Queen's Chemistry

TA MANUAL

Chemistry 112

Name: ____________________________

Department of Chemistry
Queen's University
Kingston, Ontario
2013-2014 Edition

Table of Contents

Laboratory Schedule for 2013/2014

Fall Term

Week	Date	Lab	Page
1	Sep 9–13	Lab Safety Training **All students must attend the lab**	
2	Sep 16–20	Experiment 1: Molar Mass of a Volatile Organic Liquid Lab room is CHE 206b; tutorial room is CHE202 or CHE211.	9
3	Sep 23–27	Experiment 2: Gravimetric Analysis	13
4	Sep 30–Oct 4	Experiment 3: Spectroscopic Analysis	15
5	Oct 7–11	Experiment 4: Synthesis of Metal Acetyl-acetonate Complex – Part 1	17
6	Oct 14–18	Experiment 5: Recrystallization of Metal Acetyl-acetonate Complex – Part 2	19
7	Oct 21–25	Experiment 6: Polymerization of Polystyrene as an Introduction to Green Chemistry	21
8	Oct 28–Nov 1	Experiment 7: Photochemical Reaction with Ferrioxalate	25
9	Nov 4–8	Experiment 8: Vapour Pressure and Heat of Vaporization	27
10	Nov 11–15	Experiment 9: Calorimetry – Part 1	29
11	Nov 18–22	Experiment 10: Calorimetry – Part 2	31
12	Nov 25–29	**Make-Up Week for Term 1** **This is the students' <u>one and only chance</u> to make up the lab**. If they cannot do it this week, they will fail the entire course. Exceptions may be made for unavoidable situations such as illness.	

Winter Term

Week	Date	Lab	Page
1	Jan 6–8	Experiment 11: Gas Laws	33
2	Jan 13–15	Experiment 12: Titration of Vitamin C in Orange Juice and Tang	37
3	Jan 20–24	Experiment 13: Corrosion of Metals	39
4	Jan 27–29	Experiment 14: Qualitative Analysis	43
5	Feb 3–7	Experiment 15: Titrimetric Analysis	45
6	Feb 10–14	Experiment 16: Diprotic Acid Titration	47
	Feb 17–21	**Reading Week: No labs or Tutorials**	
7	Feb 24–28	Experiment 17: Buffer Titration	51
8	Mar 3–7	Experiment 18: Rate and Order of a Chemical Reaction	53
9	Mar 10–14	Experiment 19: Rate Law Determination of the Crystal Violet Reaction	55
10	Mar 17–21	Experiment 20: Synthesis of Aspirin – Part 1	57
11	Mar 24–28	Experiment 21: Recrystallization and Analysis of Aspirin – Part 2	59
12	Mar 31–Apr 28	**Make-Up Week for Term 2** Same policies apply as before.	

General Notes

1. ***<u>SIGN</u>!*** Always sign off on students' pre-lab and data/observations before the end of every lab, and on every page.
2. For full marks in any lab report, students must:
   - ☐ answer in complete sentences;
   - ☐ show sample work calculations;
   - ☐ record and propagate errors (uncertainty calculations);
   - ☐ have the correct number of sig figs in the final answer;
   - ☐ have proper units in their final answer;
   - ☐ provide a reference page for cited sources for every lab (at least 3 sources).

Notes on Grading

- Type of mistakes that would appear repeatedly in one lab report such as not doing error propagation, incorrect number of sig figs, etc. should be penalized only once on the lab report, not per question the mistake appears.
  - For these minor mistakes, we recommend having a general system in which 2% is deducted from the overall grade of the report.
- Standard Protocol: Up to **20% can be deducted** from leaving a bench untidy.
  - Cleaning the bench after an experiment is imperative so that the next lab rotation is not delayed.

Safety Quiz

DO NOT COMPLETE THIS AT HOME. To be completed in the lab before you begin your first experiment.

Before you may perform any experiments, you must score 100% on this exam.
Please tear out this sheet and submit it, completed, to your TA.

1. Where are the fire extinguishers located in your lab room? **<u>By the doors (drench showers)</u>**
2. Where are the eyewash fountains located in you lab room? **<u>By the doors (drench showers)</u>**
3. Where is the drench Shower? **<u>By the doors</u>**
4. Match the following terms by writing the letter of the first column items beside the corresponding second-column item.

a) Cancer causing	_**B**_ Teratogen
b) Birth defects	_**C**_ Synergen
c) Hazardous in combination	_**D**_ Sensitizer
d) Allergy development	_**A**_ Carcinogen

CIRCLE THE CORRECT RESPONSE:

5. Material Safety Data Sheets (MSDS) contain information regarding…
   - a) Disposal procedures.
   - b) Health hazards.
   - c) First aid procedures.
   - d) Reactivity hazards.
   - e) All of the above.
   - **f) <u>All of the above and more</u>.**
6. Safety goggles need to be worn... Circle all that apply.
   - **a) <u>whenever you are in an area where chemicals are handled or stored</u>.**
   - b) only when working with strong acids or bases.
   - c) whenever you or your nearby fellow students are working with chemicals.
   - d) only when mixing solutions.
7. When diluting strong acids, always add the water to the acid, never the other way around.
   - a) This statement is true.
   - **b) <u>This statement is false.</u>**
   - c) It doesn't matter which way you do it.
8. Boiling stones should be added…
   - a) just before the solution starts to boil.
   - b) **<u>before applying any heat.</u>**
   - c) after the liquid begins to boil.
   - d) to the reaction mixture before heating
9. Lab coats are mandatory in all chemistry labs.
   - a) **<u>True</u>** b) False c) It's merely strongly suggested that you wear them.

10. Broken glassware should be disposed of…

a) in a wastebasket.
c) at the end of the laboratory period.
b) only if it is completely shattered.
d) **<u>only in containers designed for that purpose.</u>**

11. In the event the building fire alarm sounds, you should…
    a) check your lab manual for the location of the nearest exit from the building.
    b) ask your lab instructor for the location of the nearest exit.
    c) **<u>already know the location of the nearest exit</u>**. (do you know where the exits are?)

12. For information concerning hazards posed by a particular laboratory reagent, you should…
    a) check your lab manual.
    b) check the MSDS.
    c) ask your TA.
    d) **<u>All of the above</u>**. (But use c only after trying a and b first)

13. Before attending a laboratory, you should …
    a) carefully study the material in the laboratory manual and completed the pre-lab exercises.
    b) ask students who did the lab last week if they can think of any time-saving shortcuts.
    c) make special note of any precautions outlined in the laboratory manual.
    d) **<u>both a and c.</u>**

14. If you are uncertain about how to carry out a particular procedure, you should…
    a) try several approaches to see which one works best.
    b) ask your neighbour how to do it.
    c) **<u>seek clarification from your laboratory instructor.</u>**

15. Unused reagents should be put back into their original container. True **<u>False</u>**

16. Minor injuries (scratches, small burns) can be dealt with personally and need not be reported to your TA. True **<u>False</u>**

17. If your clothing is on fire or if you have spilled chemicals on yourself you should run quickly to the drench showers. **<u>True</u>** **<u>False</u>**
    (discuss this)

18. Drink containers are allowed in the lab as long as they are kept closed. True **<u>False</u>**

19. Before leaving the lab wipe your working area clean and dry. **<u>True</u>** False

20. Open sandals, skirts and shorts are not allowed in the lab. **<u>True</u>** False

21. Your knapsack can be kept on the floor by your lab stool. True **<u>False</u>**

Experiment 1. Molar Mass of a Volatile Organic Liquid

Notes for the Procedure

- Ensure students correctly position the snorkel over their hot plates before commencing the lab.
- It is easy to tear the aluminum foil when sealing it over the flasks with rubber bands, they should perform this step carefully.
- The goal is to heat the flasks in the warm-water baths to a gentle boil in order to *slowly* drive the air out of the flasks, leaving only the vaporized organic liquid. Thus, it's important that the students don't boil the baths quickly, they should aim to reach boiling within a 5 min timeframe.
- You will need to provide the students with the atmospheric pressure in the lab.
- Approximate ambient P= 99.5 kPa

Announcement for Students

- **On the DATA SHEET, there should be a correction made to the first column entry under** <u>Mass</u>: *flask+2.5"x2.5" Al+Cu wire.* They are NOT using a copper wire anymore. It should say <u>*Mass:*</u> *flask+2.5"x2.5" Al+**RUBBER BAND**.*

Marking Scheme

Prelab; Should include Introduction, procedure, list of equipment and a diagram of apparatus.	5	5 marks
Prelab Question 1 Prelab Question 2	2	2 marks2 marks
Observations during the experiment; Both Qualitative observations during the course of the procedure, any modifications to the procedure that were actually followed, all data.	4	5 marks
Post Lab Question 1	2	2 marks (propagation of errors not necessary)
Post Lab Question 2	2	2 marks (propagation of errors not necessary)
Post Lab Question 3	2	4 marks (each 'experimental error' should have a sentence listing the effect it might have on thenumerical answer)
Reference page	3	3 (one for each reference) Must be accompanied by a proper reference number in the text of the lab
Cleanup	5	5 marks The student must return their workstation to the state it was before they entered.
Total	25	

Pre-Lab Questions (/)

1. Assumptions:
   - *All air in the flask is displaced by the vaporized compound, thus the volume of the flask is the volume of pure gas at temperature T,* therefore one can use PV=nRT to determine the moles → molar mass of the compound.
   - *Molecular structure of organic compound in liquid phase is the same as its structure in the gas phase (no dissociation/association of molecules),* so that one can be sure that the pressure in the flask is solely exerted by the organic compound at temperature *T* in order to correctly apply PV=nRT.
   - *Mass of air in the flask is identical to what it was before heating the flask* in order to use the *weigh by difference* method to attribute the mass of the remaining liquid to the mass of the compound.
2. **Given:** P=100,000 Pa; T=323 K; *M*=58.08 g/mol ; R= 8.314 m^3 Pa K^{-1} mol^{-1} (Wikipedia)
   Solve – use Equation 20 (manual13): *M*= ρ(RT/P)

   ρ=2160 g/m^3 => **2.16 kg/m^3** (3 sig figs)

Post- Lab Questions

Ensure students include error values ( ) in measured data.

