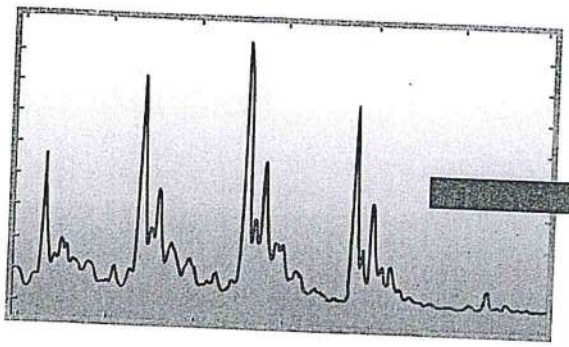
PROVA 1

1. Illustrare la gestione dei rifiuti in un laboratorio chimico/tecnologico.

2. Quali sono le applicazioni dell'analisi viscosimetrica?

3. Attività istituzionali dell'Ateneo

4. Come si imposta un semplice calcolo (somma, sottrazione...) del contenuto di più celle in Excel?



CHAPTER 5

TITRIMETRIC METHODS OF ANALYSIS

The three types of quantitative titrimetry are: volumetric, gravimetric, and coulometric. Volumetric is by far the most widely used.

Titrimetric methods are analytical procedures in which the amount of analyte is determined from the amount of a standard reagent required to react with the analyte completely.

Titrimetric methods constitute a large and powerful group of quantitative procedures that find widespread use in analytical chemistry. Volumetric methods represent one of three types of *titrimetry* in which analyses are based upon measuring the amount of a reagent of known concentration that is consumed by the analyte. The other two types are *weight* (or *gravimetric*) *titrimetry* and *coulometric titrimetry*. In volumetric titrimetry, the volume of a solution of known concentration that is needed to react essentially completely with the analyte is determined. Gravimetric titrimetry differs only in that the weight of the reagent is measured instead of its volume. In coulometric titrimetry, the "reagent" is a constant direct electrical current of known magnitude that reacts with the analyte; here, the time required to complete the electrochemical reaction is measured.

This chapter provides an introduction to volumetric methods. Chapters 9 through 16 cover the theory and applications of various types of volumetric titrimetry, although much of the material also applies to weight titrimetry. Coulometric titrimetry is considered in Section 18D–5. All these titrimetric methods are used for routine analyses because they are generally rapid, convenient, accurate, and readily automated.

5A SOME GENERAL ASPECTS OF VOLUMETRIC TITRIMETRY[1]

5A–1 Definition of Some Terms

A *standard solution* (or *standard titrant*) is a reagent of known concentration that is used to carry out a volumetric analysis. A *titration* is performed by slowly adding a standard solution from a buret or other volumetric measuring device to a solution of the analyte until the reaction

[1]For a detailed discussion of volumetric methods, see J. I. Watters, in *Treatise on Analytical Chemistry*, I. M. Kolthoff and P. J. Elving, Eds., Part I, Vol. 11, Chapter 114. New York: Wiley, 1975.

PROVA 2

1. Avendo rinvenuto in laboratorio un barattolo contenente un solido bianco incognito come procederebbe per identificare la sostanza incognita?

2. Quali sono le finalità di un processo di macinazione e come si esegue la macinazione in ambito tecnologico?

3. Il Rettore – funzioni

4. Excel: cosa si intende per filtro, e come lo si può usare?

14. AQUEOUS SOLUTIONS

Although a saturated solution, an aromatic water contains very low concentration of the solute. Other pharmaceutical solutions may contain a very high concentration of solute and yet not form a saturated solution. Syrup contains 850 g of sucrose and 450 ml of water per liter, yet it is not saturated as 1 g of sucrose dissolves in 0.5 ml of water.

A concentrated solution is usually capable of dissolving other solutes; however, the solubility of the first solute may be increased or decreased. In many pharmaceutical solutions of nonelectrolytes, the addition of a water-soluble second solute will decrease the solubility of the first solute. When the solubility of a nonelectrolyte is decreased in this manner, the effect is referred to as salting out.

