

**Indian Institute of Technology
Kanpur**

**NP-TEL
National Programme
on
Technology Enhance Learning**

**Course Title
Advanced Characterization Techniques**

Lecture-18

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So far I had discussed about infrared spectroscopy as a part of the different techniques.

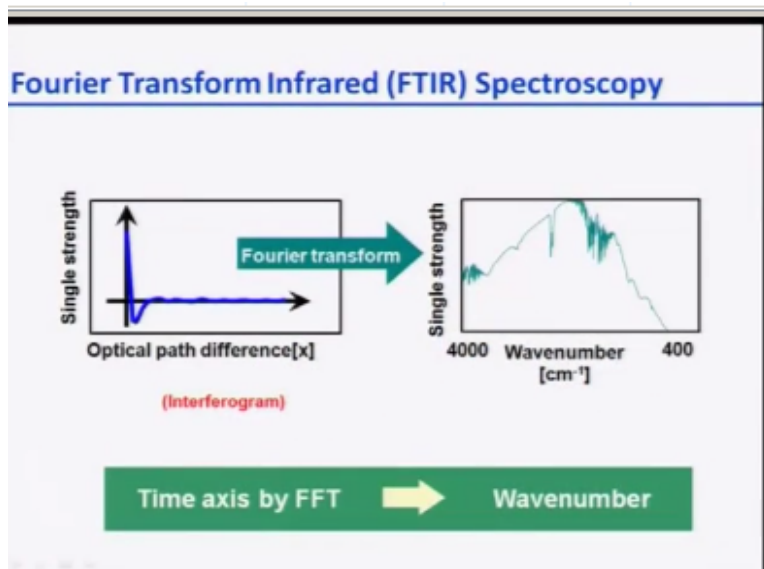
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Of the advanced spectroscopic analysis. So now I am going to move onto the Fourier transform infrared spectroscopy. Well, Fourier transformation is a very generic term. Normally it is used in different diffraction techniques like X-ray diffraction or electron diffraction where the information in the real space that is XYZ space is converted into the Fourier space or the inverse

of real space. So to give in better idea, in a spectroscopic technique we normally measure certain signal.

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Suppose this is signal strength in the Y axis is plotted as a function of optical path difference that is in distance or in X and we have a signal given in the blue color plot. So if you want to Fourier transform this, what you get is obviously X axis as inverse of distance at the centimeter inverse and we can always denote this parameters $1/\lambda$ $2\pi/\lambda$ $1/2\pi\lambda$ whichever we have to pay and define it will come back to wave number.

It can be plotted as an again single strength or signal strength. So there you can get different peaks 123456. Many peaks are present here. So that is what is called Fourier transformation of the spectroscopic data. So that means time axis is changed by a 50 and we get into wave numbers. So this path difference can be plotted or changed to wave number.

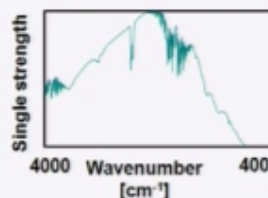
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FT-IR Terms and Definitions

Resolution (common definition) –

The separation of the various spectral wavelengths, usually defined in wavenumbers (cm^{-1}).

A setting of 4 to 8 cm^{-1} is sufficient for most solid and liquid samples. Gas analysis experiments may need a resolution of 2 cm^{-1} or better. Higher resolution experiments will have lower signal-to-noise.



Now there are many other things which you need to define before we actually go into the FTR results and the prior experiments. So the first one is a delusion. You know the resolution means the ability to resolve two distinct points in the space. So that means separation of the various spectral here evidence here, easily defined wave numbers is to what is known as resolution. So resolution means this different whatever coming spectral aspect that is the peaks, they are coming at different wave numbers. Whether can you see them separately or not?

It can be set up in a FTR machine. So a setting of 4 to 8 cm^{-1} is basically sufficient for most of solids and the liquid but gases require high resolutions. So they need resolution of 2 cm^{-1} inverse or data this is well known that high resolution experiments will always have lower signal to noise ratio. That means if you want to have high signal to know as this issue, you better do the experiments at lower resolutions and signal will be better. There are other things which you also need to know is the spectrum is what is collected in a FTR, a spectrum is collected resolution of 1 cm^{-1} inverse. If 4 data points are collected within 1 cm^{-1} spectral will develop 1 cm^{-1} inverse.

This is how this resolution has set in a machine. It is enough to acquire basically spectrum at a very high resolution and increased number of data points are required. That means long as to that of the moving, which I will show you when I discuss about the FTR requirements. High resolution instruments also requires aperture because you need to improve the parallelism of a interfere parameter which is used in the FTR.

Now FTR, we always receive signals or rather different kind of signals where there will be no axis and we need to reduce these unwanted noises or oscillations and so that the signal contributions can be increased or appropriately seen and there we need to use certain kind of mathematical operations after we receive the data from the machine and this mathematical operations are required to basically reduce oscillations or may be the noise contributions.

So one such technique or discharge technique is known as aqua digestion and this is soon here. Suppose this is the integrand or the FTR signal data points, you see as you go on the higher values of data points, the number of noise is basically increasing. So we a window and define a apodization function and then reduce this noise. So there are many functions used in the literature. They can be beyatorin cosine. These are not part of this course, so I am not going to go through each of these techniques.

But these are their inbuilt software which is attached to any FTR machine. But one must know that these are the different techniques possible for post processing of the FTR data. Another important aspect of the FTR is the scan.

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FT-IR Terms and Definitions

- **Scan(s)** - a complete cycle of movement of the interferometer mirror. The number of scans collected affects the signal-to-noise ratio (SNR) of the final spectrum. The SNR doubles as the square of the number of scans collected; i.e. 1, 4, 16, 64, 256,
- **Scan speed or optical path velocity** - the rate at which the interferometer mirror moves. For a DTGS detector, the SNR decreases as the scan speed increases.
- **Scan range** - spectral range selected for the analysis. The most useful spectral range for mid-infrared is 4000 to 400 cm^{-1} .

Scan means a complete circular movement of interferometer mirror. So number of scans actually which are collected normally, it always affects the signal to know as this issues. So that means a complete circular movement of interferometer gives one scan. So you can have many scans for a particular data to improve the signal to noise ratio and signal to noise ratio is found to double as a square of number of scans. That is if you have suppose one scan, the signal to noise ratio will be very low. It will double actually if you go for 4 sums of scans and if you keep on increasing this from 4 to 16 to 64 to 256, the quality of data will be much better.

Second important aspect in FTR, the scan speeds or scan speeds of optical path of velocity. This is nothing but rate at which the interferometer mirrors moves. So normally there are different kind of detectors, so this is DTGS as 1 scan detector or scan decrease as a scan will increases and last one which is important for you is the scan range. Scan range is the spectral range or the number range of the analysis. It can normally span from 4000 to 400 cm^{-1} . So that means the near, the median for it basically 4000 to 5 or 400, but it can go to other infrared, far infrared or near infrared region also.

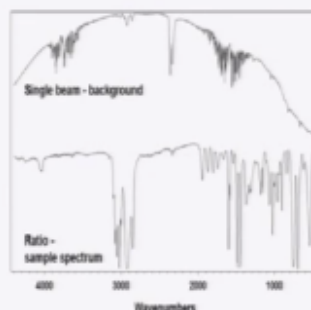
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FT-IR Terms and Definitions

Scan mode - Either single beam or ratio.

