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FTIR Spectrometer

- Basic Theory of Infrared Spectrometer
- FTIR Spectrometer
- FTIR Accessories

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What is Infrared?

- Infrared radiation lies between the visible and microwave portions of the electromagnetic spectrum.
- The Infrared region is divided into: near, mid and far-infrared.
  - Near-infrared refers to the part of the infrared spectrum that is closest to visible light and far-infrared refers to the part that is closer to the microwave region.
  - Mid-infrared is the region between these two.

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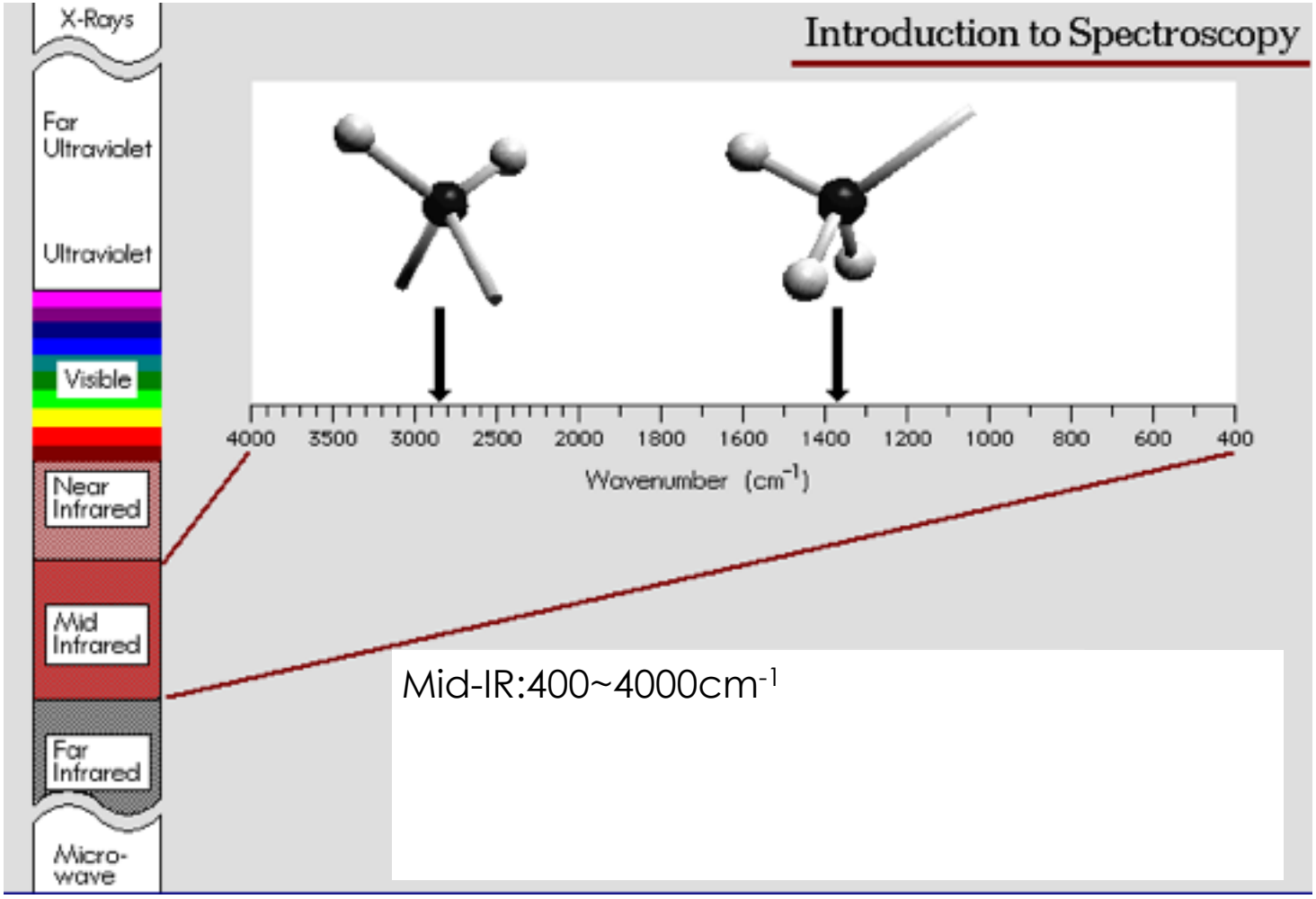
Wavelength Range of Mid-IR



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The Infrared Radiation

- It is the radiation produced by the motion of atoms and molecules in an object.
- However, not all infrared light is absorbed by the molecule.
- Very specific frequencies are absorbed by specific groups or vibrations of the molecule.

