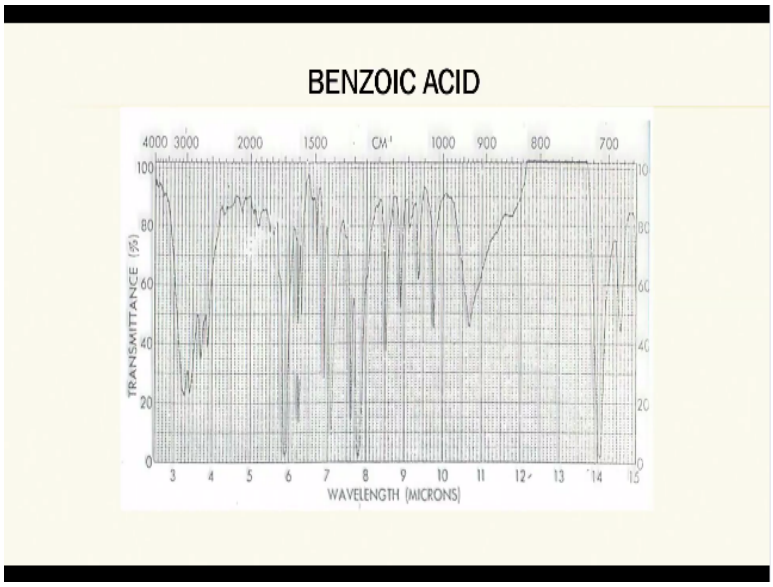
Infrared Spectroscopy for Pollution Monitoring
Prof. J.R. Mudakavi
Department of Chemical Engineering
Indian Institute of Science-Bangalore

Lecture-21
IR Gas Analysers

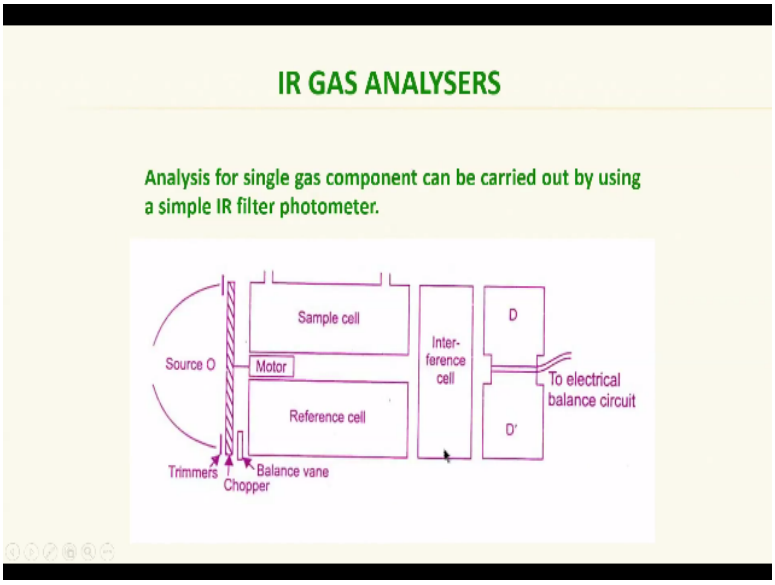
Greetings to you, probably this will be my last class in infrared spectrometer. In the next session I am planning to give a course on this electrochemical technology for pollution control monitoring. So let us continue where we had left out in the last class.

(Refer slide time: 00: 41)



I had shown you the typical IR spectrum for different compounds.

(Refer Slide Time: 00:50)



And now I want to teach you about that the gas analyser IR gas analysers, these are basically very reason of very useful nature not meant for complicated chemical identification, but for chemical analysis of the gases quantitative. So the applications of typical gas analysis such analysis are in the petrochemical industries where Methane, butane and ethylene, polypropylene such gases have been produced mass produce.

What we need is online gas analysers regarding the control of the reactions as well as to monitor the reactions and analyse. So the IR gas analyser are of recent origin to the quantitative gas analysis techniques. So here it is a very simple gas analyser schematic diagram I am showing you. These are all available commercially also, but only petrochemical complex is etc. where there is a need they will be apply using such chemical.