Single beam can be a scan of the background (no sample) or the sample.

Ratio mode always implies the sample spectrum divided by, or ratio-ed against, the single beam background.



Well to give you some other thing like important information about the single beam and the ratio sample spectrum, scan mode can be either single beam or ratio. Single beam may be a scan of a ground with no sample or may be with the sample or the single beam. Just you put the sample or no sample and then collect this for single beam. Ratio mode actually implies of beams that sample spectrum divided by the ratio or by ratio against the background. That means you have a background, you have a sample, then you basically take the ratio of the sample to the background and then plot.

This is a single background or a single beam obviously background is very bad. You cannot fit it. You can clearly see here this is a background. But the ratio of the background is very good so you signals can be easily seen.

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Qualitative Analysis

Identification of an organic compound is a two-step process.

The **first step** involves determining what functional groups are most likely present by examining the group frequency region.

Well as I said, these are the basic things about FTR you need to know, as I said the FTR actually used to do both the quantitative and the qualitative data analysis and first we will discuss the qualitative analysis. What all qualitative things you can do? As I said, this is used only mostly for identify different kinds of organic compound whereas it can be also used for inorganic compounds which are also there in my lecture today. Identification open organic compound is basically two step process in FTR.

The first step involves determine the functional group present for examining the group frequency region. What is that? You know in an organic compound there are different function possible like alcohol group, ketene group or aldehyde group or many others possible. So therefore that is the first step. First step is to determine this functional group and that is done by examining the group frequency region. Second step is then; it involves a detail composition of a spectrum of the unknown sample which you are examining.

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Qualitative Analysis

The **second step** then involves a detailed comparison of the spectrum of the unknown with the spectra of pure compounds that contain all of the functional groups found in the first step.

The **fingerprint region**, from 1200 to 600 cm^{-1} is particularly useful because small differences in the structure and constitution of a molecule result in significant changes in the appearance and distribution of absorption peaks in this region.

With the spectra of pure compounds that contain all the functional group found in the first step. So first we find these functional groups. I do not know once we have supposed 1 or 2 or more functional groups in the sample present. So if you want to really know what the best compound for is, then one needs to compare the spectrum of this unknown sample which you are experimenting to this spectra of all compounds above level in the database contain all the functional group present. So normally we use something known as finger pin region which is mentioned here. Finger pin region is spans so all 12 500 600 cm^{-1} .

This particle is fooled for because if there is a small difference in the structure of the compound or whether the constitution of the molecule, this change in the structure all the constitution of the molecule can lead to significant change in the appearance and distribution of this peaks in this region. That is why it is called the finger pin region. That means it gives the finger print of the compound.

Although we know that approximately the type of compound what it is from the first step, but in the second step when you compare us always looks for the finger pin regions to know whether any change in the structure or constitution that can lead to change in the peak positions.
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Qualitative Analysis

Group Frequencies: The approximate frequency (or wavenumber) at which an organic functional group, such as C=O, C=C, C—H, C≡C, or O—H absorbs infrared radiation can be calculated from the masses of the atoms and the force constant of the bond between them.

These frequencies, called **group frequencies**, are seldom totally invariant because of interactions with other vibrations associated with one or both of the atoms composing the group. A range of frequencies can be assigned within which it is highly probable that the absorption peak for a given functional group will be found.

Well as I said the first step we do, in the first step we do basically measure the functional group by looking at group frequencies. So let me just explain what is group frequency is. In a group frequency means approximate frequencies or other wave numbers at which an organic functional group, it can be ketene or CC double bond or C single bond or CC triple bond or OH absorb bond which absorb the infrared radiation can be calculated from the masses of the atoms and the force constants of the bonds.

That means knowing the masses of the atoms and the force constants of the atoms we can actually calculate the frequency approximately and that is what is known as group frequency. So once you know the group presents we can actually calculate it. This frequency is called group frequencies because they are seldom total invariant because of the interaction with other vibrations assertive with one or both of the atoms comprising the group. Range of frequencies can then be assigned within which this highly vibrations that absorption peak as a given functional group will appear. That is the idea.

So once you define the rate of frequencies, we assume or w can actually think that the absorption peaks will appear for that compound in that frequency range.
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TABLE 17-3 Abbreviated Table of Group Frequencies for Organic Functional Groups

Bond	Type of Compound	Frequency Range, cm ⁻¹	Intensity
C—H	Alkanes	2850–2970	Strong
C—H	Alkenes (>C=C<^{H})	1340–1470	Strong
C—H	Alkenes (>C=C<^{H})	3010–3095	Medium
C—H	Alkynes ($\text{—C}\equiv\text{C—H}$)	675–995	Strong
C—H	Aromatic rings	3300	Strong
C—H	Aromatic rings	3010–3100	Medium
O—H	Monomeric alcohols, phenols	690–900	Strong
O—H	Hydrogen-bonded alcohols, phenols	3590–3650	Variable
O—H	Monomeric carboxylic acids	3200–3600	Variable, sometimes broad
O—H	Hydrogen-bonded carboxylic acids	3500–3650	Medium
N—H	Amines, amides	2500–2700	Broad
C=N	Amines, amides	3300–3500	Medium
C=C	Alkenes	1610–1680	Variable
C=C	Aromatic rings	1500–1600	Variable
C≡C	Alkynes	2100–2260	Variable
C=N	Amines, amides	1180–1360	Strong
C=N	Nitriles	2210–2280	Strong
C=O	Alcohols, ethers, carboxylic acids, esters	1050–1300	Strong
C=O	Aldehydes, ketones, carboxylic acids, esters	1690–1750	Strong
NO ₂	Nitro compounds	1500–1570	Strong
		1300–1370	Strong

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To give you some idea, suppose if you can say alkenes in case of organic compounds, you can see the frequency range is follows in this 28,150 29,170 which give a strong intensity peak or 1348 to 1470, it will also give a strong intensity peak. Then there are alkenes, alkynes, you can look at the frequency regions in your screen and then you can go on doing amines which comes about 3300 to 3500 mediums and then we have spices like this. Amoral alkenes, aromatic grains or amines where in these regions you have per level intensity possible and then one can as well go to alcohol, etas groups.

Who had a strong intensity peaks happens in the frequency range mentioned here. So one can actually build this table. This is obtained from Thomson higher education book and table number 17. 3, you can go back and then find this book also.

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Qualitative Analysis

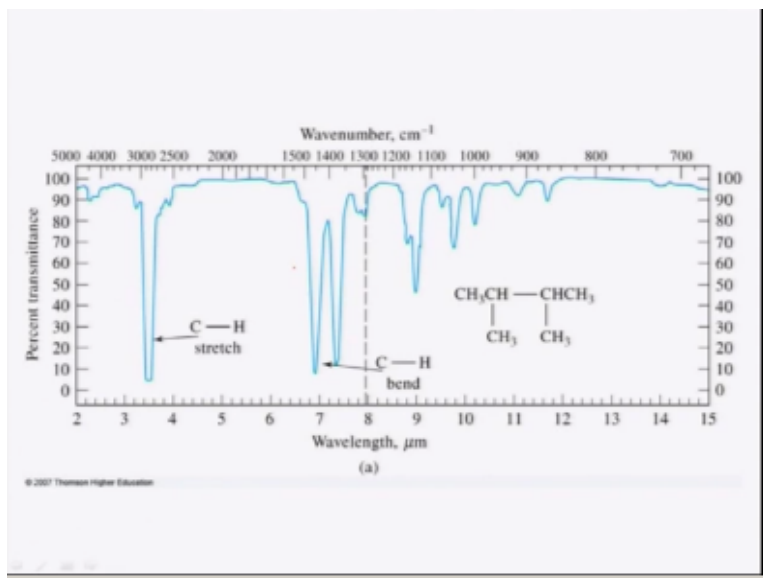
The "Fingerprint" Region: Small differences in the structure and constitution of a molecule result in significant changes in the distribution of absorption peaks in this region of the spectrum that extends from about 1200 to 700 cm^{-1} .

