

ANALYTICAL TOOL – 4

SPECTROSCOPY

(QUALITATIVE & QUANTITATIVE TECHNIQUES)

The use of light or **electromagnetic radiation** for analysis is called **spectroscopy**.

Different spectroscopic techniques utilise different parts of the electromagnetic spectrum to provide us with information about the structure, composition and amounts of substances.

ELECTROMAGNETIC RADIATION

Electromagnetic radiation is a form of energy that consists of electric and magnetic fields that travel at the speed of light.

Examples of electromagnetic radiation include light, radio waves and x-rays.

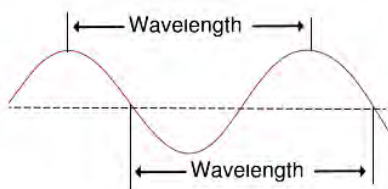
A single unit of electromagnetic radiation is called a **photon** and consists of a mass-less particle travelling in a wave-like motion.

Each photon contains a certain amount (or **quantum**) of energy, and all electromagnetic radiation consists of these photons. The only difference between the various types of electromagnetic radiation is the amount of energy found in the photons.

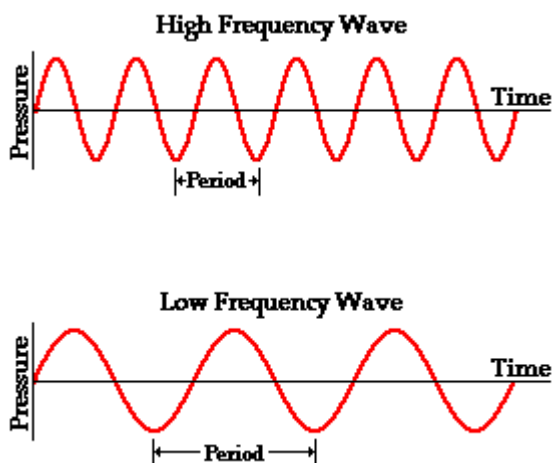
PROPERTIES OF ELECTROMAGNETIC RADIATION

As electromagnetic radiation behaves as a wave travelling at the speed of light, it is described in terms of its wavelength and frequency.

The **wavelength** (λ m) describes the distance between any two consecutive identical points on the wave.



The **frequency** (hertz, Hz) of a wave is the number of complete cycles of the wave that pass a given point in a second.



Frequency (ν) and wavelength (λ) are inversely proportional where $\nu = \frac{c}{\lambda}$ and c represents the speed of light.

The **energy** of a photon (E) is described by the rule: $\Delta E = h\nu = \frac{hc}{\lambda}$ where:

- E represents the energy
- λ represents the wavelength
- c represents the speed of light ($3 \times 10^8 \text{ m/s}$)
- h represents Planck's constant $6.63 \times 10^{-34} \text{ Js}$

Therefore, the energy of a photon (E) is inversely proportional to the wavelength i.e.

$E \propto \frac{1}{\lambda}$. The shorter the wavelength the greater the energy.

THE ELECTROMAGNETIC SPECTRUM

The electromagnetic (EM) spectrum is the range of all possible electromagnetic radiation frequencies.

Radiation from each portion of the electromagnetic spectrum has a specific frequency, wavelength and energy associated with it.

If the energy corresponds with a wavelength of light within the visible spectrum, electromagnetic radiation will appear as coloured light. (**Note:** Different colours of light consist of different energies or wavelengths).

Parameter	Radiation type						
	Gamma rays (γ)	X-rays	Ultraviolet (UV)	Visible	Infrared (IR)	Microwaves	Radiowaves
Wavelength/m	10^{-12}	10^{-10}	10^{-8}		10^{-4}	10^{-2}	10
Frequency/Hz	10^{20}	10^{18}	10^{16}		10^{12}	10^{10}	10^6
Photon energy/ kJ mol ⁻¹	10^8	10^6	10^4		1	10^{-2}	10^{-6}

Colour	Wavelength/nm
violet	420–450
dark blue	400–420
blue–green	450–490
green	490–530
yellow–green	530–545
yellow	545–580
orange	580–630
red	630–720

7×10^{14} Hz ← ————— → 4×10^{14} Hz
 ← ————— → Increasing frequency
 ← ————— → Increasing photon energy
 ← ————— → Increasing wavelength/nm

QUESTION 39

The properties of ultraviolet light are best summarised as:

- A Short wave radiation with low energy.
- B Short wave radiation with high energy.
- C Long wave radiation with low energy.
- D Long wave radiation with high energy.

QUESTION 40

The properties of radio waves are best summarised as:

- A Short wave radiation with low energy.
- B Short wave radiation with high energy.
- C Long wave radiation with low energy.
- D Long wave radiation with high energy.

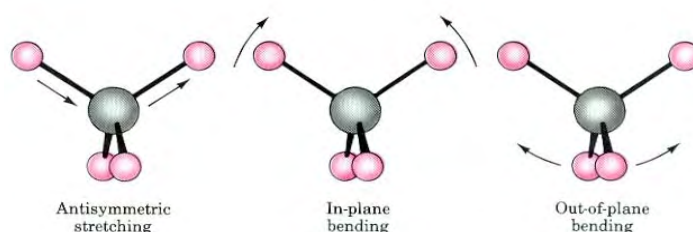
USING RADIATION IN SPECTROSCOPY

Atoms and molecules absorb and emit specific wavelengths of electromagnetic radiation.

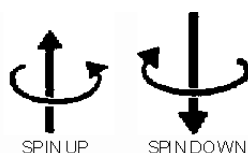
This radiation interacts with atoms and molecules in different ways. Which part of an atom or molecule is affected and what changes occur simply depends upon the wavelengths or energies employed.

For example:

- When an atom absorbs visible or ultraviolet radiation, its electrons move to higher energy levels.
- When a molecule absorbs infrared radiation, the molecules themselves move to higher energy levels by causing changes to the covalent bond in molecules. Depending upon the wavelength of infra-red radiation, covalent bonds can stretch, rock, bend or twist like a spring.



- When nuclei absorb radio wave radiation of the correct wavelength/energy they can change the direction of their spin, moving to a higher nuclear spin energy levels.



In Summary:

The different parts of the electromagnetic spectrum affect different parts of an atom or molecule and in different ways.

SUMMARY OF SPECTROSCOPIC TECHNIQUES

Technique	Part of Spectrum Used	Part of Structure Affected by Supplied Radiation
Flame Tests	Visible	Valence electrons in metal atoms
Atomic Emission Spectroscopy (AES)	Visible	Valence electrons in metal atoms
Atomic Absorption Spectroscopy (AAS)	Ultraviolet and Visible	Valence electrons in metal atoms
Colorimetry	Visible	Valence electrons in molecules
UV-Visible Spectroscopy	Ultraviolet and Visible	Electrons in molecules
Infrared Spectroscopy (IR)	Infrared	Bending and stretching of bonds in molecules
Nuclear Magnetic Resonance Spectroscopy (NMR)	Radio Waves	Nuclear spin states (nucleons in molecules)

Note:

AES and Colorimetry are not specifically listed in the VCAA Study Design. However, as the principals relating to these techniques can be examined in application style questions, these topics will be addressed in the Master Classes.

