KONGUNADU ARTS AND SCIENCE COLLEGE
B.Sc. CHEMISTRY

GRAVIMETRIC ANALYSIS

Name   :

Roll No. :

CALCULATION :

WEIGHING DETAILS :

| Object	Weights Added		Rider		Weight due to Rider Mg.	Total Weight g
	Grams	Milligram	MSD	SSD		
CRUCIBLE I Crucible with Precipitate						
Empty crucible						
Precipitate						
CRUCIBLE II Crucible with Precipitate						
Empty crucible						
Precipitate						

Experiment No.

Date :

ESTIMATION OF LEAD AS LEAD CHROMATE

Aim : *To estimate the weight of lead in the whole of the given lead acetate solution.*

PRINCIPLE: The given lead acetate solution is made up to a definite volume Lead present in a definite volume of the made up solution is precipitated as lead chromate by adding potassium solution.
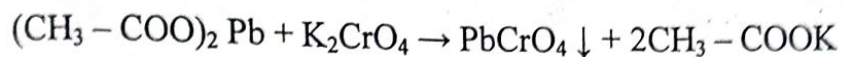
$$(CH_3 - COO)_2\ Pb + K_2CrO_4 \rightarrow PbCrO_4 \downarrow + 2CH_3 - COOK$$

The precipitate is filtered using sintered glass crucible, washed dried and weighed. From the weight of Lead Chromate, the weight of lead in the whole of the given solution is calculated knowing that 323.21g of lead chromate contains 207.21 g of lead.

PROCEDURE

The given lead acetate solution is made upto 100 ml. in a standard measuring flask. 20 ml of the made up solution is pipetted out into a 250 ml beaker. Ammonium hydroxide is added drop by drop till a permanent precipitate is formed. Dilute acetic acid is then added to make the solution slightly acidic. It is then diluted to 125 ml and heated to boiling. To the hot solution, a 4% solution (about 10 ml) of potassium chromate is added in slight excess with constant stirring. The mixture is then boiled for 5 to 10 minutes to make the precipitate granular. The precipitate is then allowed to settle. The supernatant liquid must be coloured slightly yellow. This indicates that potassium chromate solution is sufficient. The precipitate is then filtered through a previously weighed sintered glass crucible. It is washed with hot water, dried at 120° C, cooled and weighed. It is heated to constant weight. A duplicate experiment is conducted.

ESTIMATION OF LEAD AS LEAD CHROMATE

CRUCIBLE – I

Weight of precipitate lead chromate $\qquad = \qquad$ g

323.21 g of $PbCrO_4$ contains 207.21 g. of lead

Weight of lead in _____ g of $PbCrO_4$ = $\dfrac{207.21}{323.21} \times$ ___ g

Weight of lead in the Whole of the Given solution = $\dfrac{207.21}{323.21} \times$ ___ x 5g

$= \_\_\_\_$ g

CRUCIBLE – II

Weight of precipitate lead chromate $\qquad = _____$ g

323.21 g of $PbCrO_4$ contains 207.21 g. of lead

Weight of lead in _____ g of $PbCrO_4$ = $\dfrac{207.21}{323.21} \times$ ___ g

Weight of lead in the whole of the given solution = $\dfrac{207.21}{323.21} \times$ ___ x 5 g

$= _____$

From the weight of lead chromate, the weight of lead in the whole of the given solution is calculated.

RESULT

Weight of lead present in the whole of the given solution

CURICIBLE I = _____ g

CURICIBLE II = _____ g

CALCULATION :

WEIGHING DETAILS :

Object	Weights Added		Rider		Weight due to Rider Mg.	Total Weight g
	Grams	Milligram	MSD	SSD		
CRUCIBLE I Crucible with Precipitate						
Empty crucible						
Precipitate						
CRUCIBLE II Crucible with Precipitate						
Empty crucible						
Precipitate						

Experiment No.

Date :

ESTIMATION OF BARIUM AS BARIUM CHROMATE

Aim : *To estimate the weight of Barium in the whole of the given $BaCl_2$ solution.*

PRINCIPLE:

The given $BaCl_2$ solution. is made up to a definite volume. Barium present in a definite volume of the made up solution is precipitated as $BaCrO_4$ by adding K_2CrO_4 solution.

$$BaCl_2 + K_2CrO_4 \rightarrow BaCrO_4 \downarrow + 2KCl$$

The precipitate is filtered using sintered glass crucible, washed dried and weighed. From the weight of $BaCrO_4$, the weight of Ba in the whole of the given solution is calculated knowing that 253.34g of $BaCrO_4$ contains 137.34 g of Ba.

