

Chemistry 341/Analytical Techniques/ Fall 2011

LECTURES: MW 10:00-10:50, Room: Currens 202,

Lab: T:section 21: 8:00-10:50, section 22: 11-1:50, section 23: R: 2:00-4:50 all are in Currens 331

INSTRUCTOR: Dr. Tarab Ahmad

OFFICE: Currens 324B office phone: 309-298-1656 , e-mail: TJ-Ahmad@wiu.edu

OFFICE HOURS: MWF, 12:00-1:30 or by an appointment

PREREQUISITE: Chem 202 (Inorganic chemistry II)

TEXTBOOK

- 1) Quantitative chemical analysis 8th Edition, Daniel Harris, Freeman hard book or e-book. The hard book is available at the bookstore; the e-copy can be purchased from the publisher website for approximately half the price of the hard copy (new: \$165.95)
- 2) The textbook student website :www.whfreeman.com/qca8e, at this website you will find lists of experiments from journal of chemical education, downloadable Excel sheets and living Java applets that allow the student to manipulate graphs by altering data points and variables. Supplementary topics include spreadsheets and supplementary problems and their solutions. I encourage you to visit the site for sample online quizzes "Chemical Analysis on the Web",
- 3) If you choose to buy the e-book, this is the link for the e-book:

<http://ebooks.bfwpub.com/qca8e.php>

OTHER EXPENSES

SAFETY GLASSES: \$5

Lab book ~\$18, alternatively you can use composition notebook (\$1-2).

ESTIMATED TIME TO STUDY THE COURSE: 6-8 hr/week

OTHER USEFUL RESOURCES

- 1) Fundamentals of Analytical Chemistry, 8th Edition, Skoog, West, Holler, and Crouch
- 2) Analytical chemistry (ACS):e-journal /library
- 3) National institute of standards and technology NIST website
- 4) J. Chromatography A
- 5) LCGC north America, www.chromatographyonline.com
- 6) Spectroscopy magazine
- 7) Analytical abstracts e-journals/library

8) J.chemical education

OVERVIEW OF SCHEDULE AND COVERAGE.

(Note:Chaps.1&2 your responsibility, read chapters 1 and 2 before you come to the first lab)

Section I: Tools for analytical chemistry

Section II: *Aqueous Equilibria, Titrimetric methods, Acid base and Complexometric Titrations*

Section III: *Redox and Electrochemical methods*

Section IV: *Spectrochemical Methods*

Section V: *Separations*

Tentative Content	Course	Approximate Daily Schedule	Tests, HWs
<i>Tools for analytical Chem</i> Chapter 0, 3, 4, 5		August 22 August 22, 24, 29,31 Sept 7, 12, 14	HW1, HW2
<i>Aqueous Equilibria</i> Chapter 6-8		Sep 19, 21, 26, 28	Sep5:LaborDay/No Classes HW2 HW3 TEST I Sept 21
<i>Acid-base, Complexometric Titrations</i> Chapter 10-13		Oct 3, 5, 10, 12,19	HW4 HW5 TEST II Oct 12
<i>Redox& Electrochemistry</i> Chapter 14-17		Oct 24, 26, 31 Nov 2, 7	HW6 HW7 TEST III Nov 7
<i>Spectroscopy</i> Chapters 20, 21		Nov 14, 16, 28	HW8
<i>Separations</i> Chapters 23-26		Nov 30, Dec 5, 7	HW9 HW10 Nov21-25 No classes Test IV Dec 7
<i>Gravimetric analysis and precipitation titrations</i>		Will be covered in the lab in two experiments	
Monday, Dec 12 Comprehensive FINAL EXAM, 10:00^{am}-12:00			

GRADING: There will be four exams (**300 points**) and one exam will be dropped. There will be a final exam (**100 points**) which is an **analytical ACS exam** and it will cover all the material given in chem. 341. There will be 5-10 take home assignments (HW) at the end of each chapter (**100 points**) and **7-10 POP QUIZZES ON MONDAYS (100 points)**. Few quizzes and few HW assignments will be dropped from the total grade for quizzes and assignments. The total grade for the tests, HW and quizzes without the lab grades is **600 points**. Your Final Examination and your quizzes scores **MAY NOT BE DROPPED** and must be included in your final point total.