Salting out occurs because the ions of the added electrolyte combine with water to form hydrates, thus reducing the amount of water available for solution of the nonelectrolyte. The greater the degree of hydration, the greater the reduction in solubility of the nonelectrolyte.

The fact that 2 g of sugar dissolves in 1 ml of water indicates that the hydration or hydrogen bonding between sucrose and water is very strong. This strong association between the solute and solvent prevents, to any great extent, further association of the water dipoles with additional water-soluble drugs; thus, syrups have a lowered solvent power for other water-soluble drugs which may be added. This is the reason the practicing pharmacist may find it difficult to dissolve a drug in a syrup, yet the drug would readily dissolve in the same volume of water.

Dilute sucrose solutions are good media for microbial growth, and concentrated sucrose solutions retard microbial growth. If a saturated solution were employed in pharmacy, it would prevent microorganic growth, but with a change in temperature it might lead to the formation of crystals which would be difficult to redissolve. Industrially formulated syrups contain other ingredients to obtain the desired solubility for the drug as well as for improvement of taste, appearance, and stability. In developing such products it is an economic necessity to consider the additive preservative effects of ingredients such as alcohol, glycerin, sugar, propylene glycol, and dissolved solids.

Syrup U. S. P. contains 850 g of sucrose per 1000 ml of syrup or 65% sucrose by weight. The minimum amount of sucrose required to preserve a neutral syrup is about 65-68% by weight. Assuming that one wishes to formulate a syrup containing only 500 g of sucrose per 1000 ml, the amount of alcohol that must be added to preserve this product may be estimated. This requires the consideration of the U.S.P. Syrup equivalent and the free water equivalent.

If 850 g of sucrose preserves 450 ml of water in a liter of Syrup U. S. P., then 1 g of sucrose will preserve 450/850 or 0.53 ml of water. In the formulation the 500 g of sucrose will preserve 500 X 0.53 or 265 ml of water.

In Syrup U. S. P. 850 g of sucrose apparently assumes a volume of 550 ml, and 1 g of sucrose occupies a volume of 550/850 or 0.647 ml. Then, if 500 g of sucrose preserves 265 ml of water, the total volume of solution which would be equivalent to U. S. P. Syrup would be the sum of the volume occupied by the sucrose (500 X 0.647) and the volume of water preserved by this amount of sucrose (265) or 589 ml. The free-water equivalent per liter of the formulation is 1000 - 589 or 411 ml.

If this 411 ml requires 18% alcohol to preserve it, then 411 X 0.18 or 74 ml of absolute alco-

PROVA 3

1. Illustrare i principali fattori di rischio in un laboratorio chimico/tecnologico

2. Quali sono le applicazioni dell'analisi calorimetrica?

3. Il Consiglio di Amministrazione – funzioni

4. Come inserire una intestazione e delle note a piè di pagina in un documento Word?

between the two is complete. The volume needed to complete the titration is determined from the difference between the initial and final buret readings.

The *equivalence point* in a titration is reached when the amount of added titrant is chemically equivalent to the amount of analyte in the sample. For example, the equivalence point in the titration of sodium chloride with silver nitrate occurs after exactly one mole of silver ion has been added for each mole of chloride ion in the sample. The equivalence point in the titration of sulfuric acid with sodium hydroxide is reached after the introduction of two moles of base for each mole of acid.

The equivalence point is the point in a titration when the amount of added standard reagent exactly equals the amount of analyte.

It is sometimes necessary to add an excess of the standard titrant and then determine the excess by *back-titration* with a second standard titrant. Here, the equivalence point corresponds to the point where the amount of initial titrant is chemically equivalent to the amount of analyte plus the amount of back-titrant.

Back-titrations are often required when the rate of reaction between the analyte and reagent is slow or when the reagent lacks stability.