But the principle is very simple. So analysis for single gas component can be carried out by using a simple IR filter photometer. So the here we have a very simple primitive instrument which is used for quantitative analysis, what I have here is a source of IR, sources we have already discussed earlier ok. There is nothing special about it can be a silicon carbide rod or it may be thermocouple Nichrome wire or anything.

So this is the thermo source IR source I have a chopper here for one for reference, one for standard and this is a concave mirror to make the IR beam coming in parallel. Now the sample cell, this is a motor for rotating this chopper, that means at one time when it cuts it will pass through radiation will pass through the top portion and when it is rotating it will cut the top portion and sample will radiation will be passing through the bottom cell.

So the samples I have 2 sample cells of this maybe of a about 10 centimetre ok, 10, 20 depending up on the analyser sensitivity we need. I have one sample cell and 1 reference cell ok. So here I have an interference cell, these also contain some of the gases that we want to eliminate they are present along with the sample. But in the interference cell there is no sample here actual sample.

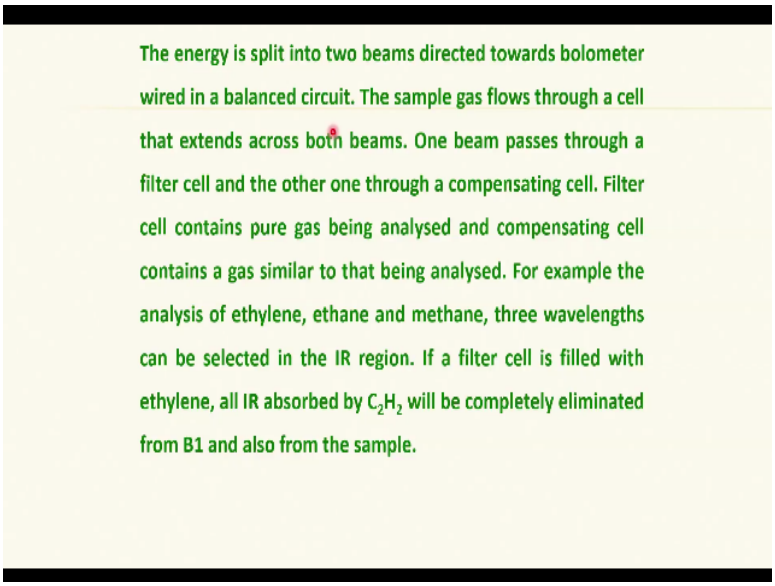
These are common will be other gases that are present in the sample cell and interference cell ok. That is the function of the interference to eliminate the IR peaks or IR quantitation coming from other component matrix components. So D and D dash are the diaphragm ok

gas filled with argon or xenon and this is a diaphragm that what you see a double bond like thing here now, so that is there this one, this is a diaphragm.

And this is the wire to an electrical balance circuit. Now this is the basic arrangement, so when the sample gas radiation is passing through once it passes through the sample cell and else all the interference cells and reaches this peak and when the reference cell contains the gas to be analysed in pure form and that is fixed here and D and D dash which contain the gases.

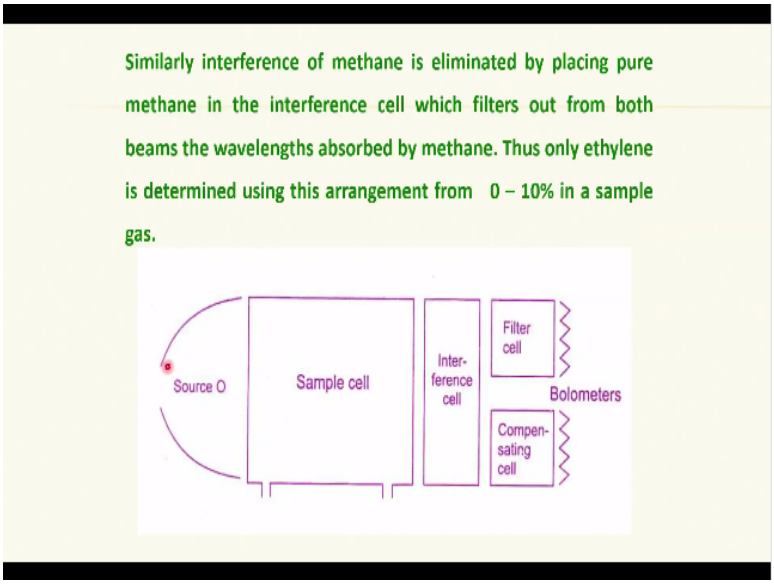
So long as the sample cell does not have any sample that is to be analysed in a sample of our entire reference cell and sample cells will be matching, that means the diaphragm will be in some sort of equilibrium ok, because the radiation will fall on this both of them will be expanding to the same extent and diaphragm will be held in equilibrium and this one the energy split into 2 beams directed towards bolometer wired in a balance circuit.

