

BT514 – ANALYTICAL CHEMISTRY

MID TERM IMPORTANT QUESTIONS

Prepared by: *Sir Mughal*

Admin: **BS Biological Sciences** (One place to all your solutions)

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1. Write a short note on sublimation.

Sublimation

Sublimation is a physical process in which a solid directly changes into vapor without passing through the liquid state. This process occurs when the vapor pressure of a solid becomes sufficiently high at a temperature below its melting point. Only substances with appreciable vapor pressure in the solid state undergo sublimation. Common examples include iodine, naphthalene, camphor, and ammonium chloride. In laboratory practice, sublimation is used as a purification method for volatile solids. The impure solid is heated, the pure substance vaporizes, and then it condenses on a cool surface, leaving non-volatile impurities behind.

2. What is Quality Assurance?

Quality Assurance (QA) is a systematic program of planned and documented activities implemented to ensure that analytical results are accurate, precise, reliable, and suitable for their intended purpose. It covers the entire analytical process, including proper sampling, use of standard operating procedures (SOPs), instrument calibration, method validation, use of reference materials, and proper documentation. The main objective of quality assurance is to prevent errors and maintain the credibility and reproducibility of laboratory results.

Key Components:

- Proper sampling procedures
- Use of standard operating procedures (SOPs)
- Calibration of instruments
- Use of certified reference materials
- Method validation
- Documentation and record keeping
- Internal and external audits

Sir Mughal: <https://wa.me/923132310940>

Sir Jawad: <https://wa.me/923036100859>

3. What is meant by replicates in chemistry?

Replicates in chemistry refer to repeated measurements or analyses of the same sample under identical experimental conditions. They are performed to assess the precision and reliability of analytical results. By comparing replicate results, random errors can be detected and minimized, and the reproducibility of the method can be evaluated. Replicates help in calculating statistical parameters such as mean and standard deviation, which indicate the consistency of the data.

4. Explain Validation Methods.

Validation Methods

Method validation is the process of proving that an analytical method is suitable for its intended purpose. It ensures that the method produces reliable, accurate, and consistent results under specified conditions. Validation involves evaluating several performance characteristics of the method. The main validation parameters include accuracy (closeness of measured value to true value), precision (repeatability of results), specificity (ability to measure analyte without interference), linearity (ability to obtain results proportional to concentration), range, limit of detection (LOD), limit of quantification (LOQ), and robustness (ability to remain unaffected by small variations in conditions). Through these parameters, method validation confirms the credibility and reliability of analytical data.

5. What is an analyte in a substance?

An analyte is the specific component or chemical species in a sample that is being measured, detected, or analyzed. It is the substance of interest in an analytical procedure, while other components present are considered matrix or interfering substances. For example, in a water sample tested for lead, lead (Pb^{2+}) is the analyte.

6. What are means of sampling?

Means of Sampling refers to the methods or procedures used to collect a representative portion of a material or substance for analysis. Proper sampling ensures that the sample accurately reflects the composition of the whole batch or population. Common means include:

1. **Random Sampling:** Selecting samples randomly to avoid bias.
2. **Systematic Sampling:** Collecting samples at regular intervals or positions.
3. **Stratified Sampling:** Dividing the material into layers or strata and sampling from each.
4. **Grab Sampling:** Taking a single sample at a specific time or location.
5. **Composite Sampling:** Combining multiple individual samples to form a single representative sample.

7. What is systematic error?

Systematic Error is a consistent, repeatable error that occurs in the same direction every time a measurement is made. It affects the accuracy of results, causing measurements to be consistently higher or lower than the true value.

Causes:

- Instrumental defects (e.g., miscalibrated balance)
- Experimental technique errors (e.g., reading a scale incorrectly)
- Environmental factors (e.g., temperature fluctuations affecting measurements)

8. Write full form of DOT.

The full form of DOT is Department of Transportation.

