

ATOMIC ABSORPTION SPECTROSCOPY

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CONTENTS:

- Principle of AAS.
- Instrumentation.
- Applications.
- Experiments.

INTRODUCTION:

- Atomic Absorption Spectroscopy is a very common technique for detecting metals and metalloids in samples.
- It is very reliable and simple to use.
- It can analyze over 62 elements.
- It also measures the concentration of metals in the sample.

HISTORY:

The first atomic absorption spectrometer was built by CSIRO scientist Alan Walsh in 1954. Shown in the picture Alan Walsh(left), with a spectrometer.



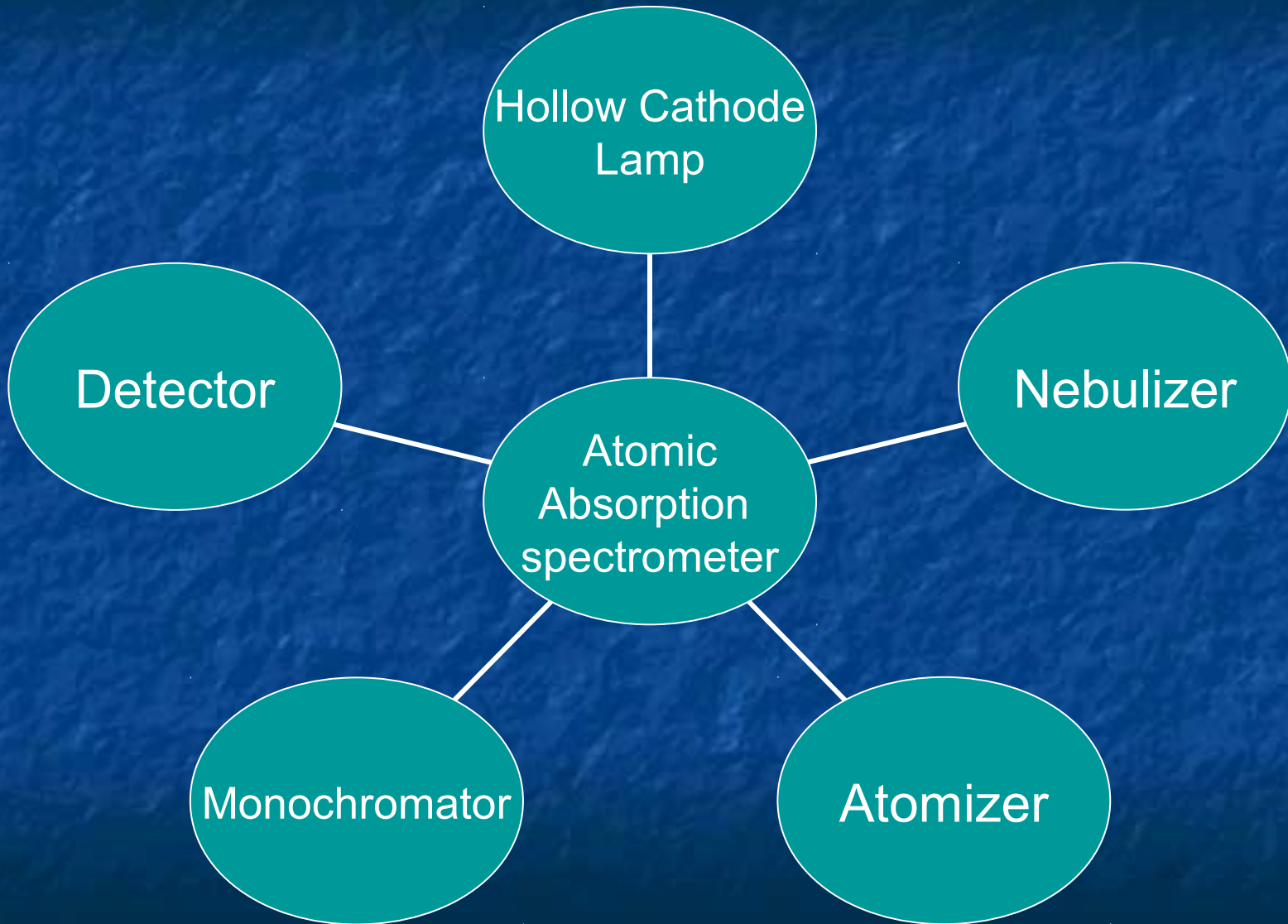
PRINCIPLE:

- The technique uses basically the principle that free atoms (gas) generated in an atomizer can absorb radiation at specific frequency.
- Atomic-absorption spectroscopy quantifies the absorption of ground state atoms in the gaseous state .
- The atoms absorb ultraviolet or visible light and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption.