As a consequence, a close match between two spectra in this fingerprint region constitutes strong evidence for the identity of compounds yielding the spectra. Exact interpretation of spectra in this region is seldom possible because of the complexity of the spectra.

Well to give you some more detail about finger pin regions, small difference in the structure and constitution as I said can lead to change in the distribution of the absorption peak and they have in this. So that means as a consequence of the finger pin region analysis, a close map between the two spectra in the spin region can be possible and this will lead to the exact identification of the compound. Exact interpretation of the data or the spectra in the region is held on possible because of the complexity of the spectra.

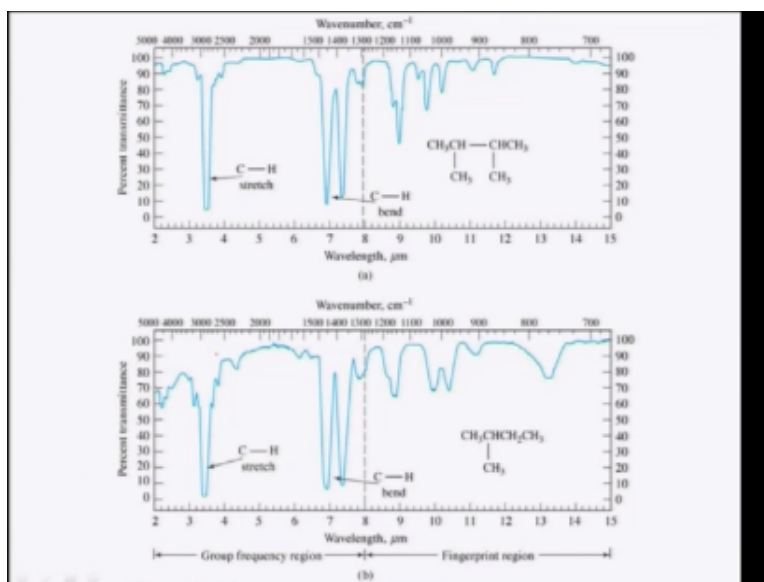
Sometime a spectrum is very complex because of the change of this small change in composition of the structure. It may not be possible even to actually pinpoint the exact structure of the compound present.

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To give you some more idea, this is taken from this kind of compound, you can clearly see there are 1,2,3,4 methyl groups and CH bond and CC bonds. So if I take an FTR spectra or from this we get CC straight bond at the wave number of 3.5 approximately micron in wavelengths. This is actually brought in terms of wavelength λ and then FCH band pins, pin can also lead to pans and then you have many other frequency schemes. So that means by looking at these vans, we can actually carefully say that what is it is.

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Now if you change it that is what I am actually going to tell you how it is to be done. So if you look at this $\text{CH}_3\text{CH}_3\text{CHCHCH}_2$, this compound you have regions which are marked as group frequency regions, regions which are marked as a finger pin regions. In the group frequency regions, you have CH stage bands, CH bands they are same. This compounds also, this compound also. There is no difference. Now although we have CH_2 , we have a CH. So there is a small change in the composition can lead and only lead to change in the finger pin region.

You see here the peaks in this place and peaks in this place, they are different. So that means this is the finger pin region where the changes in this composition or the structure little bit of the compound can give rise to changes in the peak positions or the peak number or peak's intensity of the peaks. That is why this is known as finger pin region this is known as the group frequency region where we can guess the type of compound present but any change in the structure can be revealed on in a single. I hope this is clear.

To give you more idea or even give more examples because examples are always better than this. So if you look at this compound, here there is a wage group and in this compound here there is this error and then you have CC bonds and $\text{CH}_3\text{CH}_3\text{CH}_3$, 3 CH_3 methyl groups are there. So if you look at it again, from this two, if you look at group frequency regions, obviously there is a all group here which can be clearly seen for the waste stage band here present CH stage band also can be seen here present. On the other hand, this is only CH including CCL, so CCL doesn't come in the group frequency region. So CH stage band is present here.

So by comparing and then you have CH band and CH band here also. There is little difference but more or less same. So by looking at this group frequency region you can clearly see there is a wage group here present. There is no wage group in the second compound that is this. Now if you at the finger pin regions that is very distinct. The finger pin region here and here are distinctly different. This case CCL stage band is present here because there is no CCL bond here, so there is no CCL band present here.

So by looking at these two regions, group frequency and the finger pin regions, one can classify even the finite scale changing amine in the compound structure very easily. First on the group frequency region, we can know the groups present and the finger pin region you can know the exact structures present. So this is what I like to upon that some of the analysis is done in the FTR.

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Qualitative Analysis

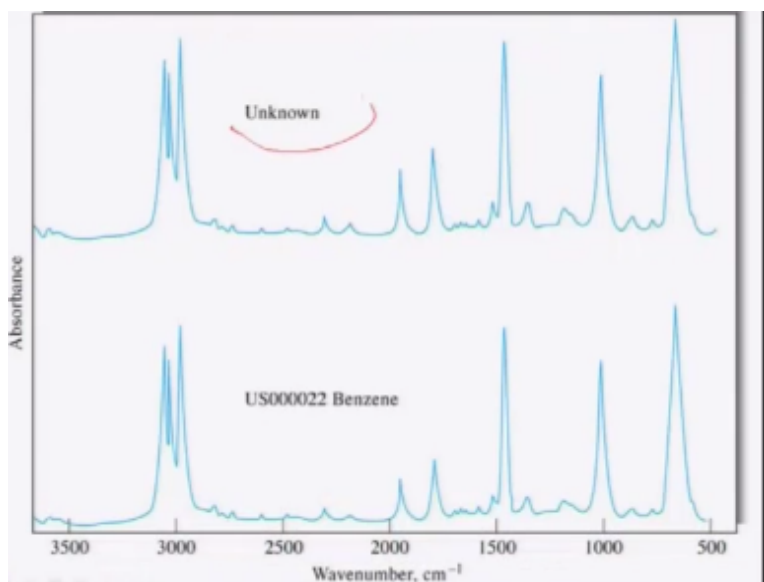
Computer Search Systems: Virtually all infrared instrument manufactures now offer computer search systems to assist you in identifying compounds from stored infrared spectral data. The position and relative magnitudes of peaks in the spectrum of the analyte are determined and stored in memory to give a peak profile, which can then be compared with profiles of pure compounds stored. The computer then matches profiles and prints a list of compounds having spectra similar to that of the analyte. Usually the spectrum of the analyte and that of each potential match can then be shown simultaneously on the computer display for comparison.

