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**Department of : chemistry**

**College of : science**

**University of : Salahaddin**

**Subject: practical molecular and atomic spectroscopy and automated analysis**

**Course Book – 4th stage**

**Lecturer's name : Wrea Mohammed Ibrahim**

**Academic Year: 2022/2023**

**Course Book**

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| **1. Course name** | practical molecular and atomic spectroscopy and automated analysis | |
| **2. Lecturer in charge** | Wrea | |
| **3. Department/ College** | Chemistry / Science | |
| **4. Contact** | e-mail: wrea.ibrahim@su.edu.krd | |
| **5. Time (in hours) per week** | Practical: 18 H | |
| **6. Office hours** | 14 H | |
| **7. Course code** |  | |
| **8. Teacher's academic profile** | BSc. ( 2006)  MSc. (2015) (assist lect. )  (Analytical chemistry – flow injection analysis ) | |
| **9. Keywords** | Quantitative analysis , Spectrophotometry ,flame photometry ,PH-meter , potentimetry, separation ,conductometry . | |
| **10. Course overview:**  Chemical analysis includes the use of instrumentation to solve an analytical problem. The use of instrumentation has now become a part of chemical analysis and is applied for all areas of pure and applied science. Any single instrument could not solve an analytical problem; instead, several instrumental techniques are required to solve the problem to a maximum extent. Hence instrumentation plays an important role in the production and evaluation of new products and in the protection of consumers and the environment. | | |
| **11. Course objective:**  Students who complete this class will understand that analytical instruments are not black boxes, but rather complex tools whose utility depends in detail on how analysts both configure and apply them. Towards that end there are three primary objectives. First, students will learn facts about major classes of instruments commonly used in chemical analysis. Their knowledge will be captured by the ability to block diagram these complex pieces of equipment, and tailor the specifications to the measurement needs. Second, the course will cover the basics of instrumental calibration and quality control. Analysts will develop the ability to apply calibration curves, internal standards and the method of standard addition as needed for various measurement problems. Finally, students must learn how to select and tailor the best instrumental method given a particular measurement need. This higher level skill involves critical evaluation of the strengths and limitations of the various method, and the ability to understand the context behind a measurement need. | | |
| **12. Student's obligation**  The student are required to do at last two closed exam at the mid of each semester for practical course beside other assignments. For every experiment the student must prepare full text paper which includes theory , calculation and discussion ( report ).  We have also do practical /practical examination  . | | |
| **13. Forms of teaching**  Power point ,data show, practical experiment in the laboratory. | | |
| **14. Assessment scheme**  Report : 8 marks  Quizzes : 6 marks  Seminars (activities) : 6 marks  Midterm exam: 15 marks. | | |
| **15. Student learning outcome:**  Any chemist has to understand how to analyze samples - whether they are water samples, soil samples or bits of a painting. Most often chemists do these using instruments of some sort. Machines like spectrophotometry or flame photometry , conductometry ….etc. can indicate both what's in a sample (qualitative) as well as how much of something there is(quantitative). | | |
| **16. Course Reading List and References**  1-Analytical Chemistry by Gary D.Christain, 5th edition  2-Fundamental of Analytical Chemistry by Douglas A.  Skoog  3-Principles of instrumental Analysis third edition  Douglas A.Skoog  4-Analytical Chemistry Principles by John H.Kennedy  5-Chemistry Experimentals for Instrumental methods  Donald T.Sawyer  6- Instumental method of chemical analysis third edition  Galen w.Ewing | | |
| **17. The Topics:** | | **Lecturer's name** |
| / | |  |
| **18. Practical Topics (If there is any)** | |  |