- Concentration measurements are usually determined from a working curve after calibrating the instrument with standards of known concentration.

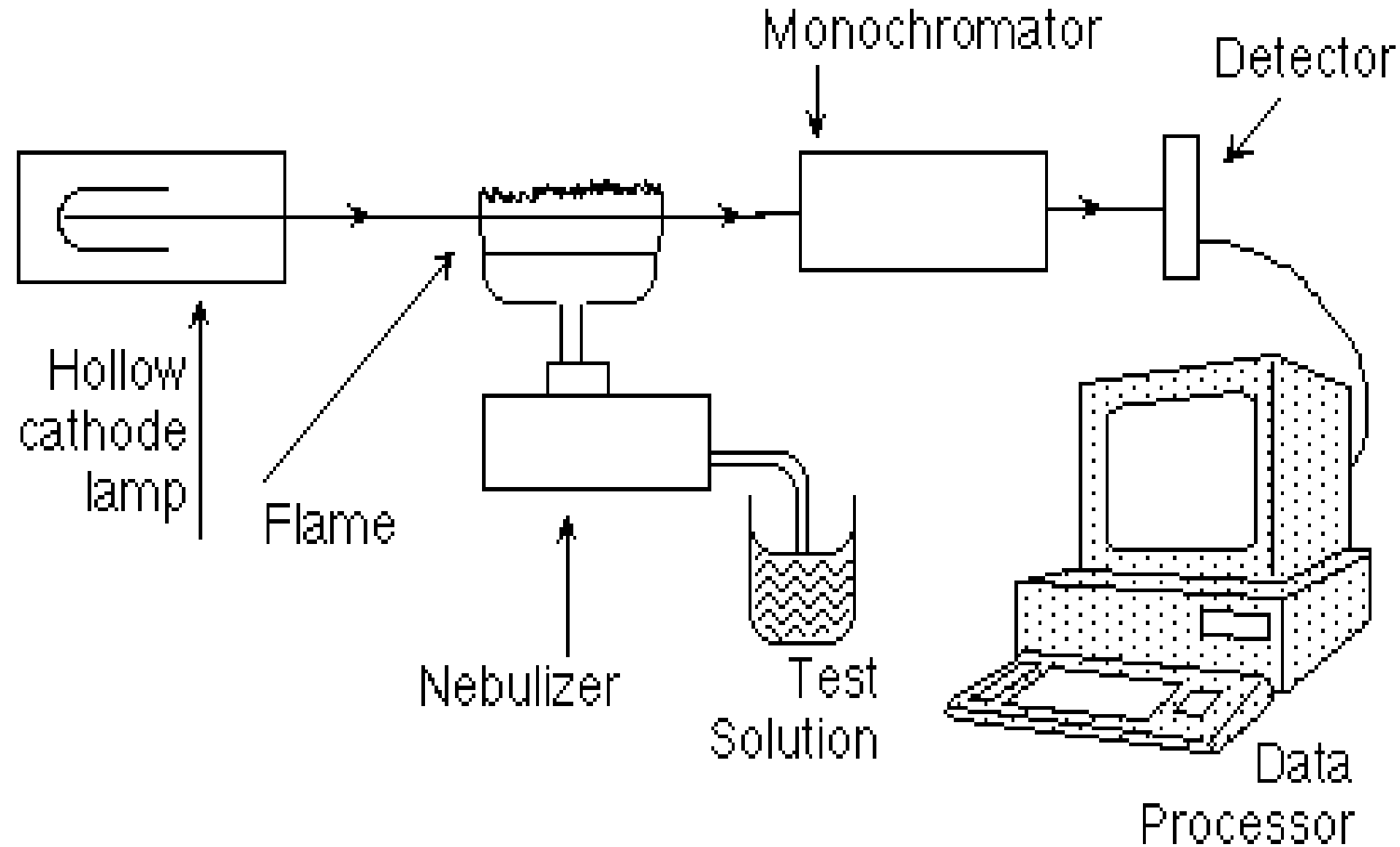
- Atomic absorption is a very common technique for detecting metals and metalloids in environmental samples.