SAMPLE DATA

		Trial 1	Trial 2	Trial 3	
Mass *flask+2.5"x2.5" Al foil+Cu wire*	=	0.09524( )	0.07732( )	0.09656( )	kg
Mass *of above + compound after heating*	=	0.09544( )	0.07744( )	0.09671( )	kg
Mass *condensed vapour*	*m*=	0.0002( )	0.00012	0.00015	kg
Vapour Data					
Volume	*V*=	0.0001585	0.0001455	0.0001600	m^3
Temperature	*T*=	372.65	372.65	372.45	K
Pressure	*P*=	99 500	99 500	99 500	Pa
Density of vapour (at *T* and *P*):	*ρ*=	1.26	0.825	0.938	kg m^{-3}
Molar Mass	*M*=	0.0392	0.0257	0.0292	kg mol^{-1}
		39.2	25.7	29.2	g mol^{-1}
		AVERAGE = 31.37			g mol^{-1}

(Theoretical value for methanol = 32.04 g/mol)

1. Students can calculate the mean and standard deviation on their calculator or on Excel. Check for completion, correctness and uncertainties (error propagated). **Example**:

   Mean = 31.37;
   Standard Dev = 5.72

2. Students should show the other 5 values they are using to re-calculate the mean and standard deviation. Check for completion, correctness and uncertainties.

   Mean = _______ ( )
   Standard Dev = _______ ( )

3. Name 3 sources of error. Answers should be ***reasonable*** and state ***how it would affect their results***. Here are some examples for this lab:

- *Sample was not heated enough to completely vaporize all the organic liquid into gaseous form* – affects accuracy of PV=nRT calculations as the experiment assumes that the volume of the flask is the volume of all the compound in gaseous form at temperature *T.*
- *An inadequate seal over the flask allowed some organic compound to escape during heating* – inaccurate measure of the amount of moles of organic compound and temperature required to occupy the volume of the flask skews PV=nRT calculations.
- *Measured liquid trapped under the foil after heating and cooling* – overestimates the mass of the compound.

Experiment 2. Gravimetric Analysis

Notes for the Procedure

- Ensure the students correctly position their snorkels over their hot plates
- It's most effective for the students to let the powder settle after mixing when it's slightly tilted (on tongs). Look at the video "Techniques for the Gravimetric Analysis Lab" on how this should be done.

Pre-Lab Questions

1. Minimum volume of 1.0 *M* Cu^{2+} needed is **<u>17.9 mL</u>** for Equation 21 and **<u>26.9 mL</u>** for Equation 22. Ensure students show their work.

Equation 21:	$Fe_{(s)}$	+	$Cu^{2+}_{(aq)}$	→	$Fe^{2+}_{(aq)}$	+	$Cu_{(s)}$
m (g)	1.00						
M (g/mol)	55.85						
n (mol)	0.0179		0.0179				
C (mol/L)			1.0				
V (L)			0.0179				
V (mL)			**<u>17.9</u>**				

Equation 22:	**2** $Fe_{(s)}$	+	**3** $Cu^{2+}_{(aq)}$	→	**2** $Fe^{3+}_{(aq)}$	+	**3** $Cu_{(s)}$
m (g)	1.00						
M (g/mol)	55.85						
n (mol)	0.0179		0.0269				
C (mol/L)			1.0				
V (L)			0.0269				
V (mL)			**<u>26.9</u>**				

2. Oxidizing Agent = Copper
   Reducing Agent = Iron
3. Iron is the stronger reducing agent; it donates its electrons to copper.

Post- Lab Questions

1. **<u>Fe^{2+}</u>** was produced. They need to use their data to calculate the mole ratio of *Fe used* and *Cu produced* to see that there is a 1:1 ratio, corresponding to reaction Equation 21. Ensure students are showing their uncertainties ( ).

Trial	**Mass of Fe (g)**	**Mol Fe**	**Mass of Cu (g)**	**Mol Cu**	**Mole Ratio Fe:Cu**
1	1.0027 ( )	0.01713 ( )	1.1598 ( )	0.01825 ( )	**1.0657**
2	1.0031 ( )	0.01713 ( )	1.2001 ( )	0.01888 ( )	**1.1023**

2. **Given:** Use the theoretical value given in Pre-Lab question 1: 1.00 g of Fe used
   n = 0.0179 mol Cu, *M* = 63.55 g/mol
   Solve: m= n · M = 0.0179 · 63.55 = **1.14 g of Cu** (3 sig fig) should be produced.

3. Students should show sample work calculation for percent yield and propagate uncertainty.
   Percent Yield $$\%\text{Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100\%$$

Trial	Measured mass of Cu (g)	Theoretical mass of Cu (g)	Percent Yield (%)
1	1.1598 ( )	1.14	**102 ( )**
2	1.2001 ( )	1.14	**105 ( )**

4. State 2 reasonable sources of error and explain how it will affect their results. Example:
   a. *Loss of sample during transfer* – cannot account for all sample produced in final weigh-in; may underestimate final yield.
   b. *Acetone incompletely evaporated* – adds weight to product; overestimates the yield of copper.

Experiment 3. Spectroscopic Analysis

Notes for the Procedure

- Caution, they will be handling STRONG ACID: 15 M phosphoric acid!!!
- Make sure the student are carefully and properly clamping their volumetric flasks into the boiling water baths

Pre-Lab Questions

1. **Given:** C_1 = 5 M, C_2 = 2 M, V_2 = 300 mL
   Solve: $n_1 = n_2 \rightarrow C_1V_1 = C_2V_2 \rightarrow$ **V_1 = 120 mL** of 5 *M* NaCl needed.
2. **Absorbed:** Every colour but purple (particularly green-yellow)
   Reflected: Purple
3. A = - log *T* → 0.47
4. Possibilities are:
   a. Over-diluting the solution
   b. Mixing won't be as effective in the volumetric flask
   c. Overflowing upon heating the flask.
5. Ensure cited reference(s) for MSDS of $KMnO_4$.
6. **7.28×10^{-4} M $KMnO_4$**. Show unit conversion analysis.

$$\frac{115\ \mu g}{1\ mL} \times \frac{1000\ mL}{1\ L} \times \frac{1\ g}{1\ 000\ 000\ \mu g} \times \frac{1\ mol}{158.034\ g} = 7.28 \times 10^{-4}\ M$$

Post- Lab Questions

1. **6.70 mL (3 sig figs)** of 60.61 µg/mL Mn^{2+} needed. Ensure students show unit conversion analysis, calculation and correct sig figs.

$$\frac{60.61\ \mu g}{1\ mL} \times \frac{1000\ mL}{1\ L} \times \frac{1\ g}{1\ 000\ 000\ \mu g} \times \frac{1\ mol}{54.94\ g} = 1.103 \times 10^{-3}\ M$$

   $n_1 = n_2 \rightarrow C_1V_1 = C_2V_2 \rightarrow V_2 = 6.699 \times 10^{-3}$ L → 6.70 mL of 60.61µg/mL Mn^{2+} needed.
2. A = εbC, but the path length is just 1 cm, so the equation becomes A = bC which is related to Beer's Law which is A = mC where m is the slope. Therefore, the slope of the line is equal to the extinction coefficient.
3. Students need to label and circle the point of maximum absorption on their Ab. vs. wavelength graph.

4. From A= εbC, (b=1), thus A= εC, students should note ε is equal to the slope of their graph, and thus state the value accordingly with proper units (M^{-1} cm^{-1}).
5. Students need to show sample work and uncertainty calculations for the determined concentration of the unknown Mn^{2+} sample.
   Sample work:
   y = mx + b → A = mC + b → A= 833.3C + 0.01545 (from graph).
   A of unknown sample = 0.190 (from experiment);
   sub-into A= 833.3C + 0.01545,
   isolate for C= 2.1×10^{-4} M ( ).
6. Full marks for stating 4 relevant sources of error and providing logical discussion on their effects. Examples:
   a. *Parallax errors* – affects volume readings
   b. *Measurement/calibration error when dealing with equipment*
   c. *Dirty cuvettes* – skew absorbance readings, affecting the graphs drawn and determination of concentration
   d. *Contamination of equipment*

Experiment 4. Synthesis of Metal Acetyl-acetonate Complex – Part 1

Notes for the Procedure

- Make sure all students have their snorkels set up before starting the lab.
- When the students are pipetting, they should take their time to add the acetylacetone solution to their 125 mL Erlenmeyer flask to allow the reaction to occur as desired (2 – 3 min). Ensure students do not take an excessive amount of time for this step, they should be efficient.
- Make sure the students don't boil their solutions.
- If students don't see crystals forming, it may be because the solution was to dilute. Unfortunately, it won't necessarily be easy to make precipitation happen. Students need to avoid adding too much solvent. If crystallization doesn't happen, cool the solution in ice, use the stirring rod to scratch the insides of the flask to induce crystallization. And patience.
- When filtering, instruct students that they don't need to measure out exactly 15 mL of cold distilled water, but just enough to filter their product. This is a step that may cause them to finish the lab late, so they should complete this as quickly as possible.

Marking Scheme

Prelab;	2	Should include Introduction, procedure, list of equipment and a diagram of apparatus. The procedure should be a VERY brief description of the procedure. Reference the lab manual for details. DO NOT write every single step into your lab notebooks please.
Prelab Questions	3	One point for each question
Observations during the experiment	2	This is largely a qualitative experiment. Keep a running track of your activities, observations and measurements directly into your notebooks, not on scrap paper.
Post Lab Question 1	1	Show the calculations, no propagation of errors calculations
Post Lab Question 2	1	Show the calculations, no propagation of errors calculations
Post Lab Question 3	1	The student should give reasonable examples of ways in which the yield is not 100%. These may include things that involve imperfect technique and ways in which even if they execute perfectly, the procedure would make 100% impossible. Each example should be accompanied by a description of how the measured yield will be affected.
Total	10	The rest of the points will be in the part B rubrics.

Pre-Lab Questions

Compound	Molar Mass (g/mol)
ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)	**270.28**

1.

acetylacetone ($CH_3COCH_2COCH_3$)	**100.11**
tris(acetylacetonato)iron(III) ($Fe(CH_3COCHCOCH_3)_3$)	**353.16**

2. State the MSDS health hazards of acetylacetone and show references.
3. Balanced equation:

$$FeCl_3{\cdot}6H_2O + \mathbf{3}\ CH_3COCH_2COCH_3 \longrightarrow Fe(CH_3COCHCOCH_3)_3 + \mathbf{3}\ HCl + \mathbf{6}\ H_2O$$

Post- Lab Questions

1. The limiting reagent is $FeCl_3{\cdot}6H_2O$.

	$FeCl_3{\cdot}6H_2O$ +	**3** $CH_3COCH_2COCH_3$ →	$Fe(CH_3COCHCOCH_3)_3$ +	**3** HCl +	**6** H_2O
m (g)	1.00	1.944	1.31		
M (g/mol)	270.28	100.11	353.16		
n	0.00369	0.0194	0.00369		
ρ (g/cm^3)		0.972			
V (mL)		2			
n_{lim}	**0.00369(lim)**	**0.00647(excess)**			

2. Show sample calculation of percent yield using their data and propagate error.

Percent Yield $\%\text{Yield} = \frac{\text{Actual Crude Yield}}{\text{Theoretical Yield}} \times 100\%$

3. Provide 3 reasonable answers as to why 100% yield was not achieved. Examples:
   a. *Product loss from transfer between apparatus* (flask, probe, weighing vial) which could not be accounted for in the final amount on the watch glass.
   b. *Impurities in the crude product* could add weight to the sample yield.
   c. *Some crystals remain dissolved in the solvent* (did not crystallize out of solution).