Well nowadays nobody uses, you cannot live without computers. So therefore all the qualitative analysis is done always with the computers or systems. Virtually all infer instruments manufacturers nowadays, how far you a compute such system. That actually has assists 95 in identifying the compounds from a large number of stored spectral data and this data actually show you the position of the relative magnitude of the peaks present in the spectrum of the different which can be later on.

So people actually take this, collect the data and store in a computer and then what you do is that, once you have a FTR spectrum from an unknown compounds, you just match the profiles and then when you find the similarity you just use it. So that is nothing but like X-ray diffraction data matching.

You have a Z impudence database in X-ray diffraction you compare your X-ray diffraction pattern from unknown sample just by database. If it matches, you are done, if it does not match, then you keep on doing this analysis final scale. Same thing is valid for this kind of analysis here also. To give an example, suppose you have got a spectrum.

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From an unknown sample like this and you want to analyze what for this compound is. So you just load this file into the computer. It will search. Whenever you find a real match like this, you can clearly see a real match, very perfect match like this. You come to know this is US000022 Benzene. This is a number or a file just like a data card, you have a number and this is Benzene. So therefore although it is very simple Benzene, everybody will probably know it and therefore by comparing this database with computer, one can easily carefully say that.

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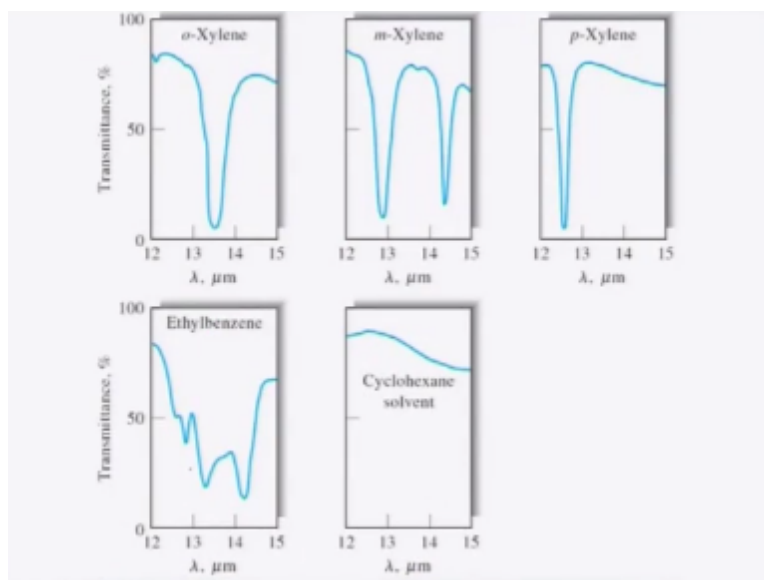
Quantitative Applications

Quantitative infrared absorption methods differ somewhat from ultraviolet/visible molecular spectroscopic methods because of the greater complexity of the spectra, the narrowness of the absorption bands, and the instrumental limitations of infrared instruments. Quantitative data obtained with infrared instruments are generally significantly inferior in quality to data obtained with ultraviolet/visible spectrophotometers.

Well now as I said in the last lecture also that infrared spectroscopy FTR can be also used to do the quantitative analysis also. What are the quantitative analyses one can do? Well quantitative analysis in this case differs extensively from the UV visible molecular spectroscopic methods because of the complexity of the spectra and also some cases you have seen even from this spectra there is bands in the spectra are very narrow and then you have instrumentation. Quantitative data obtained from the instruments are generally significantly inferior quality in the UV and visible spectrometer.

So therefore normally we do not do much of the quantification using this, they quantify in the sense of using the peak strength. The area under the peak or the peak height and can be done. So to give you some more idea, how they qualitatively things can be possible. Xylene we know, Xylene is basically a Benzene compounds. You have ortho, Meta and tyro.

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Those OMP Xylene and if you take FTR spectra very clearly you can see this is Benzene wavelength. You can see ortho Xylene gives a peak at about 13.5 micrometer wavelengths but M Xylene gives two peaks, one at close to 13, one at 14.3 micrometer. On the other hand, P Xylene gives you peaks at 12.5. So by looking at this fine scale structure, you can clearly say what kind of Benzene is present.

Let us look at ethyl Benzene. They are kind of another complex compound. You can see the peak positions. Cyclohexane solvents actually normally do not give peak that is why these are can be used for FTR study.

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TABLE 17-4 An Example of IR Determinations of Air Contaminants

Contaminant	Concn, ppm	Found, ppm	Relative Error, %
Carbon monoxide	50.0	49.1	1.8
Methyl ethyl ketone	100.0	98.3	1.7
Methanol	100.0	99.0	1.0
Ethylene oxide	50.0	49.9	0.2
Chloroform	100.0	99.5	0.5

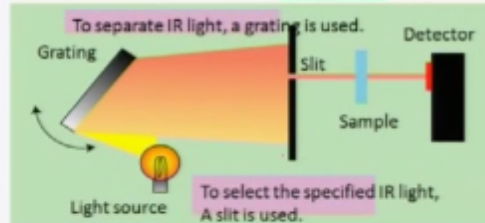
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Well nowadays as I said, if you look at literature, literature in the sense of, if in such in computer about infrared spectroscopy. Infrared spectroscopy becomes very important in 1970s when it was rather discovered or instrumentation was made for determination of the air contaminants. Air can have different pollutants like carbon monoxide, methyl and ethyl ketene or even many other contaminants can be present. Nitrous oxides or carbon dioxides, they can be measured even precisely using quantitative techniques.

To give you some idea and some of the values, again I have taken from Thomson higher division. So if you look at the suppose actual concentration of carbon monoxide in a particular system is 50 and the results actually 49.1 result will show you 50. You notice even + and 2%. Similarly for methyl ketenes, methanol, ethyl oxide chloroform. So this is what the kind of quantification one can do using the peaks area and the peaks for the infrared spectroscopy or FTR spectroscopy.

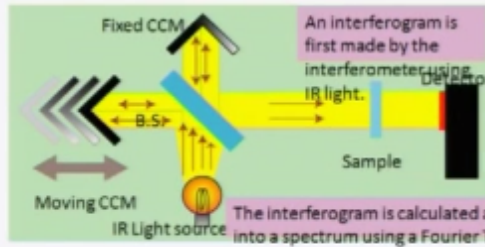
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Comparison Between Dispersion Spectrometer and FTIR



Dispersion Spectrometer

In order to measure an IR spectrum using a dispersion Spectrometer, it takes several minutes. Also the detector receives a few % of the energy of the original light source.



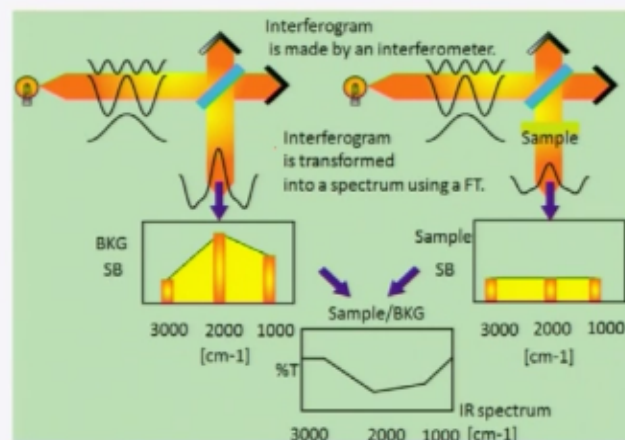
FTIR

In order to measure an IR spectrum using FTIR, it takes only a few seconds. Moreover, the detector receives up to 50% of the energy of the original light source. (much larger than the dispersion spectrometer.)