TYPES OF SPECTROSCOPIC ANALYSES

The effects of radiation on atoms and molecules can be used in spectroscopy to provide us with information about the structure, bonding and composition of substances, as well as how much of a particular atom or molecule is present.

There are two main ways in which radiation can be employed in spectroscopic analyses.

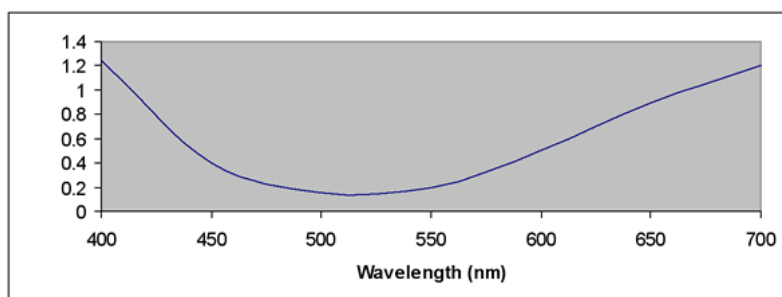
- **We can analyse the energies emitted by a substance.**

Examples include: Flame Tests and Atomic Emission Spectroscopy

- **We can analyse the energies absorbed by a substance.**

Qualitative

One approach involves the measurement of absorption as a function of wavelength to produce a spectrum of the sample that is being analysed. This approach is often used to identify a substance eg. Infrared and UV-Visible spectroscopy.



Quantitative

A second approach involves the measurement of how much energy of a specific wavelength is absorbed by a substance. This approach is often used to determine the amount of a substance.

If a substance is coloured, we can shine light of a complementary colour through the solution and measure how much of that light has been absorbed (colorimetry).

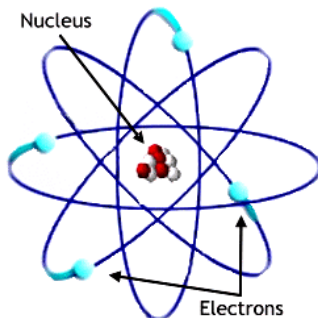
Alternatively, we can choose a wavelength from a substance's spectrum that is known to be exclusively absorbed by that substance and measure how much of that radiation is absorbed. This method is highly specific and may be used to determine the concentration of substances in complex mixtures, without interference from other sample components.

Techniques utilising this approach include atomic absorption spectroscopy (AAS) and UV- Visible spectroscopy.

Note:

A spectrophotometer (**spectrometer**) measures the amount of radiation that is absorbed by a chemical species at a specific wavelength or energy.

THE EFFECTS OF RADIATION ON ATOMS



- An atom consists of a dense positively charged nucleus containing protons and neutrons (collectively referred to as nucleons).
- The electrons move in discrete regions of space (**orbitals**) around the nucleus.
- Each orbital within an atom has a specific energy level.
- Although the pattern of energy levels is the same from atom to atom and element to element i.e. $1s$, $2s$, $2p$, $3s$ etc, their precise energy values are different. Therefore, each orbital within an atom has a specific energy level.
- All the atoms of any given element have exactly the same set of energy levels eg. Each atom of lead has the same set of energy levels as every other atom of lead. Each atom of sulfur has that same set of energy levels as every other atom of sulfur.

However, no two different elements have the same set of energy levels i.e. each element has its own unique characteristic set.

- When an electron occupies a particular orbital, it will assume the energy that is determined by that level. Orbitals that are located closer to the nucleus have lower energy levels than those that are located further away.
- As electrons closer to the nucleus experience strong electrostatic interactions with the nucleus, moving from a low energy level to a high energy level requires energy.
- Conversely, movement to a lower energy involves the release of energy.

When all of the electrons are at the lowest possible energy level they are said to be in the **ground state**. Electrons do not always stay in the ground state - they can be promoted to higher-energy levels further from the nucleus.

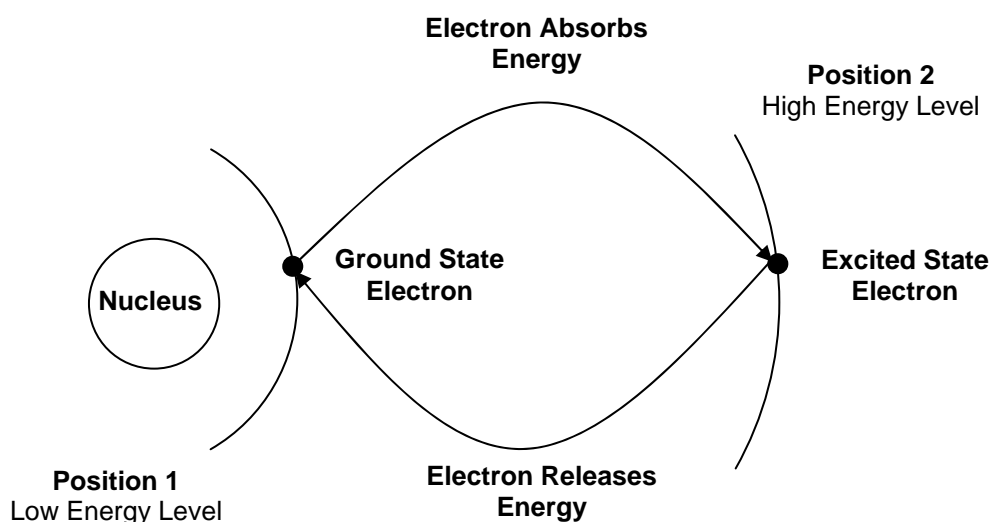
When an **atom** is excited by light, its **valence electrons** can absorb a photon of just the right amount of energy to move to a higher energy level or orbital. When an electron is in a higher-energy shell it is said to be in an **excited state**.

Atoms may also be excited by electricity or heat. In this case, their electrons can gain energy from the heat to move them to an excited state.

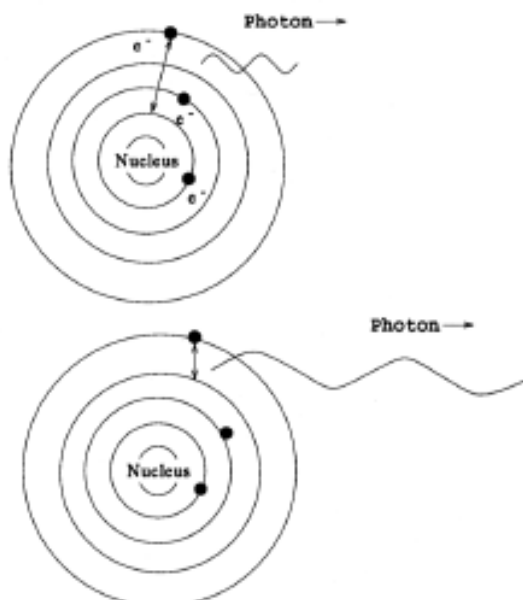
Note: The absorbed energy is used to overcome the forces of attraction between the valence electrons and the positively charged nucleus.

