Experiment 11

AIM

To separate the components of a mixture of sand, common salt and ammonium chloride.

THEORY

Sand, common salt, and ammonium chloride form a heterogeneous mixture and can be separated easily by physical methods of separation. By selecting the right order of methods of separation, the three can be easily separated:

(i) ammonium chloride sublimes on heating;

(ii) sand is insoluble in water; and

(iii) common salt can be recovered by evaporation of its aqueous solution.

MATERIALS REQUIRED

A china dish, a funnel, a beaker (250 mL), a cotton plug, burner, tripod stand, wire gauge, sand, common salt, ammonium chloride, water, and filter paper.

PROCEDURE

1. Take mixture of ammonium chloride, sand and common salt in a china dish.

Fig. 11.1 : *Separation of components of a mixture of ammonium chloride, sand and common salt. (a) Separation of ammonium chloride by sublimation; (b) Residue containing sand and common salt dissolved in water; (c) Separation of sand by filtration; and (d) Obtaining common salt by evaporation*

2. Set up a sublimation apparatus as shown in Fig. 11.1(a).
3. Heat the mixture. Ammonium chloride will be separated on the walls of the inverted funnel.
4. The residue left behind in the china dish contains sand and common salt.
5. Dissolve this residue mixture in water. Common salt will dissolve but sand will not [Fig. 11.1(b)].
6. Filter the sand from the mixture using a filter paper [Fig. 11.1(c)].
7. Sand is separated as residue and the filterate is salt solution in water.
8. Heat the filterate (salt solution) to evaporate the water and to get the dry sample of common salt [Fig. 11.1(d)].

Results and Discussion

Using methods of separation sequentially, ammonium chloride, sand and common salt have been separated from their mixture.

Precautions

- Sublimation process should be carried out carefully.
- Take care while filtering so that the filter paper does not tear off.

Note for the Teacher

- If soil is taken instead of sand for formation of mixture, it will form colloidal solution and clear filterate may not be obtained.
- As in the laboratory the sublimation apparatus is not air tight, recovery of ammonium chloride will not be 100%.
- The outer surface of funnel must be covered with wet cotton to separate sublimation product (ammonium chloride in this case) easily.
- To make the process of separation easily workable, it is desired that one water soluble component be separated by a process other than solubility and that is sublimation in this experiement.

QUESTIONS

- If in the first step the mixture had been dissolved in water what would have been the difficulty in separation.
- Instead of common salt if one component in the given mixture is sulphur, how would you carry out separation process then?
- Can the two components of a mixture that are soluble in water be separated by any technique? Justify your answer.

Experiment 12

Aim

To prepare solutions of various substances and to identify them as true solutions and suspensions.

Theory

A true solution is a homogeneous mixture of two or more substances. The solute and solvent particles can not be observed by the naked eye as they are very small (of the order of 1nm). A suspension is a hetrogeneous mixture of two or more substances. The solute particles of a suspension are often visible by the naked eyes as the size of particles is more than 0.1 mm.

Materials Required

Powder samples of sodium chloride (or common salt), sugar, baking soda, chalk powder, sand, and sulphur etc., a beaker (250 mL), water, and a glass stirrer rod.

Procedure

1. Take a small amount of a solid sample as solute.
2. Dissolve it in 100 mL water taken in a beaker with the help of a glass stirrer rod. Stir the mixture for some time.
3. Allow it to stand for some time.

4. Observe and record whether the mixture formed is homogeneous or heterogeneous.
5. Repeat the experiment for different solid solutes.

Observations

Sl. No.	Solute	Homogenous or heterogeneous	Type of mixture formed (true solution or suspension)
1.			
2.			
3.			
4.			

Results and Discussion

(a) Samples __________, __________, __________ , form true solution in water. Which are homogeneous.

(b) Samples __________, __________ form suspension in water.

Note for the Teacher

- Students may be provided fine powder samples.
- If a light beam from a torch is passed through suspensions, scattering of light may also be observed.

Questions

- Why are the particles of a true solution not visible to naked eye?
- What is the order of the size of a particle that can be seen by naked eyes?
- What different techniques of seperation can be employed for separation of components of homogeneous and heterogeneous mixture?
- What will be your observation, when a beam of light is passed through a true solution and through a suspension respectively?