5A–2 Equivalence Points and End Points

The equivalence point of a titration is a theoretical point that cannot be determined experimentally. Instead, we can only estimate it by observing some physical change associated with the condition of equivalence. This change is called the *end point* for the titration. Every effort is made to ensure that any volume difference between the equivalence point and the end point is small. Such differences do exist, however, as a result of inadequacies in the physical changes and in our ability to observe them. The difference in volume between the equivalence point and the end point is the *titration error*.

The end point is the point in a titration when a physical change occurs that is associated with the condition of chemical equivalence.

All volumetric methods are based upon a primary standard whose chemical composition and purity are known exactly.

An *indicator* is often added to the analyte solution in order to give an observable physical change (the end point) at or near the equivalence point. We shall see that large changes in the relative concentrations of analyte and titrant occur in the equivalence-point region. These concentration changes cause the indicator to change in appearance. Typical indicator changes are the appearance or disappearance of a color, a change in color, and the appearance or disappearance of turbidity.

In volumetric methods, the titration error E_t is given by

$$E_t = V_{ep} - V_{eq}$$

where V_{ep} is the actual volume used to arrive at the end point, and V_{eq} is the theoretical volume of reagent required to reach the equivalence point.

We often use instruments to detect end points. These instruments respond to certain properties of the solution that change in a characteristic way during the titration. Among such instruments are voltmeters, ammeters, and ohmmeters; colorimeters; temperature recorders; and refractometers.

5A–3 Primary Standards

A *primary standard* is a highly purified compound that serves as a reference material in all volumetric titrimetric methods. The accuracy of such methods is critically dependent on the properties of this compound. Important requirements for a primary standard are:

1. High purity. Established methods for confirming purity should be available.

PROVA 4

1. Illustrare i principali dispositivi di sicurezza in un laboratorio chimico/tecnologico

2. In quali ambiti della tecnologia farmaceutica trova applicazione la valutazione della densità?

3. Il Collegio dei revisori dei conti

4. Come si può realizzare una tabella in Word?

4

10. COMPRESSED TABLETS

Without question, the compressed tablet is the most popular dosage form today. There are approximately 380 official tablets, and about one half of the prescriptions dispensed are for tablets.

Usually one considers a compressed tablet as an oral medication; however, compressed tablets have many other uses. The sublingual tablet, the pellet, the wafer, the dental cone, the troche, and the vaginal insert are manufactured by the same procedure as an oral tablet.

PROCEDURE. There are three methods of making compressed tablets. In the direct compression method, a compressible vehicle is blended with the medicinal compound, and if necessary, with a lubricant and a disintegrating agent, and then the blend is compressed. Substances that have been suggested as directly compressible vehicles are: anhydrous and spray dried lactose, dicalcium phosphate dihydrate (Emcompress), granulated mannitol, microcrystalline cellulose (Avicel), compressible sugar (Di-Pac), starch (Sta-Rx 1500), hydrolyzed starch (Celutab), and a blend of sugar, invert sugar, starch and magnesium stearate (Nutab).

In the slugging method the ingredients in the formulation are intimately mixed and precompressed on heavy-duty tablet machines. The slug which is formed is ground to a uniform size and compressed into the finished tablet.

The wet granulation method has more operational manipulations and is more time-consuming than the other methods: however, it is widely used. The wet granulation method cannot be used for drugs which are thermolabile or are degraded by the liquid binder. The wet granulation procedure is done in the following steps:

1. The granulating solution or binder is prepared.
2. The powdered ingredients are weighed and mixed intimately.
3. The powders are granulated by adding an appropriate amount of binding solution and are kneaded to the proper consistency.
4. The wet granulation is forced through a screen or wet granulator.
5. The granules are dried in an oven.
6. The dried granules are ground to the final and suitable size for compression.
7. A lubricant and a disintegrating agent are mixed with the granulation.
8. The granulation is compressed into the finished tablet.

