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We understand commodities

Knowledge & Skill Development of Food Testing Laboratory Personnel from African Countries under IAFS– II

National Collateral Management Services Limited

Analysis of Metals in Food samples

- Obtain a representative sample from bulk
- Destruction of organic matter
- Separation and concentration and
- Determination

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Analysis of Metals in Food samples

- Fresh foods: Macerating in a blender
- Dry products: Ground and powder is sieved
- Hard foods like chocolates:

Grating/chopping finely by hand

- Meat products: Thoroughly minced and

ground in a mortar

- Fats: Melted before analysis

Analysis of Metals in Food samples

Destruction of Organic matter :

- Wet oxidation
- Dry Ashing and
- Microwave Digestion

Analysis of Metals in Food samples

❖**Wet oxidation method:**

Samples are digested with Nitric acid and with one of the following on hot plate

a) Sulfuric acid, (98%)

b) Perchloric acid (70%)

c) Hydrogen Peroxide (30%)

Analysis of Metals in Food samples

Dry Ash Method:

- Place the homogenized and ground sample in Muffle Furnace at 500° C
- Dissolve the ash in Hydrochloric acid (1+1)
- Dilute with water

Analysis of Metals in Food samples

Microwave digestion:

- 1g of finely ground sample treated with Nitric acid in a Teflon vessel
- Seal the vessels and digest as per the instructions
- Transfer the contents and dilute to the specified volume

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STANDARD PREPARATION

Preparation of Arsenic standard solution

- Take aliquot of stock solution (1000mg/l)
- Add 5ml of HCl and 5ml of each 5% KI and L-Ascorbic acid solution
- Allow the mixture to stand for 1 hour
- Make up the volume with 10% HCl

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STANDARD PREPARATION

Preparation of Selenium standard solution

- Take appropriate aliquot of stock solution (1000mg/l)
- Add equal volume of 3% Hydrochloric acid
- Heat the contents at 90°C for 20 min
- Cool and make up the volume with 3% HCl

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STANDARD PREPARATION

Preparation of Mercury standard solution

- Take appropriate aliquot of stock solution (1000mg/l)
- Dilute to mark with 3% Hydrochloric acid
- Prepare different concentrations to construct calibration curve

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STANDARD PREPARATION

Preparation of standard solutions of other elements (Multi element standard)

- Acidify aliquot of stock solution with Nitric acid (HNO_3)
- Make up the volume to obtain 2% HNO_3 in the final standard solution

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Elemental Analysis in Food

- In order to cover all of the elements that should be analyzed at the appropriate ranges, several techniques are used

- As/Se/Sn–hydride generation AAS or VGA-ICP
- Hg–cold vapor technique or VGA-ICP
- All other elements at sub ppb level GFAAS or
- All other elements at ppb–ppm-% level ICP-OES

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Instrumentation - AAS

- AAS is commonly used for metal analysis
- A solution of a metal compound is sprayed into a flame and vaporises to form atoms
- The metal atoms absorb light of a specific frequency, and the amount of light absorbed is a direct measure of the number of atoms of in the solution

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Instrumentation - AAS

- Atomic Absorption Spectroscopy is a quantitative method of analysis that is applicable to many metals and few nonmetals.
- The technique was introduced in 1955 by Walsh in Australia (A. Walsh, Spectrochim. Acta, 1955, 7, 108)