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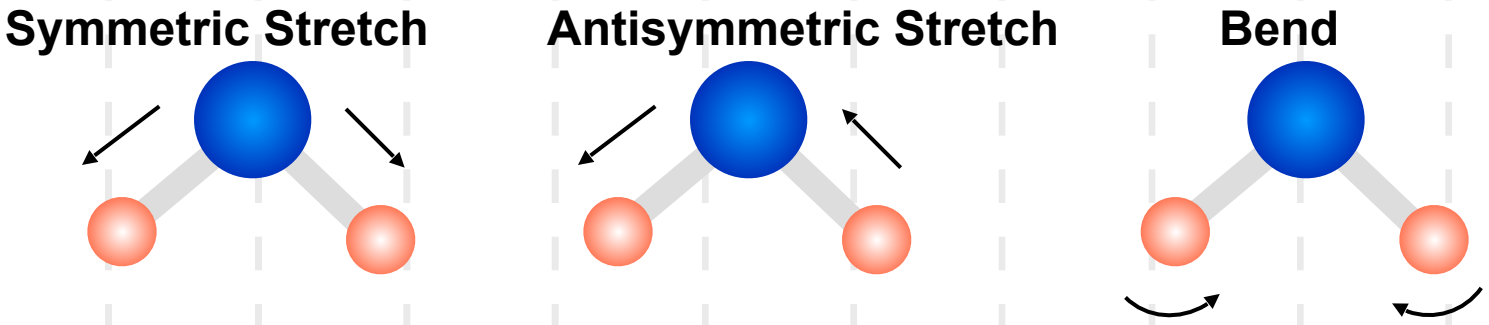
Selection Rules for Infrared Activity

The absorption is basically depending on these properties:

- The frequency of the infrared radiation must be identical to the (theoretical) frequency of the vibration of the molecule. We call this resonance.
- The dipole of the molecule, the distribution of the electrons in the molecule, has to change during the vibration. This means that even though every molecule vibrates all the time, not all vibrations absorb IR radiation.

Vibration of Molecules

- Each vibration, called a fundamental mode, has a specific energy (or frequency) which depends on the atoms involved in the motion (ie, mass of the weights), the type of motion, and the bond strengths (ie, the strength of the “spring”).



- The bonds between atoms in the molecule stretch and bend, absorbing infrared energy and creating the infrared spectrum.

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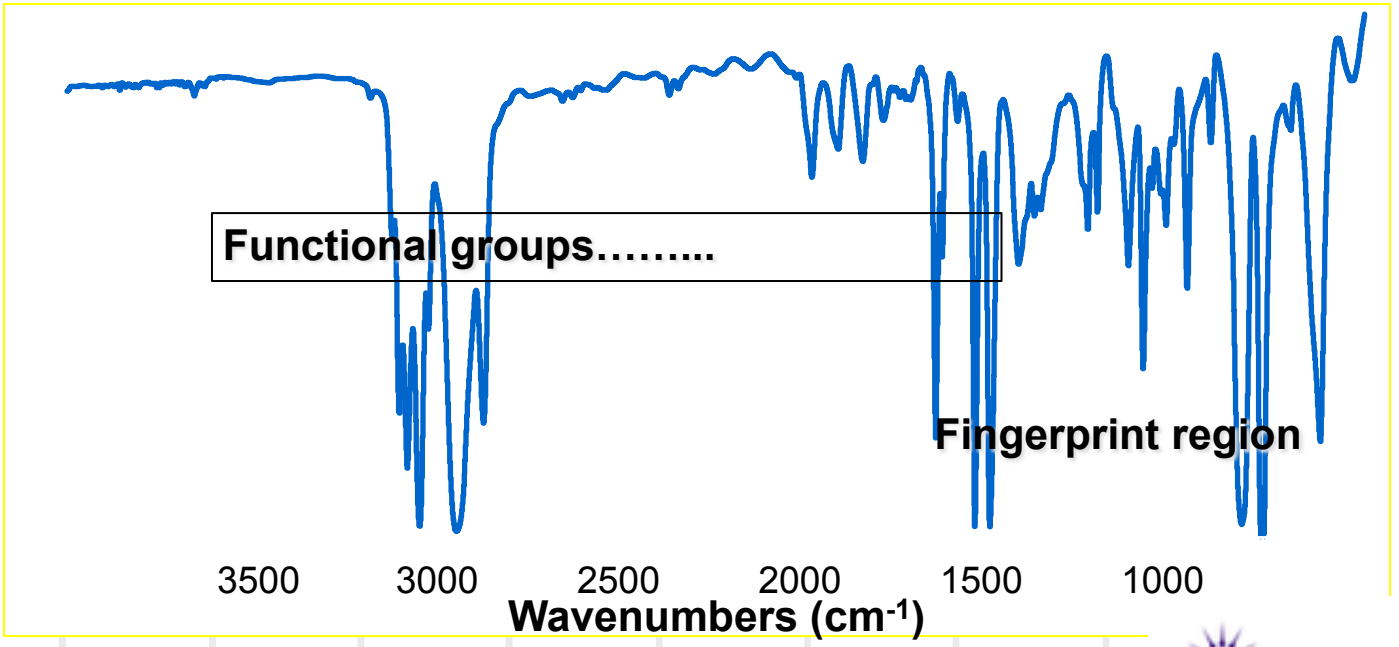
Functional Groups and Fingerprint

