KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed University Established Under Section 3 of UGC Act 1956)

COIMBATORE-21

DEPARTMENT OF CHEMISTRY

SYLLABUS

B.Sc Chemistry **2017-2020**

17CHU604A BASIC ANALYTICAL CHEMISTRY 3H 3C

Instruction Hours/week: L:3 T:0 P:0 **Marks: Internal:40 External: 60 Total:100**

Course Objectives

The course enables the student to

1. Understand the interdisciplinary nature of analytical chemistry.
2. Understand the various methods involved in the analysis of soil.
3. Understand the various methods involved in the analysis of water.
4. Understand the various methods involved in the analysis of food products.
5. Understand the various methods involved in the analysis of cosmetics.

Course Outcome

After this course the student know the

1. Interdisciplinary nature of analytical chemistry.
2. Various methods involved in the analysis of soil.
3. Various methods involved in the analysis of water.
4. Various methods involved in the analysis of food products.
5. Various methods involved in the analysis of cosmetics.

UNIT I

Introduction: Introduction to Analytical Chemistry and its interdisciplinary nature. Concept of sampling.Importance of accuracy, precision and sources of error in analytical measurements.Presentation of experimental data and results, from the point of view of significant figures.

UNIT II

Analysis of soil: Composition of soil, Concept of pH and pH measurement, Complexometric titrations, Chelation, Chelating agents, use of indicators

a. Determination of pH of soil samples.
b. Estimation of Calcium and Magnesium ions as Calcium carbonate by complexometric titration.

UNIT III

Analysis of water: Definition of pure water, sources responsible for contaminating water, water sampling methods, water purification methods.

a. Determination of pH, acidity and alkalinity of a water sample.
b. Determination of dissolved oxygen (DO) of a water sample.

UNIT IV

Analysis of food products: Nutritional value of foods, idea about food processing and food preservations and adulteration.

a. Identification of adulterants in some common food items like coffee powder, as afoetida,chilli powder, turmeric powder, coriander powder and pulses, etc.
b. Analysis of preservatives and colouring matter.

Chromatography: Definition, general introduction on principles of chromatography, paper chromatography, TLC etc.

a. Paper chromatographic separation of mixture of metal ion (Fe3+ and Al3+).
b. To compare paint samples by TLC method.

Ion-exchange: Column, ion-exchange chromatography etc. Determination of ion exchange capacity of anion / cation exchange resin (using batch procedure if use of column is not feasible).

UNIT V

Analysis of cosmetics: Major and minor constituents and their function

a. Analysis of deodorants and antiperspirants, Al, Zn, boric acid, chloride, sulphate.
b. Determination of constituents of talcum powder: Magnesium oxide, Calcium oxide, Zincoxide and Calcium carbonate by complexometric titration.

Suggested Reading:

Text Books:

1. Willard, H.H., Merritt, L.L., Dean, J. &Settoe, F.A. (1988). *Instrumental Methods of Analysis* (VII Edition). Wadsworth Publishing Co. Ltd., USA.
2. Skoog, D.A., Holler, J. & Crouch, S.R. (2009). *Instrumental Analysis* (India Edition). Cengage Learning India Private Limited, New Delhi.
3. Skoog, D.A., West, D.M. & Holler, F.J. (1992). *Fundamentals of Analytical Chemistry* (VI Edition). Fort Worth: Saunders College Publishing.
4. Harris, D. C. (2006). *Quantitative Chemical Analysis*. W. H. Freeman and Company Ltd.
5. Dean, J. A. (1992). *Analytical Chemistry Notebook*. McGraw Hill.

Reference Books:

1. Day, R. A. & Underwood, A. L. (1991). *Quantitative Analysis*. Prentice Hall of India.
2. Freifelder, D. (1982). *Physical Biochemistry* (II Edition). W.H. Freeman and Co., USA.

3. Cooper, T.G. (1977). *The Tools of Biochemistry.* John Wiley and Sons, USA.
4. Robinson, J.W. (1995). *Undergraduate Instrumental Analysis* (V Edition). Marcel Dekker Inc., New Delhi.

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Coimbatore – 641 021.

LECTURE PLAN

DEPARTMENT OF CHEMISTRY

FACULTY NAME: Dr. S. MANICKASUNDARAM
SUBJECT NAME: BASIC ANALYTICAL CHEMISTRY SUB.CODE:17CHU604A
SEMESTER: V CLASS: III B.Sc (CHEMISTRY)

S.No.	Lecture Duration Period	Topics to be Covered	Support Material/Page Nos
		UNIT-I	
1	1	Introduction to Analytical Chemistry and its interdisciplinary nature	T1: 1-4, T2: 1-2
2	1	Conceptof sampling.	T1: 153-157; T2: 2-3
3	1	Importance of accuracy, precision	T1: 82-98; T2:29-30
4	1	sources of error in analyticalmeasurements.	T1: 91, 309-340; T2:39-45
5	1	Presentation of experimental data and results, from the point of view ofsignificant figures	T2:39-42
6	1	Recapitulation	
	Total No of Hours Planned For Unit 1=06		
		UNIT-II	
1	1	Composition of soil, Concept of pH and pH measurement	T1: 342;T2:107
2	1	Complexometrictitrations,	T2: 244-246
3	1	Chelation, Chelating agents, use of indicators	T1: 295-297; T2: 230-232
4	1	Determination of pH of soil samples.	T1: 303, 322-323; T2: 107
5	1	Estimation of Calcium and Magnesium ions as Calcium carbonate by complexometrictitration.	T2: 244-246
6	1	Recapitulation	
	Total No of Hours Planned For Unit II=06		

		UNIT-III	
1	1	Definition of pure water, sources responsible for contaminating water	W1
2	1	water sampling methods,	W1
3	1	water purification methods.	W1
4	1	Determination of pH, acidity and alkalinity of a water sample.	T5: 128-132; T4:6, 14-16
5	1	Determination of dissolved oxygen (DO) of a water sample.	T4: 45-46
6	1	Recapitulation	
	Total No of Hours Planned For Unit III=06		
		UNIT-IV	
1	1	Nutritional value of foods, idea about food processing and foodpreservations and adulteration.	T3: 1-45
2	1	Identification of adulterants in some common food items like coffee powder, asafoetida,chilli powder, turmeric powder, coriander powder and pulses, etc. Analysis of preservatives and colouring matter.	T3: 102-145, 145-189
3	1	**Chromatography:** Definition, general introduction on principles of chromatography, paperchromatography, TLC etc.	T1: 861-943; T2: 702
4	1	Paper chromatographic separation of mixture of metal ion (Fe3+ and Al3+). To compare paint samples by TLC method.	T1: 940-942; T2: 692
5	1	**Ion-exchange:** Column, ion-exchange chromatography etc.Determination of ion exchange capacity of anion / cation exchange resin (using batchprocedure if use of column is not feasible).	T1: 859-861; T2: 692
6	1	Recapitulation	

	Total No of Hours Planned For Unit IV=07		
		UNIT-V	
1	1	**Analysis of cosmetics:** Major and minor constituents and their function	T5: 1-29
2	1	Analysis of deodorants and antiperspirants,	T5: 35-40
3	1	Al, Zn, boric acid, chloride, sulphate	T5: 35-40
4	1	Determination of constituents of talcum powder:	T5: 29-40
5	1	Magnesium oxide, Calcium oxide, Zincoxide and Calcium carbonate by complexometric titration.	T1: 406-429; T2:244-246
6	1	Recapitulation	
7	1	Discussion of previous year end semester question papers	
8	1	Discussion of previous year end semester question papers	
9	1	Discussion of previous year end semester question papers	
	Total No of Hours Planned for unit V=09		
Total Planned Hours	**34**		

Text Books:

1. Skoog D.A.; West D.M., Holler J, crouch J.A., (1992), Fundementals of Analytical Chemistry, Brooks/Cole, USA.
2. Christian, G. D. (2007). *Analytical Chemistry* (VI Edition). United States: John Wiley & Sons.
3. MorriesB.Jacobs (2001), The chemical analysis of foods and food products, Von NostrandReinhood Company.
4. Anand Dev Guptha, (2014), Handbook of food, water and soil analysis, International E-Publications, India.
5. G.K.Sharma, J.Gadiya, M.Bhanawat, (2018), A textbook of Cosmetic formulation, Research gate.

W1: https://www.scribd.com/doc/29103542/WATER-TREATMENT-TECHNOLOGY-TAS-3010-LECTURE-NOTES-9a-Water-Intake-Screening-Aeration-Coagulation

S.No	Question	a	b	c	d	Answer
1.	The area which deals with the identification of elements, ions, or compounds present in a sample	Qualitative analysis	Quantitative analysis	Elemental analysis	Gravimetric analysis	Qualitative analysis
2.	The area which deals with the determination of how much of one or more constituents is present	Qualitative analysis	Quantitative analysis	Elemental analysis	Volumetric analysis	Quantitative analysis
3.	Volumetric analysis is an example for	Qualitative analysis	Quantitative analysis	Elemental analysis	spectral analysis	Qualitative analysis
4.	Gravimetric analysis is an example for	Qualitative analysis	Quantitative analysis	Elemental analysis	spectral analysis	Quantitative analysis
5.	Infrared spectra will give “fingerprints” of organic compounds or their functional groups is a	Qualitative analysis	Quantitative analysis	Elemental analysis	spectral analysis	spectral analysis
6.	Certain chemical reactions will produce colors to indicate the presence of classes of organic compounds, is an example for	Qualitative analysis	Quantitative analysis	Elemental analysis	spectral analysis	Qualitative analysis

7.	The formation of a white precipitate when adding a solution of silver nitrate in dilute nitric acid to a dissolved sample indicates the presence of a halide is an example for	Qualitative analysis	Quantitative analysis	Elemental analysis	spectral analysis	Qualitative analysis
8	Determinate Errors are otherwise called as	Systematic errors	Accidental errors	Random errors	False errors	Systematic errors
9	Indeterminate Errors are otherwise called as	Systematic errors	Accidental errors	Determinant errors	False errors	Accidental errors
10	Indeterminate Errors are otherwise called as	Systematic errors	Random errors	Determinant errors	False errors	Random errors
11	Example for an Instrumental errors	uncalibrated glassware	personal errors	Inherentin the method	small differences in successive measurements	uncalibrated glassware
12	Which is an operative error	uncalibrated glassware	personal errors	Inherentin the method	small differences in successive measurements	personal errors
13	Which is called Errors of the method	uncalibrated glassware	personal errors	Experimental conditions	small differences in successive measurements	Experimental conditions
14	Experimental conditions is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
15	Coprecipitation of impurities during precipitation is	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
16	small differences in successive	Errors of the	operative error	Instrumental errors	Random errors	Random errors

	measurements	method				
17	slight solubility of a precipitate is	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
18	side reactions is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
19	Incompletereactions is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
20	Impurities in reagents is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Errors of the method
21	uncalibrated glassware is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Instrumental errors
22	Personal errors is an example for	Errors of the method	operative error	Instrumental errors	Random errors	Operative errors
23	Which is not an Errors of the method	Personal error	Impurities in reagents	Incompletereactions	slight solubility of a precipitate	Personal error
24	Which is not an Errors of the method	uncalibrated glassware	Impurities in reagents	Coprecipitations of impurities	slight solubility of a precipitate	uncalibrated glassware
25	Which is not an Errors of the method	small differences in successive measurements	Impurities in reagents	Coprecipitations of impurities	slight solubility of a precipitate	small differences in successive measurements
26	Determinant errors are	Additive and multiplicative	Subtractive	divisive	Subtractive and divisive	Additive and multiplicative
27.	diethylene glycol ethyl ether is used to scent	deoderant	Antiperspirant	Skin preservative	Anti-histamine	Antiperspirant
28.	Example for a deoderant	Aluminium chloride	zirconium chlorohydrate	Scented talcum powders	diethylene glycol ethyl ether	Scented talcum powders

29.	Example for a Antiperspirant	Aluminium chloride	dibutylpthalate	Scented talcum powders	diethylene glycol ethyl ether	Aluminium chloride
30.	Example for a Antiperspirant	dibutylpthalate	zirconium chlorohydrate	Scented talcum powders	diethylene glycol ethyl ether	zirconium chlorohydrate
31.	Antiperspirants work by	blocking the sweat ducts	kill the bacteria responsible for producing body odour	kill the virus responsible for producing body odour	blocking the nose ducts	blocking the sweat ducts
32.	Deodrants work by	blocking the sweat ducts	kill the bacteria responsible for producing body odour	kill the virus responsible for producing body odour	blocking the nose ducts	kill the bacteria responsible for producing body odour
33.	Talcum powder for sensitive skin contain	Corn starch	kaolin	Sandal wood extract	aloe vera	Corn starch
34.	Talcum powder for sensitive skin contain	tapioca starch	kaolin	Sandal wood extract	aloe vera	tapioca starch
35.	Ingredient in Talcum powder which gives skin a silky feel	tapioca starch	kaolin	Sandal wood extract	chamomile	kaolin
36.	Ingredient in Talcum powder which gives skin a silky feel	tapioca starch	Aloe vera	Sandal wood extract	chamomile	Aloe vera
37.	Ingredient in Talcum powder which gives soothing to skin, and prevent rashes and prickly heat.	tapioca starch	Aloe vera	Sandal wood extract	rose extract	Sandal wood extract
38.	Ingredient in Talcum powder which gives soothing to skin, and prevent rashes and prickly heat.	tapioca starch	Aloe vera	rose extract	chamomile	chamomile

39.	Chamomile in a talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	soothing to skin, and prevent rashes and prickly heat.
40.	Sandal wood extract in a talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	soothing to skin, and prevent rashes and prickly heat.
41.	Cornstarch in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	sensitive skin
42.	Tapioca starch in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	sensitive skin
43.	Kaolin in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	skin a silky feel
44.	Rose water in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	skin a silky feel
45.	Tocopherol is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	fat-soluble compound

46.	Tocopherol	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	Acts as an antioxidant, protecting the skin from free-radicals
47.	Dimethicone is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	A group of silicone molecules
48.	Dimethicone	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	lubricates the skin without feeling heavy.
49.	Parabens is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	man-made chemical used for preserving cosmetic ingredients
50.	Parabens	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	Prevents that expensive cosmetic from forming bacteria and fungus
51.	Titanium dioxide is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	A group of silicone molecules

52.	Titanium dioxide	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	lubricates the skin without feeling heavy.
53.	Acts as an antioxidant, protecting the skin from free-radicals	tocopherol	Dimethicone	Paraben	Titanium di oxide	tocopherol
54.	fat-soluble vitamin	tocopherol	Dimethicone	Paraben	Titanium di oxide	tocopherol
55.	A group of silicone molecules	tocopherol	Dimethicone	Paraben	Titanium di oxide	Dimethicone
56.	The compound which lubricates the skin without feeling heavy.	tocopherol	Dimethicone	Paraben	Titanium di oxide	Dimethicone
57.	man-made chemical used for preserving cosmetic ingredients	tocopherol	Dimethicone	Paraben	Titanium di oxide	Paraben
58.	Prevents that expensive cosmetic from forming bacteria and fungus	tocopherol	Dimethicone	Paraben	Titanium di oxide	Paraben
59.	natural mineral taken from oxide of titanium	tocopherol	Dimethicone	Paraben	Titanium di oxide	Titanium di oxide
60.	It acts as a filler, making the cosmetic product more opaque	tocopherol	Dimethicone	Paraben	Titanium di oxide	Titanium di oxide

UNIT-1

SYLLABUS

Introduction: Introduction to Analytical Chemistry and its interdisciplinary nature. Concept of sampling. Importance of accuracy, precision and sources of error in analytical measurements. Presentation of experimental data and results, from the point of view of significant figures.

INTRODUCTION

Analytical chemistry is concerned with the chemical characterization of matter and the answer to two important questions: what is it (qualitative analysis) and how much is it (quantitative analysis). Chemicals make up everything we use or consume, and knowledge of the chemical composition of many substances is important in our daily lives. Analytical chemistry plays an important role in nearly all aspects of chemistry, for example, agricultural, clinical, environmental, forensic, manufacturing, metallurgical, and pharmaceutical chemistry. The nitrogen content of a fertilizer determines its value. Foods must be analyzed for contaminants (e.g., pesticide residues) and for essential nutrients (e.g., vitamin content). The air we breathe must be analyzed for toxic gases (e.g., carbon monoxide). Blood glucose must be monitored in diabetics (and, in fact, most diseases are diagnosed by chemical analysis). The presence of trace elements from gun powder on a perpetrator's hand will prove a gun was fired by that hand.

The quality of manufactured products often depends on proper chemical proportions, and measurement of the constituents is a necessary part of quality assurance. The carbon content of steel will influence its quality. The purity of drugs will influence their efficacy.

Analytical Chemistry seeks ever improved means of measuring the chemical composition of natural and artificial materials. The techniques of this science are used to identify the substances which may be present in a material and to determine the exact amounts of the identified substance.

Analytical chemists serve the needs of many fields:

● In medicine, analytical chemistry is the basis for clinical laboratory tests which help physicians diagnose disease and chart progress in recovery.
● In industry, analytical chemistry provides the means of testing raw materials and for assuring the quality of finished products whose chemical composition is critical. Many household products, fuels, paints, pharmaceuticals, etc. are analyzed by the procedures developed by analytical chemists before being sold to the consumer.

● Environmental quality is often evaluated by testing for suspected contaminants using the techniques of analytical chemistry.

● The nutritional value of food is determined by chemical analysis for major components such as protein and carbohydrates and trace components such as vitamins and minerals. Indeed, even the calories in food are often calculated from its chemical analysis.

Analytical chemists also make important contributions to fields as diverse as forensics, archaeology, and space science.

The discipline of analytical chemistry consists of qualitative analysis and quantitative analysis. The former deals with the identification of elements, ions, or compounds
present in a sample (we may be interested in whether only a given substance is present), while the latter deals with the determination of how much of one or more constituents is present. The sample may be solid, liquid, gas, or a mixture. The presence of gunpowder residue on a hand

generally requires only qualitative knowledge, not of how much is there, but the price of coal will be determined by the percent of undesired sulfur impurity present

Qualitative tests may be performed by selective chemical reactions or with the use of instrumentation. The formation of a white precipitate when adding a solution of silver nitrate in dilute nitric acid to a dissolved sample indicates the presence of a halide. Certain chemical reactions will produce colors to indicate the presence of classes of organic compounds, for example, ketones. Infrared spectra will give "fingerprints" of organic compounds or their functional groups.