The amount of energy that is absorbed by the electrons corresponds to the difference between the energy levels of the orbitals.

Depending on how much energy is absorbed, the electron may move up one, two, a few, or many energy levels. If the energy is great enough, the electron may go flying out of the atom altogether (**ionisation**).

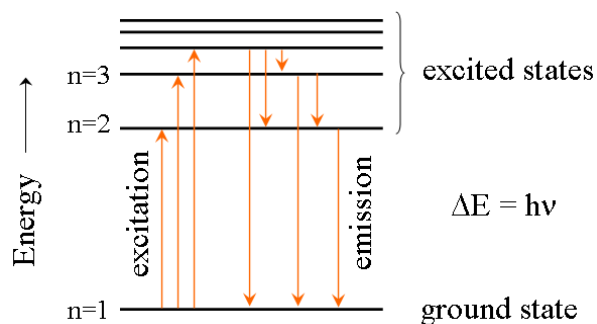


When an electron is positioned in a higher energy level it exists in an **unstable state**. These electrons can jump back to lower intermediate energy levels or their ground states, releasing a photon of light in the process. Different energy transitions result in the emission of different wavelengths of radiation.



For example:

For the simple atom shown below, 6 different energy transitions would be observed as excited electrons return to lower energy levels.



These changes in energy may be measured during spectroscopy to give us qualitative and/or quantitative information regarding the substance being analysed.

Note:

- If the quanta of energy released as electrons return to lower energy levels is proportional to a wavelength of light in the visible spectrum, the emitted radiation is observed as coloured light.
- Radiation emitted from other sections of the electromagnetic spectrum can be detected by techniques such as UV-Visible Spectroscopy and IR spectroscopy.

QUESTION 41

What factors determine the quanta of energy released when an electron moves from the excited state to the ground state?

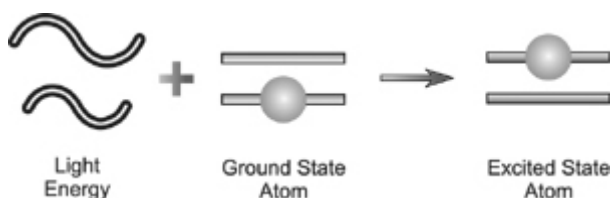
Solution**QUESTION 42**

Are the energy levels of a ground state atom the same as that for the corresponding ion?

Solution

QUANTITATIVE SPECTROSCOPIC ANALYSES

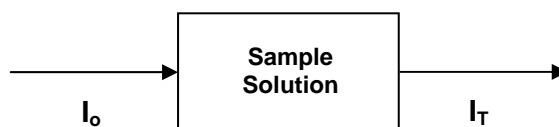
- A **spectrometer** measures the amount of radiation (light) that is absorbed by chemical species.
- Radiation of a **specific frequency** is passed through a sample of the chemical species and some of this radiation is absorbed by the ground state electrons in the atoms or molecules being analysed.



- The radiation **not absorbed** by the chemical species being analysed reaches a detector.

The detector measures the intensity of radiation that passes through the sample (i.e. the amount that is not absorbed).

Absorbance = Initial Radiation Intensity - Final Radiation Intensity



I_o = Intensity of incident radiation

I_T = Intensity of transmitted radiation

Note:

The amount of radiation absorbed is directly proportional to the concentration of that component in the solution.

i.e. The greater the concentration of the component being analysed, the higher the absorbance reading.

Absorbance \propto Concentration

- By comparing the amount of light absorbed by a solution of unknown concentration with the absorbance of solutions of known concentrations, the concentration of an unknown sample may be determined.

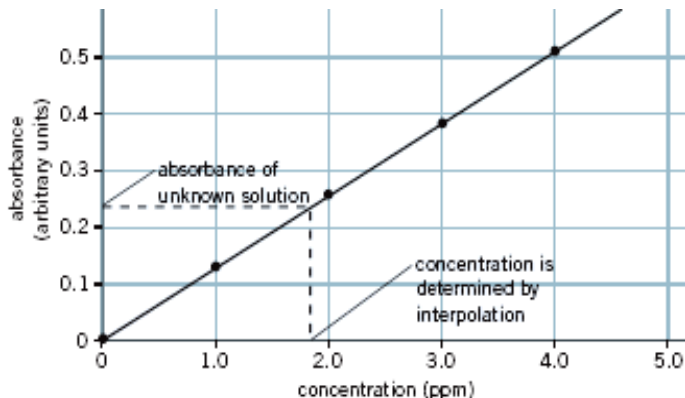
STANDARD CURVES/CALIBRATION CURVES

At low concentrations, the amount of radiation absorbed by a substance is directly proportional to the concentration of that substance in solution.

This pattern enables us to construct graphs that can be used to determine the amount of substances in samples of unknown concentration.

METHOD:

- Step 1:** Dilute the sample so that it is able to transmit some light/radiation.
- Step 2:** Select an appropriate wavelength of radiation for the analysis.
- Step 3:** Measure the amount of radiation absorbed by the sample.
- Step 4:** Measure the amount of radiation that is absorbed by known concentrations of the component being analysed under the same conditions as that employed for the sample.
- Step 5:** Plot the absorbance of each standard solution against its concentration. This curve is referred to as a **standard curve** or **calibration curve**.
- Step 6:** Use the calibration/standard curve to determine the concentration of the sample. Remember to take any dilutions performed while preparing the sample under consideration.



Note:

- At higher levels of concentration, the absorbance-concentration relationship may become non linear.
- Standards are chosen so that a linear plot is obtained.
- If plotted points are slightly scattered, we draw the line of best fit.

A line of best fit by eye is drawn through the scatterplot so that an equal number of points lie on either side of the line and/or the sum of the distances of the points above the line are roughly equal to the sum of the distances below the line.

- The concentration of the unknown solution should fall on the calibration curve.

If the absorbance reading obtained is too high, dilute the sample prior to further testing. Do not extrapolate data at high concentrations.

At low concentrations, the relationship between concentration and absorbance is constant and predictable, therefore, extrapolation can be performed.

- The spectrometer must be calibrated each time a sample is analysed using known solutions that have been prepared and measured under identical conditions to that used for the unknown(s).

