THIRUTHANGAL NADAR COLLEGE

(Belongs to the Chennaivazh Thiruthangal Hindu Nadar Uravinmurai Dharma Fund)

Selavayal, Chennai-51.

A Self-Financing Co-educational College of Arts & Science

Affiliated to the University of Madras

Accredited with 'B' Grade by NAAC

An ISO 9001: 2015 Certified Institution

NAME OF THE DEPARTMENT: CHEMISTRY

SUBJECT : GRAVIMETRIC ANALYSIS LAB

TOPIC : GRAVIMETRIC ANALYSIS

STAFF NAME : Dr.R.EPSHIBA

Syllabus

1. Estimation of Lead as Lead chromate
2. Estimation of Barium as Barium chromate
3. Estimation of Nickel as Nickel - DMG complex.
4. Estimation of Calcium as Calcium oxalate
5. Estimation of Barium as Barium sulphate
6. Estimation of sulphate as Barium sulphate.
7. Estimation of Aluminium as Aluminium oxinate (for demonstration)
8. Estimation of Silver as Silver chloride (for demonstration)

Introduction about Gravimetric Analysis

Gravimetric analysis is the quantitative determination of analyte concentration through a process of precipitation of the analyte, isolation of the precipitate, and weighing the isolated product.

Uses of gravimetric analysis…

- Chemical analysis of ores and industrial materials
- Calibration of instrumentation
- Elemental analysis of inorganic compounds

Common Procedure

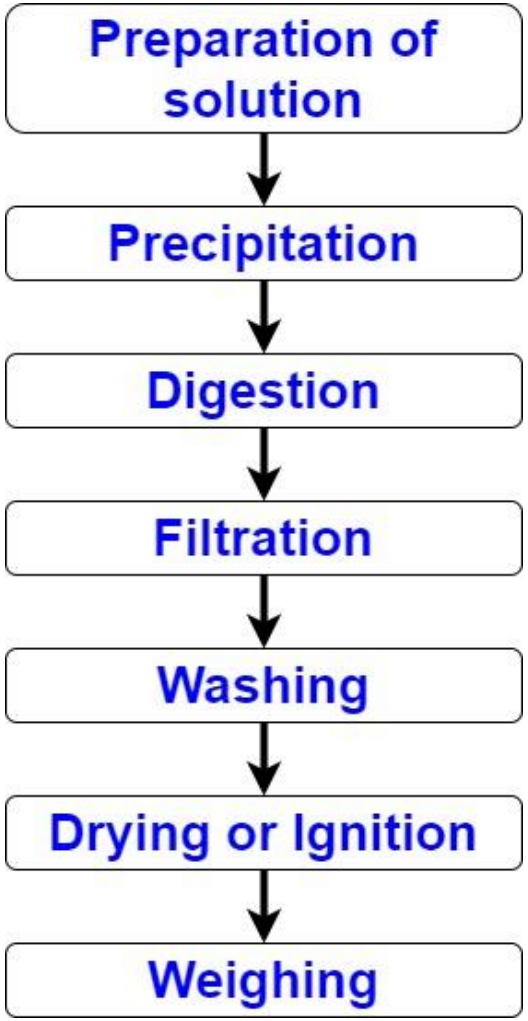
A weighed sample is dissolved

An excess of a precipitating agent is added to this solution

The resulting precipitate is filtered, dried (or ignited) and weighed

From the mass and known composition of the precipitate, the amount of the original ion can be determined

Steps to be followed



Preparation of Analyte Solution

- Make up the given solution in a standard measuring flask
- Treat this solution as a bulk
- Use this solution for further analysis

Precipitation Process

- Dilute the solution to avoid coprecipitation
- Gently heating avoids colloidal state formations and helps in giving good crystals
- Slight excess precipitating agent gives complete precipitation
- Avoid adding too much of precipitating agent

Digestion of the Precipitate

- Digestion increase precipitate size
- Digest the precipitate for minimum 30 minutes to 1 hour
- Check for completion of precipitation by adding small amount of precipitating agent on the walls of beaker

Filtration

- ✓ Isolate the precipitate from the solution
- ✓ Use filter paper (whatman no.42) or sintered glass crucible

Washing

- Removes undesired ions
- Washing solution recommended is solvent+precipitating agent/suitable reagent
- Check the filtrate for completion of filtration

Drying or Ignition

- To remove solvent and wash electrolytes
- Done by heating at 110 to 120°C for 1 to 2 hrs.
- Converts hygroscopic compound to non-hygroscopic compound
- May used high temp if precipitate must be converted to a more suitable form before weighing

Weighing

- After the precipitate is allowed to cool (preferably in a desiccator to keep it from absorbing moisture), it is weighed (in the crucible).
- Properly calibrated analytical balance
- Good weighing technique

