

- 1) Indeterminate errors are known as _____ errors.
- 2) Titrations are repeated till we get _____ values.

ANSWERS

- 1) systematic
- 2) accidental
- 3) concordant

III. State the following are True or False

- 1) The weights used in analytical balances need no further calibration.
- 2) A volume reading between 30.5 cc and 30.7 cc should be written as 30.60 cc
- 3) The median is a value about which all others are equally distributed.
- 4) The number of significant figures in 0.00042 is 6.

ANSWERS

- 1) False
- 2) False
- 3) True
- 4) False

Using the following data calculate the .

- a) mean
 - b) median
 - c) average deviation
 - d) standard deviation. Tabulate the results.
- (i) 30.26 ; 30.24 ; 30.30 ; 30.18 and 30.20 ml
(ii) 9.98 ; 9.99 ; 9.98 ; 9.95 ; 10.00 and 10.02 ml
(iii) 46.92 ; 46.47 ; 46.76 ; 46.64 and 46.53

ANSWERS

Problem No. →	(i)	(ii)	(iii)
i) Mean	30.236	9.987	46.664
ii) Median	30.24	9.985	46.64
iii) Average deviation	0.001824	0.0004556	0.02618
iv) Standard deviation	0.04830	0.0234	0.1809

GRAVIMETRIC ANALYSIS

Gravimetric analysis is a technique in analytical chemistry by which the amount of the substance present in a given sample is estimated by determining the weight of the precipitate obtained from the sample.

Explanation with example

The amount of barium present in a given BaCl₂ solution is determined by treating this solution with enough dilute sulphuric acid so as to precipitate BaSO₄ completely.



The precipitate formed is filtered off, dried and weighed. From the weight of BaSO₄ precipitate, the amount of barium present in the whole of the given solution is estimated.

Gravimetric Factor

Gravimetric factor (G.F.) is calculated using the formula,

$$\text{G.F.} = \frac{a}{b} \times \frac{\text{Formula weight of substance to be estimated}}{\text{Formula weight of the substance weighed}}$$

Here, a and b are integers which make the number of formula weights in the numerator and denominator chemically equivalent.

Explanation with examples

(i) Let us consider the estimation of barium ions. It is converted into BaSO₄ using dilute sulphuric acid.

The relevant stoichiometric equation is _____



Note

- (i) In the estimation of iron by precipitating it as Fe_2O_3 , the G.F. is given as,

$$\text{G.F.} = \frac{2 \times \text{Fe}}{1 \times \text{Fe}_2\text{O}_3} = \frac{2 \times 55.85}{1 \times 159.70} = 0.6994$$

Here, $a = 2$ and $b = 1$

- (ii) In the estimation of magnesium by precipitating it as $\text{Mg}_2\text{P}_2\text{O}_7$, the G.F. is given as

$$\text{G.F.} = \frac{2 \times \text{Mg}}{1 \times \text{Mg}_2\text{P}_2\text{O}_7} = \frac{2 \times 24.30}{1 \times 222.60} = 0.2184$$

Here, $a = 2$ and $b = 1$

From these, it is clear that the values of a and b depend on the experiment and is not always $a = b = 1$.

Characteristics of Precipitating Agent

Definition: A reagent added to a solution of a substance to be estimated so as to precipitate that substance completely is called a precipitating agent or precipitant.

For example, in the estimation of chloride in a solution of potassium chloride, we use silver nitrate solution. Thus, AgNO_3 solution is the precipitating agent.

An ideal precipitating agent should react specifically with the ion or substance to be estimated gravimetrically and give a precipitate which ———

- (i) has sufficiently low solubility so that losses due to solubility of the solid is negligible.
- (ii) is readily filtered and washed free of impurities.
- (iii) is unreactive and of known composition after drying or ignition (if required)

Choice of precipitants

Precipitating agents may be inorganic or organic. Details of some inorganic precipitating agents are given in Table 2.2

TABLE 2.2
Inorganic Precipitating Agents

S.No.	Precipitant	Substance to be estimated	Precipitate
1.	BaCl_2	SO_4^{2-}	BaSO_4
2.	NH_4SCN	Cu	$\text{Cu}_2(\text{SCN})_2$
3.	$(\text{NH}_4)_2\text{HPO}_4$	Mg	$\text{Mg}_2\text{P}_2\text{O}_7$
4.	NH_3	Fe	Fe_2O_3
5.	H_2S	Zn	ZnO or ZnSO_4
6.	$\text{H}_2\text{C}_2\text{O}_4$	Ca	CaO
7.	H_2SO_4	Ba	BaSO_4
8.	H_2SO_4	Pb	PbSO_4
9.	NaCl	Ag	AgCl
10.	AgNO_3	Cl^-	AgCl

