Analytical Chemistry (01:160:251) - Fall 2016

Chemistry 251 - Analytical Chemistry
Fall 2016
General Information

Course Coordinator: Professor Asbed Vassilian
Email: asbed@chem.rutgers.edu
Wright Rieman labs Room 126; Tel: 848-445-5879
Stockroom Telephone number: 732-932-9319, 732-932-6160

SCHEDULE: Analytical Chemistry meets once a week for laboratory in lab 005 of the Heldrich Science Building (HSB) (Douglass Chemistry) on the Douglass campus. It also meets once a week for lecture in HSB 106 on Tuesdays (10:55 – 12:15 PM)

Sec 01 Lab meets on Wednesdays from 2:15 PM to 6:55 PM in HSB 005.
Sec 02 Lab meets on Thursdays from 9:15 AM to 1:55 PM in HSB 005.
Sec 03 Lab meets on Thursdays from 2:15 PM to 6:55 PM in HSB 005.
All students meet once a week for lecture on Tuesdays from 10:55 AM to 12:15 PM in HSB 106.

REQUIRED COURSE/LABORATORY MATERIALS

Text: “Exploring Chemical Analysis-Fifth Edition”


Laboratory Notebook: A hardcover or soft cover notebook with numbered pages and tear out carbon-copy duplicate pages is required (Spiral/loose-leaf notebooks are not acceptable) for recording laboratory work. These notebooks are available in the Rutgers University Bookstore.

Safety goggles: To protect your eyes in the laboratory (must be face-fitting and form seal around eyes; ordinary glasses or other types of safety glasses including contact lenses worn under goggles are not acceptable). Goggles are available at bookstores.

Padlock: To be used for equipment drawer.

Scientific calculator: To be used for lab reports, homework problems and exams.

Lab Coat (Recommended): To wear when you are performing the experiments.

CLASSROOM MANAGEMENT SYSTEM (SAKAI): We will be using sakai (URL: http://sakai.rutgers.edu/) as a classroom management system. You should check this site regularly. If you check it now, you may find a number of documents posted. If you are registered and a Rutgers Student, you will automatically be a “member” of the online class. You will need your NetID to login. You should read the documents, especially the Syllabus, Grading Policies, the Announcements and other material very carefully. You will find instructions for the Laboratory Experiments, Pre-Labs, Safety Rules, Syllabus, Acid Washing of Glassware, Announcements, and
other important information. During the course additional documents will be posted on the web. This is where scores will be posted.

If you are having trouble accessing sakai, see the course coordinator after class.

**GRADING:**

**Exams:** 40% - We will give two examinations, each worth 20% of the final grade. The exams will take place during the regular lecture periods.

*A minimum score of about 35% is required on the exams to pass the course.*

**Laboratory Reports** 46% - To complete the course successfully, students must hand in a laboratory report for each experiment the week following its completion at the beginning of the laboratory. **Late lab reports will not be accepted.** With the permission of the lab instructor and course coordinator, only one lab report may be accepted 2 days late with 20% penalty. **Only hard copy (on paper) lab reports will be accepted.** We don’t accept lab reports by email or regular mail.

**Pre-Labs** 4% - Are due at the beginning of the specified labs. **Late pre-labs will not be accepted.** Only **hard copy** (on paper) pre-labs will be accepted. Please don’t e mail pre-labs.

**Lecture/Lab Attendance and Performance** 10% - Instructors will assign grades to students based on attendance/performance in the Lecture and laboratory.

**You need to pass both the lab portion and the theoretical portion to pass the course.**

If you have questions about the grading of lab reports/prelabs/exams etc… please contact us within one week of the day you receive your grade.

**LABORATORY NOTEBOOK:** You should record laboratory data in the specified notebook. At the end of each laboratory period, tear out the carbons, staple them together, and turn them in to your laboratory instructor. In professional laboratories, notebooks often form the evidentiary basis for patent claims and hence have considerable financial and legal importance. While we will not insist that you observe all the formalities that industrial employers require, we ask that you follow a few rules.