A. By manual means, prepare 500 tablets containing 250 mg of sulfadiazine per tablet.

	mg per tablet
Sulfadiazine, powdered	250
Starch	18
Lactose	60
Magnesium stearate	2

In a tared container prepare a 10% w/w starch paste by first making a slurry with 25 g of starch and 25 g of purified water. Add 200 g of boiling purified water to the slurry and stir. A translucent gel is formed.

PROVA 5

1. Descrivere le principali informazioni riportate sulle schede di sicurezza dei reagenti chimici necessarie alla loro gestione in laboratorio.

2. Quali applicazioni trova la filtrazione in ambito tecnologico-chimico farmaceutico?

3. Il Direttore generale e i dirigenti

4. Che cosa si può fare con PowerPoint?

24. RHEOLOGY

Rheology is the study of the flow of fluids and the deformation of solids. Resistance is offered when one part of a liquid is moved past another. The force required to slip one layer of a liquid past another with a given velocity depends directly on the viscosity of the liquid and on the areas exposed to each other, and inversely on the distances separating the two surfaces. The coefficient of absolute viscosity of a liquid, η, can be defined as the force per unit area necessary to maintain a unit velocity gradient between two parallel planes separated by a unit distance. Mathematically this may be expressed

$$\eta = \frac{\frac{F}{A}}{\frac{dv}{dx}}$$

where F/A is the force per unit area acting parallel to the planes in dynes cm^{-2}, and dv/dx is the velocity gradient perpendicular to the planes per second. Although the dimension of absolute viscosity is g cm^{-1} sec^{-1}, this unit is designated as a poise. A liquid has a viscosity of one poise when the force required to maintain a relative velocity of 1 cm sec^{-1} between two parallel planes 1 cm apart is 1 dyne cm^{-2}.

As most liquids used in pharmacy have a viscosity of less than one poise, the centipoise, equal to 0.01 poise, is commonly used to express viscosity.

Pure liquid compounds (acetone, alcohol, glycerin, water) and true solutions have a constant viscosity at a given temperature and pressure. Thus, when the liquid is placed in a rotational viscometer and subjected to a given rate of shear, the observed stress becomes constant and is directly proportional to the rate of shear. Such fluids are known as Newtonian fluids. Flow curves are obtained by plotting the rate of shear (r.p.m.) against the shear stress (divisions of rotational viscometer). Newtonian fluids produce a straight line which passes through the origin as shown in Figure 14. Viscosity is the reciprocal of the slope of the flow curve. Curve A represents a less viscous fluid than Curve B. A given stress causes a lower shear rate for fluid B than for A.

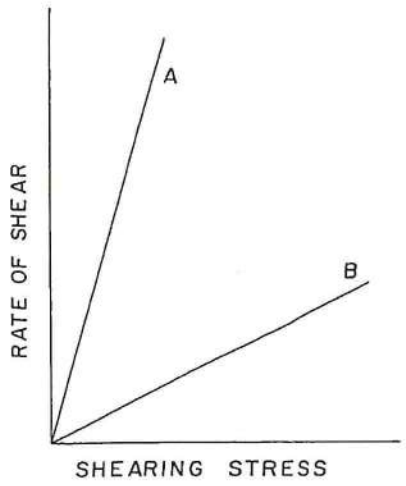


Figure 14 — Flow curve or rheogram for a Newtonian system.

The viscosity of most liquids decreases with an increase in temperature. This suggests the existence of molecular clustering or association in liquids. An increase in temperature or kinetic energy disrupts these associations wiht a resulting decrease in viscosity. Solutions of liquids which associate to a great degree or which are strong dipoles may vary greatly in viscosity. Such solutions may exhibit a maximum in their viscosity-composition curve which is greater than the viscosity of either pure liquid. The maximum represents the greatest degree of association or hydrogen bonding between the components of the solution, and this large association complex increases the resistance to movement within the solution which is apparent as an increase in viscosity.