Details of few organic precipitants are given in Table 2.3

TABLE 2.3
Organic Precipitants

S.No.	Precipitant	Metal ions precipitated
1.	Anthranilic acid	Cd(II), Co(II), Cu(II)
2.	α - Benzoin oxime (cupron)	Cu(II)
3.	α - Benzyl dioxime	Ni(II)
4.	Cupferron	Cu(II), Al(III), Fe(III)
5.	Diantipryl methane	Cd(II), Co(II), Fe(III)
6.	Dimethylglyoxime	Ni(II), Pd(II)
7.	Dipicrylamine	K(I), Rb(I), Cs(I)
8.	Neonicon	Ni(II), Pd(II)
9.	Oxine	Al(III), Mg(II), Zn(II)
10.	Captax	Ag(I), Au(III), Cu(II)
11.	β - Naphthoquinoline	Cd(II)

12.	α - Nitroso - β - naphthol	Co(II), Cu(II), Fe(II)
13.	Picrolonic acid	Pb(II), Ca(II), Mg(II)
14.	Pyrogallol	Bi(III), Sb(III)
15.	Quinaldic acid	Cu(II), Pb(II), Ag(I)
16.	Salicylaldehyde	Cu(II), Pb(II), Pd(II)
17.	Tannin	Be(II), Al(III), Ga(III)
18.	Thioacetamide	Cu(II), Cd(II), Pb(II)
19.	Thiozalide	Cu(II), Pd(II), Hg(II)
20.	Zirconon	Zr(IV)

The choice of precipitants for the estimation of metal ions is limited as only one or two precipitants are there which satisfy all the requirements of a good precipitant.

For example, in the estimation of barium, we have H_2SO_4 or K_2CrO_4 only as precipitant.

However, we can choose both an inorganic precipitant and an organic precipitant. For example, in the estimation of aluminium, we can use NH_4OH or oxine as precipitant.

When we use inorganic precipitants for estimation, co-precipitation and/or post precipitation may occur.

Organic precipitants have both advantages and disadvantages which are given below ———

Advantages

- 1) The precipitates formed by organic precipitants are insoluble in water. Hence, complete quantitative precipitation is possible.
- 2) The formula weight of the precipitate obtained in the analysis is large in comparison with the formula weight of the metal ion to be estimated. This decreases error in weighing.
- 3) Organic precipitants give precipitates with large particle size. This enables filtration and washing efficient.
- 4) Drying of precipitate is easy as organic compounds have least tendency to retain water in them. i.e., they are hydrophobic.

Disadvantages

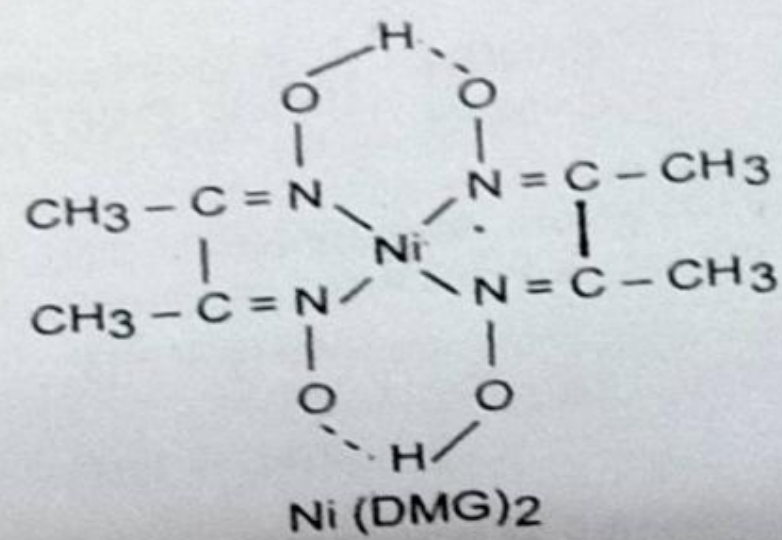
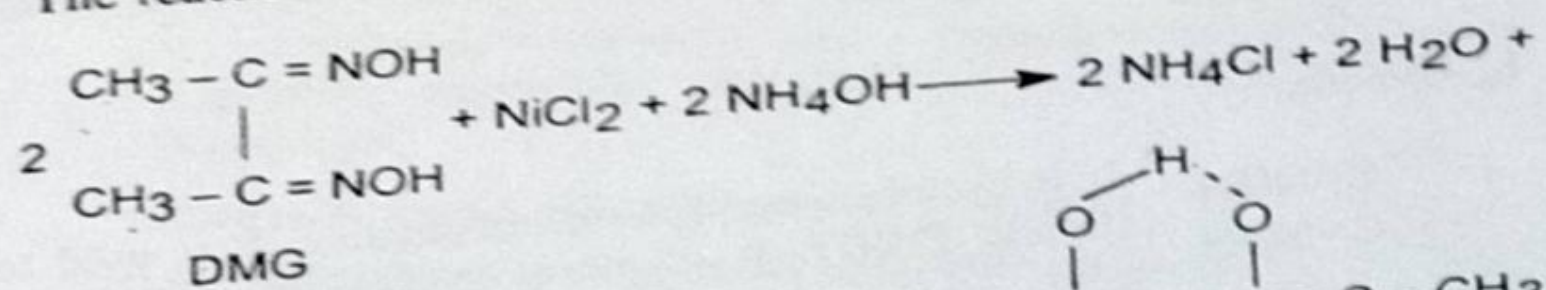
- 1) As organic substances are decomposed or volatilise on heating, drying makes the composition of the precipitate uncertain.
- 2) As the solubilities of the organic precipitates in water is less, the preparation of the solution of the precipitants is difficult. Ex : DMG is dissolved in alcohol to make the solution.
- 3) The cost of organic precipitants is high as compared to the inorganic precipitants.

