Industrial Instrumentation
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Lecture - 35
Chromatography – I

Welcome to the lesson 35 of industrial instrumentation. We will start to discuss the chromatography. In general actually we call it gas chromatography though it is more popularly known, but it is actually it is a chromatography, because the chromatography is both for the gas and the liquid. So, in this lesson and subsequent lesson that is lesson 36, we will consider the gas chromatography; we will consider the chromatography. Let us look at the contents of the lesson 35.

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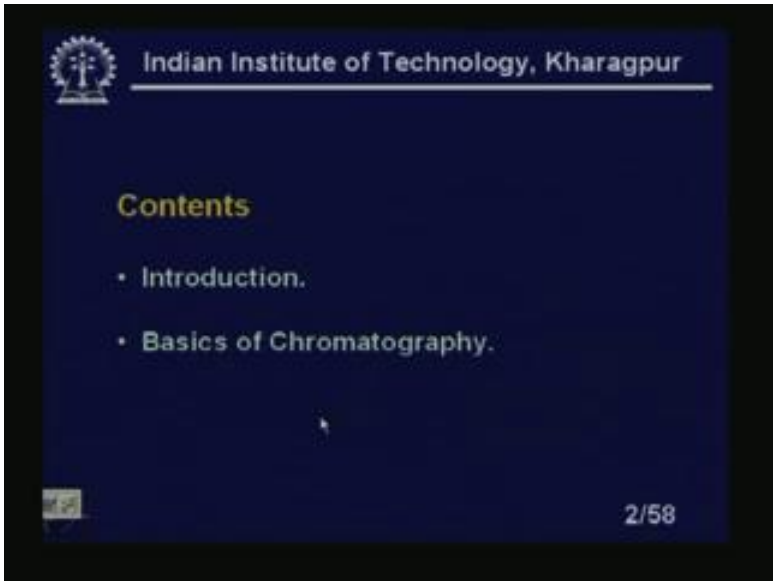

We will basic have introduction to the systems what they actually chromatography look like? What is the packing material is used and what is actually where it is used to? All those things we will discuss. Now the chromatography is basically the separations of the fluids. Because in many industrial situations as well as in the chemistries we need this type of situation when there is a mixture. So, we want to know it is very difficult to know the content of the percentage content of the mixture of the 2 gases.

So, in that case we can apply the chromatographic method to separate the 2 gases. And find the percentage content of the individual gases or individual components when it is

more than 2 and 3 components present in the gas. So, basically those things we will discuss how it works actually what is the illusions? We are talking about and how actually we will know from the peak of that the percentage of the individual components of the fluids I mean exists in the gas. So, those things we will know by in this particular lesson. Introduction then you has basic chromatography. We will discuss the basic chromatography here.

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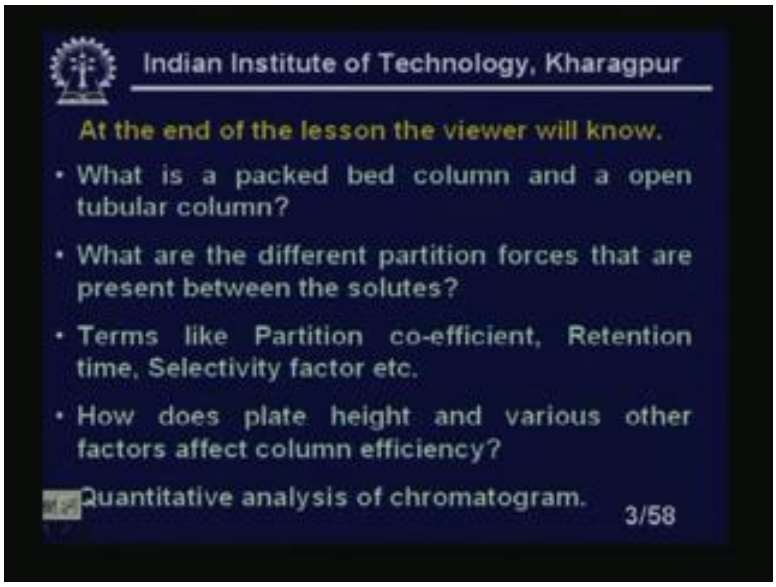

At the end of the lesson, the viewer will know: what is the packed bed column and open tubular column? Because we will see in the chromatography, we will find that the basic principle is something like this. That we will send the mixture of the gas or fluid through the through a column and it initially absorbs the both the thing absorbs or adsorbs the both the both the mixture. I mean the mixture, but we will see that depending on the characteristics of the individual component. It will elute that mean it will relieve or it will release the gas or fluid with the difference of time. So, that by that actually we can know we know we can measure the contents or we can measure the concentration of the gas.

So, this is the basic I mean philosophy of the we have different detectors. All those things will be discussed these lessons as well in the lesson 36. So, we need that I mean the column react I mean those tubular column or packed bed column, so through which the mixture will be passed. So, we will discuss what is the packed bed column and open

tubular column? Then we have what are the different partition forces that are present between the solutes? Terms like partition coefficient retention times selectivity factors all these things we will know in this particular lesson.

How does the plate height we will see, what is the height plate height and the various other factors affect the column efficiency? Because efficiency column is very important not only to know the contents of the individual components of the gas, because it depends on the material of the column. So, efficiency of the column it will also distinguish between the 2 contents 2 components very nicely. Otherwise it is very difficult to find the percentage contents of the individual components in the mixture. Quantitative analysis of the chromatogram how we can make quantitative analysis of this gas chromatography or chromatogram? The plot which will get from this chromatography we call it chromatogram.

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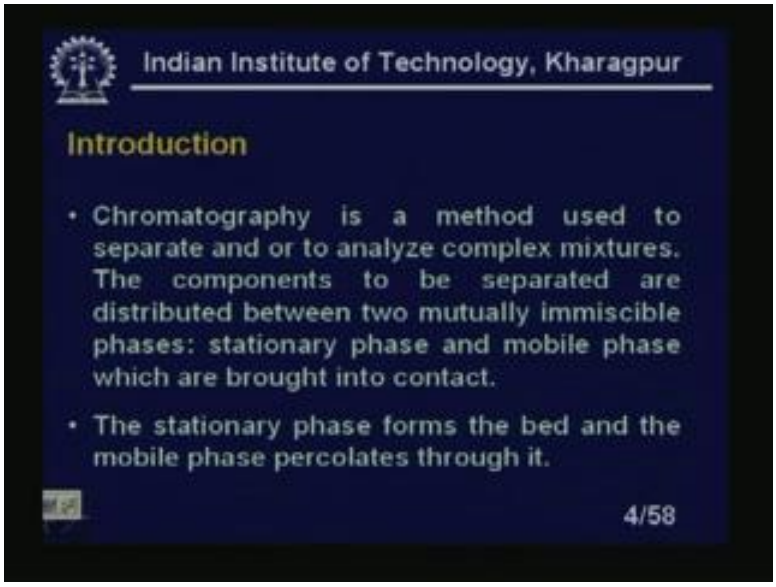

Introduction; chromatography is a method used to separate or to analyze complex mixtures. The components to be separated are distributed between 2 mutually immiscible phases; stationary phases and mobile phase which are brought into contact. So, there are 2 phases these terms we will very frequently referred throughout this lesson. That means, in stationary phase and in the mobile phase. The stationary phase forms the bed and the mobile phase percolates through it. So, we have a bed this is the packed bed column and

we are talking about we are, so that stationery phase will make that bed and the mobile phase will percolates through it.