1. Estimation barium as barium sulphate

- The given barium chloride solution is made up to 100mL in a standard flask.
- 20mL of solution is pipetted into a 400 mL beaker.
- About 5mL 2N HCl is added and diluted to 150mL with distilled water.
- The solution is heated to boiling and a hot solution of 4N H_2SO_4 (10 - 15mL) is added drop by drop with constant stirring, till the precipitation is complete.
- The solution containing the precipitate is heated in a water bath for 5 minutes.
- The precipitate is allowed to stand for an hour.
- The clear solution is decanted through an ashless filter paper (whatman No. 40).
- The precipitate is washed with hot distilled water to free sulphate ions.
- The particles adhering to the sides of the beaker and glass rod are removed by a policeman.
- Finally the precipitate is washed once again.
- The dried filter paper is folded and placed in a crucible which has been previously weighed.
- The filter paper with the precipitate is first incinerated on a Bunsen burner by a low flame and then transferred to an electric burner.
- The crucible is transferred to desiccator and cooled.
- When cold, the crucible is weighed.
- Heating, cooling and weighing are repeated till concordant values are obtained.

2. Estimation sulphate as barium sulphate

$$BaCl_2 + CuSO_4 \longrightarrow BaSO_4 \downarrow + CuCl_2$$

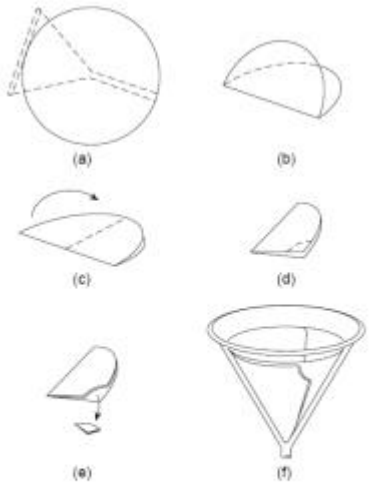
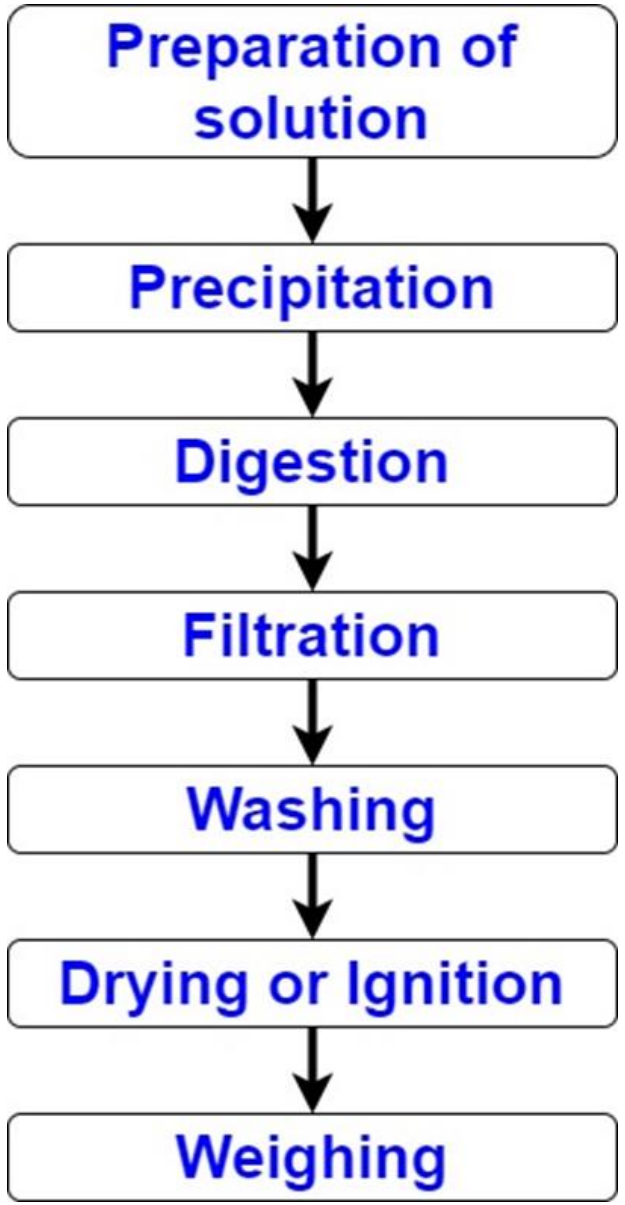