Experiment 13

Aim

To prepare a colloidal solution of sulphur and differentiate it from a true solution or from a suspension on the basis of transparency and filtration.

Theory

A collodial solution of sulphur in water can be obtained by the oxidation of hydrogen sulphide by nitric acid.

$$H_2S\ (aq) + 2HNO_3\ (aq) \longrightarrow 2H_2O\ (l) + S\ (s) + 2NO_2\ (g)$$

A true solution is homogenous and transparent. It can be completely filtered through a filter paper. It remains stable on standing.

A colloidol solution is heterogenous, it appears translucent. It can be filtered completely through filter paper. Here solute particles do not easily settle down on standing.

A suspension is heterogenous mixture. The particles of a suspension are visible to the naked eye. They settle down on standing and can be separated by filtration.

Materials Required

Kipp's apparatus to get hydrogen sulphide gas, concentrated nitric acid, common salt, chalk powder, funnel, four boiling tubes, glass rod, test tube stand, and filter paper.

PROCEDURE

(i) Preparation of Colloidal Solution of Sulphur

1. Take about 20 mL distilled water in a boiling tube and pass H_2S gas through it for about 5 minutes. The solution would smell like a rotten egg.

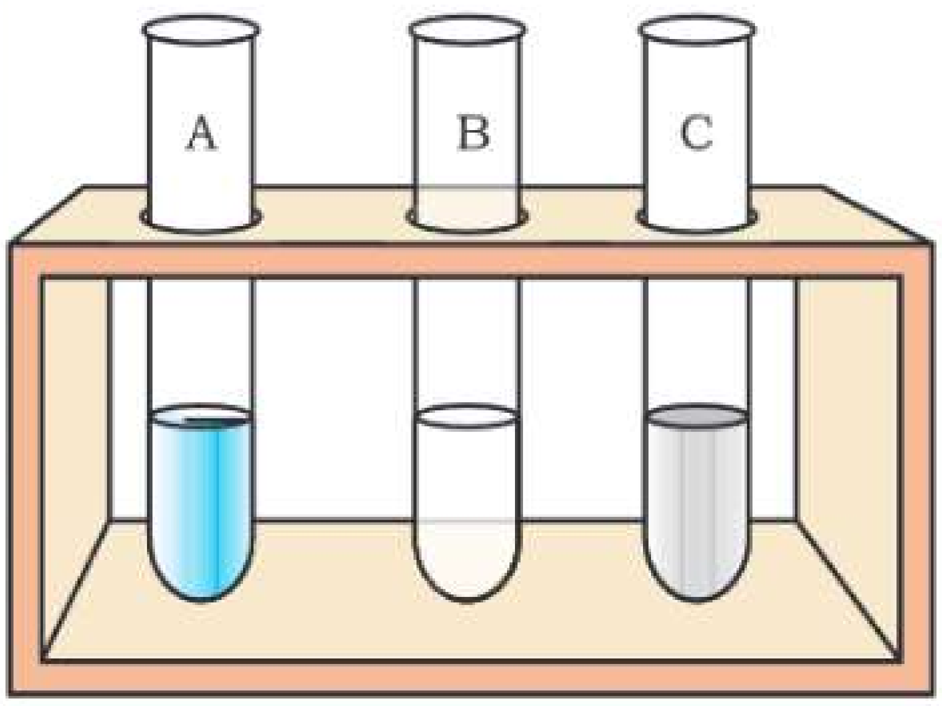

Fig. 13.1

2. Add few drops of concentrated nitric acid in it. Stir the solution. Continue adding few more drops of nitric acid untill solution becomes milky.
3. Transfer the contents into a another clean boiling tube. Label this boiling tube as tube A.

(ii) Preparation of True Solution

4. Take about 20 mL distilled water in a clean boiling tube.
5. Add approximately 1- 2 g of sodium chloride (or common salt) into it.
6. Stir the solution till it becomes clear. Label this boiling tube as tube B.

(iii) Preparation of suspension

7. Take about 20 mL of distilled water in another clean boiling tube.
8. Add approximately 1- 2 g of powdered chalk into it.
9. Stir the mixture with the help of glass rod. Label this boiling tube as tube C.