Experiment 5. Recrystallization of Metal Acetyl-acetonate Complex – Part 2

Notes for the Procedure

- Make sure all students have their snorkels set up before starting the lab.
- During the step when the students add hot methanol to their product, they only need to add a minimum amount to recrystallize.
- Determining the melting points of their recrystallized/crude product may cause students to run late as it takes time for the Mel-Temp to cool down before it can be used for the following group. If there are students running late, they may get the melting point ranges from a neighbouring group.

Marking Scheme

Prelab;	2	Should include Introduction, procedure, list of equipment and a diagram of apparatus. The procedure should be a VERY brief description of the procedure. Reference the lab manual for details. DO NOT write every step in your lab notebooks.
Prelab Question	1	Very brief explanation please.
Observations during the experiment	4	This is largely a qualitative experiment. Keep a running track of your activities, observations and measurements directly into your notebooks, not on scrap paper.
Post Lab Questions	3	Show the calculations, no propagation of errors calculations
Cleanup (Parts 1 & 2)	5	Put back all glassware, wipe surfaces, dispose of chemicals in proper containers.
Total	15	The rest of the points will be in the part 1 rubrics.

Pre-Lab Questions

1. Methanol was chosen to recrystallize the compound, because $Fe(acac)_3$ is soluble in alcohols but not in water (Lamprey). In order to purify the crude compound, the solute must be able to dissolve in the hot solvent (methanol), so that the impurities are released from the crystal structure. Then upon cooling the solvent, solid crystals slowly reform that exclude the impurities, leaving behind a pure compound of $Fe(acac)_3$.
   Ensure students cite sources where necessary.

Post-Lab Questions

1. Recrystallization was done to remove impurities from the crude product.
2. First, students will generate a ratio of mass of recrystallized product to mass of crude product used (in the recrystallization) to figure out how much of their total crude sample is pure product. This is then used to determine their percentage of the theoretical yield (stoich table for

the reaction) they got. Ensure sample calculations are shown and errors propagated.

Sample Calculation:

Sample Data: 1.21 g ( ) crude product made (in Exp 4);
0.30 g ( ) crude used for recrystallization;
0.10 g ( ) pure recrystallized.

Let *x* be amount of pure product we should yield from our total crude sample, given the recrystallization data.

$$\frac{0.10\ g\ \text{pure}}{0.30\ g\ \text{crude}} = \frac{x}{1.21\ g\ \text{crude}}$$

x = ***0.4 ( ) g*** is pure in total crude sample

	$FeCl_3 \cdot 6H_2O$	+	$3CH_3COCH_2COCH_3$	→	$Fe(CH_3COCHCOCH_3)_3$	+	3HCl	+	$6H_2O$
m (g)	1.00				***1.31***				
M (g/mol)	270.28				353.16				
n (mol)	0.00369…				0.00369…				

$$\%\text{yield of TY} = \frac{0.4\ g}{1.31\ g} \times 100\%$$

%yield = **31% (2 sig figs) (uncertainty)**

3. Students should compare qualitative/quantitative properties of their crude vs. pure product. Each comparison should be supported by a justification as to why they may be different/same.

Experiment 6. Polymerization of Polystyrene as an Introduction to Green Chemistry

Notes for the Procedure

- When the students are shaking their test tubes, makes sure they are conscious of their surroundings and don't shake too close to the lab bench tops, other students, etc.

Marking Scheme

Prelab;	5	• Purpose: 1 mark. One or two sentences describing what the student hopes to achieve. • Introduction: 1 mark. One or two paragraphs describing the background theory and perhaps literature on the subject. May need references here • Equipment and chemicals list 1 mark • Procedure, 1 mark The procedure should be a VERY brief description of the procedure. Reference the lab manual for details. DO NOT write every step in your lab notebooks. • Diagram(s) 1 mark
Prelab Question	3	One mark each question. Keep answers to one or two brief sentences at most.
Observations during the experiment	5	This is largely a qualitative experiment. Keep a running track of your activities, observations and measurements directly into your notebooks, not on scrap paper.
Post Lab Questions	7	• Q1. 1 pt. Refer to the observations to back up your statements here. • Q2. 2 pt. part A. and Part B. you may need references here to back up your statements. • Q3. 1 pt. Reference the MSDS. Where did you find it? • Q4. 2 pt. This one may take some research. References needed • Q5. 1 pt. The "other two methods" refer to question 4. HCl and NaCl. You will have done only the NaCl method but the HCl method is essentially identical save you use HCl instead of NaCl. discuss this very briefly please.
Cleanup	5	Put back all glassware, wipe surfaces, dispose of chemicals in proper containers.
Total	25	

Pre-Lab Questions

1. Resin ID code (recycling symbol) for polystyrene: 

2. List of 5 products made with polystyrene, *e.g.*, packing peanuts, disposable cutlery, water bottles, CD cases, etc…

3. Equilibrium equation of CO_2 in water: $CO_2 + H_2O \rightleftarrows H_2\,CO_3$

Post-Lab Questions

1. Compare 2 qualitative/quantitative aspects of the two samples of polystyrene made. Analysis should be supported by justification for differences/similarities.

2. Chemical reaction of water and CO_2 with surfactant:

$$H_2\,CO_3 + NaC_{18}H_{35}O_2 \rightleftarrows NaHCO_3 + HC_{18}H_{35}O_2$$

where H_2CO_3 on the reactant side is: $H_2O_{(l)}$ + $CO_{2(s)}$ [Pre-Lab Question 3]

   a. As dry ice evaporates, the reduction in CO_2 will create a tendency to drive the direction of the reaction to the left, favouring the formation of reactants.

   b. The surfactant still works in air because in natural conditions it is exposed to little atmospheric CO_2, so that the equilibrium of the reaction lies largely to the left where the surfactant is in its active state.

3. Sodium dodecyl sulfate: oval rate LD50 = 1288 mg/kg (ScienceLab).
   Sodium laurate: N/A
   Open-ended question. Students should state which detergent they would use, then justify their position with sound reasoning. They should cite references where appropriate. Examples:

   a. Sodium laurate, because it does not appear to have toxicities reported and the current information available on the web seem to favour its use as it is derived from coconut oil, which is a reasonably safe material.

   b. Sodium dodecyl sulphate, because it has been a widely used as an ingredient in many household products like shampoo, cosmetics, etc. for years.

   c. Neither, given well thought-out and sound responses.

4. Students should discuss health/environmental/industrial process issues with respect to the use of HCl, NaCl or CO_2. State whether the water treated with HCl/NaCl/CO_2 may be re-used or disposed safely after the production process. If not, what must be done to the water for it to be re-used/disposed of – responses should be tied to issues of energy consumption.

   a. **HCl:** acid is a corrosive substance. It cannot be disposed of without treatment in industrial quantities, because it can severely impair the ecosystem by creating acid lakes, causing acid rain, etc. that make natural environments inhospitable and toxic to wildlife. In order to dispose of acid safely, it must be neutralized by a base, which in itself, produces salt as a by-product, thereby adding to the production costs, energy consumption and environmental impact.

   b. **NaCl:** after salt is filtered out of solution (as mentioned above), the water may be safely used. This process seems to have a smaller carbon footprint than using HCl.

c. **CO_2:** solid CO_2 spontaneously sublimes out of solution into its gaseous form at normal atmospheric pressure, thus it appears to be the greenest production process.

5. Students should make reference to the twelve principles of green chemistry outlined in the introduction to the lab to justify/dispute how the *CO_2 method is greener than the other two methods*. Full marks are awarded for well thought out and supported answers.

Experiment 7. Photochemical Reaction with Ferrioxalate

Notes for the Procedure

- When the students are soaking their pieces of paper in the ferrioxalate solution, they must submerge them one at a time to make sure that each one is properly soaked. Putting them all in at once without individually submerging them will result in lower-quality blueprints.
- Oddly shaped objects will create more interesting blueprints. You may encourage them to try out fun designs.
- The vacuum filtration step should be done quickly. It only takes a few seconds for each piece of paper in the vacuum to be sufficiently dried.

Pre-Lab Questions

1. Given the reaction product is a dark red liquid, it absorbs every colour except dark red.
2. Bromine is a dark red-brown liquid at room temperature (Wikipedia), so it absorbs non-red light.
3. State some MSDS precautions and first-aid spills instructions for ferricyanide and potassium ferrioxalate. References must be provided.

Post-Lab Questions

1. The most amount of reaction occurred in the test tube irradiated under white light for the longest duration of time (20 sec). This is because the dissociation of ferrioxalate into ferrous ions (a reactive species) is a non-spontaneous reaction that requires light. Specifically, *violet-blue light* is absorbed by the ferrioxalate complex, (USYD), which then undergoes photoreduction to generate the ferrous ions. The ferrous ions react with ferricyanide to produce a blue pigment. Thus, an indicator of the reactivity is the intensity of the blue colour in solution, which makes sense that the darkest solution had occurred in the test tube irradiated for 20 sec under white light (containing blue wavelength light) because it was able to produce the most ferrous ions → blue pigment; while the test tubes with no reaction were observed in the tubes exposed to red light (not absorbed by ferrioxalate) or no light.
2. Provide reasonable and justified answers as to why the blueprints are rinsed and what would happen if they were not. *E.g.* Rinse off excess reagent, so that crystals of potassium ferricyanide will not appear on the dried blueprint.
3. Violet-blue colours are absorbed by the ferrioxalate (shown by the graph), which agrees with the observed data where test tubes irradiated under white light saw a colour change in the ferricyanide solution, but not under red light or with no irradiation.

