

Brazosport College
Syllabus for CTEC 1441: Instrumental Analysis I

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COURSE DESCRIPTION

Knowledge of procedures, equipment, and techniques used in industrial laboratories will be gained by students through the performance of laboratory experiments and writing of laboratory reports. Students will perform calculations, prepare standard solutions, analyze unknown samples, and operate various analytical instruments. Topics to be covered include sampling and sample preparation, gravimetric and titrimetric analysis, introduction to instrumental analysis, analytical separations, gas chromatography, and electrochemical methods.

PREREQUISITES

CHEM 1411 or CHEM 1405.

COURSE GOALS

At the completion of CTEC 1441 the student should be able to:

1. Understand and apply basic statistical methods used in analytical labs.
2. Perform gravimetric analysis calculations and analyze unknowns using gravimetric techniques.
3. Perform volumetric calculations necessary in the preparation of standard solutions.
4. Apply and understand standardization methods used in titrimetric analysis, such as the use of primary standards, indicators, and the titration curve.
5. Understand the various methods of analytical separation, such as distillation, extraction and chromatography.
6. Understand the theory and operation of the gas chromatograph.
7. Receive a grade of D or better in the laboratory portion of the course.

TEXTBOOK OR COURSE MATERIAL INFORMATION

1. Kenkel, John, Analytical Chemistry for Technicians, 3rd Edition, Published by Lewis Publishers, 2003.
2. Organic Chemistry Lab Notebook, Published by Signature Labs.
3. Scientific Calculator.

LAB REQUIREMENTS

1. Visorgogs or safety goggles, must meet ANSI Z87.1-1989 certification.

STUDENTS WITH DISABILITIES

Brazosport College is committed to providing equal education opportunities to every student. Brazosport College offers services for individuals with special needs and capabilities including counseling, tutoring, equipment, and software to assist students with special needs. Please contact the Special Populations Counselor, 979-230-3236, for further information.

ACADEMIC HONESTY

Brazosport College assumes that students eligible to perform on the college level are familiar with the ordinary rules governing proper conduct including academic honesty. The principle of academic honesty is that all work presented by you is yours alone. Academic dishonesty including, but not limited to, cheating, plagiarism, and collusion shall be treated appropriately. Please refer to the Brazosport College Student Guide for more information; this is available online at <http://www.brazosport.edu/Web%20Part%20Pages/Sched.aspx>.

ATTENDANCE AND WITHDRAWAL POLICIES

Class attendance is not graded, but you must attend class to successfully complete the course. If you are unable to complete this course, you must complete and submit a withdrawal form with the registrar. If you stop attending class and do not withdraw, you will receive a performance grade, usually an "F".

COURSE REQUIREMENTS AND GRADING POLICY

For this class you complete the following:

Exams: There will be a total of four exams. Each exam will last approximately one hour during class. The exact date of each Exam will be announced in class prior to the actual date of the exam. Students are allowed to bring a one-page, hand written notes containing equations, etc., to the exams.

Homework: As assigned by the instructor. A maximum homework grade of 10 points will be awarded as bonus points to the exam for homework assignments handed in on time. Due date for homework is the day of the exam. **All work must be shown to obtain full credit for the homework assignments.**

Lab: The laboratory portion of the course consists of weekly 3 hour labs which the student must attend. **To pass the course, the student must successfully complete the laboratory experiments with a grade of D or better.**

Final Exam: The final will be given at the end of the course. The final exam is comprehensive.

Each of the above requirements counts toward your final grade as follows:

Exams	40%
Attendance	5%
Lab	40%
Final	15%

TESTING

See the class calendar for the chapters and dates of the tests. Students are allowed to bring one page of hand written notes, containing equations, etc., to the exams. The material to be covered on each exam is as follows:

<u>Exam</u>	<u>Chapters</u>
1	Intro to Analytical Science, Sampling, Gravimetric Analysis
2	Intro to Instrumental Analysis, Analytical Separations, GC
3	Titrimetric Analysis, Acid-Base Titrations
4	Complex Ion Formation, Redox Reactions and Titrations
Final	Comprehensive Exam

MAKE-UP POLICY

There will be no make-up exams. The lowest exam grade will be replaced by the final exam grade, if higher. The final exam grade will replace **one** missed exam grade.

