



Analytical Chemistry I Laboratory – Quantitative Analysis – 84.315

Each week, we will meet in Olney 309, the analytical laboratory. We will prepare all the solutions needed in this room, and the experiment may be conducted individually or in pairs. The experiments are designed to take no longer than 4 hours each. Please be prepared before coming to class.

List of Experiments

ACID-BASE TITRATIONS

1. Preparation and Standardization of 0.1 M NaOH and 0.1 M HCl solutions using a primary standard KHP and Phenolphthalein indicator.
2. Determination of equivalence point using pH Titration of KHP solution and 0.1 M NaOH using phenolphthalein Indicator
3. Determination of Acid content in Vinegar by Volumetric and pH titration.
4. Direct versus Back Titration for the determination of Vitamin C in the tablet.
5. Evaluation of Antacid by volumetric titration.

COMPLEXATION TITRATIONS

6. a) Direct titration for the determination of Mg in the sample
b) Determination of Ca by Displacement Titration.
c) Determination of Ca by Back titration
d) Determination of Water Hardness.
7. Evaluation of Commercial dried Milk Powder by Complexation Titration.

PRECIPITATION TITRATIONS

8. Precipitation Titration: Determination of percentage of Cl^- by Mohr's Method.
Precipitation Titration: Determination of percentage of Cl^- in unknown by Gravimetric Method.

REDOX TITRATION

9. a) Preparation and Standardization of KMnO_4
b) Determination of iron in Ore.

Laboratory Safety Rules

Even though every wet chemical analytical experiment to be carried out in the lab has been tested many, many times before and every procedure has been known in details, laboratory safety cannot be overlooked. Accidents do happen from time to time. In order to protect you, your classmates, and the university properties, the following rules will be enforced **at all times**.

1. Be acquainted with the location and use of facilities and familiar with safety precautions and procedures.
2. Students **must** wear adequate eye protection at all times. Contact lenses are **NEVER** permitted to be worn in the laboratory as vapors can collect under the lenses.
3. Familiar with the method of operations and all potential hazards involved before engaging in any lab work. Know the properties, such as flammability, reactivity, corrosiveness, toxicity, etc., of the chemicals you are using.
4. Responsible behavior is required at all times.
5. No eating, drinking, or smoking in the laboratory.
6. Proper clothing (coverage from the shoulders to the knees) must be worn in the laboratory. **No sandals or open-toed shoes are permitted.**
7. Do not fill pipettes by mouth suction.
8. Do not use glassware that has been chipped or broken.
9. Dispose chemicals according to an approved procedure. Do not dump them down the sink.
10. Report all accidents including minor injuries to your TA.
11. Clean everything you used and wash your hands thoroughly before leaving the laboratory.

Lab reports

Reports are due after one week of completion of the experiment and should be handed in at the beginning of the following lab period. Though experiments are to be performed by groups, lab reports and unknowns are to be done on an individual basis. Lab reports should be prepared using a word processor using 8.5" x 11" paper and pages should be numbered. The format should be similar to a journal article (see *Analytical Chemistry*) with some differences in emphasis due to the nature of the course. More instrumental details than one normally finds in journal articles should be included in lab reports. The general format includes following sections:

- **Title Page** including the title of the experiment, your name, and the date on which the experiment were performed.
- **Abstract** summarizing the work done and reporting major results, including numerical results, instrumental technique used, instrument used, and the result of the analysis of the unknown. This is not an introduction or purpose. The abstract is very important and should only be written after your results have been evaluated completely.
- **Introduction** describing the basis for the experiment. In general, present the theory behind the technique utilized. Keep your theory pertinent to the actual measurements taken, use your own words, and use reference where appropriate. Also a block diagram of the instrument to be used should be presented. Cite your source of this diagram correctly and a description of its components and how it works should be provided.
- **Experiment** brief outline of procedure, noting where different than the lab manual: Write in past tense in complete sentences. Follow examples shown in the journal *Analytical Chemistry*. You do not need a great deal of information here; especially avoid presenting step-by-step instructions or directions. Describe equipment used including manufacturer and model, preparation of solutions, etc. Less than one page should almost always be sufficient.
- **Results** including tables of data, graphs or figures, and data analysis. A description of the data presented in tables, figures and calculations should be included to increase clarity of reading.
 - Tables should be numbered consecutively and consist of a row and column format, with a title at the top of the tables. Tables should be designed for ease and clarity of reading.
 - Figures should be numbered consecutively with a title at the bottom of the figure. The title should not just be axes labeled on the figure. The X- and Y-axes should be labeled, including units. All lines should be determined by linear regression. Data points should be made with distinct symbols.
 - Data analysis should include the determination of the concentration of the unknowns and the equation for any linear regression curves that are obtained.

- **Discussion** containing the following:
 - Any observations you make during the experiment,
 - A discussion relating your results to the theory,
 - A brief summary of any sources of error associated with results, and
 - Answers to the numbered questions in the lab manual.
- **References** should be in the style of the current chemical literature. Each reference is numbered according to when it is first used and thereafter is referred to by that number. The references are listed in order at the end of the report.

Grading

Absences from the lab will result in no points for the scheduled experiment. There will be no make-up labs. **5-point** penalty will be exercised for **each** day late. Each lab report counts 10% toward the total, and the total counts 90% of your final grade, your lab notebook counts the other 10%. Grading of the lab report is as following:

Abstract	10 %
Introduction and theory	20 %
Experiment	10 %
Result	20 %
Data analysis (10 %)	
Graphs and tables (10 %)	
Discussion and questions	30 %
References	10 %