Modern separation methods

science & technology in action
www.sta.ie
14th edition

Introduction

Separation techniques are the methods used to remove one substance from a mixture containing any number of other substances, for ***harvesting*** or ***identification***.

In the production of chemical substances, especially ***pharmaceuticals***, it is important to ensure that the products are pure and uncontaminated. If we want a particular reaction to occur we need to be sure that only the required ***reactants*** are present, otherwise unforeseen products may be formed and may change the properties of the substance we are trying to make. The un-purified ***raw materials*** are processed to the required purity so that they can be used as ***feedstock*** in a variety of pharmaceutical processes.

In almost all chemical reactions there will be more than one substance formed. The substance we want to produce is called the ***product*** and any other substances formed are called ***co-products*** or ***by-products***. These co-products and any unreacted substances must be removed before the product can be used for commercial purposes, for both safety and quality reasons. The degree of purity required varies depending on the use. If the substance is used in food or in medicines, then purity is of the highest importance.

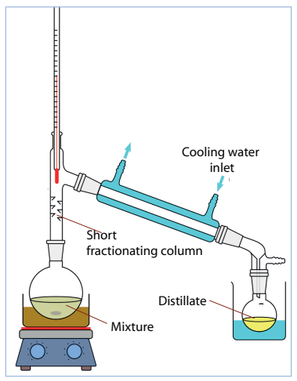
There are numerous ways to purify substances and the methods used depend on the physical and chemical properties of the substances involved. In most cases more than one method is needed to achieve the desired product purity. When a substance is separated, purified and collected for use, it is said to be ***harvested***.

Filtration

Filtration is probably the simplest method. It involves passing the mixture of an insoluble substance in a fluid (liquid or gas) through a mesh (e.g. filter paper), which will catch the solid particles and allow the fluid to pass through. The size of the pores in the mesh used will depend on the size of the particles to be removed. For example, a ***colander*** (or sieve) has large holes to separate peas from the water in which they were boiled. Filter paper has very small holes to trap sand articles, while ceramic filters have pores small enough to prevent bacteria getting through. The substance trapped by the filter is called the ***residue*** while the fluid that passes through is called the ***filtrate***. Either or both may be retained for further use.

Distillation

Sea salt is generally separated from seawater by ***evaporation***. The water is allowed to evaporate from shallow ponds called salt pans and the salt crystallises and drops out of solution. It is not very pure but this is often used as a selling point as it contains many beneficial elements.



Distillation differs from evaporation in that the liquid that has evaporated is collected by cooling its vapour in a condenser to convert it back to a liquid.

Fractional distillation is a variant of this in which a mixture of liquids that have different boiling points may be separated; e.g. petrol and diesel fuel from crude petroleum or whiskey from a mash (a weak alcohol solution made by fermenting barley).

Precipitation

Precipitation occurs when an ***insoluble*** solid is produced in a reaction. It has a number of variants, some of which are described below.

Chemical precipitation: This is done by adding a chemical that reacts with a ***soluble*** component in a mixture, causing it to become insoluble and sink to the bottom. This is used to remove phosphates and nitrates in the tertiary treatment of sewage; this is done to prevent ***eutrophication*** of ***watercourses*** into which the ***effluent*** is finally discharged.

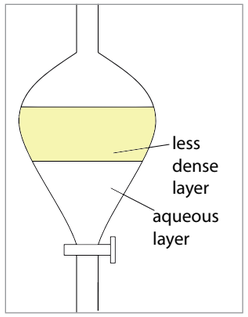
Flocculation is used in water treatment when a ***flocculating agent*** such as aluminium sulfate [$Al_2(SO_4)_3$] causes very small particles to clump together and either sink to the bottom or float to the top and so be more easily removed. Any remaining ***suspended*** particles are removed by filtration through sand.

Electrostatic precipitation is used in industry to remove dust or smoke particles from gases that are discharged into the atmosphere. Electrically charged plates induce a charge onto dust particles which are then attracted to the oppositely charged surface and removed from the air. A similar effect can be seen on older television screens which attracted dust due to their electrostatic charge.