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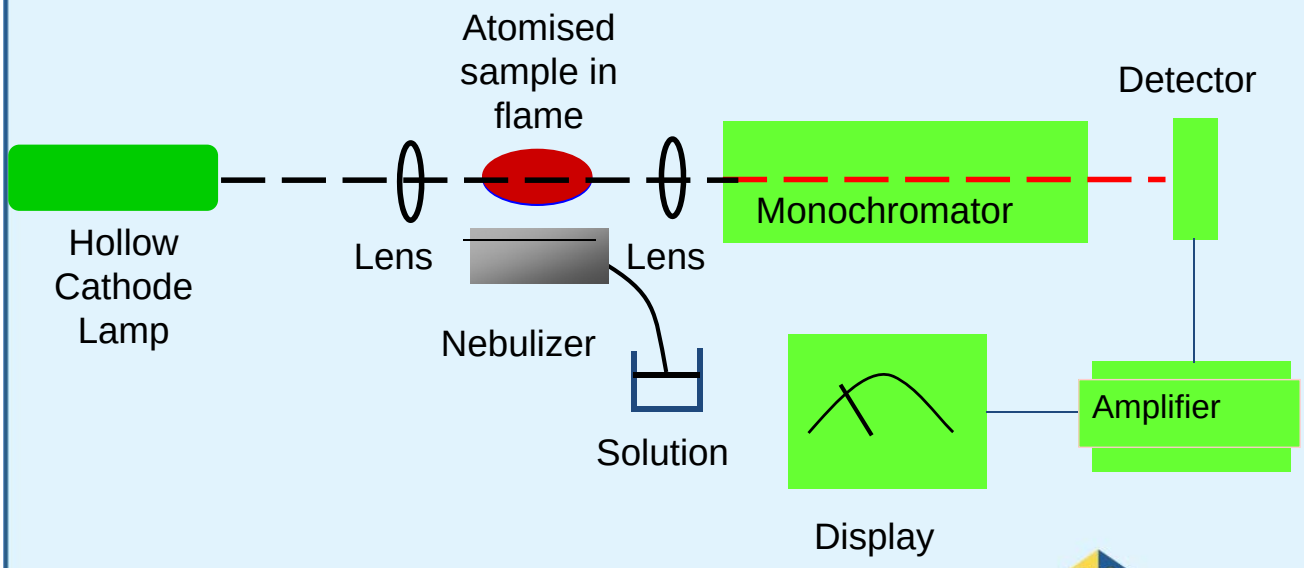
Instrumentation - AAS

AAS

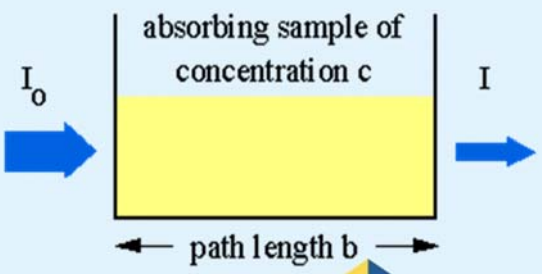


Instrumentation - AAS

- It is possible to measure the concentration of an absorbing species in a sample by applying the Beer-Lambert Law:

$$Abs = \varepsilon c b$$

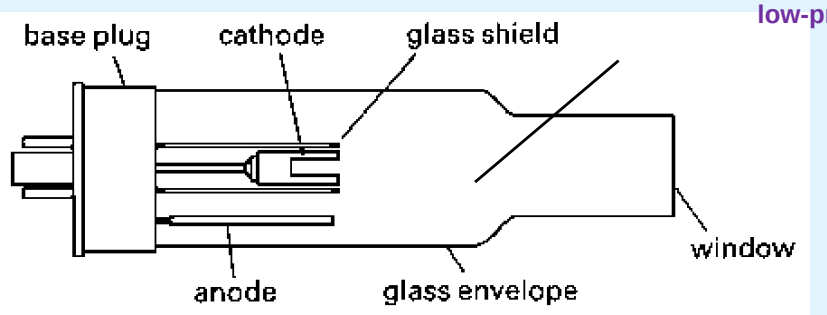
$$Abs = -\log\left(\frac{I}{I_o}\right)$$



ε = extinction coefficient

Hollow cathode lamp

Lamp



low-pressure inert gas

- Electron and ionic impact on cathode
- M(s) → M(g)
- M(g) → → → M*(g)
- M*(g) → M(g) + hν

Thin lay of cathode material

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Atomization – Flame mode

Two different types of flames are used

- Air-acetylene flame (2300°C)
- Usually 10-12cm long
- Nitrous oxide-acetylene flame (2700°C)
- 5cm long

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Atomization – Flame mode

Reaction at Flame of burner:

- $M^{n+}(aq) + anion(aq) \rightarrow salt(s)$
- $salt(s) \rightarrow salt(g)$
- $salt(g) \rightarrow atoms\ (g)$
- $M(g) + h\nu \rightarrow M^*(g)$

