Instructions for writing laboratory reports

INFRARED SPECTROSCOPY: DETERMINATION OF m-XYLENE IN COMMERCIAL XYLENES

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(Experiment date: Feb. 7, 14; Date of submission Feb. 21, 2005)

SUMMARY

Infrared spectroscopy is based on the principle of measurement of vibration frequency of a bond or a group of bonds and the corresponding absorption intensities of these vibrations. Using this principle it is possible to obtain separate absorption lines for o, m, and p-xylenes present in commercial xylene samples. In this experiment the commercial xylene sample was prepared by dilution in cyclohexane and the IR spectra were obtained in a fixed thickness (0.2 mm) liquid sample cell. A series of solutions containing 10.0, 20.0, 30.0 and 50.0 mg/mL of pure m-xylene were prepared in commercial xylene in cyclohexane. Then absorbance of the m-xylene band (λ_{max} = 650 cm^{-1}) was plotted vs. the concentration. m-Xylene concentration obtained from this standard addition plot was determined to be 24.5 ± 2.0 mg/mL. The key sources of errors in this experiment were sample preparation, background fluctuation, and the sample evaporation during measurement.[1] Error propagation calculation shows that these factors can contribute a maximum of 20% relative standard deviation.

[1]*This page should contain only the header and the summary of the experiment. Summary should include the basic principle of the technique. How it was used. Mention the most significant procedures by which the task was performed and some of the important results. The sources of errors and its effect on the experiment should also be summarized.*

INTRODUCTION

In this section you should discuss the basic principles of the experiment. It may involve physical, chemical, and instrumental principles. Provide short definitions where appropriate. You may use mathematical equations, show chemical reactions or describe the basic instrumental principle with a schematic drawing of the instrument. Do not try to draw a detailed diagram of the instrument. While describing all these you should remember the goal and the specific nature of the experiment. Therefore try to avoid generalities as far as you can. You should read the text and appropriately reference your work[2]. Remember that conciseness and correctness are valued over rambling discussion of irrelevant facts.

In the last paragraph of this section you should clearly describe the purpose of the experiment.

THEORY

It is often necessary to discuss the theory of the experiment in a separate section such as this. In this section you should discuss the relevant mathematical equations and the development of the theory. For example:

"Quantitation of toluene in commercial gasoline sample is based on a modified standard approach. Considering two peaks, the known (k) and an unknown (u) in the gasoline sample, the peak areas obtained after injection in the GC are given by

$$A_k = R_k C_k \qquad ...(1)$$
$$A_u = R_u C_u \qquad ...(2)$$

where R is response factor of the species and C is the molar concentration of the species[3]. In standard addition technique, a known volume of the known species is added to the mixture containing the known and the unknown compound."

EXPERIMENTAL

Apparatus: Briefly mention the apparatus used. Do not forget to mention the brand name, the model number and the manufacturer. This should be recorded during the experiment. However, you should mention very special capabilities of the instrument: such as a double beam spectrophotometer, diode array systems, Fourier transform spectrophotometer, computerized system etc.

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[2] *The prescribed text is Instrumental Analysis by Skoog and Leary. Most instrumental methods are described in it. However, you should consult other books on instrumental analysis. The Quantitative Chemical Analysis by D. C. Harris could be very handy for many analytical chemistry problems. All references must be placed as footnotes.*

[3] *Clearly describe the meaning of the parameters for each equation.*

Materials: List small apparatus (e.g., volumetric apparatus, pH meter, pumps etc.) chemicals used. Note the brand name, purity, type (e.g., reagent grade, UV grade, commercial grade, ultra pure, gold-label etc.) and the manufacturer. If you have prepared the reagent from stock chemicals then describe the preparation in detail. For example:

*"**Preparation of standard solution:** Standard solutions of m-xylene were prepared by dissolving 100.0, 200.0, 300.0 and 500.0 microlitres of m-xylene in commercial xylene to a final volume of 10.00 mL in a volumetric flask. Volumetric flasks were previously cleaned with cyclohexane and rinsed dry at room temperature. In this way residual water was removed form the volumetric flask[4]. Due to the high volatility of xylenes, solutions were tightly stoppered and kept in the refrigerator until further use. These solutions were used in the standard addition technique."*