Experiment 8. Vapour Pressure and Heat of Vaporization

Notes for the Procedure

- When releasing the pressure of the vessel, methanol vapor *will* spray out and may be dangerous for students. It may be a good idea to instruct students to position their snorkels and release the pressure into them.
- If students ask regarding the temperature in Prelab Question 2, the sealed flask at 101.325 kPa is at room temperature (22°C).

Pre-Lab Questions

1. **Given**: P_1= 101.324 kPa, T_1=100°C→373.15 K; T_2=25°C→298.15 K
   Solve: $P_1/T_1 = P_2/T_2$ → **P_2= 81 kPa (2 sig figs)**
2. **Given:** P_1=101.325 kPa, T_1=22°C→298.15 K; T_2= 371.4 K
   Solve: $P_1/T_1=P_2/T_2$ → **P_2= 130 kPa (2 sig figs)**

Post-Lab Questions

1. Yes, their graph of Vapour Pressure vs. Temperature (Celsius) should match the expected trend of an increasing non-linear slope, since as temperature increases, so does vapour pressure.
2. Students should show calculation determining the heat of vaporization of methanol with error propagation.
   Sample Calculation*:*
   slope of graph= - 4951 K, R = 8.314 J/mol·K
   slope = $-\Delta H_{vap}/R$ → $\Delta H_{vap} = -m \cdot R$ → ΔH_{vap} = 41.16 kJ/mol ( )
3. Theoretical value for the heat of vaporization of methanol: 38.278 kJ/mol (Wikipedia)

   Students need to use the upper and lower error limits in the regression box on their graph to calculate the upper and lower values for their experimental heat of vaporization for methanol. Full marks are awarded if the theoretical value lies within their limits of uncertainty and the %error calculation is shown with error propagation.

$$\%\,Error = \left(\frac{|Theoretical - Experimental|}{Theoretical}\right) \times \%100$$

4. State 3 sources of error and explain its effect on the results.

SAMPLE DATA

	Initial	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
P_{total} (kPa)		109.89	113.69	116.89	120.97	125.95
P_{air} (kPa)	99.15	99.25	100.36	101.33	102.60	103.71
P_{vap} (kPa)		10.64	13.33	15.56	18.37	22.24
Temp (°C)	22.7	22.85	26.15	29.05	32.85	36.15
Temp (K)	295.7	296.0	299.3	302.2	306.0	309.3

Experiment 9. Calorimetry – Part 1

Notes for the Procedure

- The students should not take an excessive amount of time collecting their sodium polyacrylate from their diapers. There should be more than enough (more than 2 times) the amount of sodium polyacrylate they need, so they do not need to waste their time trying to collect every last crystal.
- Students should clean up all debris from their desks, there may be more at the end of this lab from the cotton and leftover sodium polyacrylate from the diapers.

Pre-Lab Questions

1. State safety hazards of the following three substances, ensure references are cited. Examples include:
    - **ammonium nitrate**: skin/eye/lung irritant, strong oxidizer – may cause fire/explode, combustible at high temperature (ScienceLab)
    - **sodium acetate:** skin/eye irritant, hazardous to inhale, reactive with oxidizing agents/acids (ScienceLab)
    - **sodium polyacrylate**: respiratory irritant (Emerging Technologies Inc)
2. The calorimeter used in the experiment is assumed to mimic an isolated system for the purposes of the calculations made in the lab, but truly isolated systems do not exist in nature, so realistically it is a closed system. Definitions for the following 3 terms:
    - **isolate system**: matter nor energy can enter/exit the physical system (Wikipedia)
    - **closed system**: energy, but not matter can be exchanged between the system and surroundings (Wikipedia)
    - **open system:** matter and energy can flow in/out of the system (Wikipedia)
3. Specific heat capacities:
    - **water**: 4.1855 J g^{-1} K^{-1} (Wikipedia)
    - **Pyrex glass**: 0.75 J g^{-1} K^{-1} (Engineering ToolBox)

Post-Lab Questions

1. Students should show the sample calculations to determine the heat capacity of the two calorimeters (*CalA, CalB*). Ensure proper sig figs are used accordingly.
   Sample Work for Calorimeter A

$$\begin{aligned} C_{CalA} &= C_{H_2o} + C_{Beaker} \\ &= m_{H_2O}C_{H_2o} + m_{Beaker}C_{Pyrex} \\ &= \rho_{H_2O}VC_{H_2o} + m_{Beaker}C_{Pyrex} \end{aligned}$$

$= \left(\frac{1\,g}{1\,mL}\right)(100\,mL)\left(\frac{4.184\,J}{1\,g{\cdot}°C}\right) + (68.70\,g)\left(\frac{0.78\,J}{1\,g{\cdot}°C}\right)$

$\approx$ 470 J·°C^{-1} is the heat capacity of the calorimeter in Part A

Sample Work for Calorimeter B

$C_{CalB} = C_{H_2o} + C_{150mLBeaker} + C_{50mLBeaker}$

$= \rho_{H_2O} V C_{H_2o} + (m_{50mLBeaker} + m_{150mLBeaker})C_{Pyrex}$

$=$ 300 J·°C^{-1} is the heat capacity of the calorimeter in Part B

2. Students should show the sample calculation to determine the enthalpies of the following two reactions. Answers should be in kJ/mol and indicate whether it is exo/endothermic:

   a. Dissociation of ammonium nitrate, NH_4NO_3, in water: ~26 kJ/mol, **endothermic**.

NH_4NO_3 m= 3.00 g *M*= 80.06 g/mol n= m/*M* = *0.0374..mol NH_4NO_3*	$\Delta H^\circ_{rxn} = \frac{\Delta H'_{rxn}}{n} = \frac{-C_{cal}{\cdot}\Delta T}{n}$ $\Delta H^\circ_{rxn} = \frac{-(470\ J)(-2.1°C)}{0.0374..\ mol}$ ΔH°_{rxn} *≈26000 J/mol* → *+ 26 kJ/mol (endothermic)*

   b. Absorption of water by sodium polyacrylate: ~–15 kJ/mol, **exothermic**.

NH_4NO_3 m= 3.00 g *M*= 94.05 g/mol n= m/*M* = *0.0318..mol NH_4NO_3*	$\Delta H^\circ_{rxn} = \frac{\Delta H'_{rxn}}{n} = \frac{-C_{cal}{\cdot}\Delta T}{n}$ $\Delta H^\circ_{rxn} = \frac{-(470\ J)(-1.0°C)}{0.0318..\ mol}$ ΔH°_{rxn} *≈ –15 kJ/mol (exothermic)*

3. Students should provide at least one well-thought out answer as to how cotton as a contaminant may affect the results in their experiment. Example:

   The presence of cotton may absorb heat from the sodium polyacrylate exothermic reaction, thus this heat would not be accounted for by the calorimeter, leading to the enthalpy of reaction to be underestimated, as the observed temperature change would be smaller.

4. Students should provide 2 systematic errors in the experiment and explain how this would affect their results for full marks. Examples:

   a. *The calorimeter is not a perfectly isolated system* – heat is lost into the surroundings that cannot be accounted for by the calorimeter, thus underestimating the temperature changes.

   b. *In the procedure, the calorimeter was opened to add-in/mix materials* – heat loss into surroundings

   c. *The calorimetry calculations do not take into consideration the other materials surrounding the calorimeter that would absorb heat* – tissue paper lining, cardboard lid, air, etc.

Experiment 10. Calorimetry – Part 2

Notes for the Procedure

- You will need to designate which of the two neutralizations each bench does for Part B of the experiment. Suggestion – assign by even/odd lab bench numbers:
  - Even- *Strong base/weak acid* neutralization
  - Odd- *Weak base/strong acid* neutralization

Pre-Lab Questions

1. State the MSDS precautions in case of skin contact for HCl and NaOH– immediately flush skin under water for 15 minutes and remove contaminated clothing,(Sciencelab 1 & Sciencelab 2). Ensure references are cited.
2. **Adiabatic** refers to a chemical process where there is no heat gained/lost by the system, thus there is no heat exchange with the surroundings (Hyperphysics).
3. Since equal volumes were used in the acid-base reactions, the difference in concentration shows that there is a limiting reagent in the neutralization. Given that the concentration of the base is 1.1 *M* and the acid is 1.0 *M,* it is clear that the acid is the limiting reagent (since n=CV, and given that the acid-base react on a 1:1 mole ratio, $n_{base}>n_{acid}$). This is important to know for the stoichiometric calculations, because then it means that determining quantities like the enthalpy of the reaction will be based on the acid (limiting reagent).

Post-Lab Questions

1. Ensure students show sample work, state their answers in kJ/mol and identify whether the reaction is exothermic/endothermic.
   a. Enthalpy of neutralization of strong acid/strong base: **<u>–61 kJ/mol (exothermic)</u>**.
   <u>Sample Work:</u> **knowing that HCl is the limiting reagent, Pre-Lab Q3*

<u>HCl</u> C= 1.0 *M* V= 0.050 L n= 0.05 mol of HCl	$\Delta H^{\circ}_{rxn} = \frac{-(C_{Beaker} + C_{H_2O})\Delta T}{n}$ $\Delta H^{\circ}_{rxn} = \frac{-(\rho_{H_2O} C_{H_2O} V + m_{Beaker} C_{Pyrex})\Delta T}{n}$ $= \frac{-\left[(68.7g)\left(0.78\frac{J}{g\cdot°C}\right) + \left(1\frac{g}{mL}\right)(100mL)\left(4.184\frac{J}{g\cdot°C}\right)\right]6.5°C}{0.05\ mol}$ $\Delta H^{\circ}_{rxn} =$ <u>–61 kJ/mol</u>

   b. Enthalpy of neutralization of strong/weak neutralization – example shown for strong acid/weak base: **<u>–58 kJ/mol (exothermic).</u>**
   <u>Sample Work:</u> **knowing that HCl is the limiting reagent, Pre-Lab Q3*

HCl C= 1.0 *M* V= 0.050 L n= 0.05 mol of HCl	$\Delta H^{\circ}_{rxn} = \frac{-(C_{Cal})\Delta T}{n}$ $= \frac{-\left[(68.7g)\left(0.78\frac{J}{g\cdot°C}\right)+\left(1\frac{g}{mL}\right)(100mL)\left(4.184\frac{J}{g\cdot°C}\right)\right]6.1°C}{0.05\ mol}$ $\Delta H^{\circ}_{rxn} =$ –58 kJ/mol

2. Show the balanced overall and net ionic equations for the following reactions:

 a. Strong Acid/Strong Base

 Balanced: $NaOH_{(aq)} + HCl_{(aq)} \rightarrow H_2O_{(l)} + NaCl_{(aq)}$
 Net Ionic: $OH^-_{(aq)} + H^+_{(aq)} \rightarrow H_2O_{(l)}$

 b. and either…

	Strong Acid/Weak Base	OR	Weak Acid/Strong Base
Balanced:	$NH_{3\,(aq)} + HCl_{(aq)} \rightarrow NH_4Cl_{(aq)}$		$HC_2H_3O_{2(aq)} + NaOH_{(aq)} \rightarrow NaC_2H_3O_{2(aq)} + H_2O_{(l)}$
Net Ionic:	$NH_{3(aq)} + H^+_{(aq)} \rightarrow NH_4^+{}_{(aq)}$		$HC_2H_3O_{2(aq)} + OH^-_{(aq)} \rightarrow C_2H_3O_{2(aq)}^- + H_2O_{(l)}$

3. Provide 2 sources of systematic error and explain how it affects their results. See previous lab (part 1) for examples.