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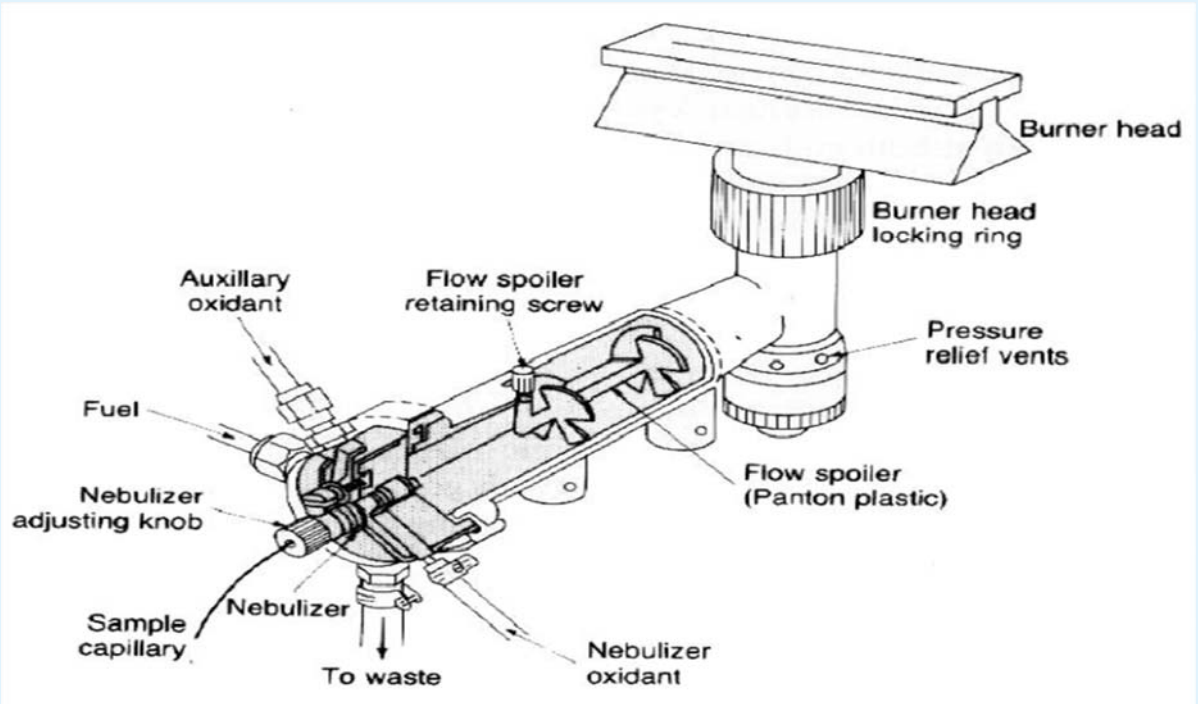
Nebulizer-Burner



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Nebulizer-Burner

- Nebulizer---to produce a mist or aerosol of the test solution
- Vaporizing chamber ---Fine mist is mixed with the fuel gas and the carrier gas
- Larger droplets of liquid fall out from the gas stream and discharged to waste
- Burner head ---The path length of flame is about 10 –12 cm

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Graphite tube atomizer (GTA)