In the case of an ideal solution in which no interactions occure, viscosity is additive. Mathematically

PROVA 6

1. Descrivere la preparazione di una soluzione tampone fosfato a pH=7.4 (pKa1=2.0, pKa2=6.8; pKa3=12)

2. Quali attrezzature di base sono necessarie in un laboratorio di tecnologia dove si allestiscono preparazioni solide?

3. Il Comitato Unico di Garanzia

4. Cosa si intende per "phishing" e come evitarlo?

of organic molecules. Nuclear magnetic resonance, which occurs in the radiofrequency part of the spectrum, is discussed in Chapters 3 and 4, while ultraviolet and visible spectroscopy are described in Chapter 5.

TABLE 2–1 Types of Energy Transitions in Each Region of the Electromagnetic Spectrum

Region of Spectrum	Energy Transitions
X-rays	Bond Breaking
Ultraviolet/Visible	Electronic
Infrared	Vibrational
Microwave	Rotational
Radiofrequencies	Nuclear Spin (Nuclear Magnetic Resonance) Electron Spin (Electron Spin Resonance)

Many chemists refer to the radiation in the vibrational infrared region of the electromagnetic spectrum in terms of a unit called *wavenumbers* ($\bar{\nu}$). Wavenumbers are expressed as cm^{-1} (reciprocal centimeters), and are easily computed by taking the reciprocal of the wavelength (λ) expressed in centimeters. They may be converted to a frequency (ν) by multiplying them by the speed of light (expressed in cm/sec).

$$\bar{\nu}\ (cm^{-1}) = \frac{1}{\lambda(cm)} \qquad \nu(Hz) = \bar{\nu}c = \frac{c(cm/sec)}{\lambda(cm)}$$

This unit has the advantage, for those performing calculations, that it is directly proportional to energy. Thus, in terms of wavenumbers, the vibrational infrared extends from about 4000 to 650 cm^{-1}. Interconversions may be made between wavelengths (μ or μm) and wavenumbers (cm^{-1}) or between wavenumbers and wavelengths by using the following relationships:

$$cm^{-1} = \frac{1}{(\mu)} \times 10{,}000 \quad \text{and} \quad \mu = \frac{1}{(cm^{-1})} \times 10{,}000$$

2.1 THE INFRARED ABSORPTION PROCESS

As with other types of energy absorption, molecules are excited to a higher energy state when they absorb infrared radiation. The absorption of infrared radiation is, like other absorption processes, a quantized process. Only selected frequencies (energies) of infrared radiation will be absorbed by a molecule. The absorption of infrared radiation corresponds to energy changes on the order of from 2 to 10 kcal/mole. Radiation in this energy range corresponds to the range encompassing the stretching and bending vibrational frequencies of the bonds in most covalent molecules. In the absorption process, those frequencies of infrared radiation which match the natural vibrational frequencies of the molecule in question will be absorbed, and the energy absorbed will serve to increase the *amplitude* of the vibrational motions of the bonds in the molecule. It should be

PROVA 7

1. Descrivere la preparazione di un litro di soluzione di HCl 0.1N a partire da HCl concentrato 37 % p/v

2. Quali attrezzature di base sono necessarie in un laboratorio di tecnologia dove si allestiscono preparazioni liquide e semisolide?

3. Il Collegio di disciplina

4. Che cosa significa PEC e in che cosa consiste?

12. COATING OF SOLIDS

An enteric coating is one which does not disintegrate in the stomach but rapidly disintegrates or dissolves in the intestine. Enteric coatings usually are formulated with ingredients which have acidic groups and are consequently insoluble in the low pH of the stomach, but they are more soluble in the higher pH of the intestine.

The enteric substance may also be hydrolyzed by enzymes of the intestine and emulsified and dispersed by the bile salts. Time-disintegration coating utilizes a waxy base blended with a hygroscopic substance which absorbs moisture, swells, and ruptures the enteric coating.

PROCEDURE. Many formulas have been suggested for the extemporaneous coating of pills and capsules; however, none have been completely satisfactory.