Sir Mughal: <https://wa.me/923132310940>

Sir Jawad: <https://wa.me/923036100859>

9. What are uncertainty measurements?

Uncertainty Measurements refer to the estimation of the range within which the true value of a measured quantity is expected to lie. It quantifies the doubt or variability in a measurement and indicates the confidence in the reported result.

Key Points:

- Expressed as $\pm$ value (e.g., 12.5 ± 0.2 g)
- Accounts for random errors and systematic errors
- Helps in comparing results and assessing reliability
- Calculated using standard deviation, confidence intervals, or propagation of errors in complex measurements

10. If product is too accurate, what do we do?

If a product or measurement is too accurate (i.e., reported with more precision than is justified by the method or instrument), it is usually rounded to an appropriate number of significant figures that reflect the true reliability of the data.

Steps to follow:

1. **Check the method's precision** – ensure the number of decimal places matches the method's capability.
2. **Round off the value** – do not report unnecessary digits beyond the method's uncertainty.
3. **Report with uncertainty** – include $\pm$ value to indicate the confidence in the measurement.

11. Explain Chromatography.

Chromatography is a separation technique used to separate, identify, and quantify components of a mixture based on their differential distribution between two phases: a stationary phase and a mobile phase.

Principle:

Different components move at different rates through the stationary phase due to differences in adsorption, partitioning, or affinity, leading to separation.

Types of Chromatography:

1. **Paper Chromatography** – stationary phase: paper; mobile phase: solvent.
2. **Thin Layer Chromatography (TLC)** – stationary phase: silica or alumina on a plate; mobile phase: solvent.
3. **Column Chromatography** – stationary phase: packed column; mobile phase: liquid or gas.
4. **Gas Chromatography (GC)** – stationary phase: coated column; mobile phase: inert gas.
5. **High-Performance Liquid Chromatography (HPLC)** – high-pressure liquid mobile phase through packed column.

Applications:

- Purification of compounds
- Identification of mixture components
- Quantitative analysis of drugs, food additives, and environmental samples

12. How do we resolve random error?

Random Errors are unpredictable variations in measurements that cause results to scatter around the true value. They can never be completely eliminated, but their effect can be minimized or resolved using statistical and procedural methods:

1. **Repeat Measurements:** Take multiple readings of the same sample to reduce the effect of random fluctuations.
2. **Average Values:** Calculate the mean of replicate measurements to get a more reliable estimate of the true value.
3. **Use Precise Instruments:** Instruments with higher sensitivity and resolution reduce random variations.
4. **Control Experimental Conditions:** Maintain stable temperature, pressure, and other environmental factors.
5. **Proper Technique:** Consistent handling and measurement procedures reduce human-induced variability.
6. **Statistical Analysis:** Use standard deviation, variance, or confidence intervals to quantify and account for random error.

13. Differentiate between Qualitative and Quantitative analysis.

Qualitative analysis focuses on identifying the components or substances present in a sample, providing descriptive information such as presence or absence of specific analytes. It answers the question “what is in the sample?” and uses techniques like chromatography, spectroscopy, and color tests. In contrast, quantitative analysis determines the exact amount or concentration of each component, answering “how much is present?” and provides numerical results. Techniques for quantitative analysis include titration, gravimetry, spectrophotometry, and HPLC. While qualitative analysis emphasizes composition, quantitative analysis emphasizes measurement and concentration.

14. What are stages of precipitation?

Stages of Precipitation refer to the steps involved when a solute forms an insoluble solid (precipitate) from a solution. The main stages are:

1. **Supersaturation:** The solution becomes saturated with the solute beyond its solubility limit, creating conditions favorable for precipitation.
2. **Nucleation:** Small clusters of solute molecules (nuclei) form, which act as seeds for crystal growth.
3. **Crystal Growth:** The nuclei grow by addition of more solute molecules from the solution.
4. **Agglomeration/Settling:** Crystals aggregate and settle out of the solution as a visible precipitate.
5. **Filtration and Washing:** The precipitate is separated from the solution and washed to remove impurities.