- In Mid-Infrared region, the frequency of vibration is divided into two portions: functional groups and fingerprint.
- Functional groups always occur at similar positions in the spectrum: OH vibrations, CH_2 vibrations, Aromatic vibrations.
- These vibrations are spread over the entire spectrum, and most of them are pretty well known.
- The region between 1350 and 650 cm^{-1} is basically built up from very specific complicated vibrations of the entire molecule.
- Therefore, this region is different for every molecule, and like a fingerprint, no two molecules have the same fingerprint region.
- We can use this part of the spectrum for the final identification.

The Region of Functional Groups and Fingerprint

- Functional groups region(4000~1350cm^{-1}): It is linked to functional groups in molecule.
- Fingerprint region(1350~650cm^{-1}): It is related to whole molecular structure.
- This is the most well-known spectrum in IR spectroscopy: A polymer with CH_2 and benzene groups.

Polystyrene



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Capabilities of Infrared Analysis

- Identification and quantitation of organic solid, liquid or gas samples.
- Analysis of powders, solids, gels, emulsions, pastes, pure liquids and solutions, polymers, pure and mixed gases.
- Infrared used for research, methods development, quality control and quality assurance applications.
- Samples range in size from single fibers only 20 microns in length to atmospheric pollution studies involving large areas.

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Applications of Infrared Analysis

- Pharmaceutical Research
- Forensic Investigations
- Polymer Analysis
- Lubricant Formulation and Fuel Additives
- Foods Research
- Quality Assurance and Control
- Environmental and Water Quality Analysis Methods
- Biochemical and Biomedical Research
- Coatings and Surfactants
- Etc.

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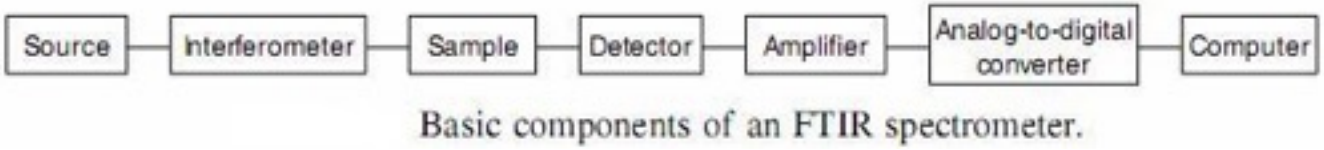
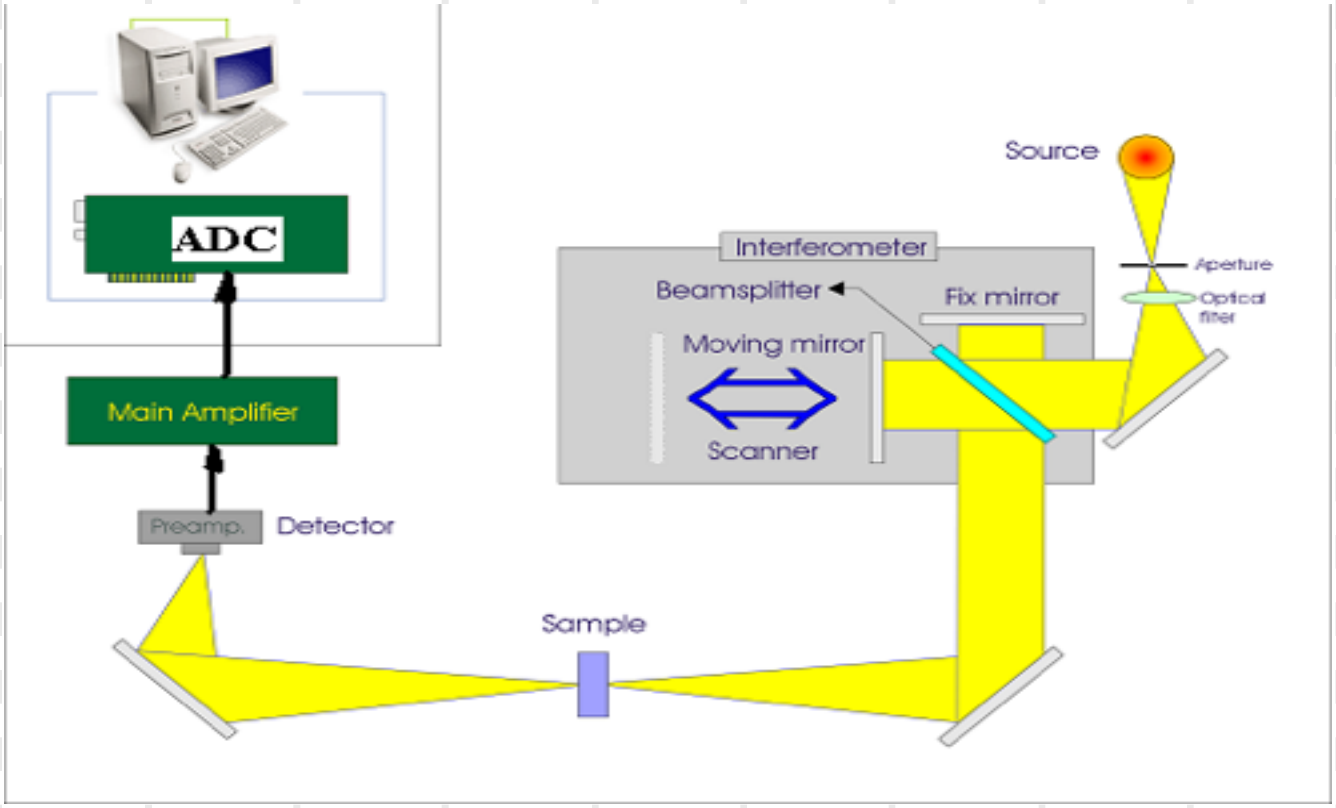
The Basic Components of FTIR Spectrometer