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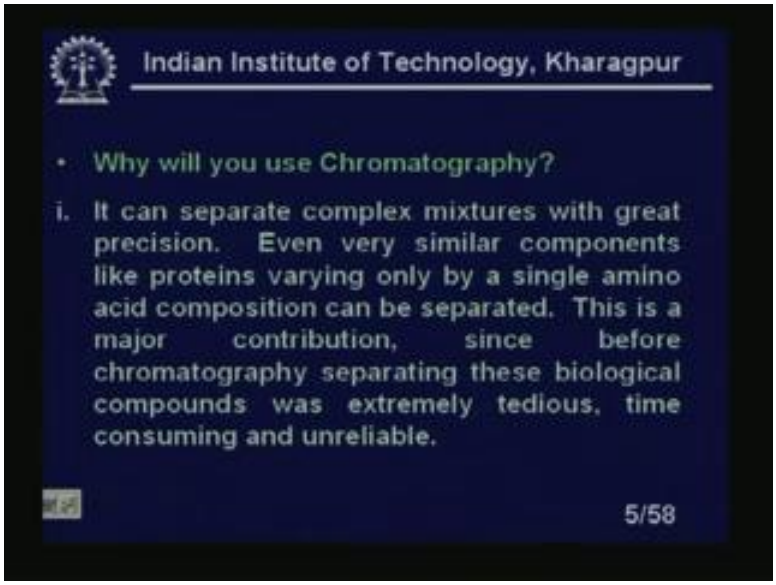

Why we use chromatography? Why do you need this gas chromatography or chromatography in general? Let us look at. It can separate complex mixtures with great precision. This is most important thing. I have 2 mixtures or 2 or 3 gases in a particular mixtures. I want to precisely know; that means, first of all you have to separate. If you cannot separate precisely you cannot measure the contents of the individual gas. So, the precision in separation is also very important in the case of gas chromatography. So, that is the reason I am telling it. It can separate complex mixtures with great precision.

Even very similar components like proteins varying only by a single amino acid composition can be separated. This is most important it will I mean in many cases we need these type of separations. So, different components with different chemical compound with even with the single amino acids differences can be separated. This is a major contribution since before the chromatography separating these biological compounds was extremely tedious and time consuming and unreliable. Chromatography is quite reliable ((Refer Time 06:50)) that since.

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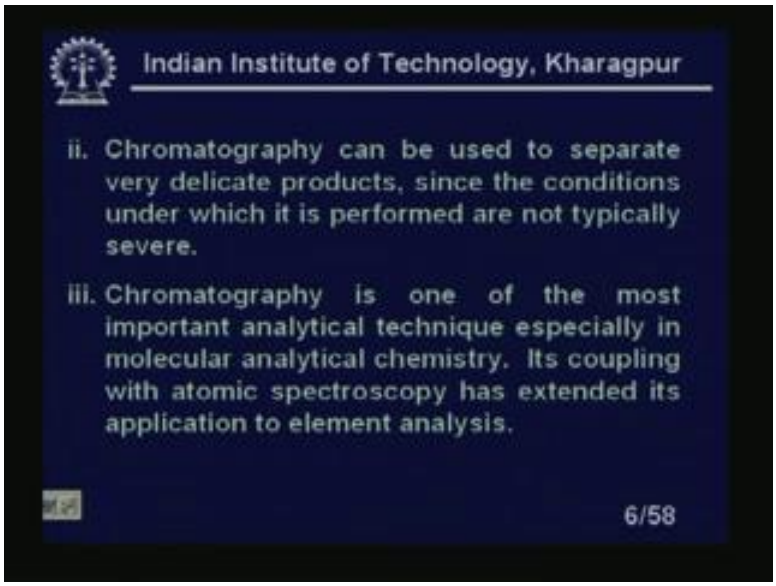

Chromatography can be used to separate very delicate products. Since the conditions under which it is performed are not typically very severe very delicate products also. That means, it is temperature is not very severe; that means, it will have that is the reasons I am telling that it is not very typically not very severe. That means, temperatures moistures everything is not very extreme. So, a normal room temperature you can do it.

So, that is the reason very delicate compound very delicate I mean complex of the gases all those things can be nicely separated in this particular system. Chromatography is one of the most important analytical techniques especially in the molecular analytical chemistry. It is coupling with atomic spectroscopy has extended it is application to element analysis. So, with the advent with the advancement I mean in the spectroscopy atomic spectroscopy we can find we can identify the element analysis also. It can be used in the element analysis.

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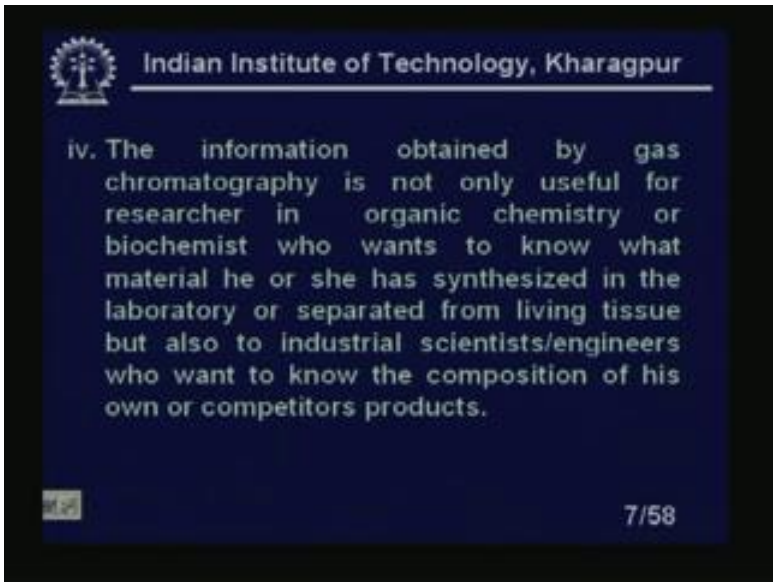

The information obtained by the gas chromatography is not only useful for the researchers in organic chemistry or biochemist who wants to know what the material he or she has synthesized in the laboratory or separated from the living tissue, but also to the industrial scientists and engineers. That is the reason we are discussing in this course of industrial instrument. It has tremendous application industrial instrumentations where we need to separate where we need 2 compositions of the gases. Especially composition of the gases is important to know I mean it is very important I mean parameters in many situations of his own or competitor's products, because suppose I mean an unknown products is coming. So, we do not know what is the composition? So, with the gas chromatography we also known not only the percentage, but also know that what is the different composition of the gas is present?