Recrystallisation

This is a method of harvesting and purifying soluble substances such as benzoic acid. Benzoic acid is first crystallised by cooling in ice after it is formed. It is then filtered to remove any unreacted material and coproducts. It is soluble in hot water but insoluble in cold water. The residue is dissolved in a minimum of hot water and filtered using a hot filter apparatus to remove any insoluble impurities. A hot filter a used to stop the solution cooling which could cause some of the benzoic acid to form crystals; thus reducing the yield. The filtrate is then cooled and most of the benzoic acid crystallises out and any soluble impurities are left in the solution. The crystals are then filtered and washed with a minimum of cold ***deionised*** water before being dried.

Solvent extraction



Non-polar organic compounds are soluble in organic solvents such as ***cyclohexane*** but not in water. When ***clove oil*** is extracted by steam distillation the result is an ***emulsion*** of the ***eugenol*** in water. When the emulsion is shaken with cyclohexane the eugenol dissolves in the cyclohexane. The cyclohexane then floats on top of the water. With the aid of a separating funnel the water is drained off leaving the solution of eugenol in cyclohexane. The cyclohexane is then evaporated to leave the pure eugenol.

Extracting DNA

Electrophoresis can be used for harvesting but it is more frequently used for identification purposes. In ***genetic profiling*** it separates DNA strands based on their different interactions with a gel under an electric potential (voltage). Restriction enzymes cut DNA at specific base sequences and the resulting strands are then separated according to their lengths. The pattern of bands is unique to the individual (or species) and is often referred to as a genetic fingerprint. It is widely used in ***forensics*** to show that someone was at the scene of a crime. It is also used to determine family relationships, to trace ancestors or identify bodily remains after a disaster.

Chromatography

This is a common and important method of separating a mixture of components in the pharmaceutical industry. The mixture of components is dissolved in a carrier fluid called the ***mobile phase***. This is then passed through another material called the ***stationary phase***. The separation of the different components is based on the fact that they travel at different speeds as they interact with the stationary phase. For example, the individual dyes in some felt markers may be separated by marking a dot on a strip of filter paper and allowing water to soak up through it. The water carries different dyes at different rates along the paper.

Chromatography is used for both identification and harvesting purposes. The stationary phase may be a solid, or a liquid coating on a solid, while the mobile phase may be a liquid or a gas. Today there is a great variety of chromatographic methods including paper, thin layer, column, HPLC and gas chromatography.

HPLC

High Performance Liquid Chromatography (HPLC) is an analytical separation technique that is used to separate and analyse ***non-volatile*** or ***thermally-unstable*** compounds. HPLC is a method that involves the separation of unknown sample components by pumping the liquid mobile phase through a column, under pressure, that contains stationary phase particles.

A HPLC instrument comprises of a reservoir (holding the mobile phase), an injector (to add the unknown sample), a column (the stationary phase), a detector (to detect the quantity of the individual components of the unknown sample in the form of a signal) and a computer (to process the signal and collect the data). There are lots of different types of detectors available such as: ***UV/VIS*** (ultraviolet/visible), fluorescence, electrochemical, mass spectrometer etc. The type of detector used depends on the sample being tested. HPLC analysis can be used in many different fields such as pharmaceutical, the food industry, cosmetics, environmental laboratories, forensic laboratories etc.

GC

Gas Chromatography (GC) is an analytical separation technique that is used to separate the components of ***volatile*** compounds. In a GC Instrument the mobile phase is a gas and the stationary phase is a nonvolatile liquid that is coated on an inert solid. A gas chromatograph consists of a carrier gas (usually helium, nitrogen or hydrogen), a column, an oven (to control the temperature) and a detector. The unknown sample is injected into the instrument and is carried through the column by the carrier gas. As the unknown sample moves through the column, it separates out into its components. Each component of the unknown sample emerges from the end of the column and moves past the detector, which identifies the sample, and produces a series of peaks. The peaks correspond to all the different components in the unknown sample. There are lots of different types of detectors available such as: ***flame ionization detector (FID)***, ***thermal conductivity detector (TCD)*** mass spectrometer etc. The type of detector used depends on the sample being tested.

Mass spectrometer

The mass spectrometer was invented by Francis Aston to detect and separate isotopes of neon. It works on the principle that ions can be separated by mass while moving in a magnetic field. Compounds are ionised and this produces many fragments whose size, mass and abundance is characteristic of the compound being ionised. The mass spectrograph can identify each of the compounds present in a mixture.