Specific Precipitants

Definition : Specific precipitants precipitate quantitatively a particular ion or a substance from a solution.

There are no specific precipitants in reality. The organic precipitants given in Table 2.3 are nearly specific. We shall discuss few of them.

1) **Dimethyl glyoxime (DMG) :** It is a specific precipitant for estimating nickel in alkaline solution and palladium in acidic solution. The reaction of DMG with Ni(II) is given as follows :



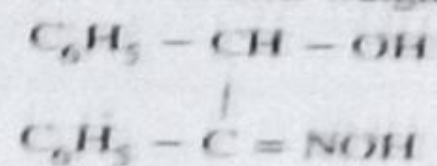
$Ni(DMG)_2$ is a bright red precipitate.

2) **8 - hydroxyquinoline (oxine) :** The reaction between Al^{3+} with oxine can be written in the pH around 3.

...takes place as slowly as possible.

About 24 metal ions give precipitates with oxime. The solubilities of metal oximates vary from cation to cation and are pH dependent. Hence, by controlling pH, we can achieve a considerable degree of selectivity.

3) **Benzoin - α - oxime (cupron)** : It is used to estimate copper in dilute ammoniacal solution in presence of tartrate. We obtain a green precipitate. It is dried at 110°C and weighed.

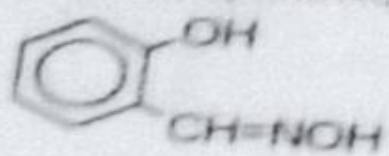


4) **Cupferron**



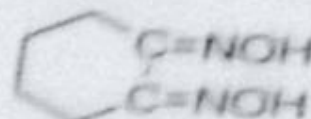
It is nothing but the ammonium salt of N - nitroso N - phenylhydroxylamine. It is employed to estimate Cu(II) and Fe(III) in cold, aqueous acid solution.

5) **Salicylaldehyde oxime**



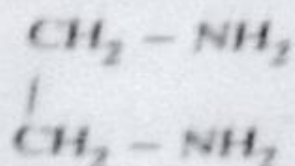
It is used to estimate Cu(II) in presence of acetic acid at pH 2.6

6) **Nioxime**



It is cyclohexane -1, 2- dioxime. It is used to estimate Pd(II). It is more soluble than DMG in water.

7) **Ethylene diamine (en)** : The IUPAC name is 1, 2 - diamino ethane. It is used to estimate Cu(II), Hg(II) and Cd(II)



Selective precipitants

Definition : The precipitants which precipitate a small group of ions from solutions containing several ions are known as selective precipitants.

Most of the gravimetric precipitants found in Table 2.2 and 2.3 are only selective.

Explanation with example : Hydroxide precipitates a few metal ions as their hydroxides from a solution containing several metal ions. The metal ions which are not precipitated also form hydroxide, but they are soluble. Hence, they are not precipitated.

Other examples of selective precipitants are - sulphide and carbonate.

It may be noted that selectivity of precipitants may be increased, when controlled conditions are adopted.

Conditions of precipitation

The following four conditions are to be adhered for successful gravimetric estimations -

1) **The solubility of the precipitate must be low.**

The ion or substance to be estimated gravimetrically should be precipitated quantitatively so that the filtrate does not contain that substance which is precipitated.