Experiment 11. Gas Laws

Notes for the Procedure

- If students notice that their pressure readings aren't changing throughout the course of their experiments, make sure they stop and restart as soon as possible!

Announcement to Students

- **Post-lab Question 4 & 5** – Skip Question 4, just answer Question 5. (question 4 is repetitive).

Pre-Lab Questions

1. **Barometric pressure:** a.k.a. atmospheric pressure, the force exerted on a surface area of Earth by the weight of the air above it (Wikipedia).
2. State the 4 gas laws mathematically and in words:

	Mathematically	In words
Charles' Law	$\frac{V_1}{T_1} = \frac{V_2}{T_2}$	Volume varies directly with temperature of an ideal gas (at constant pressure).
Boyle's Law	$P_1V_1 = P_2V_2$	Volume varies inversely with pressure of an ideal gas (at constant temperature).
Avogadro's Law	$\frac{V_1}{n_1} = \frac{V_2}{n_2}$	Volumes of gases (at constant temperature and pressure) have the same amount of molecules.
Gay-Lussac	$\frac{P_1}{T_1} = \frac{P_2}{T_2}$	The pressure of an ideal gas is directly proportional to the temperature (at constant volume).

3. Students should briefly state what kind of trend they predict to see in the graph for part 1 and part 2 with references to the shape of the graph and predict the pressure change for all three parts of the procedure.
   a. **Part 1** – Non-linear Inverse curve with pressure falling as volume is increased; this graph reflects Boyle's Law.
   b. **Part 2** – Positive linear relationship with pressure rising proportionately as temperature is increased; this reflects Gay-Lussac's law.
   c. **Part 3** – Pressure and temperature increases upon addition of the acid that initiates an exothermic reaction that generates CO_2 in the flask.

Post-Lab Questions

Part 1:

1. When the volume is doubled from 5.0 mL to 10.0 mL, pressure is halved. Students should use experimental pressure values to demonstrate this understanding of Boyle's Law ($P_1V_1=P_2V_2$).
2. The relationship between the pressure and volume of a confined gas is inverse, as pressure falls when volume is increased ($P \propto 1/V$).
3. If the volume of the syringe was increased from 10.0 mL to 40.0 mL, the pressure should be reduced by a factor of 4. Students must show calculation work with Boyle's law for full marks.
4. The relationship between the pressure and volume is inverse, so students should calculate the constant *k* using k= P·V in the third column of the Part 1 Data Sheet and remark that the calculated value of *k* is constant for their data pairs, confirming that the relationship is inverse. Ensure students also propagate their errors (state uncertainties).

Part 2:

1. The volume and the amount of moles of gas in the flask were held constant.
2. Pressure and temperature relationship is k= P/T. Knowing PV=nRT, they should see that V,n and R are constants in this experiment, thus they can be represented by *k*, arriving at the expression k= P/T. Alternatively, they can also mention that pressure-temperature has a direct relationship, so it would be represented as k=P/T, from what they have learned in Part 1, Question 4.
3. Given that the relationship is direct, students should choose k=P/T to calculate the fourth column of their Part 2 Data Sheet. Again, if they have chosen the correct formula, they should notice that the value of *k* is constant and state their uncertainties.
4. Skip. Next question is a more comprehensive version.
5. Using the equation of the line, y=mx+b, provided in the regression box of their pressure-temperature graph, the Celsius absolute zero is the y-intercept (*b*). Alternatively, they may extrapolate their graph to find the y-intercept. Students must include an estimate of the uncertainty; and comment on whether the true Celsius absolute value fits within the limits of their experimental uncertainty.

Part 3:

1. Data sheet should be complete and all calculations should be shown in the students' notebooks.
2. Dalton's Law states that the total pressure of a mixture of non-reactive gases is equal to the sum of partial pressures exerted by each gas (Wikipedia); and mathematically, it is represented as $P_{total} = P_1 + P_2 \ldots + P_n$.
   Students have the initial and final pressure readings, and knowing that CO_2 is produced in-between, then the difference between the initial and final is the contribution of the partial pressure by CO_2, which they should show using their experimental values.

3. Students should use their known values from this experiment (P_{CO2}, V, n_{CO2},T_{final}) to calculate an experimental value of *R* with the uncertainty calculated.
4. State whether the accepted value of *R* (8.3145 J K^{-1} mol^{-1}) agrees with their experimental value (fits within their limits of uncertainty).

Experiment 12. Titration of Vitamin C in Orange Juice and Tang

Notes for the Procedure

- These titrations are *much* quicker when the students allow a quicker rate of titrant to flow from the burette during the beginning of the lab.
- The endpoint colour of the titrated orange juice can be quite difficult to discern. Watch the *Techniques for the Vitamin C Titration* video to see what colour the students should expect to see once their solutions have reached the endpoint. The accepted protocol is when the blue-black indicator colour no longer dissipates from the solution.

Announcement to Students

- **Post-Lab Question 4** – To answer this question for Tang, students need to know it takes ***31 g of Tang to prepare a 250 mL (1 serving).*** This information is not provided in the manual.

Pre-Lab Questions

1. In this lab, we use the recommended daily intake determined by U.S. FDA, **<u>60 mg</u>** per day for adults (NIH). If students find a slightly different value (with cited sources), it may be accepted, though their answer to Post-lab Question 3 may differ. Mark accordingly.
2. The concentration of vitamin C in the 5.00 mL aliquot is **<u>2.9×10^{-5} *M*</u>** (2 sig figs). Show calculations:

25 mg vit C	1 g	1000 mL	1 mol	
250 mL	1000 mg	1 L	176.12 g	= 0.00057 M vit C

Known: C_1= 0.00057 *M*; V_1=5.00 mL; V_2= 100.0 mL
Solve: $C_1V_1=C_2V_2 \rightarrow C_2$=**0.000029 M vit C**

3. The main challenge of detecting the endpoint in this lab is that the blue-black colour of the indicator is difficult to see when mixed in with the orange colour of the solution. This is particularly true in OJ, where the pulp impedes visibility. In practice, the endpoint colour of the solution is brown, rather than a blue-black hue.

Post-Lab Questions

1. Students should use their titration data to determine the *concentrations* of vitamin C in OJ and Tang. Work must be shown and errors propagated.
<u>Sample Work for OJ (similar calculation for Tang)</u>:
Titrant: $V_{I2\ used}=V_1$= 30 mL ( )= 0.030 L ( ); C_1= 0.0005 *M*
OJ Aliquot: V_2=0.010 L

$C_1V_1=C_2V_2 \rightarrow C_2=0.0015\ M$ vitamin C in the aliquot of OJ... This is equivalent to the concentration of the stock OJ.

2. Students must use their data to determine the *mass* of vitamin C in OJ and Tang; then based on their answer, state which one has more vitamin C. Calculations must be shown, errors propagated, and correct amount of sig figs shown.
   Sample Work for OJ (similar calculation for Tang):

0.0015 mol vit C	176.12 g	0.100 L	= 26 mg vitamin C in 100 mL of the stock OJ prepared
1 L	1 mol		

3. Students should show calculations to determine the percentage of recommended daily intake in 1 serving (250 mL) of the OJ and Tang. Answers will vary based on experimental value and the value they found in Pre-Lab Question 1. (U.S. FDA RDI of vitamin C= 60 mg). Full marks for error propagation and correct sig figs.
   Sample Work for OJ:

$$\frac{26 \text{ mg vit C}}{65 \text{ mL pure OJ}} = \frac{x \text{ mg vit C}}{250 \text{ mL pure OJ}}$$

Isolate for *x*.
x= 100 mg vit C

$$\%\text{RDI} = \frac{x \text{ mg}}{60 \text{ mg}} \times 100\%$$

$\underline{\%\text{RDI} = 167\%}$

   Sample Work for Tang:

$$\frac{18 \text{ mg vit C}}{6 \text{ g Tang powder}} = \frac{x \text{ mg vit C}}{31 \text{ g Tang powder}}$$

Isolate for *x*.
x= 93 mg vit C

$$\%\text{RDI} = \frac{x \text{ mg}}{60 \text{ mg}} \times 100\%$$

$\underline{\%\text{RDI} = 155\%}$

4. Vitamin C is a good antioxidant because it is an electron donor (Padayatty *et al.*), thus it can donate its electrons to free radicals that often oxidize lipids, proteins, and DNA in the body to fill its open valence shell.

Experiment 13. Corrosion of Metals

Notes for the Procedure

- Make sure that the students are preparing for the following Parts (B → E) as their gel is cooling in Part A to save time.
- Final observations can be made for Part B towards the end of the lab; better results and observations are yielded when the reaction is allowed to progress longer.
- When the students are placing the nails with copper wire in their Petri dishes, they should make it sit in the same plane as the gel and flatten the copper wire as much as possible so none protrudes out from the gel.

Pre-Lab Questions

1. Definition of a **reference electrode**: an electrode with a stable and well-known electrode potential (Wikipedia). Ensure a reference is cited.
2. The reference electrode for the standard reduction table in the textbook is the **standard hydrogen electrode**.
3. Standard potential of the $Zn|Zn^{+2}|\,|Fe^{+2}|Fe$ cell is **0.32 V**. Students must show calculation.
   Calculation:

CATHODE, reduction	$Fe^{2+}_{(aq)} + 2e^{-} \rightarrow Fe_{(s}$	$E° = -0.44$ V
ANODE, oxidation	$Zn_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2e$	$E° = -0.76$ V
Cell potential	$Zn_{(s)} + Fe^{2+}_{(aq)} \rightarrow Zn^{2+}_{(aq)} + Fe_{(s)}$	$E°_{cell} = E°_{cath} - E°_{anod}$ $E°_{cell} =$ 0.32 V

4. In order to get a positive reading for the voltmeter, the positive lead should be attached to the cathode (higher potential) and the negative lead to the anode (lower reduction potential).
5. In a Cu|Ag cell, connect the positive lead to Ag (cathode) and the negative lead to Cu (anode).

