# III.4. THE PHYSICAL SEPARATION OF SUBSTANCES

There are many methods which can be used to separate one substance from another. All the methods examined in this section take advantage of differences in the physical properties of the substances involved. (Some advanced techniques rely on using differing chemical properties of the substances involved, but these techniques produce different chemical substances and are not considered here.)

## A. HAND SEPARATION

A **MECHANICAL MIXTURE** can often be separated by ***hand*** or by the use of a ***sieve*** or ***magnet***.

*Example:* Collectors in the past picked rubies out of the gravel in the streams of Burma by hand.

Sieves are used to separate fossilized dinosaur bone fragments from sand and gravel.

Magnets are used during recycling operations to separate the iron from a mixture of finely chopped iron, plastic, aluminum and copper.

## B. FILTRATION

Filtration allows the separation of liquids from solids; that is, the separation of **MECHANICAL MIXTURES** involving liquids and solids.

**Note:** Filtration ***cannot*** be used to separate DISSOLVED solids from a liquid. Therefore, you can't remove the salt from a salt water solution by filtration methods. Filtration only works when the solid particles present are big enough to be seen; smaller particles (such as dissolved salt) simply pass right through the filter paper.

The material which remains behind on **FILTER PAPER** is called the **RESIDUE**, and the liquid which passes through filter paper is called the **FILTRATE.**

*Example:* Sand can be filtered out of a sand/sea water mixture. After the filtration, the sand is the residue and the sea water is the filtrate.

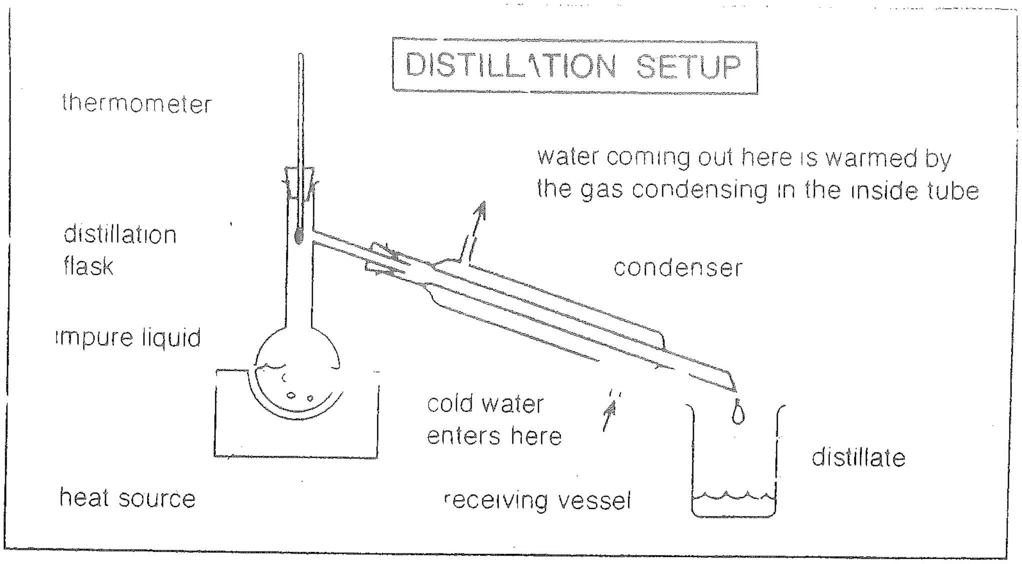
## C. EVAPORATION

Evaporation involves allowing the liquid in a **SOLID–IN–LIQUID SOLUTION** to evaporate or to be boiled away, leaving the solid.

*Example:* Evaporation of the water from salt water is used in the commercial production of salt on several seacoasts.

## D. DISTILLATION

When a **LIQUID–IN–LIQUID SOLUTION** is heated in a distillation setup (illustrated below), the liquid with the lowest boiling temperature boils first. The vapour produced is at the exact boiling temperature of the liquid and ascends to the top of the **DISTILLATION FLASK,** passes a thermometer, enters the side–arm of the flask and contacts the cold inner surface of the **CONDENSER.** The gas cools and CONDENSES back into a liquid, dropping out the end of the condenser as a purified liquid called a **DISTILLATE.** If the liquids present have boiling temperatures which are quite close to each other, one or more re–distillations of the distillate caught in the "receiving vessel" may be required in order to produce pure compounds. If the boiling temperatures are well separated, a single distillation may be sufficient to produce very pure products. In some cases, the liquid remaining in the distillation flask may be the desired liquid and the distillation is used to remove an unwanted solvent. Occasionally, a solid–in–liquid solution may be distilled to quickly separate the liquid from the solid, but liquid–in–liquid distillation is much more common.



DISTILLATION SETUP

## E. SOLVENT EXTRACTION

There are two ways in which this technique can be used.