PROCEDURE

The given $BaCl_2$ solution is made upto 100 ml. in a standard measuring flask. 20 ml of the made up solution is pipetted out into a 250 ml beaker. Ammonium hydroxide is added drop by drop till a permanent precipitate is formed. Dilute acetic acid is then added to make the solution slightly acidic. It is then diluted to 125 ml and heated to boiling. To the hot solution, a 4% solution (about 10 ml) of K_2CrO_4 is added in slight excess with constant stirring. The mixture is then boiled for 5 to 10 minutes to make the precipitate granular. The precipitate is then allowed to settle. The supernatant liquid must be coloured slightly yellow. This indicates that K_2CrO_4 solution is sufficient. The precipitate is then filtered through a previously weighed sintered glass crucible.

ESTIMATION OF BARIUM AS BARIUM CHROMATE

CRUCIBLE – I

Weight of precipitate Barium chromate = _____ g

253.34 g of $BaCrO_4$ contains 137.34 g. of Ba

Weight of Barium in _____ g of $BaCrO_4$ = $\dfrac{137.34}{253.34}$ × _____ g

Weight of Barium in the whole of the given solution = $\dfrac{137.34}{253.34}$ × _____ × 5g

= _____ g

CRUCIBLE – II

Weight of precipitate Barium chromate = _____ g

253.34 g of $BaCrO_4$ contains 137.34 g. of Ba

Weight of Barium in _____ g of $BaCrO_4$ = $\dfrac{137.34}{253.34}$ × _____ g

Weight of Barium present in the whole of the given solution

= $\dfrac{137.34}{253.34}$ × _____ 5 g

= _____ g

It is washed with hot water, dried at $120°C$, cooled and weighed. It is heated to constant weight. A duplicate experiment is conducted. From the weight of $BaCrO_4$, the weight of Ba in the whole of the given solution is calculated.

RESULT

Weight of lead ~~lead~~ Ba present in the whole of the given solution = ------------g

CURICIBLE I = _____ g

CURICIBLE II = _____ g

CALCULATION:

WEIGHING DETAILS:

| Object	Weights Added		Rider		Weight due to Rider Mg.	Total Weight g
	Grams	Milligram	MSD	SSD		
CRUCIBLE I Crucible with Precipitate						
Empty crucible						
Precipitate						
CRUCIBLE II Crucible with Precipitate						
Empty crucible						
Precipitate						

Experiment No.

Date :

ESTIMATION OF CALCIUM AS CALCIUM OXALATE

Aim : *To estimate the weight of calcium in the whole of the given calcium chloride solution.*

PRINCIPLE:

The given calcium chloride solution is made up to a definite volume. Calcium present in a definite volume of the made up solution is precipitated as calcium oxalate monohydrate, $CaC_2O_4.H_2O$ by adding ammonium oxalate solution.
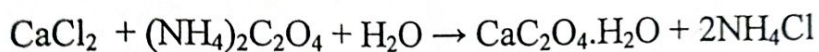
$$CaCl_2 + (NH_4)_2C_2O_4 + H_2O \rightarrow CaC_2O_4.H_2O + 2NH_4Cl$$

The precipitate is filtered using sintered glass crucible, washed, dried and weighed. From the weight of calcium oxalate, the weight of calcium in the whole of the given solution is calculated knowing that 146.08 g of $CaC_2O_4.H_2O$ contains 40.08 g of calcium.

PROCEDURE

The given calcium chloride solution is made upto 100 ml. in a standard measuring flask. 20 ml of the made up solution is pipetted out into a beaker, a drop or two drop of methyl orange indicater is added and the solution is carefully neutralized by ammonium hydroxide. About 5 ml of dilute hydrochloric acid is then added, diluted to 200 ml and heated to boiling. While continuously stirring, a moderate excess of a hot 2% solution of ammonium oxalate (freshly prepared) is gradually added. The precipitation of calcium oxalate is then completed by adding ammonium hydroxide drop by drop until the solution is alkaline. The solution is then boiled for a few minutes, care being taken to avoid bumping. It is then set aside for about an hour for cooling and settling of the precipitate.

ESTIMATION OF CALCIUM AS CALCIUM OXALATE

CRUCIBLE – I

Weight of precipitate Calcium oxalate $\quad\quad = \quad$ _____ g

146.08 g of $CaC_2O_4.H_2O$ contains 40.08 g. of Ca

Weight of Calcium present in _____ g

$$\text{of } CaC_2O_4.H_2O \quad = \quad \frac{40.08}{146.08} \text{ x } \underline{\quad\quad} \text{ g}$$

Weight of Calcium present in the whole of the

given solution $\quad\quad\quad = \quad \dfrac{40.08}{146.08} \text{ x } \underline{\quad\quad} \text{ x 5g}$

$$= \quad \underline{\quad\quad} \text{ g}$$

CRUCIBLE – II

Weight of precipitate calcium oxalate $\quad\quad = \quad$ _____ g

146.08 g of $CaC_2O_4.H_2O$ contains 40.08 g. of Ca

Weight of Calcium present in _____ g

$$\text{of } CaC_2O_4.H_2O \quad = \quad \frac{40.08}{146.08} \text{ x } \underline{\quad\quad} \text{ g}$$

