Department of Chemistry

College of Science

Salahaddin University

Subject: Statistics and Gravimetric Analysis

Course Book – (Stage Two) Course 1

Lecturer's name *Assist Prof. Dr Diyar Salahuddin Ali*

Academic Year: 2023/2024

Course Book

1. Course name	**Statistics and Gravimetric Analysis**
2. Lecturer in charge	**Assist Prof. Dr Diyar Salahuddin Ali**
3. Department/ College	**Chemistry / Science**
4. Contact	**e-mail: Diyar.ali@su.edu.krd** **Tel: 0750(xxx) xxxx**
5. Time (in hours) per week	**Theory: 2** **Practical: 3**
6. Office hours	**4**
7. Course code	
8. Teacher's academic profile	**I have more than 20 year experience teaching of Analytical Chemistry, also I have more than 15 papers are published in different local and foreign journals, I got three grand from American organization for supporting my research in Analytical field. Supervising M.Sc. student during my duty in the college. Participation in different conferences and meeting over the world. I worked in e-learning filed too, up to date I am member in Abn Sinna Center for e-learning which supporting form UNESCO.** **B.Sc. of Chemistry from 1994** **M.Sc. of Analytical Chemistry from 1998** **Ph. D of Analytical Chemistry from 2006** **Assist Lecturer** Feb 1998 – March 2006 **Instructor** March 2006 – Feb 2011 **Assist Prof.** Feb 2011 up to date
9. Keywords	**Analytical Chemistry, Analytical Techniques, Gravimetric and statistical**

10. Course overview:

First period covered all gravimetric analysis, the purpose for that for takes the gravimetric analysis in the first semester as continues for Sample Analysis, as we know all sample analysis begin with knowing which is include? (Qualitative), then we must try to calculate it (Quantitative) after that we must try to separate between them, finally if we can't do that by chemical method we changed to Instrumental Analysis.

In the second period we covered all statistical tools which used in the analytical chemistry, we try to solve a huge problems related to this issue, like accuracy, precision, detection limit, determination limit, sensitivity, selectivity , T and F- tests. In

our two periods time the students should be achieved for all of the objectives are preceded by the phrase, "The student should be able to:". As I write before I am responsible for Analytical Chemistry for second stage students which they study just gravimetric analysis and statistical tools, so before we starting with them, the students should they have a good background about Analytical Chemistry principles like meaning of this branch of chemistry and classification and all qualitative process.

11. Course objective:

The student should take all explanation about Introduction to Analytical Chemistry as general, Analytical Chemistry Classification, Simple information about Qualitative Analysis, Quantitative Analysis, Gravimetric Methods of Analysis, Types of Gravimetric Methods Precipitation Gravimetry Mechanism of Precipitate Formation
Chemical formula for precipitate and the calculations in the quantitative analysis, Factors which affected of the solubility precipitate, Mechanism for Precipitation, Factors Affecting Particle Size, History of Statistical Chemistry, Terminology of Statistical Chemistry, The Assessment of Analytical Data, Gaussian distribution, Numerical criteria for selecting an analytical method, precision, accuracy, sensitivity, selectivity, detection limit, determination limit, reproducibility, calibration graph, standard addition…..

12. Student's obligation

The students are required to do at least two closed exam at the mid of each semester besides other assignments and each student must prepare full report at the end of the year. All exams have marks, full report also has marks, the classroom activities count marks and mark for attendance too.

13. Forms of teaching

Our lecture is depend directly on showing the strong point in the lecture via data show depending on the power point program… and solve problem on the white board with the students.

14. Assessment scheme

Mid- semester exam: 10%
Classroom participation and assignments 5%
Practical Course 35%
Final Exam: 50% which include just theoretical

15. Student learning outcome:

Analytical chemistry plays a very role in the chemistry field, all student after graduate they working in some labs, industrial companies, hospitals and in all these institution they need principles of analytical chemistry and they use all assessment of data. For this reason analytical chemistry is exists in all four stages in our department. Now if we see all labs which are randomly distributed in our community all of them are depending on analysis the samples which came from different sources.