### *Extraction of a solid from a MECHANICAL MIXTURE of solids*

This method uses a liquid that dissolves one or more of the solids present but leaves others undissolved. In the ideal case, only two solids will be present so that the desired solid is either

(i) left behind, or
(ii) dissolved and subsequently separated by simple evaporation of the solvent.

In "less–than–ideal" cases, different solvents may have to be used and numerous cycles involving extraction, evaporation of the solvent and re–extraction are required. If no suitable solvent can be found after extensive experimentation, the extraction procedure cannot be used.

***Example***: Add water to a sand/sugar mixture to extract the sugar and leave the sand behind.

### *Extraction of a dissolved liquid or solid from a LIQUID SOLUTION*

Before going further, two related special terms are needed.

***Definitions***: Two liquids are **MISCIBLE** if they are mutually soluble in each other in all proportions.

***Example***: Water and alcohol dissolve each other in all proportions and thus are MISCIBLE.

Two liquids are **IMMISCIBLE** if they are INSOLUBLE in each other.

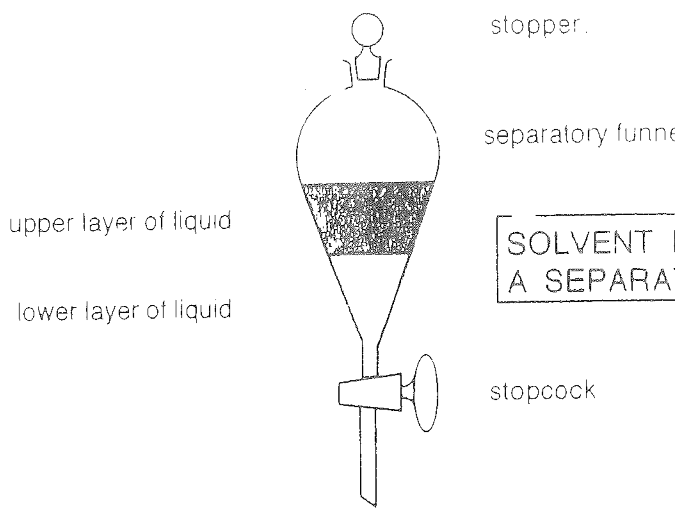
***Example***: Water and vegetable oil do not mix with each other and are IMMISCIBLE.

"Partial miscibility" situations also exist. For example, water and chloroform are partially soluble in each other. Most liquids mutually dissolve to at least a very small extent so that "IMMISCIBLE" implies that only tiny amounts of the liquids dissolve in each other.

To carry out solvent extraction on a solution with one or more solid and/or liquid substances dissolved in a liquid solution, a solvent must possesses TWO important experimentally–determined properties.

- the added solvent is IMMISCIBLE with the solvent already present.
- the added solvent dissolves one or more desired substances from the solution and leaves unwanted substances behind (or vice versa).

Depending on the relative densities of the solution and added solvent, the added solvent may form a layer above or below the original solution, as shown below.

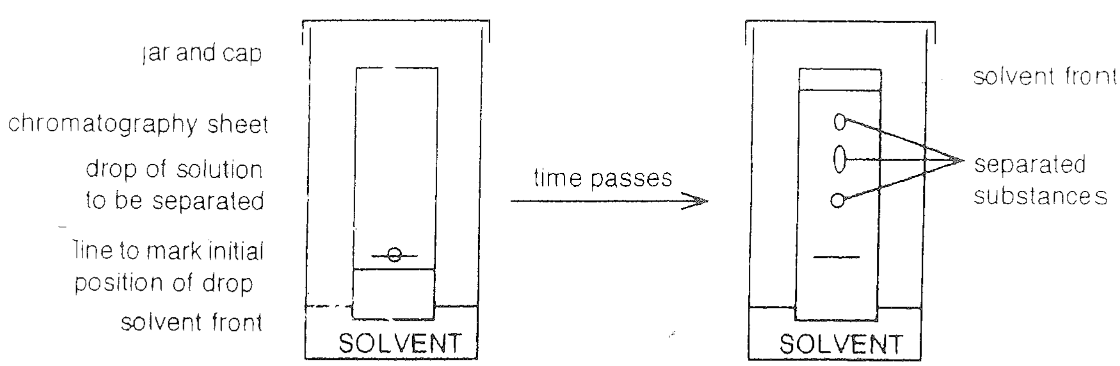



SOLVENT EXTRACTION USING A SEPARATORY FUNNEL

After a solvent is added to a solution, the mixture is shaken in a ***SEPARATORY FUNNEL***. Some substances are more soluble in the added solvent than they are in the original solvent and pass from the original solvent into the added solvent. The added solvent is then drained from the original solution and a second quantity of solvent is added to the solution. After being shaken, more of the *remaining* desired substances go into the added solvent and also can be drained.