(Refer Slide Time: 05:58)



The sample gas flows through a cell that extends across the both the beams. So 1 beam passes through a filter cell and the other one through a compensating cell. So filter cell contains pure gas been analysed and the compensating gas contains a gas similar to that being analysed without the samples. So for example in the analysis of ethylene, ethane, methane etc. 3 wavelengths can be selected in the IR region.

(Refer Slide Time: 06:47)



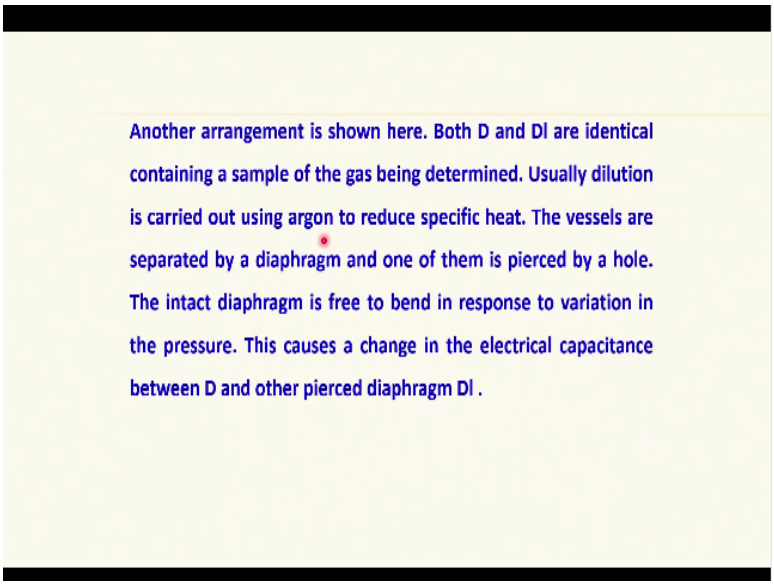
So if a filter cell is filled with ethylene all IR absorbed by ethylene will be completely eliminated from B1 and also from the sample. So something like cancelling each other ok. So interference of methane also can be similarly eliminated by place in pure methane in the interference cell. So that filters out from both the beams the wavelengths absorbed by methane. So only ethylene is being determined using this arrangements from 0 to 10% in a sample gas.

You should see note down the percentage here, we are not talking about the PPM level or milligram level or something like that trace ultra-trace, now we are talking about percentage that is why such instruments are used in the process industries petrochemical complexes. Here I am showing you another arrangement that is source is here, sample cell here, here I do not have a reference cell only the sample cell.

Here I have an interference of using the same principal which I enumerated earlier and then I have a filter cell here, a compensating cell here and a bolometer here, there is not connected with expandable diaphragm ok. So in this case this radiation falls here when the sample is there it will show some difference and the filter cell will expand and then it will abstract the IR radiation reaching the detector the bolometer.