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A clear distinction should be made between the terms selective and specific:

● A selective reaction or test is one that can occur with other substances but exhibits a degree of preference for the substance of interest.

● A specific reaction or test is one that occurs only with the substance of interest. Unfortunately, very few reactions are truly specific but many exhibit selectivity.

Selectivity may be also achieved by a number of strategies. Some examples are:

1. Sample preparation (e.g., extractions, precipitation)
2. Instrumentation (selective detectors)
3. Target analyte derivatization (e.g., derivatize specific functional groups)
4. Chromatography, which separates the sample constituents

For quantitative analysis, the typical sample composition will often be known (we know that blood contains glucose), or else the analyst will need to perform a qualitative test prior to performing the more difficult quantitative analysis. Modern chemical measurement systems often exhibit sufficient selectivity that a quantitative measurement can also serve as a qualitative measurement. However, simple qualitative tests are usually more rapid and less expensive than quantitative procedures. Qualitative analysis has historically been composed of two fields: inorganic and organic. The former is usually covered in introductory chemistry courses, whereas the latter is best left until after the student has had a course in organic chemistry.

Sampling

Collecting a representative sample is an aspect of analytical chemistry that the beginning analytical student is often not concerned with because the samples handed to him or her are assumed to be homogeneous and representative. Yet this process can be the most critical aspect of an analysis. The significance and accuracy of measurements can be limited by the sampling process. Unless sampling is done properly, it becomes the weak link in the chain of the analysis. A life could sometimes depend on the proper handling of a blood sample during and after sampling. If the analyst is given a sample and does not actively participate in the sampling process, then the results obtained can only be attributed to the sample "as it was received." And the chain of custody as mentioned earlier must be documented.

By appropriate application of experience and statistics, these materials can be sampled as accurately as the analysis can be performed. Often, however, the matter is left up to the analyst. The ease or complexity of sampling will, of course, depend on the nature of the sample.

The problem involves obtaining a sample that is representative of the whole. This sample is called the **gross sample**. Its size may vary from a few grams or less to several pounds, depending on the type of bulk material. Once a representative gross sample is obtained, it may have to be reduced to a sufficiently small size to be handled. This is called the **sample**. Once the sample is obtained, an aliquot, or portion, of it will be analyzed. This aliquot is called the **analysis sample**. Several replicate analyses on Replication in sampling and the same sample may be performed by taking separate aliquots. Analysis are key considerations. In the clinical laboratory, the gross sample is usually satisfactory for use as the sample because it is not large and it is homogeneous (e.g., blood and urine samples).The analysis sample will usually be from a few milliliters to a fraction of a drop (a few microliters) in quantity.

1. Solids. Inhomogeneity of the material, variation in particle size, and variation within the particle make sampling of solids more difficult than other materials. The easiest but usually most unreliable way to sample a material is the **grab sample**, which is one sample taken at random

and assumed to be representative. The grab sample will be satisfactory only if the material from which it is taken is homogeneous. For most reliable results, it is best to take 1/50 to 1/100 of the total bulk for the gross sample, unless the sample is fairly homogeneous. The larger the particle size, the larger the gross sample should be. The easiest and most reliable time to sample large bodies of solid materials is while they are being moved. In this way any portion of the bulk material can usually be exposed for sampling. Thus, a systematic sampling can be performed to obtain aliquots representing all portions of the bulk. Some samples follow. In the loading or unloading of bags of cement, a representative sample can be obtained by taking every fiftieth or so bag or by taking a sample from each bag. In the moving of grain by wheelbarrow, representative wheelbarrow loads or a shovelfulfrom each wheelbarrow can be taken. All of these aliquots are combined to form the gross sample.

2. Liquids. Liquid samples tend to be homogeneous and representative samples are much easier to get. Liquids mix by diffusion only very slowly and must be shaken to obtain a homogeneous mixture. If the material is indeed homogeneous, a simple grab (single random) sample will suffice. For all practical purposes, this method is satisfactory for taking blood samples. The composition of some samples vary on when it is taken. This is the case for urine samples, therefore 24-h urine sample collections are generally more representative than a single "spot sample".The timing of sampling of biological fluids is, however, very important. The composition of blood varies considerably before and after meals, and for many analysesa sample is collected after the patient has fasted for a number of hours. Preservatives such as sodium fluoride for glucose preservation and anticoagulants may be added to blood samples when they are collected.

Blood samples may be analyzed as *whole blood*, or they may be yield *plasma* or *serum* according to the requirements of the particular analysis. Most commonly, the concentration of the substance external to the red cells (the extracellular concentration) will be a significant indication of physiological condition, and so serumor plasma is taken for analysis. If liquid samples are not homogeneous, and if they are small enough, they can be shaken and sampled immediately. For example, there may be particles in the liquid that have tended to settle. Large bodies of liquids are best sampled after a transfer or,if in a pipe, after passing through a pump when they have

undergone thorough mixing. Large stationary liquids can be sampled with a "*thief*" *sampler*, which is a device forobtaining aliquots at different levels. It is best to take the sample at different depths at

a diagonal, rather than straight down. The separate aliquots of liquids can be analyze dindividually and the results combined, or the aliquots can be combined into one gross sample and replicate analyses performed. This latter procedure is probably preferred because the analyst will then have some idea of the precision of the analysis.

3. Gases The usual method of sampling gases involves sampling into an evacuated container, often a specially treated stainless steel canister or an inert polyvinyl fluoride (Tedlar) bag is commonly used. The sample may be collected rapidly (a grab sample) or over a long period of time, using a small orifice to slowly fill the bag. A grab sample is satisfactory in many cases. To collect a breath sample, for example, the subject could blow into an evacuated bag or blow up a mylar balloon. Auto exhaust could be collected in a large evacuated plastic bag. The sample may be supersaturated with

moisture relative to ambient temperature at which the sample container is. Moisture will condense in the sampling container after sample collection and the analyte of interest (e.g., ammonia in breath or nitrous acid in car exhaust) will be removed by the condensed moisture. The sample container must be heated and the sample transferred through a heated transfer line if the analyte is to be recovered.

The volume of gross gas sample collected may or may not need to be known. Often, the *concentration* of a certain analyte in the gas sample is measured, rather than the *amount*. The temperature and pressure of the sample will, of course, be important in determining the volume and hence the concentration. Gas sampling techniques mentioned here does not concern gases dissolved in liquids, such CO2 or O2 in blood. These are treated as liquid samples and are then handled accordingly to measure the gas in the liquid or to release it from the liquid prior to measurement.

Data Analysis:

Errors in chemical analysis-Defining terms: Mean median, accuracy and precision

Accuracy is the degree of agreement between the measured value and the true value. An absolute true value is seldom known. A more realistic definition of accuracy, then, would assume it to be the agreement between a measured value and the accepted true value.

We can, by good analytical technique, such as making comparisons against a known standard sample of similar composition, arrive at a reasonable assumption about the accuracy of a method, within the limitations of the knowledge of the "known" sample (and of the measurements). The accuracy to which we know the value of the standard sample is ultimately dependent on some measurement that will have a given limit of certainty in it.

Precision is defined as the degree of agreement between replicate measurements Good
of the same quantity. That is, it is the repeatability of a result. The precision may be accuracy.
expressed as the standard deviation, the coefficient of variation, the range of the data,or as a confidence interval (e.g., 95%) about the mean value. Good precision does not assure good accuracy. This would be the case, for example, if there were a systematic error in the analysis. The volume of a pipet used to dilute each of the samples may bein error. This error does not affect the precision, but it does affect the accuracy. On the other hand, the precision can be relatively poor and the accuracy may be good;admittedly, this is very rare. Since all real analyses are unknown, the higher the degree of precision, the greater the chance of obtaining the true value. It is fruitless to hope that a value is accurate despite the precision being poor; and the analytical chemiststrives for repeatable results to assure the highest possible accuracy.
These concepts can be illustrated with targets, as in Figure 3.1. Suppose you are at target practice and you shoot the series of bullets that all land in the bull's-eye (lefttarget). You are both precise and accurate. In the middle target, you are precise (steadyhand and eye), but inaccurate. Perhaps the sight on your gun is out of alignment. In theright target you are imprecise and therefore probably inaccurate. So we see that goodprecision is needed for good accuracy, but it does not guarantee it.As we shall see later, reliability increases with the number of measurements made. The number of measurements required will depend on the level of uncertaintythat is acceptable and on the known reproducibility of the method.

Determinate Errors—They Are Systematic

Two main classes of errors can affect the accuracy or precision of a measured quantity.

Determinate errors are those that, as the name implies, are determinable and that presumably can be either avoided or corrected. They may be constant, as in the case of an uncalibrated pipet that is used in all volume deliveries. Or, they may be variable but of such a nature that they can be accounted for and corrected, such as a buret whose volume readings are in error by different amounts at different volumes.

The error can be proportional to sample size or may change in a more complex manner. More often than not, the variation is unidirectional, as in the case of solubility loss of a precipitate due to its solubility (negative error). It can, however, be random in sign, i.e., a positive or negative error. Such an example is the change in solution volume and concentration occurring with changes in temperature. This can be corrected for by measuring the solution temperature. Such measurable determinate errors are classed as systematic errors.

Some common determinate errors are:

1. Instrumental errors. These include faulty equipment such as uncalibrated glassware.

2. Operative errors. These include personal errors and can be reduced by experience and care of the analyst in the physical manipulations involved.Operative errors can be minimized by having a checklist of operations. Operations in which these errors may occur include transfer of solutions,effervescence and “bumping” during sample dissolution, incomplete dryingof samples, and so on. These are difficult to correct for. Other personal errors include mathematical errors in calculations and prejudice in estimating measurements.

3. Errors of the method. These are the most serious errors of an analysis. Most of the above errors can be minimized or corrected for, but errors that are inherentin the method cannot be changed unless the conditions of the determinationare altered. Some sources of methodical errors

include coprecipitationof impurities, slight solubility of a precipitate, side reactions, incompletereactions, and impurities in reagents. Sometimes correction can be relatively simple, for example, by running a reagent blank. A blank determination is ananalysis on the added reagents only. It is standard practice to run such blanksand to subtract the results from those for the sample. But a good blank analysisalone cannot guarantee correct measurements. If the method, for example,responds to an analyte present in the sample other than the intended analyte,the method must be altered. Thus, when errors become intolerable, anotherapproach to the analysis must be made. Sometimes, however, we are forcedto accept a given method in the absence of a better one.

Determinate errors may be additive or multiplicative, depending on the nature of the error or how it enters into the calculation. In order to detect systematic errors in an analysis, it is common practice to add a known amount of standard to a sample (a "spike") and measure its recovery and note that good spike recovery cannot also correct for response from an unintended analyte(i.e., an interference). The analysis of reference samples helps guard against method errors or instrumental errors.

It is always a good idea to run a blank.

Indeterminante Errors

The second class of errors includes the indeterminate errors, often called accidental or random errors, which represent the experimental uncertainty that occurs in any measurement. These errors are revealed by small differences in successive measurements made by the same analyst under virtually identical conditions, and they cannot be predicted or estimated. These accidental errors will follow a random distribution; therefore, mathematical laws of probability can be applied to arrive at some conclusion regarding the most probable result of a series of measurements.

It is beyond the scope of this text to go into mathematical probability, but we can say that indeterminate errors should follow a normal distribution, or Gaussian curve. Such a curve is shown in. The symbol σ represents the standard deviation of an infinite population of

measurements, and this measure of precision defines the spread of the normal population distribution as shown in Figure. It is apparent that there should be few very large errors and that there should be an equalnumber of positive and negative errors.

Indeterminate errors really originate in the limited ability of the analyst to control or make corrections for external conditions, or the inability to recognize the appearance of factors that will result in errors. Some random errors stem from the intrinsic nature of things, for example, consider that a sample of the radionuclide 129I is taken. The isotope is long lived, and in a short time there will not be a perceptible change in its number. But, if a sufficient amount is taken, then based on the half-life, you can expecta decay to occur every 60 s. In reality, this may not occur every 60 s, but can fluctuate,with an average of 60 s. Sometimes, by changing conditions, some unknown error will disappear. Of course, it will be impossible to eliminate all possible random errorsin an experiment, and the analyst must be content to minimize them to a tolerable orinsignificant level.

Indeterminate errors are random and cannot be avoided.

“Undetectable errors are infinite in variety, in contrast to detectable errors, which by definition are limited.”

Significant Figures: How Many Numbers Do You Need?

The last digit of a measurement has some uncertainty. You can’t include any more digits.

The weak link in the chain of any analysis is the measurement that can be made with the least accuracy or precision. It is useless to extend an effort to make the other measurements of the analysis more accurately than this limiting measurement. The number of significant figures can be defined as **the number of digits necessary to express the results of a measurement consistent with the measured precision**.Since there is uncertainty (imprecision) in any measurement of at least ±1 in the last significant figure, the number of significant figures includes all of the digits that are known, plus the first uncertain one. In reported answers, it generally does not make sense to include additional digits beyond the first uncertain one. Each

digit denotes the actual quantity it specifies. For example, in the number 237, we have 2 hundreds, 3 tens, and 7 units. If this number is reported as a final answer, it implies that uncertainty lies in the units digit (e.g., ±1).

The digit 0 can be a significant part of a measurement, or it can be used merely to place the decimal point. The number of significant figures in a measurement is independent of the placement of the decimal point. Take the number 92,067. This number has five significant figures, regardless of where the decimal point is placed. For example, 92, 067 μm, 9.2067 cm, 0.92067 dm, and 0.092067m all have the same number of significant figures. They merely represent different ways (units) of expressing one measurement. The zero between the decimal point and the 9 in the last number is used only to place the decimal point. There is no doubt whether any zero that *follows* a decimal point is significant or is used to place the decimal point. In the number 727.0, the zero is not used to locate the decimal point but is a significant

part of the figure. Ambiguity can arise if a zero *precedes* a decimal point. If it falls between two other nonzero integers, then it will be significant. Such was the case with 92,067. In the number 936,600, it is impossible to determine whether one or both or neither of the zeros is used merely to place the decimal point or whether they are a part of the measurement. It is best in cases like this to write only the significant figures you are sure about and then to locate the decimal point by scientific notation. Thus, 9.3660 × 105 has five significant figures, but 936,600 contains six digits, one to place the decimal. Sometimes, the number may be written with a period at the end to denote all digits are significant, e.g., 936,000. to avoid amiguity.

List the proper number of significant figures in the following numbers and indicatewhich zeros are significant.

0.216; 90.7; 800.0; 0.0670

Solution

0.216 three significant figures

90.7 three significant figures; zero is significant

800.0 four significant figures; all zeros are significant

0.0670 three significant figures; only the last zero is significant _

If a number is written as 500, it could represent 500 ± 100. If it is written as 5.00×10^2,then it is 500 ± 1.

The significance of the last digit of a measurement can be illustrated as follows.

Assume that each member of a class measures the width of a classroom desk, using the same meter stick. Assume further that the meter stick is graduated in 1-mm increments. The measurements can be estimated to the nearest 0.1 division (0.1 mm) by interpolation, but the last digit is uncertain since it is only an estimation. A series of class readings, for example, might be

565.4 mm

565.8 mm

565.0 mm

566.1 mm

565.6 mm *(*average*)*

Standard Deviation—The Most Important Statistic

Each set of analytical results should be accompanied by an indication of the **precision**

of the analysis. Various ways of indicating precision are acceptable.

The standard deviation σ of an infinite set of experimental data is theoretically given by

$$\sigma = \sqrt{\frac{\sum (x_i - \mu)^2}{N}}$$

where*xi*represents the individual measurements and μ represents the mean of an infinite number of measurements (which should represent the "true" value). This equation holds strictly only as $N \rightarrow \infty$, where N is the number of measurements. In practice, we must calculate the individual deviations from the mean of a limited number of measurements, x, in which it is anticipated that $x \rightarrow \mu$ as $N \rightarrow \infty$, although we have no assurance this will be so; x is given by*_(xi/N)*. average, x = *_(xi/N)*

For a set of N measurements, there are N (independently variable) deviations from some reference number. But if the reference number chosen is the estimated mean, x, the sum of the individual deviations (retaining signs) must necessarily add up to zero, and so values of $N - 1$ deviations are adequate to define the Nth value. That is, there are only $N - 1$ independent deviations from the mean; when $N - 1$ values have been selected, the last is predetermined. We have, in effect, used one degree of freedom of the data in calculating the mean, leaving $N - 1$ **degrees of freedom** for calculating the precision.

As a result, the **estimated standard deviation** s **of a finite set of experimental data** (generally $N < 30$) more nearly approximates σ if $N - 1$, the number of degrees of freedom, is substituted for N ($N - 1$ adjusts for the difference between x and μ).

$$s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N - 1}}$$

The value of s is only an estimate of σ, and it will more nearly approach σ as the number of measurements increases. Since we deal with small numbers of measurements in an analysis, the precision is appropriately represented by s.

Example

Calculate the mean and the standard deviation of the following set of analytical results:

15.67, 15.69, and 16.03 g.

Solution

x_i	$x_i - \overline{x}$	$(x_i - \overline{x})^2$
15.67	0.13	0.0169
15.69	0.11	0.0121
16.03	0.23	0.0529
$\sum$ 47.39	$\sum$ 0.47	$\sum$ 0.0819

$$\overline{x} = \frac{\sum x_i}{N} = \frac{47.39}{3} = 15.80$$

$$s = \sqrt{\frac{0.0819}{3-1}} = 0.20 \text{ g}$$

Two mark questions

1. What are the types of error?

UNIT-2

SYLLABUS

Analysis of soil:

Composition of soil, Concept of pH and pH measurement, Complexometrictitrations, Chelation, Chelating agents, use of indicators

a. Determination of pH of soil samples.
b. Estimation of Calcium and Magnesium ions as Calcium carbonate by complexometrictitration.

COMPOSITION OF SOILS

Components and phases Soil is the natural material that covers the land surface of the earth. It is the product of mechanical, chemical and biological weathering of parent material. Its four constituent fractions or components are:

- mineral matter (both primary and secondary minerals)
- organic matter
- water solution
- soil air.