* Reserve two or three pages at the beginning for a table of contents.
* Date and sign each page.
* Within reason, write as much explanatory and descriptive text as possible as you go along. You are very likely to find what you have written helpful when you write your laboratory report.
* Erasers and white-outs are not allowed. Don't erase or obliterate erroneous data; simply put a line through them. The notebook does not have to be attractive - although of course it's nice if you can make it so - it just has to be accurate and legible. Don't throw away the pages with mistakes. Much bitter experience shows that sometimes entries thought to be erroneous turn out to be useful in surprising ways.
PURPOSE AND APPROACH: The quantitative analyst seeks to answer the question, "How much of substance (x) is in material (y)? What are the tools used and how can one do the measurements?" In *Analytical Chemistry* you will study some of the important ideas in analytical chemistry and do quantitative measurements. By the end of the course we hope that you will have an improved understanding of:

* the distinction between the goals of qualitative and of quantitative determinations.
* statistical methods for evaluating and interpreting data.
* sources of error in chemical and instrumental analysis.
* interferences in chemical and instrumental analysis.
* the concept of instrument calibration.
* the principles of chromatography and other methods of separation.
* the basic concepts of stoichiometry.
* the importance to quantitation of equilibrium theory.
* the concept of an analytical standard.

If this list seems dry to you, then you may be interested to learn that your sample for analysis during most of the semester will be approximately one gallon of seawater. Crew on a research vessel operated by the Rutgers' Department of Marine and Coastal Studies will have collected the sample shortly before you receive it. We chose seawater for two main reasons. First, the techniques and theory that you will earn for the analysis of seawater are generally applicable, not only to oceanography and the environmental sciences, but also to the various branches of chemistry and biochemistry.

Second, the sample is of interest beyond the classroom. The Department of Marine and Coastal Sciences has a project to monitor the properties of seawater from this location. If all goes well, you should be able to compare some of the results that you obtain in the laboratory with theirs and you may be in a position to contribute to ongoing research. It is our hope that in applying what you learn to samples of environmental and research significance, you will find a greater appreciation of the power of analytical chemistry. The laboratory experiments that you will do and much of the equipment that you will use to do them are new to Rutgers. Sizeable grants from the university and from the National Science Foundation have made it possible to acquire the equipment. We will do our best to see that the experiments work smoothly. As in any new venture, however, we expect to have to improvise along the way. We will need your cooperation, patience, and, perhaps at times, your help. If actual seawater is not available, we will provide artificial seawater.

COURSE POLICIES:

1. **Promptness:** Laboratory session will begin and end promptly as scheduled; students will not be allowed to work overtime or during off-hours. If a student arrives late to the lab, the instructor may not authorize the student to perform the experiment due to safety considerations. In any event, if a student arrives late to lab, he or she cannot stay longer after the lab is officially over. A record of late arrivals is kept.

2. **Lectures:** Attendance in lectures is essential in order for a student to do well in this course. Attendance will be taken and points will be deducted for missed
attendance. Significant amounts of additional material, including modifications to the lab procedure is provided in the lecture. Much of the exam material as well as what is required in the lab report will be provided during the lecture for the course in conjunction with the textbook. Lab instructors will assume that students have attended the lecture and will not provide information that has already been provided in the lecture.

3. **Make-up:** A missed lab period may be made up only for valid medical reason and with a note from the doctor and the dean’s office. The student is responsible for speaking with the stockroom personnel to find an open spot in another lab section. The authorization has to be approved by your lab instructor and course coordinator before a student will be allowed to make up a lab. The authorization will be given only when documentation for the excuse is provided (from a doctor and dean). **The lab reports must be marked clearly with student's name, lab instructor's name and section number and turned in to the lab instructor of the section you are making up the lab.** That lab instructor will pass your lab reports to your lab instructor. This should be done ASAP.

4. **Absences:** For an absence to be excused, valid documentation **MUST** be provided within a week of the absence to your lab instructor and course coordinator. Otherwise, the absence will not be excused. **NOTE:** A student with 3 or more absences (valid or otherwise) will not pass the course. Students are responsible for all the material from a missed lab and will be tested on it in exams.

5. **Weather and Other Emergencies:** Check the Rutgers website ([http://campusstatus.rutgers.edu/](http://campusstatus.rutgers.edu/)) to confirm if classes are cancelled due to weather or other emergencies. If classes are on schedule, then labs will be held as scheduled. If there is a delay in opening, as long as there are 2 hours remaining for the lab period, students should attend lab for the remaining time. Changes in schedule and other adjustments will be announced in the lecture. Students are still responsible for all the material from readings, homework problems and lecture even if a particular lab is cancelled due to weather emergency.