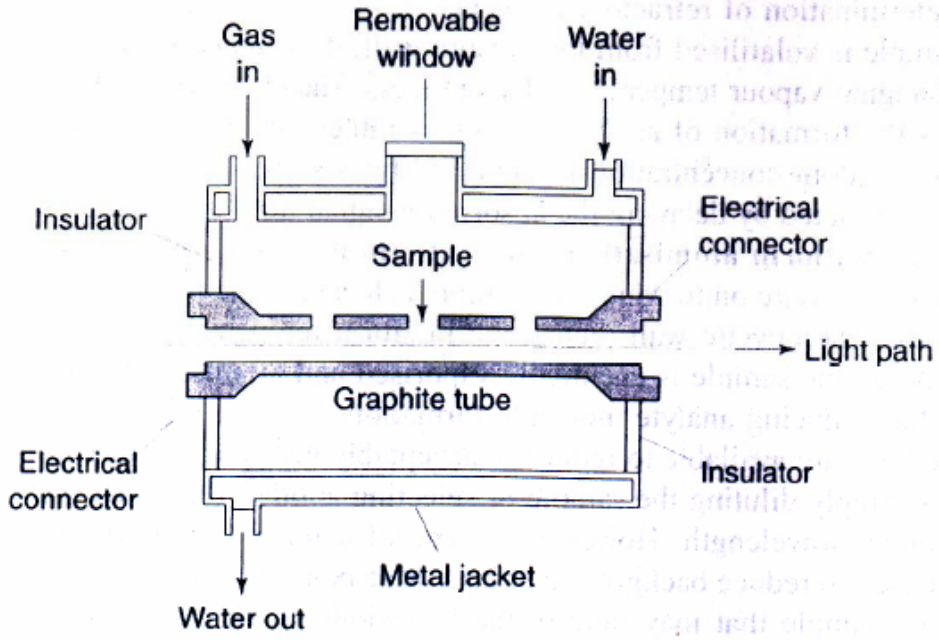

Figure 15.5 Graphite tube furnace

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Graphite tube atomizer (GTA)

It involves mainly three steps:

- ❖Drying or desolvation (105° C)
- ❖Ashing or Pyrolysis (550° C)
- ❖Atomization (depends on element)

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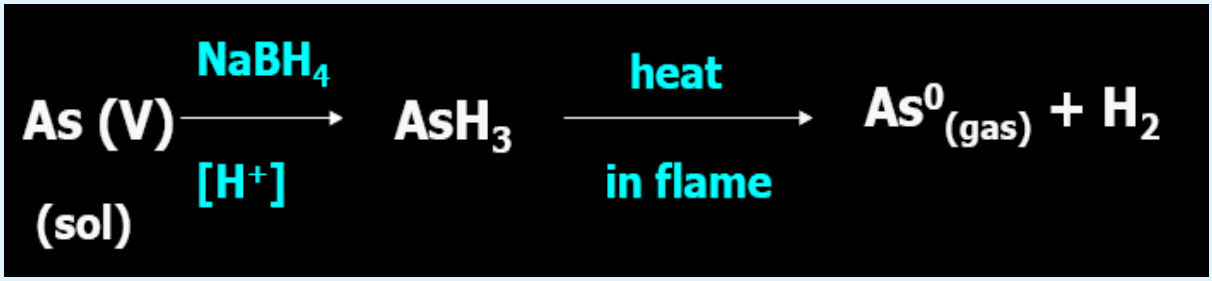
Hydride generation method-For Arsenic, Selenium & Antimony

- ❖Hydride formation of Arsenic



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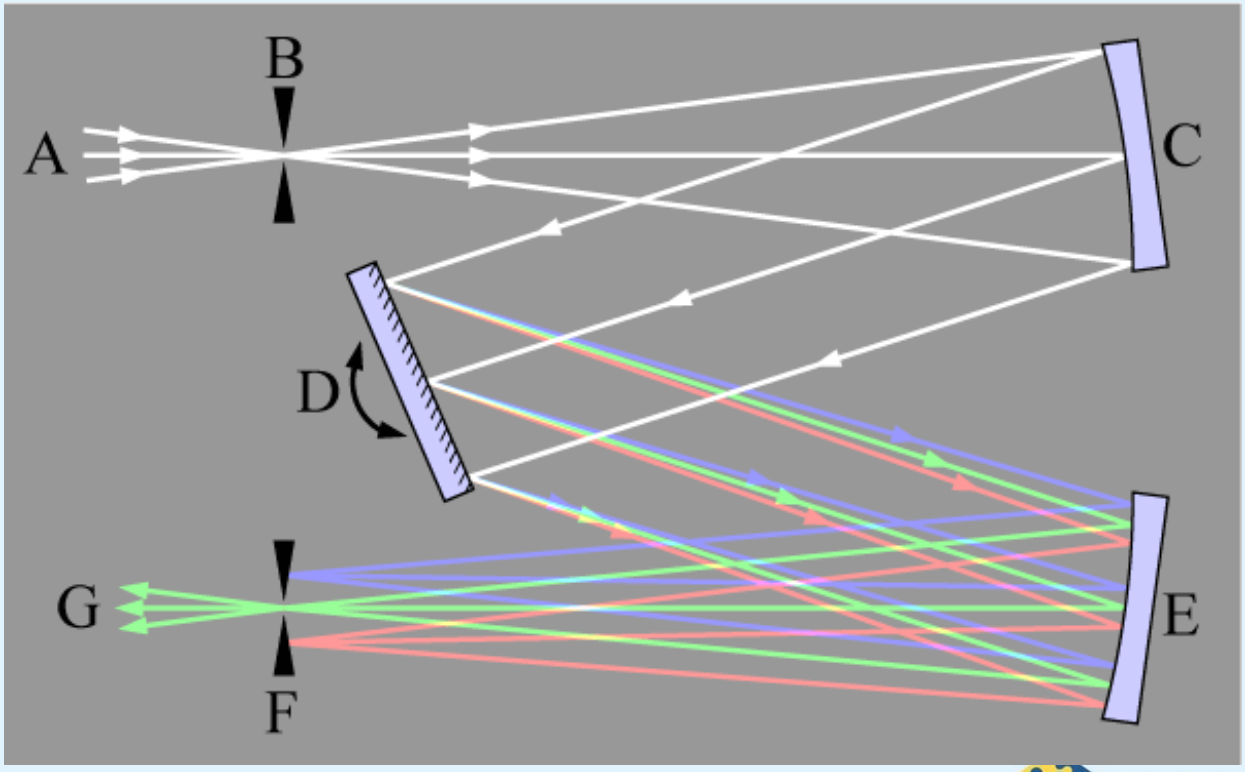
Monochromator