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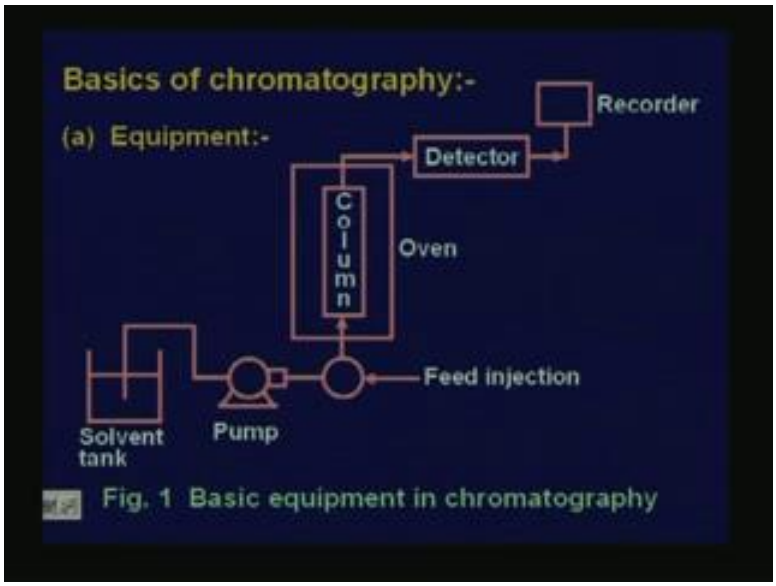

Basic chromatography equipment's what is the equipment? Let us see this is the basic equipments. We have a solvent tank you can see here. You see we have a solvent tank here we have pump. So, through a pump we are putting it and this is a feed injection it is coming here and it is through a column it is going. Now, we will see that how this column, because it will retain there for sometime after that it will dilute it will release1 by 1 the gases. So, that will be detected by the recorder and that will be automatically recorded in a recorder strip chart recorder or a xy recorder. So, so that type of cases we can find that. So, from the peak we can know the percentage composition of the gas.

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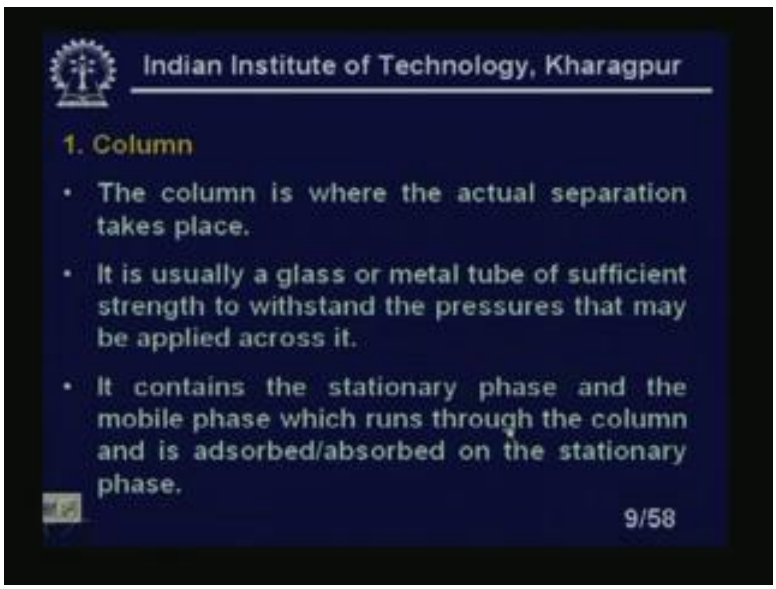

Column what is column? The column is where the actual separation takes place. Separation takes place separations takes place the usually the initially in the column all the gas or the mixture will go inside. So, it will retain depending on the properties. So, it will elute 1 after another the gas one after another. It is usually a glass or metal tube of sufficient strength to withstand the pressures that may be applied across it. It contains the stationary phase and the mobile phase, which runs through the column and is adsorbed or absorbed on the stationary phase. Adsorptions or the absorptions, so it contains the stationary phase and the mobile phase, which runs through the column and is adsorbed or absorbed depending on that stationery phase.

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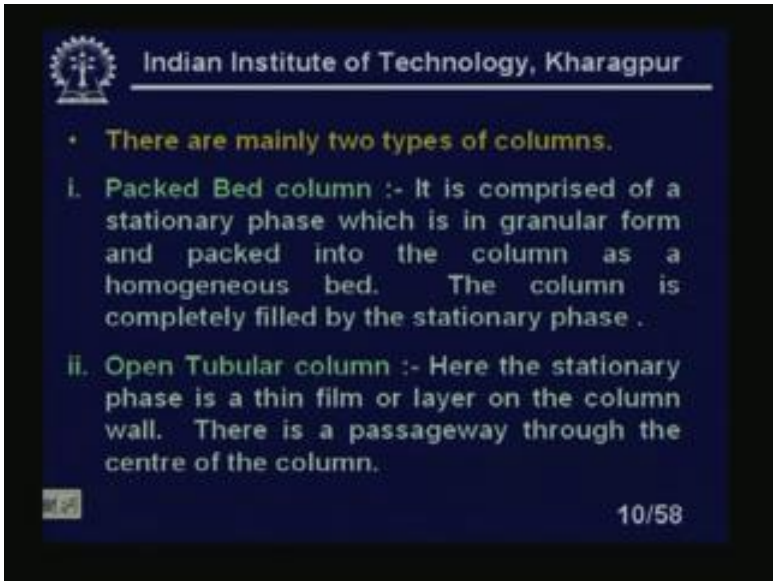

There are many mainly two types of columns. We have the packed bed column and open tubular column. So, let us discuss 1 by 1. It is comprised of a stationary phase, which is in granular form and packed bed into the column as a homogenous bed. The column is completely filled by the stationery phase. Open tubular column here the stationary phase is a thin film or layer on the column wall. And there is a passageway through the centre of the column. Through the centre of the column there is a passage through which the feed will pass through.