OBSERVATIONS

Sl.No.	Experiment	Boiling tube	Inference
1.	**Transparency**	A	
	Observe the contents of	B	
	different boiling tubes	C	
2.	**Filtration**		
	Filter the contents of	A	
	different tubes through	B	
	an ordinary filter paper	C	

Results and Discussion

Colloidal and suspension are heterogenous mixtures where as true solutions are homogenous mixtures. They differ form each other only on the basis of their particle size.

Note for the teacher

- Arrangement for the preparation of H_2S gas may be done in advance in the laboratory using the Kipp's apparatus. It requires ferrous sulphide and conc. sulphuric acid.
- When sulphur is formed by an insitu reaction the particle size is in the colloidal range as particles of sulphur remain aggregate in water, forming a colloidal solution.
- Sulphur can form true solution in carbon tetrachloride or carbon disulphide solvent.
- A suspension of sulphur can be formed in water by mixing sulphur powder in distilled water.
- Colloidal solution of sulphur can also be obtained by adding dil. HCl or conc. H_2SO_4 to sodium thiosulphate solution

$$\underset{\text{(sodium thiosulphate or hypo solution)}}{Na_2S_2O_3} + 2HCl \rightarrow 2NaCl + SO_2 + \underset{\text{(colloidal sulphur)}}{S} + H_2O$$

Questions

- What will be the effect of passing light through colloidal solution of sulphur?
- What is the difference in the particle size of colloid, true solution and suspension.
- Classify the following as a true solution, as a suspension, or as a colloid: (i) milk; (ii) $CuSO_4$ solution; (iii) jam; (iv) gum; (v) soil in water; and (vi) sand in water

Experiment 14

AIM

To study the process of separation of a mixture of two immiscible liquids.

THEORY

The separation of two immiscible liquids by a separating funnel depends on the difference in their densities. A less denser liquid floats over a liquid whose density is more.

MATERIALS REQUIRED

Separating funnel (250 mL)(with its stop-cock), two beakers (250 mL), and two immiscible liquids such as water and kerosene.

PROCEDURE

1. Take a mixture of two immiscible liquids (say water and kerosene) in a separating funnel (Fig. 14.1).
2. Allow it to stand for some time.
3. The mixture separates into two liquid layers according to their densities.
4. Collect the lower layer carefully in a beaker by opening the stop-cock of the separating funnel.
5. Similarly collect the upper layer in another beaker.

