Analytical chemistry

Lecture №14

Titrations in analytical chemistry

Qualitative Analysis	Quantitative Analysis
Analysis for identification of species in a sample.	Analysis for determination of the quantities of species in a sample.

Quantitative Analytical Methods

Classical Methods	Instrumental Methods
If the analysis is carried out solely using solutions of chemical substances, this is called as classical analysis. - Gravimetric analysis - Volumetric analysis	If the analysis is performed using a device, then it is called instrumental analysis. - Spectroscopic analysis - Electrochemical analysis - Chromatographic analysis

Volumetric analysis is a quantitative analysis based on the measurement of the volume of solutions that gives reaction.

Characteristics of Quantitative Reaction

1) Reaction must be specific and unique
2) Reaction must be in one direction
3) The reaction must be fast
4) The end of the reaction can be detected easily
5) The reaction must be repeatable yielding same results every time.

Titration methods are based on determining the quantity of a reagent of known concentration that is required to react completely with the analyte.

Titrimetry permits us to determine a compound's concentration in a given solution by quantitatively measuring the quantity of reactant reacting with it.

standard solution is added to the sample solution until the end point (the detection of the completion of the reaction) is reached.

titrate a compound A in a solution at concentration C_A. *Increasing and known volumes of its* reactant B at concentration C_B *are added to an exactly known volume* V_A *of solution A.* B is chosen to react quantitatively with A according to a stoichiometric reaction.

$$V_A C_A = V_{Bf} C_B,$$

$$C_A = V_{Bf} C_B / V_A.$$

The solution of A is called the *titrand solution, and* that of *B* is the *titrant solution.*

Classification of volumetric methods

Titrimetric methods are classified into four groups based on the type of reaction involved.

1. Acid/base reactions; an acidic or basic titrant reacts with an analyte that is a base or an acid

$$HCl + NaOH \rightarrow NaCl + H_2O$$

$$Na_2CO_3 + HCl \rightarrow NaHCO_3 + NaCl$$

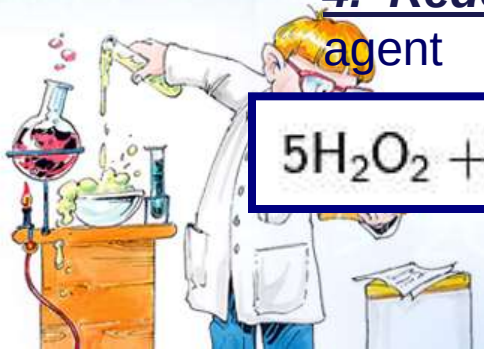
2. Precipitation reactions; the analyte and titrant react to form a precipitate

$$Ag^+ + Cl^- = AgCl\downarrow$$

3. Complexation reactions; involving a metal–ligand complexation reaction

$$Ca\text{-}EDTA^{2-}$$

4. Redox reactions the titrant is an oxidizing or reducing agent



$$5H_2O_2 + 2KMnO_4 + 3H_2SO_4 \rightarrow 5O_2 + 2MnSO_4 + K_2SO_4 + 8H_2O$$

Classification of Titrimetric Analysis Methods

Method	Technique	Titrant
Neutralisation (acid-basic titration)	Alkalimetry	MeOH
	Acidimetry	HAn
	Halometry	HAn, MeOH
Redoximety (reducing-oxidising)	Permanganatometry	$KMnO_4$
	Iodometry	I_2, $Na_2S_2O_3$
	Bromatometry	$KBrO_3$
	Cerimetry	$Ce(SO_4)_2$
	Vanadatometry	NH_4VO_3
	Titanometry	$Ti_2(SO_4)_3$
	Nitritimetry	$NaNO_2$
Precipitation titration	Argentometry	$AgNO_3$
	Mercurometry	$Hg_2(NO_3)_2$
	Rhodanometry	KSCN
Complexometry (complex compounds formation)	Mercurimetry	$Hg(NO_3)_2$
	Fluorimetry	NaF
	Complexonometry	EDTA
Nonaqueous titration	Solution of $HClO_4$ in acetic acid or nitrometane Solution of NaOH or CH_3ONa in methanol	

A **standard solution (or a standard titrant)** is a reagent of known concentration that is used to carry out a volumetric titration. The titration is performed by slowly adding a standard solution from a buret or other liquid-dispensing device to a solution of the analyte until the reaction between the two is judged complete. The volume or mass of reagent needed to complete the titration is determined from the difference between the initial and final readings.

The **titrant** solution must be standardized before the titration. In other words, its titer must be precisely known. The standardization of the titrant solution is achieved by its titration with a **standard solution**. This can be a ***primary or secondary (standard) solution***.

A **primary standard solution** is prepared simply by weighing the reagent in the pure state (called for this goal a primary standard), by dissolving it into the solvent, and making up the solution to a known volume.

A **secondary standard** is a substance that can also be used for standardizations. However, its solution content in the active substance has been found by comparison with a primary standard.

Standard Solutions

The *ideal standard solution for a titrimetric* method will

1. be sufficiently stable so that it is necessary to determine its concentration only once;

2. react rapidly with the analyte so that the time required between additions of reagent is minimized;

3. react more or less completely with the analyte so that satisfactory end points are realized;

4. undergo a selective reaction with the analyte that can be described by a balanced equation.

In a **standardization,** the concentration of a volumetric solution is determined by titrating it against a carefully measured quantity of a primary or secondary standard or an exactly known volume of another standard solution.