Weight of Calcium present in the whole of the

given solution $\quad\quad\quad = \quad \dfrac{40.08}{146.08} \text{ x } \underline{\quad\quad} \text{ x 5g}$

$$= \quad \underline{\quad\quad} \text{ g}$$

The precipitate is the filtered through a sintered glass crucible. The precipitate is washed three times by cold ammonia solution (35 ml of liq. ammonia diluted to 1000 ml.). The washing is continued until a portion of the filtrate gives no turbidity with silver nitrate solution and nitric acid.

Finally, the precipitate is washed three times with small quantities of alcohol and then with ether. The crucible is then placed in air oven maintained at 100°C for half an hour, It is then cooled and weighed.

RESULT

Weight of calcium present in the whole of the given solution = ----------

-g

CURICIBLE I = _____ g

CURICIBLE II = _____ g

CALCULATION:

WEIGHING DETAILS:

| Object	Weights Added		Rider		Weight due to Rider Mg.	Total Weight
	Grams	Milligram	MSD	SSD		
CRUCIBLE I Crucible with Precipitate						
Empty crucible						
Precipitate						
CRUCIBLE II Crucible with Precipitate						
Empty crucible						
Precipitate						

Experiment No.

Date :

ESTIMATION OF NICKEL

Aim : *To estimate the weight of nickel in the whole of the given nickel ammonium sulphate solution.*

PRINCIPLE:

The given nickel salt solution is made up to a definite volume. Nickel present in a definite volume of this solution is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime in presence of slight excess of aqueous ammonia.

$$NiSO_4 + 2C_4H_8O_2N_2 \rightarrow Ni(C_4H_7O_2N_2)_2 \downarrow + H_2SO_4$$
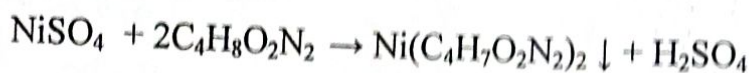
The precipitate is filtered through sintered glass crucible, washed, dried and weighed. From the weight of nickel dimethyl glyoxime obtained, the weight of nickel in the whole of the solution is calculated, knowing that 288.69 g of nickel dimethyl glyoxime contains 58.69 g of nickel.

PROCEDURE

The given nickel salt solution is made upto 100 ml. in a standard measuring flask. 20 ml of the made up solution is pipetted out into a 250 ml beaker. About 5 ml of 1:1 HCl is added and the solution diluted to 150 ml. The solution is heated to 70 - 80° C and 20 – 25 ml. of 1% dimethyl glyoxime in rectified spirit is added, immediately followed by dilute ammonia solution dropwise directly to the solution (and not along the sides of the beaker) till the solution is slightly alkaline as indicated by the smell of ammonia emanating from the solution. The solution containing the precipitate is heated on a steam bath for 30 minutes and the supernatant liquid tested for complete precipitation. The precipitate is allowed to stand for an hour. Cooling of the solution may be hastened by keeping the beaker in a basin of cold water

ESTIMATION OF NICKEL

CRUCIBLE – I

Weight of precipitate nickel dimethyl glyoxime $=$ _____ g

288.69 g of Complex contains 58.69g of Nickel

Weight of nickel present in the whole of the

given solution _____ g of the complex $= \dfrac{58.69}{288.69}$ x _____ g

Weight of Nickel present in the whole of the

given solution $= \dfrac{58.69}{288.69}$ x _____ x 5g

$= $ _____ g

CRUCIBLE – II

Weight of precipitate nickel dimethyl glyoxime $=$ _____ g

288.69 g of Complex contains 58.69g of Nickel

Weight of nickel present in _____ g

of the complex $= \dfrac{58.69}{288.69}$ x _____ g

Weight of nickel present in the whole of

the given solution $= \dfrac{58.69}{288.69}$ x _____ x 5g

$= $ _____ g

The cold solution is then filtered through a previously weighed sintered glass crucible (G4). The precipitate is washed with cold water until free from chloride. The sintered glass crucible is then placed in dry 100 ml. beaker and heated in an air oven at 110-120° C for 1 hour. It is allowed to cool in a desiccator and weighed. Heating, cooling and weighing are repeated till the weight is constant. A duplicate experiment is conducted.

From the weight of dimethyl glyoxime complex, the weight of nickel is calculated knowing that 288.69 g of complex contains 58.69 g of nickel.

RESULT

Weight of nickel present in the whole of the given solution = ---------------g.