| Week 1:  Explain the principle and theory of methods used in instrumental analysis such has potentiometric, conductometric and spectrophotometric method.  Week 2 :  Explain the theory and principle of each experimental.  Week 3 :  Explain the procedure for each experimental  Week 4:  Potentiometric titration of chloride & iodide mixture , calculation of KSP for AgCl & AgI  Week 5 :  Spectrophotometric determination of chromium and manganese in steel  Week 6 :  Determination of acetyl salicylic acid by ultraviolet spectrophotometry  Week 7 :  Conductometric titration of HCl and CH3COOH with NaOH ,determination of Ka for acetic acid  Week 8 :  Spectrophotometric determination of the PKa value of an indicator  Week 9 :  Determination of sodium and potassium by flame photometry  Week 10 :  Separate a mixture of chloride and bromide on anion exchange resin column and estimate them by complex metric titration and modified lie big method.  Week 11 :  Separate and identify a mixture of cat ions  (Cu +2,CO+2and Ni+2) by ascending paper chromatography.  Week 12:  Spectrophotometric determination of copper sulfate.  *Course program ( second semester ) :*  Week 1 :  Explain the theory of experimental  Week 2 :  Explain the calculation of experimental  Week 3 :  Evaluation of dissociation constant of acetic acid by potentiometry  Week 4 :  PH- Titration of H3PO4 , calculation of K1, K2 and K3  Week 5 :  Determination of phenol by conductometric method  Week 6 :  Spectrophotometric analysis of aspirin  Week 7 :  Determination of Ca +2 by flam photometry using standard addition method  Week 8 :  Spectrophotometric determination of methyl red concentration at isobestic point  Week 9 :  A simple spectrophotometric method for the determination of ascorbic acid in pharmaceutical preparations.  Week 10 :  Fractionate the Nickel and Cobalt as chloride complexes using a strong base anion exchanger, then to estimate then by complex metric titration and modified liebig method.  Week 11:  Separate and identify a mixture of indicators by descending chromatography. | |  |
| **19. Examinations:**  University of Salahaddin Analytical Chemistry 4th Stage  College of Science Practical Examination Date:  Chemistry Department Time: 60 min.  Q1/ What are requirements of ion exchange resin. [5mark]  Q2/ What is the flame source in flame photometry? On what factor the flame is dependence? [10mark]  Q3/ What is the reference electrode? For what it be used? Give an example for it that you were used in the laboratory; draw the shape of this electrode. [25mark]  Q4/ Compare with diagram and reaction between conductometric titration of (HCl & NaOH) and (CH3COOH & NaOH). [25mark]  Q5/ Answer with chemical reaction only. [30mark]  1- Conversion of chromium and manganese to Cr6+ and Mn7+ respectively in steel sample.  2- How can you separate (Cl- & I-)? by using AgNO3 and NH3 (complex formation).  3- Identification each ions in the separation of (Cu2+ , Fe3+ & Ni2+) by paper chromatography.  Q6/ What is (bases of separation) for the following separation techniques? [5mark]  1) masking & demasking 2) chromatography 3) distillation 4) extraction 5) controlling pH  -----------------------------------------------------------------------------------------------  University of Salahaddin Analytical Chemistry 4th Stage  College of Science Practical Examination Date:  Chemistry Department Time: 60 min.  Q1/ A potentiometric titration of acetic acid with NaOH gave the following data:  C:\Users\Chemistry\AppData\Local\Microsoft\Windows\Temporary Internet Files\Content.Word\Untitled.png  From these data:  1- Find the normality of 50mL (acetic acid) if you know the normality of NaOH is (0.02N).  2- Find the pKa value for acetic acid. [36 mark]  Q2/ Draw a graph between absorbance and wavelength showing effect of time in the determination of ascorbic acid by spectrophotometric method. [14 mark]  Q3/ Discuss the followings: [50 mark]  1- In the determination of Aspirin the absorbance were measured at 530nm.  2- Phenol must be converted to the anion before titrating with NaOH in the determination of phenol by conductometric method.  3- Extraction system.  4- Continuous extraction.  