Twelve laboratory exercises are planned for this semester. Only your ten best laboratory exercises will be counted toward your final grade. The labs will be worth a total of **400 points**. You must score a minimum of 78 % or 312 points in the laboratory exercises **to pass** Chem341.

Activity	
Best 3 of 4 Exams	300
Assignments(best 8)	100
Quizzes (best of 5 quizzes)	100
Labs	400
Final (ACS)Exam	100
Total	1000

COURSE GRADING SCALE

The following scale will be used to assign an overall grade for this class:

<u>%</u>	Grade
93-100%	A
88 - 92%	A-
85 - 87%	B+
82 - 84%	B
78- 81%	B-
75-77	C+
72-74%	C
68-71%	C-
65-67	D+
62-64	D
58-61	D-

TEST POLICY

In order to accommodate illness and /or other excused absences the comprehensive make up exam will be given at the end of the semester. You may use this to make up one, and only one, missed examination.–The time and date for the make-up exam for all missed exams will be announced in November. The make-up exam will be comprehensive over all materials covered in chem. 341. Students who miss more than one exam should drop the course. No make-up quizzes, home work, or laboratory exercises will be given. Your two lowest lab grade will be dropped and a few low quiz scores will be dropped. Missing more than one exam ; having fewer than the required number of quizzes or home work assignments; or missing three laboratory exercises will ensure a zero will be incorporated into your final grade.

DO NOT MISS MORE THAN THE ALLOWED NUMBER

Exceptions to the above rules will only be made for University sponsored functions that require your presence during the class period (i.e. WIU band trips, field trips in other WIU classes, WIU athletics, etc.), and military commitment. Notification of the official WIU sponsored function, in the form of a signed memo from the faculty sponsor or coach, must be submitted to the instructor at least 7 days prior to the expected absence. Evidence of military commitment should be presented as soon as possible.

LECTURE NOTES , QUIZZES, HOMEWORK

All materials related to the course including lecture notes, homeworks will be posted on the [western online](#)

LAB POLICY

The lab syllabus will be separate from the course syllabus. For lab instructions read the information posted on westernonline in the lab folder. Information will be posted weekly.

ATTENDANCE POLICY: You are expected to attend class regularly and punctually. You are responsible for all information and materials in sessions and classes whether you are present or not. Any student who receives six or more absences may be dropped from this course without notice. Any student arriving more than ten minutes late for any exam, laboratory exercise, or the final examination may not be allowed to take or complete the assigned work at the instructor's discretion. Any student attempting to turn in a home work assignment more than ten minutes late may not be allowed to turn in the assigned work at the instructor's discretion. Leaving the classroom prior to completion of a quiz or an exam is prohibited.

STATEMENT ON ETHICS

Western Illinois University, like all communities, functions best when its members treat one another with honesty, fairness, respect, and trust. Students have rights and responsibilities (<http://www.wiu.edu/provost/student/>). Plagiarism, cheating, and other forms of academic dishonesty constitute a serious violation of University conduct

regulations. Students who engage in dishonesty in any form shall be charged with academic dishonesty. Please remember that you are expected to do your own work at all times.

STUDENTS WITH DISABILITIES

It is the policy of Western Illinois University to accommodate individuals with disabilities pursuant to federal law and the University's commitment to equal educational opportunities. It is the responsibility of the student to inform the instructor of any necessary accommodations at the beginning of the course. Any student requiring accommodations should contact the Office of Special Student Service

EMERGENCY PREPAREDNESS: WIU Office of Risk Management and Emergency Preparedness provides resources on how to respond to emergency situations. Please view the video resources at www.wiu.edu/rmep/ (Click "Resources" on the right side of the page).