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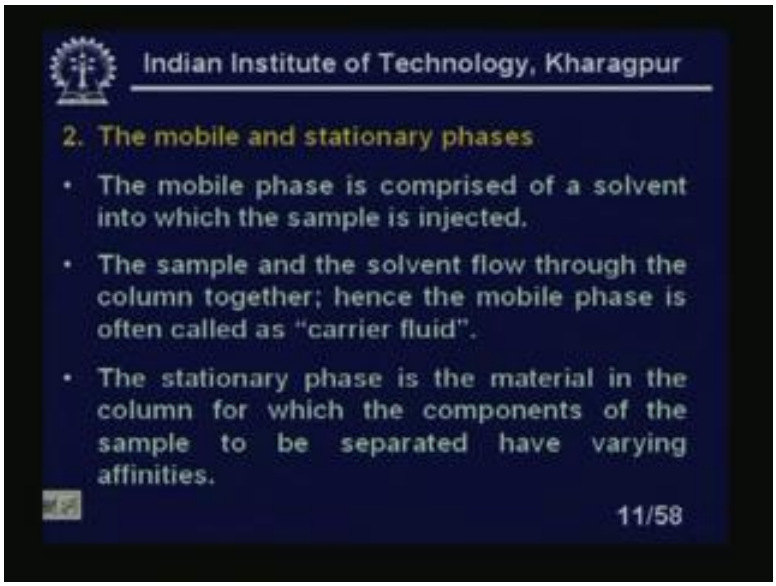

The mobile and stationary phase this is the 2 things we are repeatedly telling. The mobile phase is comprised of a solvent into which the sample is injected. So, mobile phase is a sample is a comprised of a solvent into which the sample is injected. We have seen in the in the figure in the through a pump a solvent is coming and the sample is injected through which is called the feed which is injected through this 1. So, this is the mobile phase the sample and the solvent flow through the column together. So, with these both the sample and the solvent flow through the column together.

Hence the mobile phase is often called a carrier fluid. So, sometimes we are calling it carrier fluid. The stationary phases are the material in the column for which the components of the sample to be separated have varying affinities. So, it should have a varying affinities otherwise you cannot I mean separate that is the most important thing in the chromatography. So, that is the reason third ((Refer Time: 11:56)) I am explaining the stationary phase is a material in the column for which the components of the sample to be separated having varying affinities.

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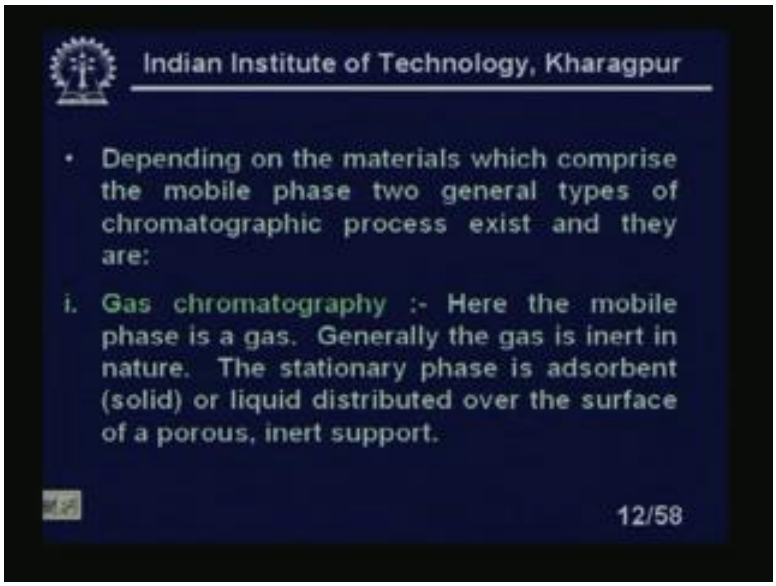

Depending on the materials, which comprise the mobile phase 2 general types of chromatographic process exist. Depending on the material, which comprised the mobile phase 2 general types of types of chromatography process exists and they are gas chromatography it depending on the mobile phase. Here the mobile phase is a gas generally the gas is inert in nature and the stationary phase is adsorbent solid or liquid distributed over a surface of a porous inert surface inert support. So, this porosity is very small in order of Armstrong please note. This is important this porosity size of the porosity or the also. We will I make the affinity of the different affinity to the different gases or components of the different gases which mixture of the gases.

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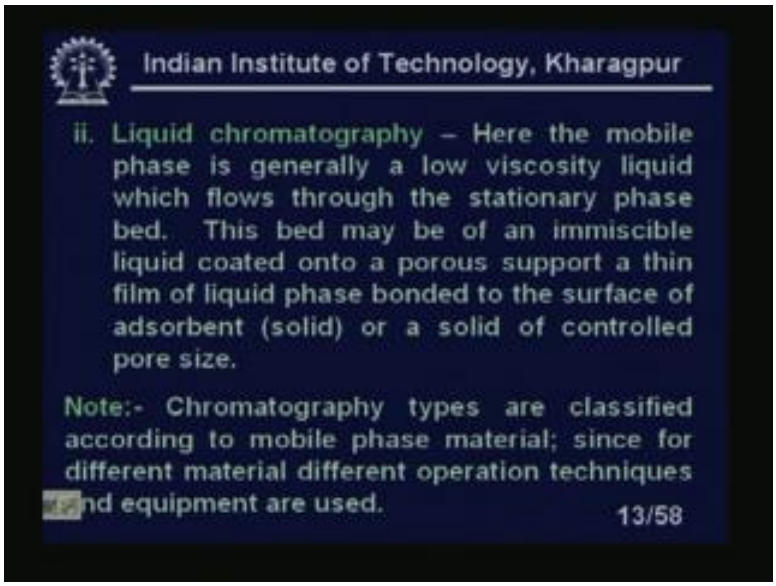

And the liquid chromatography here the mobile phase is generally a low viscosity liquid which flows through the stationary phase bed. And this bed may be an immiscible liquid coated onto the porous support a thin film of liquid phase bonded to the surface of adsorbent solid or a solid of a controlled pore size. Note chromatography types are classified according to the mobile phase materials. Since for different material different operation techniques and equipment are used.