Now I will just talk to you about.

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The Principles of FTIR Method



How the machine looks like and how things can be done and to wrap up the FTR principles. Well FTR actually, as I said is basically used to measure low concentrations in the solutions and it can be used to measure here. So first significant presence of water vapours, methane everything can be done. So there has been machine nowadays have build are very complex but I am going to talk about very simple machine which is the simple basically you are going to talk about the principles and show you some of these basic stuffs.

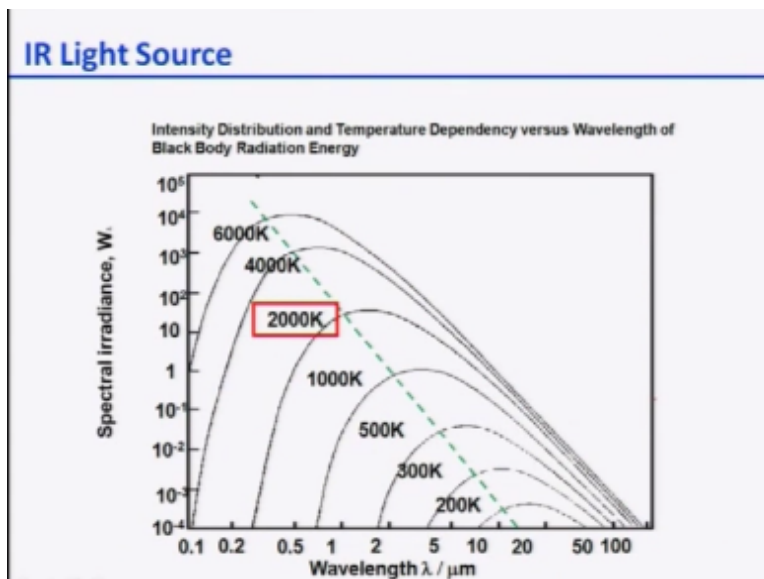
So basic components of FTR is first of all a source as you can see. There is a source here, that's a IR source and it emits a broad band of different kind of wavelengths, infrared radiations. Normally the IR source used is basically known T mate gas mate FTR CR series in the machine which people use normally in the labs are a silicon carbide compound and it has normally hitter, if you hit upto 1550 degree Kelvin, and then it emits IR radiations.

So IR radiations then go in through an interferometer. The real heart of the machine is basically interferometer. So IR radiation coming from the source passes through an interferometer which we will discuss in detail and then interferometer modulates this infrared radiations and then interferometer basically performs an optical inverse Fourier transformation on the IR radiation and this modulated IR beam then passes through the sample like modulated beams here passes through the sample and then from the sample, whatever comes out is detected in a detector.

Which is the gold detector normally and many cases, a digital signal is obviously digitized and it can be Fourier transformed in a computer to get a good spectrum. So what is done is that, you

have the infrared source, if you write infrared source and then from there it goes to interferometer and interferometer modulates and then it falls in a sample and that sample it goes to detector, detector to computer. Schematically we can write down this is what actually a machine consists of. So this is what is shown here. So you can see this is the interferometer and then sample and then we get the data.

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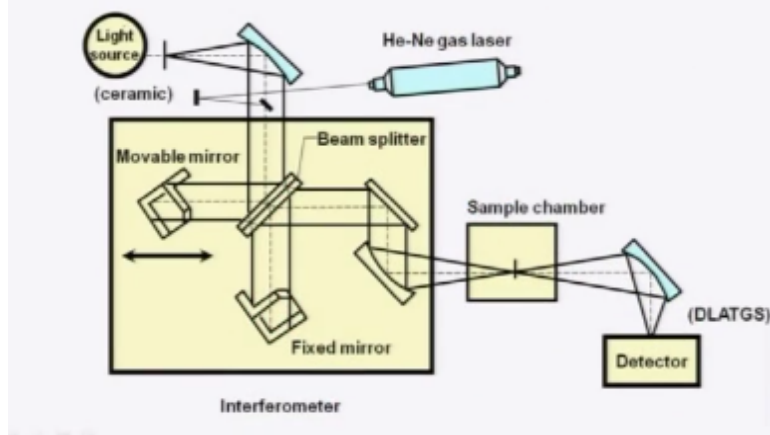


Now as I said, first IR light source and this is the plot of spectral radiant vs wavelengths as a function of temperature. As you see from 200 300 upto 1000 or 6000, the spectral radiance increases normally we use to heat the material from 1000 to 2000 degree Celsius like that. So like SIC is heated upto 1550 degree Kelvin to get IR radiations. So you get sufficient amount of spectral radiance. Radiance is nothing but energy per unit area.

Now to give an idea what the interferometer looks like, let me just tell you what exactly. This is the heart of the machine, FTR machine. So as you see there is the light source, you can also have Helium, Neon things. Light source means infrared source.

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FT Optical System Diagram



This is ceramic like silicon carbide. Now this is nothing but actually but chamber type of plain mirror interferometer and so therefore infrared radiation is collected by and collimated, this is what is done is here. You have collected and collimated by this mirror and then it falls under beam splitter. You see here there is a beam splitter. The real task of beam splitter is that it does meet one half of the radiation and then reflects the other half. So one half is gone here, other half is gone there.

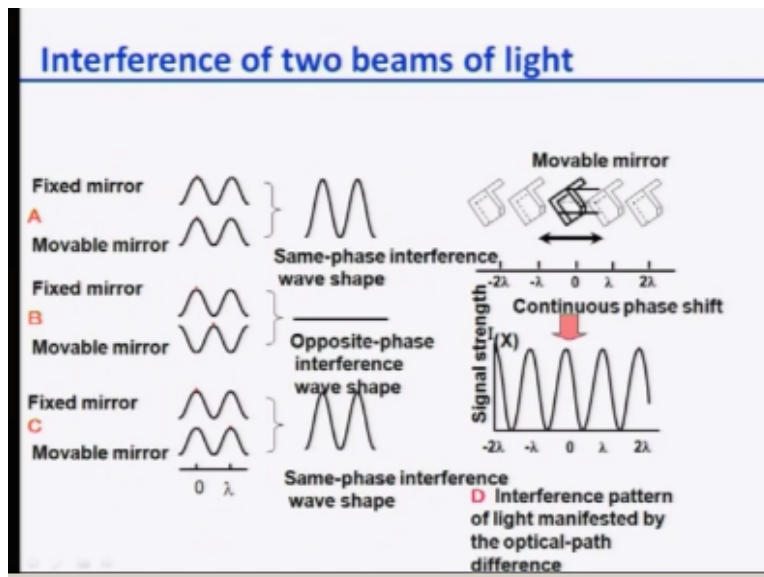
So one half is falling on a fixed mirror, other half is falling on a movable mirror. Now what actually happens if these two waves are then interfering? That is what is done, work of the interferometer. So one of the infrared radiation then finally goes to the sample, you can see that this is again goes to the sample and reflected. Again go to the sample and then before goes to the sample then gets reflected back to the beam splitter to the moving mirror, that is the moving mirror like this and then come back.