15. What are rules of significant figures?

Rules of Significant Figures determine which digits in a number are meaningful and contribute to its precision. The main rules are:

- **Non-zero digits** are always significant. Example: 245 → 3 significant figures
- **Zeros between non-zero digits** are significant. Example: 405 → 3 significant figures
- **Leading zeros** (zeros before the first non-zero digit) are not significant. Example: 0.0025 → 2 significant figures
- **Trailing zeros in a decimal number** are significant. Example: 2.500 → 4 significant figures
- **Trailing zeros in a whole number without a decimal** may or may not be significant; use scientific notation to clarify. Example: 2500 → 2, 3, or 4 significant figures ($2500 = 2.5 \times 10^3 \rightarrow 2 \text{ sig. figs}$)

Sir Mughal: <https://wa.me/923132310940>

Sir Jawad: <https://wa.me/923036100859>

16. Write importance of oxidation reactions.

Importance of Oxidation Reactions

Oxidation reactions are vital in chemistry and everyday life because they involve the transfer of electrons, leading to changes in the chemical composition of substances. They are important for:

1. **Energy Production:** Oxidation of fuels (like glucose in cells or hydrocarbons in engines) releases energy.
2. **Industrial Processes:** Used in manufacturing chemicals, bleaching, corrosion prevention, and refining metals.
3. **Analytical Chemistry:** Oxidation reactions help in titrations (redox titrations) and in detecting or quantifying analytes.
4. **Environmental Chemistry:** Oxidation reactions are essential for waste treatment, breaking down pollutants.
5. **Biological Systems:** Oxidation reactions drive metabolic pathways and cellular respiration.

17. Write basic rules of Gravimetric analysis.

Basic Rules of Gravimetric Analysis

Gravimetric analysis is a method of quantitative analysis where the amount of an analyte is determined by converting it into a pure, stable, and weighable solid. The basic rules include:

1. **Complete Precipitation:** Ensure the analyte forms a precipitate completely without losses.
2. **Purity of Precipitate:** The precipitate must be pure and free from impurities or co-precipitation.
3. **Proper Filtration and Washing:** Separate the precipitate from the solution and wash to remove adhering ions or impurities.
4. **Stable and Dry Precipitate:** The precipitate should be chemically stable and dried or ignited to constant weight.
5. **Accurate Weighing:** Use precise and calibrated balances to weigh the precipitate.
6. **Avoid Contamination:** Handle carefully to prevent loss or contamination during the procedure.

18. Write three examples of Quality records.

Examples of Quality Records:

1. **Calibration Records** – Documentation of instrument calibration details, dates, and results.
2. **Standard Operating Procedures (SOPs)** – Written procedures showing how analyses are performed consistently.
3. **Analytical Test Reports** – Records of raw data, calculations, and final results from laboratory tests.

19. What is difference between Quality Assurance and Quality Record?

Quality Assurance (QA) is a systematic process of planned activities designed to ensure that analytical results are accurate, precise, and reliable, focusing on preventing errors and maintaining credibility. In contrast, a Quality Record (QR) is a documented piece of evidence that shows QA procedures were actually followed, providing proof of compliance with standards. While QA is process-oriented, emphasizing how work should be done, quality records are document-oriented, capturing data such as calibration logs, test reports, or audit reports to demonstrate that the QA system is effective.

20. How to calibrate and handle the test item?

Calibration:

1. **Select Standard:** Use a certified reference material or standard solution appropriate for the test.
2. **Prepare Instrument:** Ensure the instrument is clean, properly set up, and at operating conditions.
3. **Perform Calibration:** Measure the standard at known values and adjust the instrument readings to match the true values.
4. **Verify Accuracy:** Check with a second standard or replicate measurements to confirm proper calibration.
5. **Document:** Record calibration data, date, operator, and any adjustments made.