2) **The precipitate must be in the form of larger particle size so that it is readily filtered.**

3) **The precipitate must be pure.**

The precipitate should not have impurities. Even if it contains some impurities, they must be removable by washing and drying.

4) **The precipitate must have a constant composition.**

It should not change its composition during filtration, drying or ignition (if required)

Techniques of precipitation

The following techniques are employed to fulfill the above four conditions of precipitation.

1) The solution of the **analyte**, substance to be estimated, is diluted. This will enable to precipitate the analyte quantitatively.

2) The precipitant added must be chosen in such a way that it forms large size particles as precipitate.

- 3) Precipitation must be carried out in hot condition. In hot condition, the solubility increases, thereby reducing super saturation.
- 4) Precipitation is made fast. Colloid formation is avoided. We obtain well formed precipitate.
- 5) The precipitant must be added slowly and in small lots with constant stirring.
This will lead to quantitative precipitation of the **analyte**.
- 6) Slight excess of the precipitant is added to ensure complete and quantitative precipitation.
- 7) Precipitation is carried out from homogeneous solutions which will minimise supersaturation and help the growth of the size of the precipitate.
- 8) The precipitate is to be digested for sufficiently long time to give precipitate of big particle size so that it is not lost during filtration.
- 9) The precipitate should be washed with suitable wash liquid containing suitable electrolyte. This will avoid **peptisation** i.e., dissolution of some precipitate.
- 10) When the precipitate is suspected to contain impurities because of co-precipitation etc., it should be dissolved in a suitable solvent and reprecipitated again.

Types of Precipitates

We come across two types of precipitates in gravimetric analyses. They are (i) Colloidal precipitates (ii) Crystalline precipitates.

Colloidal precipitates : Individual colloidal particles are so small that they are not retained on ordinary filtering media. Brownian movement prevents their settling from the solution under the influence of gravity. But the individual particles of most colloids can be coagulated to give a filterable, non crystalline mass that rapidly settles down from solution. Heating, stirring and adding an electrolyte to the medium hasten the coagulation process.

Crystalline precipitates : These are more easily manipulated than coagulated colloids. The size of the individual crystalline particles can

CO-PRECIPIATION

Definition : It is a process by which normally soluble components of a solution are carried down during the formation of a precipitate.

Explanation : In the determination of barium ions by precipitation as BaSO_4 , other barium salts such as $\text{Ba}(\text{NO}_3)_2$ and BaCl_2 **occlude** on BaSO_4 precipitate. We know that $\text{Ba}(\text{NO}_3)_2$ and BaCl_2 are soluble. Still, during the precipitation of BaSO_4 , these soluble compounds occlude on BaSO_4 particles and are carried down along with BaSO_4 precipitate. Such a process is known as co-precipitation.

Mechanism of Co-precipitation

Co - precipitation occurs either by –

- (i) surface adsorption (ii) inclusion or occlusion

Gelatinous precipitates co-precipitate by adsorption while crystalline precipitates co - precipitate by inclusion or occlusion. Adsorption is a surface phenomenon. Inclusion involves the random distribution of the contaminant through out the interior of the solid.

Occlusion involves a non - homogeneous distribution of the impurities within the imperfections in the crystal lattice of the precipitate.

Effects of Co-precipitation

Due to co-precipitation, positive or negative error may result depending on whether the co-precipitated substance has more or less molecular weight than that of the precipitate.

For example, during the precipitation of BaSO_4 , if $\text{Ba}(\text{NO}_3)_2$ is co-precipitated a positive error is observed as the molecular weight of $\text{Ba}(\text{NO}_3)_2$ (261.35) is more than that of BaSO_4 (233.34)

On the other hand, if BaCl_2 is co-precipitated, a negative error arises as the molecular weight of BaCl_2 (208.25) is less than that of BaSO_4 (233.34)

Methods adopted to minimise co - precipitation

The following methods are adopted to minimise co-precipitation —

- 1) The solution from which precipitation is carried out, should be diluted.
- 2) The precipitant is to be added in small quantities with constant stirring so that the precipitation takes place as slowly as possible.

- 3) Precipitation is carried out from hot solutions only so that ———
- the solubility of the impurity would be increased
 - formation of colloidal particles would be decreased
 - the attraction between the precipitate and impurity would be decreased.
- 4) The precipitate is digested well.
This would decrease co - precipitation due to surface adsorption.
- 5) The precipitate is dried at high temperatures so that the occluded impurity could escape.
- 6) Solutions employed in precipitation are well diluted so that when the mother liquor adheres to the precipitate, there is not much solute in it. This will decrease occlusion.
- 7) Crystal growth must be slow so that occlusion is less.

