



Chemistry Department

College of education

University of Salahaddin- Erbil

Course Book: 4th stage

Subject: Practical Instrumental Analysis

Lecturer's name: Dr. AVEEN F. JALAL

Academic Year: 2023-2024

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| 1. Course name | Instrumental Analysis |
| 2. Lecturer in charge | Dr. AVEEN F. JALAL |
| 3. Department/ College | Chemistry Department/ Education College |
| 4. Contact | E-mail: aveen.jalal@su.edu.krd or afaizulla@yahoo.com Tel: 07504069770 |
| 5. Time (in hours) per week | Theory: 3 hrs |
| 6. subject time | Laboratory : Group(A+B+C) / Wednesday 8:30am - 2:30pm Group(D+E) / Thursday 8:30pm-12:30pm |
| 7. Course code | Nothing |
| 8. Teacher's academic profile | Dr. AVEEN F. JALAL Lecturer in Analytical Chemistry B.Sc. in Chemistry , from Salahaddin University, College of Education, Chemistry Department, 2005. M.Sc. in Analytical Chemistry , Salahaddin University, College of Education, Chemistry Department, 2011. Thesis: Batch and Flow-injection Spectrophotometric determination of Meloxicam in some Pharmaceutical Formulations. Supervisor: Assist. Prof. Dr. Shirwan O. Baban. Ph.D. in Analytical Chemistry , Salahaddin University, College of Education, Chemistry Department, 2022. Thesis: Synthesis and Characterization of New Modified Magnetized Nanomaterials and Macrocyclic Compound to Remove Some Cationic Dyes in Textile Fiber Samples. Supervisor: Prof. Dr. Nabil Adil Fakhre No. of Publications: (10). |
| 9. Keywords | Practical Instrument, Analysis , Chemistry |
| 10. Course overview: | <ul style="list-style-type: none"> • This course presents a survey of instrumental methods of chemical analysis. We will focus on understanding the fundamental principles underlying instrumental methods and their realization in modern instrumentation for chemical analysis. We will focus on the following broad methodological areas: optical spectroscopy and Electrochemistry, .We focus on principles and concepts of implementation should provide greater insight both into how current instruments work as well as the basis for your understanding how they will work a decade from now. Lastly, we will pay attention to the chemical systems to which these methods are applicable and how best to obtain the chemical information desired using the most appropriate instrumental methods. The most important topics in this course are: <ul style="list-style-type: none"> • Luminescence's methods, • Basic Principles of Atomic Spectrometers, • Electroanalytical methods. |

11. Course objective:

Instrumental course concerns the practice of instrumental methods for quantitative analysis of chemical substances. Satisfactory completion of this course will afford students a working knowledge of analytical instrumentation typically employed in chemical research laboratories technique. It will also provide the student with an appreciation of the relative strengths and limitations of different instrumental based analysis methods. Specific objectives include:

Demonstrate knowledge of sampling methods for all states of matter, Assess sources of error in chemical and instrumental analysis and account for errors in data analysis, Recognize interferences in chemical and instrumental analysis, Comprehend the concept of and perform instrument and method calibration, Apply and assess concepts of availability and evaluation of analytical standards and formulate standardization methodology, Understand and be able to apply the theory and operational principles of analytical instruments At the end of the course, learner will be able to:

- Recognize the theory of operation, instrumental design and application of various instrumental methods.
- Understand and apply the various spectroscopic methods.

The main objective of this course is to introduce students to the modern instrumental methods of quantitative and qualitative analysis and to learn about the fundamental principles of operation of these methods, concentration will be drawn basically to spectroscopic and, their strengths and limitations and their application in Analysis will be discussed.

12. Student's obligation

It is important to realize that it is not possible to learn all that you need to know about Instrumental Analysis from just the text, lectures, supplemental readings, or problem sets. Rather, attention must be paid to all of these areas as the material covered in each is designed to be complementary. I think that you will find that the problems in the problem sets will be easier to solve if you've attended the lectures and done the assigned readings.

I encourage you to come to lecture having already read the assigned material as I would prefer to spend as much of our class time discussing the material, answering your questions about the material and asking you questions about the material. Obviously, our time in class will only be meaningful if you've already read the assigned readings for that particular unit.

Problem sets will not be collected and are not "due" at any particular time -- it is in your best interests, however, to work the problem sets contemporaneously with the lecture material. Detailed solutions to the problems sets will be made available online at the course website. Problems just like or very similar to those on the problem sets have a habit of finding their way onto exams, so it would be prudent of you to ensure that you can work the relevant problem set problems before you take one of the exams.

13. Forms of teaching

Different forms of practical teaching in laboratory will be used to reach the objectives of the course: power point presentations for the experimental head titles and definitions and summary of conclusions, classification of materials and any other illustrations, besides worksheet will be designed to let the chance for practicing on several aspects of the course in the classroom, furthermore students will be asked to prepare report on selective topics and summarizes articles contents published in English language, those articles need to be from printed media or internet articles. There will be laboratory discussions and the lecture will give enough background to solve, analyze, and evaluate problems sets, and different issues discussed throughout the course.

To get the best of the course, it is suggested that read the required procedures, teacher's notes regularly as all of them are foundations for the course.

Many additional, useful sources are on reserve. You are encouraged to read as many sources as possible because learning from several different viewpoints will deepen your knowledge and more closely simulate how you will learn new material in the future.

Experience has shown that the students who attend class, read more references, and complete more problems do much better! To be successful you will have to spend a significant amount of time on this course outside of the class time.