Two basic methods are used to establish the concentration of such solutions. The first is the **direct method** in which a carefully determined mass of a primary standard is dissolved in a suitable solvent and diluted to a known volume in a volumetric flask. The second is by **standardization** in which the titrant to be standardized is used to titrate (1) a known mass of a primary standard, (2) a known mass of a secondary standard, or (3) a measured volume of another standard solution. A titrant that is standardized is sometimes referred to as a **secondary standard solution.**

A **primary standard is a highly purified compound** that serves as a reference material in titrations and in other analytical methods.

Important requirements for a primary standard are the following:

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

2. Atmospheric stability.

3. Absence of hydrate water so that the composition of the solid does not change with variations in humidity.

4. Modest cost.

5. Reasonable solubility in the titration medium.

6. Reasonably large molar mass so that the relative error associated with weighing the standard is minimized.

A **secondary standard is a compound** whose purity has been determined by chemical analysis.

The point at which the titration reaction is just complete is called the ***equivalence point or the theoretical or stoichiometric endpoint***. *The volume added to the* equivalence point is simply called the *added volume to the equivalence point*

The **equivalence point** is the point in a titration when the amount of added standard reagent is equivalent to the amount of analyte.

The **end point** is the point in a titration when a physical change occurs that is associated with the condition of chemical equivalence. The point in a titration where we stop adding titrant.

We cannot determine the equivalence point of a titration experimentally. Instead, we can only estimate its position by observing some physical change associated with the condition of chemical equivalence.

In an ideal titration, the endpoint and the equivalence point coincide. In practice, a difference occurs.

The difference in volume or mass between the equivalence point and the end point is the **titration error.**

The completion of the titration reaction may be detectable with the help of a **chemical indicator**. After the titration reaction is complete, an auxiliary reagent dissolved previously in the titrand solution, called a *color indicator, causes a color* change in the solution being titrated. In this case, the indicator is called an **internal indicator.** An **external** one can also be used. A solution sample (a few drops) is taken and brought face to face with the indicator out of the titration vessel. In some cases there is no need for a color indicator since the titrant or titrand **itself** is colored. Then, at the equivalence point, a color may appear or disappear. A change in a physical property can also indicate this point. It can be detected with the help of an instrumental analysis method. The curve point at which the color changes or the physical change occurs is called the ***endpoint of the titration***.

For effect the reactions appearance indicators are ***reversible*** and ***unreversible***. Reversible indicators – changes the colour can be repeated many times as changes the system state. Unreversible indicators – colour changes ones with destruction of indicator molecule. The unreversible indicators are less comfortable and thinly use.

We often use i**nstruments** to detect end points. These instruments respond to properties of the solution that change in a characteristic way during the titration. Among such instruments are colorimeters, turbidimeters, spectrophotometers, temperature monitors, refractometers, voltmeters, current meters, and conductivity meters.

Steps in a Titration

Titrimetric analysis generally involve the following steps:

- Sampling;
- Titrant preparation;
- Standard preparation and conversion to a measurable form;
- Titrant standardization by titration of an accurately know quantity of standard;
- Sample preparation and conversion to a measurable form;
- Sample titration with the titrant solution;
- Data analysis.

Some Titration Forms

Several titration forms exist, including the following:

• **direct titrations**: the titrant solution is added to the titrand solution.

• **inverse titrations**: the solution of concern is added to a known volume of the titrant solution.

• **back titrations**: in the first step, a known volume of the titrant solution in excess is added to the solution under study. In the second step, the excess is titrated with any standard solution. Back titrations are used when the titrand cannot be directly titrated for various reasons, such as the kinetics of the titration reaction which are too slow. In this case, a higher concentration of reactant increases the reaction speed. They can also be used for practical reasons, such as having on hand an easier detection method for the equivalence point;

• **titrations by displacement or by substitution**: the titrand is replaced mole by mole with another species that is easier to titrate. For example, to the titrand M solution is added another species NA, which reacts quantitatively with it according to the reaction

$M + NA \rightarrow MA + N.$

Since the reaction is quantitative, M is replaced mole by mole with N, which is then titrated directly.

back titration

If the titration reaction is too slow, a suitable indicator is not available, or there is no useful direct titration reaction, then an indirect analysis may be possible. Suppose you wish to determine the concentration of formaldehyde, H_2CO, in an aqueous solution. The oxidation of H_2CO by I_3^- is a useful reaction, except that it is too slow for a direct titration. If we add a known amount of I_3^-, such that it is in excess, we can allow the reaction to go to completion. The I_3^- remaining can then be titrated with thiosulfate, $S_2O_3^{2-}$.

$$H_2CO(aq) + 3OH^-(aq) + I_3^-(aq) \rightleftharpoons HCO_2^-(aq) + 3I^-(aq) + 2H_2O(\ell)$$

$$I_3^-(aq) + 2S_2O_3^{2-}(aq) \rightleftharpoons S_4O_6^{2-}(aq) + 3I^-(aq)$$

displacement titration

Calcium ion plays an important role in many aqueous environmental systems. A useful direct analysis takes advantage of its reaction with the ligand ethylenediaminetetraacetic acid (EDTA), which we will represent as Y^{4-}. Unfortunately, it often happens that there is no suitable indicator for this direct titration. Reacting Ca^{2+} with an excess of the Mg^{2+}–EDTA complex releases an equivalent amount of Mg^{2+}. Titrating the released Mg^{2+} with EDTA gives a suitable end point. The amount of Mg^{2+} titrated provides an indirect measure of the amount of Ca^{2+} in the original sample.