Examples of spectroscopic techniques that utilise calibration/standard curves for quantitative analyses include:

- Colorimetry
- Atomic Absorption Spectroscopy
- UV-Visible Spectroscopy

ATOMIC ABSORPTION SPECTROSCOPY (A QUANTITATIVE TECHNIQUE)



Atomic Absorption Spectroscopy (AAS) is a technique used to identify metallic elements (qualitative) and measure their concentration (quantitative) by observing the energies absorbed as **electrons** in vaporised **metal atoms** move from the ground state to the excited state, and relating the absorption to concentration.

The sample to be analysed is heated at high temperatures by burning in a flame. The flame breaks the chemical bonds within a substance, converting molecules and ions into individual atoms in vapour form.

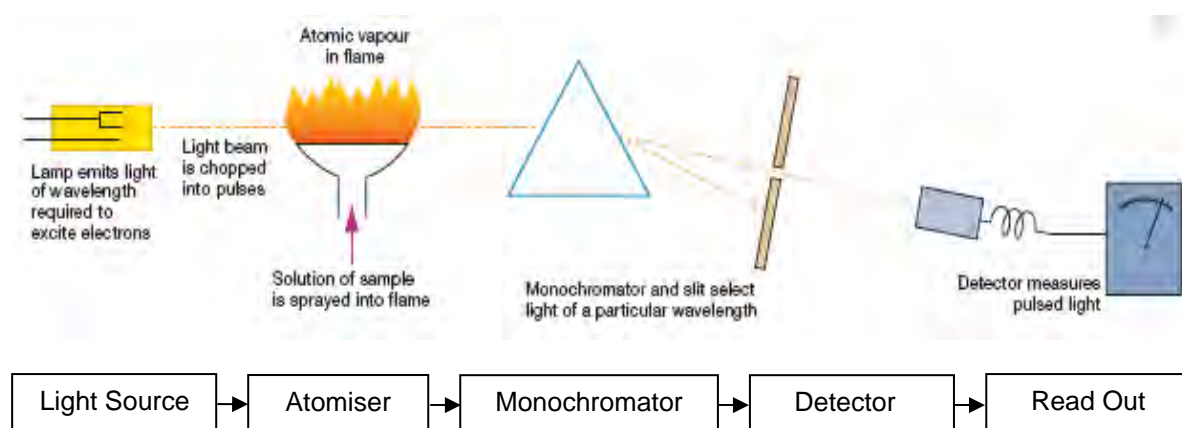
Conditions are chosen so that most atoms remain in the ground state, and it is these ground state atoms which are measured in AAS i.e. The temperature of the flame is chosen so it is low enough that the flame itself does not excite metal atoms from their ground state.

The **ground state metal atoms** in a flame are able to be excited by other means, such as radiation of a wavelength known to be absorbed by them. By measuring the fraction of radiation that is absorbed, we can determine the concentration of the metallic species.

THE COMPONENTS OF AN ATOMIC ABSORPTION SPECTROMETER

There are 5 basic components of an atomic absorption spectrometer:

1. The light source which emits the spectrum of the element of interest.
2. An absorption region (flame, graphite furnace) in which atoms of the analyte are produced and some means of introducing the sample for atomisation.
3. A monochromator, which is used to selectively focus the wavelength of light to be used for analysis.
4. A detector whose response is relative to the amount of radiation.
5. A read-out device that indicates the amount of radiation absorbed.



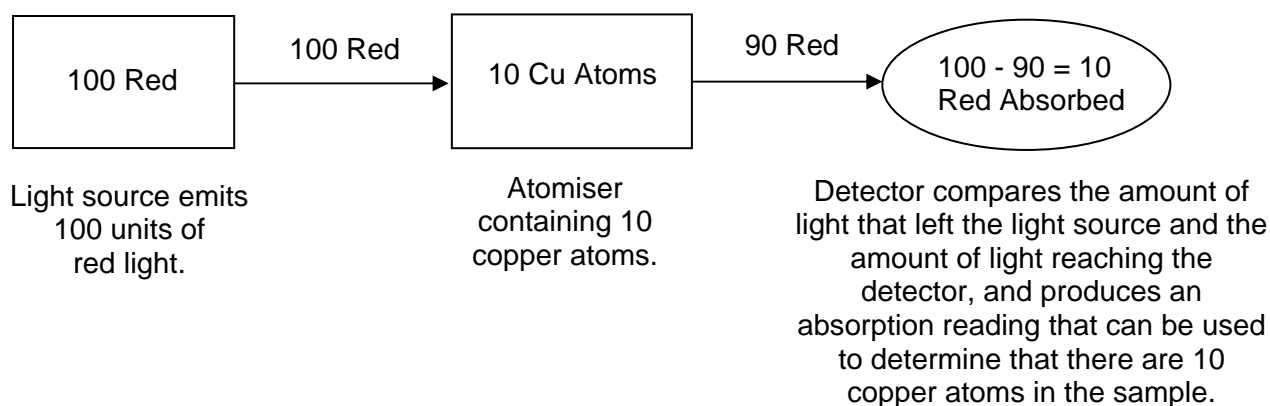
OPERATION - ANALOGY

Imagine that the light source emits 100 units of red light .

Assume that:

- Copper will exclusively absorb these red units of light i.e. no other metal will absorb this colour (wavelength of light).
- Each copper atom will only absorb one unit of red light

Under ideal circumstances, the following scenario will occur:



THE LIGHT SOURCE

The light source is chosen so that it produces the exact wavelength of light required by the atoms in the sample for excitation.

Every atom of a particular element has its own distinct pattern of wavelengths at which it will absorb energy, due to the unique configuration of electrons in its outer shell. i.e. Each metal has a characteristic wavelength that will be absorbed.

The AAS instrument quantitatively analyses a particular metal by focusing a beam of UV or visible light at a specific wavelength through a flame and into a detector. The wavelength used is one that is exclusively absorbed by the metal under investigation.

This is accomplished by using a **hollow cathode lamp** made from the metal being analysed.

In this lamp, high voltages are used to excite its metal atoms. As these excited atoms return to their ground state, they emit their characteristic wavelengths of light. It is these wavelengths that are passed through the atomiser and are absorbed by the metal atoms in the flame.

Note:

A separate lamp is required for each element.

The lamp actually produces the emission spectrum of the element to be analysed so there is an exact match of emitted and absorbed wavelengths.

THE ATOMISER

Atomic Absorption Spectroscopy requires that the analyte atoms be in the gas phase. Ions or atoms in a sample must therefore undergo desolvation (removal of solvent) and vaporisation in a high-temperature source such as a flame or graphite furnace.

Flame Atomic Absorption Spectroscopy can only analyse solutions, while graphite furnace Atomic Absorption Spectroscopy can accept solutions, slurries, or solid samples.

In Flame Atomic Absorption Spectroscopy, the sample is sprayed into the atomiser flame as an acidic solution. The acidic solution helps to dissolve the metal and produce a salt.