   Campus status information will also be available through:

   - RU-info Channel on RU-TV Channel 3, RU-info Call Center at 732/932-INFO (4636),
   - or via these local media outlets (class cancellations only): WCTC (1450 AM), WRSU (88.7 FM), WRNJ (1510 AM), WXXW (101.5 FM), WCBS (880 AM), WINS (1010 AM), News 12 New Jersey (Channel 12)

6. **Check-out for students is required of all students who complete or withdraw from the course.** Failure to check out will result in the assessment of a $50.00 fee, plus the cost of equipment to be replaced, and the withholding of grade (for those who complete the course).

7. **Laboratory Notebooks** are to be maintained according to the format described in the handout and the textbook.
8. **Written Exams** will be given on the specified dates during lecture period. The material for the exams will come from your lab experiments, pre-labs, homework problems, readings and lecture material. Make sure you do all the homework problems. The homework may be randomly collected in the lecture or the lab period. No notes, whether on paper or electronic, will be permitted on the exam. Scientific calculators are allowed. If you have a cell phone, pager, iPod, or other electronic device visible during the exam, even if it is turned off, you will receive a zero on the test and fail the course. Please bring a watch; you may not use your cell phone as a timekeeper.

9. **Quizzes:** A safety quiz will be given on the first week of lab. Be prepared for pop quizzes.

10. **Preparation:** Adequate preparation before lab will reduce frustration and prevent accidents. Attempting to perform the experiment while reading the procedure for the first time is little more than a waste of time and can lead to hazardous mistakes. Part of the evaluation points assigned by your lab instructor will be determined on the basis of your preparation.

11. **Laboratory Safety:** All persons in the lab must observe the safety rules. Non-compliance with safety rules will result in expulsion from the lab and no make-up will be allowed. It is required to wear safety goggles and lab coats in the lab.

12. **Breakage:** The student is responsible for all costs of breakages. If a student has a balance that needs to be paid when grades are assigned, the student will receive a TF grade (Temporary failure). The TF will be changed to student’s actual grade only after the balance is paid at the cashier and receipt is shown to the Chemistry Department Main Office (WL-142).

13. **Academic Integrity:** You are being graded on the work you perform. If you have previously taken the course, you cannot use the old pre-labs or the lab reports. Use of pre-labs and lab reports from other students (past or present) is expressly forbidden. Both the lender and the borrower are subject to severe penalties. Some discussion about the labs is acceptable at the discretion of the lab instructor, but you must perform all the work (including the data analysis and answering of questions) yourself. The lab instructor is free to ask you at any point to explain what you are doing so that he/she can address any confusion that you may have and assist you. You are **NOT ALLOWED TO COPY ANSWERS FROM OTHER REPORTS OR TO USE DATA FROM OTHER STUDENTS. YOU ARE NOT ALLOWED TO LOAN YOUR LAB REPORTS TO CURRENT OR FUTURE STUDENTS.** Academic honesty also applies to all exams/quizzes/pre-labs/labs and all aspects of this course. Students who suspect that other students are involved in actions of academic dishonesty should speak to the instructor and the course coordinator. A summary of Rutgers policy on Integrity is attached and can also be found at: http://academicintegrity.rutgers.edu/files/documents/AI_Policy_2013.pdf

14. **Laboratory Reports:** You must type or use a computer to print the text of all laboratory reports. We strongly urge you to use spreadsheet program for calculations and for graphing.
15. **Students with Medical Conditions**: Some medical conditions such as pregnancy, asthma, allergies to certain chemicals, or other conditions may be affected by exposure to chemicals. If you believe you are pregnant or if you have a medical problem which might be affected by chemicals, please contact the course coordinator (Dr. Vassilian) before the lab commences or as soon as you become aware of such a condition. We will fully respect your privacy and you do not need to disclose the nature of your medical condition to us. It is, however, imperative that your physician be informed of any chemicals you may be in contact with during the semester so that he/she can determine whether it is safe for you to participate in lab assignments. We will provide you with information for your physician regarding any substances you may be exposed to. We simply require that you bring in a note from your physician indicating that they have reviewed this information and whether you may safely proceed with laboratory work. Rutgers Environmental Health and Safety (REHS) is available to assist you if your doctor recommends that you avoid or minimize contact with certain chemicals. Please feel free to contact them at (732)445-2550 to request assistance.
Lecture Schedule / Homework