POST PRECIPITATION

Definition : It is a process in which an impurity is precipitated from its supersaturated solution some time after the appearance of the substance to be estimated as precipitate.

Explanation

- When calcium is determined gravimetrically as calcium oxalate, any magnesium present in the solution will be precipitated slowly on calcium oxalate.
- When Cu(II) or Hg(II) is determined as its sulphide, Zn(II) present in the solution will be precipitated slowly on CuS or HgS

Post precipitation becomes pronounced when the precipitate is allowed to be in contact with the mother liquor.

Theory of post precipitation : It occurs with sparingly soluble substances which form supersaturated solutions. These substances usually have an ion in common with the analyte. For example, post precipitation of magnesium oxalate while estimating calcium by precipitating it as calcium oxalate.

The effect of post precipitation is that it will lead to positive error.

Methods adopted to minimise post precipitation

- The precipitate is washed with suitable solution so as to remove the post precipitated impurity.
- The precipitate must be filtered off as soon as the precipitation is over.

Differences between co-precipitation and post precipitation.

S.No.	Co-precipitation	Post precipitation
1.	It decreases with time when the precipitate is allowed to be in contact with its mother liquor.	It increases.
2.	It decreases when the solution is stirred or heated.	It increases.
3.	The amount of the substance co-precipitated is far less.	It is more.

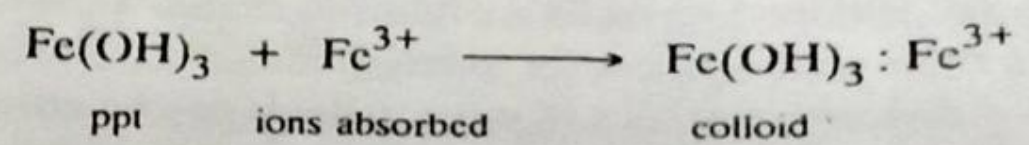
Peptisation

This is the process of passing of a precipitate into colloidal solution on adding an electrolyte.

It involves the adsorption of suitable ions (supplied by the electrolyte added) by the fine particles of the precipitate whereby their boundary potentials are raised and they form a colloidal solution.

The electrolyte added is termed a **dispersing agent or peptising agent**. Few examples of colloids obtained by peptisation are :

- A reddish brown, positively charged sol is obtained by adding small quantity of ferric-chloride solution (which furnishes Fe^{3+} ions) to a freshly precipitated and well washed $Fe(OH)_3$.

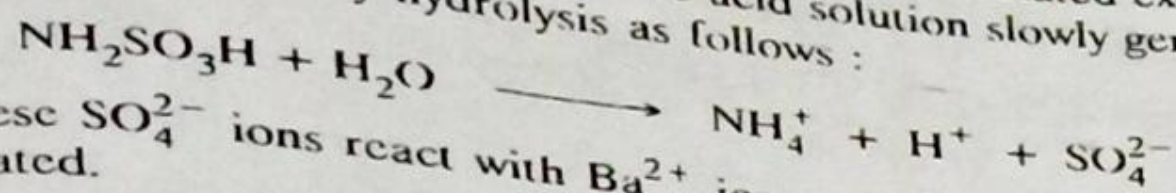


- A precipitate of Prussian blue passes into colloidal solution on adding oxalic acid solution due to peptisation.

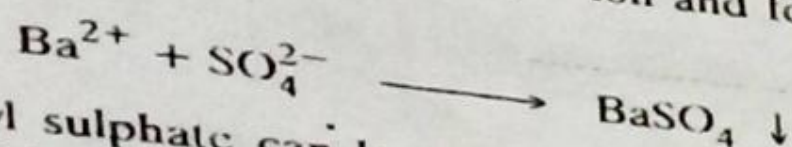
Precipitation from homogeneous solution

Definition : This is a process in which the precipitant is slowly and chemically generated in the reacting solution itself.

Explanation with example : Barium ion is precipitated as $BaSO_4$ by mixing $Ba(II)$ solution and a solution containing a calculated excess of sulphamic acid (NH_2SO_3H). Sulphamic acid solution slowly generates SO_4^{2-} ions in solution by hydrolysis as follows :



These SO_4^{2-} ions react with Ba^{2+} ion and form $BaSO_4$ which is precipitated.



Dimethyl sulphate can be used to precipitate $Ba(II)$ instead of sulphamic acid.

The following table 2.4 gives the details of precipitation from homogeneous solution.