The following action is prohibited under the Student Conduct Code:

DISORDERLY CONDUCT: Any behavior which disrupts the regular or normal functions of the University community, including behavior which breaches the peace or violates the rights of others. (<http://sjp.wiu.edu/CodeOfConduct/index.asp>)

-Simple scientific calculators without extensive memory functions (i.e. Texas Instruments model 30) may be used.

- All hats, caps, and hoods that cover your ears are not allowed to be worn during a quiz or exam.

-Assigned seating, rearrangement of seats, and closing of selected seats and areas of the classroom are the option of the instructor.

STUDENT RIGHTS AND RESPONSIBILITIES: Student rights and responsibilities are listed on the WIU website (<http://www.wiu.edu/provost/students/>).

Detailed Syllabus

Ch0: The Analytical Process
self reading

The Analytical Chemist's Job, General Steps in a Chemical Analysis, quantitative analysis, qualitative analysis, calibration curve, heterogeneous, homogeneous, sampling, sample preparation, standard solution

Ch1: Measurements

SI Units, conversion between units, mole, volume, Chemical Concentrations, Molarity and Molality, formal concentration, electrolytes, ppm, ppb, solute, solvent, volume percent, weight percent. Titrations, equivalence point, Reagent Chemicals and Primary Standards

Ch2: Tools of trade

read before coming to

lab1

Safe handling of chemicals, disposal of chemical waste, lab book, analytical balance, burets, volumetric flasks, filtration, drying, calibration of volumetric glassware, Microsoft excel, graphing with excel.

Ch3: Experimental error

Significant figures, Significant figures math, types of error, systematic errors, Random errors, precision and accuracy, standard reference material, absolute and relative uncertainty, Propagation of Uncertainty from Random Error.

Ch4: Statistics

Gaussian Distribution, Mean Value and Standard Deviation, Significant Figures in Mean and Standard Deviation, Standard Deviation and Probability, Confidence Intervals, Calculating Confidence Intervals, degrees of freedom, dynamic range, linear range, blank solution, Comparison of Means with Student's t , Case I, II and III., F test, Q test, least square method, calibration curves, variance.

Ch 5: Quality assurance and calibration methods

Assessment, calibration check, coefficient of variation, control chart, detection limit, false negative, • false positive, field blank, internal standard, linearity, lower limit of quantitation, matrix effect, method blank,, method validation, quality assurance,

range, reagent blank, response factor, robustness, selectivity, sensitivity, specification, specificity, spike, standard addition, standard operating procedure.

Ch6 : Chemical equilibrium

Acid, acid dissociation constant(K_a), acidic solution, amine, ammonium ion, aprotic solvent, autoprotolysis, base, base hydrolysis constant, basic solution, Brønsted-Lowry Acids and Bases, salts, conjugate acids and bases, antire of H^+ , Autoprotolysis, carboxylate anion, carboxylic acid, common ion effect, complex ion, conjugate acid – base pair, coprecipitation, endothermic, exothermic, entropy, enthalpy change, Gibbs free energy, hydronium ion, ion pair, Lewis acid, Lewis base, ligand, neutralization, overall formation constant, solubility product, standard state, stepwise formation constant, saturated solution, salt, reaction quotient, protic solvent, polyprotic solvent, polyprotic base, pH.

Ch7: Activity and the Systematic Treatment of Equilibrium

The Effect of Ionic Strength on Solubility of Salts, The Effect of Ionic Strength on Solubility of Salts
Ionic strength, activity coefficient, Systematic Treatment of Equilibrium.

Ch8: Monoprotic Acid-Base Equilibria

Strong acids and bases, Weak Acids and Bases, weak acid equilibria, Buffers, Henderson-Hasselbalch Equation, preparing buffers, buffer capacity, fraction of dissociation of acids and bases.

Ch9: Polyprotic Acid-Base Equilibria

Diprotic Acids and Bases, Diprotic Buffers 10-3 Polyprotic Acids and Bases 10-4 Which Is the Principal Species? 10-5 Fractional Composition Equations, Isoelectric and Isoionic pH, Isoelectric Focusing, Zwitterion, hydrolysis, amino acid.