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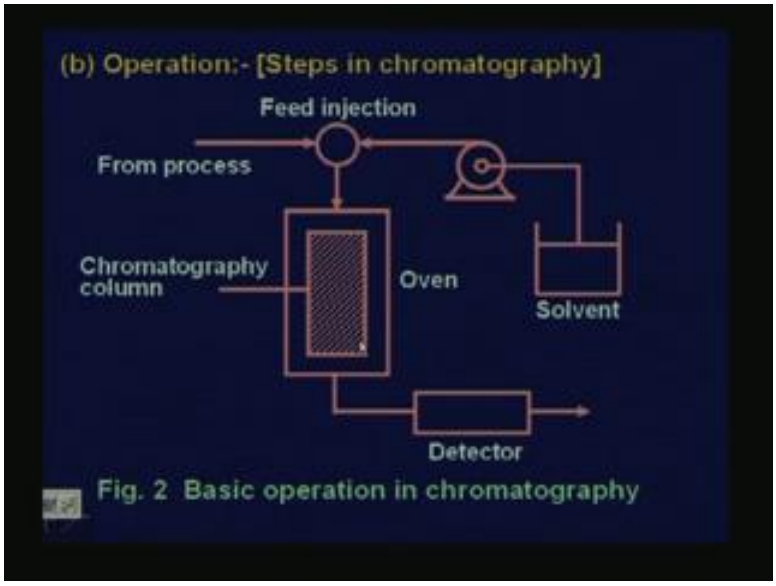

Operation let us look at steps in the chromatography. So, you see here we are I mean just change the directions. So, that from the feed injections we have solvent here we are feed injections it can be other way also. In this case it may come through gravity, so other case we have to use to the pump. So, basic operations in this after this it will go to a detector after this it will go to a sorry it after this it will go to a recorder, so this is a detector same thing solvent oven. So, it is if oven is necessary suppose in many some situations we have to have to use a control temperature, so in that type of situation we need an oven. So, that is the reason we put in the oven, but it is most of the cases we do not temperature is not very I mean crucial in that cases we can use the normal room temperature.

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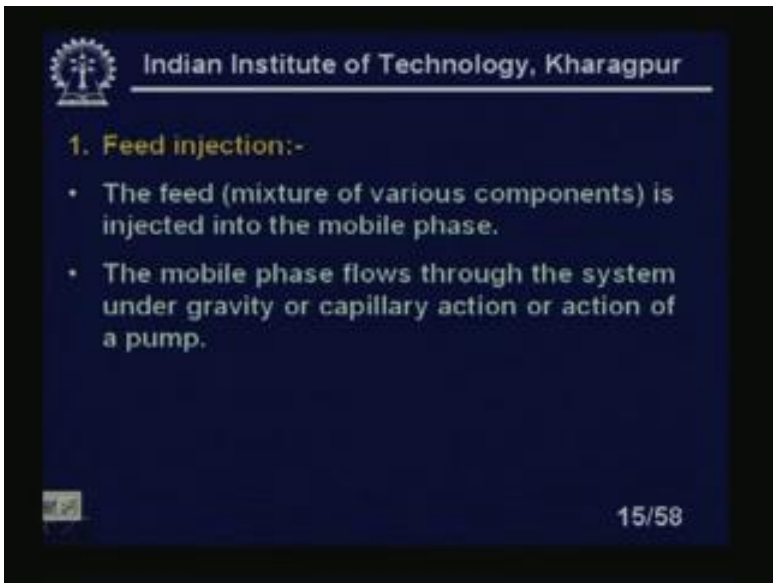

Feed injection the feed mixture of various components is injected into the mobile phase. Obviously, it is a mixture individual component, if it is a single gas, I mean single component; obviously; we are not using going to use a chromatography or gas chromatography. Number 2; the mobile phase flows through the system under gravity or capillary action or action of the pump. So, there are 3 possibilities either it will through gravity it will move or through capillary actions it will move or the action of the pump it will move.

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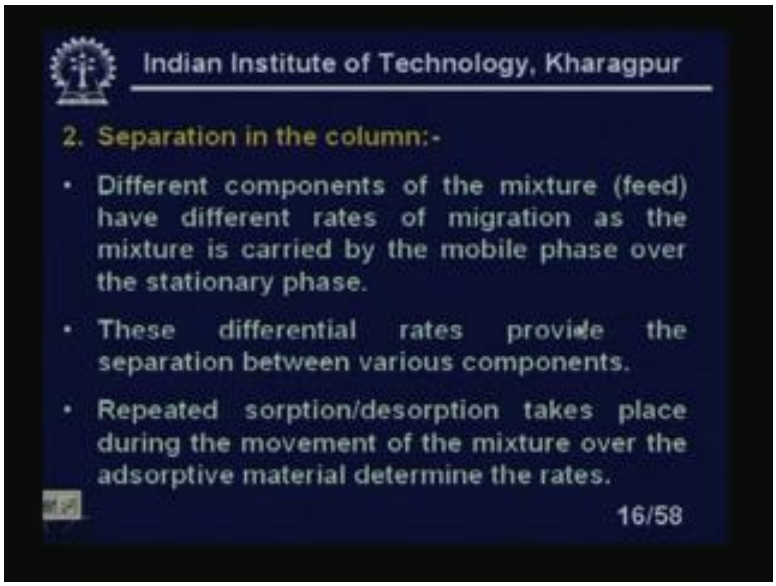

Separation in the column the different components of mixture feed have different rates of migration as the mixture is carried by the mobile phase over the stationary phase. These differential rates provide the separation between the various components. Repeated sorption and desorption takes place I mean I do not know whether it desorption is in dictionary. But it is opposite to desorption's takes place during the movement of the mixture over the adsorptive material determine the rates.

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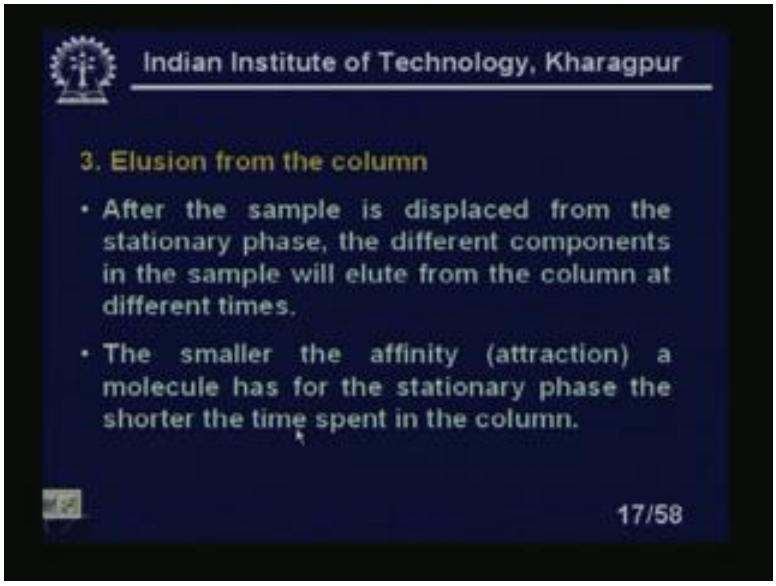

Elusion from the column you see. After the sample is displaced from the stationary phase the different components in the sample will elute from the column at different times. This is the important thing in gas chromatography. Sample will elute from the column at different times if it is not in different times we cannot separate. Since, it is I mean coming at different times we can separate we can know the percentage we can know the individual concentrations of the gas component.