Soils are very complex in their composition and quite variable in their occurrence and properties. Their complex nature, changing behavior upon use, and variable spatial distribution are the result of nonhomogeneous mixing and interacting of all their components. In spite of this intricate nature, soils can be handled and studied systematically. Their components, like all other matter in nature, exist in the three states of matter, i.e. solid, liquid and gas. With some exceptions, all three states occur in soils side by side. Hence, these states are called 'phases' of the soil. Strictly speaking, the term 'phase' is not used properly here, since in chemistry it is reserved for a portion of a system

having definite geometrical boundaries and uniform properties. The latter is not true here. Nevertheless, we will adhere to this terminology and discuss, successively,

1. The solid phase
2. The liquid phase
3. The gas phase.

PH

pH is calculated as the negative log of a solution's hydrogen ion concentration:
$PH = - \log_{10} [H^+]$

The **pH scale** is used to rank solutions in terms of acidity or basicity (alkalinity). Since the scale is based on pH values, it is logarithmic, meaning that a change of 1 pH unit corresponds to a ten-fold change in H^+ ion concentration. The pH scale is often said to range from 0 to 14, and most solutions do fall within this range, although it's possible to get a pH below 0 or above 14. Anything below 7.0 is acidic, and anything above 7.0 is alkaline, or basic.

Soil PH

Sample Preparation with Reagent Water

1 Air dry the soil sample.

2 Sieve the soil sample through a No. 10 sieve (2 mm mesh) to remove the coarser soil fraction.

3 Weigh out approximately 10 g of the air-dried and sieved soil sample.

4 Place the soil into a glass container and add approximately 10 mL of distilled or deionized water.

5 Mix thoroughly and let stand for 1 hour.

OR

Sample Preparation with Calcium Chloride Solution

1 Weigh out approximately 10 g of the air-dried and sieved soil sample.

2 Place the soil into a glass container and add approximately 10 mL of 0.01 M calcium chloride solution. 3 Mix thoroughly and let stand for 1 hour.

1 Measure the temperature of the suspended soil sample. Set the temperature dial on the pH meter to match the measured temperature in C.

2 Rinse the probes with distilled or deionized water. Blot dry.

3 With the meter on, place the electrode in the partially settled sample suspension to be measured.

4. If the meter is calibrated using pH 4.00 and pH 7.00 buffers and the sample reading is >7.00, the meter must be recalibrated using pH 7.00 and 10.00 buffers.

Many metal ions form slightly dissociated complexes with various ligands (complexing agents). The analytical chemist makes judicious use of complexes to mask undesired reactions. The formation of complexes can also serve as the basis of accurate and convenient titrations for metal ions in which the titrant is a complexing agent.Complexometric titrations are useful for determining a large number of metals.Selectivity can be achieved by appropriate use of *masking agents* (addition of othercomplexing agents that react with interfering metal ions, but not with the metalof interest) and by pH control, since most complexing agents are weak acids orweak bases whose equilibria are influenced by the pH. In this chapter, we discussmetal ions, their equilibria, and the influence of pH on these equilibria. The EDTA titration of calcium plusmagnesium is commonly used to determine water hardness. In the food industry,calcium is determined in cornflakes. In the plating industry, nickel is determinedin plating solutions by complexometric (also called chelometric) titration, and inthe metals industry in etching solutions. In the pharmaceutical industry, aluminium hydroxide in liquid antacids is determined by similar titrations. Nearly all metals can beaccurately determined by complexometric titrations. Complexing reactions are usefulforgravimetry, spectrophotometry, and fluorometry, and for masking interfering ions.

Complexes play important roles in many chemical and biochemical processes. For example, the heme molecule in blood holds iron tightly because the nitrogen atoms of the heme form strong ligating or complexing bonds. In general, the nitrogen atom derived from an amino group is a good donor atom or complexer. The iron [asiron (II)] in turn bonds readily with oxygen to transport oxygen gas from the lungsto elsewhere in the body and then easily releases it because oxygen is a poor donor atom or complexer. Carbon monoxide kills because it is a strong complexer and displaces oxygen; it binds to heme 200 times more strongly than does oxygen, forming carboxyhemoglobin.

Many cations will form complexes in solution with a variety of substances that have a pair of unshared electrons (e.g., on N, O, S atoms in the molecule) capable of satisfying the coordination number of the metal cation. [The metal cation is a Lewisacid (electron pair acceptor), and the complexer is a Lewis base (electron pair donor).]The number of molecules of the complexing agent, called the **ligand**, will depend on the coordination number of the metal cation and on the number of complexingon the ligand molecule.

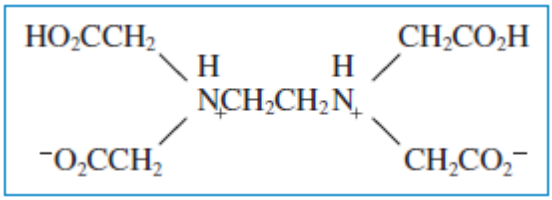
Chelates: EDTA—The Ultimate Titrating Agent for Metals

Simple complexing agents such as ammonia are rarely used as titrating agents because a sharp end point corresponding to a stoichiometric complex is generally difficult to achieve. Certain complexing agents that have two or more complexing groups on the molecule, however, do form well-defined complexes and can be used as titrating agents. Schwarzenbach demonstrated that a remarkable increase in stability is achieved if a bidentate ligand (one with two complexing groups) is used (see Reference 4 for his many contributions). For example, he showed replacing ammonia with the bidentateethylenediamine,$NH_2CH_2CH_2NH_2$ (en), results in a highly stable $Cu(en)_2^{2+}$ complex.

The most generally useful titrating agents are aminocarboxylic acids, in whichthe amino nitrogen and carboxylate groups serve as ligands. The amino nitrogensare more basic and are protonated $(-NH_3^+)$ more strongly than the carboxylategroups. When these groups bind to metal atoms,

they lose their protons. The metalcomplexes formed with these multidentatecomplexing agents are often 1:1, regardlessof the charge on the metal ion, because there are sufficient complexing groups on onemolecule to satisfy all the coordination sites of the metal ion.

An organic agent that has two or more groups capable of complexing with a metal ion is called a **chelating agent**. The complex formed is called a **chelate**. The chelating agent is called the *ligand*. Titration with a chelating agent is called a **chelometrictitration**, perhaps the most important and practical type of complexometrictitrations.The most widely used chelating agent in titrations is **ethylenediaminetetraaceticacid (EDTA)**. The formula for EDTA is



Each of the two nitrogens and each of the four carboxyl groups contains a pair of unshared electrons capable of complexing with a metal ion. Thus, EDTA contains six complexing groups. We will represent EDTA by the symbol H4Y. It is a tetraprotic acid, and the hydrogens in H4Y refer to the four ionizablehydrogens belonging to the four carboxylic acid groups. At sufficiently low pH, the nitrogens can also be protonated and this diprotonated EDTA can be considered a hexaprotic acid. However, this occurs at a very low pH and EDTA is almost never used under such conditions. It is the unprotonated ligand Y^{4-} that forms complexes with metal ions, that is, the protons are displaced by the metal ion upon complexation.

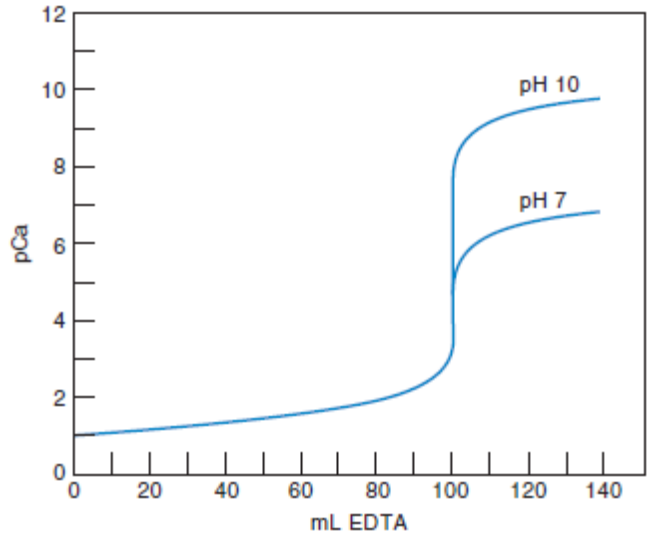
Metal–EDTA Titration Curves

A titration is performed by adding the chelating agent to the sample; the reaction occurs as in Equation

Consider the formation of the EDTA chelate of Ca^{2+}. This can be represented by:

$$Ca^{2+} + Y^{4-} \rightleftharpoons CaY^{2-} \quad ($$

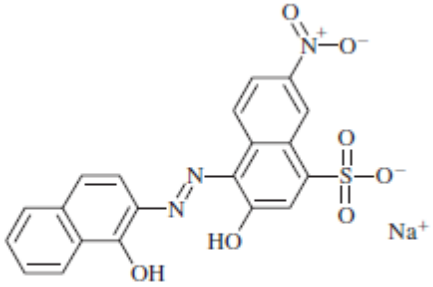
Figure below shows the titration curve for Ca2+ titrated with EDTA at pH 10. Before the equivalence point, the Ca2+ concentration is nearly equal to the amount of unchelated unreacted) calcium since the dissociation of the chelate is slight (analogous to the amount of an unprecipitated ion).



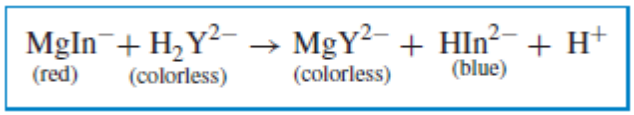
Detection of the End Point: Indicators—They Are Also Chelating Agents

We can measure the pMpotentiometrically if a suitable electrode is available, for example, an ion-selective electrode, but it is simpler if an indicator can be used. Indicators used for complexometric titrations are themselves chelating agents.

Eriochrome Black T is a typical indicator. It contains three ionizable protons, so we will represent it by H3In. This indicator can be used for the titration of Mg2+ with EDTA. A small amount of indicator is added to the sample solution, and it forms a red complex with part of the Mg2+; the color of the uncomplexed indicator is blue. As soon as all the free Mg2+ is titrated, the EDTA displaces the indicator from the magnesium, causing a change in the color from red to blue:



Eriochrome Black T



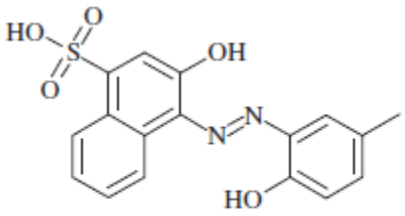
This will occur over a range of pMg values, and the change will be sharper if the indicator is kept as dilute as possible but is still sufficient to give a good color change. Of course, the metal–indicator complex must be less stable than the metal–EDTA complex, or else the EDTA will not displace it from the metal. On the other hand, it must not be too weak, or the EDTA will start replacing it at the beginning of the titration, and a diffuse end point will result. In general, *the Kf for the metal–indicator complex should be 10 to 100 times less than that for the metal–titrant complex*.

The formation constants of the EDTA complexes of calcium and magnesium are too close to differentiate between them in an EDTA titration, even by adjusting pH. So they will titrate together, and the Eriochrome Black T end point can be used as above. This titration is used to determine **total hardness of water**,.Eriochrome Black T cannot be used to indicate the endpoint of a direct EDTA titration of calcium in the absence of magnesium, however, because the indicator forms too weak a complex with calcium to give a sharp end point. Therefore, a small measured amount of Mg2+ is added to the Ca2+ solution; as soon as the Ca2+ and the small amount of free Mg2+ are titrated, the end-point color change occurs as above. (The Ca2+ titrates first since its EDTA chelate is more stable.) A correction is made for the amount of EDTA used for titration of the Mg2+ by performing a “blank” titration on the same amount of Mg2+ added to the buffer.

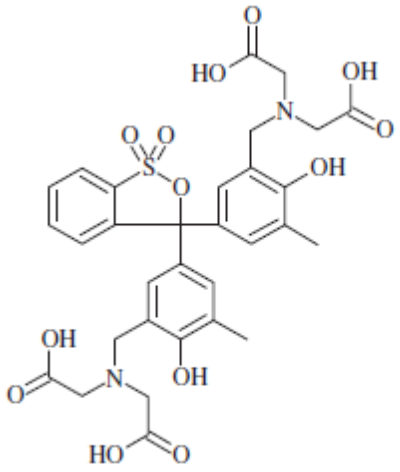
The titration of calcium and magnesium with EDTA is done at pH 10, using an ammonia–ammonium chloride buffer. The pH must not be too high or the metal hydroxide may precipitate, causing the reaction with EDTA to be very slow. Calcium can actually be titrated in the presence of magnesium by raising the pH to 12 with strong alkali; $Mg(OH)_2$ precipitates and does not titrate.

Since Eriochrome Black T and other indicators are weak acids, their colors will depend on the pH because their ionized species have different colors. For example, with Eriochrome Black T, H_2In^- is red (pH <6), HIn^{2-} is blue (pH 6 to 12), and In^{3-} is yellow orange (pH >12). Thus, indicators can be used over definite pH ranges.It should be emphasized, though, that although complexometricindidators respond to pH, their mechanism of action does not involve changes in pH, as the solution is buffered. But the pH affects the stability of the complex formed between the indicator and the metal ion, as well as that formed between EDTA and the metal ion. An indicator is useful for indication of titrations of only those metals that form a more stable complex with the titrant than with the indicator at the given pH. This may sound complex but suitable indicators are known for many titrations with several different chelating agents.

Calmagite gives a somewhat improved end point over Eriochrome Black T forthe titration of calcium and magnesium with EDTA. It also has a longer shelf life.Xylenol orange is useful for titration of metal ions that form very strong EDTAcomplexes and are titrated at pH 1.5 to 3.0. Examples are the direct titration ofthorium(IV) and bismuth(III), and the indirect determination of zirconium(IV) andiron(III) by back-titration with one of the former two metals. There are many other indicators for EDTA titrations.

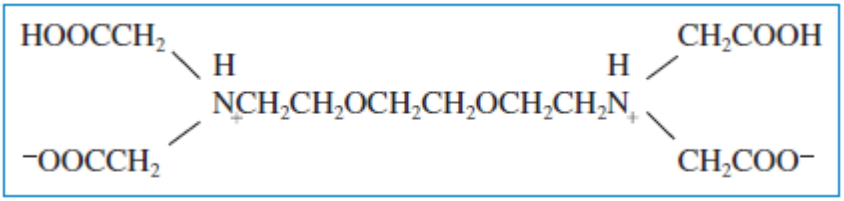


Calmagite



Xylenol orange

There are a number of other useful reagents for complexometric titrations. A notable example is ethyleneglycolbis (*β*-aminoethyl ether)-N, N, N_, N_-tetraaceticacid (**EGTA**). This is an ether analog of EDTA that will selectively titrate calcium inthe presence of magnesium:



Complexing agents having ether linkages have a strong tendency to complex the alkaline earths heavier than magnesium.

Complexometric titrations in the clinical laboratory are limited to those substances that occur in fairly high concentrations since volumetric methods are generally not too sensitive. The most important complexometric titration is the determination of calcium in blood. Chelating agents such as EDTA are used in the treatment of heavy-metal poisoning, for example, when children ingest chipped paint that contains lead. The calcium chelate (as Na_2CaY) is administered to prevent complexation and use of Ca-EDTA, rather than Na_2EDTA, prevents leaching of calcium in the bones. Heavy metals such as lead form more stable EDTA chelates than calcium does and will displace the calcium from the EDTA. The chelated lead is then excreted via the kidneys.

S.No	Question	a	b	c	d	Answer
1.	What is a a soil horizon	A factor influencing how soil is formed	A layer of soil	An organism found within the soil	A technique used to map the soil	A layer of soil
2.	Which three layers form the soil profile	Air. water and soil	Minerals, organic matter and living organisms	Clay, silt and sand	The topsoil, subsoil and parent material	The topsoil, subsoil and parent material
3.	How does a 'sandy' soil feel like to touch?	Sticky	Gritty	smooth	muddy	Gritty
4.	How does a 'sandy' soil feel like to touch?	It helps to improve water infiltration	It can break down organic pollutants	It converts nitrogen in the air into nitrates used by plants	It is rich in nutrients, which is important for fertility	It is rich in nutrients, which is important for fertility
5.	Which of the following is NOT a common reason why soil maps are used	To determine the land drainage capabilities of an area	To determine the suitability of soils for particular crops	To identify soils and their properties	To record how soils are used by people	To record how soils are used by people
6.	Approximately how many micro-organisms can be found in a teaspoonful of soil	4 billion	50 million	500,000	1000	4 billion
7.	Which of the following creatures will you NOT find in the soil	Earthworm	Mites	springtail	lemur	lemur
8.	Which of the following is NOT a threat commonly faced by soils	Soil erosion	Soil percolation	deforestration	Climate change	Soil percolation
9.	What is soil erosion	It is the process by which soil is formed	A harmful process that involves the removal and transport of soil by wind and water	A natural method of filtering harmful pollutants	A process often referred to as the 'greenhouse' effect	A harmful process that involves the removal and transport of soil by wind and water

10.	A harmful process that involves the removal and transport of soil by wind and water	Soil erosion	Soil percolation	deforestration	Climate change	
11.	What effect can soil have on health if eaten or inhaled	Nothing-it is perfectly safe	It can be good for your health	It can have serious health implications like cancer	If inhaled it is poisnous	It can have serious health implications like cancer
12.	Which soil layer is made up of small lumps of rocks	A-horizon	B-horizon	C-horizon	Bed rock	C-horizon
13.	Which soil layer contains humus	A-horizon	B-horizon	C-horizon	Bed rock	A-horizon
14.	In addition to rock particles, the soil contains,	Air and water	Water and plant	Minerals, organic matter, air, water	Water air plants	Minerals, organic matter, air, water
15.	What is the key component of the soil which makes it more cohesive	clay	sand	silt	humus	clay
16.	Soil profile refers to an arrangement within a soil of	Its horizontal layout	Vertical layout	Diagonal layout	Size of soil particles	Its horizontal layout
17.	Which of the following soil is loosely packed with large air spaces	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Sandy soil
18.	Percolation rate of water is least in	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Clayey soil
19.	Lentils and other pulses are grown in	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Loamy soils
20.	Toys, statues, and pots are made up of	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Clayey soil
21.	Water holding capacity is highest in	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Clayey soil
22.	Which top soil is best for growing plants	Sandy soil	Clayey soil	Loamy soil	Rocky soil	Loamy soil