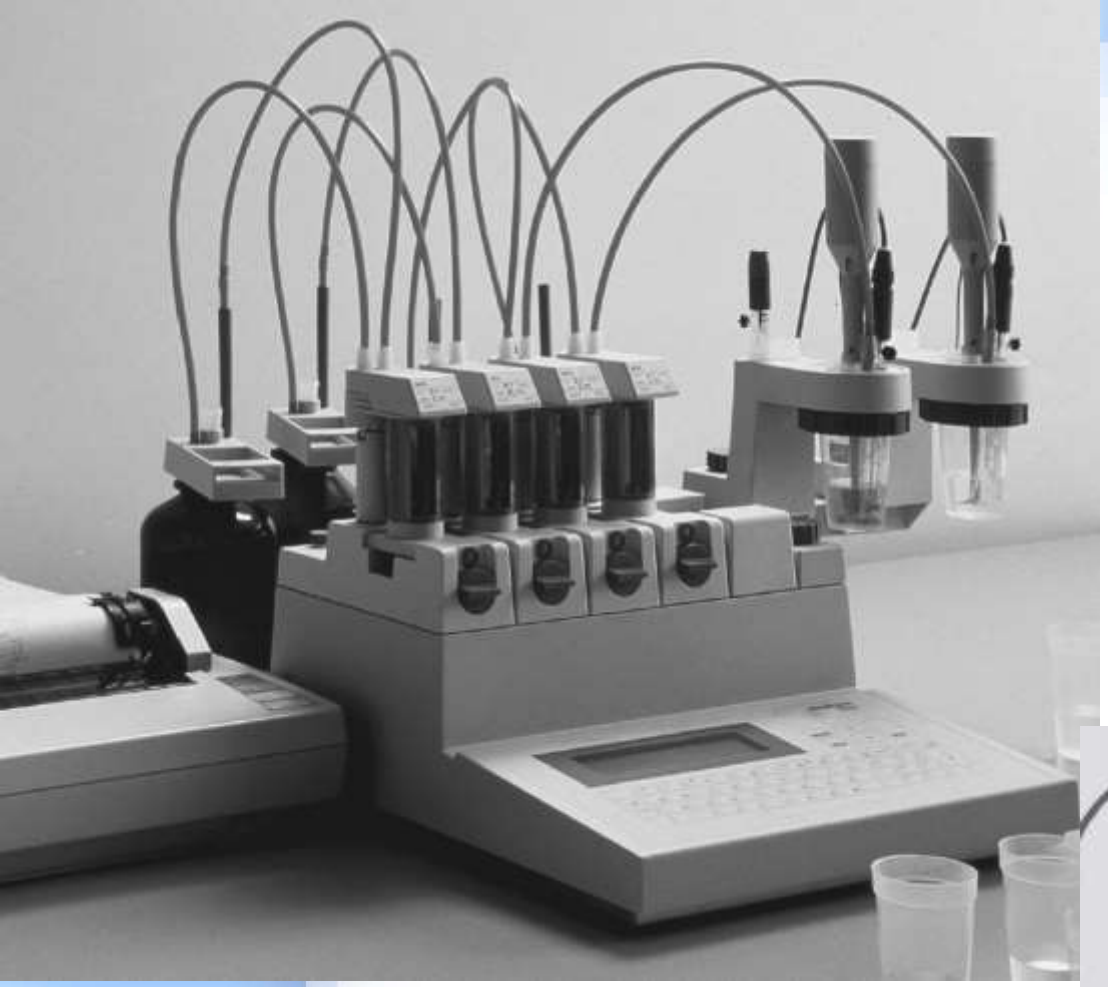
$$Ca^{2+}(aq) + Y^{4-}(aq) \rightleftharpoons CaY^{2-}(aq)$$

$$Ca^{2+}(aq) + MgY^{2-}(aq) \rightleftharpoons CaY^{2-}(aq) + Mg^{2+}(aq)$$

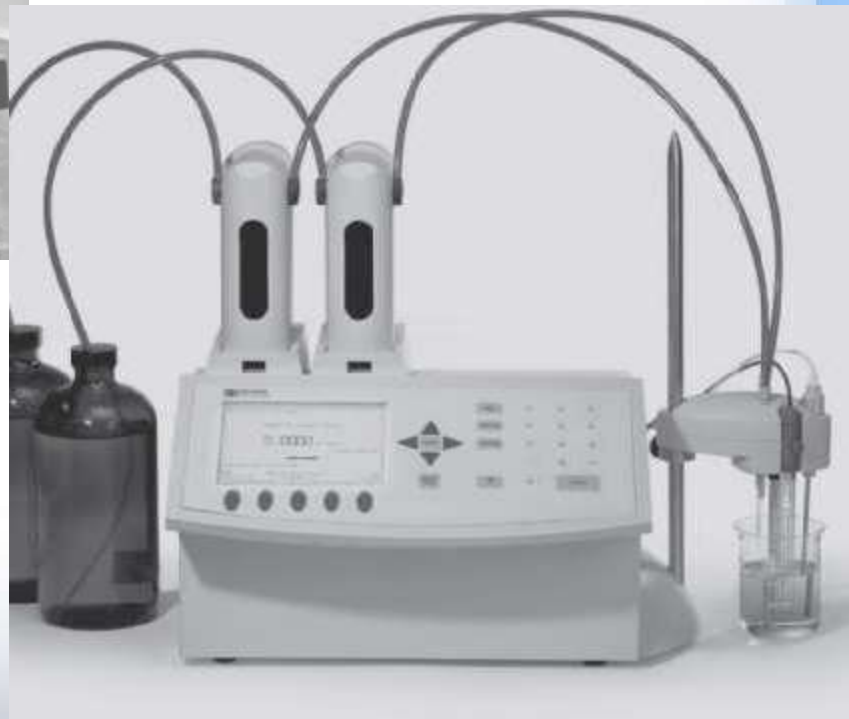
$$Mg^{2+}(aq) + Y^{4-}(aq) \rightleftharpoons MgY^{2-}(aq)$$

The **basic requirements** or components of a volumetric method are:

1. A standard solution (i.e., titrant) of known concentration, which reacts with the analyte with a known and repeatable stoichiometry (i.e., acid/base, precipitation, redox, complexation);
2. A device to measure the mass or volume of sample (e.g., pipet, graduated cylinder, volumetric flask, analytical balance);
3. A device to measure the volume of the titrant added (i.e., buret);
4. If the titrant-analyte reaction is not sufficiently specific, a pretreatment to remove interferents;
5. A means by which the endpoint can be determined. This may be an internal indicator (e.g., phenolphthalein) or an external indicator (e.g., pH meter).