A. Coat the hexamethylenetetramine and ammonium chloride capsules previously prepared with the following coating:

n-butyl stearate	45 g
Carnauba wax	30 g
Stearic acid	25 g

Fuse the mixture in a water bath at 75° and keep at this temperature during the coating process. Hold the capsule by means of a forceps and dip it into the molten mixture. Withdraw the capsule and gently touch its end to the lip of the beaker to remove the excess coating. Allow the coating to congeal, reverse the capsule, and repeat the operation.

B. With the above enteric coating, coat eight sodium salicylate tablets which you prepared.

C. A 10% cellulose acetate phthalate solution will be supplied by the instructor. Coat eight sodium salicylate tablets with cellulose acetate phthalate. Place 25-30 ml of the solution in a small beaker and drop in a tablet. Remove the tablet immediately with a forceps, allowing the excess coating to drain away. Place the tablet on a paper and allow it to dry. When the tablet is dry, repeat the process until a continuous coat has been applied.

D. Using a U.S.P. disintegration apparatus, test the disintegration time of the two types of enteric coating applied to the sodium salicylate tablets.

Immerse the basket containing six coated sodium salicylate tablets in simulated gastric fluid T.S. at 37° and operate the apparatus for one hour. A properly enteric coated tablet will show no distinct evidence of dissolution or disintegration.

Replace the simulated gastric fluid with simulated intestinal fluid T. S. and operate the apparatus for one hour at 37° using six tablets that have withstood exposure to the gastric fluid. At the end of this time, the coatings of all six tablets must be broken and the contents dissolved or softened so that all material remaining on the screen is soft.

In vitro test methods may indicate that a dosage form possesses the desired characteristics of drug release, but ultimately the pattern of availability of a drug must be determined in humans. The resistance of an enteric formulation to simulated gastric fluid in a U. S. P. disintegration apparatus strongly suggests an enteric coating is satisfactory; however, only by admin-

PROVA 8

1. Illustrare le procedure per ottimizzare una separazione mediante flash-cromatografia

2. Quali sono i principali metodi applicati nei processi di essiccamento dei liquidi in ambito tecnologico?

3. Le funzioni del Direttore di Dipartimento

4. Che cos'è la Firma Digitale?

SOLIDS

4. CHARACTERISTICS OF PARTICLES

The reduction of particle size of drugs is important in pharmacy. Not only do small particles of various solid drugs mix to form a more uniform dose than larger particles, but properties of the drug itself are modified by a reduction in particle size.

As the particles of a drug are reduced in size there is a more rapid release of the drug from the solid, because with smaller particle size there is a greater specific surface area. The dissolution of a given weight of a drug is directly proportional to the surface area. Thus, an increase in area causes more rapid solution, which is followed by increased absorption from the gastrointestinal tract. This is especially important with sparingly soluble drugs, such as the corticosteroid and sulfa drugs. Microcrystalline sulfadiazine given orally appears more rapidly and in higher concentrations in the blood than ordinary sulfadiazine powder. The experimental results shown in Figure 1 are actually due to an increase in exposed surface. It has been demonstrated that 0.5 g of microcrystalline griseofulvin produced blood concentrations equal to or higher than 1.0 g doses of regular griseofulvin. A high fat diet administered with microcrystalline griseofulvin will more than double the blood concentration.

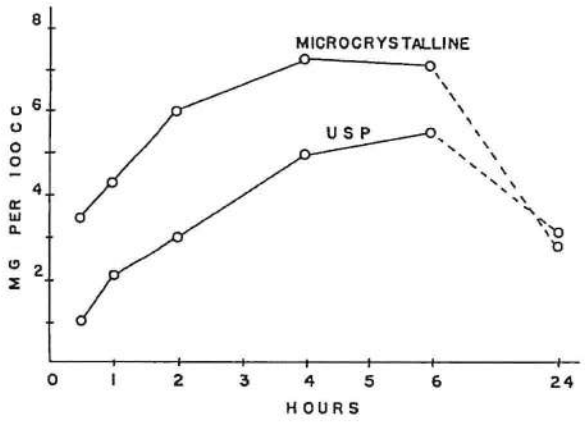


Figure 1 — Serum levels of sulfadiazine and microcrystalline sulfadiazine after oral administration of 3 g to humans, showing that a higher serum level is obtained when the drug has a smaller particle size (J. G. Reinhold, F. J. Phillips, H. F. Flippin, and L. Pollack: Am. J. Med. Sci., 210, 141, 1945).