Figure 1. Folding filter paper

Figure 2. Proper filtering technique

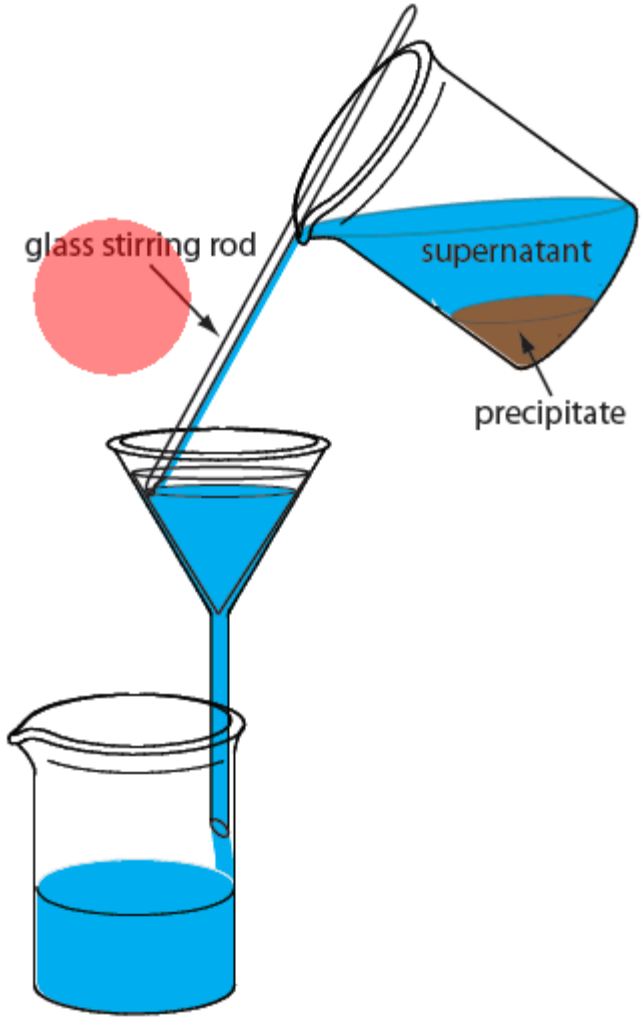
3. Estimation barium as barium chromate

$$BaCl_2 + K_2CrO_4 \longrightarrow BaCrO_4 + 2KCl$$



Procedure

•The given barium chloride solution is made up to 100mL in a standard flask.
•20mL of solution is pipetted into a 400 mL beaker.
•About 1mL 6M acetic acid & 10 mL 3M ammonium acetate are added and diluted to 100mL with distilled water.
•The solution is heated to boiling and a hot solution of 4% potassium chromate (20 mL) is added drop by drop with constant stirring, till the precipitation is complete.
•The solution containing the precipitate is heated in a water bath for 30 minutes.
•The precipitate is allowed to stand for an hour.
•The clear solution is decanted through sintered glass crucible.
•The precipitate is washed with hot distilled water to chromate.
•The particles adhering to the sides of the beaker and glass rod are removed by a policeman.
•Finally the precipitate is washed once again.
•The dried filter paper is folded and placed in a crucible which has been previously weighed.
•The filter paper with the precipitate is first incinerated on a Bunsen burner by a low flame and then transferred to an electric burner.
•The crucible is transferred to desiccator and cooled.
•When cold, the crucible is weighed.
•Heating, cooling and weighing are repeated till concordant values are obtained.