16. Course Reading List and References:

1- Analytical Chemistry by Gary D. Christain, 5th edition

2- Chemical Separation principles, Techniques and

Experiments by Clifton E. Meloan

3- Fundamentals of Analytical Chemistry by Douglas A. Skoog

4- Quantitative Chemical Analysis by Kolthofe- Sanell

5- Analytical Chemistry Principles by John H. Kennedy

6- Modern Analytical Chemistry by David Harvey

7- Analytical Chemistry, Theoretical and Metrological Fundamentals K. Danzer

8- Principles and Practice of Analytical Chemistry, F.W. Fifield

9- Validation and Qualification in Analytical Laboratories,

17. The Topics:	Lecturer's name
1- Curriculum of Analytical Chemistry for Second Stage Chemistry 2- Meaning of Analytical Chemistry 3- Classification of Analytical Chemistry 4- Types of Analysis 5- Electrolytes, acids and bases	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week one
1- Quantitative Analysis 2- Chemical Methods 3- Gravimetric Methods of Analysis 4- Types of Gravimetric Analysis 5- Terms and Examples	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week two
1- Types of Gravimetric Analysis (Explanation) 2- Precipitation Gravimetry 3- Important Attributes for Precipitation Gravimetric 4- Solubility Considerations 5- Avoiding Impurities	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week three
1- Mechanism of Precipitate Formation 2- Induction Period 3- Sample Classification 4- Conditions Required for Prepare any Sample to Gravimetric Analysis 5- Precipitate and weighing form properties which are used in Quantitative gravimetric analysis. 6- The advantageous and disadvantageous for organic and inorganic precipitant.	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week four

1- Chemical formula for precipitate and the calculations in the quantitative gravimetric analysis. 2- Examples	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week five
1- Factors which affected of the solubility precipitate 2- Common Ion Effect…. 3- Example for Common Ion Effect 4- Ionic Strength Effect	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week six
First Examination	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week seven
1- Examples of Ionic Strength Effect 2- Activity and activity coefficient…… 3- Examples Activity and activity coefficient……	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week eight
1. pH Effect 2. Examples of pH Effect	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week nine
1. Mechanism for Precipitation 2. Factors Affecting Particle Size 3. Techniques to minimize	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week ten
Second Examination	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week eleven
1- Induction Period 2- Examples	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week twelve
Gravimetric Theory for Precipitation	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week thirteen
1. Conditions need for Analytical Precipitation 2. Gravimetric Overview	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week fourteen

Factors which affecting on the Precipitation Process	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week fifteen
Review for all subject with solving problems	Assist Prof. Dr. Diyar S. Ali (2 hrs) Week sixteen
18. Practical Topics (If there is any)	

Class: Sunday 8:30 –11:00 / 11:00-1:30 / 1:30- 4:00 Practical

Monday 8:30 –11:00 / 11:00-1:30 / 1:30- 4:00 Practical

Course Objective

The following objectives should be achieved by the student during first course. All of the objectives are preceded by the phrase, "The student should be able to:" All Gravimetric and Statistical experiments for the course included

Grading

The students are required to do at least two closed exams for practical course besides other assignments. For every experiment the student must prepare full text paper which includes theory, calculation and discussion. All exams have 3 marks which meaning summation of it equal to 8 marks. 4 marks on the reports and seven marks on the quizzes.

Mid- course exam:

1st exam :8

2nd exam: 8

Classroom participation and assignments

Which distributed as follows?