23.	Soil formation is a	Slow process	Fast process	Rapid process	Ultra fast process	Slow process
24.	Soil is filled with water is termed as	soluble	hydrated	saturated	H-bonded	saturated
25.	Soil exists in how many states	one	two	three	four	four
26.	Most soils have a particle density of	2.6 g/cc	3.6 g/cc	4.6 g/cc	5.6 g/cc	2.6 g/cc
27.	In oven drying method for the determination of water content, the temperature maintained is	100 – 105^{0}C	150 – 165^{0}C	105 – 110^{0}C	110 – 120^{0}C	105 – 110^{0}C
28.	How many tests can be performed in the lab to get permeability of a soil	2	3	4	5	2
29.	How many grades of soil are there	4	5	6	7	4
30.	How many types of pores are present in soil mass	3	4	5	6	5
31.	*Which of the following soil pH levels indicates an alkaline soil*	PH = 5.5	PH = 6.5	PH = 7.5	PH = 8.5	PH = 8.5
32.	If pH value is greater than 7, then solution is	acidic	Basic	Neutral	salty	Basic
33.	PH of water is	3	5	7	9	7
34.	Amount of Hydrochloric Acid (HCl) secreted daily by gastric glands is	1 lit	2 lit	3 lit	4 lit	2 lit
35.	As an electrolyte, water is	neutral	strong	weak	A good insulator	weak
36.	What is the hydrogen ion concentration in pure water	1 x 10^{-7}M	1 x 10^{7}M	1 x 10^{-14}M	1 x 10^{14}M	1 x 10^{-7}M

37.	Water is having a neutral PH, the best explanation is	PH of pure water is 8	The hydrogen ion and hydroxyl ion concentration are same	The hydrogen ion and hydroxyl ion concentration are different	Water will never ionise	The hydrogen ion and hydroxyl ion concentration are same
38.	EDTA method is also called as_____	Complexometric titration	Complex titration	Complement titration	Simple acid base titration	Complexometric titration
39.	The indicator used in the EDTA method is_____	Methyl orange	phenopthalein	Erichrome black T	Phenol red	Erichrome black T
40.	EDTA has the ability to form _____ with metal ions	Stable complexes	Unstable complexes	salts	acids	Stable complexes
41.	The colour of dye metal complex and dye are _____	Same	Not known	different	Same in few cases	different
42.	In EDTA titrations the sharp colour change occurs at PH	1	3	5	10	10
43.	At PH=10, the metal dye complex has the colour_____	Wine red	Blue	green	pink	Wine red
44.	_____drops of indicator is used in the EDTA method.	1 to 2 drops	4 to 5 drops	5 to 6 drops	10 drops	1 to 2 drops
45.	The buffer used in the EDTA solution must have the PH of_____	5	10	15	20	10
46.	The standard hard water is prepared such that each ml must contain _____ mg of $CaCO_3$.	1	2	3	4	1
47.	EDTA has _____ binding sites and therefore it is also called as multidentate ligand.	four	five	six	seven	four

48.	_____agent forms the complex with the metal ions that are not required in the estimation	Masking agent	Demasking agent	chelates	analytes	Masking agents
49.	What conditions should be respected in order to carry out the complexometric titration	presence of a buffer solution	presence of a catalyst;	Low temperature	[metal-EDTA] complex should be more stable than [metal-indicator] complex;	[metal-EDTA] complex should be more stable than [metal-indicator] complex;
50.	Which of the following can be used to determine the end point in a complexometric titration	Redox indicators;	Adsorption indicators;	Metallochromic indicators	Specific indicators	Metallochromic indicators
51.	The titration brake of the complexometric titration curve depends on	Concentration of the analyte	Concentration of the titrant	PH	Order of a titration	PH
52.	Which of the following would cause the sharpest change at the equivalent point of the complexometric titration	Increasing the concentration of an analyte	decreasing the concentration of an analyte	Increasing the stability of the [metal-EDTA] complex	Increasing the concentration of the EDTA solution	Increasing the stability of the [metal-EDTA] complex
53.	Which of the following soil compounds can be analyzed by direct complexometric titration	CH2O	FeSO4	NH4Cl	MgSO4	MgSO4
54.	Which of the following soil compounds can be analyzed by direct complexometric titration	CH2O	FeSO4	NH4Cl	$CaCO_3$	$CaCO_3$
55.	Which of the following drug compounds CANNOT be analyzed by direct complexometric titration	ZnSO4	CuSO4	KI	Calcium gluconate	KI

56.	Which of the following direct methods can be applied for the quantitative determination of CaSO4	Iodometric titration	Complexometric titration	Gravimetric analysis	Acidimetric titration	Complexometric titration
57.	Which of the following direct methods can be applied for the quantitative determination of MgSO4	Iodometric titration	Complexometric titration	Gravimetric analysis	Acidimetric titration	Complexometric titration
58.	Which of the following analysis refers to a complexation titration	titration of halides with Hg2(NO3)2	titration of halides with Hg(NO3)2	titration of barium salts with H2SO4	titration of calcium chloride with EDTA	titration of calcium chloride with EDTA
59.	Which of the following analysis refers to a complexation titration	titration of halides with Hg2(NO3)2	titration of halides with Hg(NO3)2	titration of barium salts with H2SO4	titration of calcium chloride with EDTA	titration of magnesium sulphate with EDTA
60.	Which of the following statements about EDTA solution are correct	It is always prepared by a standardization method	It can be standardized against primary standard solution of MgSO4	It can be standardized against primary standard solution of NaCl	It should be standardized 7-10 days after preparation	It can be standardized against primary standard solution of MgSO4

UNIT-3

SYLLABUS

Analysis of water:

Definition of pure water, sources responsible for contaminating water,water sampling methods, water purification methods.

a. Determination of pH, acidity and alkalinity of a water sample.
b. Determination of dissolved oxygen (DO) of a water sample.

INTRODUCTION:

Pure water, also known as purified water, is water from a source that has removed all impurities. Distilled water is the most common form of pure water. Pure water can be purified by carbon filtration, micro-porous filtration and ultraviolet oxidation. Some places use a combination of purification processes.

COMMON IMPURITIES

Suspended Particles

Sand, silt, clay and other suspended particles cause water to be turbid. These suspended particles can interfere with instrument operation, plug valves and other narrow flow paths, and foul reverse osmosis membranes. They typically range from 1 to 10µ in size.

Colloids

Colloidal particles typically have a slightly net negative charge, range in size from 0.01-1.0µm, and can be either organic or inorganic. Unlike suspended particles, colloids do not settle out by gravity, but remain suspended in the liquid that carries them. Colloids clog filters, interfere with instrument operation, foul reverse osmosis membranes and can bypass ion exchange resins, resulting in lower resistivity in deionized water systems.

Inorganic Ions

Impurities such as silicates, chlorides, fluorides, bicarbonates, sulfates, phosphates, nitrates and ferrous compounds are present as cations (positively charge ions) and anions (negatively charged ions). Water with a high concentration of ions will conduct electricity readily and have high conductivity and low resistivity, as conductivity and resistivity are inversely related. Ions will adversely affect the results of inorganic analyses such as IC, AA, ICP/MS, and may retard cell and tissue growth in biological research. They can also affect the cartridge life in deionized water systems.

Dissolved Organics

Organic solids are present from plant and animal decay and from human activity. They may include proteins, alcohols, chloramines, and residues of pesticides, herbicides and detergents. They foul ion exchange resins, interfere with organic analyses including HLC, gas chromatography and fluoroscopy. They will also hinder electrophoresis, tissue and cell culture.

Dissolved Gases

Water naturally contains dissolved gasses such as carbon dioxide, nitrogen and oxygen. Carbon dioxide dissolves in water to form weakly acidic carbonic acid (H^2CO^3), which can alter the pH of the water. Additionally, oxygen, the most common non-ionized gas, may cause corrosion of metal surfaces.

Microorganisms

Bacteria, fungi and algae are found in all natural water souorces. Chlorinatin eliminates harmful bacteria, but tap water still contains live microoganisms which interfere with sterile applications, such as cell and tissue culture.

Pyrogens and Viruses

Pyrogens or bacterial endotoxins are lipopolysaccaride molecules incorporated in the cell membrane of gram negative bacteria. Viruses are considered to be non-living nucleic acids. Both can adversely affect laboratory experiments often hindering cell and tissue growth in culture.

Nucleases

RNase and DNase are naturally occurring enzymes that are instrumental in regulating bodily functions. As important as these enzymes are to the life process, they can be devastating to nucleic acid experiements. If these contaminants are present in the pure water used the ability to amplify DNA molecules will be severely limited. Likewise, experiments utilizing RNA can be ruined.

WATER PURIFICATION TECHNOLOGIES

Water purification is a step-by-step process often requiring a combination of technologies, each of which varies in it's ability to remove specific contaminants.

DISTILLATION

Distillation has the broadest removal capabilities of any single form of water purification. Water is boiled and undergoes phase changes during the distillation process, changing from liquid to vapor and back to liquid. It is the change from liquid to vapor that separates the water (in various degress) from many dissolved impurities, such as ions, organic contaminants with low boiling points (<100ºC / 212ºF), bacteria, pyrogens, and particulates. Distillation can not be used on its own to remove inorganic ions, ionized gases, organics with boiling points higher than 100°C, or dissolved non-ionized gases.

Benefits

- Offers the broadest removal capabilities of any single form of water purification
- Requires no consumables

Limitations

- Requires periodic maintenance and manual cleaning of system to maintain water purity
- Requires water for cooling

Systems that utilize this Technology

- Classic Still, Mega-Pure Stills, and FI-Streem Stills

FILTRATION

Thermo Scientific Barnstead water products offer both depth (nominal) and membrane (absolute) filters.

Depth filters are most commonly used as a pretreatment and are manufactured by winding fibers around hollow and slotted tubes. As water passes through the wound fiber matrix toward the center tube, particles are retained on the fibers. Traditionally this type of filter removes most of the impurities above the rated pore size of the filter. Most often these filters are rated to remove larger particles (>1 µm) to protect the technologies that follow.

Membrane filters are often termed absolute, meaning that they are designed to remove all particles above the rated pore size of the filter. These filters use a membrane (in flat sheet or hollow fiber form) and are most often used at the end of a system to remove bacteria or other particles that are not removed by the preceding technologies. Traditionally membrane filters in laboratory water systems have a rated pore size below 0.45 µm, most often to 0.2 µm.

Benefits

- Efficient operation
- Maintenance is change out only

Limitations

- Clogging
- Will not remove organics, nucleases, pyrogens, dissolved gases or dissolved inorganics

Systems that utilize this Technology

- Nanopure, Easypure II, TII, E-Pure and Barnstead RO

ULTRAFILTRATION (UF)

In water purification, ultrafiltration is used to remove pyrogens (bacterial endotoxins) and nucleases, which is critical for tissue culture, cell culture and media preparation.

Ultrafilters use size exclusion to remove particles and macromolecules. By design, ultrafilters operate similar to reverse osmosis membranes; particles are captured on the surface of the membranes and are flushed from the membrane via a reject stream. Ultrafilters are used at the end of systems ensuring the near total removal of macromolecular impurities like pyrogens, nucleases and particulates.

Benefits

- Effectively removes molecules (pyrogens, nucleases, microorganisms, particulates) above their rated size
- Long life
- Helps to remove pyrogens and nucleases

Limitations

• Will not remove dissolved inorganics, dissolved gases and organics

Systems that utilize this Technology

• Nanopure and Easypure II

REVERSE OSMOSIS

Reverse osmosis is the most economical method of removing up to 99% of your feed water's contaminants.

To understand reverse osmosis we must first understand osmosis. During natural osmosis, water flows from a less concentrated solution through a semipermeable membrane to a more concentrated solution until concentration and pressure on both sides of the membrane are equal.

In water purification systems, external pressure is applied to the more concentrated (feed water) side of the membrane to reverse the natural osmotic flow. This forces the feed water through the semipermeable membrane. The impurities are deposited on the membrane surface and sent to drain and the water that passes through the membrane as product water is, for the most part, free of impurities.

A reverse osmosis membrane has a thin microporous surface that rejects impurities, but allows water to pass through. The membrane rejects bacteria, pyrogens, and 90-95% of inorganic solids. Polyvalent ions are rejected easier than monovalent ions. Organic solids with a molecular weight greater than 200 Daltons are rejected by the membrane, but dissolved gases are not as effectively removed.

Reverse osmosis is a percent rejection technology. The purity of the product water depends on the purity of the feed water. The product is typically 95-99% higher in purity than that of the feed water.

Due to the restrictive nature of the membrane, the flow rate is much slower than other purification technologies. This slow flow rate means that all RO systems require a storage tank to provide a constant supply of RO water ready when you need it.

Benefits

• To varying degrees, removes most types of contaminants, bacteria, pyrogens, and 90-95% of inorganic ions

• Requires minimal maintenance

Limitations

• Limited flow rates through the membrane require intermediate storage devices to meet user demand

• Does not remove dissolved gases

• Requirespretreatment to avoid damaging the membrane

>Oxidation - Chlorine

>Scaling - $CaCO^3$

>Fouling - Organics and Colloids

>Piercing - Hard particles

Systems that utilize this Technology

• Barnstead RO, TII, and EasypureRoDi

DEIONIZATION

Deionization is also referred to as demineralization or ion exchange. The process removes ions from feed water with the use of synthetic resins. These resins are chemically altered to have an affinity for dissolved inorganic ions and are divided into two classifications: cation removal resins and anion removal resins.

Cations have a positive charge and include sodium (Na^+), Calcium (Ca^{+2}), and Magnesium (Mg^{+2}). Anions have a negative charge and include chloride (CI-), sulfates ($SO4^{-2}$), and bicarbonates (HCO^{-2}). The ions are removed from the water through a series of chemical reactions. These reactions take place as the water passes through the ion exchange resin beds. cation resin contains hydrogen (H^+) ions on the surface which are exchanged for positively charged ions. Anion resin contains hydroxide (OH^-) ions on its exchange sites which are exchanged for negatively charged ions. The final product of these two exchanges is H^+ and OH^-, which combine to form water (H^2O).

Deionization is the only technology which produces the resistivity requirement for Type 1 reagent grade water. In laboratory water systems, cation and anion resins are most often mixed together allowing them to achieve maximum ionic purity.

Two bed deionization - The cation and anion resin are in separate halves of a cartridge. In general, this method is less effective deionizing water as compared to the mixed bed deionization, however, it is more tolerant of other types of impurities.

Mixed bed deionization - We use semi-conductor grade mixed bed deionization resin to achieve maximum resistivity and low TOC. Mixing the cation and anion resin drives the deionization to completion, making it more efficient and more effective at the removal of ions. This is the most effective method of removing ions.

Benefits

- Removes dissolved inorganics ions very effectively
- Produces product water with a resistivity above 18Ω-cm

Limitations

- Finite capacity - once all ion binding sites are occupied ions are no longer retained and the cartidge must be replaced
- Does not remove organics, particles, pyrogens or bacteria

Systems that Utilize this Technology

- Nanopure, Easypure II, EasypureRoDi, E-Pure, TII, Barnstead RO, Bantham Cartridges, Hose Nipple Cartridges and B-Pure Cartridges

ADSORPTION

Adsorption uses high surface area activated carbon to remove organics and chlorine from feed water. It is used as a first or second step in most water purification systems and may be used as a final step, in combination with ion exchange resins, to achieve ultra low Total Organic Carbon (TOC). Organics and chlorine adhere to the surface of the activated carbon and remain attached to the carbon.

Mixed bed deionization and adsorption - We use a combination semiconductor grade mixed bed deionization resins and synthetic carbon in a single cartridge to achieve maximum resistivity and low Total Organic Carbon (TOC).

Benefits

- Removes dissolved organics and chlorine

• Long Life

Limitations

• Will not remove ions and particulates

Systems that Utilize this Technology

• Nanopure, Easypure II (including RF [Reservoir Feed]), EasypureRoDI, E-Pure, TII, Barnstead RO, Bantham Cartridges, Hose Nipple Cartridges and B-Pure Cartidges

ULTRAVIOLET (UV) OXIDATION

Photochemical oxidation with ultraviolet light eliminates trace organics and inactivates microorganisms in feed water. THe UV lamps in our pure water systems generate light at two wavelengths, 185 and 254 nm. The light generated at 254 nm has the greatest anti-bacterial action, reacting with their DNA, resulting in inactivation. The combination 185/254 nm light oxidizes organic compounds, allowing for total oxidizable carbon levels of less than 5 ppb.

Benefits

• Effective method to prevent bacterial contamination

• Oxidizes organics to produce pure water with low TOC levels

Limitations

• Will not remove ions, colloids, and particulates

Systems that Utilize this Technology

• Nanopure, Easypure II, EasypureRoDi, TII, and select reserviors

COMBINATION ULTRAVIOLET OXIDATION AND ULTRAFILTRATION (UV/UF)

The use of ultraviolet oxidation and ultrafiltration technologies in conjunction with adsorption and deionization in the same system produces water virtually free of all impurities. These technologies have demonstrated the ability to remove nucleases such as RNase and DNase as well as pyrogens when challenged with known concentrations of the material. The Type 1 systems with UV/UF options produce reagent grade water with resistivity up to 18.2 MΩ-cm, TOC of 1-5 ppb, pyrogens<0.001 EU/ml and no detectable RNase, DNase or DNA.

Benefits

- Removes nucleases and DNA
- Produces water with low TOC and pyrogen levels

Limitations

- Must be used in the same system

Direct sources include effluent outfalls from factories, refineries, waste treatment plants etc.. that emit fluids of varying quality directly into urban water supplies.

Categories of Water Contaminants

Water contaminants fall into four basic categories:

1. Aesthetic: Offensive tastes and odors that come from natural and unnatural sources; sediment, dirt, sand or particulates that affect taste; and minerals that affect taste and can become a nuisance for plumbing fixtures;

2. Biological: Pathogens that have serious or deadly effects on human health, including bacteria (such as E. coli, Salmonella, Shigella and Legionella), cysts and parasites (such as Giardia, Cryptosporidium and tapeworms), and viruses (such as hepatitis A and poliovirus);

3. Chemical: Volatile organic compounds, chlorine, chloramines, pesticides, herbicides, and inorganic chemicals, such as nitrates; and

4. Dissolved solids: Minerals (such as calcium and magnesium) and heavy metals (such as iron, manganese, lead, mercury, cadmium, chromium, arsenic, aluminum, copper, radon and barium).

Water Sample collection

Sampling is to collect a portion of material small enough in volume to be transported comfortably and yet large enough for analytical purposes while still representing the material being sampled.

Selection of sample containers

• Selection of sample container is utmost importance in sampling. Containers are generally made of glass or plastic. Some sample analytes may get absorbed into the walls of plastic containers and/or some contaminants may leach into samples.

• Trace level of some metals and pesticides may get adsorbed and/or absorbed onto the walls of the glass container. In the same way, silica, sodium, and boron may be leached from soft glass.