Typical instrumentation for performing an automatic titration



Glassware Used in Titrimetry

Apparatus for Precisely Measuring Volume

Volume may be measured reliably with a **pipet, a buret, or a volumetric flask.** Volumetric equipment is marked by the manufacturer to indicate not only the manner of calibration (usually TD for “to deliver” or TC for “to contain”) but also the temperature at which the calibration strictly applies. Pipets and burets are usually calibrated ***to deliver specified volumes***. Volumetric flasks, on the other hand, are calibrated ***to contain a specific volume***.

Glassware types include Class A and Class B. Class A glassware is manufactured to the highest tolerances from Pyrex, borosilicate, or Kimax glass. Class B (economy ware) tolerances are about twice those of Class A.

Pipets and burets must be kept absolutely clean. It is good practice in general to see that all glassware is absolutely clean. To test for cleanliness, fill the object with water and then empty it. If the remaining water forms a uniform thin film on the walls with no beads, the object is clean.

To clean burets rinse repeatedly with DW. Pipets and burets with hard-to-remove residues must be cleaned using soap and water. A buret brush may be used for cleaning a buret. Take care not to scratch the insides of the buret. Rinse well with DW after cleaning or use. Always rinse pipets well with DW after use and store them flat.

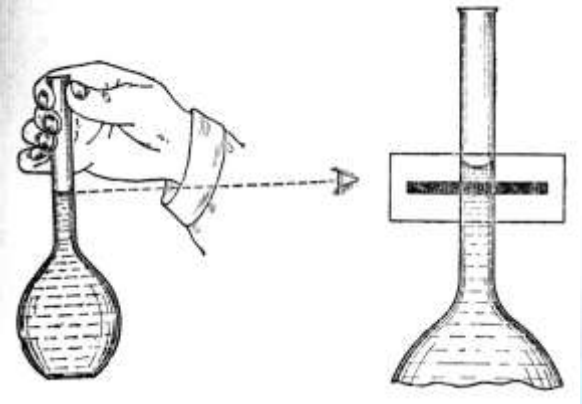
VOLUMETRIC FLASKS

Volumetric flask.

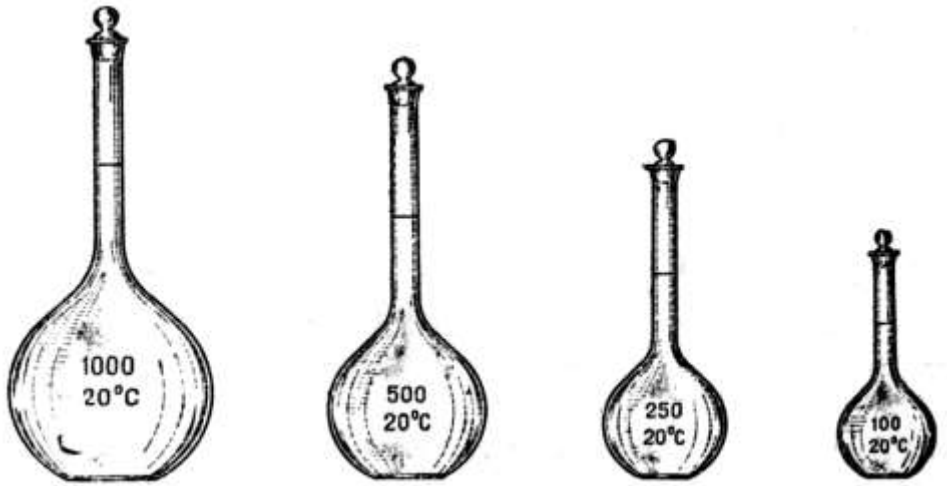
Volumetric flasks are used for the preparation of standard solutions and for the dilution of samples to a fixed volume prior to taking aliquots with a pipet.. They come in a variety of sizes, from 1mL to 2 L or more.

The volumetric shown below is calibrated to contain exactly 250 mL at 20°C and should be recorded as 250,0 mL.

In reading volumes, the eye must be at the level of the liquid surface to avoid an error due to **parallax.** Parallax is a condition that causes the volume to appear smaller than its actual value if the meniscus is viewed from above and larger if the meniscus is viewed from below.