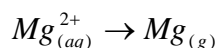
For example: $Mg \rightarrow Mg^{2+} \rightarrow MgCl_2$

QUESTION 65

Why is the analyte usually introduced into the atomiser as a metal chloride?

Solution

The energy of the flame (thermal energy) removes the solvent and reduces the metal to neutral atoms in the ground state. The heat of the flame further transforms the sample into an atomic vapour.



QUESTION 66

Why are metal ions converted into an atomic vapour in atomic absorption spectroscopy?

Solution

The choice of fuel and oxidant used in the atomiser depends largely on the flame temperature required to atomise the sample.

The most common system utilises acetylene (C_2H_2) as a fuel and air as the oxidant, enabling the atomiser to reach temperatures in the order of $1800^\circ C$. If hotter flames are required, nitrous oxide (NO_2) may be used as the oxidant to generate temperatures up to $3000^\circ C$.

When the light from the hollow cathode lamp enters the flame, some of it is absorbed by the ground state metal atoms. The metal atoms absorb the wavelengths of light (from the light source) that are needed for the **outer** electrons to move to higher energy levels. The greater the concentration of metal atoms, the greater the amount of light absorbed.

Note: The absorption of light elevates the valence electrons to their excited states.

QUESTION 67

What is the principal function of the atomiser?

Solution

QUESTION 68

Why do some metal analytes require higher temperatures in the atomiser?

Solution

QUESTION 69

A sample of copper chloride solution was sprayed into the flame of an atomic absorption spectrometer.

- (a) Write an equation to represent the reaction that occurs in the atomiser.
- (b) State the reducing agent in AAS.
- (c) State the oxidant(s) used in flame AAS.

Solution**QUESTION 70**

Which one of the following processes does not occur to a great extent in the atomiser of an atomic absorption spectrometer or the flame of Bunsen burner whilst metal containing samples are being analysed?

- A Evaporation
- B Atomisation
- C Excitation
- D Emission of radiation

THE MONOCHROMATOR/SLIT

The wavelength of light chosen for the analysis and emitted from the cathode lamp passes through the flame and into a prism or monochromator.

The monochromator separates the emerging wavelengths of light so as to ensure that only the wavelength that is emitted from the light source and is not absorbed reaches the detector. In this manner, the monochromator prevents interference from light emissions as electrons return to their lower energy levels.

If the monochromator was omitted from the spectrometer, other sources of light (eg. emissions) would reach the detector. The amount of light reaching the detector would be higher, and this in turn would be interpreted as being due to a lower concentration of metal species in the sample.

i.e. The calculated concentration of the analyte would be lower than the true value.

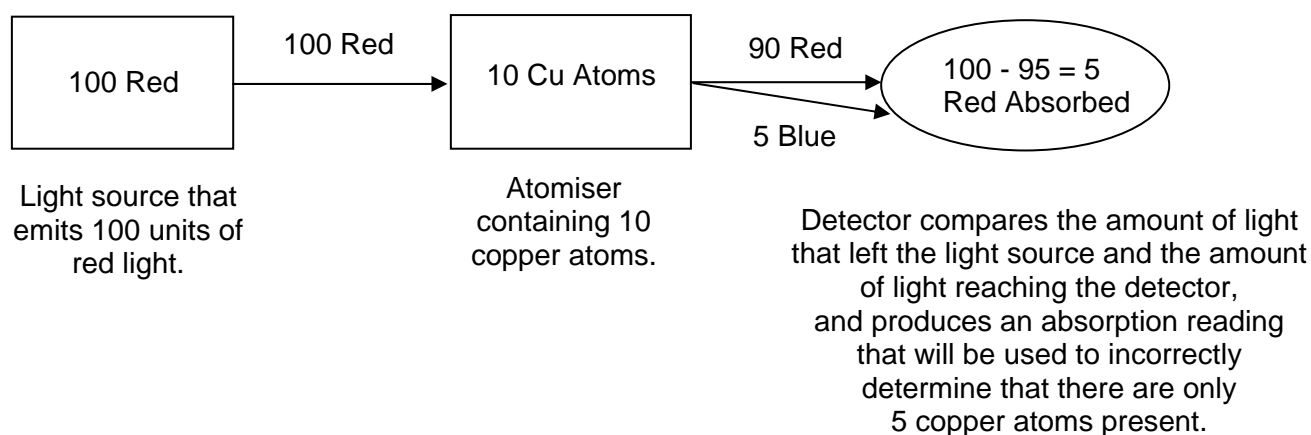
OPERATION ANALOGY - THE IMPORTANCE OF THE MONOCHROMATOR

In the absence of a monochromator:

Assume that 5 of the copper atoms in the atomiser drop to a lower energy level and release blue light in the process.

As the detector cannot distinguish between the different wavelengths of light, 95 pulses will reach the detector (90 units of red light as well as 5 units of blue light), producing a reading that indicates that 5 copper atoms are present in the sample.

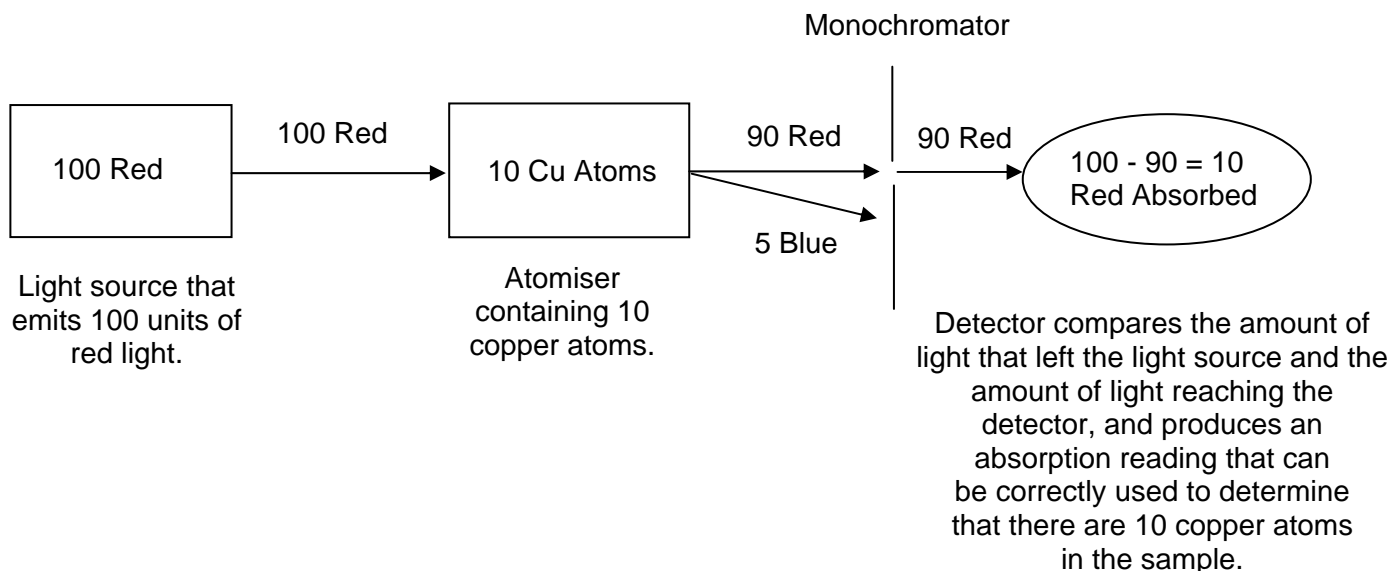
i.e. A smaller absorption value will be obtained. Therefore, the calculated concentration of copper ions will be lower than the true value.



