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FT-IR SPECTROSCOPY: PRINCIPLE, TECHNIQUE AND MATHEMATICS

Corresponding Author

S. D. SAWANT

Sinhgad Technical Education Society's Smt. Kashibai Navale College of Pharmacy. Kondhwa (Bk), Pune, India.

Co Authors

A. A. BARAVKAR[1], R. N. KALE[1]

[1]SVPM's College of Pharmacy, Malegaon (Bk), M.S., India.

ABSTRACT

Fourier Transform Infra Red spectroscopy (FT-IR) has wide applicability in structure elucidation, which are either synthesized chemically or of natural origin. Now a day, Fourier Transform Infra Red (FT-IR) Spectroscopy is extensively used for quantitative as well as for qualitative analysis in almost all fields of science. It has many advantages and applications as compared to dispersive infra red technology.

KEYWORDS

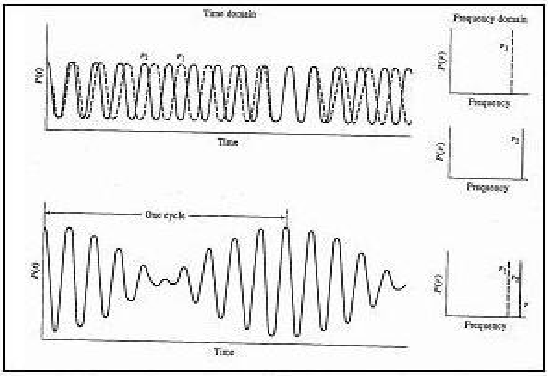
FT-IR spectroscopy, quantitative analysis, qualitative analysis.

INTRODUCTION

In 1887, Albert Michelson (German born American physician) perfected this instrument and used it for several measurements in his study of light and relativity [1].

PRINCIPLE

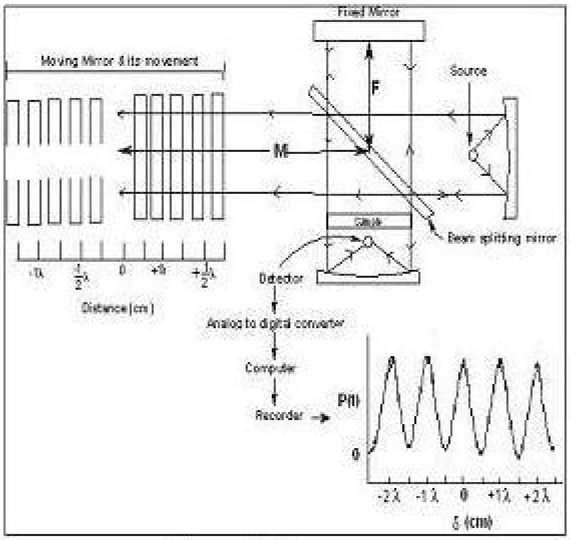
Conventional spectroscopy is frequency domain spectroscopy in which radiant power data are recorded as function of frequency. In time domain spectroscopy, which is achieved by Fourier Transform (FT), radiant power data is recorded as a function of time. In previous case, radiant power (v) is plotted against frequency (v_1) (Hz) while in later, against the time [2].



"Figue 1 : Time domain and frequency domain spectra"

Michelson interferometer (MI) changes the frequency of electro magnetic radiation (EMR) from source to proportionately slower oscillating signal. The sum of slower oscillating signal is carried to the computer which mathematically separates the signal into individual oscillations and calculate the oscillations of corresponding frequencies of observed radiation. This data is continuously recorded. The amplitude of each resolved oscillations is a function of intensity of radiation. A mathematical method called Fourier Transform (FT) is used to convert time domain spectrum to conventional frequency domain spectrum[3].

CONSTRUCTION AND WORKING[3]



"Figure 2 : Instrumentation"

FT-IR consists of a moving mirror, fixed mirror, beam splitter, IR radiation source and detector. Instead of using monochromator, Michelson interferometer is used for analysis of IR radiation after passing through sample. Radiation fro IR source is collimated by mirror and the resultant beam is divided at beam splitter. Half of beam passes through mirror (fixed) and half refracted to moving mirror. After reflection by these two mirrors, two beams recombined at beam splitter and passes through cell and after that radiation is focused to detector.

Movable mirror, moves back and forth at a distance of 21 cm. If round trip distance between beam splitter and fixed mirror is identical to that of beam splitter and movable mirror, then only the radiation from two mirrors arise in phase at beam splitter, cell and to detector. As the movable mirror changes its position, the distance between mirror and beam splitter no longer identical and radiation of fixed wavelength will arrive in phase only to cell and detector.

