Name:

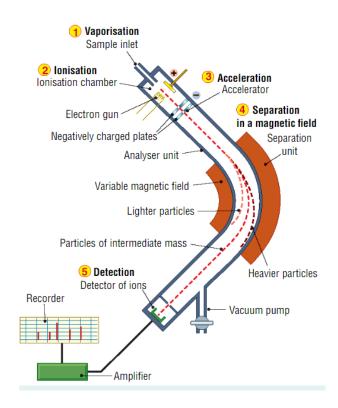
| Instrumentation | Objectives  |
|-----------------|---|
|                 | -describe how a mass spectrometer can be used to determine relative atomic mass                                   |
|                 | -describe the principles on which the Mass Spectrometer is based  |
|                 | -explain the fundamental processes that occur in a mass spectrometer  |
|                 | -describe chromatography as a separation technique in which a mobile phase carrying a                             |
|                 | mixture is caused to move in contact with a selectively absorbent stationary phase                                |
|                 | -separate a mixture of indicators using paper chromatography or thin-layer  |
|                 | chromatography or column chromatography   |
|                 | -describe the use of thin-layer chromatography (TLC) in the separation of dyes taken from fibres in forensic work |
|                 | -describe GC and HPLC as more advanced separation techniques  |
|                 | -give examples of instrumental methods of separation or analysis referring  |
|                 | briefly to the principles involved in each case for the following:  |
|                 | mass spectrometry- analysis of gases from a waste dump, trace organic pollutants in water                         |
|                 | • gas chromatography (GC)- drug tests on athletes, blood alcohol tests  |
|                 | high-performance liquid chromatography (HPLC)- growth-promoters in meat,     vitamins in foods                    |
|                 | • infra-red absorption spectrometry (IR)- identification of organic compounds, e.g.                               |
|                 | plastics and drugs  |
|                 | <ul> <li>ultraviolet absorption spectrometry- quantitative determination of organic</li> </ul>                    |
|                 | compounds (e.g. drug metabolites, plant pigments)   |
|                 | -Brief reference to the principles of each method. interpretation of spectra etc. not                             |
|                 | required (It should be noted that these techniques are applicable not only to organic                             |
|                 | chemistry but also to many other areas of chemistry)  |
|                 |   |

## **MASS SPECTROMETRY:**

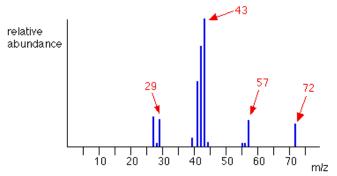
*Def*<sup>\*</sup>: The **Principle of Mass Spectrometry** is that charged particles moving in a magnetic field are deflected by different amount due to their masses. This separates the particles according to their masses.

## Stages in Mass Spectrometry: (Need to learn the names)

- 1. **Vaporisation**: The sample material is vaporised into a gas.
- 2. **Ionisation:** An electron gun fires high-energy electrons at the gaseous sample. This knocks electrons off the sample particles, leaving the sample as a group of positively charged ions.
- 3. **Acceleration:** A negatively charged plate attracts the positive ions. This accelerates the particles so that they travel at high speed through the spectrometer.
- 4. **Separation:** A magnetic field of a particular strength is used to deflect the particles. Particles that are too light are deflected too much and hit the side of the spectrometer. Particles that are too heavy are not deflected enough and hit the side of the spectrometer. Only particles that have a certain mass are deflected by just the right amount and make it through the spectrometer to the detector.
- 5. **Detection:** A detector senses the number of positive ions hitting it and displays the result on a mass spectrum.



simplified mass spectrum of pentane - CH3CH2CH2CH2CH3



The peaks shown in the mass spectrum have units of mass on the horizontal axis. The height of each peak represents the relative abundance of particles of that mass. E.g. the peak at 43 is roughly 4x higher than the peak at 72. This means there are 4x as many particles of mass 43 as there are of mass 72.

**Uses:** Analysis of gases in waste dumps, identification of organic pollutants in water, extraterristrial rock composition.