<table>
<thead>
<tr>
<th>Date</th>
<th>Event</th>
<th>Chapter</th>
<th>Subject</th>
<th>Problems</th>
</tr>
</thead>
<tbody>
<tr>
<td>9/6</td>
<td>Lecture 1</td>
<td>1-4</td>
<td>Tools of analytical chemistry</td>
<td>1:8,11,12,14,16,18,21,22,24</td>
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<td></td>
<td></td>
<td></td>
<td>Reagents and laboratory</td>
<td>2:1,4,6,9,10</td>
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<td></td>
<td></td>
<td></td>
<td>Equipment</td>
<td>3:12,18</td>
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<td></td>
<td></td>
<td>4:13</td>
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<tr>
<td>9/13</td>
<td>Lecture 2</td>
<td>6</td>
<td>Titrimetry</td>
<td>6:1,10,11,12,13</td>
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<tr>
<td>9/20</td>
<td>Lecture 3</td>
<td>8,9</td>
<td>Acid-base Titration/Buffers</td>
<td>8:1,3,5,6,7,9,11,14,19,21</td>
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<td></td>
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<td></td>
<td>9:2,4,8,10,14</td>
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<tr>
<td>9/27</td>
<td>Lecture 4</td>
<td>10, 11</td>
<td>Acid-base Titration</td>
<td>10:6-9,11,12,13; 11:26</td>
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<td>10/04</td>
<td>Lecture 5</td>
<td>18</td>
<td>Spectroscopy(Colorimetry)</td>
<td>18:2,6,7,11,13,15,17,20,21</td>
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<td>Turbidimetry</td>
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<tr>
<td>10/11</td>
<td>Lecture 6</td>
<td>21 (pp. 455-465) Separation Techniques-</td>
<td>21:1,2,11(a,b)</td>
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<td></td>
<td></td>
<td>22 (pp. 489-505) Chromatography</td>
<td>22:9,12,13</td>
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<tr>
<td>10/18</td>
<td>Lecture 7</td>
<td>7</td>
<td>Gravimetry and Ion- Exchange Chromatography</td>
<td>7:4,7,8,14</td>
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<td>23 (pp. 511-517)</td>
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<td><strong>10/25</strong></td>
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<td></td>
<td><strong>Exam I</strong></td>
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<tr>
<td>11/01</td>
<td>Lecture 8</td>
<td>23</td>
<td>Anion Chromatography Suppressed Ion Chromatography</td>
<td>23: 1,4,8</td>
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<tr>
<td>11/08</td>
<td>Lecture 9</td>
<td>13</td>
<td>Complexometric Titrations</td>
<td>13:1,8,9,13</td>
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<tr>
<td>11/15</td>
<td>Lecture 10</td>
<td>23</td>
<td>Cation Chromatography</td>
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<td></td>
<td>Suppressed Ion Chromatography</td>
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<tr>
<td>11/29</td>
<td>Lecture 11</td>
<td>17</td>
<td>Dissolved Oxygen</td>
<td>17:16</td>
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<td></td>
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<td></td>
<td>Data Synthesis/Cross Comparison</td>
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<tr>
<td>12/6</td>
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<td></td>
<td>Exam II</td>
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</table>
The short report for the Synthesis of Experimental Data is due on the last day of the lab. Note: The draft Alkalinity Laboratory reports will either be full (5-6 pages maximum) or short (1-2 pages maximum) as indicated below. Note: The draft Alkalinity – I & II report is due the week following the completion of Alkalinity II. The short report for the Synthesis of Experimental Data is due on the last day of the lab.

### Laboratory Schedule

Laboratory reports are due one week after the completion date of each laboratory. The completion dates are given on the lines in which the titles are marked with an asterisk (*). Laboratory reports will either be full (5-6 pages maximum) or short (1-2 pages maximum) as indicated below. Note: The draft Alkalinity – I & II report is due the week following the completion of Alkalinity II. The short report for the Synthesis of Experimental Data is due on the last day of the lab.