Basic components of an FTIR spectrometer.



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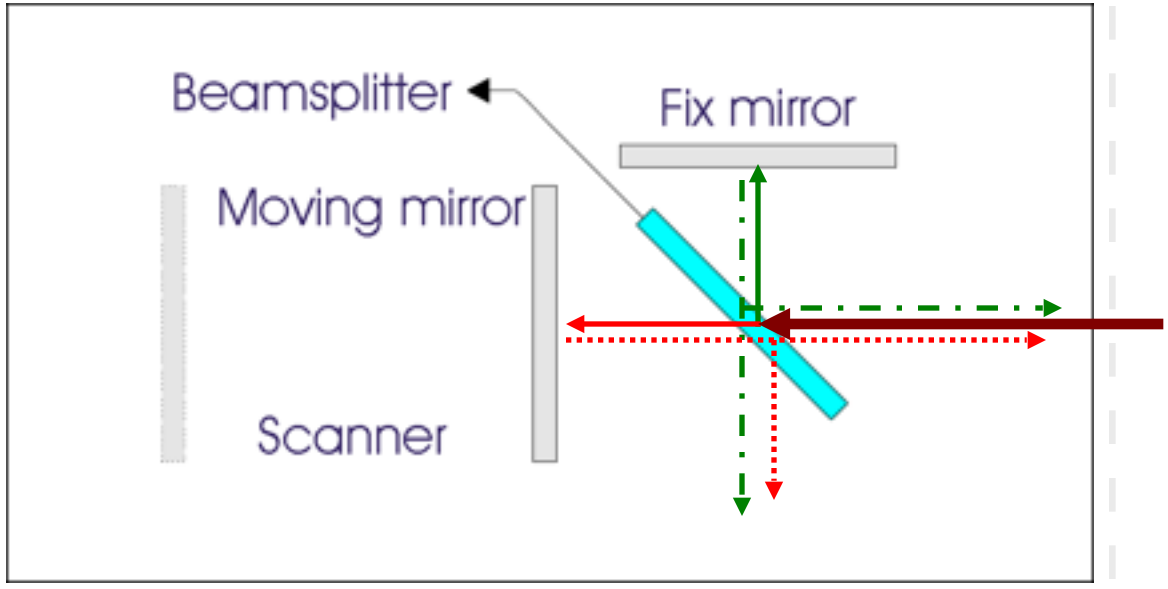
The Interferometer

- IR radiation passed into beamsplitter,50% of IR radiation be transmitted to the moving mirror,50% be reflected to the fix mirror. The two beams are reflected from these two mirrors, returning to the beam splitter where they recombine and interfere.



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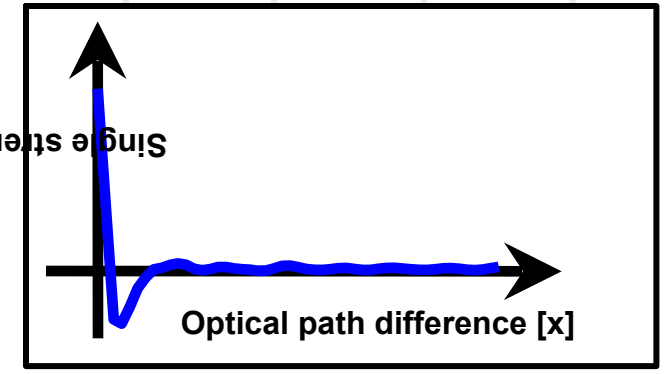
Fourier-Transform Infrared Spectrometers

- Fourier-transform infrared spectroscopy is based on the idea of the Interference of radiation between two beams to yield an interferogram.
- The interferogram is a signal produced as a function of the change of pathlength between the two beams.
- The two domains of distance and frequency are interconvertible by the mathematical method of Fourier-transformation.
- The radiation emerging from the source and passed through an interferometer to the sample before reaching a detector.

