Electronics And Instrumentation

8th Semester

Subject: Analytical Instrumentation
Subject code: BT 808

Unit-3

Flame Photometers

Let us learn about the different components of a flame photometer.

6.1 Flame Atomiser

The role of atomiser is to generate the vapours of analyte which get excited by the thermal energy of the flame and then emit characteristic radiation that is measured. The flame atomiser assembly consists of two components. The prior is a nebuliser where the sample in the form of a solution is drawn in and converted into a fine mist or an aerosol. It is then passed onto the second component i.e. the burner along with air or oxygen and a fuel gas. In the flame a number of processes (Section 7.4) occur that convert the analyte into excited species. Let us learn about the nebulisers used in flame photometers. You would learn about the burners in the next subsection.

Nebuliser is a device used for sample introduction into the flame. The process is called nebulisation and consists of thermal vapourisation and dissociation of aerosol particles at high temperatures producing small particle size with high residence time. A number of nebulisation methods are available. A few are listed below.

• Pneumatic nebulisation
• Ultrasonic nebulisation
• Electrothermal vapourisation
• Hydride generation (used for certain elements only)

However, we would discuss about the pneumatic nebulisation only. It is the most commonly employed nebulisation method in flame photometers.

Pneumatic nebuliser is the most commonly used nebuliser for introducing aqueous/ liquid samples. In this the sample solution is fed or aspirated into the nebuliser which converts liquid into a fine mist, or aerosol which is then fed into the flame. A common type of pneumatic nebuliser is called **concentric pneumatic nebuliser**, as shown in Fig. 9. The concentric pneumatic nebuliser consists of a fine capillary surrounded by concentric tube with a small orifice near one end of the capillary. The capillary is dipped into a solution of the analyte while the outer tube is connected to a high pressure gas supply. The analyte is sucked into the capillary by the high pressure gas stream flowing around the tip of the capillary using the Bernoulli effect. The process is called **aspiration**. The high velocity gas breaks up the liquid into various sized fine droplets. The other types of the pneumatic nebulisers also work on the same principle.
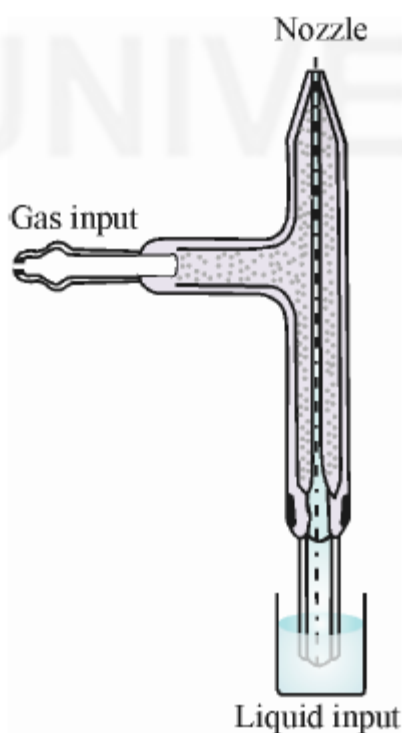
**Fig. 9:** Concentric type pneumatic nebuliser

7.6.2 Atomiser Burners

The sample is introduced in the form of a fine spray at a controlled rate into the flame of a burner with the help of nebuliser. In the burner, the analyte undergoes a number of processes as mentioned earlier. Two types of atomisation burners have been used in flame photometry which are given below and explained in the following paragraphs..