In the presence of a monochromator:

To avoid errors such as these, a monochromator is included into the equipment design.

The monochromator ensures that only light of a specific wavelength reaches the detector (in this case, only red light will reach the detector). The detector compares the amount of pulsed light that left the light source and the amount of pulsed light reaching the detector, and produces an absorption reading that indicates that there are 10 copper atoms in the sample.



THE DETECTOR

The detector used in Atomic Absorption Spectroscopy is a **photomultiplier tube** which converts light that comes in contact with it into electrical energy.

This device measures the amount of light that passes through the sample in the atomiser.

LIGHT CHOPPING

The light coming from the atomised sample will include both the transmitted (unabsorbed) light and the light emitted as excited electrons return to their ground state.

To ensure that the detector does not record the amount of light emitted from excited electrons (which is continuous in nature), the incident light is pulsed so that the detector can differentiate between the two sources of radiation.

i.e. The light emitted from the sample does NOT reach the detector because it is not the same as the chopped light.

Note:

If the incident light was not chopped, the detector would record both the transmitted (unabsorbed) light and the light emitted as excited electrons return to their ground state.

Therefore, the absorbance reading would be lower than what it should be and the calculated concentration of unknown would be lower than the true value.

OPERATION ANALOGY - THE IMPORTANCE OF LIGHT CHOPPING

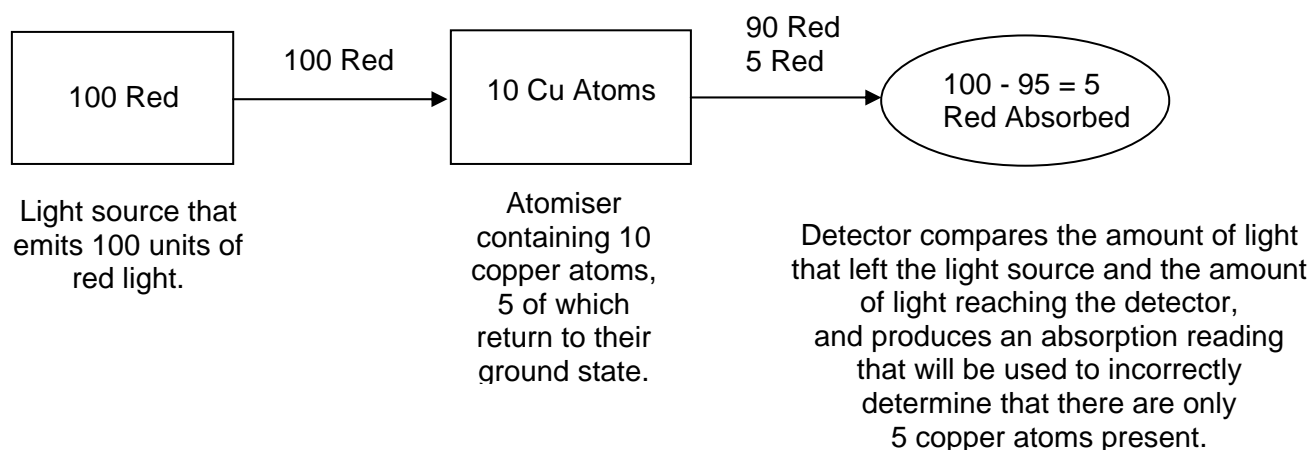
In the absence of light chopping:

Assume that 5 of the copper atoms return to their ground states, emitting green light in the process.

The light coming from the atomised sample will include both the transmitted (unabsorbed) light and the light emitted as excited electrons return to their ground state.

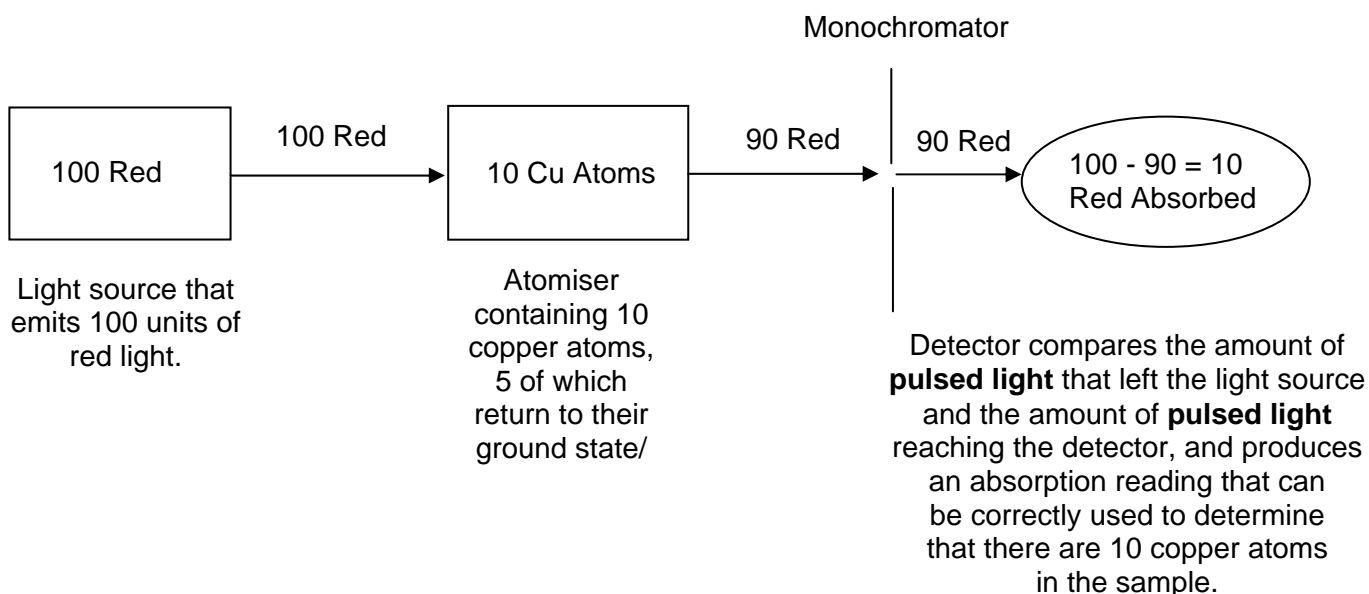
The detector would therefore record 95 pulses of red light and produce an absorbance reading that indicates that there are only 5 copper atoms present.

i.e. A smaller absorption value will be obtained. Therefore, the calculated concentration of copper ions will be lower than the true value.