$Ag^{+}_{(aq)} + e^{-} \rightarrow Ag_{(s)}$	$E° = +0.80$ V	higher reduction potential → CATHODE (will be reduced)
$Cu^{2+}_{(aq)} + 2e^{-} \rightarrow Cu_{(s)}$	$E° = +0.34$ V	lower reduction potential → ANODE (will be oxidized)

   In a Cu|Pb cell, connect the positive lead to Cu (cathode) and the negative lead to Pb (anode).

$Cu^{2+}_{(aq)} + 2e^{-} \rightarrow Cu_{(s)}$	$E° = +0.34$ V	higher reduction potential → CATHODE (will be reduced)
$Pb^{2+}_{(aq)} + 2e^{-} \rightarrow Pb_{(s))}$	$E° = -0.13$ V	lower reduction potential → ANODE (will be oxidized)

6. Question 6 and 7. Students must show the balanced half reactions of the following two reactions (Q6) then determine the standard reduction potential.
   a. $Fe^{+2}_{(aq)} + Cu_{(s)} \rightarrow Cu^{+2}_{(aq)} + Fe_{(s)}$

Reduction	$Fe^{+2} + 2e^{-} \rightarrow Fe_{(s)}$	$E° = -0.44$ V
Oxidation	$Cu_{(s)} \rightarrow Cu^{2+}_{(aq)} + 2e^{-}$	$E° = -0.34$ V
Overall Balanced	$\mathbf{Fe^{+2}_{(aq)} + Cu_{(s)} \rightarrow Cu^{+2}_{(aq)} + Fe_{(s)}}$	$E°_{cell} = E°_{red} + E°_{ox}$ **$E°_{cell} = -0.78$ V**

b. $Zn^{+2}_{(aq)} + Fe_{(s)} \rightarrow Fe^{+2}_{(aq)} + Zn_{(s)}$

Reduction	$Zn^{+2} + 2e^{-} \rightarrow Zn_{(s)}$	E°= -0.76 V
Oxidation	$Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e^{-}$	E°= +0.44 V
Overall Balanced	**$Zn^{+2}_{(aq)} + Fe_{(s)} \rightarrow Fe^{+2}_{(aq)} + Zn_{(s)}$**	$E°_{cell} = E°_{red} + E°_{ox}$ **$E°_{cell}$= -0.32 V**

7. Students should show calculations as in the third column on the tables above in Q6 to determine the standard electric potentials, (a) -0.78 V; (b) -0.32 V.

Post-Lab Questions

1. The cathode reaction in Part B (corrosion of iron) is the reduction of oxygen gas to hydroxide ions. **Cathode:** $O_{2(g)} + 2H_2O_{(l)} + 4e^{-} \rightarrow 4OH^{-}_{(aq)}$.
   Some blue colour may be detected near the iron metal (from the oxidation of $Fe_{(s)} \rightarrow Fe^{2+}_{(aq)}$, which is detected by potassium hexacyanoferrate (III), producing a blue pigment (Prussian blue).

2. Students are to identify the anode and cathode and explain the distribution of colours for the experiments in Part C (corrosion of iron with other metals). The third column of the following table is a rough guide of what would be seen in the lab, but **ideal answers** would also indicate *where* the colour is distributed (e.g. at points of the needle, sharp edges, etc.) as discussed in the introduction to the lab:

	Electrodes	**Inferences**
Fe only (Bare nail)	Anode: $Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e$	Some blue colour visible.
	Cathode: $O_{2(g)} + 2H_2O_{(l)} + e \rightarrow 4OH^{-}_{(aq)}$, then $Fe^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow Fe(OH)_{2(s)}$ then rust $Fe(OH)_{2(s)} + O_2 \rightarrow Fe(OH)_{3(s)}$	Pink colour not present, because the OH^{-} ions go on to react with Fe^{2+}
	∴Fe rusts a little, more so in regions where the electric field is higher (points & sharp edges), where the electrons can leave the metal surface easier.	
Fe & Zn	Anode: $Zn_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2e$	Blue is not present. (Fe is not oxidized)
	Cathode: $O_{2(g)} + 2H_2O_{(l)} + e \rightarrow 4OH^{-}_{(aq)}$,	Intensity of pink in the gel is high.
	∴Since Zn has a lower reduction potential than Fe, it is preferentially oxidized by O_2, thus Fe^{2+} is not made and the nail does not rust.	
Fe & Cu	Anode: $Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e$	Intensity of blue colour is high.
	Cathode: $O_{2(g)} + 2H_2O_{(l)} + e \rightarrow 4OH^{-}_{(aq)}$, then $Fe^{2+}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow Fe(OH)_{2(s)}$ then rust $Fe(OH)_{2(s)} + O_2 \rightarrow Fe(OH)_{3(s)}$	Pink colour is not present.
	∴ Fe rusts a lot. Since Fe has a lower reduction potential than Cu, it is preferentially oxidized. Further, the rate at which Fe rusts is faster when in contact with a metal of greater reduction potential.	

3. Students should compare the extent of colour development around the nails in Part D and explain the differences. The main concept to identify is that the rate of galvanic corrosion of the

Fe (anode) is affected by the surface area ratio of the cathode to anode (S_{cath}/S_{anod}) (McGraw-Hill Professional). The more copper wire (cathode) there is to covering the Fe nail (anode surface), the greater the rate of corrosion because the flow of electron is concentrated out from the nail (ETHZ).

4. Students should set up and complete a table similar to the following one shown below for Part E:

Metal pair	Leads	Voltage	Theoretical V	Agree/Disagree—Why?
Cu & Fe	Red: Cu Black: Fe		0.78	
Zn & Fe	Red: Fe Black: Zn		0.32	

5. Steel gas pipelines are often protected from corrosion by placing a lump of zinc that is electrically attached to the pipe at regular intervals. Since steel is an alloy of Fe, Zn may be used as a sacrificial anode because it has a lower reduction potential than iron. Thus between the two metals, zinc would be oxidized/corroded in preference to the steel structure. This was observed in Part C of the lab with an Zn-protected Fe nail.

Experiment 14. Qualitative Analysis

Notes for the Procedure

- Make sure students add the ammonia solution drop-wise and observe as instructed in the manual as the reaction progresses in such a way that students must document what happens after each drop that they add.

Pre-Lab Questions

1. Solubility of the following reagents in water at 25°C:

Reagent	**Solubility in Water, 25°C**
NaF	soluble
CaF_2	soluble
$(NH_4)_2SO_4$	soluble
$BaSO_4$	insoluble
$FeCO_3$	insoluble
K_2CO_3	soluble
MnS	insoluble
Na_2S	soluble
$Ni(OH)_2$	insoluble
$Ba(OH)_2$	soluble

Post-Lab Questions

1. Some reactions produce precipitate while others don't depending on whether the end products are soluble in water. For instance, if both products are soluble, like in the reaction between $Cr(NO_3)_3 + KI \rightarrow CrI_3 + KNO_3$, both the products are all soluble in room temperature water (Table 6 of the manual), so they will remain as ions in aqueous solution and no precipitate will form. On the other hand, in a reaction like $Cr(NO_3)_3 + Na_2CO_3 \rightarrow NaNO_3 + Cr_2(CO_3)_3$, carbonates are generally insoluble, so the $Cr_2(CO_3)_3$ forms a precipitate and the solution changes to a grey colour.
2. If the contents of the test tubes with precipitates were dumped into a large container of water, the amount of precipitate would not change because the concentration of pure solids is fixed and is not considered in Le Chatelier's Principle.
3. Chromates are generally insoluble, with the exception of K^+ (alkali metals).

Experiment 15. Titrimetric Analysis

Notes for the Procedure

- Since the students will be completing 4 titrations, it's best to advise them to allow larger portions of their titrant to flow from the burette to the reaction beaker at the beginning of the titration to quicken the process.

Pre-Lab Questions

None.

Post-Lab Questions

1. Students will use their titration data of V_{NaOH} used to determine the exact concentration NaOH in the burette. Sample work must be shown with error propagation and reference values cited.
   Example: with a 39.2 mL NaOH titration
   Given: $V_{NaOH}=V_1= 0.0392$ L; $V_{KHP}=V_2=0.050$ L; m_{KHP} used=0.80 g; $M_{KHP}= 204.22$ g/mol
   Solve: $n_{KHP}=m_{KHP}/M_{KHP}= 0.003917$ mol KHP
   $C_1V_1=n_{KHP} \rightarrow$ $C_1= 0.10$ (2 sig figs) *M* NaOH
2. The **equivalence point** is the stage in titration where the amount of titrant added is stoichiometrically equivalent to react with all the analyte in solution (ChemEd). The **endpoint** of titration is when the indicator of the solution changes colour to signal the equivalence point (Wikipedia). An ideal titration will use an indicator so that the endpoint occurs as close to the equivalence point as possible. Students should also compare their theoretical equivalence point vs. their actual endpoint and explain why it may be same/different.
3. Provide 3 sources of errors and explain how it would affect their results. Examples:
   a. *Contaminated burette* **–** may ruin the base and alter the amount of base needed to titrate the KHP, affecting concentration calculations.
   b. *Parallax error* – incorrect volume readings would skew the calculated results of titration.
   c. *Splashing* – may have left some KHP solution unaccounted for in the titration.

Experiment 16. Diprotic Acid Titration

Notes for the Procedure

- There are no specific technical issues that are particular to this experiment; however, be ready to help students and answer any questions they may have about the experiment.

Pre-Lab Questions

1. The **equivalence point** is the stage in titration where the amount of titrant added is stoichiometrically equivalent to react with all the analyte in solution (ChemEd). The **endpoint** of titration is when the indicator of the solution changes colour to signal the equivalence point (Wikipedia).