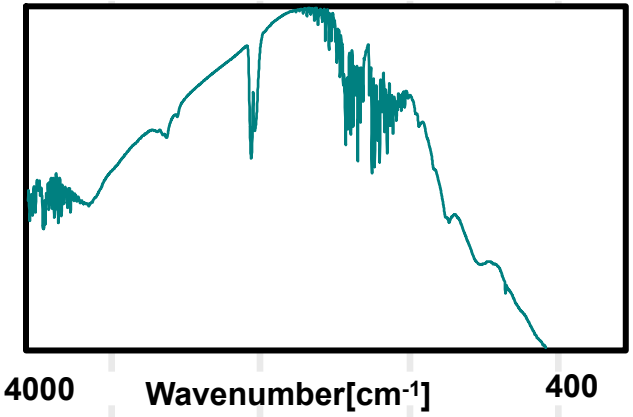
Fourier Transform

- Integrate the frequency, obtain the intensity of power.



spectrum

- And then obtain the intensity of power by the mathematical method of Fourier-transformation.



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Advantages of FT Instruments

Why would we rather use FT instruments than dispersive ones? There are three major advantages of FT instruments:

- Multiplex: since all wavelength are measured simultaneously ,the measurement time is greatly reduced.
- Throughput: FT instruments don't use slits, therefore the amount of light entering into the instrument is much larger, and the measurement is less sensitive to noise.
- Precision: since we use an extremely accurate laser for the position determination of the mirror, the precision is very high, better than 0.01 cm^{-1}. This is better for quantitative analysis and library searching.

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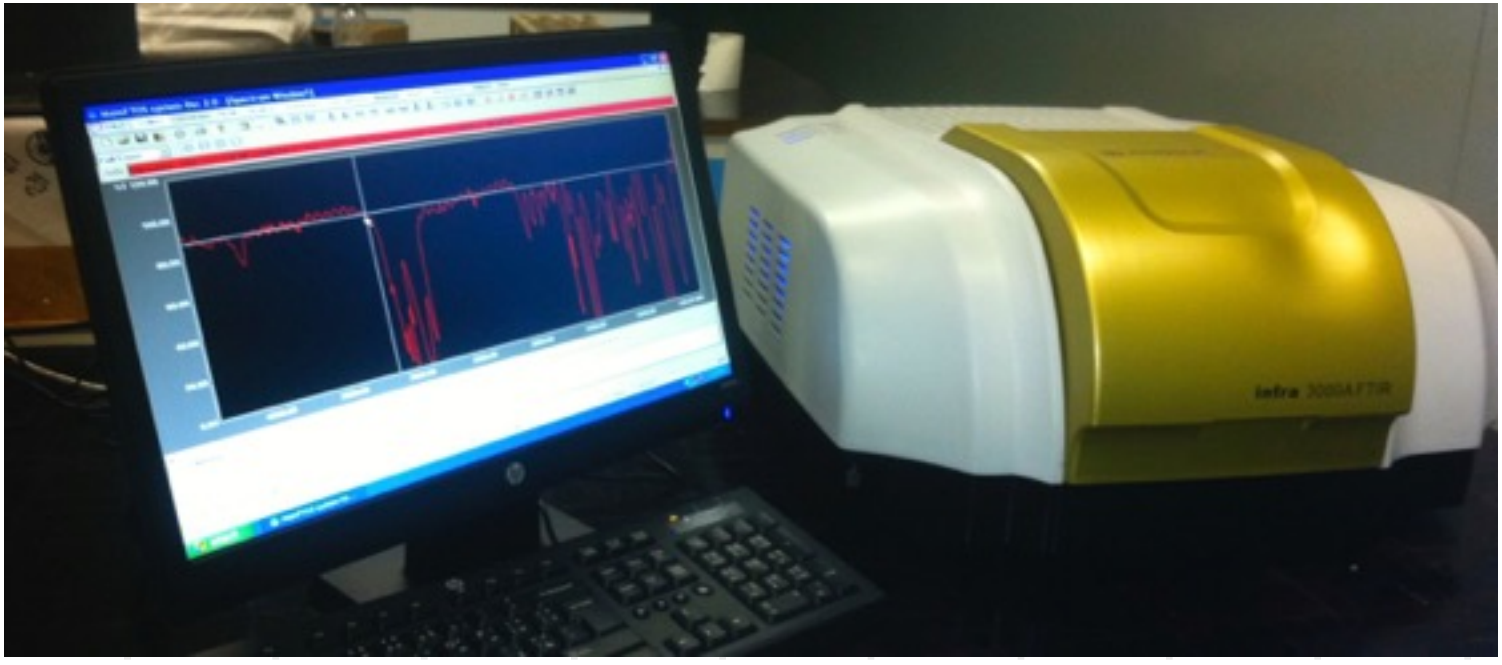
infra 3000 Series FTIR Spectrometer