So we have to add or subtracts whole number of multiple of wavelength of radiation in round trip distance between splitter and fixed mirror. If movable mirror moves by a factor λ/4, then round trip distance is altered by λ/2 reflected radiation if **out of phase** with that from stationary mirror and interferes destructively while movable mirror moves by a factor λ/2, then round trip distance is altered by λ/2 reflected radiation if **in phase** with that from stationary mirror and interferes **constructively**. The radiation striking the detector, after passing through MI will be of lower frequency that source frequency. One cycle of the signal occurs when the mirror moves at a distance that corresponds to half of wavelength (λ/2,). If the mirror is moving at constant velocity V_m and we define τ as the time required for mirror to move distance of λ/2, then we can write [4]

$Vm.\ \tau = \lambda/2$ So, $\tau = \lambda/2\ Vm$

But frequency of signal at detector is

$f = 1/\tau = 2\ Vm/\lambda$

But, $\lambda = C/v$

So, $f = 2\ Vm.\ v/C$ ————————————— 1

v=Frequency of radiation, C=Velocity of light (3×10^{10}cm/s)

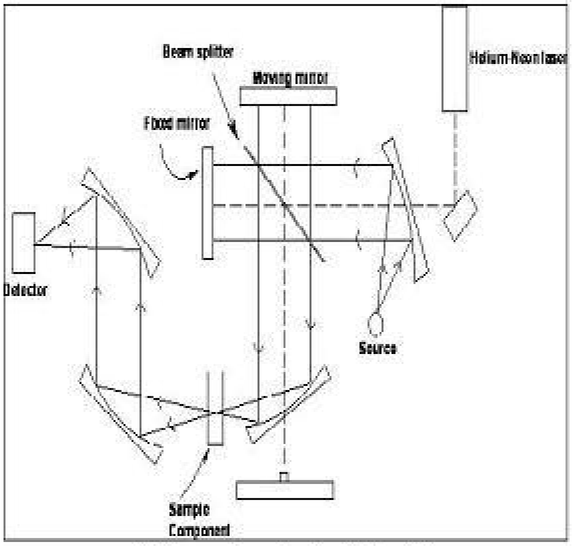
When Vm constant, f (interferogram frequency) is is directly proportional to optical frequency v.

Equation 1 shows the relationship between optical frequency of radiation and the frequency of interferogram (f).

We can relate, f with wave number as follows

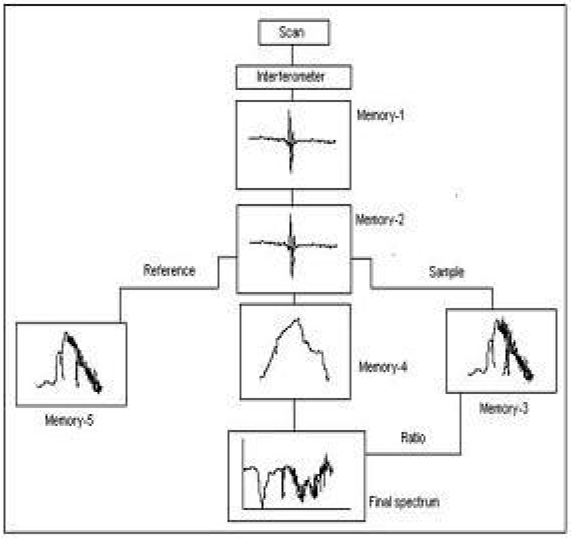
$1/\lambda = \bar{v}$ = Wave number of radiation

So, $f = 2\ Vm.\ \bar{v}$



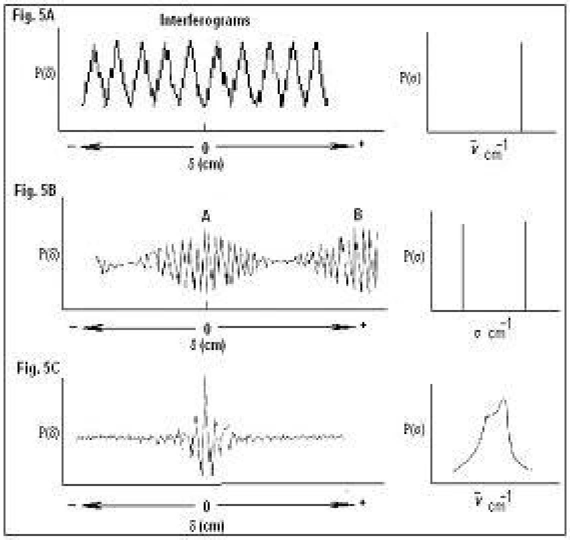
"Figure 3 : Optical Path"

So as the distance changes, wavelength of radiation beams becomes in phase or out of phase depending on wavelength of incident radiation and rate at which movable mirror moves. So that by controlling the rate of mirror motion a series of simultaneous signals that oscillate frequency which is directly proportional to EMR frequency arrive at detector and oscillate slowly for detector to measure. The detector simultaneously measures all of frequencies that pass through the cell and routes the information to computer and this information is decoded by FT and decoded spectrum is directed to read out device. Time from insertion of sample to recording of plot is about 2 min.



