4480 or 1.4450 – 1.4459</td><td>15</td><td>9.5 – 10.9</td></tr>
<tr><td>1.4481 – 1.4490 or 1.4440 – 1.4449</td><td>10</td><td>7.5 – 9.4</td></tr>
<tr><td>≥ 1.4491or ≤ 1.4430</td><td>5</td><td>≤ 7.5</td></tr>
</table>
• Answers to questions
1. What role does xylenes play in this distillation? What kind of distillation is this considered?
Azeotropic Distillation: The higher boiling solvent (xylenes) pushes the lower boiling solvent (cyclohexene) to distill, increasing the product yield.
2. What steps did you take to maximize your yield and purity?
Rate of distillation, observe Temperature vs. Volume graph to stop distillation before the temperature increases signifying distillation of xylenes
3. How can you determine your yield while minimizing loss of product?
Taring flask, measuring the mass of product, and using the density to calculate the volume.
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis.
A reasonable answer should be given.
• Waste Tag</td><td>80
40
5
10
10
10
5</td></tr>
<tr><td>Conclusion
• 3rd person past tense in paragraph form with passive voice
• Data-based conclusion</td><td>5
2
3</td></tr>
<tr><td>Format – Deduct after finishing grading everything else
• Not written in full sentences
• Written in first person
• Written in present tense
• Numbers without units</td><td>
-5
-2
-2
-2</td></tr>
<tr><td>Total Points</td><td>100</td></tr>
</table>

CEM 355 US 2018
Grading Rubric

Thin Layer Chromatography	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**40**
• Data	
○ Correct ID of Component 1 of Unknown	10
○ Correct ID of Component 2 of Unknown	5
• Answers to questions	
1. If your compound does not travel far by TLC in dichloromethane, what solvent should you try next to get good development? In general, a nonpolar developing solvent should be used for nonpolar compounds, and a polar solvent for polar compounds. Dichloromethane is somewhat polar; therefore, a more polar solvent should be chosen (i.e. ether, ethyl acetate, methanol).	5
2. What will happen if your solvent in the solvent chamber is above your origin and spots? Your compound may dissolve in the developing solvent, possibly resulting in no spots and contamination of the developing solvent.	5
3. How might TLC be useful in ways other than identifying an unknown compound when comparing with standards? TLC can be used to follow the course of an organic reaction and column chromatography, as there may be different mixtures present at different time points. It can also be used to identify how many different products are formed in a reaction.	5
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	5
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**60**

CEM 355 US 2018
Grading Rubric

Column Chromatography	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**40**
• Data	
○ Melting Point of Fluorene (114-116 °C), ± 5 degrees	7.5
○ Melting Point of Fluorenone (82-85 °C), ± 5 degrees	7.5
• Answers to questions	
1. What steps can you take as you prepare your column to ensure a good separation of your sample? Choose the proper solvent to ensure a good separation (can be tested by TLC), prepare the column so all layers are level, load the sample carefully, don't let the column run dry or crack.	5
2. When performing column chromatography on a new/unknown mixture of compounds, what should you do first to ensure you can get a good separation? Use concentrated solution of sample, consider the polarity of the compounds undergoing separation and test by TLC.	5
3. How do you determine an appropriate recrystallization solvent? Test small amounts of the compound. If it is a good solvent, the compound will dissolve at high temperatures but precipitate at lower temperatures.	5
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	5
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**60**

CEM 355 US 2018
Grading Rubric

Extraction	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**40**
• Data	
○ Melting Point of Acid (139-142 °C), ± 5 degrees	7.5
○ Melting Point of Ester (78-80 °C), ± 5 degrees	7.5
• Answers to questions	
1. How does the extraction performed in this experiment enable a separation of two organic liquids? Acid-base chemistry is used in this experiment to convert the organic acid into its conjugate base, which is water-soluble, thus allowing for separation of the two compounds.	5
2. How can you determine which layer is the aqueous and which is the organic in an extraction? Comparing the density of the solvents being used can help determine which layer is which. The denser liquid will always be the bottom layer.	5
3. Theoretically, how could you determine how many extractions you need to do to achieve a good separation? Calculate using the partition coefficient.	5
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	5
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**60**