a) Pre-mix or Lundegarh burner
b) Total consumption burner

a) Premix or Lundegarh burner

In the premix burner, fuel and oxidant are thoroughly mixed inside the burner housing before they leave the burner ports and enter the primary-combustion or inner zone of the flame. In this type of burner, the solution of the analyte is aspirated with the help of a nebuliser from the sample container into the mixing chamber in which the fuel gas is also introduced. The larger drops are stopped by baffles in the mixing chamber and are drained off. Pressure and density fluctuation of the aerosol due to atomisation are smoothened in the mixing
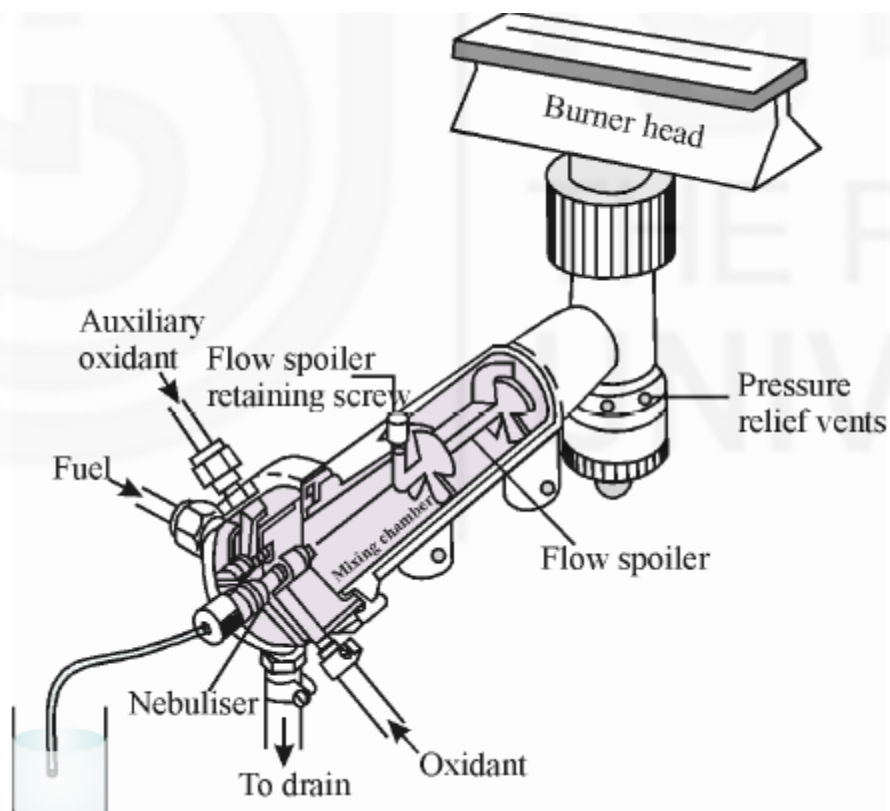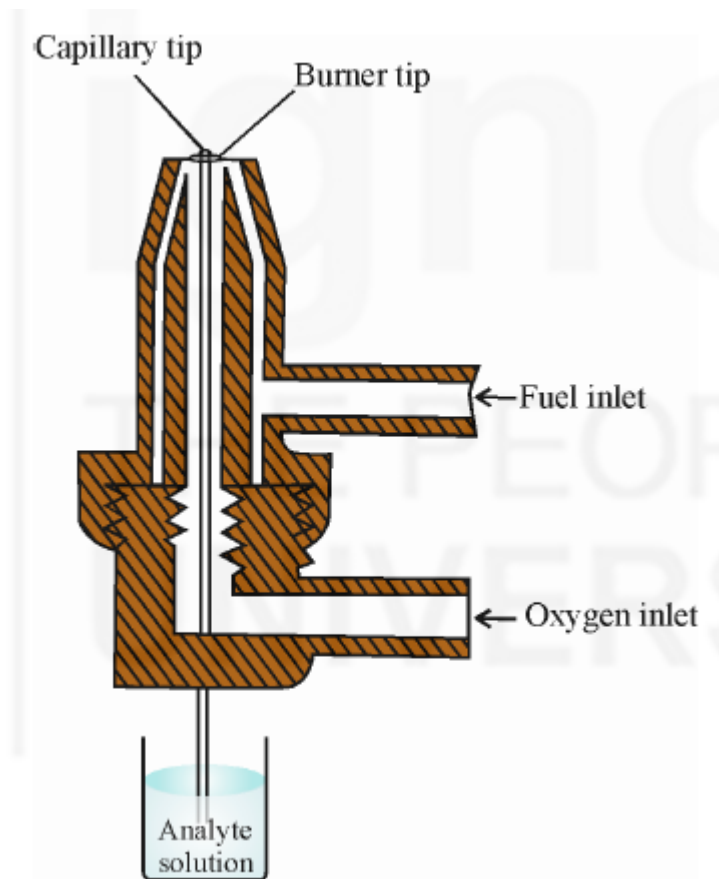
Fig.10: Schematic diagram of a premix burner

chamber and mixture of aerosol, fuel gas and oxidant burns to yield stable noiseless flame. The solvent of the smaller droplets evaporates partly first in mixing chamber, then in the conduit of the burner and finally in the flame. Only drops of a certain size can reach the burner and are completely burnt. Thus, the efficiency of the premix burner is low and only 5% of the sample reaches the burner. The efficiency of this burner has been improved by putting an impact bead immediately after the nebuliser. The impact bead has the effect of breaking up large droplets as they come out from the nebuliser, thereby increasing the number of smaller droplets and the efficiency of nebulisation. The schematic diagram of a premix burner is given in Fig.10.