• Always use hard glass containers for all organics analyses such as pesticides, volatile organics, PCBs, and oil & grease.

• Some of the analytes like pesticides, PAH etc. are light sensitive. Hence collect them in amber-coloured glass containers to minimize photo degradation.

Selection of type of sampling

• Grab

• Composite

• Integrated

Grab sampling

• Grab samples are also called as spot or catch samples. Grab samples are single samples collected at a specific spot at a site in specified time. Grab samples are to be collected only when the source is known to be constant in composition for an extended period of time. Examples are, ground water samples, well mixed surface waters, large lakes, rivers, estuaries, shorelines, wastewater streams that are expected to be constant in composition over an extended period of time, like spent wash line in a distillery.

• When the source composition varies from location to location, like upstream and downstream of a river, then grab samples can be collected from appropriate locations. This helps in finding out the extent of variation and duration of variation.

Composite sampling

• Composite sampling is carried out when the liquid matrix is expected to be heterogeneous and varies from time to time or depth or at many sampling locations. This type of sampling provides a representative sampling for this type of matrix and is carried out by combining portions of multiple grab samples collected at regular intervals. If the flow is expected to be constant, then volume based sampling can be carried out. If the flow varies, like sewerage line, then sampling can be done by flow based composite, i.e., collecting sample that is proportional to the discharge. Time composite sampling represents a 24- hour period, with interval being 1-3 hours.

Use composite samples only for parameters that will remain unchanged under the sampling conditions, preservation and storage. For parameters like pH, temperature, residual chlorine, carbon dioxide, alkalinity, sulfide, dissolved oxygen, Oil&Grease etc. avoid composite sampling

and analyse individual samples as soon as possible, preferably in the field itself, except for sulfide and Oil &Grease.

Integrated sampling

• Integrated sampling is carried out by collecting mixture of grab samples collected from different points simultaneously. The points may be horizontal or vertical variation. Examples include river, stream or reservoir or lake that varies in composition across the width and depth. Also in industries that have different streams and combined treatment is proposed, than integrated sampling of different streams can be made to understand the significant effect on treatment.

Selection of sampling points

• Selection of sampling points plays an important role in sampling. The site selection should be based on the objective of the study. If the monitoring is carried out for judging suitability of water for drinking purpose, then the sampling point shall be near the intake point. Always samples must be taken from locations that are representative of the source, treatment plant, storage facilities, point of discharge, and point of use. Eventhough there is no methodology for site selection on a cook book basis some general basic rules can be followed to have a sound sampling programme

Always have a reference station upstream of any discharge point like industrial outfall, city sewage drain etc. The reference point allows us to ascertain the background water quality. Additional downstream stations can be fixed to assess the extent of influence of discharge and to find the recovery point The points chosen should generally yield samples that are representative of the system as a whole. For this it is important to select a well-mixed zone. When samples are collected from a river or stream, the observed results may vary with depth, flow and distance from the shore. Hence, if equipments are available collect an integrated sample from top to bottom in the middle of the lake or river or from side to side at mid-depth. Otherwise, preferably collect samples at various points of equal distance across the water body. If only one sample can be collected, collect it in the middle of the water body at mid depth Avoid areas of turbulence and at weirs. Generally collect samples beneath the surface with the mouth directed towards the current. For oil and grease, collect sample at the surface. In case of groundwater sampling, select wells that are in continuous use.

Sample labeling

• Labeling is an important part in sampling programme. The following information should be included in the label. Use water proof ink to record all the information.

• Date and time of sampling

• Sample field code

• Sampling point

• Nature of sample: Effluent / Surface water / Ground water / Others

• Type of sample (Grab/Composite/Integrated) • Pre-treatment or preservation carried out on the sample

• Any special notes for the analyst

• Name and sign of sample collector.

Dissolved oxygen measurement in biological flocs

This modification is used to determine DO for biological flocs such as activated sludge process, which have high oxygen utilization rate.

Requirements: 1 lit. reagent bottle, siphon tube, BOD bottle, reagent Add 10mL Copper sulphate-sulfamic acid inhibitor to a 1 litre glass stoppered bottle. Collect sample, stopper and mix by inverting. Allow the suspended solids to settle and siphon the supernatant into a 300mL BOD bottle. Add 1 mL manganoussulphate solution, followed by 1mL alkali-iodide-azide reagent by holding pipette just above liquid surface. Stopper carefully and mix by inverting bottle few times.

(i) Alkalinity:

The alkalinity of the water is a measure of its capacity to neutralize acids. The alkalinity of natural waters is due primarily to the salts of week acids. Bicarbonates represent the major form of alkalinity. Alkalinity can be expressed as follows:

Alkalinity (mol/L) = $[HCO_3^-] + 2[CO_3^{2-}] + [OH^-] - [H^+]$ (1)

Figure 1 presents the carbonate speciation diagram at different pH values. Waters rich in bicarbonates (HCO_3^-) have high acid neutralizing capacity (high alkalinity).

Alkalinity is significant in many uses and treatments of natural waters and waste waters. As alkalinity of many surface waters constitute of carbonates, bicarbonate and hydroxide contents, it is assumed to be an indicator of these constituents as well. Alkalinity in excess of alkaline earth metal concentrations is significant in determining the suitability of water for irrigation. Alkalinity measurements are used in the interpretation and control of water and wastewater

treatment processes. Raw domestic wastewater has an alkalinity less than or only slightly greater than that of the water supply.

(ii) Acidity:

Acids contribute to corrosiveness and influence chemical reaction rates, chemical speciation and biological processes. Acidity of water is its quantitative capacity to react with a strong base to a designated pH. The measured value may vary significantly with the end point pH used in the determination. When the chemical composition of the sample is known study mineral acids, weak acids such as carbonic and acetic and hydrolyzing salts such as iron or aluminumsulfate may contribute to the measured acidity according to the method of determination.

Mineral acidity: It is measured by titration to a pH of about 3.5, the methyl orange end point (also known as methyl orange acidity).

Total acidity: Titration of a sample to the phenolphthalein end point of pH 8.3 measures mineral acidity plus acidity due to weak acids, thus this is called as total acidity (or phenolphthalein acidity).

In water analysis, this test does not bear significant importance because methyl orange acidity invariably remains absent in the raw water and even phenolphthalein acidity (that too principally due to the excessive-prevalence of dissolved carbon dioxide and carbonic acids) normally does not exist to a significant extent in the raw water.

Lab Procedure

(i) Alkalinity:

pH meter; Reagents for alkalinity (H_2SO_4 (0.02N); Methyl Orange Indicator;

Phenolphthalein Indicator)

1. Collect 50 mL water sample, add 3 drops of phenolphthalein indicator, titrate the 50 mL sample with 0.02N sulfuric acid to pH 8.3 and estimate phenolphthalein alkalinity (Eq. 2a) (phenolphthalein indicator will change color, from pink to clear, at pH 8.3).

Phenolphthalein Alkalinity (in mg/L as $CaCO_3$) = ($A1 \times N \times 50{,}000$) / V (2a)

Where: A1 = volume of sulfuric acid used in mL; N = normality of acid used to titrate;

V= volume of sample used in mL

2. Use the same sample. Add 3 drops of bromcresol green indicator. Titrate the 50 mL sample with 0.02N sulfuric acid to pH 4.5 and estimate total alkalinity (bromcresol green indicator will change color, from blue to yellow, at pH 4.5). Amount of acid used at this moment starting from step1 (i.e., A2) is used to react with the hydroxide, carbonate, and bicarbonate and it constitutes of total alkalinity (Eq. 2b):

Total Alkalinity (in mg/L as $CaCO_3$) = ($A \times N \times 50{,}000$) / V (2b)

Where: A2 = volume of acid used in mL starting from step 1 (i.e., A2>A1); All other parameters are defined in Eq. 2a.

(Note: If after adding phenolphthalein indicator no colour develops, it means no phenolphthalein alkalinity and it can be reported as "Phenolphthalein alkalinity absent".)

Calculation from Alkalinity and pH measurements:

Hydroxide alk. (mg/L as $CaCO_3$)= $50{,}000 \times 10[pH-pKw]$; pKw= 15 at 24°C (3a)

Carbonate alk. (mg/L as $CaCO_3$)= 2 × [Phenolphthalein alk.-hydroxide alk.] (3b)

Bicarbonate alk. (mg/L as $CaCO_3$)= Total alk.-[Carbonate alk.+ hydroxide alk.] (3c)

(ii) Acidity:

pH meter; Reagents: Sodium hydroxide titrant (0.02 N); Phenolphthalein Indicator; Methyl Orange Indicator

1. Take 50 ml sample in a conical flask and add 2-3 drops of methyl orange indicator solution.

2. Fill the burette with 0.02 N NaOHsolution and titrate till the colour of solution just changes to faint orange colour, indicating the end point. Record the volume of titrant consumed as V1 in ml. Calculate the methyl orange acidity using Eq (4a):

Methyl orange acidity (or Mineral Acidity) = (V1×1000)/(Sample volume) (4a)

When the 0.02 N NaOH solution, used in titration is not standardized, mineral acidity is calculated using following Eq (4b):

Methyl orange acidity= (V1×N×50×1000)/ (Sample vol.) (4b)

3. For phenolphthalein acidity test, add 2-3 drops of phenolphthalein indicator solution to water sample from step 2 and continue the titration till the faint pink colour develops in the solution (i.e., the end point of titration). Record the volume of titration consumed as V2 (mL) and calculate total acidity or phenolphthalein acidity using Eq.(5):

Total acidity (or Phenolphthalein Acidity)= (V2×N×50×1000)/ (Sample vol.) (5)

S.No	Question	a	b	c	d	Answer
1.	The following unit is not used to measure turbidity of water?	NTU	ATU	JTU	FTU	ATU
2.	The water temperature should preferably be less than __ degree Celsius.	10	15	25	30	25
3.	technique used to determine the concentration of odour compounds in a sample is known as	Stripping	Settling	Flushing	Chlorination	Stripping
4.	In filtration, the amount of dissolved solids passing through the filters is	Difference between total solids and suspended solids	Sum of total solids and suspended solids	Independent of suspended solids	Independent of total solids	Difference between total solids and suspended solids
5.	The Total dissolved solids (TDS) can be reduced by the following method	Distillation	Reverse osmosis	Ion exchange	Ion exchange, Reverse osmosis and distillation	Ion exchange, Reverse osmosis and distillation
6.	water having less than 1000 ml/litre of total dissolved solids is	Fresh water	Slightly saline	Moderately saline	Brine water	Fresh water

7.	The following cause alkalinity in natural water.	Potassium carbonate	Potassium bicarbonate	Sodium carbonate	Potassium carbonate, Potassium bicarbonate and Sodium carbonate	Potassium carbonate, Potassium bicarbonate and Sodium carbonate
8.	The following cause alkalinity as well hardness in natural water	Calcium carbonate, Calcium bicarbonate and Magnesium carbonate	Calcium carbonate	Calcium bicarbonate	Magnesium carbonate	Calcium carbonate, Calcium bicarbonate and Magnesium carbonate
9.	The following cause hardness in natural water	Calcium carbonate, Calcium bicarbonate and Magnesium carbonate	Calcium carbonate	Calcium bicarbonate	Magnesium carbonate	Calcium carbonate, Calcium bicarbonate and Magnesium carbonate
10.	Temporary hardness is caused due to	Magnesium carbonate	Calcium sulphate	Magnesium sulphate	Magnesium chloride	Magnesium carbonate
11.	Permanent hardness is caused due to	Magnesium carbonate and Magnesium bicarbonate	Magnesium carbonate	Magnesium bicarbonate	Magnesium sulphate	Magnesium sulphate
12.	According to WHO, the soft water has 0 to _____ milligram per litre as $CaCO_3$.	30	60	90	120	60
13.	The excess presence of which of the following	Fluorides	chlorides	Hardness	Iodides	Fluorides

	cause the teeth of children mottled and discoloured?					
14.	Fluorides can be removed by	Reverse osmosis	Reverse osmosis, Lime softening and Ion exchange	Lime softening	Ion exchange	Reverse osmosis, Lime softening and Ion exchange
15.	The source of Arsenic in water is	Industrial waste	Fertilizers	Phosphate rocks	Fertilizers, industrial wastes and Phosphate rocks	Fertilizers, industrial wastes and Phosphate rocks
16.	The process of nutrient enrichment is termed as	Eutrophication	Limiting nutrients	Enrichment	Schistosomiasis	Eutrophication
17.	Freshwater lakes are most often limited by	Nitrogen	phosphorous	carbon	potassium	Phosphorous
18.	Which of the following is not a water borne disease?	Typhoid	Scabies	Cholera	Hepatitis	Scabies
19.	Which of the following is not a water hygiene disease?	Leprosy	conjuctivities	Trachoma	Diarrhoea	Diarrhoea
20.	The introduction of chemical, physical or biological agents into the water is	desalination	potable water	Point source pollution	Water pollution	Water pollution
21.	Fertilizer from farms can cause	increase oxygen in water	artifical eutrophication	potable water	desalination	artifical eutrophication
22.	Artifical eutrophication can	increase oxygen in water	never be reversed	increase fish's reproductive rate	harm commerical fisherman's income	harm commerical fisherman's income
23.	Pathogens are	fertilizers	Disease causing bacteria and	Drinkable water	Chemical pollutants	Disease causing bacteria and virus

			virus			
24.	A likely pH of acid rain could be	6.9	4.7	7.6	5.8	4.7
25.	The effects of water pollution on an ecosystem	can become worse due to biomagnification	are concentrated in one area	result mostly from point source locations	are always immediate	can become worse due to biomagnification
26.	Most of the pollution in the ocean comes from	commerical boats	waste from land	leaking tankers	Oil spills	Oil spills
27.	Chemical substance used to destroy weeds in crops is called	herbicides	monoxides	carob-oxides	pesticides	pesticides
28.	Natural source of pollution is	Rain forest	Mining of minerals	Forest fire	Falling of meteroites	Forest fire
29.	In extreme cases of eutrophication, water bodies no longer support	Pollution	Fishes	Nutrients	algae	Fishes
30.	What are most common metals found in industrial processing	Mercury, Lead & Copper	Manganese, Nickel, & Arsenic	Toxic industrial waste	Mercury, Lead &Copper,Manganese, Nickel, & Arsenic	Mercury, Lead &Copper,Manganese, Nickel, & Arsenic
31.	Fluoride is also added to water, which helps in preventing	Infection	Sickness	fever	Tooth decay	Tooth decay
32.	Tanks that supply water to towns are built at	Roof of buildings	High towers	Town level	High lands	High towers
33.	An addition of small dose of chlorine gas to filtered water is known as	coagulation	sedimentation	filtration	chlorination	chlorination
34.	Process in which water is passed through filter beds of sand and gravel to	coagulation	sedimentation	filtration	chlorination	filtration

	remove smaller particles of dust is called					
35.	Huge strainers are used to	force water to the treatment plant	increase the water pressure	filter large particles in the water	filter bacteria and other harmful organisms	filter large particles in the water
36.	The acceptable value of pH of potable water is	6.5 to 8.5	7.0 to 9.5	6 to 8.5	6.5 to 10	6.5 to 8.5
37.	Which three factors reduce dissolved oxygen levels in water?	high Temperture	decay of organic matter by bacteria	Respiration by aquatic organisms	High temperature, decay of organic matter by bacteria and Respiration by aquatic organisms	High temperature, decay of organic matter by bacteria and Respiration by aquatic organisms
38.	Dissolved oxygen levels are important because	Light penentration is affected	Salinity can increase	It is important for aquatic animals	Not required by living things	It is important for aquatic animals
39.	The way in which oxygen can enter water is	Respiration of aquatic organisms	By rain	Photosynthesis of green aquatic organisms	Water mixing with air in waterfalls, rapids and rain	Water mixing with air in waterfalls, rapids and rain
40.	Biological oxygen demand indicates	Dissolved oxygen levels	turbidity	salinity	Organic matter levels in water	Organic matter levels in water
41.	What is the relationship between dissolved oxygen level and BOD	High BOD reduces dissolved oxygen as organic matter decays	High BOD means High DO	BOD has no impact on DO	Inversly proportional to each other	High BOD reduces dissolved oxygen as organic matter decays
42.	_____ is the amount of oxygen required to oxidize only organic matter in sewage	Turbidity	BOD	COD	DO	BOD
43.	The full form of BOD is	Biodegradable oxygen demand	Biological oxygen	Biochemical oxygen demand	Bandwidth on demand	Biological oxygen demand

			demand			
44.	The biochemical oxygen demand is computed by	Dissolved oxygen / Dilution factor	Dissolved oxygen + Dilution factor	Dissolved oxygen – Dilution factor	Dissolved oxygen x Dilution factor	Dissolved oxygen x Dilution factor
45.	The maximum desirable limit Bureau of Indian Standards (BIS) of lead in the drinking water is	0.05 mg/l	0.09 mg/l	0.1 mg/l	1.0 mg/l	0.05 mg/l
46.	Zeolite softening process removes	only temporary hardness of water	only permanent hardness of water	both temporary and permanent hardness of water	the dissolved gases in permanent hard water	both temporary and permanent hardness of water
47.	Conventional tertiary treatment is	chemical coagulation and flocculation	filtration	sedimentation	synthesis	chemical coagulation and flocculation
48.	The maximum desirable limit (BIS) of total hardness (as $CaCo_3$) in drinking water is	600 ppm	300 ppm	500 ppm	1000 ppm	300 ppm
49.	The chemical oxygen demand (COD)measures the	amount of oxygen required for growth of microorganisms in water	amount of oxygen that would be removed from the water in order to oxidize pollution	amount of oxygen required to oxidize the calcium present in waste water	amount of nitrogen required to oxidize the calcium present in waste water	amount of oxygen that would be removed from the water in order to oxidize pollution
50.	Hardness of water does not	have any bad effect in boiler	make cooking of foods difficult	make it unfit for drinking	cause difficulty in the washing of clothes with soaps	make it unfit for drinking

51.	Permanent hard water may be softened by passing it through	sodium silicate	sodium bicarbonate	sodium hexametaphosphate	sodium phosphate	sodium hexametaphosphate
52.	Zeolite used in zeolite softening process for the treatment of hard water gets exhausted after certain time of usage but can be regenerated by flushing it with	10% calcium chloride solution	10% magnesium sulfate solution	10% magnesium chloride solution	10% sodium chloride solution	10% sodium chloride solution
53.	Temporary hardness of water is caused by the presence of	chlorides of calcium and magnesium	sulfates of calcium and magnesium	bicarbonates of calcium and magnesium	carbonates of sodium and potassium	bicarbonates of calcium and magnesium
54.	Secondary treatment uses _____ to consume wastes.	Microorganisms	chemicals	filteration	Peptisation	Microorganisms
55.	Sublimation, dissolving and filtration can only be carried out in	Soluble substances in solution	Insoluble substances in liquids	Liquid-liquid mixtures	Solid-solid mixtures	Solid-solid mixtures
56.	Process quicker than filtration but not so effective is	decanting	centrifuging	crystillation	Fractional distillation	decanting
57.	Anhydrous agent used to remove water from ethanol distillate may be	Iodine	Hydrocarbon	Calcium chloride	Napthalene	Calcium chloride
58.	Acid used mostly for removal of milk stone is	Phosphoric acid	Nitric acid	Gluconic acid	Tartaric acid	Nitric acid

59.	Which of the following chemical is sometime added in the process of coagulation and flocculation?	Aluminum sulphate	Aluminium oxide	Calcium chloride	Magnesium sulphate	Aluminum sulphate
60.	The common methods used for disinfection in waste water treatment plants are	Chlorination	UV-light	UV-light and chlorination	Phenolic solvent	UV-light and chlorination

S.No	Question	a	b	c	d	Answer
1.	Chromatography is based on the	Different rate of movement of the solute in a column	Separation of one solute from other constituents by being captured on the adsorbent	Different rate of movement of the solvent in the column	Movement of adsorbents in a solvent	Different rate of movement of the solute in a column
2.	Ion exchange chromatography is based on the	Electrical mobility of ionic species	Adsorption chromatography	Partition chromatography	Electrostatic attraction	Electrostatic attraction
3.	Which of the following statements about chromatography is correct?	Paper chromatography and gas chromatography are both routinely used for qualitative analysis only.	Paper chromatography is usually considered to be quantitative only, while gas chromatography can be qualitative or quantitative	Paper chromatography is usually considered to be qualitative only, while gas chromatography can be qualitative or quantitative.	Paper chromatography and gas chromatography are both routinely used for quantitative analysis only.	Paper chromatography is usually considered to be qualitative only, while gas chromatography can be qualitative or quantitative.

