

Instructor: Dr. Heather Bullen  
SC 454; 572-5411  
Office Hours: I have an open door  
policy. Formal office hours:  
M, W: 2:00-3:00 pm; T: 9-10 am;  
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**CHE 340L-012**  
**ANALYTICAL CHEMISTRY LABORATORY**  
**FALL 2004**

**COURSE OBJECTIVES:**

In this course laboratory techniques will be refined. Emphasis will be placed on collection, analysis, and interpretation of quantitative data. Students will be able to obtain accurate and precise data and evaluate the reliability of data through error analysis. They will also understand the limitations of the measurements and analysis employed.

**LAB MANUAL:** "Analytical Chemistry Laboratory Manual"; Vinay Kumar and Roger Blanchard; 2003 edition; available at the NKU Bookstore.

**LAB NOTEBOOK:**

A bound notebook of approximately 10" X 7 1/2" is required for submitting weekly reports. For instructions about maintaining the notebook refer to page 1 in the lab manual. A notebook with a spiral binding will not be permitted.

Please read the following information very carefully as your grade will depend upon it.

1. Listed on page 2 are the 12 experiments and the dates on which the results of each analysis are due.
2. Access to the lab (SC 427) will be limited to the scheduled 4 hours and 50 minutes (Thursday 1:00-5:50 pm) per week. No one will be allowed to work outside these hours. Students with excused absence may be given consideration to complete their work at other times.
3. For the instructions concerning the unknown samples, "lateness" in turning in the results and repeating the experiment, see page 3.
4. Lab Grade: Each experiment will be graded on the basis of 100 points. This numerical grade will depend upon the precision, accuracy, and the report write-up (including neatness, grammar, spelling, use of units, and significant figures). Up to 15 points may be deducted for sloppiness in writing the report (in the notebook!) and for not following the proper format. For the lab report format for an experiment refer to pages 1-2 in the lab manual.

The letter grade for the course will be based on the numerical average as follows:

|          |   |   |
|----------|---|---|
| 88 - 100 | A |   |
| 78 - 87  | B |   |
| 68 - 77  |   | C |
| 58 - 67  | D |   |
| <58      | F |   |

### Tentative Lab Schedule Fall 2004

| <u>Exp #</u> | <u>Title of Exp. and Page No.</u>   | <u>Date</u> | <u>Results(Notebook)<br/>Due Dates (gen. Mon.)</u> |
|--------------|---|-------------|--|
| 1            | Check in and Calibration of Pipet (p7)  | 8/26        | 8/30*  |
| 2a           | Soda Ash Detmn. (Indicator Method p9)   | 9/2         | 9/13   |
| 2b           | Soda Ash Detmn. (Potentiometric Method; p12)  | 9/9         | 9/13   |
| 3            | Cation Exchange Determination of K <sup>+</sup> and H <sup>+</sup> ions in a solution (p15) | 9/16        | 9/27   |
| 4            | Determination of Ca and Mg in Water (p19)   | 9/23        | 10/4   |
| 5            | Determination of Iron in iron ore (p23)   | 9/30        | 10/11  |
| 6            | Potentiometric detmn. of iron in FAS (p27)  | 10/7        | 10/18  |
| 7            | Microscale Determination of Ascorbic Acid in Vit. C Tablets (p29)                           | 10/14       | 10/25  |
| 8            | Determination of Mn Spectrophotometrically (p33)  | 10/28       | 11/8   |
| 9**          | HPLC (p47)/AA (p40)/Cu ( p37)/F <sup>-</sup> (p43)  | 11/4        | 11/15  |
| 10**         | AA (p40)/Cu (( p37)/F <sup>-</sup> (p43)/ HPLC (p47)  | 11/11       | 11/22  |
| 11**         | Cu ( p37)/F <sup>-</sup> (p43)/ HPLC (p47)/ AA (p40)  | 11/18       | 11/29  |
| 12**         | F <sup>-</sup> (p43)/ HPLC (p47)/ AA (p40)/ Cu (( p37)                                      | 12/2        | 12/6   |
|              | Open lab (make-up day)/Checkout   | 12/9        | 12/9   |

\* This experiment will not be graded; the report of this and all other experiments (to be written in the notebook) are due in my office, SC 454, by 12:00 pm on the due dates shown above. Note: except Dec. 9, all other due dates are on Mondays.