In the presence of a light chopping:

The incident light is pulsed and the detector is set to record only this pulsed light, and not the continuous light emitted by the excited atoms.



THE READOUT

The intensity of the transmitted light is compared to the intensity of the incident light, and the amount absorbed is determined. The greater the concentration of metal atoms in the sample, the higher the absorbance reading.

$$\text{Absorbance} = \text{Initial Light Intensity} - \text{Final Light Intensity}$$

QUESTION 71

Samples were being analysed for lead by atomic absorption spectroscopy. The samples also contained significant concentrations of copper and barium. Would these ions interfere with the analysis for lead? Give a reason for your answer.

Solution

QUESTION 72

In what phase are the samples introduced into the atomic absorption spectrometer?

Solution

QUESTION 73

In what phase are the analytes analysed in the atomic absorption spectrometer?

Solution

QUESTION 74

Which statement is FALSE about Atomic Absorption Spectroscopy?

- A Hollow cathode lamps are used as light sources.
- B The sample is mixed with an oxidant and fuel in the atomiser.
- C The electrons in the ground state metals in the atomiser are solely excited by the radiation emitted from the cathode lamp.
- D The energies of the transitions observed are in the UV-visible region.

QUESTION 75

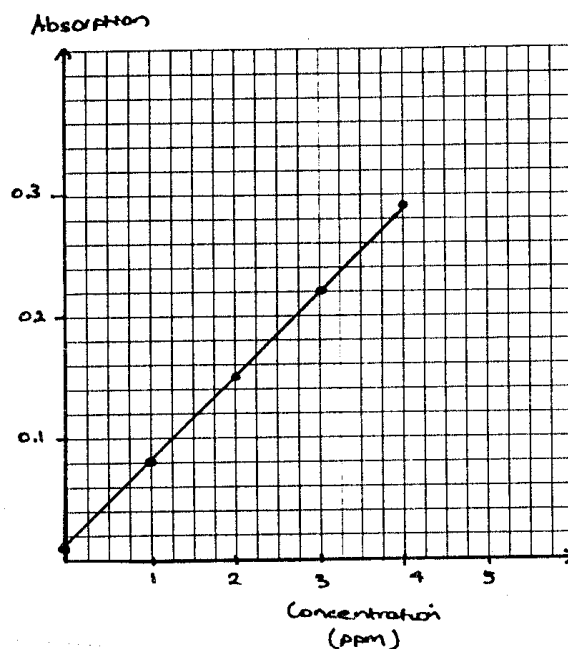
Calibration curves will correct for those atoms that were excited by the heat of the flame, and as a consequence, did not absorb the energy emitted by the light source. What would be the effect on the calculated concentration of an unknown sample if these atoms were not taken into consideration?

Solution

QUESTION 76

Iron is essential to our health. To determine the iron content in a "Milo" milk drink, a 5.0 ml sample was diluted to 50.0 ml . The absorption of the diluted solution and of several standard solutions were measured using AAS. The results are shown below:

Solution concentration (ppm)	Absorption
0.00	0.010
1.00	0.080
2.00	0.150
3.00	0.220
4.00	0.290
Sample	0.190



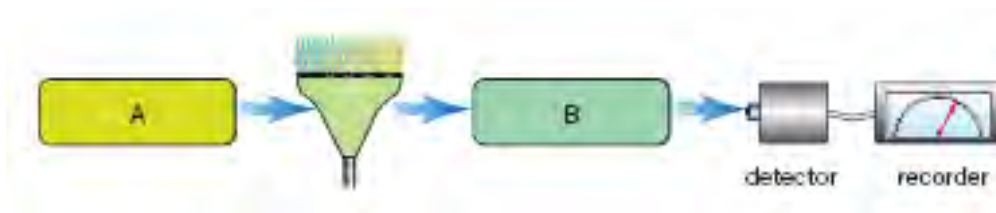
- (a) What is the concentration of iron, in ppm, in the diluted Milo?
- (b) Calculate the concentration of iron, in ppm, in the undiluted Milo.
- (c) What mass of iron would you consume by drinking a 250 ml glass of Milo?

(d) The recommended daily allowance (RDA) of iron for people over the age of 11 years is 18 mg . What percentage of your daily needs does a 250 ml glass of Milo provide?

(e) The 0.00 ppm standard, which contained no added iron, gave a small absorption reading. Suggest an explanation for this.

(f) Suggest why the sample of Milo was diluted in order to measure its absorption.

(e) The diagram below shows the main components of an atomic absorption spectrophotometer in schematic form.



(i) In what state is the sample when analysed using AAS?

(ii) What is the function of component **B**?

(iii) How is component **A** chosen?

QUESTION 77

Atomic absorption spectroscopy was used to measure the concentration of iron in several natural water samples. The samples were filtered then sprayed into the flame of the instrument at a carefully regulated rate; absorbance by the iron atoms produced in the flame was measured using the appropriate lamp for iron. Results are tabulated below.

Sample	L	M	P	Q
Absorbance	0.74	0.05	0.53	0.28

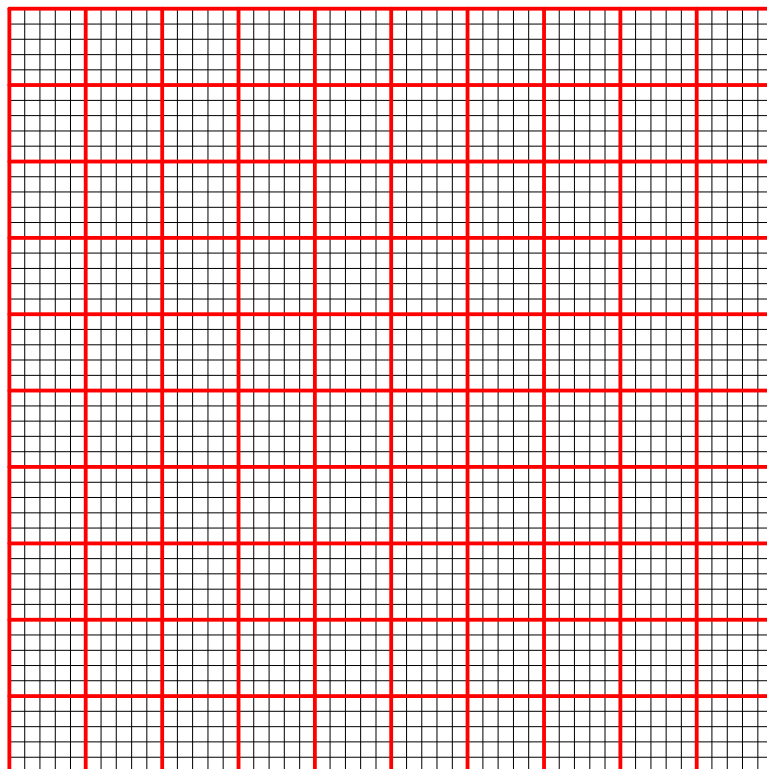
A calibration curve was then constructed as described below:

3.62 g of hydrated iron(II) ammonium sulfate, a very pure compound of iron with formula $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, was dissolved in dilute acid solution and the volume made up to 0.500 L. Volumes of this solution were then accurately diluted to 1.000 L. These diluted solutions were analysed in the instrument in exactly the same way as was used for the samples for analysis. Results are recorded below.