STUDENT RESPONSIBILITIES

Students are expected to fully participate in this course. The following criteria are intended to assist you in being successful in this course:

1. understand the syllabus requirements
2. use appropriate time management skills
3. communicate with the instructor
4. complete course work on time, and
5. utilize online components (such as Desire2Learn) as required.

PROJECTS, ASSIGNMENTS, PORTFOLIOS, SERVICE LEARNING, INTERNSHIPS, ETC.

None.

OTHER STUDENT SERVICES INFORMATION

Information about the Library is available at

www.brazosport.edu/sites/CurrentStudents/Library/default.aspx or by calling 979-230-3310.

Information about study skills and tutoring for math, reading, writing, biology, chemistry, and other subjects is available in the Learning Assistance Center (LAC); see

www.brazosport.edu/sites/CurrentStudents/LAC/default.aspx or call 979-230-3253.

To contact the Physical Sciences and Process Technology Department call 979-230-3427.

The Student Services provides assistance in the following:

Counseling and Advising	979-230-3040
Financial Aid	979-230-3294
Student Activities	979-230-3355

To reach the Information Technology Department for computer, email, or other technical assistance call the Helpdesk at 979-230-3266.

CTEC 1441 – Fall 2011 – Tentative Schedule *

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WEEK of	LECTURE	LAB
	TT 5:35 – 6:50 PM	Tuesday 7 – 9:50 PM
8/29	Chapter 1 – Introduction to Analytical Science	Safety and check-in. Exp. 1 – Excel Spreadsheet
9/5	Chapter 2 – Sampling Chapter 3 – Gravimetric Analysis	Exp. 2 – Gravimetric Sulfate
9/12	Chapter 3 – Gravimetric Analysis Quiz 1	Exp. 2 – Gravimetric Sulfate
9/19	Chapter 6 – Introduction to Instrumental Analysis	Exp 3a – GC of Beer
9/26	Chapter 11 – Analytical Separations	Exp 3a – GC of Beer
10/3	Chapter 11 – Analytical Separations Chapter 12 – Gas Chromatography	Exp. 3b- GC of Gasoline
10/10	Chapter 12 – Gas Chromatography Quiz 2	Exp. 3b- GC of Gasoline
10/17	Chapter 4– Introduction to Titrimetric Analysis	Exp. 4 – Esterification (Synthesis)
10/24	Chapter 4– Introduction to Titrimetric Analysis	Exp. 4 – Esterification (Extraction and Analysis)
10/31	Chapter 4– Introduction to Titrimetric Analysis Chapter 5 – Applications of Titrimetric Analysis	Exp. 5a – Titration (Standardization of NaOH) Exp. 5b – Standardization of HCl
11/7	Chapter 5.2 – Acid –Base Titrations Quiz 3	Exp. 5c – KHP unknown
11/14	Chapter 5 – (5.3 – Complex Ion Formation Reactions)	Exp. 5d – Soda Ash unknown
11/21	Thanksgiving Holiday	
11/28	Chapter 5 – (5.4 – Oxidation-Reduction Reactions)	Exp. 6 – Water Hardness
12/5	Quiz 4 Review for Final	Clean up
12/12	Final – Tuesday Dec. 13, 5-7 PM	

*This schedule is subject to change.

**CTEC 1441 – Applied Instrumental Analysis
Fall 2011**

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Homework Assignments: A maximum homework grade of 10 points will be assigned to completed homework assignments handed in on time (prior to the start of the exam). These points will be added to the corresponding exam grade as bonus points.