The effect of particle size on plasma concentration of phenacetin is shown in Figure 2 for fine ($< 75\ \mu m$), medium (150-180 μm), and coarse ($> 250\ \mu m$) particles. The fine particles produce the greatest plasma concentration followed in decreasing order by medium and coarse particles. The addition of 0.1% Polysorbate 80 to the fine particles increased the plasma concentration of phenacetin presumably due to the surface-active agent wetting the particles to provide a greater effective surface from which dissolution could occur.

The plasma concentration of phenacetin at any time is the resultant of the simultaneous processes of dissolution, absorption, tissue equilibrium and metabolism by the hepatic microsomal enzymes since negligible phenacetin appears in the urine. The major metabolite of phenacetin is N-acetyl-p-aminophenol. Although the concentration of N-acetyl-p-aminophenol in the plasma indicates a less marked difference between the fine, medium and coarse particles, the plasma concentrations as shown in Figure 3 are related in the same order to the size of the particles as the unmetabolized drug.

The effect of particle size upon solution and absorption has also been demonstrated with novobiocin. When the crystalline acid form of novobiocin is given orally, practically no absorption occurs as indicated by blood levels. If this antibiotic is micronized or precipitated in a finely divided, amorphous form, it becomes an orally effective drug. As the sodium and calcium salts of novobiocin are water soluble and have a rapid dissolution rate, their particle size does not significantly influence plasma concentration.

PROVA 9

1. Descrivere la preparazione di una soluzione 0.1 M di H_2SO_4 a partire da una soluzione di H_2SO_4 1.0 M e una soluzione di H_2SO_4 0.02 M.

2. Quali metodi di controllo delle dimensioni particellari dei solidi possono essere utilizzati?

3. Il Codice Etico e la Commissione etica

4. Perché comprimere un file prima di inviarlo, e come fare?

lary action or under the influence of gravity. The stationary phase can also be an immobilized liquid which is immiscible with the mobile phase. Several procedures are employed to fix the stationary liquid in place. For example, a finely divided solid, coated with a thin layer of liquid, may be held in a glass or metal tube through which the mobile phase flows or percolates. Ordinarily the solid plays no direct part in the separation, functioning only to hold the stationary liquid phase in place by adsorption. Alternatively, the inner walls of a capillary tube can be coated with a thin layer of liquid; a gaseous mobile phase is then caused to flow through the tube. Finally, the stationary liquid phase can be held in place on the fibers of paper or on the surface of finely ground particles held on a glass plate.

Table 24-1 classifies common chromatographic methods according to the nature of the stationary and mobile phases. Most of the theoretical discussions in this chapter will be concerned with partition and gas-liquid chromatography. With suitable modification, these concepts can be adapted to the other type as well.

Linear Chromatography

All chromatographic separations are based upon differences in the extent to which solutes are partitioned between the mobile and the stationary phase. The equilibria involved can be described quantitatively by means of a temperature-dependent constant, the *partition coefficient* K:

$$K = \frac{C_S}{C_M} \qquad (24\text{-}1)$$

where C_S is the analytical concentration of a solute in the stationary phase and C_M is its concentration in the mobile phase. In the ideal case, the partition ratio is constant over a wide range of solute concentrations; that is, C_S is directly proportional to C_M. More often than not, however, nonlinear relationships occur. Some typical distribution curves are shown in Figure 24-1.