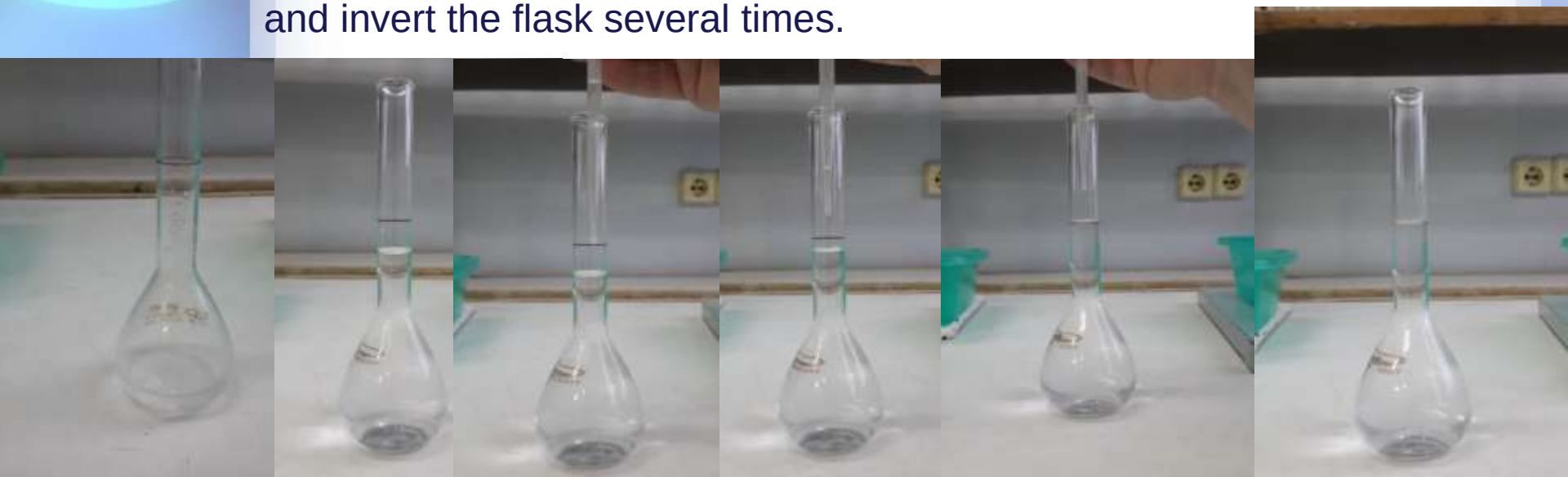
meniscus



1. Solid reagent (the solute) is transferred to the volumetric flask by using a funnel. The weighing boat is rinsed with the solvent used (usually DW) into the funnel. Then the funnel is rinsed with solvent while still inserted in the flask so that any remaining solute particles will be washed into the flask. In this way, the solute is transferred quantitatively - nothing is left behind.

2. After the solute has been added, the flask should be filled about **halfway**, and then swirled until all the solute has dissolved. Then solvent is added near to the calibration mark but not up to it.

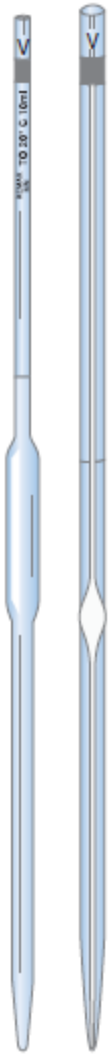
3. Final addition is accomplished using a medicine dropper. If the liquid level goes past the calibration mark the solution must be discarded. Make sure your eyes are leveled with the liquid surface, and add solvent drop-wise until the bottom of the meniscus is touching the calibration line. It is important that the solution is well mixed. Stopper and invert the flask several times.



Pipets

Pipets permit the transfer of accurately known volumes from one container to another.

Transfer or volumetric pipets



Measuring pipets

There are two common types of pipets, the **volumetric, or transfer, pipet and the measuring or graduated pipet. Variations of the latter are also called clinical, or serological, pipets.**

Handheld Eppendorf micropipets deliver adjustable microliter volumes of liquid.

digital pipet





A volumetric pipet has one calibration mark and is designed to deliver one fixed volume. Various capacities (i.e. 5,00 mL, 20,00 mL, 50,00 mL etc.) are available and are accurate to two digits after the decimal. The pipet shown above is calibrated to deliver exactly 10 mL at 20°C and should be recorded as 10.00 mL. Measuring pipets include Serological and Mohr pipets. They deliver various volumes to varying degrees of accuracy.

Pipets and burets should be rinsed at least twice with the solution with which they the are to be filled. If they are wet, they should be rinsed first with water, if they have not been already, and then a minimum of *three times with the solution to be used; about* one-fifth the volume of the pipet or buret is adequate for each rinsing.

No solution should be pipetted by mouth!

1. Squeeze the air out of the ***pipet bulb*** and press the opening of the bulb against the opening of the pipet. Do not push the pipet into the bulb. The tip of the pipet must be kept under the surface of the liquid during suction is applied or air will be sucked into the pipet.
2. Rinse the pipet well with at least two portions of the desired solution.
3. Fill the pipet above the calibration mark using a pipet bulb.
4. Quickly remove the bulb and place your index finger (not your thumb) over the end of the pipet.
5. Wipe the pipet tip clean of any excess solution.
6. Drain the solution in the pipet down to the calibration mark by gently reducing the pressure of your index finger. You do not have to remove your index finger. Tip the pipet against the beaker to remove any excess solution. If your finger "leaks" and allows the level in the pipet to drop below the calibration line, moisten your finger slightly and try again.
7. Reset the pipet tip against the wall of the container into which the solution is to be transferred and allow the solution to drain. Volumetric pipets are allowed to drain completely. Measuring pipets should not be allowed to go past the desired volume levels. Leave the pipet in this position for at least 10 seconds after all the solution appears to have drained out and touch the pipet tip to the side of the flask to remove any droplets. Remove the pipet. **DO NOT BLOW OUT THE SOLUTION REMAINING IN THE PIPET.**
8. Rinse the pipet well with DW and store it when finished.

BURETS

A **buret** is used for the accurate delivery of a variable amount of solution.