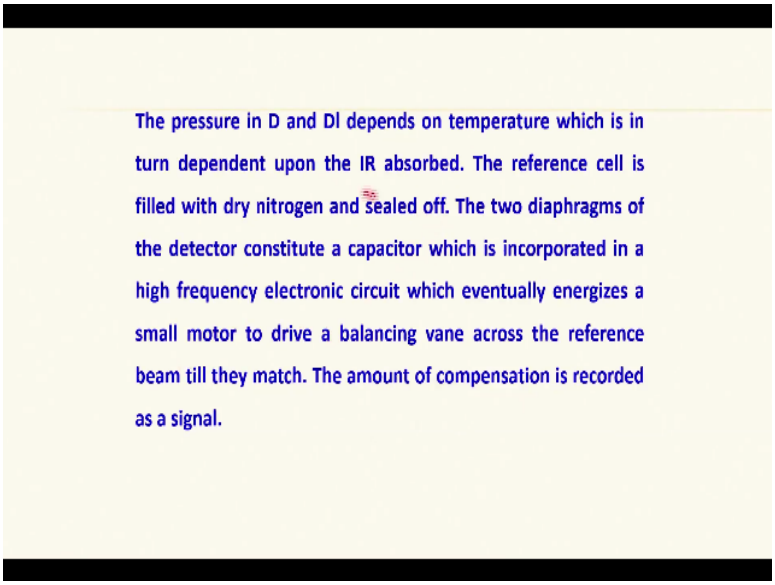
And there are so much quantity, the imbalance will be registered as a signal. So both these thing instruments designs are both popular, the more primitive one is second one which I have shown you, the previous one is the this seems to be more appropriate to the current one because the reference cell and samples and both are being determined simultaneous ok.

(Refer Slide Time: 08:57)



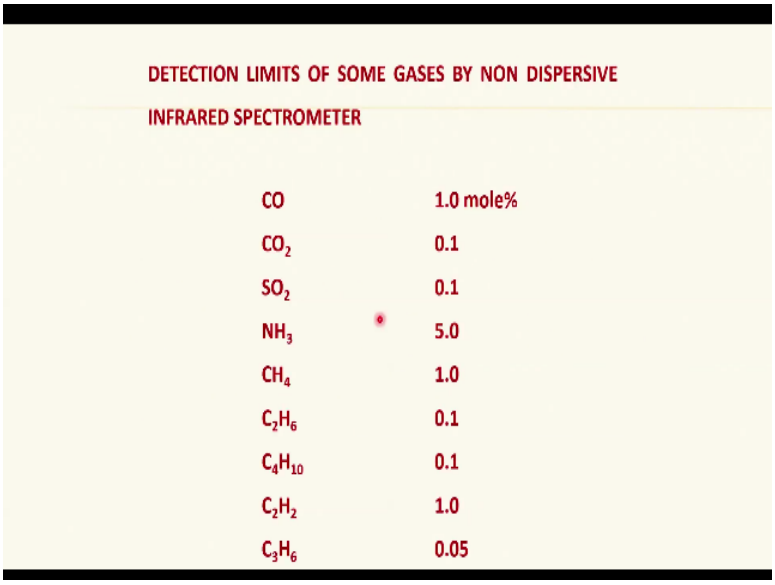
Now another arrangement that arrangement I have shown here I have already explained to you.

(Refer Slide Time: 09:06)



So this is also essentially regarding the explanation for the 2 types ok, first type this is explanation for the first type.

(Refer Slide Time: 09:19)