THEORY:





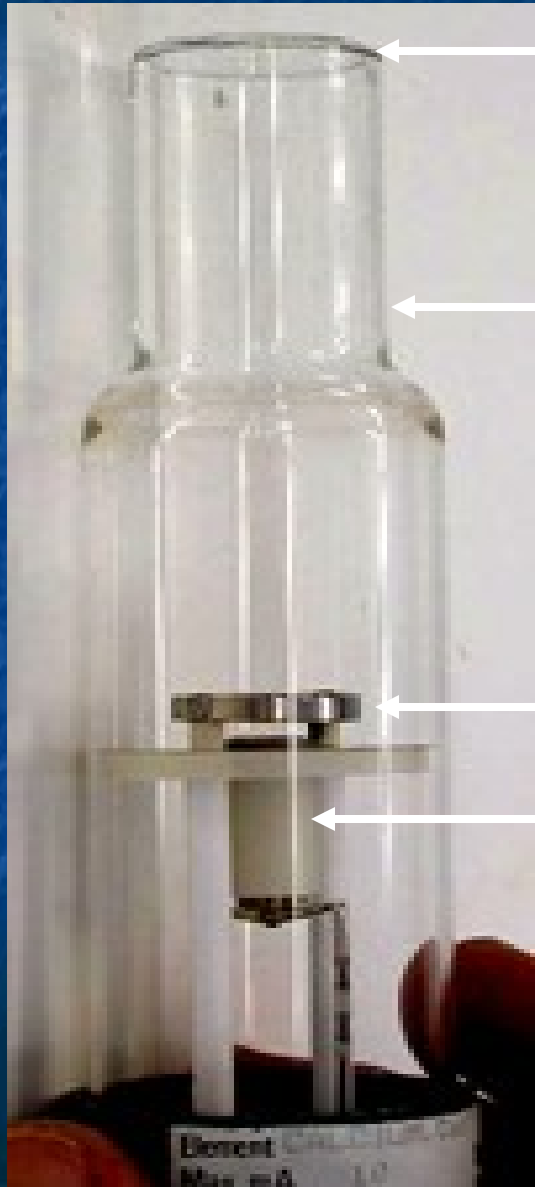
Schematic diagram of AAS:



LIGHT SOURCE:

- Hollow Cathode Lamp are the most common radiation source in AAS.
- It contains a tungsten anode and a hollow cylindrical cathode made of the element to be determined.
- These are sealed in a glass tube filled with an inert gas (neon or argon) .
- Each element has its own unique lamp which must be used for that analysis .

Hollow Cathode Lamp:



Quartz window

Pyrex body

Anode

Cathode



cathode

NEBULIZER:

- suck up liquid samples at controlled rate.
- create a fine aerosol spray for introduction into flame.
- Mix the aerosol and fuel and oxidant thoroughly for introduction into flame.

Atomizer

- **Elements** to be analyzed needs to be in atomic state.
- **Atomization** is separation of particles into individual molecules and breaking molecules into atoms. This is done by exposing the analyte to high temperatures in a flame or graphite furnace .