Report: 4

Quiz: 7

Final Exam: 8 for practical and theoretical

Course Material

1. Analytical Chemistry by Gary D. Christain, 5th edition
2. Chemical Separation principles, Techniques and Experiments by Clifton E. Meloan
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4. Quantitative Chemical Analysis by Kolthofe- Sanell
5. Analytical Chemistry Principles by John H. Kennedy
6. Modern Analytical Chemistry by David Harvey
7. Analytical Chemistry, Theoretical and Metrological Fundamentals K. Danzer
8. Principles and Practice of Analytical Chemistry, F.W. Fifield
9. Validation and Qualification in Analytical Laboratories, Ludwig Huber
10. A Text Book of Quantitative Analysis; By: Vogel.
11. Quantitative Chemical Analysis; By: Kolthoff.
12. Quantitative Analysis; By: Alexeyev.

Course Program

Week 1:

Introduction of gravimetric analysis.

Week 2:

Explanation of main concepts in G.A

Week 3:

Gravimetric determination of water of hydration in crystallized CuSO4.5H2O or BaCl2.2H2O.

Week 4:

Gravimetric determination of Water of Hydration in crystallized substance

(Unknown)

Week 5:

Gravimetric determination of hygroscopic water in hygroscopic substances.

Week 6:

Gravimetric determination of Chloride as silver chloride

Week 7:

Gravimetric determination of Chloride as silver chloride (unknown)

Week 8:

Gravimetric determination of Sulphate as barium sulphate (BaSO4)

Week 9:

Gravimetric determination of Sulphate as barium sulphate (unknown)

Week 10:

Gravimetric determination of fluoride as lead chlorofloride.

Week 11:

Gravimetric determination of fluoride as lead chlorofloride (unknown).

Week 12.

Gravimetric determination of Calcium as calcium oxalate.

Week 13:

Gravimetric determination of Calcium as calcium oxalate (unknown).

First Lab Examination

Week 14:

Gravimetric determination of Cobalt.

Week 15:

Gravimetric determination of Cobalt (unknown)

19. Examinations:

1) Organic sample weighed 0.185 g was ignited in an excess of oxygen, then all carbon dioxide were collected in barium hydroxide solution. Calculate the percentage of carbon in the sample, if you know that the precipitate formed (barium carbonate) weighed 0.526 gm. What the name of this type of gravimetric analysis?

Typical Answer for 1):

Volatilization Method

$Ba(OH)_2 + CO_2 \rightarrow BaCO_3 + H_2O$

Wt. of C = Wt. of $BaCO_3$ x (At.Wt. C/M.Wt. $BaCO_3$)

Wt. of C = 0.526 x (12/197) =0.0320 gm

C% = (Wt. of C/Wt. of Sample) x 100 = (0.0320/0.185) x 100 = 17.29%

2) Calculate the weight of carbon dioxide liberated from 1.5 g sample include 34% magnesium carbonate?

Typical Answer for 2):

$MgCO_3$ % = (Wt. $MgCO_3$/Sample weight) x 100

34 % = (Wt. $MgCO_3$/1.5) x 100

Wt. $MgCO_3$ = 0.51 gm

Wt. of CO_2 = Wt. of $MgCO_3$ x (Mt.Wt. CO_2/M.Wt. $MgCO_3$)

Wt. of CO_2 = 0.51 x (44/84)

Wt. of CO_2 = 0.267 gm

3) Calculate the barium sulphate solubility in 0.1 M HCl, if you know that solubility product constant for the precipitate is equal to 1.08×10^{10} and the second dissociation constant for sulphuric acid equal to 1.2×10^{-2}

Typical Answer for 3):