Premix burners are called **Bunsen, Méker**, or **slot burners** depending on whether they have one large hole, a number of small holes, or a slot as outlet port(s) for the gas mixture, respectively. In

case the burner contains several parallel slots it is called a **multislot burner** (e.g. a **three-slot burner***). The small diameter of the holes in the Méker burner or the narrowness of the slot in the slot burner usually prevents the unwanted **flash back** of the flame into the burner housing. The **advantage** with the premix burner is that it is quiet to operate and the analytical signal is significantly less noisy than that of total consumption burner (described below). However, it also has certain **disadvantages** like; most of the sample solution goes down the drain which leads to loss of sensitivity in the determination of a given analyte. Further, special precautions are necessary to avoid a flash back as the fuel and oxidant both are present and well mixed in the mixing chamber before combustion.

b) Total consumption burner

A total consumption burner combines the functions of nebuliser and burner. In this type of burner, the suction created by the compressed oxidant streaming past the inner capillary, introduces the sample directly into the flame even if it included suspended particles or large solvent droplets; hence the name "**total consumption burner**". Here the oxidant and fuel emerge from separate ports and are mixed above the burner orifices through their turbulent motion. The flame produced by such a burner is **turbulent**. As the aspirated the sample solution as such is put into the flame, IUPAC recommends the term **direct-injection burner** in place of total consumption burner. However, due to its prevalent use the earlier term still continues to be in use. A schematic diagram of total consumption burner is given in Fig 11

Fig. 11: Schematic diagram of a total consumption burner

The total consumption burner has the following advantages. • There are no losses by condensation of spray; all the material enters the flame. For this reason, the emission for a given concentration of analyte in solution is higher than that from a premix flame (with indirect atomisation).

• No fractionation of the sample takes place during aspiration. This eliminates errors due to loss of nonvolatile components.

• Combustible sample e.g. petroleum can be directly aspirated into the flame without any danger of explosion. However, the total consumption burner suffers from the following disadvantages.

• Droplets of widely varying sizes are formed during aspiration. Many of the larger droplets are frequently blown right through the flame without totally evaporating or without pyrolysis of the solute.

• The flame is more strongly cooled by heavier load of the liquid.

• The burner tip can become incrusted with salts left after evaporation of solvent leading to a change in the aspiration rate of the solvent.

• The burners are very noisy both physically and electronically which lead to poor reproducibility of analytical results. For the above reasons, the total consumption burner is not much used except for explosive flames e.g. hydrogen with other oxidants.

6.3 Monochromator

You have learnt about monochromators in Unit 2 on UV-VIS spectrophotometry. Generally a grating or a prism monochromator is employed. The role of the monochromator is to disperse the radiation coming from the flame and falling on it. The dispersed radiation from the exit slit of the monochromator goes to the detector. In case a low temperatures flame is used, the spectral lines from only a few elements are emitted. In such a case, for most routine analyses, a filter can be used as a monochromator to isolate a particular spectral line. Filters are generally made from materials which are transparent in a small selective wavelength region. The filter chosen is one which has a wavelength range in which it is transparent to emission from the element of interest. In such a case, a condenser lens system is employed to collect the emitted light and send the rays through the filter as an approximately collimated (parallel) beam to reach the detector. Filters have been designed for use in the determination of lithium, sodium, potassium, calcium and other elements.

6.4 Detector

The function of a detector is to measure the intensity of radiation falling on it. Photoemissive cells or photomultiplier tubes are commonly employed for the purpose. These detectors are also used in UV-VIS spectrophotometers. You have learnt about these in Unit 2. You are advised to have a relook at subsection 2.4.3 to recall the functioning of different types of detectors.

6.5 Amplifier and Readout Device

The output from the detector is suitably amplified and displayed on a readout device like a meter or a digital display. The sensitivity of the amplifier can be changed so as to be able to analyse samples of varying concentrations. Nowadays the instruments have microprocessor controlled electronics that provides outputs compatible with the printers and computers thereby minimising the possibility of operator error in transferring data..