Methods and Procedure: Report only those methods and procedure not described in the instruction manual. You may already have noticed that we modify procedure before or during the experiment. This is done in order to meet the demand of resource and time so that you can finish the experiment. Report all these changes and modifications. For example:

*"**Procedure modification:** Procedure #4 of this experiment was modified to use 30:70 v/v% tetrahydrofuran: water instead of the prescribed solvent mixture. Here both the nature (e.g., tetrahydrofuran instead of acetonitrile) and the composition were changed. This new solvent is supposed to increase the elution time of the components for a faster experiment[5]*.

You should also note instrumental procedures that you have learned during the experiment (e.g., injection technique in GC, LC, handling micropipet etc.)

DATA AND RESULTS

This is the most important section of all. You should give a careful consideration while writing this section. A general strategy is to gather all the raw data, process the raw data according to the instruction, prepare all graphs and tables and then arrange them before you start writing this section. You should check the following items while on this section (may not be in order):

- Do not include raw output from computer or computerized instruments. Show only representative raw data. These should be attached in the appendix. All numbers must have proper significant numbers.

- For a straight line fit, you must show the least-squares results. This includes the best slope, the best intercept, their errors and the correlation coefficient. If the line was used for calibration or for standard addition to determine the concentration of an unknown you must report the error in your calculation.

- Understanding experimental errors and their sources is one of the most important functions of an experimentalist. Terms like: systematic error, random error, uncertainties, standard

[4] *Describe the purpose of some of the procedures. Everything that you have been doing in the lab has a purpose. Some of these may seem obvious to you at the time but it may not be clear to others. Therefore, you should record them.*
[5] *You should note why these changes were made.*

deviation, t-test, and Q-test should be known and used whenever appropriate. The term "human error" should be used as a last resort with an explanation. Error propagation calculation should be included in this section.

- Graphs must have a title and appropriate labels. Tables should be numbered with a descriptive title. If equations are used in your calculation then describe the terms and show an example calculation with real data. Do not forget to show units of measurement.

- You may include short comments or pertinent observations in this section as they apply.

DISCUSSION

Start this section with what you set out to do and what was the outcome. Discuss the main points of the experiment, the final results and the sources of errors. The effect of these sources should be discussed as thoroughly as possible. You should also discuss the difficulties of the experiment in a manner that the method could have been improved or a procedure could have been done differently.

THINGS TO REMEMBER

- Keep a thorough record of the experiment in your lab notebook. Make sure you have obtained all the raw data and instrument records from your partners. Lab notebook carbon must be submitted with your final lab report. You are only allowed to use your lab notebook in the final lab exam.
- Present all data with appropriate significant figures.
- Present data with an error (measured or estimated) whenever necessary.
- Do error propagation calculations whenever necessary.
- Replicate measurements should be made whenever possible even if it is not instructed.
- A straight line calibration must be fitted to a linear regression model. The errors of slope, and intercept should be used to estimate the measurement error.
- All final results must be presented with an error.

HOW TO ORGANIZE FOR FINAL WRITE-UP

- Gather all the raw data in sequence. Make tables of raw data in Excel (XL).
- Process the raw data, make graphs, do regressions etc. Sometimes a printout of these final results is convenient to handle during write-up.
- Start writing the experimental section first. Once you have a clear idea of what was done, it would be easier to write other sections.
- Then do the results and discussion. The lab instruction requires you to answer some questions. Answer them in sequence (number them if necessary). Your instructor looks for these answers.
- You should try to integrate the Tables and Graphs from XL to your word document. Complicated equations can be hand written instead of typing.
- Once you have satisfied with your results and discussion section, write the introduction. A meaningful introduction can be written only when you have done the experiments and obtained results.
- Finally, write the summary.