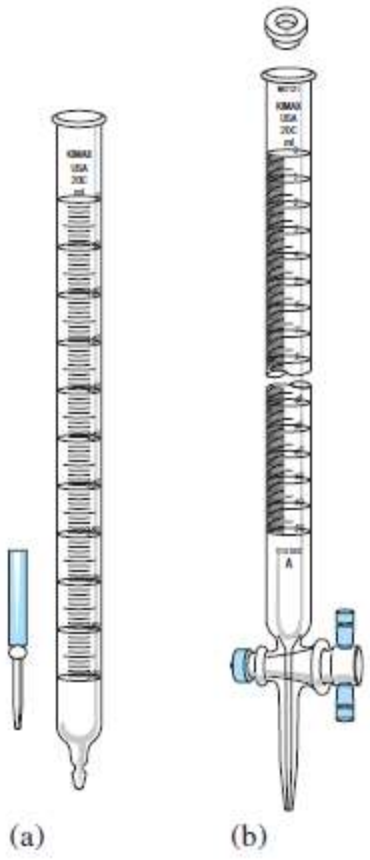
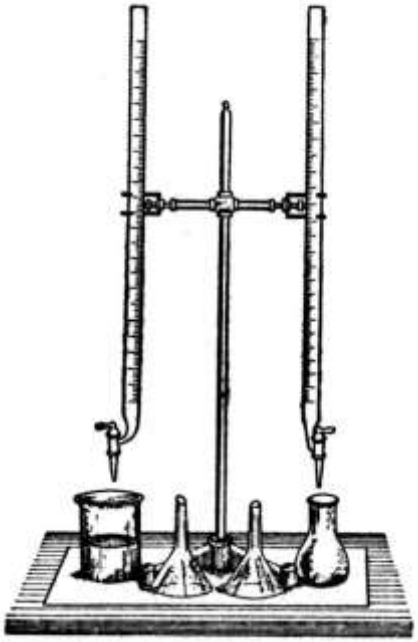
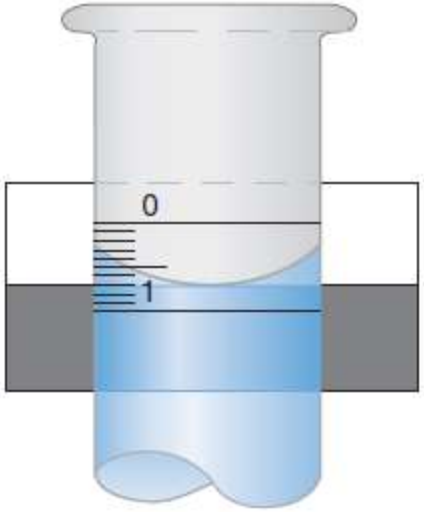
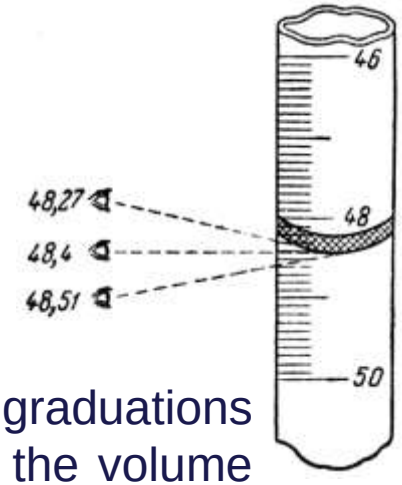


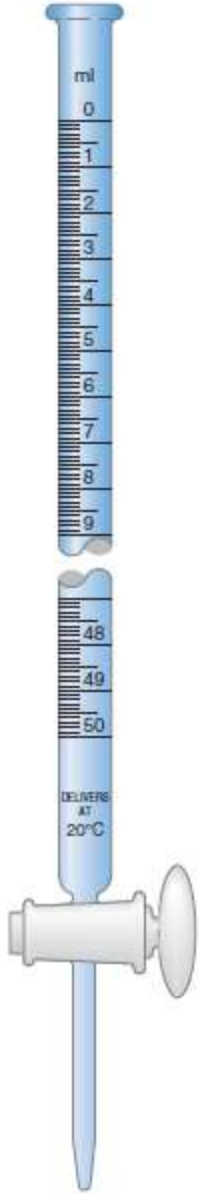
Figure 2-19 Burets: (a) glass-bead valve, (b) Teflon valve.







A buret is a glass tube with graduations that can be used to determine the volume of a solution added to a receiving vessel.
A buret is calibrated to be read from the top down. To determine the volume dispensed from the buret, use the graduation that is closest to the lowest point of the meniscus.



Typical buret.

Reading a buret. (a) The student reads the buret from a position *above a line perpendicular* to the buret and makes a reading (b) of 12.58 mL. (c) The student reads the buret from a position *along a line* perpendicular to the buret and makes a reading (d) of 12.62 mL. (e) The student reads the buret from a position *below a line perpendicular to the buret* and makes a reading (f) of 12.67 mL.