TABLE 2.4

S.No.	Precipitant	Reagent which generates the precipitant	Metals precipitated
1.	OH^-	Urea	Al, Fe, Sn
2.	SO_4^{2-}	Dimethyl sulphate	Ba, Pb
3.	SO_4^{2-}	Sulphamic acid	Ba, Pb
4.	PO_4^{3-}	Trimethyl phosphate	Zr, Hf
5.	$C_2O_4^{2-}$	Ethyl oxalate	Ba, Ra
6.	S^{2-}	Thioacetamide	Cu, Cd, Mo

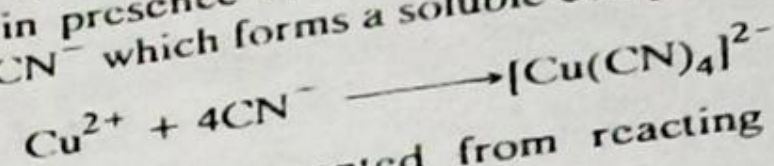
Advantages

- 1) Precipitation from homogeneous media is carried out to avoid local reagent excess, as the precipitant appears slowly and homogeneously throughout the entire solution.
- 2) The relative supersaturation is kept low.
- 3) This method results in the marked increase in particle size of the precipitate and its purity.

Sequestering agents

Definition : The reagents added to eliminate the interference by a substance in an estimation are called sequestering agents. These are also called masking agents.

Explanation with example : In the gravimetric estimation of $Mg(II)$ using oxine in presence of $Cu(II)$, we have to eliminate $Cu(II)$. For this, we use CN^- which forms a soluble complex with $Cu(II)$ as follows.



Thus, $Cu(II)$ is prevented from reacting with oxine. In this example, CN^- is the sequestering agent.

Other examples

- 1) $Ca(II)$ can be estimated with suitable reagent in presence of $Cu(II)$ using cyanide ion as sequestering agent.
- 2) In the presence of EDTA, beryllium may be precipitated with ammonia in presence of Cr, Co, Cd, Fe, Cu, Pb, Mn, Zn, Al, Bi etc.
- 3) Uranium can be separated from numerous other ions by precipitation with oxine from an EDTA solution at pH 5.3.
- 4) Copper has been masked with EDTA in the polarographic determination of antimony in alloys.
- 5) Precipitation of soaps as their calcium and magnesium salts by hard water can be checked by transforming these metals into soluble complexes of EDTA.

DETAILS OF SOME GRAVIMETRIC EXPERIMENTS**I. Estimation of barium as barium sulphate**

The given solution is made upto 100 ml using distilled water in a 100 ml standard flask. 20 ml of the made up solution is pipetted out into a 400 ml beaker. 2 ml of 2N HCl is added. The solution is diluted to 100 ml with distilled water. The solution is heated to boiling in a water bath. To the boiling solution 10 ml of 2N H_2SO_4 is added slowly in drops with stirring. The precipitate obtained is digested for about 30 min in a water bath. The precipitate is filtered off using Whatman filter paper.

The precipitate is washed with hot water. The filtrate is tested for the absence of Cl^- and SO_4^{2-} . The precipitate along with the filter paper and the funnel is dried over an aluminium cone on a sand bath. The dried filter paper along with the precipitate is taken in a weighed silica crucible. It is ignited.

When the entire filter paper has been burnt out, the precipitate is heated for about 30 min. The crucible along with the precipitate is

Types of Wash-Solutions

Wash solutions are of three types:

1. **Solutions which prevent the precipitate from becoming colloidal and passing through the filter.** This tendency is observed with gelatinous or flocculated precipitates. A solution of an electrolyte like an ammonium salt is used for washing such precipitates. If the wash solution has an ion common to an ion in the precipitate, the solubility of the precipitate would be less in the wash solution. For example, dilute ammonium oxalate solution is used for washing calcium oxalate. Sometimes, organic solvents like ethanol and ether may be used for these.
2. **Solutions which reduce the solubility of the precipitates.** If the wash solution has an ion common to an ion in the precipitate, the solubility of the precipitate would be less in the wash solution. For example, dilute ammonium oxalate solution is used for washing calcium oxalate. Sometimes, organic solvents like ethanol and ether may be used for these.
3. **Solutions which prevent the hydrolysis of salts of weak acids and bases.** A tendency to hydrolyse is shown by a precipitate which is a salt of a weak acid. The product of such hydrolysis will be a base. The wash liquid must therefore be basic. For example, $MgNH_4PO_4$ hydrolyses to give acid phosphate ion HPO_4^{2-} and hydroxide ion and therefore it should be washed with dilute aqueous ammonia.