For Quiz 1: Due before Quiz 1

1 (1, 4, 6, 8, 20, 22, 24, 27, 28, 30, 31)
2 (3, 5, 18)
3 (10, 13, 16, 19, 23, 25, 27, 29, 37, 39)

For Quiz 2: Due before Quiz 2

6 (6, 9, 23, 25, 27, 28, 29, 39, 45, 47, 51)
11 (7, 9, 11, 14, 16, 24, 27, 28, 39, 41, 49, 50)
12 (1, 6, 10, 11, 13, 17, 26, 29, 31, 43, 45)

For Quiz 3: Due before Quiz 3

4 (5, 7, 9, 11, 12, 19, 22, 32, 33, 43)
5 (7, 10, 11, 13, 18, 27)

For Quiz 4: Due before Quiz 4

5 (37, 40, 41, 46, 50, 54a, 54g, 55a)
5 (57, 66, 68[q-u], 75)

CTEC 1441 Lab – Fall 2011

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Experiments for Applied Instrumental Analysis Lab

- 1) Graphing using Excel spreadsheet. (Handout)
- 2) The Gravimetric Determination of Sulfate in a Commercial Unknown (Kenkel, page 57)
- 3) GC - Gas Chromatography
 - a) Experiment 42: Determination of Ethanol in Wine (or Beer) by Gas Chromatography and the Internal Standard Method. (Kenkel, page 359, and Handout)
 - b) Experiment 45: The Gas Chromatography Determination of a Gasoline Component by the Method of Standard Additions and the Internal Standard Method. (Kenkel, page 361, and Handout)
- 4) Esterification Pilot Process
 - a) Synthesis of methyl and isopropyl benzoate (Handout)
 - b) Determination of the relative yields of methyl and isopropyl benzoate using the GC (Handout)
- 5) Titrations Using the pH Probe
 - a) Experiment 8: Standardization of NaOH solution against Potassium Phthalate. (Kenkel, page 92, and Handout)
 - b) Experiment 8: Standardization of HCl solution against NaOH. (Kenkel, page 92, and Handout)
 - c) Experiment 10: Titrimetric Analysis of a Commercial KHP Unknown for KHP. (Kenkel, page 135, and Handout)
 - d) Experiment 11: Titrimetric Analysis of a commercial Soda Ash Unknown for Sodium Carbonate. (Kenkel, page 135, and Handout)
- 6) EDTA Titration
 - a) Experiment 14: Determination of Water Hardness (Kenkel, page 138)

Applied Instrumental Analysis Lab

Safety:

1. Safety goggles must be worn at all times in the laboratory.
2. Know the locations of eyewashes, showers, fire extinguishers and exits.
3. Use common sense.
4. Bare feet are NOT allowed into the laboratory. Open sandals and shoes are discouraged.

Laboratory Housekeeping:

1. Arrange apparatus neatly and compactly. Keep all books except the laboratory manual and laboratory notebook off the laboratory workbench.
2. Do not throw paper or solid materials into the water troughs or sinks.
3. Keep all reagent bottles clean (especially acids and bases).
4. Keep the lab bench area clean. Pay particular attention to keeping the balances clean and in order. If you spill chemicals, clean them up immediately. Put caps back on reagent containers.
5. At the end of the laboratory period, clean off your workspace with a sponge or wet paper towel. Perform proper shutdown of the instruments. Check to see that the gas and water have been turned off. You are responsible for keeping the area neat. Repeated failure to do so may result in loss of credit.

CLEAN UP AND INSTRUMENT SHUTDOWN STARTS 10 MINUTES BEFORE THE OFFICIAL END OF THE CLASS PERIOD.

When the time is up, you are supposed to be out of the laboratory. Failure to properly budget your time is presumptive of poor planning and your grade may suffer.

Grading:

1. Come prepared to the lab. The introduction (objective, discussion), safety, material and apparatus, should be written in the Laboratory Notebook prior to the beginning of the experiment. The format for the lab notebook is described in detail in the handout (Report Format for CTEC 1441). This pre-lab write-up (30 points) must be checked and initialed by me prior to the start of each experiment.
2. Experimental data and observations must be recorded in the Laboratory Notebook. The data sheets and observations (20 points) must be completely filled out in ink and initialed by me before you leave the lab. When you make an error, cross it out with a single line. Do not use liquid paper or obliterate the error. For example: ~~error~~ error
3. After a lab is finished, write the date that you finished the lab on the lab sign-up sheet. This signifies that your lab report will be due one week from that date.
4. Analysis of the data, calculations, including any tables and graphs, and summaries and conclusions (50 points) are due one week after the conclusion of the experiment.
5. Carbon copies of the complete experimental write-up are due the week after you finish the experiment.
6. Grading will be based on completeness of the experiments and the submitted report write-up.