- **Spectral range:**7800cm^{-1}~400cm^{-1}
- **Resolution**:
  better than 1cm^{-1}(3000A)
  better than 0.5cm^{-1}(3000B)
- **Wavenumber accuracy**:±0.01cm^{-1}
- **Signal to noise ratio:** better than 15000:1
- **Beam splitter**: Ge be coated on KBr
- **Detector**: DTGS,MCT(optional)
- **IR source:** air-cooled, high efficiency



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Accessories

- Hydraulic Press
- Gas Cell
- Diffuse/Specular Reflectance
- Liquid Cell
- Attenuated Total Reflection

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Hydraulic Press

- Generally, you need to get a spectrum of powder samples through halogenide tablet pressing method.
- The most common preparation method is KBr tablet pressing method. (there are about 90% spectrums in pharmacopoeia selecting this method.)
- Hydraulic press is the essential tool for halogenide tablet pressing method.
- Samples are pressed to a transparent tablet after grind.
- It can get a very good infrared spectrum through transmitting method.

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KBR Pellet for Solids

- To mix a solid sample with dry KBR powder. The little sample is needed and around 1 to 2mg sample with about 200mg KBR.
- Place mixed powder in the mould, and subjected to a pressure of 8~10MPa with one Minute.
- Sample and KBR must be dry to avoid the effect from water.
- The diameter of particles must be less than 0.2um, in order to avoid scattering results.

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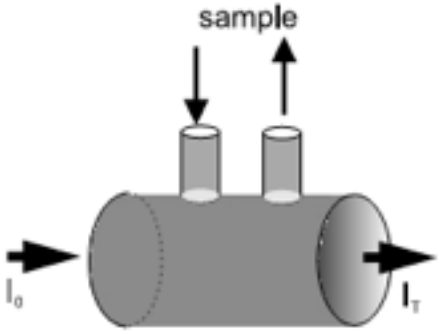
Gas Cell



Gas Cell

- Generally, it is composed by a glass cylinder with two pistons for gas in or out and two windows transmitting infrared. The length is 50mm or 100mm. The diameter is 30~40mm.
- The cylinder must be pumped into vacuum state first when sampling, then blow into the gas sample.
- The volatile liquid sample can also be put into the cell to measure its steam spectrum.

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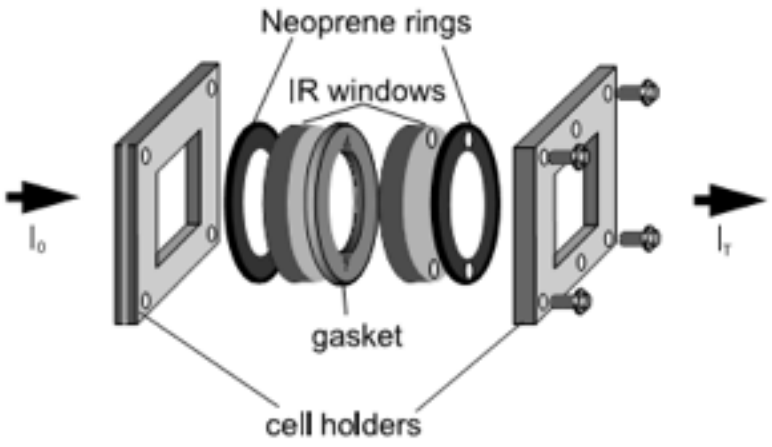
Liquid Cell