$BaSO_4 + H^+ \rightarrow Ba^{2+} + HSO_4$

$HSO_4^- \rightarrow H^+ + SO_4^{2-}$ K2

$K2=[H^+][SO_4^{2+}]/[HSO_4^-]= 1.2x10^{-2}$ 1

$Ksp=[Ba^{2+}][SO_4^{2-}]= 1.08 \times 10^{-10}$ 2

$[Ba^{2+}]= [HSO_4^-]+[SO_4^{2-}]$.....3

$1.2 \times 10^{-2} = [0.1][SO_4^{2+}]/[HSO_4^-]$

$[HSO_4^-]= 8.3 [SO_4^{2+}]$

$[Ba^{2+}]= 8.3 [SO_4^{2+}]+[SO_4^{2-}]$

$[Ba^{2+}]= 9.3 [SO_4^{2+}]$

$[SO_4^{2+}]=[Ba^{2+}]/9.3$

$Ksp=[Ba^{2+}][Ba^{2+}]/9.3= 1.08 \times 10^{-10}$

$[Ba^{2+}]^2 = 9.3 \times 10^{-10}$

$[Ba^{2+}] = 3 \times 10^{-5}$ mol/l

4) Calculate the barium fluoride solubility in 0.1 M HCl, if you know that solubility product constant for the barium fluoride is equal to 1.7×10^{-6} and the dissociation constant for fluoric acid equal to 7.4×10^{-4}.

Typical Answer for 4):

$BaF_2 + 2H^+ \rightarrow Ba^{2+} + 2HF$

$HF \rightarrow H^+ + F^-$

$Ka= [H^+][F^-]/[HF]= 7.4 \times 10^{-4}$

Square the both side

$Ka^2 = [H^+]^2[F^-]^2/[HF]^2= (7.4 \times 10^{-4})^2$

$Ksp=[Ba^{2+}][F^-]^2 = 1.7 \times 10^{-6}$

Dividing eq 2 on eq 1

$Ksp/Ka = (1.7 \times 10^{-6})/(7.4 \times 10^{-4}) = [Ba^{2+}][F^-]^2[HF]^2/[H^+]^2[F^-]^2$

Assume $[Ba^{2+}] = X$

[HF]=2X

Subsitiuted in eq 3

$(1.7 \times 10^{-6}) \times 0.01/(7.4 \times 10^{-4})^2 = [x][2x]^2$

$0.031=4X^3$

X= 0.197 mole/l

5) Why solubility of calcium oxalate precipitate was increased when adding hydrochloric acid?

Typical Answer for 5):

Because the negative ion which produced from salt dissociation combined with the positive ion and produced a weak acid unstable, then the defect could be happed in the equilibrium state…

According to Lee- Shatelia principle the reaction orientation depart from the left to the right…..

6) Calculate the ionic strength for 0.05 M KNO3 and 0.1 M Na2SO4 solution?

Typical Answer for 6):

$$Na_2SO_4 \rightarrow 2Na^+ + SO_4^{2-} \qquad KNO_3 \rightarrow K^+ + NO_3^-$$
$$0.1M \qquad 0.2M \quad 0.1M \qquad 0.05M \qquad 0.05M \quad 0.05M$$

$$I = \frac{1}{2}\sum_{i=1}^{n} c_i z_i^2$$

$$I = \left[\frac{[0.05x(1)^2]+[0.05x(1)^2][0.2x(1)^2]+[0.1x(2)^2]}{2}\right] = 0.35$$

7) Calculate the solubility of Ba(IO3)2 in 0.02 M of KIO3 , solubility product of Ba(IO3)2 equal to 1.57 x10-9

Typical Answer for 7):

$$Ba(IO_3)_2 \rightarrow Ba^{2+} + 2IO_3^-$$
$$Ksp = [Ba^{2+}][IO_3^-]^2$$
$$1.57x10^{-9} = [X][2X + 0.02]^2$$
$$1.57x10^{-9} = 4x10^{-4}X$$
$$X = 3.9x10^{-6}M$$

8) What are the main differences between specific and selective reagents?

Typical Answer for 8):

Specific reagents, which are rare, react only with a single chemical species.

Selective reagents, which are more common, react with only a limited number of species.

9) Show the properties of colloidal and crystalline suspensions?

Typical Answer for 9):

(1) Colloidal suspensions (10-7 to 10-4 cm in diameter)

show no tendency to settle from solution and are not easily filtered

(2) Crystalline suspension

Tend to settle spontaneously and are easily filtered.

20. Extra notes:

21. Peer review