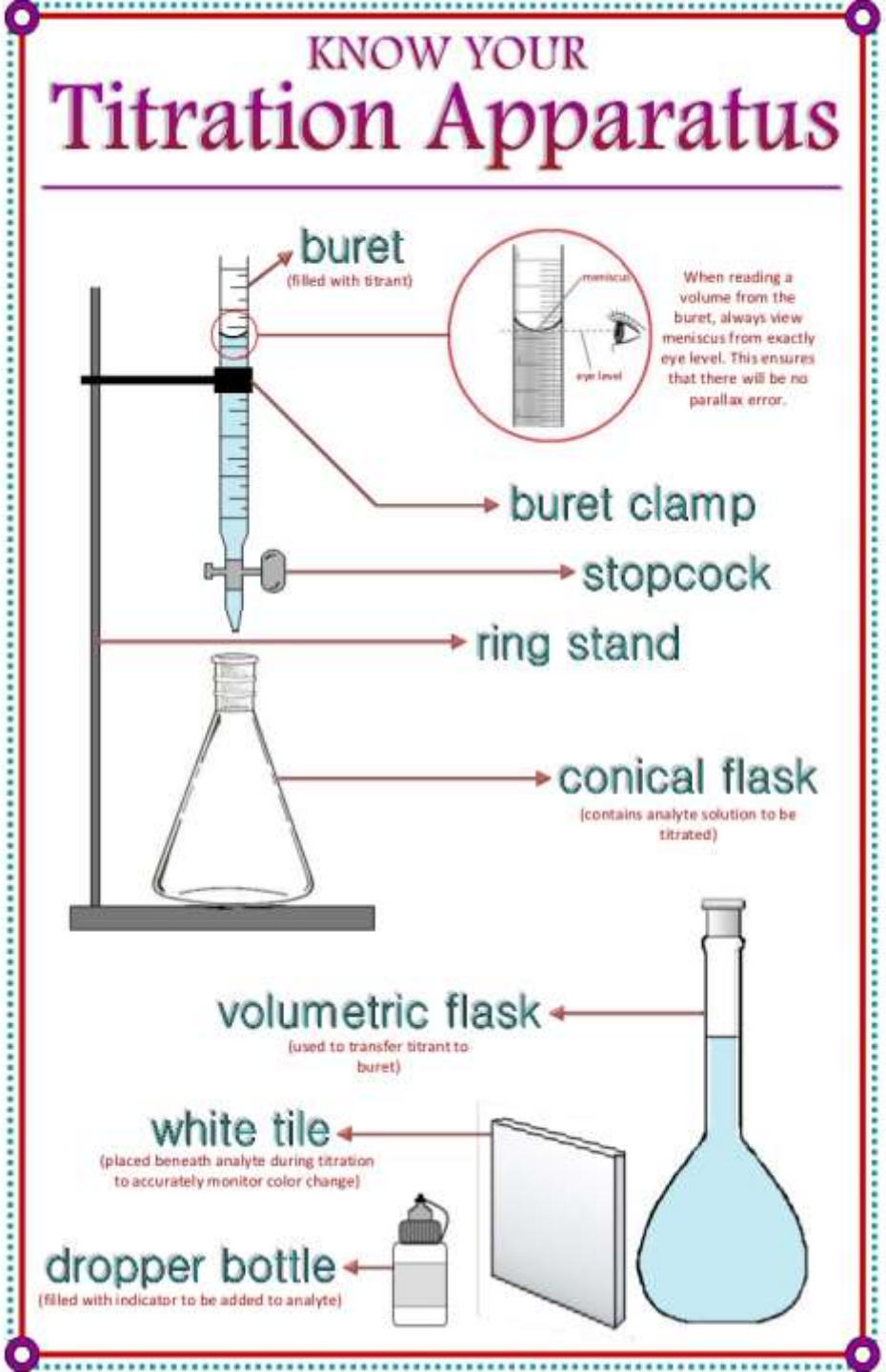
To avoid the problem of parallax, buret readings should be made consistently along a line perpendicular to the buret, as shown.

1. Be sure the buret is clean by rinsing several times with DW. Drain the buret well and rinse at least twice with the desired solution and discard the washings.
2. Fill the buret and drain to or below the 0,00 mL mark.
3. After a suitable time for drainage along the walls, wipe any excess solution from the buret tip and be sure that no air bubbles are trapped in either the tip or on the sides of the buret. If air bubbles are present in the tip, open the stopcock completely to force them out. If there are air bubbles on the sides of the buret, gently tap the buret to get them to rise to the surface. Read the buret to 2 places after the decimal.
4. Deliver the required amount of solution and read the buret again. If you have emptied the buret rapidly, wait for the solution on the walls to drain before making a reading.
5. During titrations the titrant, solution in the buret, should be added slowly to the titrated solution. A flow rate of 1-2 drops per second is recommended. It is essential that the titrant be mixed thoroughly with the solution being titrated throughout the titration. Be sure to stir or swirl the solution into which the titrant is being added to insure complete mixing of the constituents.
6. When approaching the end point of a titration with a color change that is not sharp, note down the volume and color after each addition to avoid overrunning the endpoint. Approach the end point of all titrations with extreme care. After each addition contact the buret tip with the wall of the titration vessel to remove any drop of titrant remaining on the tip and be sure to wash down the sides of the receiving vessel.
7. Always estimate the buret reading to 0,10 mL.
8. When finished, rinse the buret well with DW.

Erlenmeyer flask

beaker

graduated cylinder



Typical setup for carryingout a titration. The apparatus consists of a buret, a buret stand and clamp with a white porcelain base to provide an appropriate background for viewing indicator changes, and a wide-mouth Erlenmeyer flask containing a precisely known volume of the solution to be titrated. The solution is normally delivered into the flask using a pipet. Before the titration begins. The solution to be titrated, an acid in this example, is placed in the flask, and the indicator is added.

During titration. The titrant is added to the flask with swirling until the color of the indicator persists. In the initial region of the titration, titrant may be added rather rapidly, but as the end point is approached, increasingly smaller portions are added; at the end, point, less than half a drop of titrant should cause the indicator to change color.

Lab Glassware Cleaning Procedures

Cleaning Procedures:

1. Remove all labels using sponge or acetone.
2. Wash with hot tap water and a brush to scrub inside of glassware, stopcocks, and other small pieces, if possible, using a suitable laboratory-grade detergent.
3. Rinse thoroughly with hot tap water.
4. Rinse thoroughly with deionized water.
5. Rinse thoroughly with pesticide grade Acetone.
6. Rinse thoroughly with pesticide grade Methanol.
7. Rinse thoroughly with pesticide grade Hexane.
8. Rinse or soak with 1:1 HCl (Hydrochloric Acid).
9. Rinse or soak with >10% HNO_3 (Nitrate Acid).
10. Bake at 105°C for 1 hour.
11. Bake at 180°C (prior to use as per method).
12. Drain, then heat in muffle furnace for 30-60 minutes at 400°C.
13. Clean, dry glassware should be sealed and stored in dust-free environment.
14. Soak in oxidizing agent (Chromic acid or equivalent); preferably hot (40-50°C).
15. Last step (prior to use) should be a rinse with the solvent used in analysis.
16. Drain, then heat in muffle furnace for 1 hour at 550°C.
17. Heat 1 hour in EDTA solution at 90-100°C.
18. New glassware must be soaked overnight in 10% HNO_3 or HCl.
19. New glassware must be soaked overnight in seawater.
20. Rinse thoroughly with DW.