\*\* For these experiments involving specialized instrumentation, the class will be divided into groups of 2/3 students each. Each group will perform only one of these experiments per week. The report for that experiment will be due on the due date shown above.

**All items on this syllabus are subject to change by the instructor. Students are responsible for reading and understanding all items on this syllabus. Any items not understood must be brought to the attention of the instructor within the first two weeks of class.**

**Honor Code:** All students are expected to honor the NKU Honor Code.

<http://www.nku.edu/~deanstudents/HonorCode.htm>

The instructor reserves the right to determine type of punishment for not honoring this code.

### Policies and Procedures for Analytical Chemistry Laboratory

1. Reporting Results:

Results must be reported to the correct number of significant figures (four in most cases). Please staple a results reporting slip, provided by the instructor, on the last page of the write-up for that determination, allowing the slip to extend a quarter of an inch or so beyond the top edge of the notebook so that the result and unknown number can be easily located.

2. Due Dates:

The lab notebook must be turned in by noon on the date indicated in the lab schedule. A penalty of ten points per week or fraction thereof will be imposed for lateness (*e.g.*, a report that is late by any length of time up to one week receives a ten point penalty, a report that is late by any length of time between one week and two weeks receives a twenty point penalty). A report that is more than two weeks late will be assigned a grade of 60 (out of 100). A student completing an experiment, but never handing in the report will be assigned a grade of 50 for that experiment.

3. Grading and Rejection of a Result:

For each experiment only the reported average value of the results will be graded; however, the student should include all individual values obtained, indicating which, if any, have been omitted in calculating the final "average" value. (The decision to reject a result rests entirely with the student. In no case will the student be told whether his/her results are high or low or what the correct value is. However, at the time of returning the notebooks, the "grading scale" for each experiment will be made available.)

4. Recalculations:

A. Before Grading

If the instructor detects or suspects that a reported value is erroneous due to an error in calculations, he will return the notebook ungraded and marked "Recalculate." The subsequently reported (recalculated) result will be graded with a ten-point penalty for recalculation.

B. After Grading

It is not the responsibility of the instructor to detect calculation errors. Therefore, it is possible that a student may discover a calculation error after the reported value has already been graded, the error having gone undetected by the instructor. In this event the student may turn in a recalculated result on a separate page (to be left blank for this purpose--at the end of each experiment) labeled "Recalculation", along with a clear, concise note explaining where the calculation error occurred, noting the location of the error in the notebook. It is the student's responsibility to convince the instructor that a bonafide calculation error was made and that the student is not simply "second-guessing" to achieve a higher grade. Again, as in case A above, a ten-point penalty for recalculation will be assessed.

5. Repeating Determinations:

A. Before Reporting a Result

If a triplicate determination yields values so widely scattered that the student considers them all unreliable, he/she may repeat determination on the same sample, provided he/she has not turned in the results, and been graded on these results. If an additional portion of the sample is required, this may be obtained at a 10 point penalty from the instructor by submitting the sample vial. The due date for the determination in question remains unaltered, *i.e.*, the result must be turned in by the due date to avoid a 10 point penalty for lateness.

B. After Reporting a Result

If a student receives a low grade on an experiment, he/she may elect to repeat the determination on a different sample. This may be obtained from the instructor. The final grade recorded for the determination will be the grade earned on the repeat analysis minus 10 points. The repeat determination has to be carried out during regular lab hours. No extra time will be allowed. No more than two experiments may be repeated. No penalty for lateness will be imposed on the repeat determination.