So, this is most important terms in on gas chromatography elute. The smaller the affinity attraction a molecule has for the stationary phase the shorter the time spent in the column. So; obviously, we will find that there will be lesser affinity I mean affinity will be different I mean if you measure the affinity. So, it will be affinity 1 has a larger affinity than other. So, this will make our separation possible otherwise the separation was impossible.

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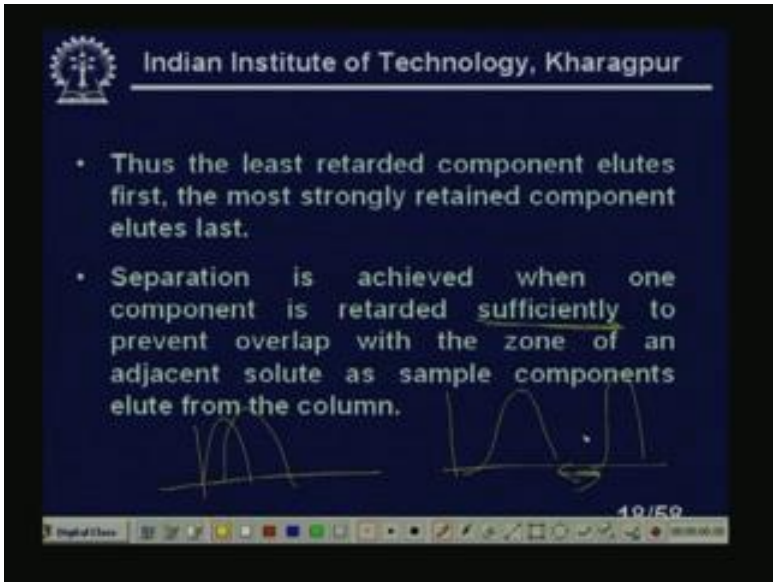

Thus, the least retarded component elutes first. The least retarded component elutes first and most strongly retained component elutes last. It will come at the end and the most least retarded component will come at the first. Separation is achieved when one component is retarded sufficiently to prevent overlap with the zone of an adjacent solute as sample components elute from the column. This is most important thing if there is a overlapping there is a problem. So, separation is achieved when the 1 component is retarded sufficiently.

So, this is the word you see sufficiently to prevent overlap with the zone of an adjacent solute. As a sample component elute from the column we will see later on it will come like this 1 peak like this 1. However, if the curves looks like this it is very difficult to it is very difficult to I mean separate. So, in that case, so that is the reason we have to sufficiently to prevent overlap. So, there is a sufficient gap or time with the zone of an adjacent solute as sample component elutes from the column.

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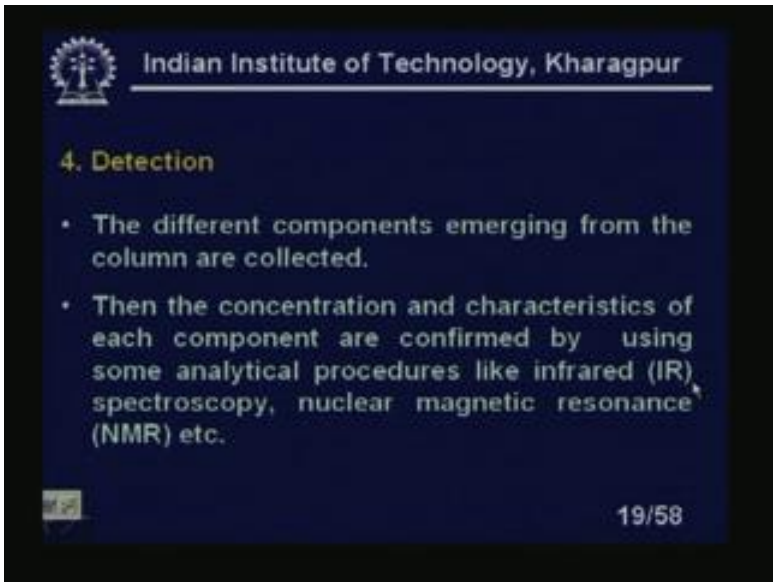

Detection the different components emerging from the column are collected. And then the concentrations and the characteristics this is the most important. The concentration and the characteristics of each component are confirmed by using a same some analytical procedures like infrared IR spectroscopy or nuclear magnetic resonance NMR techniques. So, we can identify the element we can find the concentrations. So, the concentrations and the characteristics of the gas which is coming out which elutes; obviously, with the time difference when it is coming out I can measure it I can detect the elements also.

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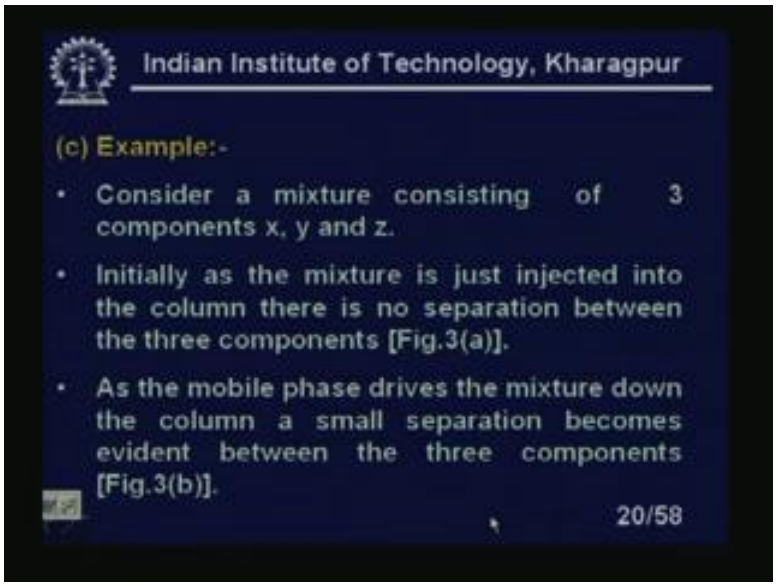

Now, for an example a we considers a mixture of component xyz 3 gases are there. 3 components are x y and z. Initially as the mixture is just injected into the column there is no separation between the 3 components. Let us see otherwise, it is very difficult you see here.

