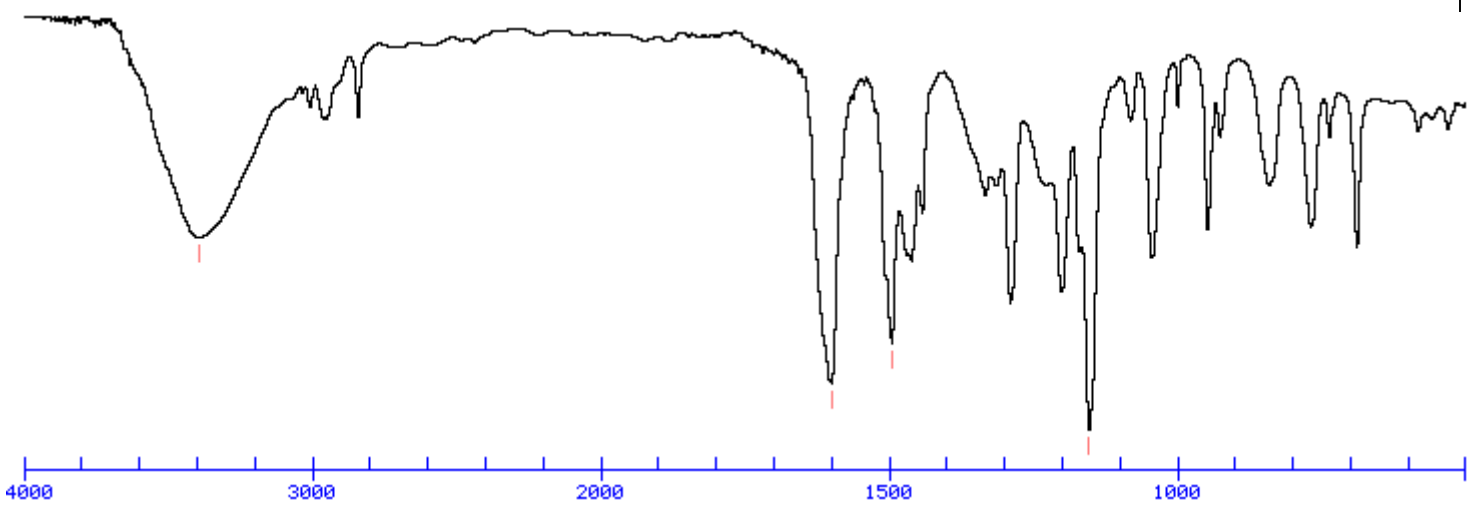
Infrared Spectroscopy

Sample Preparation

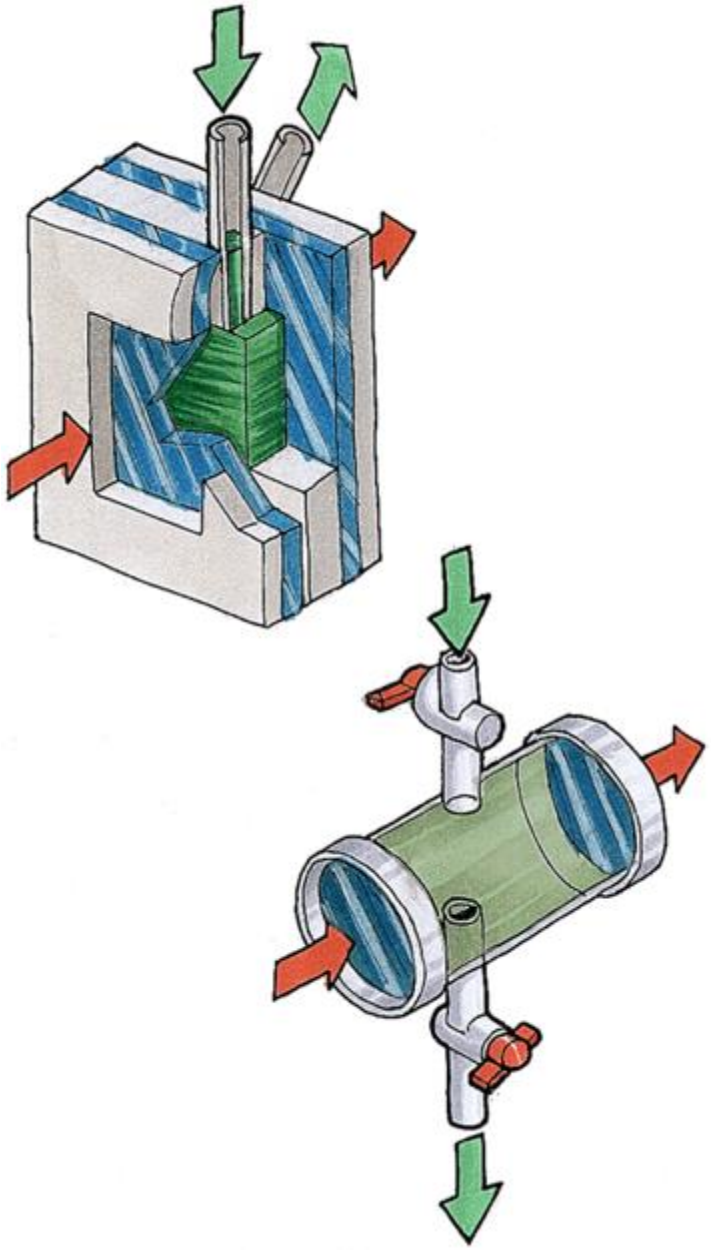


Sample Preparation

- There are a variety of techniques for sample preparation dependent on the physical form of the sample to be analyzed.
- Most time consuming part in IR analysis.

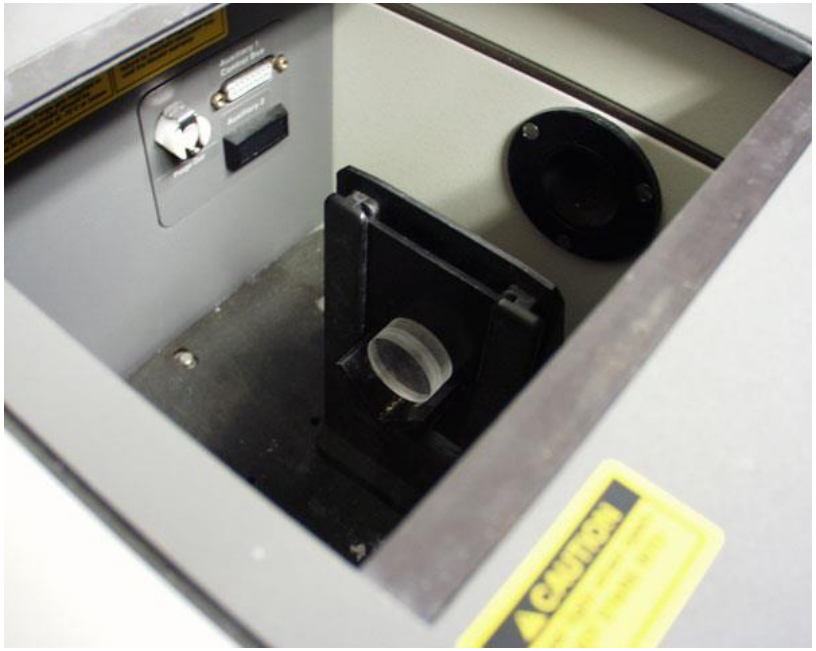
Gases

- Getting an infrared spectrum of a gas requires the use of a cylindrical gas cell with windows at each end composed of an infrared inactive material such as KBr, NaCl or CaF2.
- The cell usually has an inlet and outlet port with a tap to enable the cell to be easily filled with the gas to be analyzed.



Liquids

- This is possibly the simplest and most common method of sample preparation.
- A drop of the sample is placed between two potassium bromide or sodium chloride circular plates to produce a thin capillary film. The plates are then placed in a holder ready for analysis.



Solids

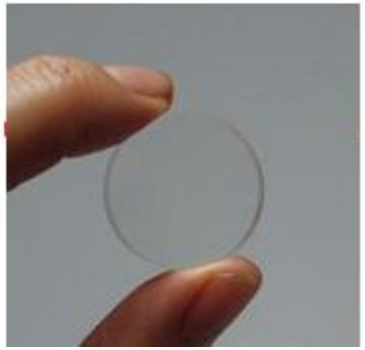
1) Nujol Mull

- The sample is ground using an agate mortar and pestle to give a very fine powder.
- A small amount is then mixed with nujol to give a paste.
- several drops of this paste are then applied between two sodium chloride plates (these do not absorb infrared in the region of interest).
- The plates are then placed in the instrument sample holder ready for scanning.