So reflected back and other half of the radiation basically going to the sample first gone through this beam splitter and then reflected from a fixed mirror. So one half is going to this, another half is going to this. I have marked it like this. So interference happens because of these two different, they are all same wavelengths because beam splitter only reduces the energy, what is called the absorption amount allows certain amount to pass through and certain amount to be reflected.

So wavelength is same and then gets interfered and you create interference pattern and then finally this interference pattern from beam falls on sample and then it is collimated on a detector

whatever is coming from the sample. That is the basically the heart of the machine. So this is what is shown here and the arrows it goes back.

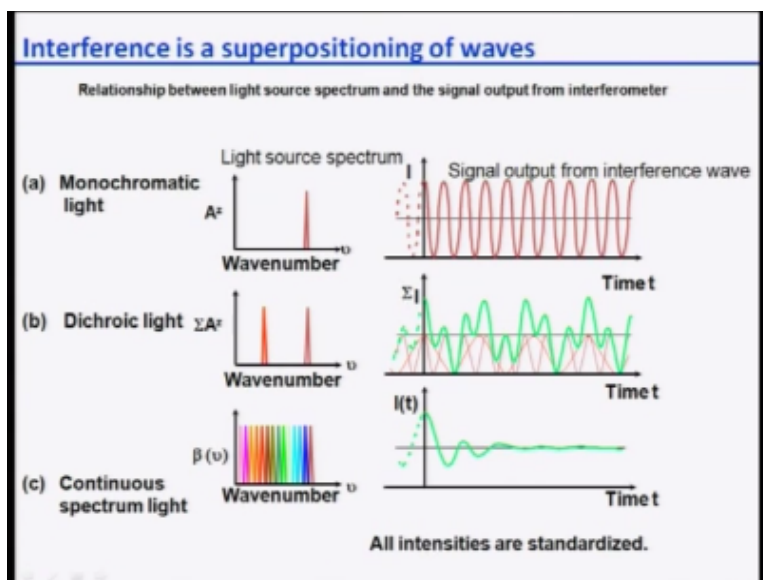
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So now if I have to talk about the interference in little bit detail, if I have supposed fixed mirror and movable mirror you have seen. So if both these waves are very similar in terms of the spatial and the temporal part so they can interfere and produce same speed interference this kind of. If you have opposite, then there will be nothing. There will be destructive interference and if you have again similar looking but displaced by λ wavelength, so you have interference pattern like this.

So whatever the situations, in a real situation, the interference pattern will look like this because of continuous phase shift and you get very strong increase in the amplitude of the waveform when it comes out from the interferometer. So that is the basically idea to create a strong interference of the waves coming from the source before it falls on a sample and then one can actually do the similar analysis for monochromatic and the continuous spectrum. If you do monochromatic.

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Once you get the signal from the sample after detector it absorbs, then this can be confided into Fourier transforms. So this is like $AJ + \text{wave number}$ on a monochromic light and then this is interference wave. This is a Dichroic light, you have two. You can see then it, this is this kind of signal you can get through interference wave. You have a continuous large number of waves and you get a, this is what you normally get in the FTR spectrometer or in the FTR plots.

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Sample Handling

Solutions: A convenient way of obtaining infrared spectra is on solutions prepared to contain a known concentration of sample. This technique is somewhat limited in its applications, however, by the availability of solvents that are transparent over significant regions in the infrared.

Solvents: No single solvents is transparent throughout the entire mid-infrared region. Water and alcohols are seldom employed, not only because they absorb strongly, but also because they attack alkali-metal halides, the most common materials used for cell windows.

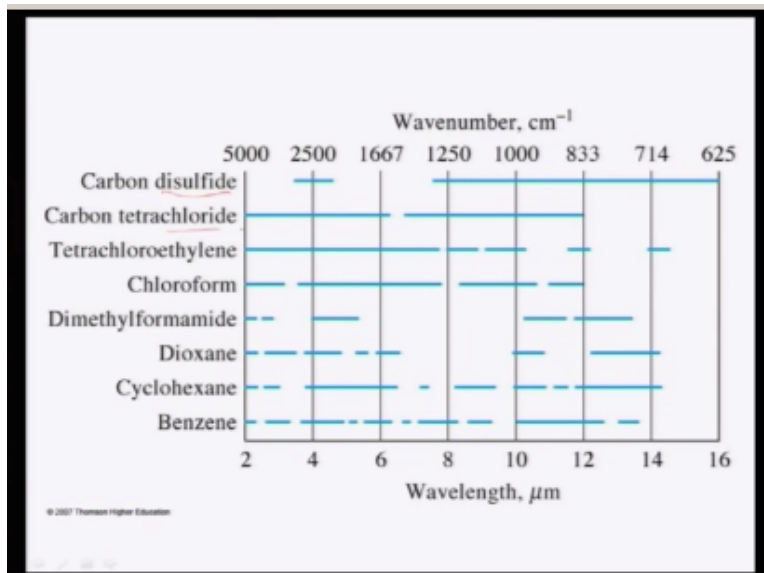
So that is about the machine and how things are done. Now in the last stage what I want to discuss is basically how the samples to be handled because many cases you need to know how

the samples to be handled otherwise you may not get good data. Normally we need solutions for doing this analysis. It is a convenient way of obtaining infrared spectra is on a solution prepared to contain a known amount of a concentration of a sample and this is basically to get data from a known sample and known concentration.

This technique is somewhat limited in its application however, if you know large number of solvent presents and they are transparent to the infrared regions, then this can be done. Then second thing which is used FTR is solvent. How to handle solvents? Normally no solvent, because you have to have solvent to dissolve this unknown compound which you are going to analyze. No single solvent is found to be transparent through the entire infrared regions. That is a big problem.

What are alcohols are seldom employed, you cannot employ them because they absorb infrared and they absorb infrared and they can create problem. Not only that, they can also attack many of the alkali metal elides and many other materials which is used for the windows of the infrared spectroscopy.

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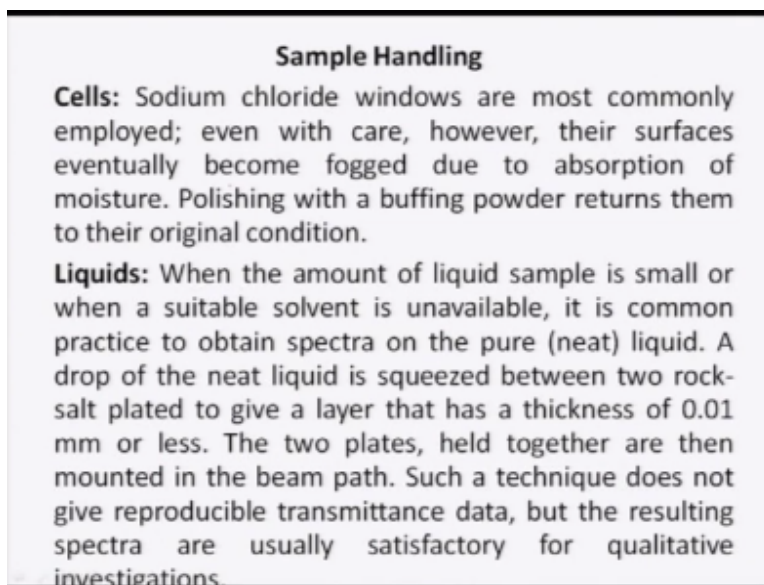
So what are the things we can use? Well this is covered different compounds written carbon disulphide, carbon tetrachloride, and tetra Chloee ethylene chloroform, diethyl for mine, dioxin, cylcohexene and benzene. If you look at it, the absorbent as a function of wavelengths or wave

numbers, this is absorbed here absorbed there but not absorbed here. So you can use it if you want to analyze in history consistency.