14. Assessment scheme

This course is divided into two portions: lecture and laboratory. The lecture portion constitutes 65% of the overall final grade, while the laboratory portion constitutes 35%.

Lecture Grade

The students are required to do closed book exam at the mid of the semester besides other assignments including daily quizzes, exams and all reports. The exam has 5 marks, the attendance, classroom activities; quiz exam and one report count 10 marks. Constructive classroom participation, submitting assignments, and attending class will be evaluated by the lecturer over the semester and used in borderline cases to determine the final grad.

There will be a final exam on 20 marks. So that the final grade will be based upon the following criteria:

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|---|-----|
| Mean of at least two-semester exam: | 20% |
| Classroom participation, Quiz, Assignments & Laboratory reports : | 5% |
| Reports: | 10% |
| Total ----- | 35% |

15. Student learning outcome:

By the end of the course students will be able to:

1. Acquire a detailed understanding of the principles of operation of principle of some technique in instrumental analysis.
2. Demonstrate critical thinking, quantitative reasoning, and scientific knowledge r elated to the wide variety of choices that must be made when using instrumental methods to solve analytical problems.
3. Acquire more advanced laboratory skills associated with the collection, handling, and evaluation of analytical data. The students will also demonstrate competence in writing scientific reports.
4. Interpret data and to use the appropriate statistical methods in their evaluation.

16. Course Reading List and References:

- **Principles of Instrumental Analysis, Skoog, 6th ed.; Thompson: Belmont, CA, 2007.**
- **Practical Analytical chemistry Gary Christian**
- **Fundamentals of analytical chemistry , Skoog & West**
- **Quantitative chemical analysis: D.C. Harris.**
- **Undergraduate instrumental analysis : Robinson & Frame**

| 17. The Topics: Experiment Title | Lecturer's name |
|--|------------------------|
| Course outline, general observations. | 1 st week |
| Introduction to AAS and AES methods, and the Use of the | 2 nd week |
| Determination of Potassium in Cement by flame photometry | 3 rd week |
| Determination of sodium in tap water by Flame photometry | 4 th week |
| Determination of Calcium in water sample by Flame – AAS using standard addition method | 5 th week |
| Determination of hardness of water sample by Flame – AAS | 6 th week |
| Indirect determination of chloride in soil sample by Flame - AAS | 7 th week |
| Determination each of HCl & H ₃ PO ₄ in a mixture Potentiometric titration | 8 th week |
| Determination of aspirin by conductometric titration | 9 th week |
| Determination of aspirin by conductometric back titration. | 10 th week |
| Simultaneous determination both of chloride and iodide by Potentiometric method | 11 th week |
| Flow injection potentiometric technique for determination of silver | 12 th week |
| Ion selective analysis for determination of Nitrate | 13 th week |
| Ion selective analysis for determination of sulphate using standard addition method | 14 th week |
| Examination | 15 th week |

19. Examinations:

Q1/ 10g soil sample dissolved and the filtrate complete to **50mL** using D.W. **5mL** of the sample solution have been taken into **25mL** volumetric flask and we should add **1mL** of the -----solution then completed to final volume by D. water. The later solution titrated with **0.25M EDTA** solution using photometric titration. The laboratory results obtained shown in the table below (at $\lambda=755$ nm) :

| | | | | | | | | | | | |
|--------------------------|-------------|---------------|---------------|---------------|---------------|---------------|--------------|--------------|--------------|--------------|--------------|
| Vol.(mL) EDTA | 0 | 0.50 | 1.0 | 1.5 | 2.0 | 2.5 | 3.0 | 3.5 | 4.00 | 4.50 | 5.0 |
| Abs. | 0.00 | -0.008 | -0.015 | -0.030 | -0.012 | -0.003 | 0.020 | 0.067 | 0.087 | 0.100 | 0.109 |

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|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| 5.5 | 6.0 | 6.5 | 7.0 | 7.5 | 8.0 | 8.5 | 9.0 |
| 0.119 | 0.123 | 0.128 | 0.130 | 0.129 | 0.125 | 0.123 | 0.121 |

- a-** Fill the blank.
b- Draw a graph to find the end-points.
c- Calculate the percentage of Ca^{2+} & Cu^{2+} in the soil sample.

Atomic weights: Ca=40 , Cu= 63.5

Q2/ Answer the following questions briefly:

- a) Write the principles for the determination of Sodium by Flame photometer instrument.

Q3/ For determination of SO_4^{2-} in water sample by the turbidimetric method. Take **5ml sample** with adding **1.0ml** of ----- reagent then diluted to **25ml** with D.W finally add **0.1g**-----and mixed for fixed time, the transmittance read **80**. The transmittances for a series of sulphate standard solution are shown in the table below. - Calculate the concentration of sulphate in original sample?

Not: drawing graph not allowed.

| Conc. (ppm) | T% |
|-------------|----|
| 5 | 95 |
| 10 | 88 |
| 15 | 83 |
| 20 | 78 |
| 25 | 72 |
| Sample | 80 |

Q4/ Calculate the concentration of chloride ion in water sample, which was determined by quenching of the fluorescence intensity of quinine, from the following data. [6 marks]

| | | | | | | | |
|----------------------------|------|-------|------|------|------|------|--------|
| [Cl^{-1}] mol/L | 0.00 | 0.01 | 0.02 | 0.04 | 0.06 | 0.08 | Sample |
| Fluorescence intensity | 100 | 30.05 | 18.0 | 11.0 | 6.7 | 5.4 | 25.0 |

20. Extra notes:

21. Peer review

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Best Wishes