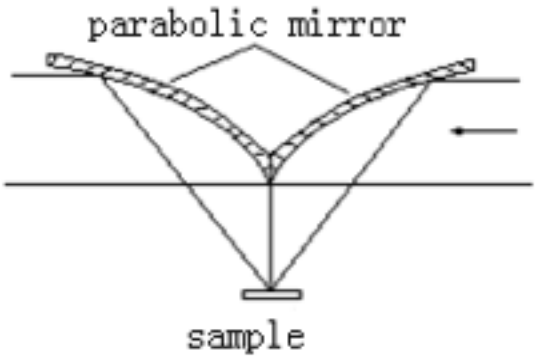
Fixed Liquid cells

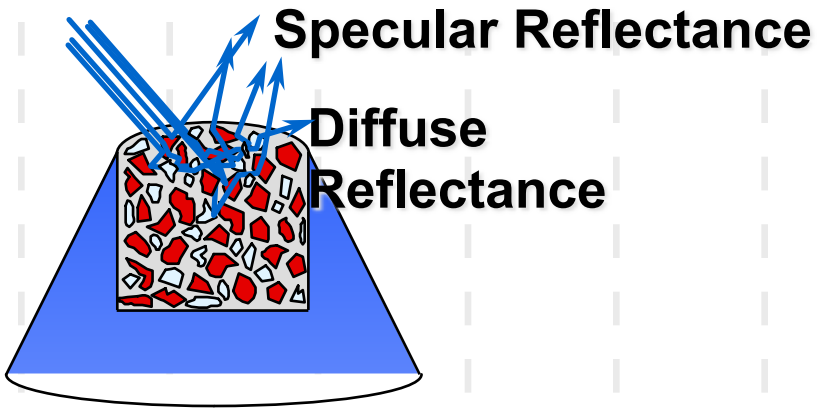
- Fixed liquid cell, that is to say the thick of the cells is fixed. It is used for qualitative or quantitative analysis of volatile liquid and solution. The common material of windows is NaCl, KBr, CsI, KRS-5 and so on. The padding between the windows is Al, Pb or PTFE. The common thickness is 0.025, 0.05,0.1 , 0.2, 0.5, 1.0mm.

Demountable liquid cell

- When analysing non-volatile liquid sample, you can choose Demounted cells.
- It is composed by a metal holder and two windows between which there is a suitable neoprene or PTFE padding.
- Put the liquid into the cells with syringe when measuring.
- Please open the cells to clear when finishing measurement or changing sample.

Diffuse Reflectance





- When the incident light get to sample surface, may be partly reflected regularly, partly scattered diffusely, and partly enter the substrate. The latter part may undergo absorption with particles, emerge at the sample surface and intermingle with reflected parts.

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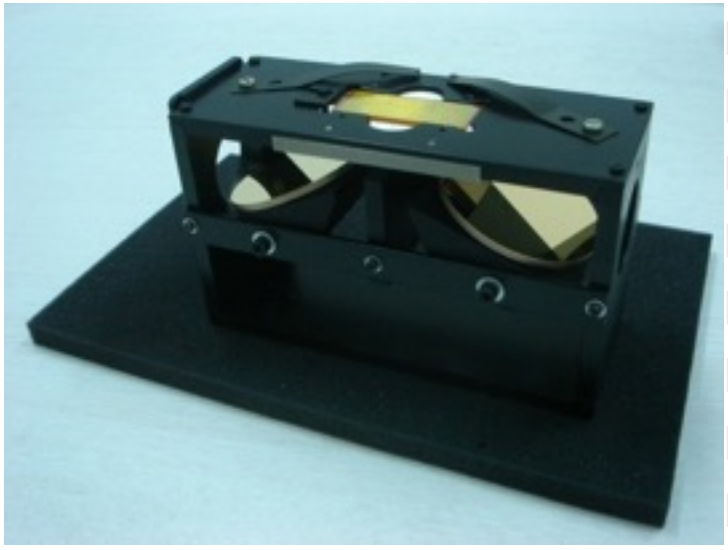
Attenuated Total Reflection(ATR)



- Horizontal attenuated total reflectance accessory is used in analysis of liquid, semi-liquid materials and a number of soft solids.
- In general, sampling is achieved by placing the sample onto the HATR crystal—generally eliminating sample preparation.

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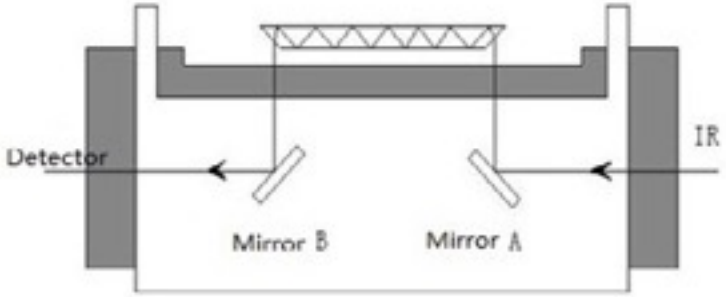
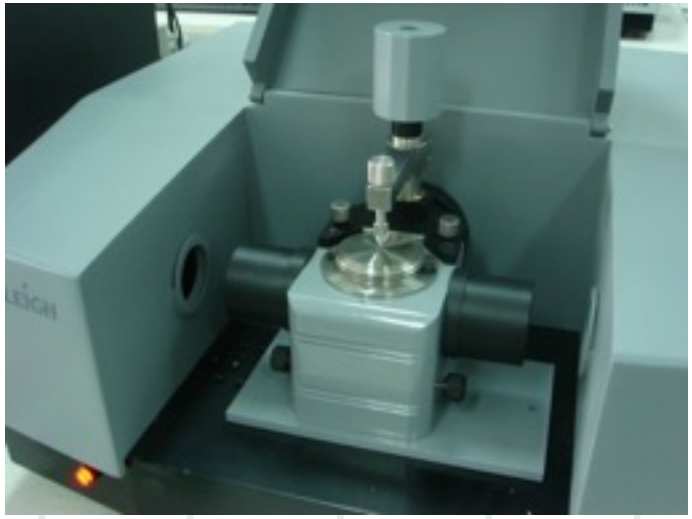
Horizontal ATR