4.	Which of the following statements about paper Rf and gas chromatography Rt is correct?	The Rf and Rt values of a substance are determined solely by the interaction of the substance with the stationary phase.	A substance with a long retention time in gas chromatography is likely to have a high Rf value in paper chromatography.	A high Rf value is indicative of a substance that adsorbs strongly onto the stationary phase.	A long retention time in gas chromatography is indicative of a substance with a strong adsorption on to the stationary phase.	A long retention time in gas chromatography is indicative of a substance with a strong adsorption on to the stationary phase.
5.	Thin layer chromatography can be used to distinguish between different amino acids. If a particular amino acid has low solubility in the mobile phase used, then the other amino acid	will spend more time dissolved in the mobile phase than attached to the stationary phase.	will have a low Rf value.	will move at a speed close to that of the solvent.	must have a high molecular mass.	will have a low Rf value.
6.	Column chromatography separates molecules according to their	Molecular size	Solubility	Polarity	Matrix	polarity
7.	Chromatography is a physical method that is used to separate and analyse _____	Simple mixtures	Complex mixtures	Viscous mixtures	metals	Complex mixtures
8.	In which type of chromatography, the stationary phase held in a narrow tube and the mobile phase is forced	Column chromatography	Planar chromatography	Liquid chromatography	Gas chromatography	Column chromatography

	through it under pressure					
9.	In chromatography, the stationary phase can be _____ supported on a solid	Solid or liquid	Liquid or gas	Solid only	Liquid only	Solid or liquid
10.	In chromatography, which of the following can the mobile phase be made of	Solid or liquid	Liquid or gas	Gas only	Liquid only	Liquid or gas
11.	Which of the following cannot be used as adsorbent in Column adsorption chromatography	Magnesium oxide	Silica gel	Activated alumina	Potassium permanganate	Potassium permanganate
12.	Which of the following types of chromatography involves the separation of substances in a mixture over a 0.2mm thick layer of an adsorbent	Gas liquid	column	Thin layer	paper	Thin layer
13.	In Column chromatography, the stationary phase is made of _____ and the mobile phase is made of _____	Solid, liquid	Liquid, liquid	Liquid, gas	Solid, gas	Solid, liquid
14.	In Thin layer chromatography, the stationary phase is made of _____ and the mobile	Solid, liquid	Liquid, liquid	Liquid, gas	Solid, gas	Solid, liquid

	phase is made of _____					
15.	In which of the following type of paper, chromatography does the mobile phase move horizontally over a circular sheet of paper?	Ascending paper chromatography	Descending paper chromatography	Radial paper chromatography	Ascending – descending chromatography	Radial paper chromatography
16.	For a typical adsorbent such as silica gel, the most popular pore diameters are	10 and 50 A°	60 and 100 A°	100 and 150 A°	150 and 200 A°	60 and 100 A°
17.	Column efficiency is measured in terms of number of plates which is	inversely related to the square of the peak width	directly related to the square of the peak width	inversely related to the cube root of the peak width	directly related to the square of the peak width	inversely related to the square of the peak width
18.	The eluent strength is a measure of	solvent adsorption energy	solvent absorption energy	solvent diffusivity	solvent mixing index	solvent adsorption energy
19.	Which of the following is a factor that affects the storage stability of food	Type of raw material used	Quality of raw material used	Method of packing	Type of raw material, Quality of raw material used and method of packing	Type of raw material, Quality of raw material used and method of packing

20.	Which of the following sentence is true with respect to food storage/preservation	Each food type has a potential storage life	Each food type don't have a potential storage life	Not due to the mechanical abuse that food has received during storage/distribution affects its storage stability	Preservative is not added	Each food type has a potential storage life
21.	Which of the following sentence is true with respect to food storage/preservation	Each food type has a different and varied potential storage life	Each food type don't have a potential storage life	The mechanical abuse that food has received during storage/distribution affects its storage stability	Preservative is not added	The mechanical abuse that food has received during storage/distribution affects its storage stability
22.	The mechanical abuse that food has received during storage/distribution affects its storage stability	Food storage and preservation is observed to be better/easier in parts of the world that have civilizations prevalent there	Proteins are held in an emulsion state in a water system	Fats are in colloidal state	Fats are in solution state	Food storage and preservation is observed to be better/easier in parts of the world that have civilizations prevalent there
23.	Which of the following statement with respect to food preservation is true?	Leafy vegetables perish slow due to their high moisture content	Cereals have the lowest requirements of moisture and soil types	Cereals have the highest requirements of moisture and soil types	Leafy vegetables perish slow due to their low moisture content	Cereals have the highest requirements of moisture and soil types

24.	Which of the following is an advantage of food processing	Availability of seasonal food throughout the year	Availability of seasonal food throughout the year	Processed food adds empty calories to food constituting junk	Some chemicals make the human and animal cells grow rapidly which is unhealthy	Availability of seasonal food throughout the year
25.	Which of the following is an advantage of food processing	Removal of toxins and preserving food for longer	Availability of seasonal food throughout the year	Processed food adds empty calories to food constituting junk	Some chemicals make the human and animal cells grow rapidly which is unhealthy	Removal of toxins and preserving food for longer
26.	Which of the following is an advantage of food processing	Adds extra nutrients to some food items	Availability of seasonal food throughout the year	Processed food adds empty calories to food constituting junk	Some chemicals make the human and animal cells grow rapidly which is unhealthy	Adds extra nutrients to some food items
27.	Which of the following is a disadvantage of food processing	Adds extra nutrients to some food items	Removal of toxins and preserving food for longer	Availability of seasonal food throughout the year	Availability of seasonal food throughout the year	Availability of seasonal food throughout the year
28.	Which of the following is a disadvantage of food processing	Adds extra nutrients to some food items	Removal of toxins and preserving food for longer	Availability of seasonal food throughout the year	Processed food adds empty calories to food constituting junk	Processed food adds empty calories to food constituting junk
29.	Which of the following is a disadvantage of food processing	Adds extra nutrients to some food items	Removal of toxins and preserving food for longer	Availability of seasonal food throughout the year	Some chemicals make the human and animal cells grow rapidly which is unhealthy	Some chemicals make the human and animal cells grow rapidly which is unhealthy

30.	A substance needed by the body for growth, energy, repair and maintenance iscalled a _____.	Nutrient	carbohydrate	calorie	fattyacid	Nutrient
31.	All the following are nutrients except	Plasma	proteins	carbohydrates	vitamins	Plasma
32.	A diet high in saturated fat can be linked to which of the following	Kidney failure	bulimia	diabetes	Cardiovascular disease	Cardiovascular disease
33.	Amylases in saliva breakdown carbohydrates into	fattyacids	polypepides	Amino acids	Simple sugars	Simple sugars
34.	Body needs vitamins and minerals because	They need body energy	They help carryout metabolic reactions	They insulate body organs	They withdraw heat from the body	They help carryout metabolic reactions
35.	About half of the diet should be made up of	Grains and vegetables	Fruits and milk	Milk and cheese	Fats and sugars	Grains and vegetables
36.	Which is not considered as a nutrient	vitamins	minerals	fibers	proteins	fibers
37.	The adulterant present in chille powder is	Brick powder	Soap stone starch	Coloured saw dust	chicory	Brick powder
38.	The adulterant present in asafoetida is	Brick powder	Soap stone	Coloured saw dust	chicory	Soap stone
39.	The adulterant present in turmeric is	Brick powder	Soap stone	Coloured saw dust	chicory	Coloured saw dust
40.	The adulterant present in coffee is	Brick powder	Soap stone	Coloured saw dust	chicory	chicory

41.	Brick powder is added as an adultrant to	coffee	Turmeric	Chille powder	asafoetida	Chille powder
42.	Soapstone is added as an adultrant to	coffee	Turmeric	Chille powder	asafoetida	asafoetida
43.	Coloured saw dust is added as an adultrant to	coffee	Turmeric	Chille powder	asafoetida	Turmeric
44.	chicory is added as an adultrant to	coffee	Turmeric	Chille powder	asafoetida	coffee
45.	The adulterant present in chille powder is	Talc powder	Soap stone starch	Coloured saw dust	chicory	Talc powder
46.	The adulterant present in asafoetida is	Brick powder	Starch	Coloured saw dust	chicory	Starch
47.	To a little powder of chilli add small amount of concHCl and mix to the consistency of paste,dip the rear end of the match stick into the paste and hold over the flame,brick red flame colour due to the presence	Brick powder	Salt powder	Talc powder	chicory	Brick powder
48.	To asafoetidaAdd tincture of iodine, appearance of blue colour shows the presence of	Brick powder	Salt powder	Talc powder	starch	starch
49.	Ion-exchange chromatography is used for the separation of	Polar molecules	Non polar molecules	Neutral molecules	Assymetric molecules	Polar molecules

50.	Ion exchange chromatography is based on the	Electrostatic attraction	Electrical mobility of ionic species	Adsorption chromatography	Partition chromatography	Electrostatic attraction

UNIT-4

SYLLABUS

Analysis of food products:

Analysis of food products: Nutritional value of foods, idea about food processing and foodpreservations and adulteration.

a. Identification of adulterants in some common food items like coffee powder, asafoetida,chilli powder, turmeric powder, coriander powder and pulses, etc.
b. Analysis of preservatives and colouring matter.

Chromatography: Definition, general introduction on principles of chromatography, paperchromatography, TLC etc.

a. Paper chromatographic separation of mixture of metal ion (Fe3+ and Al3+).
b. To compare paint samples by TLC method.

Ion-exchange: Column, ion-exchange chromatography etc.Determination of ion exchange capacity of anion / cation exchange resin (using batchprocedure if use of column is not feasible).

Nutritional Value

Definition: An indication of the contribution of a **food** to the **nutrient** content of the diet. This **value** depends on the quantity of a **food** which is digested and absorbed and the amounts of the essential **nutrients** (protein, fat, carbohydrate, minerals, vitamins) which it contains.

Why nutrition is important.

Eating a balanced diet is vital for good health and wellbeing. Food provides our bodies with the energy, protein, essential **fats**, **vitamins** and minerals to live, grow and function properly. We need a wide variety of different foods to provide the right amounts of **nutrients** for good health.

What is the basic nutrition

Food is essential—it provides vital **nutrients** for survival, and helps the body function and stay healthy. ... Food also supplies micronutrients (vitamins and minerals) and phytochemicals that don't provide calories but serve a variety of critical functions to ensure the body operates optimally.

Food processing and preparation activities cover three main fields: (1) the preservation of foods by (a) modern methods such as refrigeration, canning and irradiation, and (b) traditional methods such as drying, salting, smoking and fermentation; (2) the development of protein - rich foods; (3) food additives.

The Nutrition Division's interest was mainly, in their nutritional implications, in particular in reducing wastage of food, in preventing losses in nutritive value and in conserving or enhancing palatability.

Food processing is a way or technique implemented to convert raw food stuff into well-cooked and well preserved eatables for both the humans and the animals. All these methods are used by food processing industry to give out processed or preserved foods for our daily consumption. Best quality harvested, slaughtered and butchered and clean constituents are used by food processing industry to manufacture very nutritious and easy to cook food products. Following are some techniques and methods used to convert food into processed or preserved food.

Preservation process: this includes heating or boiling to destroy micro-organisms, oxidation, toxic inhibition, dehydration or drying, osmotic inhibition, freezing, a sort of cold pasteurization which destroys pathogens and various combinations of all these methods.

Drying: this is probably the most ancient method used by humans to preserve or process their food. Drying reduces the water content in the product and lack of water delays the bacterial growth very much. Drying is the most common technique to preserve or process cereal grains like wheat, maize, oats, rice, barley, grams and rye etc.

Smoking: many foods such as meat, fish and others are processed, preserved and flavored by the use of smoke mostly in big smoke houses. This process is very simple as the combination of smoke to preserved food without actually cooking it and the aroma of hydro-carbons generated from the smoke processes the food and makes it even tastier to eat.

Freezing: probably, it is the most common technique used in modern world to preserve or process the food both on commercial and domestic basis. This freezing is conducted in big cold storages which can stockpile huge amount of food stuffs which can be further used in some natural emergencies. A very big range of products can be frozen to preserve and process which includes some which do not need freezing when are in their natural condition. For example potato chips and potato wafers requires freezing whereas a potato does not.

Vacuum packs: in this method, food is packed in airtight bags and bottles in a vacuum area. This method is used in processing the food as the air-tight environment doesnâ€™t provide oxygen needed by germs especially bacteria to survive. This then, prevents food from getting rotted. This method is very commonly used for preserving processed nuts.

Salting: the method of salting is used in food processing as it sucks out the moisture from the food. This is done through the process of osmosis. Meat is the best example of the food processed by salting as nitrates are used very frequently to treat meat.

Sugaring: the method of using sugar to preserve or process food is very frequent where it comes to preserve fruits. In this method fruits such as apples, peaches and plums are cooked with sugar until they are crystallized and then it is stored dry. Now days, sugar is also used in combination of alcohol to make some branded alcohol and spirits.

Pickling: in this method of preserving or processing food, food is cooked in chemicals and materials which destroy micro-organisms. This is very strictly kept in mind that these chemicals or materials are fit to eat for humans. Normally, these include brine, vinegar, ethanol, vegetable

oil and many other types of oils. Pickling is very commonly seen in vegetables such as cabbage and peppers. Corned beef and eggs are the non vegetarian eatables that are pickled.

Food processing mainly talks about how raw food could be changed into another form so that it would be edible at the same time it should be variative, mass-produceable, cost-efficient production, have long shel-life, and many other industrial- related things.

On the other hand, food preservation focuses on the things needed to prolong shelf-life of food but it should be edible and safe to consume.

So, the main point for **food processing** is **edible** and **food preservation** is prolonged **shelf-life**.

Food adulteration is the act of intentionally debasing the quality of **food** offered for sale either by the admixture or substitution of inferior substances or by the removal of some valuable ingredient.

Chillies powder

1. Brick powder, salt powder or talc,powder.

Take a teaspoon full of chillies powder in a glass of water. Coloured water extract will show the presence of artificial colour. Any grittiness that may be felt on rubbing the sediment at the bottom of glass confirms the presence of brick powder/sand, soapy and smooth touch of the white residue at the bottom indicates the presence of soap stone.
To a little powder of chilli add small amount of concHCl and mix to the consistency of paste,dip the rear end of the match stick into the paste and hold over the flame,brick red flame colour due to the presence of calcium slats in brick powder.

2. Artificial colours

Sprinkle the chilli powder on a glass of water. Artificial colorants descend as coloured streaks

3. Water soluble coal tar colour

Water soluble artificial color can be detected by sprinkling a small quantity of chillies or turmeric powder on the surface of water contained in a glass tumbler.

The water soluble colour will immediately start descending in colour streaks

Asafoetida

1. Soap stone or other earthy material

Shake little portion of the sample with water and allow to settle.Soap stone or other earthy mailer will settle down at the bottom.

In compounded asafoetida due to presence of starch, a slight turbid solution may be produced.However, this will settle down after keeping

2. Starch

Add tincture of iodine, appearance of blue colour shows the presence of starch.

Compound of asafoetida contains starch which is declared on the label. This test is not applicable for compound asafetida

3. Foreign resin

Burn on a spoon, if the sample burns like camphor, it indicates the sample is pure.

Pure hing burns like aromatic camphor

Turmeric powder

1. Coloured saw dust

Take a tea spoon full of turmeric powder in a test tube. Add a few drops of concentrated Hydrochloric acid. Instant appearance of pink colour which disappears on dilution with water shows the presence of turmeric If the colour persists, metanil yellow (an artificial colour) a not permitted coal tar colour is present.

This test is only for Metanil yellow

1. Turmeric whole
   Lead chromate Appears to be bright in colour which leaves colour immediately in water.

2. Chalk powder or yellow soap stone powder

   Take a small quantity of turmeric powder in a test tube containing small quantity of water. Add a few drops of concentrated Hydrochloric acid, effervescence (give off bubbles) will indicate the presence of chalk or yellow soap stone powder

Pulses

Lead Chromate

Shake 5 gm.Of pulse with 5 ml. Of water and add a few drops of HCl. Pink colour indicates Lead Chromate.

Coffee

Chicory

Gently sprinkle the coffee powder sample on the surface of water in a glass.The coffee floats over the water but chicory begins to sink down within a few seconds.The falling chicory powder particles leave behind them a trail of colour, due to large amount of carame.