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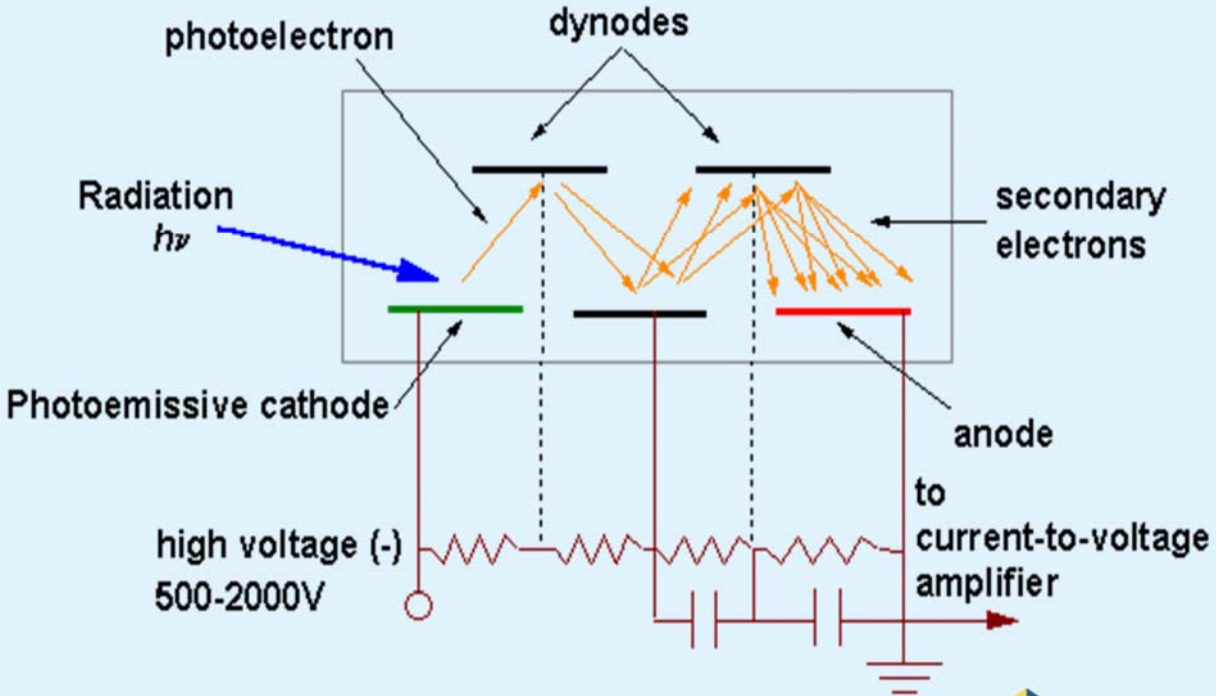
Photomultiplier tube (PMT)



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