<table>
<thead>
<tr>
<th>Lab #</th>
<th>Title</th>
<th>Subject</th>
<th>Section 01 Wed</th>
<th>Section 02/03 Th</th>
<th>Report Due Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Check in; Safety Quiz; Integrity Document; Familiarization with Lab</td>
<td>Laboratory Safety – Washing of Glassware: Discussion of Lab Notebook: Practice Volumetric Glassware Practice Weighing</td>
<td>9/07/16</td>
<td>9/08/16</td>
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<tr>
<td>2</td>
<td>Suspended and Dissolved Particulate Matter</td>
<td>Sampling, filtration</td>
<td>9/14/16</td>
<td>9/15/16</td>
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<tr>
<td>4</td>
<td>Alkalinity - II* Spectrophotometric Det of Phosphate (Glassware Prep only)</td>
<td>pH titration – Glassware Acid Wash for Spectrophotometric Det of Phosphate</td>
<td>9/28/16</td>
<td>9/29/16</td>
<td>Typed Draft Alk I &amp; II 10/05 &amp; 10/06</td>
</tr>
<tr>
<td>5</td>
<td>Alkalinity – I and II* (Draft Alkalinity Report Due)</td>
<td>Discussion of Alkalinity Report - Report Format and Calcs</td>
<td>10/05/16</td>
<td>10/06/16</td>
<td>Full 10/12 &amp; 10/13</td>
</tr>
<tr>
<td>6</td>
<td>Colorimetric (Spectrophotometric) Determination of Phosphate*</td>
<td>Anions - I Phosphate</td>
<td>10/12/16</td>
<td>10/13/16</td>
<td>Short 10/19 &amp; 10/20</td>
</tr>
<tr>
<td>7</td>
<td>Turbidimetric Det. of Sulfate*</td>
<td>Anions - II Sulfate (Make arrangements w/ instructor for receipt of reports before exam)</td>
<td>10/19/16</td>
<td>10/20/16</td>
<td>Short 10/26 &amp; 10/27</td>
</tr>
<tr>
<td>8</td>
<td>Gravimetric Analysis of Chloride and Sulfate*</td>
<td>Anions - III Sulfate and Chloride Using Gravimetry and Ion Exchange Chromatography</td>
<td>10/26/16</td>
<td>10/27/16</td>
<td>Full 11/02 &amp; 11/03</td>
</tr>
<tr>
<td>9</td>
<td>Ion chromatography - Anions - I</td>
<td>Anions - IV Halides et al</td>
<td>11/02/16</td>
<td>11/03/16</td>
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<tr>
<td>11</td>
<td>Complexometric titration – Analysis of Ca2+/Mg2+.</td>
<td>EDTA Titration of Calcium/Magnesium</td>
<td>11/16/16</td>
<td>11/17/16</td>
<td>Full 11/30 &amp; 12/01</td>
</tr>
<tr>
<td>12</td>
<td>Ion chromatography – Cations*</td>
<td>Cations</td>
<td>11/30/16</td>
<td>12/01/16</td>
<td>Full 12/07 &amp; 12/08</td>
</tr>
</tbody>
</table>
Material Covered on Exam 1

Understanding and Knowledge of the work involved in the lab and lecture. You need to show the work to get full credit.

1. Laboratory Safety
2. All material covered in the following Pre-Labs:
   LEO-15
   Acid/Base
   Colorimetry

3. All material covered in the following Labs, Lab Procedures, and Lab Reports
   Dissolved and Suspended Solids
   Alkalinity (Indicator titration and pH titration)
   Phosphate (Spectrophotometry)
   Sulfate (Turbidimetry)

4. All material covered in the following Lectures
   Lecture 1 Chemical Measurements, Tools of the Trade, Math, and Error Analysis
   Lecture 2 Titrimetry,
   Lecture 3 Acids Bases, Buffers
   Lecture 4 Acid Base Titrations, Polyprotic Acids and Bases
   Lecture 5 Spectroscopy, Turbidimetry

5. Concepts and Understanding of Assigned Homework Problems or Similar Problems, Review Problems, etc… Refer to the chapters in your textbook.
   Chapters 1,2,3,4,6,8,9,10,11,18
Material Covered on Exam 2

Understanding and Knowledge of the work involved in the lab and lecture. You need to show the work to get full credit.