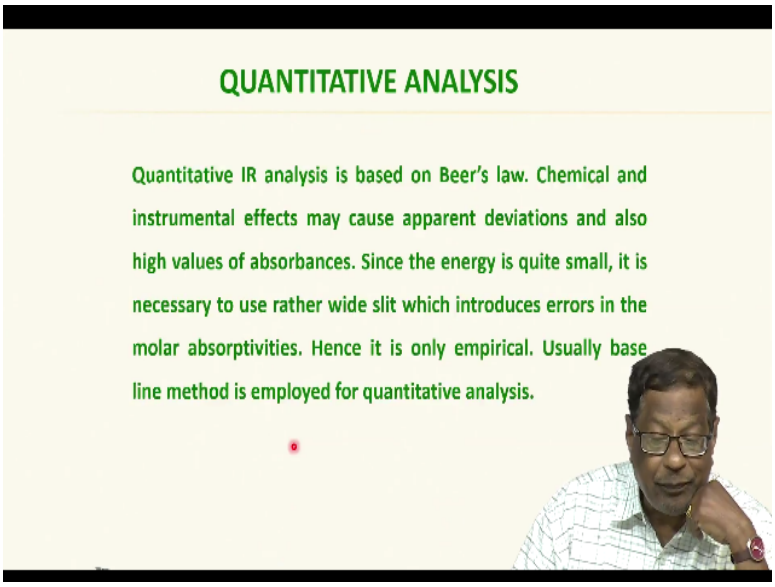


So detection limits of some gases both the instruments are called as non-dispersive infrared spectrometer why because we are not going to use any dispersion, prism, gratings nothing like that, only total IR radiation, it is like a filter photometer, you must have seen many infrared instrument in your gym, if you are in a habit of going to gym or going to doctor there for a pain relieve pain relieving they give you and IR lamp ok.

The IR lamp normally has only a filter that will focus it on the pain. So that the molecules will respond to the pain and functional groups etc. So the pain reduces, so in this case also both these are quantitative spectrum what I have defined early and they are all filter photometers or I just have a small filter to remove unwanted radiation, but all IR radiation I am focusing. So detection limits for some gases of non-dispersive infrared spectrometer.

These are called as non-dispersive infrared spectrometer. If you want to determine carbon monoxide you can determine 1mole% and carbon dioxide is 0.1 mole, SO2 is 0.1 mole, ammonia is 5, CH4 is 1, and 1 more percent and ethylene C2H6 that is ethane is 0.1 mole, C4H10 is 0.1 mole, C2H2 is 1 mole, 1% and C3H6 is this is CnH2n, this is methane, ethane, methane etc. ok.

(Refer Slide Time: 11:22)

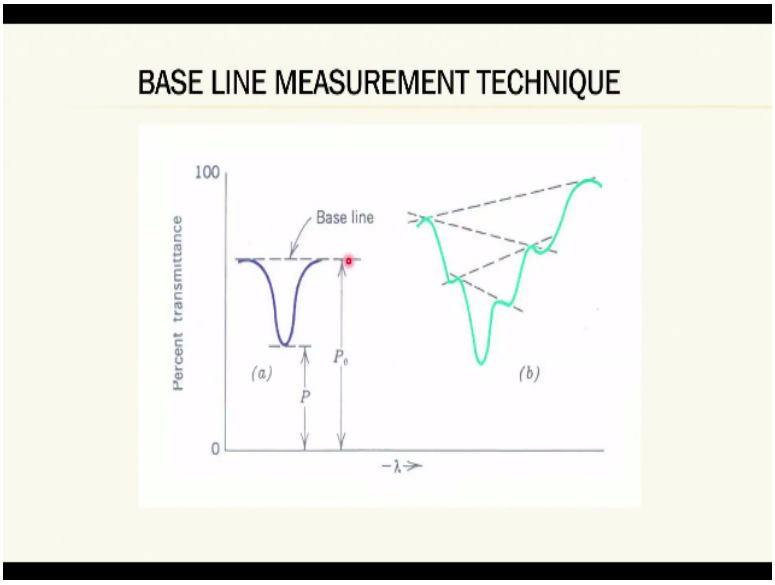


So this is one type of quantitative analysis we use in processes instruments. Now if you want to do quantitative analysis of infrared what type of what you will do in the laboratory for a given specific system is just like what we do in a spectrophotometer quantitative IR analysis is based on Beer Lambert law. So chemical and instrumental effects may cause apparent deviation.

There is certain limit of a Beer Lambert law, there you cannot use very high absorbance values also because earlier in my spectrophotometric course I have taught you that more than 1.2 absorbance should not be used for Beer Lambert law application that will not apply him but some other limit should be applying since the energy changes are quite small in infrared compared to ultraviolet or visible range see.

The energy changes I have already described that electronic straight energy changes or of the order of about 35 to several hundred kilocalories in IR it is only a few kilocalories few calories ok. So it is necessary to use rather wide slit there which introduce errors in the molar absorptivity. So that is what I was making a statement that you cannot use molar absorptivity as an exact quantity because there is always a certain amount of uncertainty involved in this.

