Single Beam Spectrophotometer.

A single beam spectrometer records the energy of the radiation reaching the detector after passing through a sample and spectrometer for each spectral element along the scanning range. These are simple, sensitive, accurate, versatile and are used to study fine details but have following **disadvantages.**

- *When the spectra of the solution is recorded, the absorption bands due to solvent are also obtained, thus making the interpretation of bands more difficult.*
- The base line (line obtained without the use of the sample in the light path) slopes because the intensity of the source changes continuously with the wavelength.

DOUBLE BEAM SPECTROPHOTOMETER

The optical null and ratio recording spectrophotometers are most common. The main difference between these two instruments lies in the manner in which the energy measured by the detector is recorded as spectra.

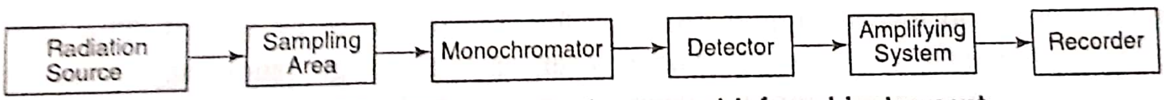

Fig. 14. Schematic diagram of a general infrared instrument.

Components of Double Beam Infrared Spectrophotometer.

1. Radiation source 2. Monochromator and optical material 3. Sampling area
4. Detector 5. Instrument for the measurement of response of the recorder.

1. Radiation Source.

Infrared radiation is produced by electrically heating a source, usually a Nernst glower or a **Globar** to 1273–2073K. The Nernst filament is fabricated from a binder and oxides of thorium, cerium, zirconium and yttrium. The disadvantage of Nernst glower is that it is non conducting at ambient temperature. Globar is a small rod of silicon carbide usually 5 cm in length and 0·5 cm in diameter. The maximum radiation for the Globar occurs in the 5500–5000 cm^{-1} region and drops off by a factor of about 600 as the 600 cm^{-1} region is approached. Nichrome wire, carbon arc, rhodium wire and tungsten filament lamp are also used as light source.

2. Monochromator and Optical Material.

The separation of desired frequencies can be achieved by means of monochromators. Prisms and gratings are used for this purpose. A monochromator (i) *disperses the radiation according to its wave number components, (ii) restricts the radiation falling on the detector into a narrow wave number range, (iii) maintains the energy incident on the detector to a constant level when sample is present throughout the wave number range of the instrument.* Most IR spectrophotometers use prisms of alkali halides. Prisms of LiF or CaF_2 give more resolution in the region where the significant stretching vibrations are located. However, recent made spectrometers are of grating type. Prisms, windows and cell faces are cut from crystals of optical materials such as KBr, CsI and LiF etc. The most common prism material used is NaCl (rock salt) for the entire region from 4000 – 650 cm^{-1} (2·5 – 15·4 μ). *Crystalline KBr and CsBr are satisfactory for the* **far infra red region** (15 μ – 40 μ) while LiF provides prism material in the near infra red region (1 to 5 μ). CaF_2, BaF_2, AgCl, fused silica and glass are also used as optical materials.

Prisms suitable for use below 250 cm^{-1} are not commercially available. It is, therefore, necessary to use gratings in order to obtain spectra below 250 cm^{-1}. *Gratings disperse the energy into more than one order and the undesirable wavelengths are separated with high-quality filters.* **Reflecting gratings** have several advantages as dispersing elements such as (i) *Gratings provide more nearly linear dispersion. (ii) They resist the attack by water. (ii) Prisms or filters can be employed in conjugation with the grating.*

Both transmission and interference filters have been developed for infra red region.