Chicory. - One of the common adulterants of coffee is the prepared root of the chicory plant, Cychoriumintybus. There are several chemical methods for the detection of chicory, depending

upon positive and negative tests. Ground chicory when thrown on cold water sinks quickly, coloring the water, and is soon softened, whereas ground roasted coffeefloats, imparting no color. Chicory is easily bleached by chlorinated soda (labarraque solution); coffee is but slowly affected by this bleaching agent. The coloring 1 matter of chicory is not precipitated by iron salts, while that of coffee is colored green and is partially precipitated. G. C. Wittstein2 employs the following method:

Boil 30 drops of the coffee infusion in a test tube with 2 drops of concentrated hydrochloric acid; add 15 drops potassium ferrocyanide solution (1 part of the salt to 8 of water), and again boil until the liquid becomes a dark green; add 6 drops of potassium hydroxide solution and boil; if chicory is present the liquid will become brown and murky, otherwise a precipitate will separate and settle to the bottom of the tube, leaving the supernatant solution of a light-yellow color.

Preservatives:

Preservatives are the compounds used to prevent and retard the microbial spoilage of food. Preservative as "a substance which when added to food is capable of inhibiting, retarding or arresting the process of fermentation, acidification or other decomposition of food" They are classified into Class I and Class II preservatives.

Class I preservatives are

1. Common salt
2. Sugar
3. Dextrose
4. Glucose
5. Spices
6. Vinegar or acetic acid
7. Honey
8. Edible vegetable oils

Class II preservatives are

1. Benzoic acid including salts thereof

2. Sulphurous acid including salts thereof
3. Nitrates or Nitrites and/or Sodium and Potassium in respect of foods like ham, Pickled meat
4. Sorbic acid and its sodium,
5. Potassium and calcium salts
6. Propionates of Calcium or sodium,
7. Sodium, Potassium and Calcium salts of Lactic acid.
8. Nisin
9. Methyl or Propyl parahydroxy Benzoates
10. Sodium Diacetate.

Benzoic Acid:

Qualitative Methods

(A) Ferric Chloride Test:

Acidify the food product with hydrochloric acid (1+3) and extract with diethyl ether. Evaporate the solvent on a hot water bath removing last traces of solvent under a current of air. Dissolve the residue in few ml of hot water and add few drops of 0.5% ferric chloride solution. Salmon colour precipitate of ferric benzoate indicates the presence of benzoic acid.

(B) Modified Mohler's Test:

To the aqueous solution of the residue obtained as given under method 'A' add one or two drops of 10% sodium hydroxide solution and evaporate to dryness. To the residue add 5-10 drops of sulphuric acid and a small crystal of potassium nitrate. Heat for 10 min in a glycerol bath at 120 – 130 º C. Cool, add 1 ml of water and make distinctly ammonical. Boil the solution to decompose any ammonium nitrite (NH_4NO_2) formed. Cool and add a drop of fresh colourless ammonium sulphide [$(NH_4)_2S$] solution. The sulphide solution can be made by passing hydrogen sulphide in 0.88 ammonia. Do not let the layers mix. Red brown ring indicates benzoic acid. On mixing, colour diffuses throughout the liquid and on heating finally changes to greenish yellow.

This change differentiates benzoic acid from salicylic acid cinnamic acid. Salicylic acid and cinnamic acid form coloured compounds which are destroyed on heating.

Spectrophotometric method:

Principle:

Benzoic acid is extracted from prepared sample using diethyl ether and the absorbance of the ether layer is measured at 272 nm, 267.5 nm and 276.5 nm in the UV region. From the corrected absorbance and the calibration graph obtained using standard benzoic' acid solution, the amount of benzoic acid is determined.

Reagents:

1. Diethyl ether distilled
2. Hydrochloric acid (1+3)
3. Saturated sodium chloride solution
4. Ammonium hydroxide (0.1%)
5. Standard benzoic acid.

Procedure:

(a) Preparation of standard curve: Prepare solution of benzoic acid in ether containing 50 mgs/l. Determine absorbance of this solution in tightly stoppered cell in Beckman DU or recording spectrophotometer between 265 and 280 nm at 1 nm intervals. Plot absorbance against wavelength and record wavelength of minimum at approximately 267.5 nm as point B. Other minimum at approximately 276.5 nm as point D and highest maximum at approximately 272 nm as point C.

Prepare solution of benzoic acid in ether containing 20, 40, 60, 80,100 and 120 mg/l. Determine absorbance of these solutions in a spectrophotometer at points B, C and D. For each concentration average absorbance at Band D subtract from absorbance at C.

Plot difference against concentration to get the standard curve.

(b) Preparation of sample:

Mix sample thoroughly. Transfer 10 gm or 10 ml to separator and dilute to 200 ml with saturated sodium chloride solution. Make solution definitely acidic to litmus with hydrochloric acid and mix well.

(c) Determination: Extract prepared solutions with 70, 50, 40, and 30 ml portions of diethyl ether, shaking well to ensure complete extraction (break emulsions by standing, stirring or centrifuging). Drain and discard aqueous phase. Wash combined ether extracts with 40 and 30 ml portions hydrochloric acid (1+1000) and discard hydrochloric acid washings (if extraction requires no purification,. proceed to next para). Extract ether solution with 50, 40, 30, and 20 ml portions of 0.1% ammonium hydroxide and discard ether. Neutralize combined ammonium hydroxide extracts with hydrochloric acid and add 1 ml excess. Extract the acidified solution with 70, 50, 40 and 30 ml ether.

Dilute combined ether extracts to 200 ml with ether and determine absorbance in stoppered cell in spectrophotometer at wavelengths B, C and D, diluting with ether if necessary to obtain optimum concentration of 20-120 mg/l. Average the absorbance's at B and D, subtract this value from absorbance at C. Determine the concentration of benzoic acid from standard curve correcting for dilutions.

FOOD COLOURS

The colouring matter in food may be (a) natural and (b) Synthetic colours. They may also be classified as (a) water soluble and (b) oil soluble. They have to be separated from food before identification can be done. Natural colours consist of chlorophyll, carotenes, cantaxanthene, riboflavin, annatto, saffron, turmeric, curcumin, caramel etc. Synthetic colours are of importance as they are widely used in different foods. They are classified as acidic and basic dyes. Only 8 coal-tar food colours are permitted to be used in certain food products under the provisions of FSS (Food Product Standards & Food additives) Regulations, 2011. They include three red

shades namely Carmoisine, Ponceau 4 R, Erythrosine, two Yellow shades namely Sunset Yellow FCF and Tartrazine, two blue shades i.e. Brilliant Blue FCF and Indigo Carmine and one green shade i.e. Fast Green FCF. However certain unpermitted colours such Metanil yellow, Rhodamne B, Orange G, Blue VRS, Auramine and certain unidentified water and oil soluble colours (such as Sudan red colours) often appears as adulterants in foods.

Turmeric (curcumin):

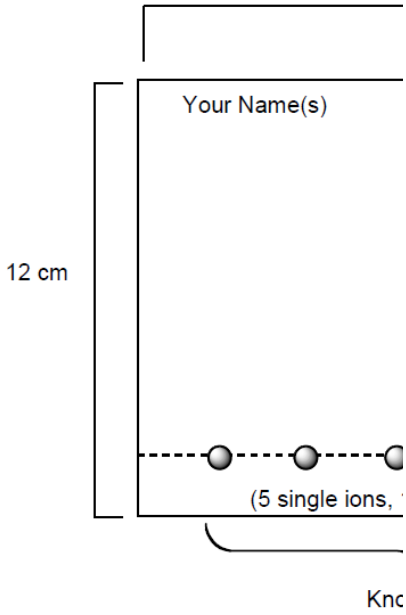
Evaporate an alcoholic extract of the material almost to dryness on the water bath with a piece of filter paper. Moisten the dried paper with a few drops of weak solution of boric acid to which some drops of hydrochloric acid have been added. Dry the paper again. If turmeric is present, the dry paper will be cherry red in colour which changes to bluish green by a drop of sodium hydroxide or ammonium hydroxide.

TABLE SHOWING ABSORPTION MAXIMA OF PERMITTED FOOR COLOURS

Sl No	Name of Colour	Absorption maxima (nm)
1	Carmosine	516
2	Ponceau 4 R	507
3	Erythrosine	527
4	Green FCF	624
5	Indigo Carmine	609
6	Brilliant Blue FCF	630
7	Tartrazine	427
8	Sunset yellow FCF	482

Paper chromatography:

A diagram showing how to prepare the paper is shown below. Standard solutions containing each of these ions will be spotted onto the paper using a capillary tube, along with a standard solution containing all five ions. An unknown will also be spotted onto the paper. Once the paper is prepared, it will be developed by placing the paper into the eluent. After 75-90 minutes, the paper is visualized by wetting it with an aqueous solution containing potassium iodide, KI, and potassium ferrocyanide, K4[Fe(CN)6]. The unique color observed for each ion is produced by a chemical reaction with the visualization solution. This is one useful way to identify which ions are present in an unknown mixture.



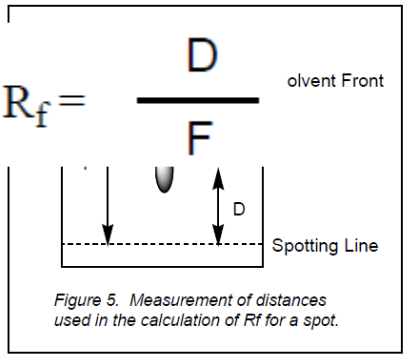


Figure 5. Measurement of distances used in the calculation of Rf for a spot.

The distance the ion moves up the paper can also be used to identify the ion. However, since students will develop their chromatography experiments for different amounts of time and under slightly different conditions, each student will have somewhat different measured distance for a given ion. The ratio of the

distance moved by an ion (D) to the distance moved by the solvent (F, *solvent front*) is characteristic and should be nearly the same for all students. This ratio is called $\mathbf{R}_f$, or "retention factor."

Thin-layer chromatography (TLC) has been used with some success on household paints Thin-layer chromatography was carried out on Merck DC aluminium sheets, precoated with silica gel 60F254 and previously activated for 1 h at 110°C. The Solvent systems used were:
For paint pigments
(1) Chlorobenzene/l,2dichloroethane/toluene (1 : 1: 1 v/v)
(2) 1,2-dichlorobenzene/l,2dichloroethane/toluene (2 : 1: 1 v/v)
(3) Acetone

Paint samples were extracted initially with dichloromethane. Insoluble pigments were spotted on to TLC plates as suspensions. Soluble pigments were run in suitable TLC systems.

Ion exchange is the reversible interchange of ions between a solid (ion exchange material) and a liquid in which there is no permanent change in the structure of the solid. Ion exchange is used in water treatment and also provides a method of separation in many non-water processes. It has special utility in chemical synthesis, medical research, food processing, mining, agriculture and a variety of other areas. The utility of ion exchange rests with the ability to use and reuse the ion exchange material. For example, in water softening:

$$2RNa^+ + Ca^{2+} \rightleftharpoons R_2Ca^{2+} + 2Na^+$$

The exchanger R in the sodium ion form is able to exchange for calcium and thus, to remove calcium from hard water and replace it with an equivalent quantity of sodium. Subsequently, the calcium loaded resin may be treated with a sodium chloride solution, regenerating it back to the sodium form, so that it is ready for another cycle of operation. The regeneration reaction is reversible; the ion exchanger is not permanently changed. Millions of liters of water may be softened per cubic meter of resin during an operating period of many years. Ion exchange occurs in a variety of substances and it has been used on an industrial basis since circa 1910 with the introduction of water softening using natural and later, synthetic zeolites. Sulfonated coal, developed for industrial water treatment, was the first ion exchange material that was stable at low pH. The introduction of synthetic organic ion exchange resins in 1935 resulted from the synthesis of phenolic condensation products containing either sulfonic or amine groups which could be used for the reversible exchange of cations or anions. A variety of functional groups have been added to the condensation or addition polymers used as the backbone structures. Porosity and particle size have been controlled by conditions of polymerization and uniform particle size manufacturing technology. Physical and chemical stability have been modified and improved. As a result of these advances, the inorganic exchangers (mineral, greensand and zeolites) have been almost completely displaced by the resinous types except for some analytical and specialized applications. Synthetic zeolites are still used as molecular sieves.

Capacity.

Ion exchange capacity may be expressed in a number of ways. Total capacity, i.e., the total number of sites available for exchange, is normally determined after converting the resin by chemical regeneration techniques to a given ionic form. The ion is then chemically removed from a measured quantity of the resin and quantitatively determined in solution by conventional analytical methods. Total capacity is expressed on a dry weight, wet weight or wet volume basis. The water uptake of a resin and therefore its wet weight and wet volume capacities are dependent an the nature of the polymer backbone as well as an the environment in which the sample is placed. Variations of dry weight and wet volume capacities with cross-linkage are shown in Figure 2 for a sulfonic resin. Operating capacity is a measure of the useful performance obtained with the ion exchange material when it is operating in a column under aprescribed set of

conditions. It is dependent on a number of factors including the inherent (total) capacity of the resin, the level of regeneration, the composition of solution treated, the flow rates through the column, temperature, particle size and distribution. An example is shown in Figure 3 for the case of water softening with a standard sulfonic resin at several regenerant levels.

It is defined as the process of separation of the individual components of a mixture based on their relative affinities towards stationary and mobile phases. Principle: The samples are subjected to **flow** by mobile liquid onto or through the stable stationary phase.

Chromatography is based on the principle where molecules in mixture applied onto the surface or into the solid, and fluid stationary phase (stable phase) is separating from each other while moving with the aid of a mobile phase. The factors effective on this separation process include molecular characteristics related to adsorption (liquid-solid), partition (liquid-solid), and affinity or differences among their molecular weights. Because of these differences, some components of the mixture stay longer in the stationary phase, and they move slowly in the chromatography system, while others pass rapidly into mobile phase, and leave the system faster.

Based on this approach three components form the basis of the chromatography technique.

- Stationary phase: This phase is always composed of a "solid" phase or "a layer of a liquid adsorbed on the surface a solid support".
- Mobile phase: This phase is always composed of "liquid" or a "gaseous component."
- Separated molecules

The type of interaction between stationary phase, mobile phase, and substances contained in the mixture is the basic component effective on separation of molecules from each other. Chromatography methods based on partition are very effective on separation, and identification of small molecules as amino acids, carbohydrates, and fatty acids. However, affinity chromatographies (ie. ion-exchange chromatography) are more effective in the separation of macromolecules as nucleic acids, and proteins. Paper chromatography is used in the separation of proteins, and in studies related to protein synthesis; gas-liquid chromatography is utilized in the separation of alcohol, esther, lipid, and amino groups, and observation of enzymatic

interactions, while molecular-sieve chromatography is employed especially for the determination of molecular weights of proteins. Agarose-gel chromatography is used for the purification of RNA, DNA particles, and viruses.

Thin-layer Chromatography uses an absorbent material on flat glass or plastic plates. This is a simple and rapid method to check the purity of an organic compound. It is used to detect pesticide or insecticide residuesin food. Thin-layer chromatography is also used in forensics to analyze the dye composition of fibers.

Paper Chromatography is one of the most common types of chromatography. It uses a strip of paper as the stationary phase. Capillary action is used to pull the solvents up through the paper and separate the solutes.

UNIT-5

SYLLABUS

Analysis of cosmetics:

Analysis of cosmetics: Major and minor constituents and their function

a. Analysis of deodorants and antiperspirants, Al, Zn, boric acid, chloride, sulphate.
b. Determination of constituents of talcum powder: Magnesium oxide, Calcium oxide, Zincoxide and Calcium carbonate by complexometric titration.

Antiperspirant

Antiperspirants work by blocking the sweat ducts whereas most deodorants inhibit or kill the bacteria responsible for producing body odour and/or cover the odour with a fragrance. Antiperspirants therefore affect a body function and because of this many countries classified them as over-the-counter (OTC) drugs and regulated them accordingly, legally distinguishing them from deodorants whose effect is merely cosmetic.

Deodorants intended for use on the body fall readily into two main types, i.e., those that are meant merely to deodorize the perspiration and those which contain a sufficiently appreciable content of astringents [so] as temporarily to check the flow of perspiration altogether.

Over the years many theories have been proposed to explain the action of antiperspirants with an early view being that they acted as astringents to close the pores. The simplest method of inhibiting perspiration is by the use of an astringent which acts partly by coagulating the proteins in the surface of the epidermis and partly on the nerve reflexes by closing the pores, thus

achieving the desired result. Astringent mixtures act by precipitating protein, reversibly or irreversibly, thus partially closing pores and stopping the flow of perspiration at its source.

Using astringents as antiperspirants was not a new idea. Alum – a naturally-occurring astringent containing aluminium and potassium sulphates – had been used in the West as an antiperspirant from Greek and Roman times. It was also employed to treat excessive sweating (hyperhidrosis). Although astringent aluminium salts proved to be the most effective antiperspirants and were widely used, cosmetic chemists examined a wide range of substances for similar capabilities.

Aluminium salts

In 1916, the ability of aluminium salts to control perspiration received scientific support and it was reported that aluminiumhexa chloride could be used to control localised hyperhidrosis. However, the first aluminium salt to be used in a commercial antiperspirant was aluminium chloride.

Using aluminium chloride is a mystery but it has been suggested that in the late nineteenth century actors and actresses experimented with aluminium chloride to reduce perspiration when working in heavy costumes under hot electric lights. However, I also note that aluminium chloride was a widely used nineteenth-century external disinfectant. Chloralum for example – made with aluminium chloride – was a well-known disinfectant and numerous formulas for it were published. So, it is possible to use aluminium chloride as a deodorant rather than an

Antiperspirant

Most of the early antiperspirants were made with aluminium salts with aluminium chloride, aluminiumsulphate and alum being most commonly employed. If scented, diethylene glycol ethyl ether could be added to help incorporate the perfume into the mixture, giving the final product some additional odour-covering capability.

Alum	20.0
Water	76.5
Ethyl ether of diethylene glycol	3.0
Perfume	0.5

Aluminium chloride was generally regarded as the most effective antiperspirant. However, personal-care products made with it were relatively acidic and were liable to irritate the skin and rot clothing fabric.

Two solutions to this problem were to use less acidic aluminium salts such as aluminiumsulphate and/or to buffer the antiperspirant with agents such as urea or borax. Both of these fixes made the antiperspirant less acidic but the trade-off was that they also made it less effective.