Mix sample with liquid (mineral oil)

Spread it over one of the pair of NaCl plates

Make a sandwitch of sample between two plates



2) Potassium Bromide disk

- A very small amount of the solid (approximately 1-2 mg) is added to pure potassium bromide powder (approximately 200 mg) and ground up as fine as possible.
- This is then placed in a small die and put under pressure mechanically.
- The pressure is maintained for several minutes before removing the die and the KBr disk formed. The disk is then placed in a sample holder ready for scanning.
- The success of this technique is dependent on the powder being ground as fine as possible to minimize infrared light scattering off the surface of the particles. It is also important that the sample be dry before preparation. KBr has no infrared absorption in the region 4000 - 650 cm-1

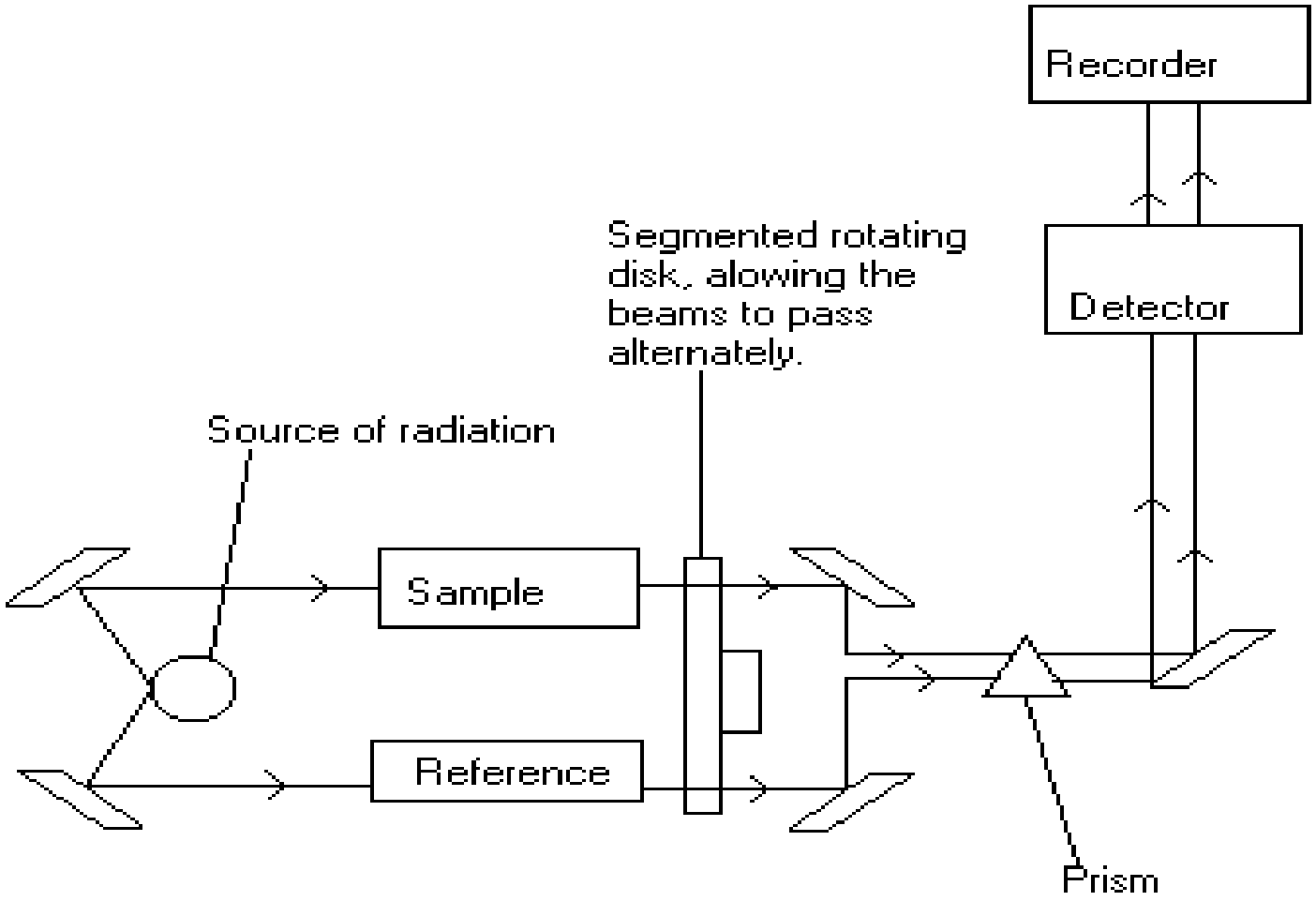
Sources of IR instrument:

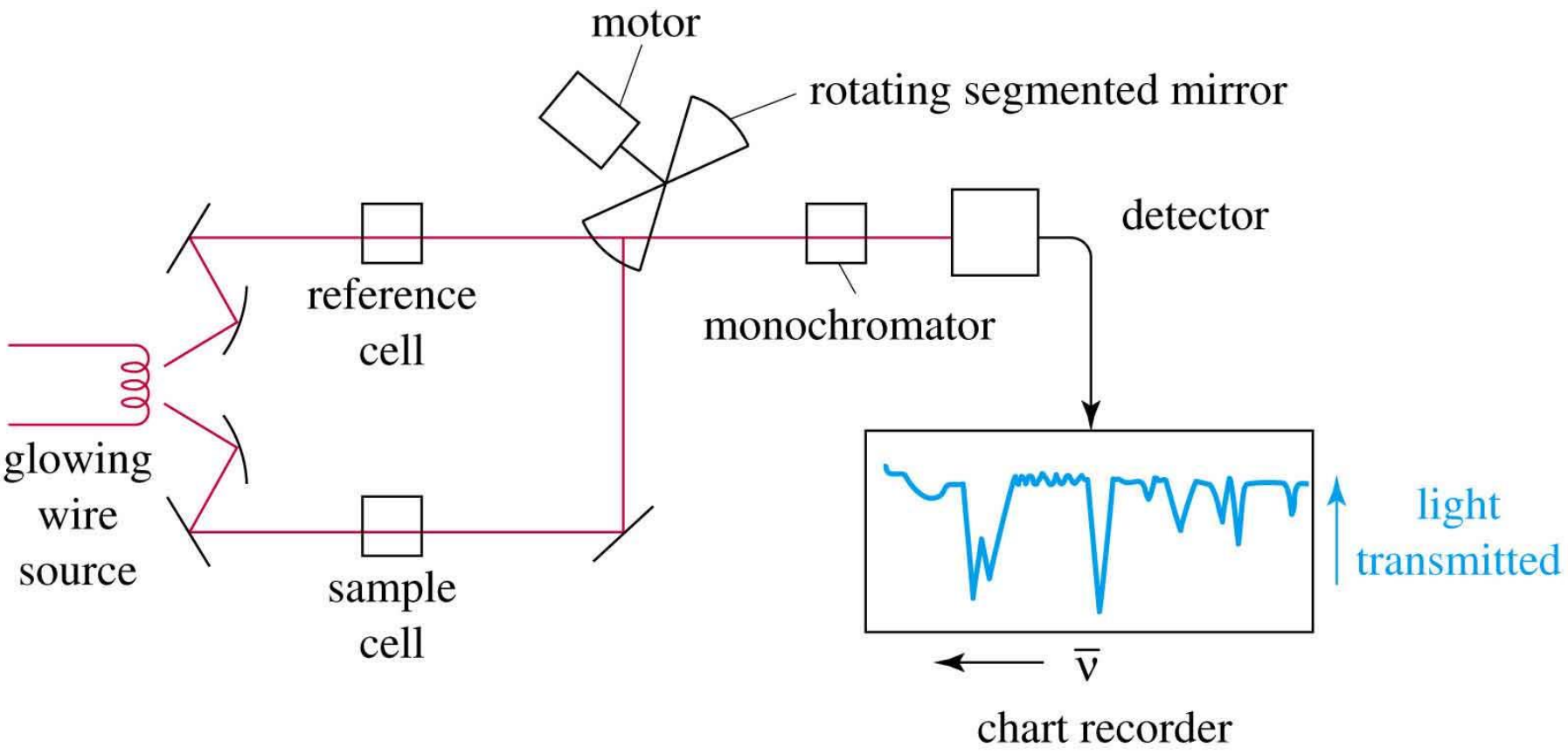
Nernst Glower	1-10 µm
Globar	1-10 µm
W filament lamp	0.78-2.5 µm
Hg arc lamp	>50 µm
CO_2 laser	9-11 µm

Instrumentation

1) The Double Beam Infrared Spectrometer.

