

Dr. Jeff Altig

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Hours: By Appointment

Laboratory Manual & Schedule

www.nmt.edu/~jaltig/Laboratory.html

Grading

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| 10% | Pre-Lab Safety Questions
Each lab will have associated with it a series of Safety Questions. These must be completed before coming to class and turned in at the beginning of the lab session. These questions will likely require a short "web" search for answers. |
| 10% | Pre-Lab Quizzes
Prior to coming to the lab, you must view the Pre-Lab Video and read the Lab Handout. You will not be allowed start the laboratory if you have not done this. At the beginning of each laboratory exercise, a short quiz covering material in the video and the handout will be given. This quiz will be a record of your lab attendance. |
| 5% | Lab Report Discussion
Prior to turning in your second lab report you are required to meet with a chem tutor to discuss the structure of your report. The tutor can be any one of chemistry tutors assigned to the Jones Hall chemistry tutoring center. You are responsible for allowing enough time to have this discussion prior to the report being due. |
| 30% | Laboratory Notebook
Details are provided below. (Carbon copies due at the end of the Laboratory.) |
| 40% | Laboratory Reports
Details are provided below. (Due, with certain announced exceptions, one week following completion of the Laboratory.) Each student is responsible for completing their own lab reports. |
| 5% | Lab Safety, Proper Disposal of Chemicals and Tidiness
All safety rules (separate handout) must be followed, all chemicals must be disposed of properly, and your laboratory workstation must be kept tidy and left clean. At the end of each laboratory session, your laboratory instructor will issue a grade according to how well you are following the appropriate guidelines in this regard. |

Notes:

- 1) Missing more than two laboratory exercises will result in a failing grade for the course.
- 2) The "late papers" policy will be set by your laboratory teaching assistant. However, no work will be accepted more than two weeks after the due date. And, no work will be accepted after the last laboratory session of the semester.
- 3) If you are observed abusing a balance, including using an incorrect balance, you will be asked to leave the lab and you will receive a score of zero for that lab exercise.

Laboratory Notebook

The Laboratory Notebook is a record of your observations and data taken during the laboratory period. The notebook itself should be constructed with permanently sewn or fixed pages. In general terms, the notebook should contain a Table of Contents listing all the laboratory exercises completed. Each page of the notebook should be numbered, dated and initialed. All entries should be made in ink. Each page of the notebook should have a carbon-copy that is turned in at the conclusion of each laboratory exercise. Lab notebook entries should be completed before leaving the lab.

Each laboratory exercise should have data in the notebook in the following format:

I. Title

Each laboratory exercise should begin with a descriptive title. This should be completed before coming to the laboratory.

II. Statement of Purpose

The purpose of the exercise should be described in one or two sentences. This should not be a lengthy recitation of all the things to be done, but instead should be a simple statement of the one main purpose of the lab exercise. For our first experiment, a good statement of purpose would be:

To separate and identify the metal cations in a solution possibly containing Fe^{3+} , Ni^{2+} , Cu^{2+} or Co^{3+} . The separation will be carried-out via Paper Chromatography.

The statement of purpose should not be a repetition of the learning goals listed at the top of each laboratory exercise in the laboratory manual.

This should be completed before coming to the laboratory.

III. Procedure

A “bullet point” list of the main procedural steps to be carried-out during the laboratory exercise should be provided. This should not be a copy of the “Procedure” in the laboratory manual. It should be a very short list of the main things which need to be done. Procedural details can be obtained from the lab manual. The purpose of this list is to simply familiarize yourself with the steps required to complete the exercise. For our first experiment, a good procedure would be:

- *Prepare eluting solution from Acetone and 8M HCl.*
- *Dot unknown solution and known solutions onto the chromatography paper.*
- *Run chromatogram.*
- *Develop chromatogram with Ammonia vapor and DMG.*
- *Determine the Retention Factor for each metal cation.*

Notice the list is short and contains no details. Further, it is expected that if some interesting result is found, or if something goes wrong, there may be deviations from this procedure.

This should include as few items as possible. Each bullet point should be no more than a single sentence. (You do not want to waste your time copying out a procedure that is already available in the lab manual. Just hit the highlights.) You should, however, reference the source of the complete laboratory procedure. This may appear as:

Altig, Jeff (Accessed August 1, 2010) Paper Chromatography of a Metal Cation Mixture, Revision 2.0. Retrieved from www.nmt.edu/~jaltig/Chromatography.pdf.

This should be completed before coming to the laboratory.

IV. Data

Experimental details are provided in this section. Here is where you report what actually occurs during the laboratory exercise. This includes:

- i) Observations concerning all reagents and products.
- ii) Observations concerning all changes observed during the experiment.
- iii) Diagrams or descriptions of all experimental apparatus used.
- iv) Physical data taken.
- v) Record of the model and serial number for any commercially constructed equipment and calibration data, if available.

The data should be presented in a logical order and should be recorded as it is taken. Data tables should be used where appropriate. Only simple calculations should be performed at this point. For instance, tare weights can be subtracted:

weight flask & reagent	=	12.3435 g
weight flask empty	=	10.6987 g
weight reagent	=	1.6448 g

Or, calibration curves can be constructed and presented. However, lengthy calculations and data analyses should not be presented here.