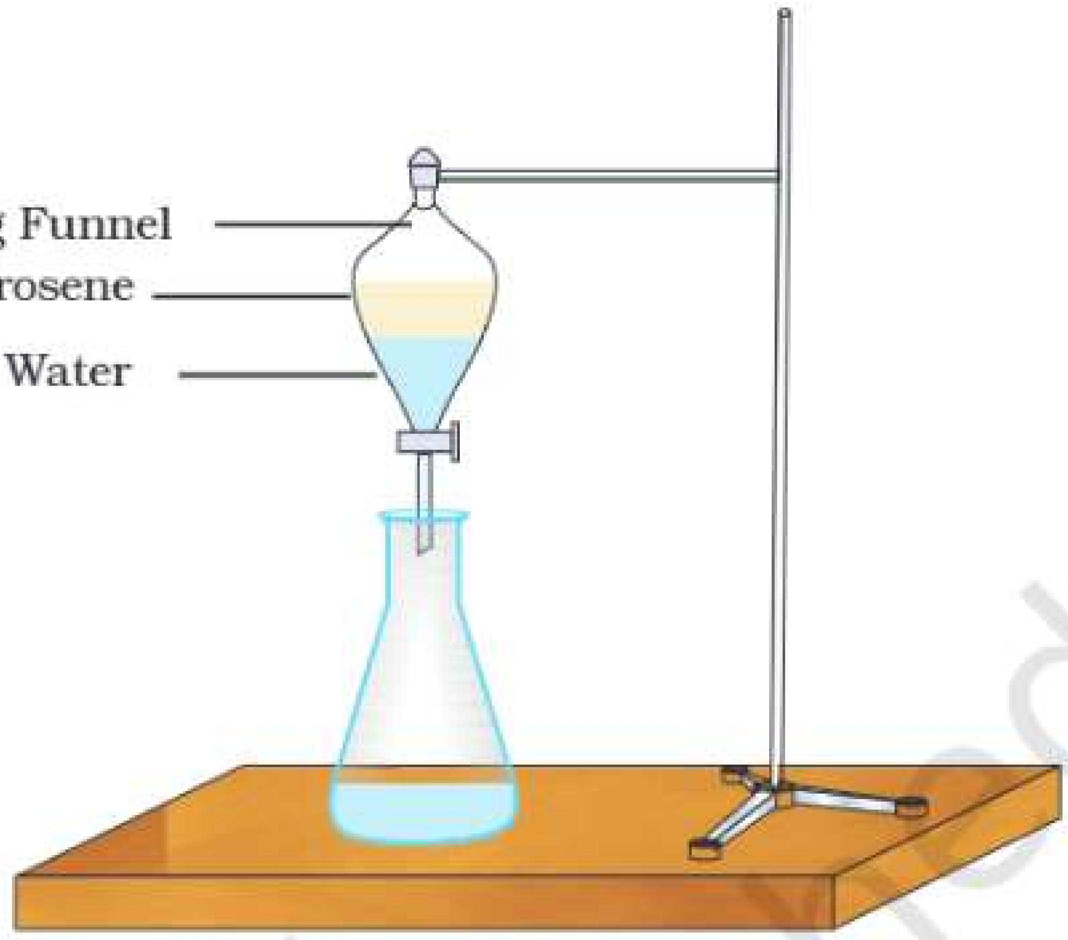

***Fig. 14.1 :** Separation of immiscible liquids*

Results and Discussion

Water and kerosene are immiscible and can be separated by using separating funnel. Miscibility and immiscibility of liquid pairs depends upon the effectiveness of intermolecular interaction.

Note for the Teacher

- It is essential to remove the lid of the separating funnel while opening the stop cock of the separating funnel.
- In order to get the two components in pure form, it is advisable to discard a part of the mixture at the junction of the two layers.

Questions

- Arrange the two liquids used in the above experiment according to the increasing order of their densities?
- Think of a technique that you can use to separate the above immiscible liquids if separating funnel is not available?
- Is the mixture used in the above experiment heterogenous or homogenous in nature?
- Which of the two sea water or pure water has got higher density?
- Can there be any way of varifying that lower layer in the separating funnel is water layer. Explain either way.

Experiment 15

AIM

To separate a mixture of two miscible liquids by simple distillation.

THEORY

The separation of two miscible liquids (having at least a difference in their boiling points of 25 K) can be separated by a simple distillation method. Distillation depends on the difference in their boiling points. The liquid which has lower boiling point evaporates first and faster than the liquid which has a higher boiling point.

MATERIALS REQUIRED

Mixture of two miscible liquids (water and acetone), measuring cylinder, a round bottom flask (250 mL), thermometer (-10 °C - 110 °C), condenser, two beakers (250 mL), burner, tripod stand, and a wire gauge.

PROCEDURE

1. Take a mixture of 50 mL water and 50 mL acetone in a round bottom flask.
2. Arrange the apparatus as shown in the Fig. 15.1.
3. Heat the mixture of acetone and water slowly and carefully monitor the rise in temperature.

4. Observe and note the temperature at which the first component of the mixture distils out, that is, vapours get cooled and collected in a beaker kept at the other end of the condenser.
5. Continue heating and similarly observe and note the temperature at which the second component distills out.