The ideal relationship, shown by curve C in this figure, is often approximated by distribution equilibria between two immiscible liquids, provided association or dissociation reactions do not occur in one of the solvents. Where such equilibria do exist, a relationship

TABLE 24-1 CLASSIFICATION OF CHROMATOGRAPHIC SEPARATIONS

Name	Type Mobile Phase	Type Stationary Phase	Method of Fixing the Stationary Phase
Gas-liquid	Gas	Liquid	Adsorbed on a porous solid held in a tube or adsorbed on the inner surface of a capillary tube
Gas-solid	Gas	Solid	Held in a tubular column
Partition	Liquid	Liquid	Adsorbed on a porous solid held in a tubular column
Adsorption	Liquid	Solid	Held in a tubular column
Paper	Liquid	Liquid	Held in the pores of a thick paper
Thin layer	Liquid	Liquid or solid	Finely divided solid held on a glass plate; liquid may be adsorbed on particles
Gel	Liquid	Liquid	Held in the interstices of a polymeric solid
Ion exchange	Liquid	Solid	Finely divided ion-exchange resin held in a tubular column

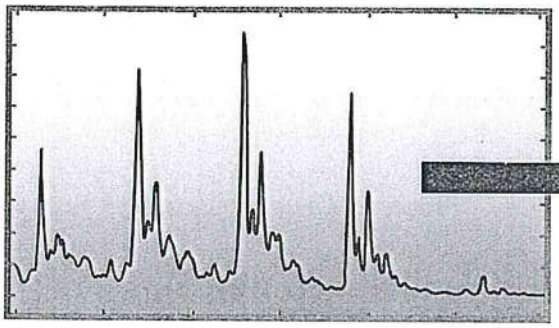
PROVA 10

1. Descrivere la preparazione di una soluzione a concentrazione esatta di idrossido di sodio.

2. A quali preparati farmaceutici viene applicato il test di disaggregazione e in cosa consiste?

3. Il Consiglio di Dipartimento e la Giunta: competenze.

4. A cosa serve il backup dei dati?



CHAPTER 4

GRAVIMETRIC METHODS OF ANALYSIS

Gravimetric methods of analysis are based upon mass measurements with an analytical balance, an instrument that yields highly accurate and precise data. In fact, if you perform a gravimetric chloride analysis in the laboratory, you may make some of the most accurate and precise measurements of your life.

Gravimetric methods of analysis, which are based upon the measurement of mass, are of two major types.[1] In *precipitation methods*, the analyte is converted to a sparingly soluble precipitate that is filtered, washed free of impurities, and converted to a product of known composition by suitable heat treatment. This product is then weighed. For example, in a precipitation method for determining calcium in natural water recommended by the Association of Official Analytical Chemists, an excess of oxalic acid, $H_2C_2O_4$, is added to a carefully measured volume of the sample. The addition of ammonia then causes essentially all the calcium in the sample to precipitate as calcium oxalate. The reaction is

$$Ca^{2+}(aq) + C_2O_4^{2-}(aq) \rightarrow CaC_2O_4(s)$$

The precipitate is collected in a weighed filtering crucible, dried, and then ignited at red heat. This process converts the precipitate entirely to calcium oxide:

$$CaC_2O_4(s) \rightarrow CaO(s) + CO(g) + CO_2(g)$$

The crucible and precipitate are cooled, weighed, and the weight of calcium oxide is determined by subtraction of the known weight of the crucible. The calcium content of the sample is then computed from the stoichiometry of the process as shown in the various examples in Section 4B.

In *volatilization methods*, the analyte or its decomposition products are volatilized at a suitable temperature. The volatile product is then collected and weighed, or alternatively the weight of the product is determined indirectly from the weight loss of the sample. An example of a gravimetric volatilization procedure is the determination of the sodium

[1]For an extensive treatment of gravimetric methods, see C. L. Rulfs, in *Treatise on Analytical Chemistry*, I. M. Kolthoff and P. J. Elving, Eds., Part I, Vol. 11, Chapter 13. New York: Wiley, 1975.