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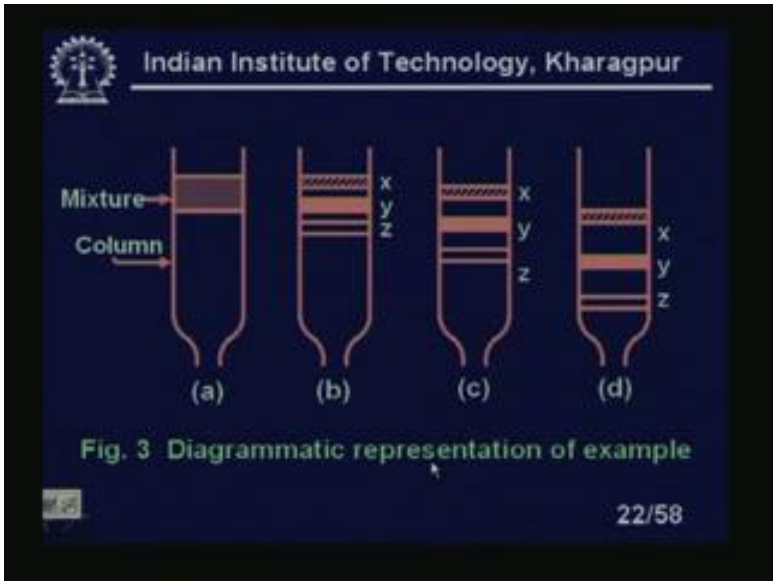

So, in the mixture you see initially 3 gases are here. This is the time axis we are plotting. This is the time x axis is the time y axis is the separation between the 3 gases; that means, elutes at it elutes. You see as the time goes by x y z the 2 I mean x has a least z

has a least affinity. So, it is coming out first. You see as the time goes by, so the separation between the I mean separation means the time required time needed to come out of the I mean column is more and more. So, 3 components now, here the 3 components are mixtures x y z 3 are mixture here you see as the time goes by, so if the separation; that means, elution time is becoming more and more.

So, it is coming out of the gas 1 by 1. So, z will be detected then y will be detected then x will be detected. So, I can go back. Consider a mixture consisting of a 3 component x y and z initially as the mixture is just injected when it is just injected in the column. There is no separation between the 3 components that we have seen there is no separation between the 3 components as the mobile phase drives the max the mixture down. The column mixture down the column a small separation becomes evident when the mobile phase is moving through the column. A small difference a small separation becomes evident between the 3 components which is shown in figure 3 b.

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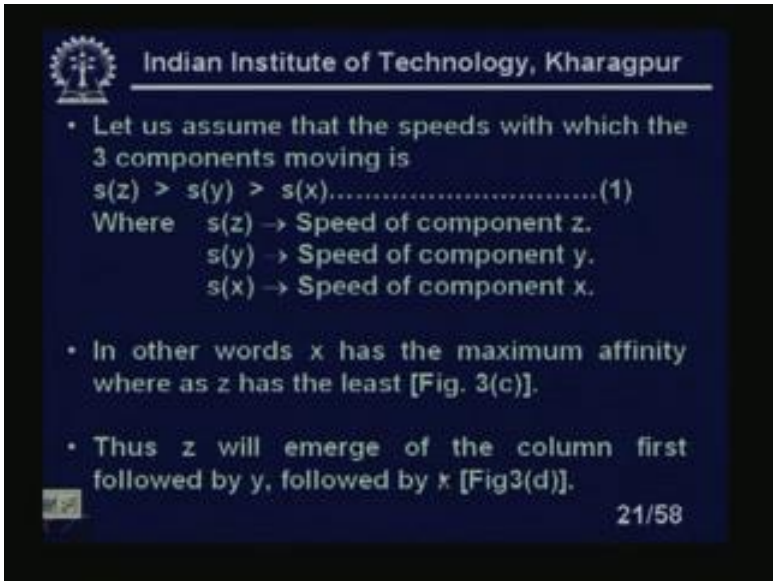

Let us assume that the speeds with which the 3 components moving is sz more than sy more than sx where sz is the speed of the component z, where sz is the speed of the component z; sy is the speed of the component y and sx is the speed of the component x In the other words x has the maximum affinity. So, the affinity is different for the different components where z has the lease affinity this is partially shown in figure 3 c. Thus, z will emerge from the column first followed by y and followed by x.

This is given in 3 this the things which we have there is a mixture there is no separation. When the mobile phase is moving through this 1 slowly there is a separation. So, at the end there is a large separations between separations; that means, of the of the elusion time; that means, elusion times becoming more and more in the in the case of the x. It is retention time is most and in the case of y the retention time is medium and in the case z the retention time is minimum. So, there is a diagrammatic representation of a ((Refer Time: 21:03)) of example.

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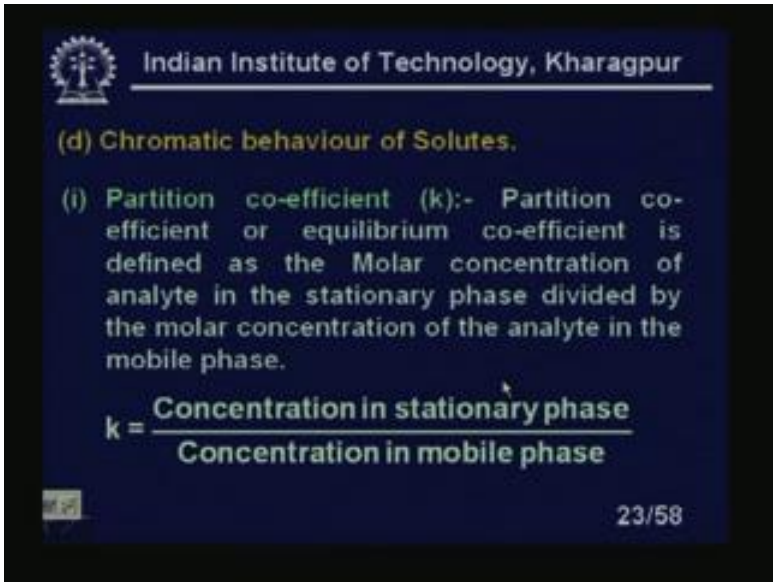

Chromatic behavior of a solutes let us look at partition coefficient; there is some terminologies we are going to introduce. Partition coefficients or equilibrium coefficient is defined as the molar concentration of analyte in the stationary phase divided by the molar concentration of the analyte in the mobile phase. Analyte means this is which we are going to make the analysis, but k is given by concentration in stationary phase upon concentration in mobile phase.