Solubility losses are minimized by using the least quantity of wash solution. Washing should be carried out efficiently by using many small portions of liquid than a few large portions. Qualitative tests must be made upon portions of the filtrate for some foreign ion which is present in the original solution and washing stopped at the appropriate stage. For example, in the estimation of silver chloride, the washing of the precipitate is continued with dilute nitric acid until the filtrate is free from chloride ion.

Ideal Characteristics of Wash Liquids

1. It should have no solvent action upon the precipitate, but dissolve foreign (impurity) substances easily.
2. It should have no dispersive action on the precipitate.
3. It should form no volatile or insoluble product with the precipitate.
4. It should be easily volatilized at the temperature of drying of the precipitate.
5. It should not contain any substance which is likely to interfere with subsequent determination of constituents in the filtrate.

Drying of Precipitate

The temperature at which a precipitate is to be dried can be ascertained from the knowledge of the thermogravimetric curve for that substance. The drying temperature range used should be

sufficiently high to volatilize water and other volatile impurities but must be well below precipitate decomposition temperature. The precipitate is brought to a specified temperature and maintained at that temperature for a definite time to ensure a constant weight precipitate.

Types, Care and Use of Crucibles

Different types of crucibles are used for collecting and weighing the precipitates, mainly of two types.

1. Crucibles made of silica or porcelain are used when the precipitates are ignited in air.
2. Sintered crucibles are used for precipitates which are weighed after drying in air.

Crucibles are expensive. They should be handled properly for obtaining reliable data. They should never be touched but must always be handled with a pair of tongs. They should not be placed on the table but only on a clean tile.

Porcelain Crucibles. These are usually of 3 to 4 cm in diameter. These should be heated with a small flame in order to avoid fracture before being heated to a high temperature. These are suited for heating the precipitates to very high temperatures. Porcelain crucibles are suitable for use up to temperatures 1000-1200°C. Hydrofluoric acid, sodium hydroxide, peroxide, and alkali carbonates attack porcelain and therefore substances containing these materials should not be handled in such crucibles. These crucibles can be cleaned with concentrated acids.

Silica Crucibles. These are more expensive than porcelain crucibles. However, they are safely exposed to sudden changes of temperature without any risk of fracture. Silica crucibles must not be used for alkalis or hydrofluoric acid because these chemicals attack silica. Precipitates tend to adhere strongly to these crucibles and hence these crucibles should be carefully cleaned immediately after use.

Platinum Crucibles. Platinum crucibles are marketed in various shapes and sizes. The choice of the crucible for an experiment is decided by the quantity of precipitate handled. Platinum crucibles are very expensive and hence their use is very much restricted to cases where they are necessary. These crucibles can be more readily and more uniformly heated to redness than other crucibles. The following rules and precautions must be observed with regard to these crucibles.

1. These must not be exposed to a luminous flame since this will cause the loss of lustre and become brittle owing to the formation of platinum carbide.
2. Compounds of lead, silver, zinc, tin, bismuth, arsenic and antimony must not be heated in these when there is any possibility of reduction to the metallic state. These compounds, having low melting points, may alloy with platinum. Phosphates should not be heated in a platinum crucible. If phosphorus is formed during ignition it will attack platinum rendering it brittle.
3. Hydroxides, nitrates, nitrites and alkali cyanides should not be heated in platinum crucibles.
4. Evaporation or fusion involving the formation of chlorine, bromine or iodine should not be performed in platinum crucibles because these elements attack platinum.
5. The crucible should not be squeezed or scratched. A bright surface should be obtained by burnishing it with sand.

6. Adhering substances or stains can be removed by heating a little concentrated sulphuric acid in the crucible. Then it should be rinsed with distilled water and dried.

The Gooch Crucible. A Gooch crucible (Fig. 9.1(a)) is made of porcelain with the bottom part of a narrow rubber ring, the adaptor passing through a glass adaptor of such diameter that the bottom of the crucible is covered with asbestos. This covering is done by shaking asbestos fibre bits with water and pouring the mixture into the crucible. The water is then allowed to evaporate as a thin cover at the bottom. A Gooch crucible is used usually for drying precipitates only up to about 250°C, because above this temperature the asbestos in it loses its weight. This crucible is preferable to filter paper for filtering solutions, such as potassium permanganate, which attack paper.