glass stirring rod
supernatant
precipitate

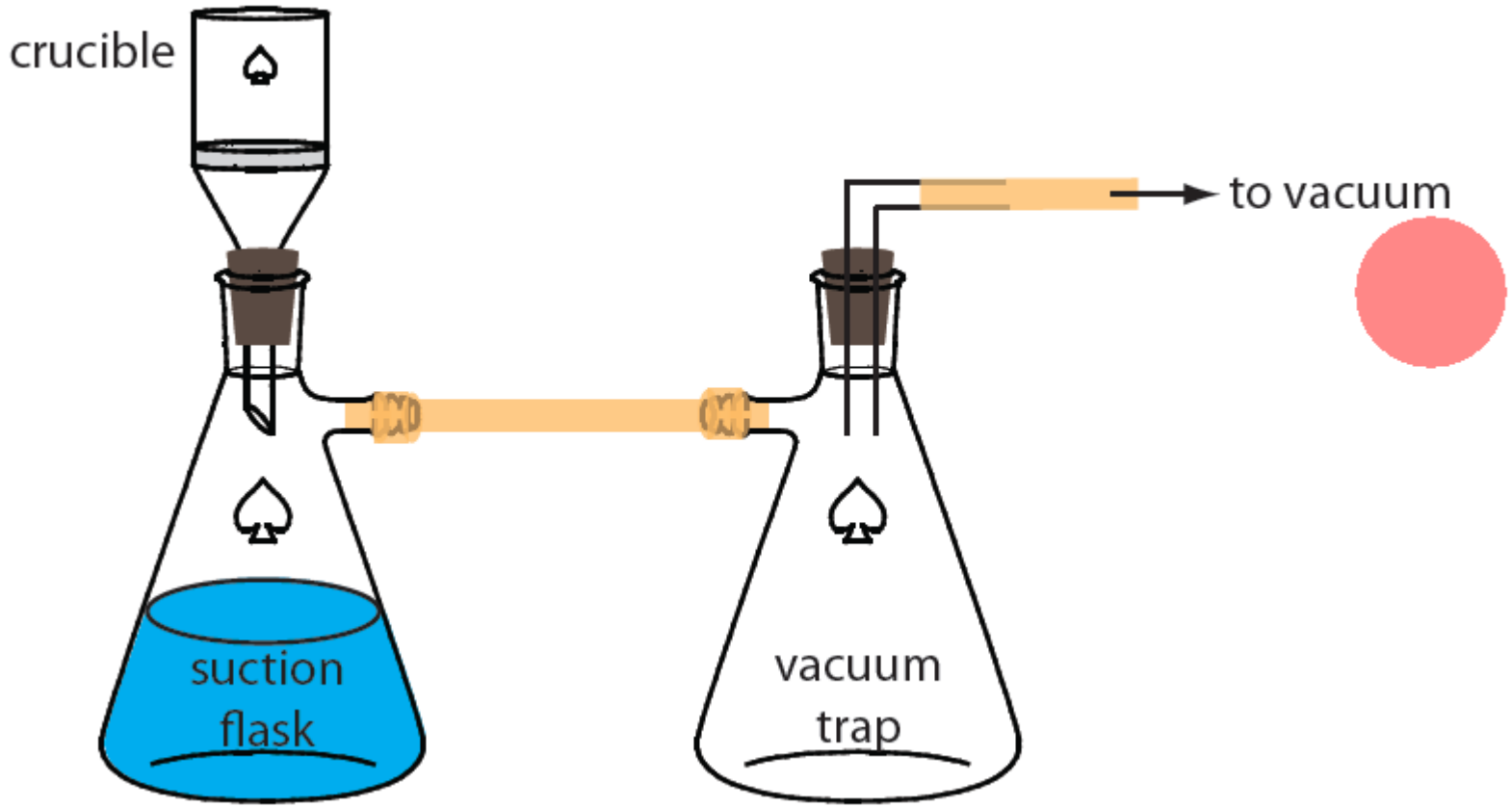

crucible
to vacuum
suction
flask
vacuum
trap

Table 8.1 Selected Precipitation Gravimetric Methods for Inorganic Cations and Anions (Arranged by Precipitant)			
Analyte	Precipitant	Precipitate Formed	Precipitate Weighed
Ba^{2+}	$(NH_4)_2CrO_4$	$BaCrO_4$	$BaCrO_4$
Pb^{2+}	K_2CrO_4	$PbCrO_4$	$PbCrO_4$
Ag^+	HCl	AgCl	AgCl
Hg_2^{2+}	HCl	Hg_2Cl_2	Hg_2Cl_2
Al^{3+}	NH_3	$Al(OH)_3$	Al_2O_3
Be^{2+}	NH_3	$Be(OH)_2$	BeO
Fe^{3+}	NH_3	$Fe(OH)_3$	Fe_2O_3
Ca^{2+}	$(NH_4)_2C_2O_4$	CaC_2O_4	$CaCO_3$ or CaO
Sb^{3+}	H_2S	Sb_2S_3	Sb_2S_3
As^{3+}	H_2S	As_2S_3	As_2S_3
Hg^{2+}	H_2S	HgS	HgS
Ba^{2+}	H_2SO_4	$BaSO_4$	$BaSO_4$
Pb^{2+}	H_2SO_4	$PbSO_4$	$PbSO_4$
Sr^{2+}	H_2SO_4	$SrSO_4$	$SrSO_4$
Be^{3+}	$(NH_4)_2HPO_4$	NH_4BePO_4	$Be_2P_2O_7$
Mg^{2+}	$(NH_4)_2HPO_4$	NH_4MgPO_4	$Mg_2P_2O_7$
Zn^{2+}	$(NH_4)_2HPO_4$	NH_4ZnPO_4	$Zn_2P_2O_7$
Sr^{2+}	KH_2PO_4	$SrHPO_4$	$Sr_2P_2O_7$
CN^-	$AgNO_3$	AgCN	AgCN
I^-	$AgNO_3$	AgI	AgI
Br^-	$AgNO_3$	AgBr	AgBr
Cl^-	$AgNO_3$	AgCl	AgCl
ClO_3^-	$FeSO_4/AgNO_3$	AgCl	AgCl
SCN^-	$SO_2/CuSO_4$	CuSCN	CuSCN
SO_4^{2-}	$BaCl_2$	$BaSO_4$	$BaSO_4$