ANALYTICAL CHEMISTRY
LABORATORY MANUAL

I hear and I forget.
I see and I remember.
I do and I understand.

Confucius

Middle East Technical University,
Chemistry Department

ANKARA

September 2014
EXPERIMENTS

1. Gravimetry
   - Determination of Sulfate in a Soluble Sample

2. Gravimetry
   - Determination of Nickel in a Soluble Sample

3. Neutralization Titrations
   - Preparation and Standardization of HCl and NaOH
   - Analysis of Weak Acids
   - Selection of Proper Indicator(s)
   - Analysis of Mixtures of Weak and Strong Acids
   - Determination of Acetic Acid in Commercial Vinegar

4. Analysis of Carbonate Mixtures
   - Determination of NaOH, NaHCO₃ and Na₂CO₃ alone
   - Determination of NaHCO₃ and Na₂CO₃ Mixture
   - Determination of NaOH and Na₂CO₃ Mixture
   - Analysis of Unknown Sample

5. Precipitation Titrations
   - Determination of Chloride by Mohr Method
   - Determination of Chloride by Fajans Method
   - Analysis of chloride mixtures (MgCl₂ and NaCl mixture)

6. Oxidation-Reduction Titrations (Permanganometry)
   - Preparation and Standardization of KMnO₄
   - Determination of Iron
   - Determination of Calcium and Hydrogen Peroxide

7. Complex Formation Titrations
   - Determination of Magnesium by Direct Titration
   - Determination of Calcium by Displacement Titration and Back Titration
   - Determination of Water Hardness/Statistical Treatment of Analytical Data

8. Nitrogen Determination by Kjeldahl Method

9. Iodometry
   - Preparation and Standardization of Na₂S₂O₃ Solution
   - Determination of Copper

10. Potentiometric Titrations
    - Standardization HCl
    - Analysis of H₃PO₄ solution

11. UV-VIS Spectrophotometry
    - Determination of Iron

12. Flame Photometry
    - Determination of Sodium and Calcium in tap water

Analytical Chemistry laboratories are performed in the following laboratory: A-104

COURSE OBJECTIVES

This course provides a one semester survey of basic analytical laboratory techniques, instrumental methods and approaches to data analysis used in quantitative analytical chemistry. Students must have completed CHEM 106 before taking this course. By the end of the course the students should be able to (i) understand the proper use of analytical glassware and associated lab equipment, (ii) use statistics to analyze experimental data, (iii) understand the chemical principles behind various analytical methods including gravimetric analysis, titrations (acid-base, precipitation, redox and complexometric), electrochemistry. (iv) understand the basic principles and operation of absorption spectrophotometers.

GENERAL INFORMATION

1) Attendance is compulsory. A medical report is required to have a make-up. Otherwise, no credits will be given for that experiment. Student is not allowed to enter to the laboratory when she/he is late. Make-up will not be given for being late.

2) More than 1 make-up will not be given and the student will fail both from the laboratory and the course. The course grade will be NA.

3) The student will fail both from the laboratory and the course if the overall lab grade is less than 50%. The course grade will be NA.

4) Total grade for your laboratory work will be evaluated based on 100 points. Distribution of these points are;

<table>
<thead>
<tr>
<th>Quiz</th>
<th>25</th>
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<tbody>
<tr>
<td>Report</td>
<td>35</td>
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<tr>
<td>Midterm</td>
<td>15</td>
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<tr>
<td>Final</td>
<td>25</td>
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<td>Total</td>
<td>100</td>
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5) Students must behave in limits of common sense in the lab. A student who does not obey the rules will be asked to leave the lab and no credits will be given for that experiment.

6) No food, drink or smoking is allowed in the laboratories.

7) You have to switch off your mobile phones during the lab. period.
8) The use and care of the following equipment will be illustrated by your laboratory instructors and you are expected to strictly follow these instructions.

- Analytical balances
- Glassware, Pipettes, Burettes, etc.
- Keeping sink environment clean.
- Transfer and care of solutions.