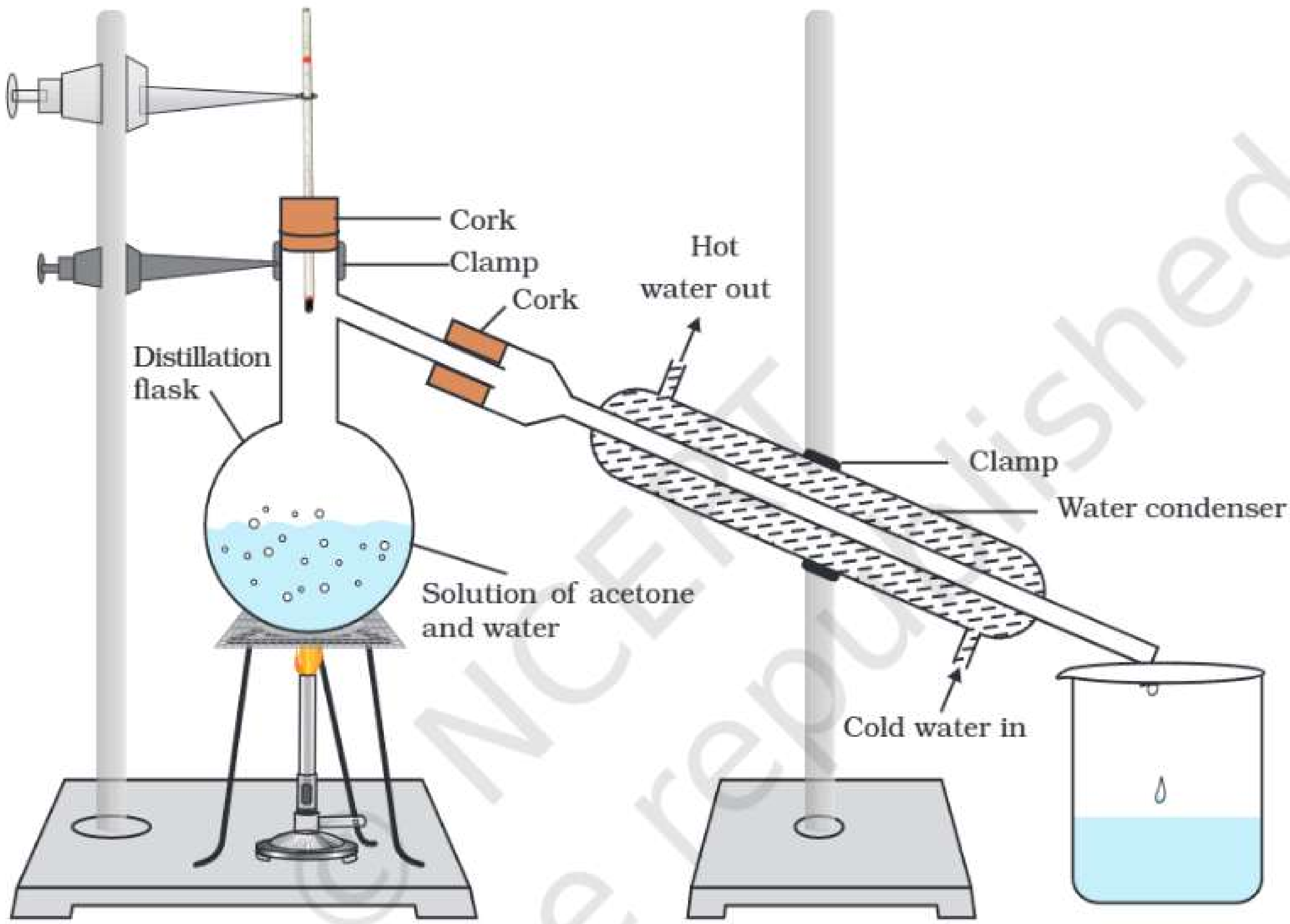

***Fig. 15.1 :** Laboratory apparatus for distillation*

OBSERVATIONS

	Component	Component
	I	II
Temperature Name of the component		

RESULTS AND DISCUSSION

The two components of the miscible liquids are separated by distillation. The difference in the boiling points of the liquids depend upon the attraction between the particles of the liquid.

NOTE FOR THE TEACHER

- The intermediate fraction may be rejected as it may contain both the components.
- For distillation, in place of round bottom flask and condenser, a distillation apparatus is easy to use.
- The process of distillation can be stopped after the separation of first component, as the second component of the mixture is left in the round bottomed flask.
- A discussion on the difference between bottled mineral water and distilled water may be initiated in the class room.

QUESTIONS

- In the above experiment you have found the boiling points of water and acetone. Use this information to arrange acetone and water in the order of (i) increasing force of attraction between the particles of water (water-water); particles of acetone (acetone-acetone); and (ii) increasing densities.
- You are provided with a mixture of methanol and ethanol having boiling points 61 °C and 78 °C respectively. Can you separate the two components by simple distillation method? Explain.
- You are given a sample of tap water, suggest a technique for obtaining pure and salt free water (distilled water) from it?
- What is the natural technique of obtaining distilled water from the nature?
- What is the utility of acetone in daily life?