ATOMIZERS:

ATOMIZER

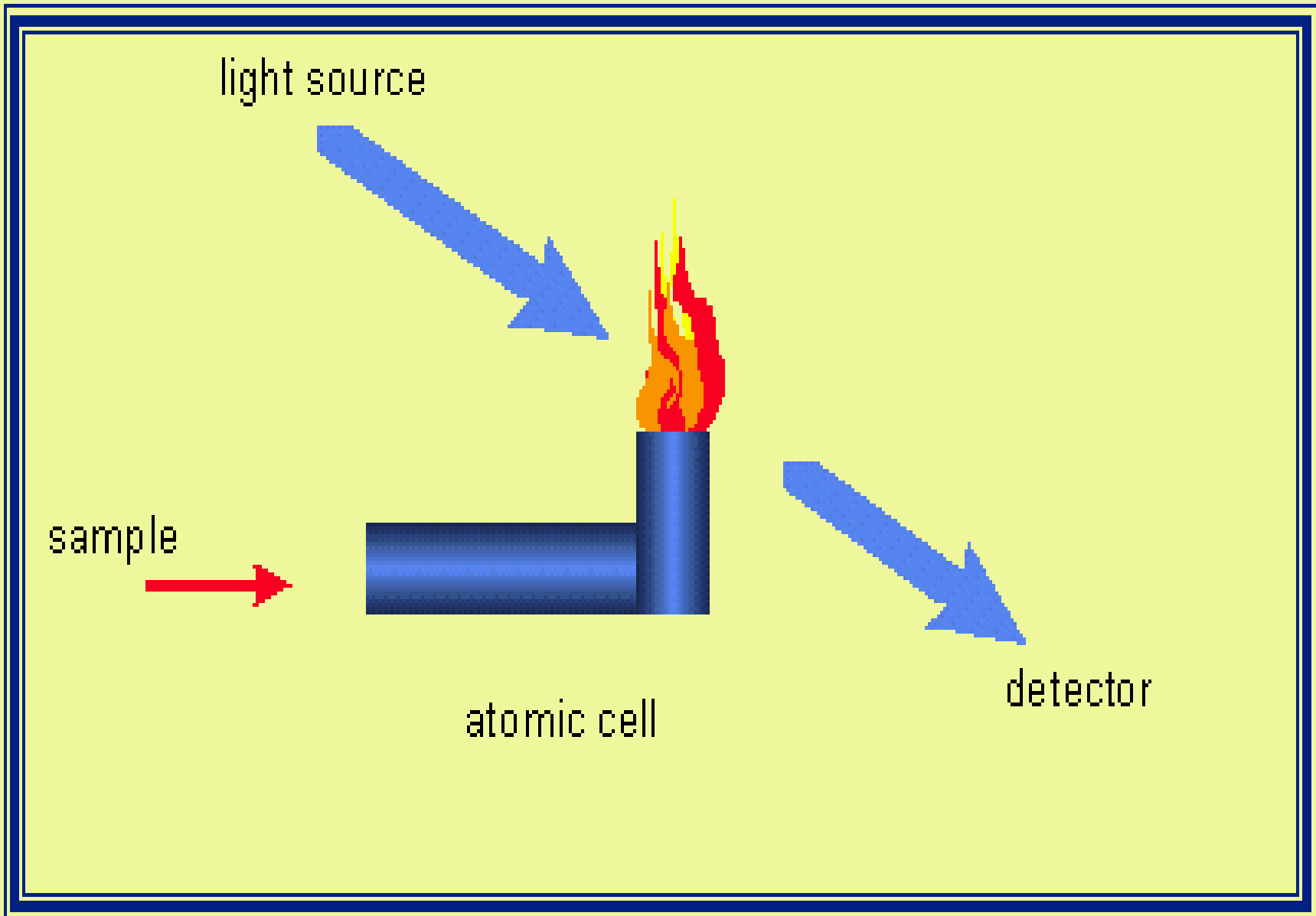
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graph TD; A[ATOMIZER] --> B[FLAME ATOMIZERS]; A --> C[GRAPHITE TUBE ATOMIZERS]
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FLAME
ATOMIZERS

GRAPHITE TUBE
ATOMIZERS

FLAME ATOMIZER:

- To create flame, we need to mix an oxidant gas and a fuel gas.
- in most of the cases air-acetylene flame or nitrous oxide-acetylene flame is used.
- liquid or dissolved samples are typically used with flame atomizer.



GRAPHITE TUBE ATOMIZER:

- uses a graphite coated furnace to vaporize the sample.
- In GFAAS sample, samples are deposited in a small graphite coated tube which can then be heated to vaporize and atomize the analyte.
- The graphite tubes are heated using a high current power supply.