Data should always be recorded using an ink pen. If an error is made, the erroneous data should be crossed-out with a single line and initialed. It is not uncommon to realize later the data was not erroneous and this allows for recovery of the correct piece of information. Spectra, graphs and other diagrams should be taped into the notebook securely.

For our first experiment, we could start taking data as:

The chromatographic eluting fluid is prepared by mixing:

*19.0 mL Acetone; a clear liquid with a sweet odor.
6.0 mL 8M HCl; a clear liquid with a very sharp odor.*

This is added to an 800mL beaker which serves as a chromatography tank. The tank is covered with a piece of aluminum foil.

etc.

Report what you have done, including any deviations from the recommended procedure. If you take the time to accurately record your observations, your analysis of the data will proceed much more smoothly.

Also, note that some verbiage is provided to put the data in context. You should not be chatty, but a minimalist approach with only recorded data is also unacceptable.

Laboratory Report

Unless instructed otherwise, you should complete a laboratory report for each lab exercise performed. This will be turned-in one week following completion of the exercise. This should be presented as a separate document.

Logically, the report should start general, placing the experiment in context. It should then proceed to the specifics of your experiment. It should then end generally; broadly, what has been learned. Grammatically, "You should report your work in the *past* tense and the work of other scientists in the *present* tense." [Rodney J. Sime; Physical Chemistry; Saunders (1988) 170.]

The laboratory report should have the following elements:

I. Title

This is the same title used in the laboratory notebook.

II. Abstract

This is a very, very short description of the experiment completed. It should not exceed three sentences in length. It should answer the following three questions:

- i) What was measured or determined?
- ii) How was the measurement performed?
- iii) What were the results?

Only one or two key results should be presented at this point.

For our first experiment, an example would be:

The metal cations in an unknown solution were separated and identified using Paper Chromatography. We found Unknown solution A contains the cations Fe^{3+} and Cu^{2+} .

III. Introduction

This should be a short introduction as to why we are performing the current laboratory exercise. This should be a description of what was measured and why. It should not be a listing of the learning goals for the experiment. All relevant chemical reactions should be listed.

In effect, this should be a prelude to the experimental procedure outlined in the next section. Here, you outline why you are performing this experiment and in the next section, you outline how the experiment was conducted.

For our first experiment, this could be:

Paper Chromatography is a quick and useful technique for separating pigments and other substances. It is possible to use this technique to separate many different metal cations in a mixture. By comparing the separated species with results for standard solutions of known composition, the cations in the mixture can be identified.

We present here the results of our chromatographic separation of a mixture of cations that possibly contained Ni^{2+} , Cu^{+} or Fe^{3+} .

IV. Experimental Data

A “bullet point” list of the procedure with the key data taken during the lab. Unless there were significant departures from the laboratory manual, this should be very similar to the procedural list provided as part of the Pre-Lab exercise. Data tables and graphs should be used where appropriate.

Only key results should be included. Tare weights and individual buret readings should be omitted. Only final weights or volumes used should be included.

For our first lab exercise, this may look like:

- Prepared eluting solution from 19mL Acetone and 6mL 8M HCl.
- Dotted unknown solution and known solutions onto the chromatography paper.
- Ran the chromatogram.
- Developed the chromatogram with Ammonia vapor and DMG.
- Results were:

Distance Solvent Front Runs = 25.0 cm

<u>Sol'n Used</u>	<u>Color of Spot</u>	<u>Dist. Run [cm]</u>
Fe^{3+}	Rust	24.5
Ni^{2+}	Pink	4.5
Cu^{2+}	Blue-Green	12.0
Unknown	Rust	24.0
	Blue-Green	12.5

V. Data Analysis

An analysis of the data should be provided. This analysis should be performed in a logical order, culminating with the key piece of information being sought.

Data Tables and graphs should be used wherever possible.

All calculations should begin with the formula needed, followed by insertion of the data with appropriate units. The numerical result, using the correct number of significant digits and appropriate units, should then be presented. Intermediate algebraic details should be omitted. Repetitive calculations should be presented only once, with the case being illustrated clearly highlighted.

For example:

$$R_f = \text{Dist. from Origin} / \text{Dist. of Solvent Front} = 24.0 \text{ cm} / 25.0 \text{ cm} = 0.960$$

VI. Conclusion

A brief statement summing up what was learned is provided. This should not exceed three or four sentences. Briefer is better. This should not include statements like “I liked this lab because ...” or “I learned a lot”. The focus should be on the content of the lab exercise.

A brief analysis of the major sources of error should also be provided. Single out the one or two key measurements which were the likely source of error in the final result. For instance, in a density determination, the mass measurement is typically very good, whereas the volume measurement is problematic.

Speculation as to how this experiment might be improved or performed differently is appropriate at this point.

VII. Post Lab Questions

Finally, answer all post lab questions posed in the laboratory manual. Each problem must be worked out fully, with all steps needed to complete the problem shown. Where appropriate, provide needed formulas, followed by insertion of data with appropriate units.