"Figure 4 : Block diagram of instrument function"

MATHEMATICS OF FOURIER TRANSFORMATION



"Figure 5 : Fourier transformation of interferogram"

The cosine wave of interferogram shown in figure A can be described theoretically by following equation

$P(\delta)=\frac{1}{2}P(\bar{v})\cos 2\pi.f.t$

$P(\bar{v})$=Radiant power of beam

$P(\delta)$ =Radiant power of interferogram signal

But interferometer will not split the source exactly in half. So it is useful to introduce a new variable $B(\bar{v})$ in place of $\frac{1}{2}P(\bar{v})$

So, $P(\delta) = B(\bar{v}) \cos 2\Pi.f.t$

But, $f = 2\,Vm.\,\bar{v}$

So, $P(\delta) = B(\bar{v}).\cos 4\Pi\,.Vm.\bar{v}.t$

Above equation shows that magnitude of interferogram signal as function of wave no. of optical signal.

But, $Vm = \frac{\delta}{2t}$ ----Mirror velocity

So, $P(\delta) = B(\bar{v}).\cos 2\Pi.\delta.\bar{v}$

Interferogram in figure-B can be described by 2 terms, one for each wave number

$P(\delta) = B_1(\bar{v})\cos 2\Pi.\delta.\bar{v}_1 + B_2(\bar{v})\cos 2\Pi.\delta.\bar{v}_2$

Interferogram in figure-C can be presented as a sum of infinite number of cosine terms

$P(\delta) = \int_{+\infty}^{-\infty} B(\bar{v}).\cos 2\Pi.\delta.\bar{v}$

Fourier Transform of this equation is

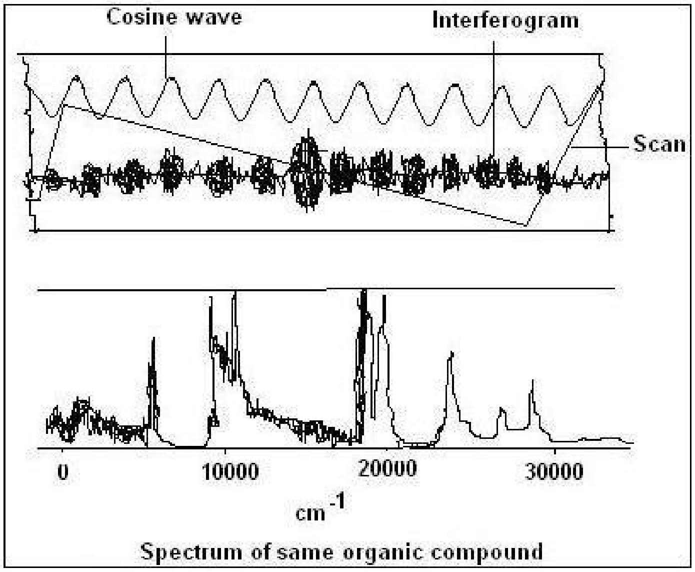
$\mathbf{B(\bar{v}) = \int_{+\infty}^{-\infty} P(\delta).\cos 2\Pi.\delta.d\delta}$

Resolution of FT spectrometer is given by

$\Delta(\bar{v}) = \bar{v}_1 - \bar{v}_2 = \frac{1}{\delta}$ --2

$\bar{v}_1 - \bar{v}_2$ =Wave number for resolved lines.

Equation 2 shows that resolution is inversely proportional to distance travelled by mirror.



Spectrum of same organic compound

"Figure 6 : Presentation of interferogram"

DIFFERENCE BETWEEN DISPERSIVE IR AND FT-IR [2]

ADVANTAGES OF FT IR:

1) Fellgett's advantage[5]-It makes use of all frequencies from the source simultaneously, rather that sequentially as in scanning instruments/dispersive IR. So that all wavelengths are detected through out the scan and so MI gives same spectral signal to noise (S/N) ratio as in dispersive IR this ratio occurs in fractions of time.

2) Jacquinot advantage[5]- High sensitivity than dispersive IR because more radiation enters slit less system.
3) Only one moving part involved. So less / no slippage and wear.
4) Dispersion / filtering is not required. So slits are not required.
5) Better absolute frequency accuracy (0.01 cm^{-1}) due to use of Helium-neon laser as a reference, which provides red light of wavelength of 632.8 nm for continuous spectral calibration.
6) Intensity of radiation falling on detector is much much more.
7) Flexibility in spectral print outs (Multiplex advantage)[4].

APPLICATIONS OF FT-IR:

1) Mainly used for quantitative analysis due to Fellgett's advantage.
2) In astronomical measurements.
3) In case where spectra and low concentration of sample is required.
4) Used where the spectrum is required to obtain quickly.
5) Can be used as a detector for chromatography.

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