(Refer Slide Time: 13:24)

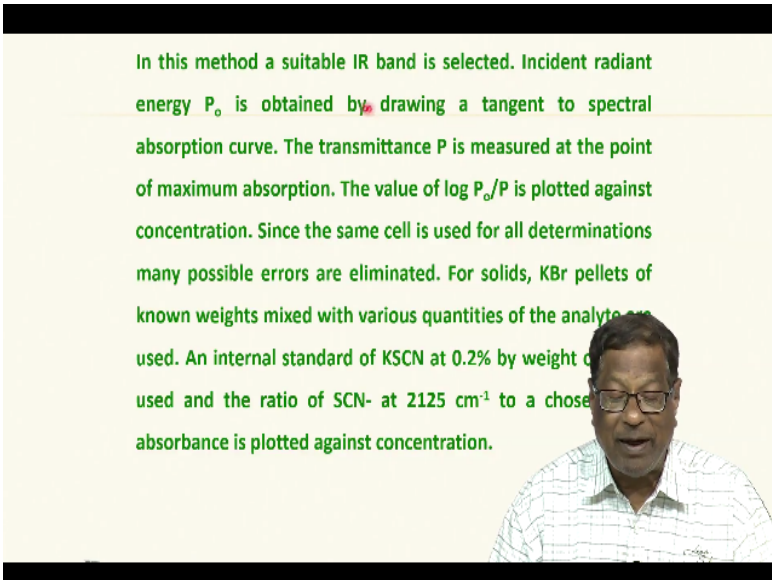


Hence it is only empirical usually base line method is employed for quantitative analysis, how do we do that is a very simple system, so you pick up any IR peak ok. This is in terms of transmittance you know, so IR peak will this is 100%, 100% transmittance means it is the baseline ok and then you choose the percent transmittance from the bottom of the peak, this is the absorbed ok, the amount of this is the incident radiation P and this is P0, P0 is the original radiation and P is the transmitted radiation.

What is P0-P is what is observed, so that you correlated to concentration, this is 0%, this is 100%, this is wavelength, wavelength has no meaning if you want to do a particular quantitative analysis. Now a typical IR spectrum would be so simple at all, but it will be like what I am showing you there in b, here you see IR peak is quite complex and if you are choosing this peak you do not know whether to use this as the base peak or 100% transmittance or this as the base peak or this as the base peak or this is a base peak.

But you can be consistent and use whichever when you want to use, use it, but be consistent and use the same base peak for all these things, that is important. So IR and you have to take different concentrations weigh them accurately and then take the IR spectrum and then correlate to the absorbance, draw a separate plot and then determine the unknown, so it is quite a bit complicated structure but it can be done ok.

(Refer Slide Time: 15:20)



So in this method what to do with a suitable IR band is selected incident radiant energy P0 is obtained by draw a tangent, transmittance is measured, P log, P0/P is plotted against concentration. Same cell is used for all determinations many possible errors are eliminated. For solids where to again weight the KBr's because we are going to make the KBr pellet. So when we use the KBr quantity also should be exact.

So they are mixed with various quantities of the analyte and we take the IR spectrum, I can use an internal standard of potassium thiocyanate at 0.2 degree or 0.2% by weight of KBr and that shows a peak of 2125cm inverse to a chosen band is plotted against concentration like as simple as it ok.

(Refer Slide Time: 16:24)

So this is where most of our infrared discussion ends and I hope I have given you fairly good amount of knowledge regarding the atomic absorption sorry regarding the atomic structure, electromagnetic radiation, interaction of electromagnetic radiation and then instrumentation followed by application of IR. I wish you all the best in your endeavours for your future studies or investigations or learning procedure, learning programs.

And I just want to end my lecture, I wanted to show you this IR spectrometer, the current IR spectrometer look something like this, the left side is the IR peak, IR source, and optics this is the sample and here is the microprocessor. So everything looks very sophisticated and beautiful, but the interpretation is also complicated, but it can be done.

So I wish you all the best in this course and I hope you have learnt something more useful than what they teach you at the universities or something like that, because there is nothing like learning from a teacher, whatever doubts you have etc. you please contact me I will be able to help you and the best way to learn is to study, study and study. So thank you very much, and all the best, bye, bye, good luck.