2. Show work up of how the pH must be equal to pKa at the half-titration point, as follows:
At half-titration, the initial x concentration of H_2X is reduced by ½…

	$H_2X_{(aq)}$	$\rightleftharpoons$	$H^+_{(aq)}$	+	HX
I	*x*		-		-
C	–0.5*x*		+0.5*x*		+0.5*x*
E	*x* – 0.5*x* =0.5*x*		0.5x		0.5*x*

$$K_{a1} = \frac{[\mathrm{H^+}][\mathrm{HX^-}]}{[\mathrm{H_2X}]}$$

$$K_{a1} = \frac{[0.5x]\cancel{[0.5x]}}{\cancel{[0.5x]}} = 0.5x$$

$$K_{a1} = [\mathrm{H^+}]$$

$$-\log(K_{a1}) = -\log([\mathrm{H^+}]) \rightarrow \mathbf{pK_{a1} = pH}$$

3. The K_a and pK_a values for sulfurous acid are: K_a=$1.5×10^{-2}$, pKa = 1.8.

4. The pH range for the colour change of phenolphthalein is: 8.3 – 10.0.

5. The net ionic equations for equations 51 – 53 are shown below:

(Eq. 51) $H_2X_{(aq)} + NaOH_{(aq)} \rightleftharpoons H_2O_{(l)} + NaHX_{(aq)}$

$H^+_{(aq)} + HX^-_{(aq)} + Na^+_{(aq)} + OH^-_{(aq)} \rightleftharpoons H_2O_{(l)} + HX^-_{(aq)} + Na^+_{(aq)}$

NET IONIC: $\mathbf{H^+_{(aq)} + OH^-_{(aq)} \rightleftharpoons H_2O_{(l)}}$

(Eq 52) $NaHX_{(aq)} + NaOH_{(aq)} \rightleftharpoons H_2O_{(l)} + Na_2X_{(aq)}$

$Na^+_{(aq)} + HX^-_{(aq)}\ Na^+_{(aq)} + OH^-_{(aq)} \rightleftharpoons H_2O_{(l)} + 2Na^+_{(aq)} + X^{2-}_{(aq)}$

NET IONIC: $\mathbf{HX^-_{(aq)} + OH^-_{(aq)} \rightleftharpoons H_2O_{(l)} + X^{2-}_{(aq)}}$

(Eq 53) $H_2X_{(aq)}$ + $2NaOH_{(aq)}$ $\rightleftharpoons$ $2H_2O_{(l)}$ $Na_2X_{(aq)}$

$H^+_{(aq)}$ + $HX^-_{(aq)}$ + $2Na^+_{(aq)}$ + $2OH^-_{(aq)}$ $\rightleftharpoons$ $2H_2O_{(l)}$ + $2Na^+_{(aq)}$ $X^{2-}_{(aq)}$

NET IONIC: $H^+_{(aq)}$ + $HX^-_{(aq)}$ + $2OH^-_{(aq)}$ $\rightleftharpoons$ $2H_2O_{(l)}$ + $X^{2-}_{(aq)}$

Post-Lab Questions

1. Students need to use their titration data for V_{NaOH} used to calculate the moles NaOH used to reach endpoint. Sample work with error propagation and correct sig figs should be shown.
   Example Calculation: if 9.50 mL NaOH was used to reach the first endpoint
   Given: C_{NaOH}=0.10 M, V_1=0.00950 L ( )
   Solve: n=CV → **n= 0.00095 mol NaOH ( ) (2 sig figs)**

2. Students have a choice of using Equation 51 (first equivalence point) or Equation 53 (second equivalence point) to determine the mole of diprotic acid present. Sample work should be shown with error propagation.
   Equation 51: $H_2X_{(aq)}$ + $NaOH_{(aq)}$ $\rightleftharpoons$ $H_2O_{(l)}$ + $NaHX_{(aq)}$
   Solve: 1 mol $H_2X_{(aq)}$: 1 mol $NaOH_{(aq)}$ at the first equivalence point, ∴ **0.00095 mol** of unknown diprotic acid is present

3. Sample work and error propagation should be shown to determine the molecular weight of the diprotic acid (in g/mol) using the mass of diprotic acid that was measured out.
   Example Calculation: if 0.1200 g ( ) of unknown acid was measured out
   Given: m= 0.1200 g ( ), n= 0.00095 g ( )
   Solve: M=m/n → M_{exp}= **126 g/mol** is the molar mass of the unknown diprotic acid

4. Answers may vary based on the sample acid students chose. Mark accordingly based on the sample they chose. *E.g.* based on the example given in Question 3, students would conclude their sample was oxalic acid (M_{TY}=126 g/mol).

5. Percent error calculation of their molecular weight with error propagation should be shown with correct sig figs.

$$\%\ error = \left(\frac{|\mathrm{M_{TY}} - \mathrm{M_{exp}}|}{\mathrm{M_{TY}}}\right) \times 100\%$$

6. Students must show calculation and error propagation with correct sig figs to determine the precise V_{NaOH} of the first and second half-titration points.
   Sample Work: if V_{equiv1}= 9.50 mL ( )and V_{equiv2}= 18.50 mL ( )
   Given: V_{equiv1}= 0.00950 L, V_{equiv2}= 0.01850 L
   Solve:

V_{half1}=0.5V_{equiv1}	V_{half2}=0.5(V_{equiv1}+V_{equiv2})
V_{half1}=0.5(0.00950)	V_{half2}=0.5(0.0095+0.01850)
V_{half1}=**0.00475 L (3 sig figs) ( )**	V_{half2}=**0.0140 L (3 sig figs) ( )**

7. The equivalence point of the titration results in a basic solution, since the reaction involves a strong base and weak acid, wherein the fully deprotonated weak acid undergoes a hydrolysis reaction: $X^{2-}_{(aq)} + H_2O_{(l)} \rightarrow HX^{-}_{(aq)} + OH^{-}_{(aq)}$, producing OH^- ions that increase the pH of the final solution. Thus, a good indicator would stop the titration (endpoint) when the solution is basic to signal the equivalence point. The phenolphthalein would signal the endpoint across a pH range of 8.3 – 10.0; this is best suited for the given titration reaction compared to bromothymol blue, which changes colour in a acidic to neutral pH range: 6.0–7.6 (Wikipedia).

8. Students should provide a few points on the advantages and disadvantages of using a pH probe vs. indicator. Here are some examples:

	PROS	**CONS**
pH probe	• Very precise and accurate • No need to arrange the use of an appropriate indicator • Can use graphing software to see how close to equivalence point	• Very expensive • More difficult to complete a titration alone (must watch reaction beaker as well as the computer monitor)
indicator	• Cheaper than pH probes • Effective when a proper indicator is chosen • One can focus on just the titration apparatus, unlike the pH probe	• The effectiveness of indicators are dependent on the knowledge (pH ranges of the indicators)

Experiment 17. Buffer Titration

Notes for the Procedure

- As students are titrating their buffers and are approaching the endpoint, students should add the titrant minimally to get as many points in that region for their graph. Once they hit the endpoint the pH will spike dramatically and they won't have another opportunity to get data points within that region to produce a nice curve. As such, instruct students to be patient during that point in their titration.

Pre-Lab Questions

1. Calculation work to prepare **Buffer A** with solid sodium acetate, ensure correct sig figs:

$$pH = pK_a + log\frac{[conj.\ base]}{[acid]}$$

$$4 = 4.74 + log\frac{[x]}{[0.1]}$$

$x = 0.0182\ M$ of $OAc^{=}$

0.0182 mol	82.0343 g	0.100 L	=**<u>0.150 g (3 sig figs) of NaOAc required.</u>**
1 L	1 mol		

2. Calculation work to prepare **Buffer B** with solid sodium acetate, ensure correct sig figs:

$$pH = pK_a + log\frac{[conj.\ base]}{[acid]}$$

$$4 = 4.74 + log\frac{[x]}{[1.0]}$$

$x = 0.182\ M$ of $OAc^{=}$

0.182 mol	82.0343 g	0.100 L	=**<u>1.50 g (3 sig figs) of NaOAc required.</u>**
1 L	1 mol		

Post-Lab Questions

1. From Pre-Lab Questions 1 & 2:
   Buffer A: [Acid] = 0.1 M, [Conjugate base]= 0.0182 M

   Buffer B: [Acid] = 1.0 M, [Conjugate base] =0.182 M

2. need to revisit

Comment [DL1]: try to solve again

3. Buffer B would be a better choice to keep the pH steady at 4, because it has a greater capacity to resist the changes introduced by an acid/base. In the lab, it was observed that 20 mL of NaOH was added before the Buffer B system changed pH; on the other hand, it took only 2 mL of NaOH to change the Buffer A system. This is because there is quantitavely more acetic acid present in the Buffer B solution. Upon addition of NaOH, the OH^- shifts the equilibrium of the buffer system forward, so that acetic acid is dissociated to form the H^+ ions that can neutralize OH^-. Buffer A has smaller capacity to buffer acids/bases simply, because it has less acid and conjugate base to do that.

Experiment 18. Rate and Order of a Chemical Reaction

Notes for the Procedure

- As students complete each trial, they should quickly mix the chemicals together, stir the reaction with a glass rod for a few seconds, then efficiently rinse and transfer it into the cuvette for analysis in the SpectroVis. They want to get the data toward the beginning of the chemical reaction, so being timely with mixing, stirring and transferring the solution to the cuvette will provide optimal results.
- For the rate order calculations in this lab, ensure that the students are aware that they must *ROUND* up (≥0.5) or down (<0.5) their decimal to a whole number to determine the order of the reaction.
- **Example 1**: *An order of 2.3 was calculated for the rate with respect to $FeCl_3$, therefore the order is 2.*
- **Example 2**: *An order of 1.6 was calculated for the rate with respect to KI, therefore the order is 2.*

Pre-Lab Questions

None!

Post-Lab Questions

1. Students should complete a table of concentration for $FeCl_3$ and KI as shown below. They should include one sample dilution calculation for concentration.
   Sample Data:

Trial	[$FeCl_3$] (*M*)	[KI] (*M*)	Initial Rate (s^{-1})
1	0.0075	0.0075	0.0026
2	0.0075	0.00375	0.0015
3	0.00375	0.0075	0.0021
4	0.00565	0.00375	0.0014
5	0.00375	0.00565	0.0015

Sample Calculation of Dilution: Trial 1 [$FeCl_3$]:
Given C_1=0.020 *M*; V_1=0.015 L; V_2=0.040 L
Solve $C_1V_1 \rightarrow$ **C_2= 0.0075 M**

2. The order of reaction with respect to $FeCl_3$ is 0^{th}; for KI, it is 1^{st} order. Students must show the calculation to arrive at this conclusion.