LABORATORY PROGRAM

Each student has two lab periods a week to complete an experiment. Each student should read the necessary parts from the book at home and be ready for the laboratory.

1) Please bring the following items with you.
   - Report of the last experiment (Must be submitted at the beginning of the experiment)
     (No Late Lab Reports will not be accepted!!)
     (Note: Do not ask to your assistants to postpone laboratory reports due to midterms etc.)
   - 3/4 or full length white lab coat must be kept CLEAN and BUTTONED in the lab. Students without lab. coats and goggles will not be allowed in the lab.
   - You have to bring your laboratory manual, water proof pen for writing on the glass. A scientific calculator capable of handling arithmetic and logarithmic functions is required (graphing calculators not required, nor suggested)

2) For each experiment, you are expected to write a summary as homework and take it to the lab at the beginning of the experiment. Summary must include the answers of the pre-lab questions for each experiment. Consider these your “admissions” tickets. These will typically consist of a few calculations or questions that are relevant to the lab that is about to be performed. You will given a ZERO for the experiment if the summary are not completed before starting the experiment.

3) You will take an entrance quiz at the beginning of each lab period. The quizzes will be approximately 10 minutes in length and will cover the pre-lab material, procedure and previous experiment content. A good understanding of the procedure and any calculations necessary for the lab will be needed.
4) Data sheets and calculations will be processed in the lab using a pen. **Summary and data sheet must be signed by the assistants** at the beginning of each laboratory period.

5) Students should prepare the lab. reports by themselves. Otherwise, no credit will be given for that report.

**REPORT FORMAT (Total : 100 points)**

<table>
<thead>
<tr>
<th>POINTS</th>
<th>PART</th>
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| 1. A heading page:  
Title of the experiment  
Name and Surname of the student  
Date of the experiment | 2.5 pts |
| 20 pts | 2. Purpose |
| 2.5 pts | 3. Summary (answer pre-lab questions) |
| 35 pts | 4. Data sheet (signed by the assistant) |
| 40 pts | 5. Graphs and calculations (**Note**: If there are not many calculations and graphs in your experiment, this part will be evaluated together with the discussion part) |
| 40 pts | 6. Discussion and conclusion (**The discussion part must offer include postlab questions with all discussible things in procedure**). (25 pts postlab. questions and 15 pts discussion of experimental results) |
**Academic Integrity**

It is your responsibility to maintain a high degree of integrity in your work. Cheating of any kind will not be tolerated and will result in a failure in the course! The following are considered cheating: (a) Sharing of results and answers on lab reports, graded assignments, quizzes and exams; (b) Use of unauthorized materials during an exam; (c) Plagiarism, including copying a fellow student’s lab report or homework. When in doubt, both parties involved in plagiarism (both the copier and the copyee) will be held responsible for the integrity violation.

**Safety**

The safety of everyone is the highest priority in any laboratory setting. You will be required to attend a lecture where lab safety is discussed (the first pre-lab lecture of the semester) and to sign a lab safety agreement. Please make sure you abide by the following points:

- Personal protective equipment: Safety glasses, lab coats and gloves must be worn at all times whilst working in the lab.
- Dress: Open-toed shoes are forbidden, pants or skirts must reach the ankle, long hair should be tied back, not exposed midsections or torn pants. If you are dressed inappropriately you will be sent home to change!
- All eating and drinking is forbidden in the laboratory. If you need a drink, you may go outside (with the permission of the TA).
- Only experiments authorized by the lab director may be performed in the lab.
- Waste should always be disposed of in the appropriate container. If you are unsure where to pour it, check with the TA.
- Talking on cellphones will not be allowed in lab. If you need to make or answer an urgent call, you should go outside (with the permission of the TA).
- Use of iPODs (or similar devices) is not allowed in lab. Listening through headphones makes you less aware of your surroundings, prevents you from hearing instructions and warnings, and endangers the safety of you and others.
- If you are unsure about any aspect of what you are doing, ask your TA for help!

Failure to heed these instructions, or the instructions of a TA, may result in you being sent home and forfeiting the points from that particular lab exercise. Safety is the most important part of a lab.