Carbon tetrachloride doesn't absorb at low wave numbers. Same thing is valid for tetra Chloro ethylene chloroform again same thing. Dichloride fermadide can be used in this frequency range. Dioxin can be used here between 1600 to 1250 or may be upto 1000s. Benzene can be used at low wave numbers or high wavelengths, so there by tellurian this in a different compounds, one can actually study the what is called different compounds or different wave number range using different solvents.

Then you have cells. Sodium chlorides actually Windows have most commonly used in the machine.

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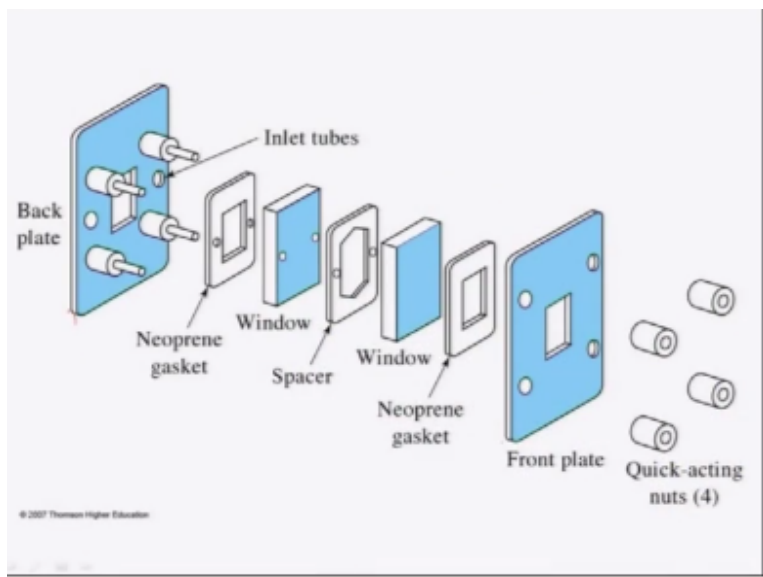
Because they do not absorb IR radiation. But surfaces can get chloride where sodium hydro chloride can be absorbed moisture and then can reduce where they lead to absorption of the infrared radiation and then signal strength will reduce. Sometime polishing with buffering powder can help but sometime may not. Liquids which can be there. So when the amount of liquid sample is small, a suitable solvent is unavailable.

Many times you can use pure liquid of that compound without solvent. A drop of that neat liquid is squeezed between the two rock salt plates or sodium chloride plates to give a layer that is

thickness of very small layer of the 0.1 mm or less. These two plates are then held together and mounted in the beam path. Such techniques do not give reproducible responsible data but one can do qualitative analysis with this.

That is possible. So whenever you do not find any suitable solvent, you can actually do this such a kind of analysis.

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To give you this, this is the back plate sodium chloride front plate and they have windows and these are the newer print gaskets and these are the actually can be fitted into this. So you can see and sometime you can use fitted so this infrared radiation goes through and comes back like this front plate to the back plate and then and these are the knots. This has to be all made up of special quality materials so that it does not absorb these infrared radiations.

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TABLE 17-2 Common IR window materials

Window Material	Applicable Range, cm^{-1}	Water Solubility, g/100 g H_2O , 20°C
Sodium chloride	40,000–625	36.0
Potassium bromide	40,000–385	65.2
Potassium chloride	40,000–500	34.7
Cesium iodide	40,000–200	160.0
Fused silica	50,000–2,500	Insoluble
Calcium fluoride	50,000–1,100	1.51×10^{-3}
Barium fluoride	50,000–770	0.12 (25°C)
Thallium bromide-iodide, KRS-5	16,600–250	<0.0476
Silver bromide	20,000–285	1.2×10^{-7}
Zinc sulfide, Irtran-2	10,000–715	Insoluble
Zinc selenide, Irtran-4	10,000–515	Insoluble
Polyethylene	625–30	Insoluble

And what kind of things windows can be used. Sodium chloride, it can be used by large range. That's the best material of a level but it can be solvable in water. That is the problem. Otherwise you can use potassium chloride, potassium bromide. But solubility is very bad. Cesium iodide can be used. The important thing to use is fused silica whereas which is insoluble in water and alcohol and it can be used by large, the wave number range or you can use even zinc sulphide which is insoluble but you can also use that. The polymers cannot be used here, do not absorb high radiation is very small wave number range.

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Sample Handling

Solids: Most organic compounds exhibit numerous absorption peaks throughout the mid-infrared region, and finding a solvent that does not have overlapping peaks is often impossible. As a consequence, spectra are often obtained on dispersions of the solid in a liquid or solid matrix.

Pelleting: One of the most popular techniques for handling solid samples has been KBr pelleting. A milligram or less of the finely ground sample is intimately mixed with about 100 mg of dried potassium bromide powder. The mixture is then pressed in a die at 10,000 to 15,000 pounds per square inch to yield a transparent disk. The disk is then held in the instrument beam for spectroscopic examination.

Well I have already told about that, how do you use liquids. Let us talk how to use solids. Most organic compound exhibits numerous absorption peaks through the solids in the median for region. So finding out a solvent does not there is absorption peaks is impossible. So as a consequence, spectra are obtained on dispersion of the solids in a liquid or solid matrix. Some cases you can use pallets. Most commonly techniques for the handling solution are to use KBR pelleting.

Okay you must know that. A very small amount of the finally ground sample is intimately mixed with 100 mg of the diode production bromide and mixture is placed in a die about 10000 to 15000 amounts per square range to yield a disk and then this can be used. But we will do in our lab classes. You can use spectroscopic moves also.

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Sample Handling

Mulls: Infrared spectra of solids that are not soluble in an infrared-transparent solvent or are not conveniently pelleted in KBr are often obtained by dispersing the analyte in a mineral oil or fluorinated hydrocarbon mull. Mulls are formed by grinding 2 to 5 mg of the finely powdered sample in the presence of one or two drops of a heavy hydrocarbon oil (Nujol). If hydrocarbon bands interfere, Fluorolube, a halogenated polymer, can be used. The resulting mull is then examined as a film between flat salt plates.

They are not soluble in confidently transparent solvent and conveniently not can be pelleted in KBR open obtained by dispersing the mineral in the mirror in a while are fluorinated hydro carbon mules. So mules are actually formed by 2 to 5 mg of finely dispersed powder in the presence of 1 or 2 drops of heavy hydrocarbon a while they can be hydrocarbon . If hydrocarbon balance interfere loop hydrogen a polymer can be used and then examines. So there are many such techniques.

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MID-INFRARED ABSORPTION SPECTROMETRY

Sample Handling

No good solvents exist that are transparent throughout the region of interest. As a consequence, sample handling is frequently the most difficult and time-consuming part of an infrared spectrometric analysis.

Gases: The spectrum of a low-boiling liquid or gas can be obtained by permitting the sample to expand into an evacuated cylindrical cell equipped with suitable windows.

Gases can also be done, spectrum low volume liquids can be obtained by permitting sample to expand and evacuate cylindrical cells possible.