Aluminium chloride	90
Aluminiumsulphate	40
Borax	10
Water	860
	1000

In the long run there was widespread industry adoption of aluminiumchlorohydrate (ACH), it was less acidic that other aluminium salts so it was less likely to irritate the skin. It was also kinder to fabrics and a good antibacterial.

Clothing stains continued to be long-term issue. Alkaline soaps and detergents reacted with aluminium salts to generate yellow stains which could be set by ironing the fabric between washes. If the aluminium salt was contaminated with iron the problem could be even worse.

Zirconium salts

In the 1950s, zirconium salts were introduced. Early forms gave way to zirconium chlorohydrate with modern formulations commonly using aluminium zirconium chlorohydrate (AZCH). This has a higher antiperspirant efficacy than aluminiumchlorohydrate although the later is still used in some modern day antiperspirants.

Deodorants

Toilet waters had been used for centuries to mask body odour. Some of had some antiseptic value that helped to curb the activity of the Coryne bacteria that are the main cause underarm odour. Scented talcum powders were another popular deodoriser. The perfume helped cover odour, talc absorbed some of the underarm perspiration, zinc or magnesium stearate helped the powder adhere, and there was a selection of other ingredients – such as zinc peroxide, boric acid and sodium perborate – that acted as deodorisers. A simple recipe for a talcum powder is listed below:

	Per Cent
Purified zinc peroxide	40.0
Boric acid	20.0
Talc	39.5
Perfume	0.5

Composition of talcum powder depends on the company manufacturing the product. Different brands have different compositions and qualities. Here is the list of chemical compounds that constitute talcum powder.

- Silicon dioxide
- Magnesium oxide
- Calcium oxide
- Iron(III) oxide
- Aluminum oxide
- Zinc oxide
- Benzoin
- Calcium carbonate

The ingredients also include organic extracts and various essential oils. They are combined with chemicals having antibacterial and antifungal properties. It also contains vitamin E oil and almond oil. Talcum powder for sensitive skin contain arrowroot, cornstarch, oat starch, or

tapioca starch. Talcum powders with kaolin, rose extract, bentonite, myrrh, aloe vera, and slippery elm extracts give the skin a silky feel. Sandal wood extract and chamomile are soothing to skin, and prevent rashes and prickly heat.

Complexometric titrations

Many metal ions form slightly dissociated complex ions. The formation of these can serve as the basis of accurate and convenient titrations for such metal ions. Such determinations are referred to as complexometric titrations. The accuracy of these titrations is high and they offer the possibility of determinations of metal ions at concentrations at the millimole level. Many cations will form complexes in solution with a variety of substances that have a pair of unshared electrons (e.g. on N, O, S atoms in the molecule) capable of satisfying the coordination number of the metal. The metal ion acts as a Lewis acid (electron pair acceptor) and the complexing agent is a Lewis base (electron pair donor). The number of molecules of the complexing agent, called the ligand, will depend on the coordination number of the metal and on the number of complexing groups on the ligand molecule.

Simple complexing agents such as ammonia are rarely used as titrating agents because a sharp end point corresponding to a stoichiometric complex is generally difficult to achieve. Certain ligands that have two or more complexing groups on the molecule, however, do form well-defined complexes and can be used as titrating agents. One such reagent that is widely used is ethylenediaminetetraacetic acid (EDTA).

An organic agent which has two or more groups capable of complexing with a metal ion is called a chelating agent. The complex which is formed in this manner is called a chelate. Titration with such a chelating agent is called a chelometric titration which is a particular type of complexo metric titration. A pair of unshared electrons capable of complexing with a metal ion is located on each of the two nitrogen atoms and each of the four carboxyl groups. Thus there are six complexing groups in EDTA. We represent EDTA by the symbol H_4Y, which recognizes the fact that it is a tetraprotic acid. The four hydrogens in the formula refer to the four acidic hydrogens

on the four carboxyl groups. It is the unprotonated ligand Y^{4-} that is responsible for the formation of complexes with metal ions.

The present analysis is concerned with the determination of Ca by the use of a complexometric titration of the type that is described above. The titration is performed by adding a standard solution of EDTA to the sample containing the Ca. The reaction that takes place is the following:

$Ca^{2+} + Y^{4-} <===> CaY^{2-}$

Before the equivalence point, the Ca^{2+} concentration is nearly equal to the amount of unchelated (unreacted) calcium since the dissociation of the chelate is slight. At the equivalence point and beyond, pCa is determined from the dissociation of the chelate at the given pH. The equivalence point is detected through the use of an indicator which is itself a chelating agent. The specific indicator used is Eriochrome Black T. It contains three ionizable protons and we will represent it by the formula H_3In. In neutral or somewhat basic solutions, it is a doubly dissociated ion, HIn^{2-}, which is blue in color. Eriochrome Black T cannot be used as an indicator for the titration of calcium with EDTA, since it forms too weak a complex with calcium to give a sharp end point. Therefore, a solution containing the magnesium complex of EDTA, MgY^{2-}, is introduced into the titration mixture. Since Ca^{2+} forms a more stable complex with EDTA than magnesium, the following reaction occurs:

$MgY^{2-} + Ca^{2+} <===> CaY^{2-} + Mg^{2+}$

The magnesium that is released in this manner then reacts with the doubly ionized ion of the Eriochrome Black T. The complex that is formed between magnesium and that ion is red, hence at the start of the Ca titration the solution is red. This reaction can be written as follows:

$Mg^{2+} + HIn^{2-} <===> MgIn^{-} + H^{+}$
(blue) <===> (red)

The solution is then titrated with a standard solution of EDTA. At the beginning of the titration, the EDTA reacts with the remaining calcium ion that has not been complexed. After all the calcium has reacted the next portion of EDTA reacts with the magnesium complex which was formed earlier. The added EDTA competes favorably with the red magnesium-indicator complex ($MgIn^-$), to give MgY^{2-} and HIn^{2-} and thereby giving a blue color at the end point.

$MgIn^- + H^+ + Y^{4-}$ <===> $MgY^{2-} + HIn^{2-}$

(red) <===> (blue)

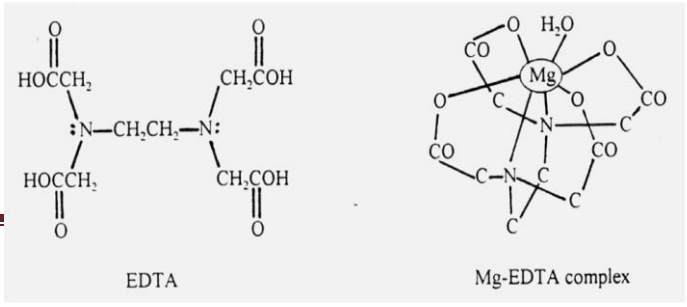
Determination of Mg by Titration with EDTA

Many metal ions react with electron pair donors to form coordination compounds or complex ions. The formation of a particular class of coordination compounds, called chelates, are especially well suited for quantitative methods. A chelate is formed when a metal ion coordinates with two (or more) donor groups of a single ligand. Tertiary amine compounds such as ethylenadiaminetetraacetic acid (EDTA) are widely used for the formation of chelates. Complexometric titrations with EDTA have been reported for the analysis of nearly all metal ions.

Because EDTA has four acidic protons, the formation of metal-ion/EDTA complexes is dependent upon the pH. For the titration of $Mg^{2+,}$ one must buffer the solution to a pH of 10 so that complex formation will be quantitative. The reaction of Mg2+ with EDTA may be expressed as:

$$Mg2+ + H2Y2- = MgY-2 + 2H+$$

The structure of EDTA and the magnesium-EDTA complex (without the hydrogen atoms) is



The endpoint of the titration is determined by the addition of Eriochrome Black T, which forms a colored chelate with Mg^{2+} and undergoes a color change when the Mg^{2+} is released to form a chelate with EDTA. While it is possible to achieve relatively good results by titration with EDTA prepared directly from the solid, better results should be obtained when the EDTA is standardized against a solution containing a known amount of metal ion. You will be provided with a standard solution of Zn^{2+} which you will use to standardize your EDTA solution.

1. Weighed m 50ml g of talc was placed in a nickel crucible, adding IO mg of solid NaOH, placed in a muffle furnace melting 30min at 700-750 ° C;

2. cooling water leaching, the leaching solution is poured in a previously added 20ml6mol / L hydrochloric acid 250ml volumetric flask, dilute to the mark; suction 2 parts, per ml of the test solution in k 250ml beaker for the determination of calcium and magnesium;

The determination of calcium

3. 250ml aliquot of test solution in the beaker, triethanolamine were added 3ml, 3ml mass concentration of 0.5% o-phenanthroline solution, 10ml200g / L KOH solution and a suitable amount of calcium carboxylate indicator, the triethanolamine is triethanolamine solution with water in a volume ratio of 1: 2 mixed, after shaking standard solution was titrated with ethylenediaminetetraacetic acid (EDTA), when the color of the solution turns from red to blue when is the titration end point, referred to as the consumption volume V1, in units of ml; k while withdrawing ml of distilled water in step (3) a blank test, titration of blank test calcium consumed volume of EDTA recorded as V. , In units of ml; EDTA is calcium consumption volume referred to as V2 = V1-Vtl;

4. the total amount of calcium and magnesium determination: taking another portion of the test solution in the 250ml beaker were added 3ml triethanolamine, 3ml mass concentration of 0.5% o-phenanthroline solution, IOmlpHIO ammonia - chloro 1_3 and ammonium buffer solution

dropwise Eriochrome black T, the triethanolamine is triethanolamine solution with water in a volume ratio of 1: 2 mixed, after shaking with EDTA standard solution titration, when the solution color changed from red is the end point of titration is blue, referred to as a volume V3 consumed, units of ml; the same time, the suction k milliliters of distilled water in step (4) blank test, titration test blank sum of calcium and magnesium is consumed volume of EDTA recorded as Vc / ml units; Han and magnesium is consumed by the total volume of EDTA recorded as V4 = V3-Vc /, talc, magnesium oxide content is calculated as:

5. where C is the concentration of EDTA standard solution, in units of mol / L, m is the mass of talc, in units of g, n = 250 / k.

6. The masking agent and while using triethanolaminePhenanthroline determined that the combination of two masking agents.

7. The method of the present invention has a more accurate measurement of talc, magnesium oxide, easy to operate, economical and time-saving.

S.No	Question	a	b	c	d	Answer
1.	Titanium dioxide is commonly present in	Vanishing cream	Sunscreen cream	Aqueous calamine cream	Opthalmic cream	Sunscreen cream
2.	The high concentration of _____ substances in lipstick formulation gives a dull appearance and develop bloom on storage.	Cetyl alcohol	Isopropyl alcohol	Wool alcohol	Isopropyl myristate	Cetyl alcohol
3.	_____ is used in hair tonic preparation	capsicum	Cantharides	resorcinol	Formaldehyde	capsicum
4.	_____ preparation produces both protective and cooling effect to relieve the sun burn.	Sunscreen	pallative	simulative	Vanishing cream	Sunscreen
5.	_____ is the evaluation of the relative screening activity of the sunscreen compounds.	Dihydroxyacetone	Juglone	Erythrulose	Benzoic acid	Dihydroxyacetone
6.	_____ wax is melts at 60-80 degree celcius temperature.	Carnuba wax	Ozokerite wax	Bees wax	Ceresin wax	Carnuba wax
7.	In compact rouges for colouring effect of powder, pigment content usually ____ %	one to five	Five to ten	Five to twenty	Two to forty	one to five

8.	_____ is the temperature at which the lipstick, lying flat, melts within the case and ooze out oil or flatten out.	Droop point	Flash point	Kraft point	Cloud point	Droop point
9.	_____ can readily remove the chemical substances of facial makeup.	Cold cream	Foundation cream	Cleansing cream	Night cream	Cold cream
10.	_____ rays stimulate blood circulation in the derma, cause the development of vitamin D from provitamins.	Infrared rays	UV rays	Gamma rays	Alpha rays	Infrared rays
11.	_____ % aqueous solution of sodium sulphide at pH 12 shows a strong depilatory action and can degrade hair in six to seven min.	One	Two	Three	Five	one
12.	In shaving creams total fatty substances should be minimum _____ %.	Ten	twenty	thirty	fifty	ten
13.	The after shave lotions are the clear solutions containing ____ % of alcohol.	Ten to twenty	Twenty five to fifty	Twenty to fourty	eighty	eighty

14.	The following primary film forming agent used in Nail lacquer folrmulation.	Acacia gum	Methyl cellulose	Nitro cellulose	Tragacanth	Acacia gum
15.	___ is caused by yeast like fungi in which nail folds becomes red and swollen.	Paronychia	Leukonychia	Brittleness	Koilonychia	Paronychia
16.	____ super fatting agents used in shaving preparations to make lather softer and give emollient effects on skin.	Mineral oil	PEG	Lanolin	parafin	Mineral oil
17.	The materials used to impart frosted look in face powder ____	Mica	Aluminium	Bronze	copper	Mica
18.	_____ cream is used in skiing and outdoor activities.	Sports	Vanishing	Cold	Body	sports
19.	_____ is hardest wax melting point is 85 0C is small percentage improve soft, strength of lipstick.	Beeswax	carnauba wax	Candella wax	Paraffin wax	Beeswax
20.	_____ plasticizer used in Nail lacquers.	Dibutyl phthalate	Camphor	Triethyl citrate	Trimethyl citrate	Dibutyl phthalate
21.	In moist environment covering power of titanium dioxide is ____ of dry powder.	25%	30%	37%	40%	25%

22.	Which is not an ingredient in talcum powder	Benzoic acid	Silicon dioxide	Magnesium oxide	Calcium oxide	Benzoic acid
23.	Scented talcum powders is a	deoderant	Antiperspirant	Skin preservative	Anti-histamine	deoderant
24.	Aluminium chloride is used as	deoderant	Antiperspirant	Skin preservative	Anti-histamine	Antiperspirant
25.	zirconium chlorohydrate is used as a	deoderant	Antiperspirant	Skin preservative	Anti-histamine	Antiperspirant
26.	Chloralum is a well known	deoderant	Antiperspirant	Skin preservative	disinfectant	disinfectant
27.	diethylene glycol ethyl ether is used to scent	deoderant	Antiperspirant	Skin preservative	Anti-histamine	Antiperspirant
28.	Example for a deoderant	Aluminium chloride	zirconium chlorohydrate	Scented talcum powders	diethylene glycol ethyl ether	Scented talcum powders
29.	Example for a Antiperspirant	Aluminium chloride	dibutylpthalate	Scented talcum powders	diethylene glycol ethyl ether	Aluminium chloride
30.	Example for a Antiperspirant	dibutylpthalate	zirconium chlorohydrate	Scented talcum powders	diethylene glycol ethyl ether	zirconium chlorohydrate
31.	Antiperspirants work by	blocking the sweat ducts	kill the bacteria responsible for producing body odour	kill the virus responsible for producing body odour	blocking the nose ducts	blocking the sweat ducts
32.	Deodrants work by	blocking the sweat ducts	kill the bacteria responsible for producing	kill the virus responsible for producing body odour	blocking the nose ducts	kill the bacteria responsible for producing body odour

			body odour			
33.	Talcum powder for sensitive skin contain	Corn starch	kaolin	Sandal wood extract	aloe vera	Corn starch
34.	Talcum powder for sensitive skin contain	tapioca starch	kaolin	Sandal wood extract	aloe vera	tapioca starch
35.	Ingredient in Talcum powder which gives skin a silky feel	tapioca starch	kaolin	Sandal wood extract	chamomile	kaolin
36.	Ingredient in Talcum powder which gives skin a silky feel	tapioca starch	Aloe vera	Sandal wood extract	chamomile	Aloe vera
37.	Ingredient in Talcum powder which gives soothing to skin, and prevent rashes and prickly heat.	tapioca starch	Aloe vera	Sandal wood extract	rose extract	Sandal wood extract
38.	Ingredient in Talcum powder which gives soothing to skin, and prevent rashes and prickly heat.	tapioca starch	Aloe vera	rose extract	chamomile	chamomile
39.	Chamomile in a talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	soothing to skin, and prevent rashes and prickly heat.

40.	Sandal wood extract in a talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	soothing to skin, and prevent rashes and prickly heat.
41.	Cornstarch in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	sensitive skin
42.	Tapioca starch in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	sensitive skin
43.	Kaolin in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	skin a silky feel
44.	Rose water in talcum powder gives	soothing to skin, and prevent rashes and prickly heat.	skin a silky feel	sensitive skin	Roughness to skin	skin a silky feel
45.	Tocopherol is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	fat-soluble compound
46.	Tocopherol	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	Acts as an antioxidant, protecting the skin from free-radicals
47.	Dimethicone is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	A group of silicone molecules

48.	Dimethicone	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	lubricates the skin without feeling heavy.
49.	Parabens is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	man-made chemical used for preserving cosmetic ingredients
50.	Parabens	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	Prevents that expensive cosmetic from forming bacteria and fungus
51.	Titanium dioxide is a	fat-soluble compound	A group of silicone molecules	man-made chemical used for preserving cosmetic ingredients	natural mineral taken from oxide of titanium	A group of silicone molecules
52.	Titanium dioxide	Acts as an antioxidant, protecting the skin from free-radicals	lubricates the skin without feeling heavy.	Prevents that expensive cosmetic from forming bacteria and fungus	It acts as a filler, making the cosmetic product more opaque	lubricates the skin without feeling heavy.
53.	Acts as an antioxidant, protecting the skin from free-radicals	tocopherol	Dimethicone	Paraben	Titanium di oxide	tocopherol
54.	fat-soluble vitamin	tocopherol	Dimethicone	Paraben	Titanium di oxide	tocopherol
55.	A group of silicone molecules	tocopherol	Dimethicone	Paraben	Titanium di oxide	Dimethicone
56.	The compound which lubricates the skin without	tocopherol	Dimethicone	Paraben	Titanium di oxide	Dimethicone

	feeling heavy.					
57.	man-made chemical used for preserving cosmetic ingredients	tocopherol	Dimethicone	Paraben	Titanium di oxide	Paraben
58.	Prevents that expensive cosmetic from forming bacteria and fungus	tocopherol	Dimethicone	Paraben	Titanium di oxide	Paraben
59.	natural mineral taken from oxide of titanium	tocopherol	Dimethicone	Paraben	Titanium di oxide	Titanium di oxide
60.	It acts as a filler, making the cosmetic product more opaque	tocopherol	Dimethicone	Paraben	Titanium di oxide	Titanium di oxide