CURICIBLE I = _____ g

CURICIBLE II = _____ g

CALCULATION :

WEIGHING DETAILS:

| Object	Weights Added		Rider		Weight due to Rider Mg.	Total Weight g
	Grams	Milligram	MSD	SSD		
CRUCIBLE I Crucible with Precipitate						
Empty crucible						
Precipitate						
CRUCIBLE II Crucible with Precipitate						
Empty crucible						
Precipitate						

Experiment No.

Date :

ESTIMATION OF BARIUM AS BARIUM SULPHATE

Aim : *To estimate the amount of barium in the whole of the given solution of barium chloride.*

PRINCIPLE:

The given barium chloride solution is made up to a definite volume. A measured volume of it is then treated with dilute H_2SO_4 and the barium precipitated as $BaSO_4$.

$$BaCl_2 + H_2SO_4 \rightarrow BaSO_4 \downarrow + 2HCl$$
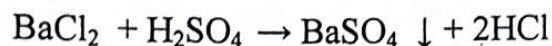
The precipitated $BaSO_4$ is separated and weighed. From the weight of $BaSO_4$, the weight of barium in the whole of the solution is calculated, knowing that 233.6 g $BaSO_4$ contains 137.36 g of barium.

PROCEDURE

The given barium chlorides solution is made up to 100 ml. in a standard measuring flask. 20 ml of the made up solution is pipetted out into a 250 ml. beaker. About 5 ml. of NH_4Cl is added and diluted to 100 ml. with distilled water. The solution is heated to boiling and a hot solution of 4N sulphuric acid (10-15 ml) is added drop by drop with constant stirring, till the precipitation is complete. The solution is boiled for about 5 minutes. The precipitate is allowed to settle and the supernatant liquid tested for complete precipitation by adding a few drops of hot 4N sulphuric acid through the sides of the beaker. The solution is kept hot (but not boiling) for half an hour in order to allow time for complete precipitation.

The clear solution is decanted through an ashless filter paper (Whatman No. 40). The precipitate is washed with small portions of hot distilled water until the washings does not give precipitate with barium chloride solution (free from sulphate ions). The precipitate is then transferred completely into the filter paper.

ESTIMATION OF BARIUM AS BARIUM SULPHATE

CRUCIBLE – I

Weight of precipitate barium sulphate $\qquad$ = _____ g

233.36 g of $BaSO_4$ contains 137.36 g of Ba

Weight of Barium in _____ g of $BaSO_4$ $\qquad = \dfrac{137.36}{233.36} \times$ _____ g

Weight of Barium present in the whole of the

given solution $\qquad = \dfrac{137.36}{233.36} \times$ _____ × 5g

$\qquad$ = _____ g

CRUCIBLE – II

Weight of precipitate barium sulphate $\qquad$ = _____ g

233.36 g of $BaSO_4$ contains 137.36 g of Ba

Weight of Barium in _____ g of $BaSO_4$ $\qquad = \dfrac{137.36}{233.36} \times$ _____ g

Weight of Barium present in the whole of the

given solution $\qquad = \dfrac{137.36}{233.36} \times$ _____ × 5g

$\qquad$ = _____ g

Any particles stricking to the sides of the beaker are removed by a policeman. Finally the precipitate is washed once again. After the water has completely drained off, the funnel is kept in the air oven for drying. When dried, the filter paper is removed from the funnel and folded in the form of a packet so as to enclose the precipitate taking special care not to tear the paper. The packet is placed point down in a crucible which previously heated to constant weight.

The crucible is then mounted on a clay pipe triangle partially covered by the lid and heated by a low flame until the paper charrs and volatile matter is expelled. The paper should not be allowed to inflame as it causes mechanical expulsion of find particles. If the paper catches fire, the flame should be extinguished by momentarily placing the lid on the mouth of the crucible. When the charring is complete, the temperature is raised to dull redness and the carbon burnt off with free access of air. When the precipitate is white, the crucible is cooled, two drops of dilute H_2SO_4 is added and carefully heated, gently first and then strongly for about 10 minutes. If there is any deposit of carbon on the lid, it could be heated separately by holding it in the flame with a pair of tongs. The crucible is then transferred into a desiccator. The lid of the desiccator is kept partially opened for about 3 minutes, to avoid air current inside the desiccator and then closed completed. When cold, the crucible is weighted. Heating, cooling and weighing are repeated till concordant results are obtained.

A duplicate experiment is conducted. From the weight of $BaSO_4$ the weight of Ba in the whole of given solution is calculated knowing that 233.36 g of $BaSO_4$ contains 137.36 g of Ba.

RESULT

Weight of Barium present in the whole of the given solution = ---------------g.

CURICIBLE I = _____ **g**

CURICIBLE II = _____ **g**

21.200