MONOCHROMATOR:

- This is a very important part in an AA spectrometer. It is used to separate out all of the thousands of lines.
- A monochromator is used to select the specific wavelength of light which is absorbed by the sample, and to exclude other wavelengths.
- The selection of the specific light allows the determination of the selected element in the presence of others.

DETECTOR:

- The light selected by the monochromator is directed onto a detector that is typically a photomultiplier tube , whose function is to convert the light signal into an electrical signal proportional to the light intensity.
- The processing of electrical signal is fulfilled by a signal amplifier . The signal could be displayed for readout , or further fed into a data station for printout by the requested format.

Calibration Curve

- A calibration curve is used to determine the unknown concentration of an element in a solution. The instrument is calibrated using several solutions of known concentrations. The absorbance of each known solution is measured and then a calibration curve of concentration vs absorbance is plotted.
- The sample solution is fed into the instrument, and the absorbance of the element in this solution is measured. The unknown concentration of the element is then calculated from the calibration curve

APPLICATIONS:

Determination of even small amounts of metals (lead, mercury, calcium, magnesium, etc) as follows:

- Environmental studies: drinking water, ocean water, soil.
- Food industry.
- Pharmaceutical industry.

EXPERIMENTS

- DETERMINATION OF VANADIUM IN LUBRICATING OIL
- DETERMINATION OF TRACE ELEMENTS IN CONTAMINATED SOIL

VANADIUM IN LUBRICATING OIL:

- THEORY:

High temperature corrosion and fouling can be attributed to vanadium in the fuel. During combustion, the element oxidize and form semi-liquid and low melting salts (vanadium pentoxide), which adhere to exhaust valves and turbochargers. In practice, the extent of hot corrosion and fouling are generally maintained at an acceptable level through temperature control, an operational solution, and material selection.

- the oil is dissolved in white spirit and the absorption of this solution is compared with the absorption of standard.
- **STANDARD SOLUTION:** the standard solutions are made up from vanadium naphthenate in white spirit which contain about 3% of vanadium.
- (weigh out 0.6 g of vanadium naphthenate into a 100 ml flask and made up to mark with white spirit. dilute portions of this stock solution to obtain a series of working standards containing 10-40 mg ml⁻¹ of vanadium).

PROCEDURE:

- weigh out accurately about 5 g of the oil sample, dissolve in small volume of white spirit and transfer to 50 ml flask.
- using same solvent, make up the sol. to the mark.
- set up a vanadium hollow cathode lamp selecting a resonance line of wavelength 318.5 nm.
- adjust gas controls to give a fuel rich acetylene-nitrous oxide flame.
- aspirate successfully into the flame the solvent blank, standard solutions and finally the test solution
- in each case recording the absorbance reading .plot the calibration curve and ascertain the vanadium content of the oil.

LEAD IN CONTAMINATED SOIL:

- **SAMPLING:** samples of approx. 50g should be taken from specified sampling points on the site.
- The sampling point should include surface soil and two further samples taken at depth, at 0.5 and 1.0m.
- The exact location of these points should be noted, for it may be necessary to take further samples.

PROCEDURE:

- weight out about 1g of sieved soil and transfer to a 100ml beaker. add 20 ml of 1:1 nitric acid .
- boil gently on a hot plate until the volume of nitric acid is reduced to 5ml.
- add 20ml of deionised water and boil gently again until the volume is 10ml.

- cool the suspension and filter through a whatman filter paper, washing the beaker and filter paper with deionised water until a volume of about 25ml is obtained.
- transfer the filtrate to a 50ml flask and make up to the mark with deionised water.
- setup acetylene-air flame with resonance line 217.0 nm.
- standard lead solutions containing 1-10 mg ml⁻¹ are suitable for measurement

REFERENCES:

- *Vogel's Textbook of Quantitative Analysis*, G. Svehla, Pearson.
- *Principles of Instrumental Analysis*, Skoog.
- *Basic Concepts Of Analytical chemistry*, S M Khopkar.

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