Handling the Test Item:

1. **Proper Sampling:** Collect a representative sample without contamination.
2. **Labeling:** Clearly label the sample with identification, date, and storage instructions.
3. **Storage:** Store under recommended conditions (temperature, light, humidity) to maintain stability.
4. **Minimize Loss or Contamination:** Use clean containers and avoid unnecessary exposure.
5. **Documentation:** Record sample handling steps to ensure traceability and reproducibility.

21. Describe the deviation.

Deviation refers to the difference between a measured value and a reference or mean value. It indicates how much an individual measurement varies from the expected or average result.

Types of Deviation:

1. **Positive Deviation:** Measured value is greater than the reference value.
2. **Negative Deviation:** Measured value is less than the reference value.

Calculation:

$$\text{Deviation} = \text{Measured Value} - \text{Mean or True Value}$$

22. Describe why the QC of product is important for clients?

Quality Control (QC) of a product is important for clients because it ensures that the product meets specified standards and is safe, effective, and reliable. QC verifies that the product performs as intended, free from defects or contamination, which builds client trust and satisfaction. It also ensures consistency between batches, reduces the risk of failures or recalls, and provides confidence that the client receives a product that complies with regulatory and contractual requirements.

23. How to remove the chemometric errors in analyzers?

Removing Chemometric Errors in Analyzers

Chemometric errors arise from instrumental, environmental, or mathematical processing issues in analytical measurements. They can be minimized or removed by:

1. **Instrument Calibration:** Regularly calibrate instruments using certified standards to correct systematic errors.
2. **Use of Blanks and Controls:** Run blanks and control samples to detect and correct baseline shifts or interference.
3. **Proper Sample Preparation:** Ensure samples are homogeneous and free from interfering substances.
4. **Signal Processing and Data Correction:** Apply mathematical corrections, smoothing, or baseline adjustments to remove noise or drift.
5. **Maintenance and Environmental Control:** Keep instruments clean, stable, and under controlled temperature and humidity to reduce variability.

Sir Mughal: <https://wa.me/923132310940>

Sir Jawad: <https://wa.me/923036100859>

24. Write significance of demasking in chemical analysis.

Significance of Demasking in Chemical Analysis

Demasking is important because certain ions or substances in a sample can interfere with the detection or precipitation of the analyte. By using demasking agents, these interfering species are neutralized or removed, allowing the analyte to react properly and form a clear, accurate result. This improves accuracy, selectivity, and reliability of the analysis, especially in gravimetric, titrimetric, or complexometric methods.

25. What is scatter diagram? What does it explains?

Scatter Diagram

A scatter diagram is a graphical representation of data points plotted on a coordinate system to show the relationship between two variables. Each point represents a paired observation of the variables.

Explanation:

- It helps to visualize trends, correlations, or patterns between the variables.
- A positive correlation shows that as one variable increases, the other also increases.
- A negative correlation shows that as one variable increases, the other decreases.
- No clear pattern indicates no correlation.

26. How stationary phase impacts on the movement of molecules?

Impact of Stationary Phase on the Movement of Molecules

In chromatography, the stationary phase is the phase that does not move and interacts with molecules in the mixture. Its properties directly affect the rate and extent of movement of molecules:

1. **Affinity/Adsorption:** Molecules with higher affinity for the stationary phase move more slowly, while those with lower affinity move faster.
2. **Polarity:** Polar stationary phases retain polar molecules longer; non-polar phases retain non-polar molecules longer.
3. **Surface Area:** A larger surface area increases interaction, slowing down the movement of analytes.
4. **Phase Type:** In liquid chromatography, partitioning between stationary liquid and mobile phase controls migration; in gas chromatography, adsorption on solid stationary phase dominates.