1. ORGANIC, Semi-Volatile: (Pesticides, Herbicides, Oil & Grease) Solvents: 5, 1-4, 5 or 6, 13, 15 or Muffle: 5, 1-4, 12, 13, 15
Or Oxidizer: 5, 1-3, 14,
2. INORGANICS, Trace Metals: 1-4, 9, 8 (optional), 4. Nutrients, Minerals: 1-4, 8, 4. Solids: 1-4, 11. Non-Metals, Physical Properties: 1-4, 14
3. MICROBIOLOGY: 1-4, (Sterilize per approved method)
4. BIOASSAY: Freshwater: 18, 2, 3, 9 or 8, 4, 5, 4, 20. Marine & Estuarine: 19, 2, 3, 9 or 8, 4, 5, 4, 20.
5. RADIONUCLIDES : 17, 3, 8, 4

Titration

Recommended method for manipulating a buret stopcock.

Proper technique for titration

1. It is needed to choose volume of an unknown solution and put it in an Erlenmeyer flask.
2. Fill a buret with a standard reagent of known concentration and read the initial volume of the solution.
3. Add a couple of drops of an indicator in the flask for titration. An indicator is a soluble dye that changes its color noticeably over a fairly short range of pH. Different indicators show color changes at different pH values and it is important to determine an indicator to be used according to the expected equivalence point.
4. Slightly open the cork of the buret and add the standard reagent into the unknown solution. Around the expected equivalence point of the titration, you need to drop the solution very slowly and mix the solutions very well because, around the equivalence point, just one drop of solution from the buret can make a radical pH change in the mixed solution.
5. If the color of the solution in the Erlenmeyer flask changes, record the volume of the solution in the buret and add a few drops of the solution to make sure the equivalence point you found is correct.

Volumetric Calculations

Titr (T) is a special unit for measuring of concentration connected with chemical quantitative analysis. Titr is the number of grams of solute in 1 milliliter of solution. For example, the titr of a solution made from 0,3 g of Silver Nitrate ($AgNO_3$) dissolved in 150 mL of solution is found as follows:

$$T = \frac{\text{Mass of solute } m_S \text{ in grams}}{\text{Volume of solution in mililiters}} =$$

$$= \frac{0{,}3 \text{ g } AgNO_3}{1150 \text{ mL of soution}} = 0{,}002 \text{ g/mL}$$

$$\text{amount A (mol)} = \frac{\text{mass A (g)}}{\text{molar mass A (g/mol)}}$$

$$\text{amount A (mol)} = V\text{(L)} \times c_A \left(\frac{\text{mol A}}{\text{L}} \right)$$

Titer established the relationship between volume of titrant and amount of analysed substance present. The most commonly titer is in units of mg analysed substance per ml of titrant. This system was developed to assist in doing routine calculations.

$$\text{weight percent (w/w)} = \frac{\text{weight solute}}{\text{weight solution}} \times 100\%$$

Titre of the titrant by the substance to be determined

The mass (g) of the substance to be determined, which is equivalent to 1 ml of the titrant with the theoretical molar concentration of the equivalent substance (dimension unit is g/ml)

To calculate the titre of the titrant solution $KMnO_4$ by Fe^{2+}-ions the following formula is used:

$$T = \frac{c \cdot 5 \cdot M(Fe^{2+})}{1000}$$

Describe the preparation of 2.000 L of 0.0500 M $AgNO_3$ (169.87 g/mol) from the primary-standard-grade solid.

Solution

$$\text{amount } AgNO_3 = V_{soln}(L) \times c_{AgNO_3}(mol/L)$$

$$= 2.00 \cancel{L} \times \frac{0.0500 \text{ mol } AgNO_3}{\cancel{L}} = 0.100 \text{ mol } AgNO_3$$

To obtain the mass of $AgNO_3$, we rearrange Equation 13-2 to give

$$\text{mass } AgNO_3 = 0.1000 \cancel{\text{ mol } AgNO_3} \times \frac{169.87 \text{ g } AgNO_3}{\cancel{\text{mol } AgNO_3}}$$

$$= 16.987 \text{ g } AgNO_3$$

Therefore, the solution should be prepared by dissolving 16.987 g of $AgNO_3$ in water and diluting to the mark in a 2.000 L volumetric flask.

Several basic ***requirements*** can be reported for a well-defined titrimetry:

1. A quick and quantitative reaction A + B is necessary.
2. A standard titrant solution of accurately known concentration of A to react with the analyte with a well-known and repeatable stoichiometric procedure.
3. An indicator (external or internal) to identify the endpoint. The random and systematic error resulting from the empirical estimation of the endpoint may be estimated by conducting a blank titration.
4. When the chemical for the titrant solution is not available in a kinetically stable form of pure and well-defined composition, the titrant must be standardized. This operation is an independent titration against stable, pure chemical known as a primary standard.
5. Accurate measurements of the sample and added titrant volumes.
6. It should be interesting if possible to have an A + B reaction sufficiently selective to avoid a previous sample treatment to remove interferences.