REPORT FORMAT for CTEC 1441

- I. Introduction
 - A. Objective
 - B. Discussion
 - I. Theory of operation performed (what happens in the analysis to obtain results).
 - II. Brief write-up of procedure.
 - III. Reactions if any.
 - IV. Method used to calculate or to obtain results.
- II. Safety precautions involved.
- III. Materials and Apparatus
 - A. Reagents used. Equipment used (glassware etc.) used.
 - B. Instrument (type - model number etc.) used.
- IV. Results
 - A. Raw Data and Observations

This is the data obtained in the procedure (weights, titrations, sample volumes, any data used to obtain results). The read out charts from the instrument determinations and graphs are attached to the reports. The data from them is shown here too, such as peak sizes, etc. No results here! Any deviation from the procedure should be included here.
 - B. Calculations and/or Results

All calculations, if any, are shown here.
Results are shown here.
- V. Summary and Conclusion
 - A. Short summary of experiment. Results are included in the summary too!
 - B. Comments pertaining to: Was objective obtained? How did the experiment go? Any problems? Your evaluation of the experiment, etc.

Grading:

Objective	10 points
Discussion	10 points
Safety	5 points
Materials & Apparatus	5 points
Raw Data	20 points
Calculations & Results	30 points
Summary & Conclusion	20 points

Reports on experiments are due one week after they are finished. Each week that the report is turned in late, 5 points will be taken off the grade.

An Example Report:

Experiment ##

I. Introduction

A. Objective

To determine % ppm or what is in an unknown.

B. Discussion

1. Theory

In a chromatographic determination, the components are separated by their interaction between the mobile and the stationary phases as they move through the column. Temperature, carrier gas flow rate and the type of packing in the column affect the separations. (This is in your book as are all theories of the other methods. If it is a titration, tell what a titration is.)

2. Procedure

Short procedure. Do not put all the steps you do in order to get an instrument ready for the analysis.

3. Reactions

Reactions if any.

4. Calculation formulas

How results are arrived at, calculations etc.

II. Safety

III. Materials and Apparatus

IV. Results

A. Raw Data and Observations

Sample weights.

Area counts and calculations as in the internal standards method.

Milliliters of titrations.

Linear regression calculations if needed.

Anything that is used to get the results.

Dilutions.

Any deviations from the written procedure.

B. Calculations and Results

Calculations for results.

Titration calculations.

Results are shown here. Do not refer to charts, graphs or readouts. They are backup data only.

V. Summary and Conclusion

A. Summary

Short write up of what was done. Results are shown here too.

B. Conclusion

How did it go? Was objective obtained? Problems? Anything good or bad about the experiment.

Working With Your Lab Partner

Lab Partner's Name _____

Best way to contact (phone, email...) _____

To become a productive lab partner, develop and fine-tune the following skills and abilities:

1. **RESPONSIBILITY.** Before leaving the lab, make sure both you and your lab partner have completely filled out both you and your partner's data sheets. This is your insurance policy.
2. **LISTENING SKILLS.** You must be able to put your own thoughts aside and listen without interrupting or interpreting what your partner is saying. Try it - it's not easy.
3. **SELF-CONFIDENCE.** You must believe in yourself and in the worth of your contributions. Speak up!
4. **OPEN-MINDEDNESS.** Welcome change, and listen to the ideas others bring.
5. **CREATIVITY.** Try stretching yourself outside of your routines. Try a different method. It might work better than your current method.
6. **THOUGHT.** Keep your goal in sight. Instead of following the lab manual like a recipe, consider the instructions to be a guide. When you make an error, how can you adapt the manual's procedure to still reach your goal? Which type of balance will give you enough significant digits?
7. **RELIABILITY.** Do what you say you're going to do.
8. **OBJECTIVITY.** Assess ideas, thoughts, and opinions from all sides, not just yours.
9. **OPTIMISM.** Look at problems as opportunities. Knocking over the beaker containing your product can lead to learning about purification techniques.
10. **COOPERATION.** You must be able to accept team decisions and work just as hard on other people's ideas as you do on your own.

Adapted from Ern, B. L. and Lawley, C. M. (1992). The office professional as a team player. Office Hours, 229, 1.