Experiment 16

AIM

To differentiate between a mixture (containing two components) and a pure compound.

THEORY

The components of a mixture retain their individual properties. In a mixture these components can have any ratio while components of a mixture lose their individual properties once the compound is formed. Ratio of components in a compound is fixed. Merely mixing two components is a physical change and converting these into a compound is a chemical change.

MATERIALS REQUIRED

Sulphur powder, iron filings, dil. hydrochloric acid (or dil. sulphuric acid), lead acetate solution, carbon disulphide solvent, a bar magnet, two beaker (100 mL), three test tubes, china dish, watch glass, glass rod, filter paper, tripod stand, burner, wire gauge, splinter (or candle), and a mortor and pestle.

PROCEDURE

1. Take iron fillings (5.6 g) and sulphur powder (3.2 g) in a beaker. Mix them properly. Label this mixture as A.

2. Take half of this mixture A in a china dish and heat it slowly with constant stirring untill black mass is formed.
3. Cool the contents of china dish.
4. Grind the black mass with the help of mortar and pestle and put it in another beaker and mark it as B.
5. Perform various tests (as suggested in the table below) with samples A and B and record your observations.

OBSERVATIONS

Sl. No.	Experiment	Observation with sample		Inference
		A	B	
1.	**Magnet test** Move a magnet over the sample A and powdered black mass in sample B several times.			
2.	**Gas test** Take a small amount of mixture from sample A in a test tube and add 5 mL dil. hydrochloric acid or dil. H_2SO_4 carefully. Test the evolved gas by (a) bring a burning splinter or lighted candle near the mouth of the test tube; (b) bring a filter paper dipped in lead acetate solution near the mouth of the test tube. Repeat the same test with sample B.			
3.	**Solubility test** Take a pinch of A and B in test tubes. Add 5 mL carbon disulpide (CS_2) and shake the mixture. Filter the solution.			

PRECAUTIONS

- Do not inhale hydrogen sulphide gas as it a poisonous gas.
- Carbon disulphide is inflammable, so keep it away form the flame.

Note for the Teacher

- In order to simplify the performance part of this experiment, it is suggested that 5.6 g of iron filings and 3.2 g of sulphur powder may be provided to the students.
- It is important to maintain the proper stoichiometry in the reaction of iron with sulphur. In case of excess iron the product will be attracted towards magnet.
- A mixture containing iron and sulphur shows the properties of both its contituents. Iron filings are attracted towards a bar magnet and react with dil. hydrochloric acid or dil. sulphuric acid to liberate hydrogen. Hydrogen burns with the pop sound at the mouth of the test tube. This reaction is highly exothermic and therefore, must be performed carefully.

$$2Fe\ (s) + 6HCl\ (aq) \longrightarrow 2FeCl_3\ (aq) + 3H_2\ (g);$$
$$2Fe\ (s) + 3H_2SO_4\ (aq) \longrightarrow Fe_2(SO_4)_3\ (aq) + 3H_2\ (g);$$

Sulphur is soluble in non polar solvent like carbon disulphide (CS_2). The compound iron sulphide (FeS) is formed on heating iron (Fe) and sulphur (S).

$$Fe\ (s) + S\ (s) \xrightarrow{\text{heat}} FeS\ (s)$$

Iron sulphide reacts with dilute hydrochloric acid forms hydrogen sulphide (H_2S) gas which turns lead acetate paper into shiny black. Iron sulphide is insoluble in carbon disulphide solvent.

$$2FeS\ (s) + 2HCl\ (aq) \longrightarrow 2FeCl_2\ (aq) + H_2S\ (g);$$
$$2FeS\ (s) + H_2SO_4\ (aq) \longrightarrow 2FeSO_4\ (aq) + H_2S\ (g);$$
$$(CH_3COO)_2\ Pb\ (aq) + H_2S\ (g) \longrightarrow \underset{\text{(Black)}}{PbS\ (s)} + 2CH_3COOH\ (aq).$$

Questions

- How would you proceed to test that a mixture of NH_4Cl and $CuSO_4$ give test for NH_4^+, Cl^-, Cu^{2+} and SO_4^{2-} ions?
- Which one is the more appropriate statement amongst the following and Why? (i) Air is an oxidising agent; (ii) Oxygen of the air is an oxidising agent.
- Why does brass react with dilute hydrochloric acid and is corroded in rainy season to form $CuCO_3 . Cu(OH)_2$?