Volume (in mL) of concentrated solution diluted to 1.000 L	1.00	2.00	5.00	10.00
Absorbance	0.07	0.13	0.34	0.69

- (a) Calculate the concentration (in ppm) of iron in each of the standard solutions and draw a graph of absorbance versus concentration.

- (b) Use the calibration curve to estimate the iron concentration in each of the unknown samples.



- (c) Does this analysis measure iron(II) or iron(III) or both? Explain your answer.

QUESTION 78

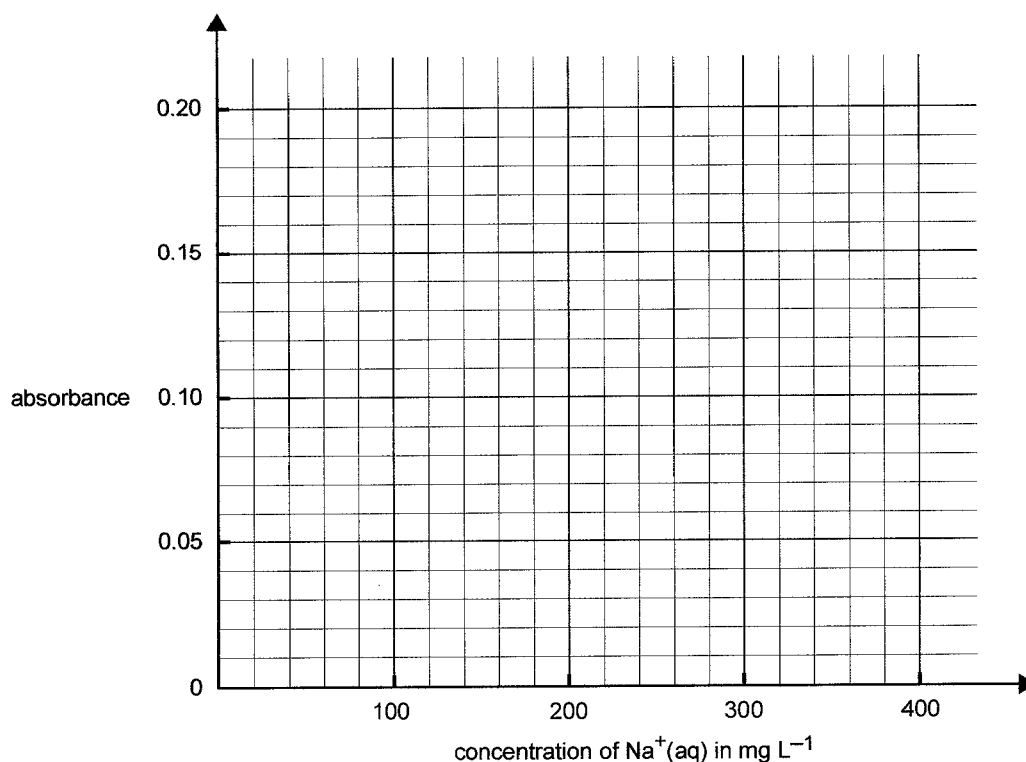
Sodium is an essential element in our diets. However, the amount of sodium present in some foods is often much higher than levels recommended by doctors. A sauce was analysed using atomic absorption spectroscopy to determine the sodium content.

A 25.00 ml sample of the sauce was diluted to 1.00L with deionised water.

Four aqueous samples of known NaCl concentration were also prepared as standard solutions. The absorbances of the four standard solutions and the diluted sauce solution were measured. The results are given in the table below.

concentration of $\text{Na}^+(\text{aq})$	absorbance
100 mg L^{-1}	0.051
200 mg L^{-1}	0.100
300 mg L^{-1}	0.149
400 mg L^{-1}	0.199
diluted sauce	0.185

- (a) Use the above data for the $\text{Na}^+(\text{aq})$ standards to plot a calibration line on the graph below.



(b) Use your calibration graph to determine the sodium ion concentration in the diluted sample of the sauce and in the original sauce in mg/L .

(c) (i) What important assumption must you make in order to calculate the $NaCl$ content of the sauce from the Na^+ concentration?

(ii) Calculate the concentration of $NaCl$ in the original (undiluted) sauce in g/L .

- (iii) The maximum recommended daily $NaCl$ intake for a healthy adult is 2.5 g . What percentage of a maximum daily recommended intake would be consumed by a person who eats 10 ml of the original (undiluted) sauce?
- (d) Why is it that atomic absorption spectroscopy will measure only the sodium ion concentration in your sample and not the concentration of some other substance or substances as well?
- (e) If the monochromator/slit was omitted from the atomic absorption spectrometer, what effect would this have on the calculated concentration of Na^+ ? Give a reason for your answer.

USES OF AAS

Atomic absorption spectroscopy is widely used to monitor:

- Low concentrations of metals eg. The detection of copper and aluminium in waterways, zinc in oysters, lead fallout beside highways and mercury in fish.
- Concentrations of micro-nutrients in soils.
- Small amounts of contaminants in foods (particularly processed foods) and medicines and other manufactured goods
- The concentration of trace elements (elements that are required by living organisms in very small amounts - generally in the 1 to 100 ppm range).

ADVANTAGES OF AAS

- Apparatus is relatively easy to operate.
- The technique is rapid, allowing large numbers of samples to be measured in a short period of time.
- Very small quantities of sample may be analysed.
- This technique may be used to identify many metals that cannot be excited using atomic emission spectroscopy.

AAS can be used to detect metals in a variety of samples, including urine, blood, soil, food, drink. eg. Lead in petrol, mercury in fish.

- AAS is so selective for metals that there is no need to separate them from other sample components. This makes AAS ideal for analysing ores, blood and urine.
- Very sensitive – AAS can detect concentrations as low as ppb. mg / L to $\mu g / L$ detection is routine

DISADVANTAGES OF AAS

- The technique is restricted to approximately 70 metal elements.
- AAS cannot be used on atoms that are too easily excited by the heat of the flame. These atoms are better analysed using emission spectroscopy.