Sample Work:

$$\frac{rate_1}{rate_2} = \frac{k[FeCl_3]_1{}^m[KI]_1{}^n}{k[FeCl_3]_2{}^m[KI]_2{}^n}$$

KI	$FeCl_3$
Using Trials 1 & 2 data ($[FeCl_3]$ constant) $\frac{rate_1}{rate_2} = \frac{[KI]_1{}^n}{[KI]_2{}^n}$ $1.73 = (2)^n$ $\boldsymbol{n = 0.79 \cong 1}$ ∴ The reaction is **1st order** with respect to KI.	Using Trials 1 & 3 data ([KI] constant) $\frac{rate_1}{rate_3} = \frac{[FeCl_3]_1{}^m}{[FeCl_3]_3{}^m}$ $1.24 = (2)^m$ $\boldsymbol{m = 0.31 \cong 0}$ ∴ The reaction is **0th order** with respect to $FeCl_3$.
Also possible with Trials 3 & 5.	Also possible with Trials 2 & 4.

3. Rate law expression for the reaction is: **rate = k[KI].**

4. Students must calculate the rate constant, *k*, using their experimental data and the expression derived in Question 3 for each five trials.

$$k = \frac{rate}{[KI]}$$

Sample Work: Trial 1

$k_1 = \frac{0.0026}{0.0075} = 0.0035 \frac{L}{mol \cdot s}$

5. Students must determine the mean rate constant and provide the calculation and error propagation.

$$k_{average} = \frac{k_1 + k_2 + k_3 + k_4 + k_5}{5}$$

6. Provide 3 sources of error and explain how it would affect the results. Examples:

   a. *Taking too long to mix the chemicals and record the data before each run* would have collected data too far in the chemical reaction or when the reaction is already finished, this would throw off the slope obtained from the graph and the final value of *k.*

   b. *Dirty/stained cuvettes* may alter the absorbance readings and tweaked the values of the slopes that were determined from measuring the data.

   c. *Parallax errors* may skew the amount of solution added; this would have changed the final rate law expression determined for this reaction.

Experiment 19. Rate Law Determination of the Crystal Violet Reaction

Notes for the Procedure

- It's important that students don't spend too much time waiting for their hot-water baths to reach the correct temperature range. If students must wait to reach the required temperature, they can analyze the data from their room temperature bath while waiting, (Logger Pro will also save the lnA and 1/A functions when they analyze the next runs). If students have their hot-water baths at the correct temperature from the start, they can complete all 3 runs and then return to analyze them all together at the end.

Pre-Lab Questions

1. If the temperature is increased for a chemical reaction, the rate of the reaction will also increase. At higher temperatures, molecules have more kinetic energy, which increases the frequency of collisions for chemical reactions to occur (Clark 1).
2. With regards to the Arrhenius equation, the value of k depends on the temperature, not concentration.
3. The value of R that should be used is **R = 8.314×10^{-3} kJ/(mol·K).** This is because the activation energy in the given Arrhenius equation is reported in kJ/mol , thus to obtain the correct units for the value of the rate constant *k*, kJ/(mol·K) must be used in R.

Post-Lab Questions

1. The room temperature data produced a linear slope when lnA vs. time was plotted, thus the reaction is **first order**.
2. Students must calculate the rate constant *k* using the appropriate slope from the regression of their graph for all 3 temperature ranges. (**k'= –slope** for all temperatures in this lab)
Sample Data:

	Slope	Pseudo Rate Constant (k')
Room temp	-0.007534	0.007534
30.3°C	-0.01128	0.01128
35.7°C	-0.01548	0.01548

3. Rate law expression in terms of crystal violet: **rate=k[CV^+]**

4. Students must compare their experimental half-life value to the theoretical half-life value calculated from the expression t=(ln2)/k for their room temperature run. Sample work must be shown and errors propagated. Full marks are awarded if the values agree to within the limits of uncertainty.
   Sample Work:

   Graphical method:
   Select two points on the graph where one has an absorbance value half of the other.
   e.g. p_1(134 s, 0.2), p_2(224 s, 0.1)

   $t_{1/2} = t_2 - t_1$

   $t_{1/2} = 224 - 134$

   $t_{1/2} = 90$ sec

   Half-Life Formula:

   $$t_{1/2} = \frac{ln2}{k}$$

   $$t_{1/2} = \frac{ln2}{0.007534}$$

   $$t_{1/2} \approx 92 \text{ sec}$$

5. **Students must calculate the activation energy using the values from the room temperature and ~35°C data. Sample work and error propagation must be shown.**
   Sample Work:
   Given: R= 8.314 x 10^{-3} kJ mol^{-1} K^{-1}
   T_1= 24.5°C= 297.5 K, k_1'= 0.007534 s^{-1}
   T_2= 35.7°C= 308.7 K, k_2'= 0.01548 s^{-1}
   Solve:

   $$\ln\left(\frac{k_1}{k_2}\right) = \frac{E_a}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right)$$

   $$E_a = R\left[\frac{ln(k_1'/k_2')}{\frac{1}{T_2} - \frac{1}{T_1}}\right]$$

   $$E_a = \mathbf{49.1\ kJ/mol}$$

6. Students must plot a ln(k) vs 1/T (Kelvin) graph with 5 points. Graphs may be done on computer or by hand. Full marks are awarded for proper formatting: axes labelled with units, good title, regression shown, accurate data, etc.
   Using the graph and regression, the activation energy must be calculated with work shown and error propagation.
   Sample Work:
   Given: slope= -7989 K/s
   Solve:

   $$\text{slope} = \frac{-E_a}{R}$$

   $$E_a = -R(\text{slope})$$

   $$E_a = \mathbf{66.4\ kJ/mol}$$

7. Students are to compare the E_a determined in Question 5 and 6 and discuss which value is more accurate with logical support.

8. As rate = k[CV^+], if the concentration of crystal violet was doubled, so would the rate.

Experiment 20. Synthesis of Aspirin – Part 1

Notes for the Procedure

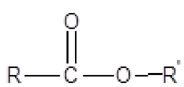
- If crystals are not forming for students while they are synthesizing their aspirin, you can help them by scratching the inside of their beakers with a glass stir rod.

Announcement to Students

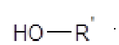
- **POST-LAB QUESTION:** There is one additional question to answer– *Calculate the percent yield of your crude product based on the theoretical value you determined in Question 2*.

Pre-Lab Questions

1. Balanced equation for the production ASA:
   $C_7H_6O_{3(s)} + C_4H_6O_{3(aq)} \rightleftharpoons C_9H_8O_{4(s)} + C_2H_3O_{2(aq)}$
2. A catalyst is a substance that increases the rate of reaction without itself undergoing any permanent chemical changes. (Dictionary.com)
3. General formula of an ester and alcohol:



ester



alcohol

Post-Lab Questions

1. Students must use the data in their experiment to determine the amount of salicylic acid used. Sample work must be shown with error propagation and correct sig figs.
   Sample Work:
   Given: m=5.00 g SA, M= 138.12 g/mol
   Solve: n=m/M → n= **0.0362** mol of salicylic acid was used

2. Students are to determine the theoretical yield of aspirin. Their work must be shown and errors propagated with correct sig figs.

   Sample Work:

	$C_7H_6O_{3(s)}$	+	$C_4H_6O_{3(aq)}$	→	$C_9H_8O_{4(s)}$	+	$C_2H_3O_{2(aq}$
m (g)	5.00				6.53 (3 sig figs)		
M (g/mol)	138.12				180.157		
n (mol)	0.0362				0.0362		

3. Students must calculate the percent yield of their crude sample. Work should be shown with errors propagated and correct sig figs.

$$\%yield\ of\ crude = \frac{\text{Actual crude yield}}{\text{Theoretical yield}} \times 100\%$$

Experiment 21. Recrystallization and Analysis of Aspirin – Part 2

Notes for the Procedure

- Ensure students are efficient when using the vacuum filter to let other groups use it as well.
- When determining the melting points of their crude and recrystallized aspirin, students should place both tubes in the Mel-Temp to save on time.
- The second groups to use the Mel-Temp may be run late, as they must wait for the apparatus to cool down before they can take their melting points. If it is too late in the lab, these groups may take melting points ranges from another group.

Pre-Lab Questions

None.

Post-Lab Questions

1. Students will use the ratio of mass-recrystallized:mass-crude to estimate the purity of the crude sample. Sample work, error propagation and correct sig figs must be shown. They should also use this value to comment on the percent yield they calculated in the previous lab, *e.g. the percent yield was inaccurate and overestimated, because only 52% of the sample was pure product.*
   Sample Calculation:
   Given: 1.00 g ( ) crude used for recrystallization;
   0.52 g ( ) pure recrystallized.

$$\%purity = \frac{\text{mass pure}}{\text{mass recrystallized}} \times 100\%$$

$$\%purity = \frac{0.52}{1.00} \times 100\%$$

$$\underline{\%purity = 52\% \text{ (2 sig figs)}}$$

2. The melting point range of the crude aspirin was lower than that of the recrystallized product because impurities lower the intermolecular forces of attraction between molecules of the pure product. By physically interrupting the bonds between aspirin crystals, they can't build up a relatively strong crystal lattice structure. This means that less heat is needed to overcome the solid-state intermolecular forces.

3. Students must calculate the percent yield of their pure sample. Work should be shown with errors propagated and correct sig figs.
   Sample Work:

Let *x* be amount of pure product we should yield from our total crude sample.

$$\frac{0.52\ g\ \text{pure}}{1.00\ g\ \text{crude}} = \frac{x}{7.20\ g\ \text{crude}}$$

x = ***3.7* g** is pure in total crude sample

	$C_7H_6O_{3(s)}$ +	$C_4H_6O_{3(aq)}$ →	$C_9H_8O_{4(s)}$	+ $C_2H_3O_{2(aq)}$
m (g)	5.00		***6.52***	
M (g/mol)	138.12		180.157	
n (mol)	0.00362		0.00362	

$$\%yield\ of\ pure = \frac{\text{Actual crude yield}}{\text{Theoretical yield}} \times 100\%$$

$$\%\text{yield of pure} = \frac{3.7\ g}{6.52\ g} \times 100\%$$

%yield of pure= <u>57% (2 sig figs)</u>

4. Provide 3 sources of error from this lab and explain how it would affect their results.

List of Lab Equipment Errors

This is a list of the instrumental errors inherent in the lab equipment. These values are used to propagate errors for uncertainty calculations.

Equipment	**Error**
Analytical balance	0.0002 g
Top-loading balance	0.01 g
10 or 50 mL Dispensette	0.5 %
50 mL Burette	0.05 mL
10 mL Graduated cylinder	0.1 mL
100 mL Graduated cylinder	0.6 mL
20 mL Syringe	0.05 mL
100 mL Volumetric flask	0.08 mL
Plastic cuvette	0.05 cm