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TITRIMETRIC ANALYSIS AND ITS APPLICATION IN QUANTITATIVE DETERMINATIONS

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Abstract: This article discusses titrimetric (volume) analysis, one of the main methods of quantitative chemical analysis. It presents the essence of the method, the classification of titration types, the basic requirements for reactions used in titrimetry, and a description of working solutions and standard substances.

Keywords: titrimetric analysis, titration, equivalent point, indicator, standardization, titrant, fixanal, quantitative analysis

Introduction: Titrimetric analysis is widely used in laboratory and industrial practice to determine the concentration of substances and control the quality of chemical compounds.

The method is based on the precise measurement of the volume of a reagent solution of known concentration required for complete interaction with the substance being determined. Due to its high accuracy, speed of execution, and simplicity of equipment, titrimetric analysis occupies an important place among quantitative methods of analytical chemistry.[1,2]

The essence of the method and its advantages: The titrimetric (volume) method of analysis involves determining the quantity of a substance by measuring the volume of a solution of known concentration required for a reaction with the component being analyzed.[2]

Calculations in titrimetric analysis are based on the law of equivalents.

The method is characterized by:
• speed of analysis;
• simplicity of equipment;
• automation capability;
• high accuracy (relative error of 0.1–0.01%).
Classification of titrimetric analysis methods
Depending on the type of chemical reaction, the following types of titration are distinguished:
• acid-base titration (neutralization);
• oxidation-reduction titration (redoximetry);
• precipitation titration;
• complexometric titration (complexometry).
Based on the reagents used, methods are divided into:
• acidometric (titrant is an acid, such as HCl);
• alkalimetric (titrant is an alkali, such as NaOH);
• permanganometric (titrant is $KMnO_4$);
• chromatometric (titrant is $K_2Cr_2O_7$);
• iodometric (titrant — I_2 or KI), etc.

Theoretical Foundations of Titration

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Titration is the gradual addition of a titrated reagent solution (titrant) to the analyzed solution until the equivalence point is reached—the point at which the reactants are in an equivalent ratio. The equivalence point is determined using indicators—substances that change the color, structure, or physical properties of the medium near the equivalence point.

To minimize errors, it is important that the equivalence point coincides with the titration endpoint. The following requirements apply to reactions used in titrimetric analysis:

• strict stoichiometric ratio of reagents;
• high equilibrium constant ($K > 10^8$);
• absence of side reactions;
• high reaction rate (1–3 s);
• the ability to accurately determine the equivalence point.[2,3]

The titer or normality of the prepared solution is determined by titrating solutions of so-called adjusting substances with it. An adjusting substance is a chemically pure compound of precisely known composition used to establish the titer of a solution of another substance.

Based on the titration data of the adjusting substance, the exact titer or normality of the prepared solution is calculated.[3]

A solution of a chemically pure adjusting substance is prepared by dissolving a calculated amount of it (weighed on an analytical balance) in water and then bringing the solution volume to a specified value in a volumetric flask. Individual (aliquot) portions of the solution prepared in this way are pipetted from the volumetric flask into conical flasks and titrated with the solution whose titer is being established. The titration is performed several times, and the average result is taken.[1,2,3]

Calculating the titer using the working substance. This is the mass of a solute contained in a milliliter of aqueous solution, expressed in grams. The titer is calculated as the ratio of the mass of the solute to the volume of the solution (g/ml).

$T = m / V$

where: m is the mass of the solute, g; V is the total volume of the solution, ml;

$T = E * N / 1000$ (g / ml)

Sometimes, a correction factor or correction factor (K) is used to indicate the exact concentration of titrated solutions. K = actual sample taken / calculated sample.

The correction factor indicates the number by which the volume of a given solution must be multiplied to bring it to the volume of a solution of a given normality.

Obviously, if the correction factor for a given solution is greater than one, then its actual normality is greater than the normality taken as the standard; if the correction factor is less than one, then the actual normality of the solution is less than the standard normality. Example: 200 ml of a solution are prepared from 1.3400 g of reagent grade NaCl. Calculate the correction factor to bring the concentration of the prepared solution to exactly 0.1 N.

Solution: 200 ml of 0.1 N. The NaCl solution should contain

$58.44 * 0.1 * 200/1000 = 1.1688$ g

Hence: $K = 1.3400 / 1.1688 = 1.146$

The correction can be calculated as the ratio of the titer of the prepared solution to the titer of a solution of a given normality:

K = Titer of the prepared solution / Titer of a solution of a given normality

In our example, the titer of the prepared solution is 1.340 / 200 = 0.00670 g/ml

The titer of a 0.1 N NaCl solution is 0.005844 g/ml

Hence $K = 0.00670 / 0.005844 = 1.146$

Conclusion: If the correction for a given solution is greater than one, then its actual normality is greater than the normality taken as the standard; if the correction is less than one, then its actual normality is less than the standard.[3]

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Titration Methods: The following titration methods are distinguished:

Direct titration - the substance being determined reacts directly with the titrant: $KOH + HCl = KCl + H_2O$

Back titration - an excess of titrant-1 is used, which is then titrated with titrant-2. For example:

$NaCl + AgNO_3 \rightarrow AgCl + NaNO_3$

$AgNO_3 + NH_4SCN \rightarrow AgSCN + NH_4NO_3$

Substitution titration is used when direct interaction is impossible, e.g.:

$K_2Cr_2O_7 + 6\ KI + 7\ H_2SO_4 \rightarrow Cr_2(SO_4)_3 + 3\ I_2 + 4\ K_2SO_4 + 7\ H_2O$

$I_2 + 2\ Na_2S_2O_3 \rightarrow 2\ NaI + Na_2S_4O_6$

Working solutions and standardization: Solutions used in titrimetric analysis are called titrants (secondary standard solutions). Their concentration is determined using primary standards—substances with a constant composition and high purity.

Requirements for standard substances:

• high molar mass (to reduce weighing errors);

• constant chemical composition;

• easy purification (by crystallization);

• resistance to oxidation, moisture, and atmospheric CO_2.[3,4]

Standardization example: $NaOH + HCl = NaCl + H_2O$

The concentration of HCl is determined by weighing Na_2CO_3 or $Na_2B_4O_7$ $10H_2O$.

For convenience, fixanals—standard ampoules containing a known amount of equivalent substance (0.1 mol/L)—are used, significantly simplifying the preparation of titrated solutions.[3]

Practical recommendations: When conducting titrimetric analysis, it is necessary to:

• maintain accuracy when weighing and measuring volumes (up to 0.01–0.02 cm^3);

• Use titrants with a concentration of 0.1 N;

• Take sample weights of at least 0.2 g;

• Calibrate equipment regularly;

Perform mathematical processing of the obtained data. [4]

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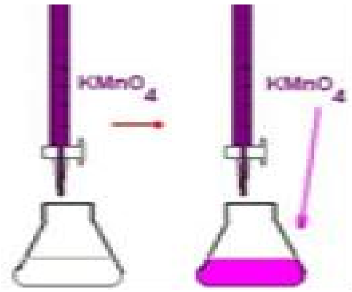

Conclusion: Titrimetric analysis is a versatile, rapid, and accurate method for the quantitative determination of substances. It is widely used in inorganic, organic, and analytical chemistry, as well as in pharmaceutical analysis for quality control and standardization of chemicals.

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