1. Laboratory Safety
2. Basic Concepts Covered on the first exam
3. All material covered in the following Labs, Lab Procedures, and Lab Reports

Gravimetric Analysis of Chloride and Sulfate
Ion Chromatography - Anions
Complexometric Titration
Ion Chromatography - Cations
Measurement of Dissolved Oxygen
Synthesis of Experimental Data

4. All material covered in the following Lectures
   Lecture 6 Separation Techniques, Chromatography
   Lecture 7 Gravimetry and Ion Exchange Chromatography
   Lecture 8 Anion Chromatography – Suppressed Ion Chromatography
   Lecture 9 Complexometric Titrations
   Lecture 10 Cation Chromatography – Suppressed Ion Chromatography
   Lecture 11 Dissolved Oxygen – Data Synthesis

5. Concepts and Understanding of Assigned Homework Problems or Similar Problems, Review Problems, etc… Refer to the chapters in your textbook.
   Chapters 22,7,13,17
POLICY ON ACADEMIC INTEGRITY SUMMARY

"Academic freedom is a fundamental right in any institution of higher learning. Honesty and integrity are necessary preconditions to this freedom. Academic integrity requires that all academic work be wholly the product of an identified individual or individuals. Joint efforts are legitimate only when the assistance of others is explicitly acknowledged. Ethical conduct is the obligation of every member of the university community and breaches of academic integrity constitute serious offenses" (Academic Integrity Policy, p. 1).

The principles of academic integrity entail simple standards of honesty and truth. Each member of the university has a responsibility to uphold the standards of the community and to take action when others violate them. Faculty members have an obligation to educate students to the standards of academic integrity and to report violations of these standards to the appropriate deans.

Students are responsible for knowing what the standards are and for adhering to them. Students also should bring any violations of which they are aware to the attention of their instructors.

Violations of Academic Integrity

Any involvement with cheating, the fabrication or invention of information used in an academic exercise, plagiarism, facilitating academic dishonesty, or denying others access to information or material may result in disciplinary action being taken at either the college or university level. Breaches of academic integrity can result in serious consequences ranging from reprimand to expulsion.

Violations of academic integrity are classified into four categories based on the level of seriousness of the behaviors. Brief descriptions are provided below. This is a general description and is not to be considered as all-inclusive.

Level One Violations

These violations may occur because of ignorance or inexperience on the part of the person(s) committing the violation and ordinarily involve a very minor portion of the course work. These violations are considered academic merit and not as disciplinary offenses.

Examples: Improper footnoting or unauthorized assistance on academic work.

Recommended Sanctions: Makeup assignment.

Level Two Violations

Level two violations involve incidents of a more serious nature and affect a more significant aspect or portion of the course.

Examples: Quoting directly or paraphrasing without proper acknowledgment on a moderate portion of the assignment; failure to acknowledge all sources of information and contributors who helped with an assignment.

Recommended Sanctions: Probation, a failing grade on the assignment, or a failing grade in the course.

Level Three Violations

Level three offenses involve dishonesty on a significant portion of course work, such as a major paper, an hourly, or a final examination. Violations that are premeditated or involve repeat offenses of level one or level two are considered level three violations.

Examples: Copying from or giving others assistance on an hourly or a final examination, plagiarizing major portions of an assignment, using forbidden material on an hourly or final examination, using a purchased term paper, presenting the work of another as one's own, altering a graded examination for the purposes of regrading.

Recommended Sanctions: Suspension from the university for one or more terms, with a notation of "academic disciplinary suspension" placed on a student's transcript for the period of suspension, and a failing grade in the course.

Level Four Violations

Level four violations are the most serious breaches of academic integrity. They include repeat offenses of level three violations.

Examples: Forging of grade change forms, theft of examinations, having a substitute take an examination, dishonesty relating to senior thesis, master's thesis, or doctoral dissertation, sabotaging another's work, the violation of the ethical code of a profession, or all infractions committed after return from suspension for a previous violation.

Recommended Sanctions: Expulsion from the university and a permanent notation on the student's transcript.

Faculty who believe that violations have occurred should immediately contact the Office of the Dean. Students who suspect that other students are involved in actions of academic dishonesty should speak to the instructor of the course. Questions on reporting procedures may be directed to the Office of the Dean.