- This instrument uses a source of infrared radiation such as a nichrome wire or cooled rod of silicon carbide
- The beam produced is then split into two, one passes through the sample whilst the other is used as a reference beam.

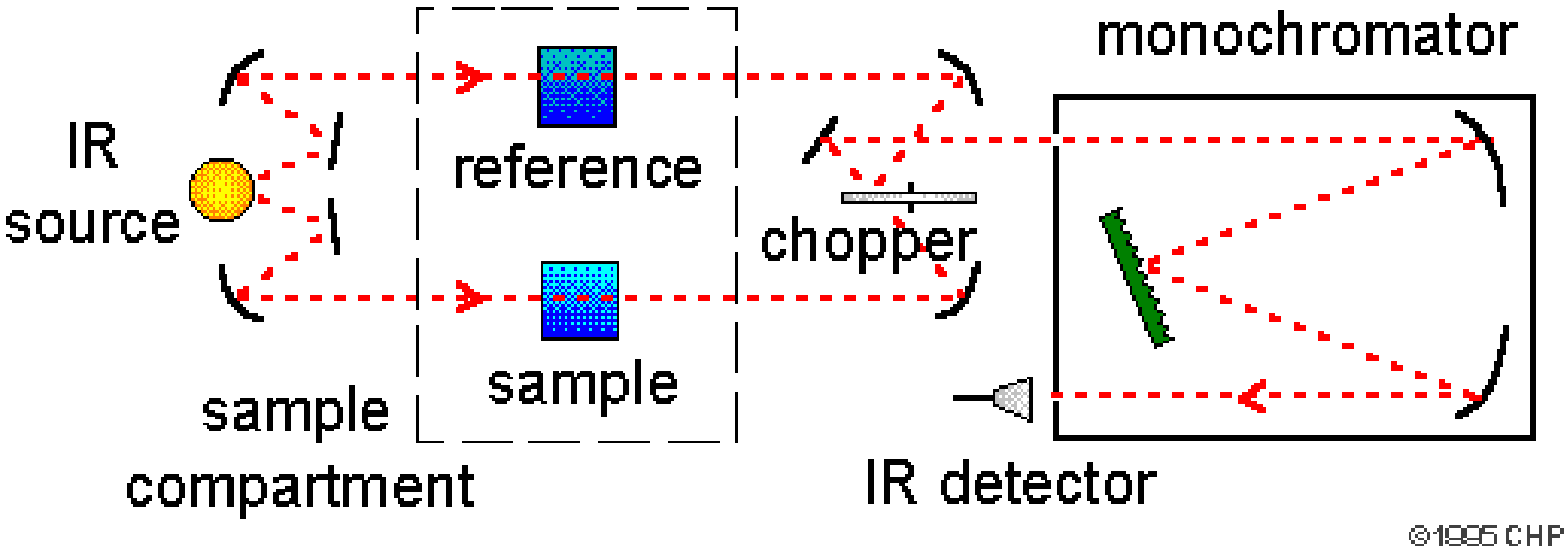

Recorder
Detector
Segmented rotating disk, alowing the beams to pass alternately.
Source of radiation
Sample
Reference
Prism


motor
rotating segmented mirror
detector
reference cell
monochromator
glowing wire source
sample cell
light transmitted
$\bar{\nu}$
chart recorder

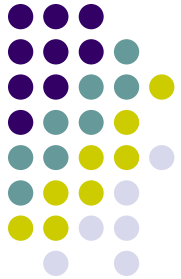
2) Fourier Transform Infrared Spectrometer (FTIR Spectrometer)

- Has better sensitivity.
- Less energy is needed from source.
- Completes a scan in 1-2 seconds.
- Takes several scans and averages them.
- Has a laser beam that keeps the instrument accurately calibrated.
- A reference or “background” single beam is collected first without a sample and the sample single beam is ratio-ed to the background single beam to produce a transmittance or “%T” spectrum

FTIR


monochromator
IR
source
reference
chopper
sample
sample
compartment
IR detector
©1995 CHP

Quantitative Analysis



IR is more difficult to be used in quantitative analysis than UV/Vis because:

- Narrow Bands.
- Complex Spectra
- Weak Incident Beam
- Low transducer sensitivity
- Solvent absorption

Qualitative analysis

- Identify functional groups (functional group region).
- Compare with standard spectra containing these functional groups (fingerprint region) to identify the compound.