CEM 355 US 2018
Grading Rubric

Recrystallization	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data:	
○ Melting Point of Unknown after recrystallization	
▪ ± 5 degrees	35
▪ ± 10 degrees	-5
▪ ± 20 degrees	-10
• Answers to questions	
1. What steps did you take to determine an appropriate recrystallization solvent or solvent pair? Students should explain that the solubility of small amounts of their purified sample was tested in different solvents to determine in which solvents the compound was soluble when warm but insoluble when cold.	10
2. How do you determine how much solvent is required to recrystallize your sample? If you have more sample, will you need to use more solvent? Use the minimum amount of solvent needed to dissolve the liquid, amount used will depend on the amount of solid you are attempting to recrystallize.	10
3. Once you have obtained crystals, how should you decide which solvent to use to rinse your crystals? It is best to use the mother liquor from the recrystallization, but a solvent that your compound is insoluble in is also a good option.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	10
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**100**

CEM 355 US 2018
Grading Rubric

Triphenylmethanol (Grignard)	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data	
○ Melting Point of Triphenylmethanol (159-163 °C)	35
▪ ± 5 degrees	-5
▪ ± 10 degrees	-10
▪ ± 20 degrees	
• Answers to questions	
1. Why were efforts made to exclude water from this reaction? Draw the mechanism of what would occur if water was present.	10
Water could react with the Grignard reagent.	10
2. Are you convinced that you synthesized the correct product? How does the data support your conclusion? What other analysis could be performed to gain more confidence?	
Students should have completed IR, NMR, and melting point.	10
3. Why is the order of addition important in this reaction?	
The Grignard reagent needs to be prepared before the addition of methylbenzoate. It is prepared first to minimize the number of transfers.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis.	
A reasonable answer should be given.	
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**100**

CEM 355 US 2018
Grading Rubric

Benzopinacolone	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data	
○ Conversion to product as evidenced in IR spectrum	25
○ IR Characterization	10
• Answers to questions	
1. This experiment is analyzed using IR spectroscopy. Discuss the spectra you obtained, identifying all relevant peaks. How do the spectra of the starting material and product differ? Students should discuss that the starting material contains an -OH band at ~3500 cm^{-1}, whereas the product does not contain -OH, but instead exhibits a -C=O band at ~1700 cm^{-1}. Other relevant bands that should be identified are sp2 -C-H (3000 cm-1).	10
2. Draw out and discuss the mechanism of benzopinacolone formation from benzopinacol. See reverse.	10
3. The pinacol rearrangement is well known in organic chemistry. Explain the driving force for this rearrangement. Consider using a reaction coordinate diagram to compare the relative energies of the intermediates you identified above. A tertiary carbocation is more stable than a secondary carbocation.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	10 5
• Waste Tag	
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**100**

CEM 355 US 2018
Grading Rubric

2-Chloro-5-nitrobenzamide (Multi-Step Synthesis)	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data	
○ Melting Point of 2-chloro-5-nitrobenzamide (170-175 °C)	
▪ ± 5 degrees	25
▪ ± 10 degrees	-5
▪ ± 20 degrees	-10
○ NMR Characterization	10
• Answers to questions	
1. The overall yield of a multi-step synthesis is the product of each percent yield multiplied together. Calculate the theoretical yield for each step, and the theoretical percent yield, and compare with your results. See reverse.	10
2. Why does the nitration yield the nitro group in the 5 position on the aromatic ring? What modifications could be made to position it in the 4 position? Electrophilic Aromatic Substitution – different order of addition or starting material would be needed to put the nitro group in the 4 position.	10
3. Incomplete reactions may produce a mixture of products, which are sometimes difficult to isolate/interpret. What observations might suggest that you have a mixture of products? Why is it important to identify this early in your multi-step synthesis? Small peaks in the NMR spectrum, unexpected peaks in the NMR spectrum. If not identified and addressed early, the undesired product(s) can propagate.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	10
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**100**