- A beam of radiation entering a crystal will undergo total internal refection when the angle of incidence at the interface between the sample and crystal is greater than the critical angle ,where the latter is a function of the refractive indices of the two surfaces .the beam penetrates a fraction of a wavelength beyond the reflecting surface, when a material that selectively absorbs radiation is in close contact with the reflecting surface ,the beam loses energy at the wavelength where the material absorbs.

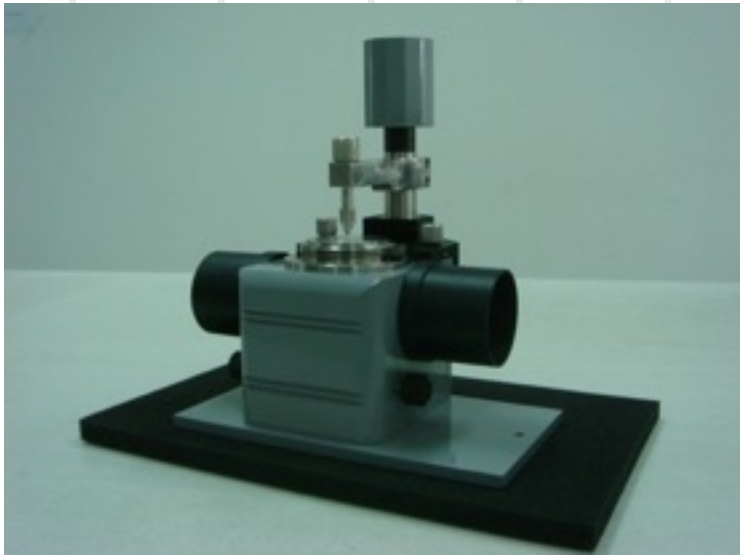
Single- reflection ATR

- Single-reflection ATR further simplify sample preparation and also reduce the complexity of traditional FTIR measurements.
- This is achieved through the reduction of the sampling area, easy of cleaning and the introduction of more rigid, diamond-protected sampling interfaces.



Single reflection ATR

- Capable of withstanding higher pressures and harder samples.
- Capable of analyzing a wide variety of samples-including rigid solids and hard powders-which are difficult to achieve with a multi-reflection ATR.
- Also it works very well for the analysis of relatively strong absorbing substances, (e.g. polymers, rubber, paint chips, fibers, etc.)



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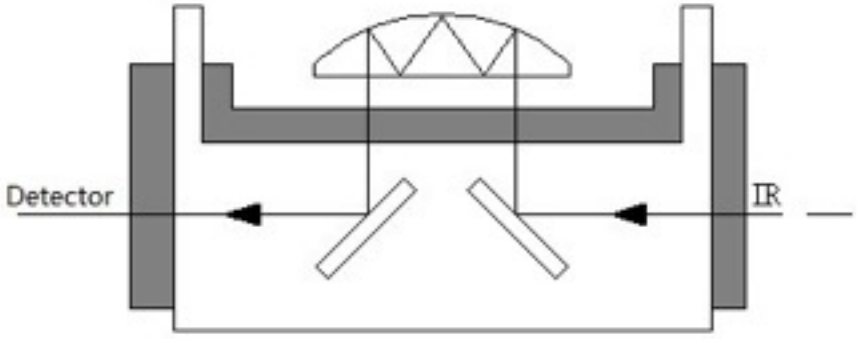
Single-reflection ATR



- It's unique optical design provides exceptional sensitivity and IR throughput.
- The compact design of the accessary employs a transfer mirror to direct the infrared beam to one end of an IR transmitting ATR crystal(or specular reflectance mirrors).
- A second mirror directs the beam emitted from the other end of the crystal to the detector built into your FTIR spectrometer.

Experimental Method

1) Gas —— Gas Cell

2) Liquids
- Volatile Liqiud —— Fixed Liqiud Cell
- Demounted Liquid Cell
- Involatile Liqiud — Attenuated Total Reflection(ATR)
- Coating Liquid on KBr Disc

3) Solids
- Powder
  - d < 0.2um —— Kbr Discs
  - d > 0.2um —— Diffuse Reflectance
- Pliable Solids —— Attenuated Total Reflection(ATR)

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Thanks !