Ch10 :Acid-Base Titrations

Titration of Strong Base with Strong Acid, Titration of Weak Acid with Strong Base, Titration of Weak Base with Strong Acid, Titrations in Diprotic Systems, Finding the End Point with a pH Electrode, Finding the End Point with Indicators, Gran plot, indicator error, leveling effect, transition range.

Ch11: E DTA Titrations

Metal-Chelate Complexes EDTA properties, complexes, Conditional Formation Constant, EDTA Titration Curves, Metal Ion Indicators, EDTA Titration Techniques, direct and back titrations, masking.

Ch13: Fundamentals of Electrochemistry

Galvanic Cells, Standard Potentials, Nernst Equation, E° and the Equilibrium Constant, Cells as Chemical Probes

Ch14: Electrodes and potentiometry

Reference electrode, indicator electrode, junction potential, ion selective electrodes, sensors.

Ch15: Redox Titration

-1 The Shape of a Redox Titration Curve, Finding the End Point, Oxidation with Potassium Permanganate,

Redox titrations Oxidation with Ce^{4+} , Oxidation with Potassium Dichromate, Methods Involving Iodine.

Ch16: Electroanalytical techniques

Fundamentals of electrolysis, electrogravimetric analysis, Coulometry, amperometry, oxygen sensors, voltametry, Karl Fisher titration of H_2O .

Ch17: Fundamental of Spectrophotometry

Properties of light, absorption of light, measuring absorbance, Beer's law, Luminescence.

Ch20: Atomic spectroscopy

Mercury analysis by cold vapor atomic fluorescence, Flames, furnaces, plasmas, instrumentation.

Ch21: Mass spectrometry

Electron ionization, molecular mass and nominal mass, resolving power, molecular ion and mass patterns, identifying the molecular ion peak, interpreting fragmentation pattern, quadrupole MS, time of flight MS, Quadrupole Ion-Trap Mass Spectrometer, Chromatography–Mass Spectrometry, Electrospray, Atmospheric Pressure Chemical Ionization

Ch22: Introduction to Analytical Separations

Extractions, Types of chromatography,plumber view of chromatography, scaling up. Retention time, hold up time, partition coefficient, efficiency of separation, resolution, diffusion, plate height, broadening, van Deemter equation.

Ch23: Gas chromatography

The separation process, Open tubular column, packed columns, capillary columns, carrier gas, injectors, split less injection, split injection, detectors (TCD, FID, ECD, other detectors) retention index, Temperature and Pressure Programming, GCMS.

Ch24: High-Performance Liquid Chromatography

The Chromatographic Process, particle size and efficiency, the column,the stationary phase, the elution process, Isocratic and Gradient Elution, Selecting the Separation Mode, Solvents, Bandshapes, Pumps and Injection Valves, Spectrophotometric Detectors, RI detector, ELs,electrochemical, optimization of the separation, solven optimization, tempearture as a varaibale, computer optimization, gradient separations

Ch 25: Chromatography-electrophoresis

Capillary Electrochromatography, Ion-Exchange Chromatography, Ion Chromatography, Molecular Exclusion Chromatography, Affinity Chromatography, Principles of Capillary Electrophoresis, Micellar Electrokinetic Chromatography, capillary electrophoresis, Capillary Gel Electrophoresis.

Ch 26: Gravimetric Analysis, Precipitation titrations and Combustion analysis

Examples, precipitation, crystal growth, homogenous precipitation, effect of electrolytes, digestion, co precipitation, gravimetric calculations, combustion analysis.

The Precipitation Titration Curve, Titration of a Mixture, Calculating Titration Curves with a Spreadsheet, End-Point Detection, Efficiency in Experimental Design, adsorption indicator, Argentometric titration, back titration, blank titration, co-precipitation, direct precipitation, end point, equivalence point, Fajan's titration, Gravimetric titration, indicator, Kjeldahl nitrogen analysis, Primary standard, Reagent-grade chemical, Standardization, Primary standard, titrant, titration error, Trace analysis, Volhard titration, volumetric analysis.