#### **CHROMATOGRAPHY:**

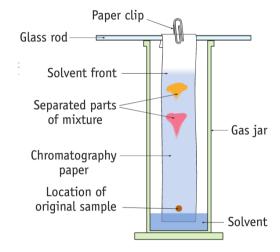
*Def*<sup>n</sup>: **Chromatography** is a separation technique in which a mobile phase carrying a mixture moves in contact with a selectively adsorbant stationary phase.

Paper Chromatography: (need to know this in detail, with diagram. TLC and column chromatography don't need as much detail, or a diagram)

<u>Stationary Phase:</u> Chromatography paper <u>Mobile Phase:</u> Water (or other solvent)

- 1. Spot of sample mixture is placed around 1cm above the water line.
- 2. The mobile phase soaks up through the stationary phase and dissolves the materials in the mixture.
- 3. Each material dissolves to a different extent.
- 4. Less soluble materials will appear as a spot closer to the bottom of the paper.
- 5. More soluble materials will appear as a spot higher up the paper.
- 6. This separates the components in the mixture.

**Uses:** Separating dyes in ink/food colouring/indicators.



# Thin Layer Chromatrography (TLC):

Stationary Phase: Silica Gel Plates Mobile Phase: Methanol (or other organic solvent)

**Uses:** Test drug purity, Separate colours in clothes dyes in forensic science.

### **Column Chromatography:**

Stationary Phase: Silica Gel in glass tube

Mobile Phase: Water/Ethanol (or other appropriate solvent)

Uses: Separate dyes in food colouring.

Passing a solvent through a tube/column is called *elution*. The word for the solvent used is the *eluent*.

#### Gas Chromatography (GC):

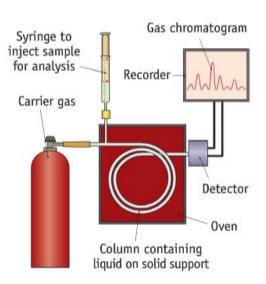
Stationary Phase: Long coiled column filled with coated silica gel.

*Def*<sup>n</sup>: The **Principle of Gas Chromatography** is that a mixture of components is carried by a gaseous mobile phase is separated based on their different interactions with a solid stationary phase and the gaseous mobile phase.

**Uses:** Alcohol levels in urine samples, drug testing in athletes.

**GC-MS:** Gas Chromatography is often used with Mass Spectroscopy. After separation by GC, each component is put through a Mass Spectrometer to identify each component.

Mobile Phase: Inert carrier gas (N<sub>2</sub>)

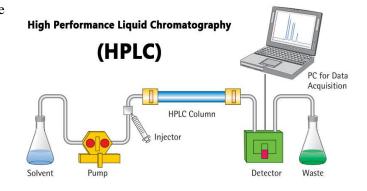


#### High Performance Liquid Chromatography (HPLC):

Stationary Phase: Coated Silica Gel Mobile Phase: Suitable liquid solvent (ethanol) under high pressure

The pump supplies the high pressure needed for the mobile phase. The reason the mobile phase needs high pressure is that the column is packed very tightly and the elution would take too long if the pump wasn't used.

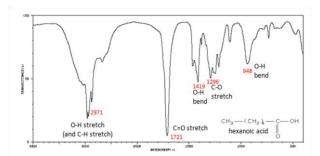
**Uses:** Test for growth hormones/additives/vitamins in food, caffeine in soft drinks etc.



#### **SPECTROSCOPY:**

## **Infra-red Spectroscopy (IR):**

Organic compounds absorb infrared-radiation. Different frequencies of IR light are absorbed by different types of bonds. So a C=O would absorb a different frequency of IR then an O-H or a C=C bond. By looking at how an organic compound absorbs IR light, we get an IR spectrum, which is used as a "fingerprint" for that compound.



Uses: Identifying plastics, illegal drugs. Used to back up breathalyser results.

# **Ultraviolet Spectroscopy (UV):**

This instrument measures how a compounds absorbs UV light of different wavelengths/frequencies. Like IR spectroscopy, this also results in a "fingerprint" for each compound. However, unlike IR, UV spectroscopy can also measure the *concentration* of the compound, making this a *quantitative* analysis.

UV spectroscopy is commonly used with HPLC in order to identify each compound in a mixture, and their concentrations.

**Uses:** Drug concentrations in blood/cells, plant pigments in plant cells etc.