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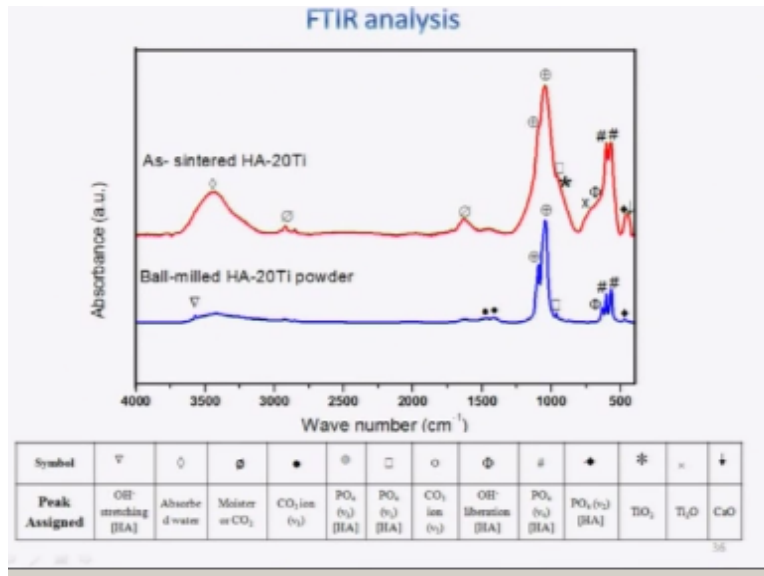
Sample Handling

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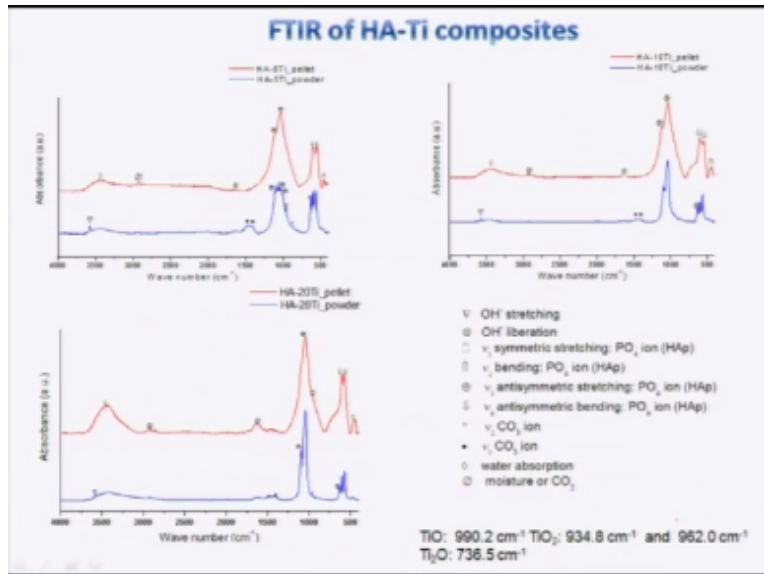
Solution solvent I have already discussed.

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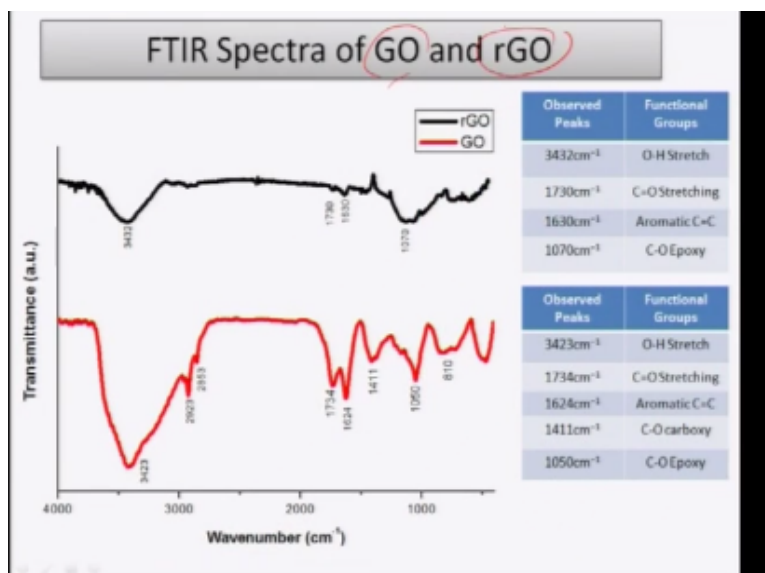
Well now in the last, I will show you some solid sample data because what people normally face. These are actually As sintered composites. You know the problem with solid sample is you have to use, mix with KBR pellete and do that. You will clearly see the different stretching bands like way stretching bands, absorb water, moisture, CO₂ics and then for hydrapeties , these are the bands present there. Titanium can be present in titanium dioxide, titanium Ti O or calcium oxide.

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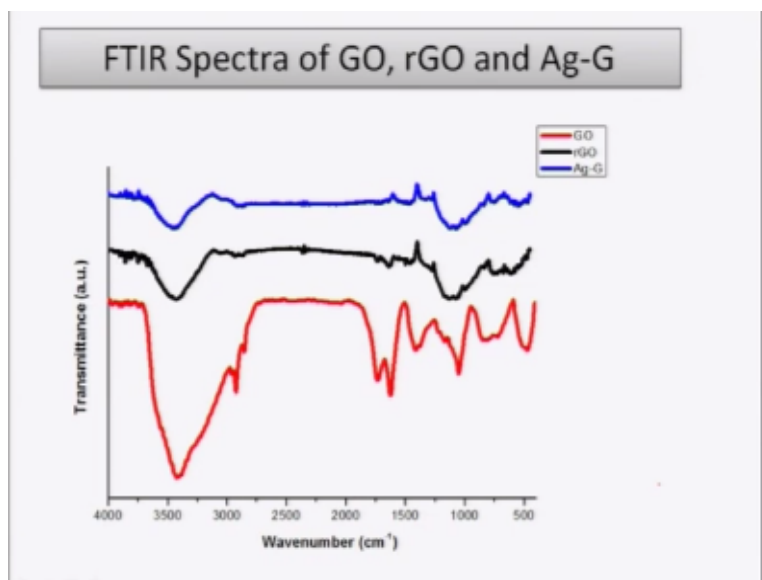
One can actually do such analysis for different kinds of pellets with 39% of 5 Ti or 10 Ti and 20 Ti and get all kinds of data like bonds presence in the samples very easily. Lastly to give you a data about the graphene oxide and reduce graphene oxide.

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This can also be studied by using FTR. See graphene oxide you have, this is basically. I think wastage speak and then these are actually coming from C-O stretching or CC and last one C-O Epoxy. If you have looked at, did use graphene oxide. Graphene oxide obviously, they are viewed larger wise stretch and then CO Approx, CO carboxyl bonds are present or then all those bonds present in the graphene oxide.

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And then even though it is silver, you obviously will not get any peaks from the silver but you will get very large stretch of the wastage band much compared to that. So one can actually do this kind of analysis on solid samples very easily and finish it all. Okay with this I complete my things in the FTR.

Acknowledgement

Ministry of Human Resources & Development

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Co-ordinator, NPTEL IIT Kanpur

Prof. Satyaki Roy
Co Co-ordinator, NPTEL IIT Kanpur

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