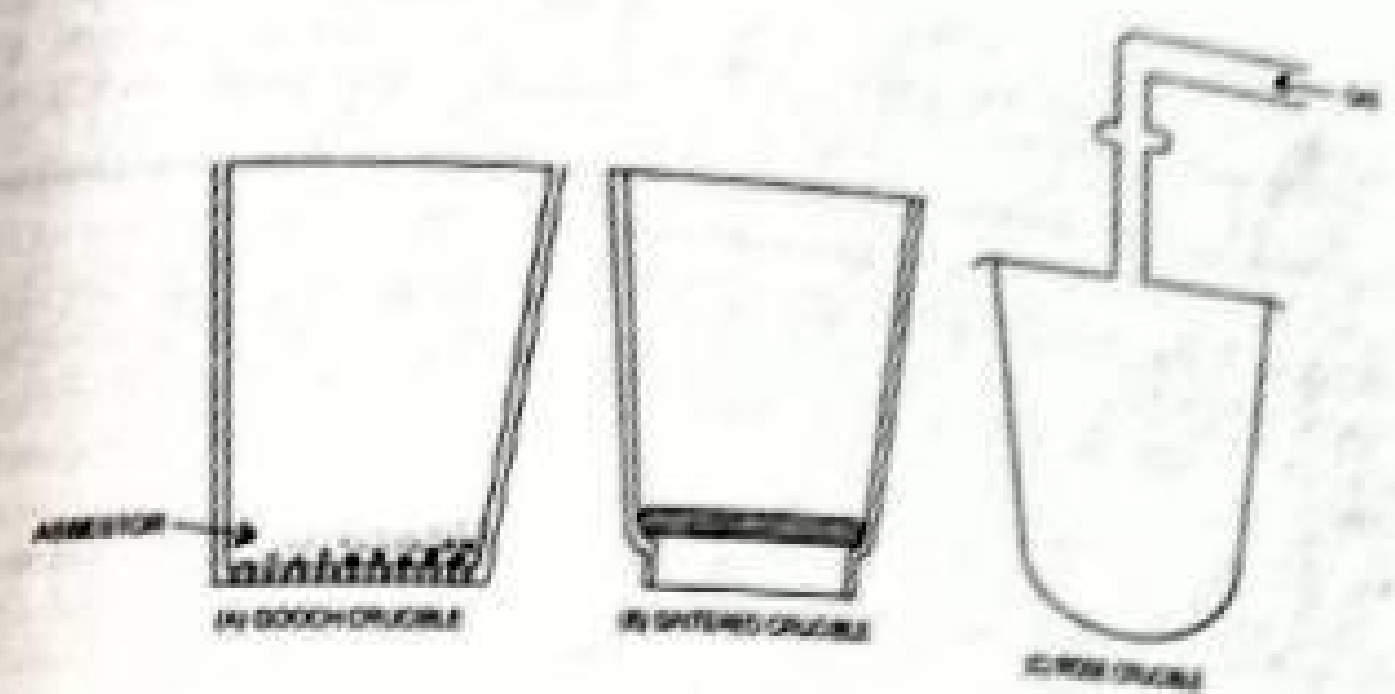


Fig. 9.1 Types of Crucibles

Sintered Crucible. In this crucible, instead of asbestos fibre a fixed layer of sintered glass particles is used as the filtering medium. The body of the crucible is made of resistance glass and a porous disc of sintered glass is fused into the body (Fig. 9.1 (b)). The filter disc is made in varying porosities indicated by numbers from zero to five.

Porosity:	0	1	2	3	4	5
Pore diameter (mm):	200-250	100-120	40-50	20-30	5-10	1-2

Smaller the porosity number, the sinter is less suitable for filtering fine particles of precipitate. For filtering precipitates of moderate particle size, porosity 3 is suitable. For fine particles such as BaSO₄, porosity 4 must be used.

A sintered crucible is also fitted into an adaptor and then into a flask in the same manner as a Gooch crucible. Sintered crucibles are convenient to handle because they are sturdy and remain nearly constant in weight. However, they should not be heated above 200°C. Gelatinous precipitates cannot be filtered through sintered crucibles because the filter pores tend to clog.

Precipitates from a sintered crucible may be gently removed by means of the bristles of a test tube brush and water. The sintered portion should never be scratched with glass rod, spatula, etc., because scratching will permanently damage the filtering medium. Particles in the pores of the filter can be dislodged by drawing water through in the reverse direction or by dissolving the

solid in a suitable solvent. Caustic alkali should not be used for cleaning because it attacks sinter. Concentrated acids may be used for cleaning. Acetone is convenient for cleaning because of its rapid drying. EDTA solution is particularly useful for removing precipitate such as barium sulphate. A new crucible should always be conditioned before being used in a desiccator. This is done by treating it repeatedly with warm hydrochloric acid, washing and drying. Weight constancy is attained.

Rose Crucible. A Rose crucible [Fig. 9.1 (c)] is used when a precipitate has to be weighed in an atmosphere of a gas, say, hydrogen, carbon dioxide or oxygen.