Find this and other lessons on www.sta.ie

MSD INVENTING FOR LIFE

SSPC SYNTHESIS AND SOLID STATE PHARMACEUTICAL CENTRE Innovation Through Collaboration

Modern separation methods

science & technology in action 14th edition

www.sta.ie

Syllabus References

The main syllabus references for the lesson are:

Leaving Certificate Chemistry

Chromatography and Instrumentation in Organic Chemistry

- Chromatography as a separation technique.
- Use of thin-layer chromatography (TLC) in the separation of dyes taken from fibres in forensic work. GC and HPLC as more advanced separation techniques. (pp. 22 & 59)
- Instrumental methods of separation or analysis, or both: Mass spectrometry (p. 7). Gas chromatography (GC). High-performance liquid chromatography (HPLC). (p. 23)

Option 1A: Additional Industrial Chemistry

- Characteristics of effective and successful industrial chemical processes, such as: (i) feedstock, (ii) rate, (iii) product yield, (iv) co-products (separation, disposal or sale) p. 27

Science and Technology in Action is also widely used by Transition Year classes.

Learning Outcomes

On completion of this lesson, students should be able to:

- Explain why obtaining pure substances is particularly important in medicines and food products
- Explain the difference between raw materials, feedstock, products and co-products
- Understand in broad terms how electrophoresis works
- Distinguish between solute, solvent, filtrate and residue
- State the principles by which chromatography works
- Describe a number of separation techniques and what they are used to separate.

General Learning Points

These are additional relevant points which are used to extend knowledge and facilitate discussion.

- Purifying substances usually involves a number of different techniques. The particular technique used depends on the properties of the substance.
- Purification can also be used to identify substance and/or to harvest (collect) them.
- The degree of purification depends on the proposed use of the product.
- Almost all chemical reactions produce co-products.
- Separation can involve chemical, physical, electromagnetic and electrostatic forces.
- In order to obtain pure products it is necessary to start with pure reactants.

Student Activities

1. Steam distillation is used to extract essential oils from a variety of plants. How many can you discover? Can you explain why it is used and how it works?
2. The mass spectrometer is widely used in forensic science. Describe some of these uses.
3. The different smells of oranges and lemons are caused by optical isomers. Find the structures of these and other optical isomers.
4. Describe the processes involved in treating drinking water to make it safe for human consumption.
5. Desalination of seawater can be done by evaporation followed by condensation but is can also be carried out using reverse osmosis. Explain how and where this is done.
6. Gas chromatography has many uses, as does High Performance Liquid Chromatography. Write notes on how either of these techniques works and what it are used for.
7. What is destructive distillation? Find examples of some of the substances that were discovered using destructive distillation.
8. Find out the various fractions produced from crude petroleum and their uses. Most of these are long chain hydrocarbons and are not in great demand. How can they be converted to the more wanted short chain hydrocarbons?
9. Investigate how the mass spectrometer works in as much detail as you can understand.
10. Sewage treatment involves purification of biological waste to make it safe and prevent eutrophication. Make a presentation or a poster to describe the different stages of the process.

True/False Questions

a) To obtain a pure substance it is normal to use more than one method of separation. **T F**

b) Eutrophication is a method of separating fertilisers from water. **T F**

c) In electrophoresis a potential (voltage) is used to separate the substances. **T F**

d) The liquid that passes through a filter is called the residue. **T F**

e) Fractional distillation depends on the differing solubility of the components. **T F**

f) Francis Aston invented the mass spectrometer. **T F**

g) Petrol does not dissolve in water because petrol is non-polar while water is polar. **T F**

h) Recrystallization could be used to remove salt which had been added to sugar. **T F**

i) A magnet could be used to purify a sample of sulfur contaminated by iron. **T F**

j) Raw materials are purified feedstock. **T F**

Check your answers to these questions on www.sta.ie.

Examination Questions

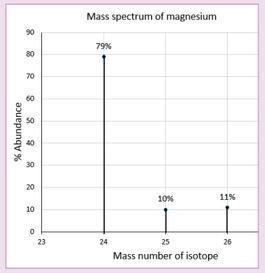
Leaving Certificate Chemistry (HL) 2018, Q. 5

(a) Define (i) mass number of an atom, (ii) relative atomic mass of an element.