27. What is the titrant in acid base titration? Explain its process.

Titrant in Acid-Base Titration

The titrant is the solution of known concentration that is added from a burette to react with the analyte (solution of unknown concentration) in a controlled manner until the reaction is complete.

Process of Acid-Base Titration:

1. **Preparation:** Fill the burette with the titrant and place the analyte in a conical flask, adding a suitable indicator.
2. **Addition of Titrant:** Slowly add the titrant to the analyte while continuously swirling the flask.
3. **End Point Detection:** Observe a color change of the indicator (or use a pH meter) to determine when the reaction is complete.
4. **Volume Measurement:** Record the exact volume of titrant used to reach the end point.
5. **Calculation:** Use the titrant's known concentration and the measured volume to calculate the concentration of the analyte.

28. What are ligands? Also explain their role.

Ligands

Ligands are ions or molecules that can donate a pair of electrons to a central metal atom or ion to form a coordination complex.

Role of Ligands:

1. **Complex Formation:** Ligands bind to metal ions, stabilizing them in solution and forming coordination compounds.
2. **Selectivity in Analysis:** Certain ligands preferentially bind specific metal ions, aiding in qualitative and quantitative determinations.
3. **Solubility Control:** Ligands can increase solubility of metal ions in solvents.
4. **Stabilization:** They prevent precipitation or hydrolysis of metal ions by forming stable complexes.
5. **Enhance Detection:** Ligands can produce colored or fluorescent complexes for spectrophotometric measurements.

29. How degree of variability in a dataset can be estimated?

The degree of variability in a dataset can be estimated using statistical measures that describe how spread out the data points are from the mean or expected value. Common measures include:

1. **Range:** Difference between the maximum and minimum values.
2. **Mean Deviation:** Average of the absolute differences between each data point and the mean.
3. **Variance (σ^2):** Average of the squared differences between each data point and the mean.
4. **Standard Deviation (σ):** Square root of the variance; indicates how much data typically deviates from the mean.

$$\text{Standard Deviation, } \sigma = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n}}$$

30. Write the significance of preventing mistake in a quality management system.

Significance of Preventing Mistakes in a Quality Management System

Preventing mistakes ensures that all processes are **consistent, accurate, and reliable**, reducing the risk of defective products or erroneous analytical results. It helps maintain **client trust**, avoids financial losses due to rework or recalls, ensures **regulatory compliance**, and promotes **efficiency** in operations by minimizing errors before they occur.

31. Define histogram.

Histogram

A histogram is a graphical representation of the frequency distribution of a dataset. The data is divided into intervals (bins) along the x-axis, and the frequency of data points in each interval is represented by the height of vertical bars. Histograms are used to visualize the distribution, spread, and patterns of numerical data.

32. What is Analytical Chemistry? Write its uses in biological studies and laboratory.

Analytical Chemistry

Analytical chemistry is the branch of chemistry that deals with the identification and quantification of chemical substances in a sample. It focuses on understanding what a substance is (qualitative analysis) and how much is present (quantitative analysis).

Uses in Biological Studies:

1. Determining concentrations of biomolecules like proteins, sugars, and nucleic acids.
2. Detecting and monitoring metabolites and hormones in biological fluids.
3. Studying enzymatic reactions and metabolic pathways.
4. Analyzing trace metals and electrolytes in tissues and cells.

Uses in Laboratory:

1. Quality control of reagents, pharmaceuticals, and chemicals.
2. Performing titrations, gravimetric, and spectroscopic analyses.
3. Monitoring purity of compounds in synthesis and extraction processes.
4. Supporting research and development by providing accurate and reliable data.

33. What is importance of precipitation?

Importance of Precipitation

Precipitation is important because it allows the separation and isolation of specific components from a solution. It is widely used in gravimetric analysis to determine the amount of an analyte by converting it into a pure, weighable solid. Precipitation also helps in removing impurities, purifying substances, and preparing compounds for further chemical reactions or analysis.