5- Extraction of Uncharged Metal Chelates  --------------------------------------------------------------------------------------  University of Salahaddin Practical Instrumental Analysis 4th Stage  College of Science Final Examination Date:  Chemistry Department Time: 90 min.  Q1/ Explain the followings: [50 mark]  1-In the determination of phenol by conductometric method at the beginning the conductance varies very little.  2-Standard addition method is better than direct calibration curve.  3-Absorbance decrease by increasing the concentration of ascorbic acid in the spectrophotometric determination of ascorbic acid.  4-Flame photometry is applied for a limit number of elements.  5-Using salt bridge in electrochemical cell.  Q2/ A sample contain calcium, how can you determine calcium in this sample by flame photometry? Write in detail with necessary diagrams. [30 mark]  Q3/ A 100ppm (250mL) stock solution of ascorbic acid was prepared (How prepared)?, from this stock solution 5,10,15,20 and 25mL were placed in five volumetric flask then completed to 50mL with water. The absorbance of each volumetric flask were measured the results are shown in the table below. Find the concentration of ascorbic acid in the unknown.  C:\Users\Chemistry\AppData\Local\Microsoft\Windows\Temporary Internet Files\Content.Word\2.png    [35 mark]  Q4/ Answer the followings: [35 mark]  1-How selectivity increased in quantitative analytical methods (in general)?  2-Separate Fe(II) and Cu(II) in a sample (use a reducing agent and 1,10-phenanthroline as a reagent).  3-Hydrochloric acid used with the mobile phase for separation Fe3+, Cu2+ and Ni2+ by paper chromatography. Why?  4-In paper chromatography the paper must be handled from the side edges or with gloves. Why?  5-What are main stages in the performance of ion exchange experiments?  6-What is the relation between partition coefficient (Kd) and extraction efficiency? How can you increase extraction efficiency?  7-Explain the extraction of Ion-association Complexes.  University of Salahaddin Practical Instrumental Analysis 4th Stage  College of Science Final Examination Date:  Chemistry Department Time: 90 min.  Q1/ Describe the following preparations: [60 mark]  a)A solution (900 mL) of 3M HNO3 from the commercial reagent that is 70.5% (w/w) HNO3 and has a specific gravity of 1.42 .  b)A solution (500mL) contain (70 ppm) of Ca2+ from pure Ca3(PO4)2 .  Q2/ Discuss the followings: [ 35 mark]  1-Limitation of flame photometry. [16 mark]  2-Detection the end point in potentiometric titration. [10 mark]  3-Calibration of a pH-meter before use. [9 mark]  Q3/ Draw the absorption spectrum for a solution containing both dichromate and permanganate ion. [20 mark]  Q4/ Define the followings:  Combined glass electrode - Atomization - Separation  [15 mark]  Q5/ Give short notes on the followings: [20 mark]  1-Uses of potentiometric method [12 mark]  2-Factors affecting on conductivity[8 mark]  Q6/ Answer the followings: [50 mark]  a)Using the fume of concentrated ammonia solution in the separation of Fe3+, Cu2+ and Ni2+ by paper chromatography. Why? [12 mark]  b)What are Retention factor (Rf - value) and partition coefficient (Kd)? [10 mark]  c)What are properties of extracting solvent? [15 mark]  d)What are applications of ion exchange chromatography? [8 mark]  e)Write about batch extraction method. [5 mark]  [At. Wt. for Ca = 40 , P = 31 , O = 16 , N = 14 , H = 1 mol/L ] | | |
| **20. Extra notes:**  Here the lecturer shall write any note or comment that is not covered in this template and he/she wishes to enrich the course book with his/her valuable remarks. | | |
| **21. Peer review پێداچوونه‌وه‌ی هاوه‌ڵ**  This course book has to be reviewed and signed by a peer. The peer approves the contents of your course book by writing few sentences in this section.  *(A peer is person who has enough knowledge about the subject you are teaching, he/she has to be a professor, assistant professor, a lecturer or an expert in the field of your subject).*  ئه‌م کۆرسبووکه‌ ده‌بێت له‌لایه‌ن هاوه‌ڵێکی ئه‌کادیمیه‌وه‌ سه‌یر بکرێت و ناوه‌ڕۆکی بابه‌ته‌کانی کۆرسه‌که‌ په‌سه‌ند بکات و جه‌ند ووشه‌یه‌ک بنووسێت له‌سه‌ر شیاوی ناوه‌ڕۆکی کۆرسه‌که و واژووی له‌سه‌ر بکات.  هاوه‌ڵ ئه‌و که‌سه‌یه‌ که‌ زانیاری هه‌بێت له‌سه‌ر کۆرسه‌که‌ و ده‌بیت پله‌ی زانستی له‌ مامۆستا که‌متر نه‌بێت.‌‌ | | |