## H. PAPER, COLUMN and THIN LAYER CHROMATOGRAPHY

Each of **PAPER, COLUMN** and **THIN LAYER CHROMATOGRAPHY** works similarly and is used to separate **small** amounts of **SOLID–IN–LIQUID SOLUTIONS** containing two or more dissolved solids which a coloured or can be reacted to form colours. ***Paper chromatography*** uses a sheet of absorbent paper and ***thin layer chromatography*** (TLC) uses a thin absorbent layer of dried silica gel on a sheet of glass or plastic. (Column chromatography is discussed below.) A drop of the solid–in–liquid mixture is put near one end of a chromatography sheet and allowed to dry. Another liquid, the "**developing solvent**", is allowed to absorb into the lower end of the chromatographic sheet containing the mixture and the liquid is absorbed up the sheet. A "***solvent front***" is seen as the liquid slowly moves upwards.




PAPER OR THIN LAYER CHROMATOGRAPHY

As the solvent is absorbed up the sheet, two opposing tendencies come into play: the dissolved solids tend to stay absorbed onto the sheet ***but*** the solids also tend to dissolve in the solvent. Chemicals which have a greater tendency to stick to the sheet and less tendency to dissolve in the solvent travel only a short distance up the sheet. Solids which have a greater tendency to dissolve in the solvent will travel almost as fast as the upward–flowing ***solvent front***. After drying the sheet, the areas containing the separated solids can be individually cut out (if using a paper sheet) or scraped off (if using a silica gel TLC sheet). Each individual solid can then be dissolved out of the paper or silica gel, and the solvent evaporated to leave the solid in its pure form.

## G. GRAVITY SEPARATION

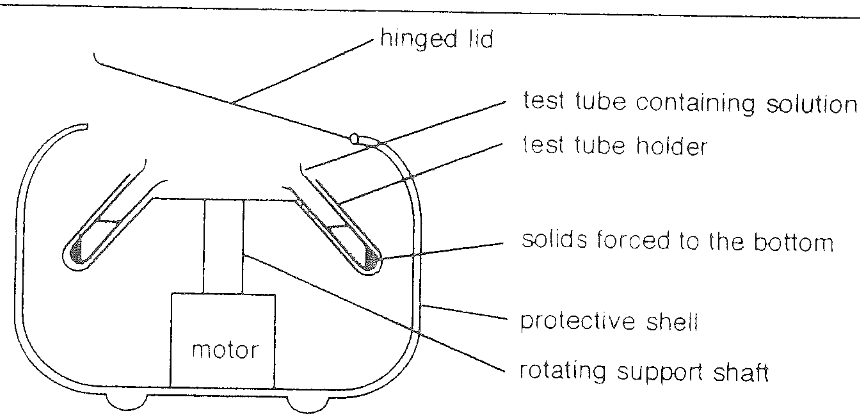
This method is used to separate desired solids from a MECHANICAL MIXTURE, based on their ***DENSITY***.

*Example:* Gold pans are used to trap dense particles of gold (density = 19.3 g/mL) and allow less dense rock and gravel (average density = 2–3 g/mL) to be removed. Gold in streams is often found in cracks in the underlying bedrock since it rapidly sinks under the lighter sand and gravel.

Mechanical shakers agitate a mixture of low and high density materials, allowing the high density substances to sink to the bottom and the low density substances to move to the top.

In the "froth flotation" process, finely pulverized ores are stirred into tubs of liquid through which fine air bubbles are blown. A "frothing agent" dissolved in the liquid produces a foam as the air passes through the stirred ore–liquid mixture. Rock particles rich in a desired ore are trapped in the froth, carried to the surface and removed by skimming off the foam. Rock particles having little or no ore content sink to the bottom and are eventually discarded.

Another gravity separation method involves the use of a ***CENTRIFUGE***, which whirls a test tube around at extremely high speeds forcing finely dispersed solids to the bottom of the test tube. (The closed end of the test tube faces away from the central rotating axis; the huge force arising as a result of the rotation pushes the solids to the bottom of the tube.)




CROSS–SECTION OF A CENTRIFUGE

Centrifuges are frequently used when a chemical reaction forms small amounts of solid particles in a solution and the solid must be separated. A solid formed in a liquid solution as a result of a chemical reaction is called a ***PRECIPITATE***.

**Note:** Both filtration and centrifuging can produce a separation with similar solid–in–liquid mechanical mixtures. However, filtration works best with relatively large volumes of liquid, whereas a centrifuge works best with volumes that can be held in a small test tube.

# A SYNOPSIS OF SEPARATION METHODS

Rather than attempting to give a "one–method–does–all" recipe, it is preferable to see what separation methods ("tools") can be used when you are confronted with particular situations.