Experiment 17

Aim

To verify the law of conservation of mass in a chemical reaction.

Theory

Law of conservation of mass states that the mass remains conserved during a chemical reaction. In this experiment we shall verify the law of conservation of mass using a precipitation reaction. This reaction is considered as the simplest method to verify this law.

Materials Required

Barium chloride ($BaCl_2.2H_2O$), sodium sulphate ($Na_2SO_4.10H_2O$), distilled water, two beakers (150 mL), one beaker (250 mL), physical balance, spring balance (0 – 500 g) and a polythene bag, two watch glasses of known masses, and a glass stirrer.

Procedure

1. Pour 100 mL distilled water in two beakers (150 mL).
2. Using the physical balance and a watch glass of known mass, weigh 7.2 g of $BaCl_2.2H_2O$ and dissolve it in a beaker (150 mL) containing 100 mL distilled water.
3. Similarly, weigh 16.1 g of $Na_2SO_4.10H_2O$ in another watch glass of known mass and dissolve it in another beaker (150 mL) containing 100 mL distilled water.

4. Take the third beaker (250 mL) and weigh it using a spring balance and polythene bag (see Experiment no. 3 for details).
5. Mix both solutions of 150 mL beakers in the third beaker (250 mL). Mix the contents using a glass stirrer.
6. On mixing white precipitate of $BaSO_4$ appears due to precipitation reaction.
7. Weigh the beaker containing the reaction mixture again to determine the mass of the precipitation reaction products.
8. Compare the masses of before and after the chemical reaction.

OBSERVATIONS

(i)	Mass of 100 mL distilled water (The density of distilled water is 1 g/mL.)	= 100.0 g
(ii)	Mass of $BaCl_2.2H_2O$	= 7.2 g
(iii)	Mass of $BaCl_2$ solution	= 107.2 g
(iv)	Mass of $Na_2SO_4.10H_2O$	= 16.1 g
(v)	Mass of Na_2SO_4 solution	= 116.1 g
(vi)	Total Mass of reactants (solutions of $BaCl_2$ and Na_2SO_4)	= 223.3 g
(vii)	Mass of empty 250 mL beaker, m_1	= _____ g
(viii)	Initial mass of reaction mixture and empty beaker (before the precipitation), $m_2 = (m_1 + 223.3\ g)$	= _____ g
(ix)	Final mass of reaction mixture in the beaker after the precipitation, m_3	= _____ g.

RESULTS AND DISCUSSION

Compare the initial mass (m_2) of the reaction mixture (before the precipitation) with the final mass (m_3) of the reaction mixture (after the precipitation). Are they same? If the two masses are same within the reasonable limits, then the law of conservation of mass stands verified. The verification of the law rests on accurate mass measurements in the laboratory.

The chemical reaction involved is

$$BaCl_2\,(aq) + Na_2SO_4\,(aq) \longrightarrow \underset{\text{White precipitate}}{BaSO_4\,(s)} + 2NaCl\,(aq)$$

and more precisely

$$Ba^{2+}\,(aq) + SO_4^{2-}\,(aq) \longrightarrow BaSO_4\,(s)$$

PRECAUTIONS

- The spring balance should be held vertical while taking measurements.
- Before making use of the spring balance it must be ensured that its pointer is at zero mark. If not then ask your teacher to help.
- The readings of the spring balance should be noted only when its pointer comes to rest.
- Mixing of barium chloride and sodium sulphate solutions be done slowly with constant stirring.

NOTE FOR THE TEACHER

A physical balance is a sophisticated equipment. It is advised that students may be trained to use a physical balance and they must be properly supervised while they use the physical balance. This experiment involves several weighings that may take lot of time. In case if the weighing measurements takes lot of time, the required quantities of barium chloride and sodium sulphate may be provided to the students on two separate watch glasses. This will facilitate students to concentrate on the reaction dynamics rather than on the weighing skills.

QUESTIONS

- What are the other precipitation reactions that can be conveniently studied in the laboratory to verify this law?
- How can the law be verified by studying combination reaction? Suggest a procedure for the same.