CEM 355 US 2018
Grading Rubric

Isolation of Caffeine	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**30**
• Data	2
○ IR and NMR Characterization	3
• Answers to questions	
1. How does the caffeine content in a given mass of coffee grounds compare with the same mass of black tea leaves?	5
The coffee should contain more caffeine by mass than tea. Students' data should reflect this.	5
2. Does the brewing method influence caffeine content (i.e. cold brew vs. warm water brew vs. French press)? Explain.	
Yes. Caffeine is poorly soluble in cold water, but more soluble in hot water, therefore warm brewing methods will result in higher caffeine content.	5
3. What other compounds might be present in coffee/tea? How does the extraction procedure you performed address their presence and isolate caffeine from the other compounds? There are over 1000 in coffee and tea (see http://www.compoundchem.com/2015/02/17/coffee-aroma/ and http://www.compoundchem.com/2014/01/30/why-is-coffee-bitter-the-chemistry-of-coffee/), though not all are extracted in the brewing process. This extraction uses dichloromethane and brine to enhance the aqueous layer's ability to extract polar compounds from the organic layer, though too much mixing can cause emulsions and poor separation in this case.	5
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis.	
A reasonable answer should be given.	5
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**50**

CEM 355 US 2018
Grading Rubric

Identification of an Unknown Carbonyl	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data	
○ Correct ID of Unknown	15
○ NMR Characterization	10
○ Derivative Melting Point	10
• Answers to questions	
1. How confident are you in the identification of your unknown compound? Explain the results you obtained from each portion of the experiment. Students should discuss their boiling point, elemental analysis (and calculations of molecular formula), NMR spectra, and melting point of at least one solid derivative.	10
2. Could you have come to the same conclusions using only one or two of these methods (i.e. only NMR and boiling point, only boiling point and elemental analysis)? Perhaps, but more evidence is desired to increase certainty.	10
3. Why is the preparation of solid derivatives useful in the identification of unknown compounds? A solid derivative can provide more data to narrow down several possible unknowns that may be isomers and/or have similar boiling points.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	10
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
• **Total Points**	**100**

CEM 355 US 2018
Grading Rubric

Azo Dye	Points
Pre-Lab	**5**
• Turned in at beginning of lab	5
• Copied from laboratory handout	-5
Introduction	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Briefly explain the experiment and relevant background	3
• References where appropriate	-1
Experimental	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Mass/volume, moles for all reagents included	3
• Copied from laboratory handout	-5
Results and Discussion	**80**
• Data	
○ Quality of crystals	20
○ Residue on paper	15
• Answers to questions	
1. Draw a detailed, arrow-pushing mechanism of the synthesis of the azo dye. See reverse.	10
2. Why is the conjugate base of 2-naphthol required? The conjugate base is needed in order to encourage formation of a resonance form for the reaction to proceed.	10
3. The compound you produced, 1-(4-chlorophenylazo)-2-naphthol, is part of a class of dyes known as azo dyes. Draw the compound and discuss which parts of the molecule contribute to the color you observe. The double bonds and aromaticity of these components contribute to its visible color.	10
4. If you had the opportunity to perform this experiment again, what would you modify to improve your results? Consider the experimental set up, process, and analysis. A reasonable answer should be given.	10
• Waste Tag	5
Conclusion	**5**
• 3rd person past tense in paragraph form with passive voice	2
• Data-based conclusion	3
Format – Deduct after finishing grading everything else	
• Not written in full sentences	-5
• Written in first person	-2
• Written in present tense	-2
• Numbers without units	-2
Total Points	**100**