## A. Mechanical Mixtures

| MIXTURE | METHOD | WHEN TO USE METHOD |
|---|---|---|
| SOLID in SOLID | Hand separation | Large chunks present among other solids |
| | Gravity separation | The density of the desired solids is much different from the density of the other solids |
| | Solvent extraction | One solid preferentially dissolves in a particular solvent |
| | Chromatography | The solids are coloured, present in small amounts and are soluble in some solvent or mixture of solvents |
| SOLID in LIQUID | Hand separation | A few large pieces of solid are present in the liquid |
| | Gravity separation | Solid particles are present in a ***small*** amount of liquid |
| | Filtration | Solid particles are present in a ***large*** amount of liquid |

## B. Solutions

| MIXTURE | METHOD | WHEN TO USE METHOD |
|---|---|---|
| SOLID in LIQUID | Evaporation | The solid is wanted and the liquid is not |
| | Distillation | The liquid is wanted; the solid may or may not be wanted |
| | Solvent extraction | An immiscible added solvent preferentially dissolves at least one but not all of the solids present |
| | Recrystallization | One dissolved solid is much less soluble than the others present (if any); the liquid is not wanted |
| | Chromatography | Small amounts of more than one coloured solid are present; the liquid present is not wanted |
| LIQUID in LIQUID | Distillation | Two or more liquids are present and have different boiling temperatures |
| | Solvent extraction | An immiscible added solvent preferentially dissolves at least one but not all of the liquids present |

***EXERCISES:***

45. A red–brown solution of bromine in water (density = 1.01 g/mL) is poured into a separatory funnel. Trichloroethane (density = 1.34 g/mL) is added and the mixture shaken thoroughly. Afterwards, two liquid layers are seen in the funnel: a clear layer of water and a reddish–orange layer of bromine in trichloroethane. Which layer will be on the top?

46. (a) If you wished to completely remove and save the liquid from a solid–in–liquid solution, which separation method(s) could be used?
    (b) If you wished to completely remove and ***not*** save the liquid from a solid–in–liquid solution, which separation method(s) could be used?

47. Which separation method(s) could be used to separate the following?
(a) two miscible liquids
(b) two immiscible liquids
(c) a flour–like solid floating around in water
(d) a mixture of three water–soluble dyes
(e) a mixture of sand, salt and water

48. Copper(II) sulphate, potassium nitrate and sodium chloride are solids which dissolve in water to about the same extent. If a mixture contains about 100 g of copper(II) sulphate, 0.5 g of potassium nitrat and 0.1 g of sodium chloride, which separation method(s) might be used to recover most of th copper(II) sulphate from the mixture?

OMIT

49. A single solvent extraction removes 90% of a desired chemical from a solution. What percentage of the chemical is ***left*** in the solution after two successive extractions?

50. A single solvent extraction removes 60% of a desired chemical from a solution. What percentage of the chemical would be ***removed*** from the solution after four successive extractions?

51. Why shouldn't the solvent completely evaporate in the recrystallization method of purification?

52. When an aqueous alum solution is left uncovered some of the water present evaporates, forming perfect alum crystals. The perfect crystals are scattered among many imperfect crystals. What separation technique(s) can be used to separate the perfect crystals from the rest of the mixture?

53. How can you separate all the components in a mixture containing sand, iron filings, water, gasoline, red water–soluble dye and blue water–soluble dye? In pure form the dyes are powders.

54. How can you separate a mixture of white sand (density = 2.2 g/mL), black sand (density = 5.2 g/mL), liquid methanol (MP = –94°C, BP = 65°C), and liquid hexanol (MP = –47°C, BP = 158°C)? Methanol and hexanol are miscible.

55. How can you separate a mixture of the three solids: potassium sulphate (MP = 1069°C, soluble in water, insoluble in alcohol), calcium carbonate (MP = greater than 1000°C, insoluble in water, insoluble in alcohol) and naphthalene (MP = 81°C, insoluble in water, soluble in alcohol)?

56. How can you separate a mixture of liquid chloroform (density = 1.48 g/mL, BP = 62°C, soluble in alcohol, insoluble in water), water (BP = 100°C), sugar (decomposes at 185°C, soluble in water, insoluble in alcohol and chloroform), powdered aluminum(insoluble in water, alcohol and chloroform) and liquid benzene (boils at 80°C, soluble in chloroform and alcohol, insoluble in water)? [Hint: what phases or layers will be observed in this mixture?]

57. You have a few milligrams of a mixture of powdered crystals containing green copper(II) chloride, pink cobalt(II) chloride and yellow iron(III) chloride. Suggest a method to separate the mixture.

58. How could you separate a mixture consisting of 500 kg of white sand (density = 2.2 g/mL), 50 kg of pennies (density = 8.96 g/mL), 10 kg of small nails (density = 7.86 g/mL) and 1 kg of fine platinum granules (density = 21.45 g/mL)? The sand and platinum granules are the same size.