(b) A sample of magnesium metal was introduced into a mass spectrometer and vaporised. What were the next three fundamental processes that occurred in the mass spectrometer?

(c) The mass spectrum of the sample, in the picture, shows that magnesium has three naturally occurring isotopes.

Use the data given to calculate the relative atomic mass of magnesium correct to two decimal places.



Leaving Certificate Chemistry (OL) 2017, Q. 4 j

Name the separation technique in which a mobile phase carrying a mixture is caused to move in contact with a selectively adsorbent stationary phase. The mixture may contain coloured or colourless components.

Leaving Certificate Chemistry (OL) 2014, Q. 11 a

Consider the following six terms: refluxing, fractionation, recrystallisation, distillation, chromatography, steam distillation. Match the terms above with their corresponding descriptions (A to F) in the table below.

A	Separation of the components of a mixture using a solvent as the mobile phase and a suitable solid as the stationary phase
B	Separation of two liquids on the basis of their different boiling points
C	Dissolving impure crystals in the minimum of hot solvent and then filtering the mixture after allowing it to cool
D	Separation of several substances in crude oil on the basis of difference in boiling points
E	Boiling a reaction mixture without loss of material, thus giving time for reaction to occur

Leaving Certificate Chemistry (HL) 2008, Q. 2 a, b

Chromatography is widely used in chemistry as a separation technique.

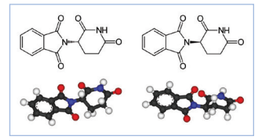
(a) Describe how you would set up and carry out an experiment to separate the components in a mixture of indicators using paper chromatography, thin-layer chromatography or column chromatography.

(b) Explain why the different components of the mixture travel different distances along the paper or along the thin-layer or through the column in a given time.

Did You Know?

Perhaps the most famous case involving a lack of purity of pharmaceuticals was the Thalidomide scandal of the 1960s. Thalidomide was a drug given to pregnant women to counteract morning sickness. It was soon discovered that the children of women taking the drug had a high incidence of major birth defects, many being born without limbs.

It was eventually discovered that Thalidomide exists in two mirror-image forms: it is a mixture of (R)- and (S)-enantiomers. This may seem a rather insignificant difference but it is of major importance. The (R)-enantiomer, has sedative effects, whereas the (S)-isomer is teratogenic, i.e. disturbs the development of the foetus and causes deformities. Under biological conditions, the isomers interconvert, so separating the isomers before use is actually ineffective.



Biographical Note

Francis W Aston (1877 to 1945)

Aston was born in Birmingham England and first trained as a chemist investigating fermentation chemistry in the Birmingham School of Brewing. He was later appointed as a lecturer in the University of Birmingham but the discovery of X-rays and radioactivity drew him more and more into physics. He was invited by J J Thompson (the discoverer of electrons and author of the 'plum pudding' model of the atom), to join him in the Cavendish Laboratory in Cambridge.

He was awarded the Nobel Prize for Chemistry in 1922 for his discovery of a large number of isotopes using the mass spectrometer which he invented. This instrument separates ions of different mass by their deflection in a magnetic field. Heavier ions are deflected less than lighter ones. He discovered that neon has two isotopes and the spectrometer enabled him to work out both their mass and their relative abundance. He discovered in the course of his work 212 of the 287 naturally occurring isotopes of the non-radioactive elements.

Revise The Terms

Can you recall the meaning of the following terms? Revising terminology is a powerful aid to recall and retention.

by-products, clove oil, colander, co-products, cyclohexane, deionised, distillation, effluent, electrophoresis, emulsion, eugenol, eutrophication, evaporation, feedstock, filtrate, flame ionization detector (FID), flocculating agent, flocculation, forensics, fractional distillation, gas chromatography (GC), genetic profiling, harvested, harvesting, high performance liquid chromatography (HPLC), identification, insoluble, mobile phase, non-polar, non-volatile, pharmaceuticals, precipitation, product, raw materials, reactants, residue, soluble, stationary phase, suspended, thermal conductivity detector (TCD), thermally-unstable, UV/VIS, volatile, watercourses

Check the Glossary of terms for this lesson on www.sta.ie