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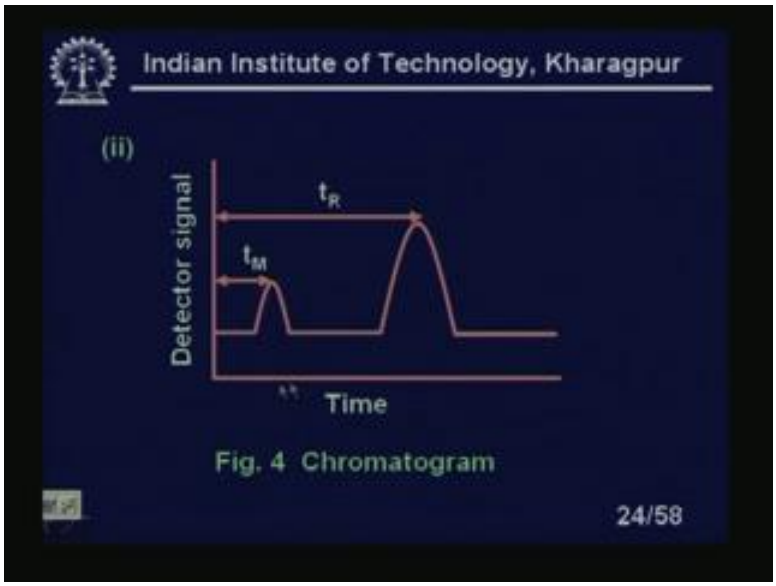

You see here is the chromatogram we are talking about the plot you see there here there are detected signal. So, x axis we are plotting time. You see the retention time this is the time, because after injection. So, see when it elutes the tM is coming first then tR will come in with the peak. So, these are the 2 peaks. So, this is called this type of plot is called the chromatogram in the case of gas chromatography.

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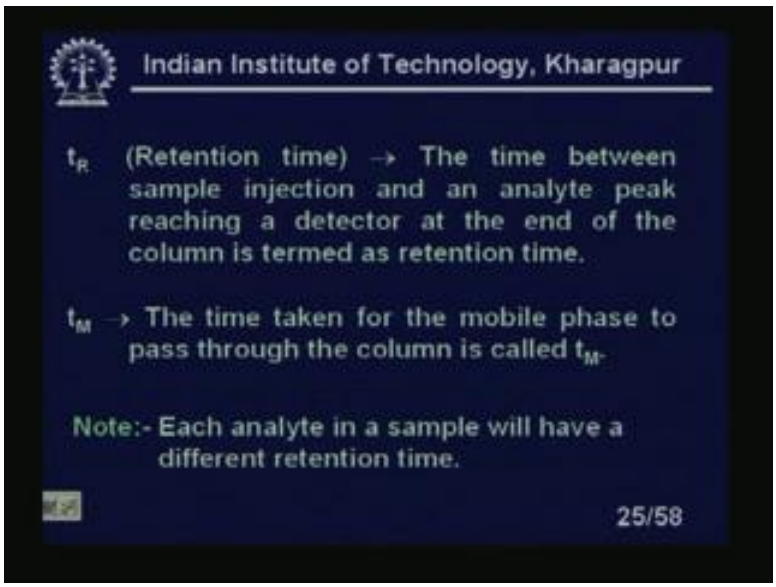

Retention time you see here we have written tR and tM. Retention time the time between the same sample injection and analyte peak reaching a detector at the end of the column

is termed as retention time. So, is the time between the sample injections and an analyte peak reaching a detector at the nd of the column? This term is retention; that means, here it is reached the detector. So, here from here it reached the detector TM is the time taken for the mobile phase to pass through the column is called tM time taken by the, for the mobile phase to pass the column is called tM. So, this individual gas we have shown 1 component of the gas we have shown that it is retention time tR and tM is the time.

Obviously, you can see that the tR is; obviously, that tR should be more than tM in all the cases. So, the time taken for the mobile phase to pass through the column is called tM. So, we will get similar graphs similar type of tR. That means, we will get similar tR this is for 1 gas we can get you will get these for the several other gas or the mixture of the components. Each analyte is a in a sample we will have a different retention time. That's I am saying, so with this we are showing for a single element. So, there will be if you have a 2 such I mean 2 such gas I mean gas which is making mixture. So, we will have 2 retention time tR 1 tR 2, if you have 3 so on.

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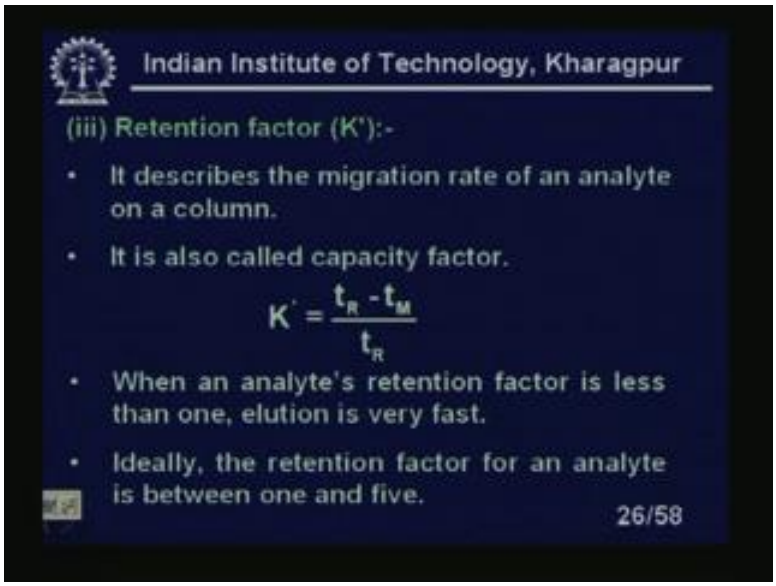

Retention factor K dash it describes the migration rate of an analyte on a column. It is also called a capacity factor. K dash equal to tR minus tM by tR. Obviously is a positive, because tM is always less than tR. When an analytes retention factor is less than 1 elution is very fast that is very undesirable I mean elution should normally be very fast. If the elution is fast, so it is very difficult to I mean make the analysis. Typically ideally

or I should say typically retention factor for an analyte is between 1 and 5 for to be 1 and 5.

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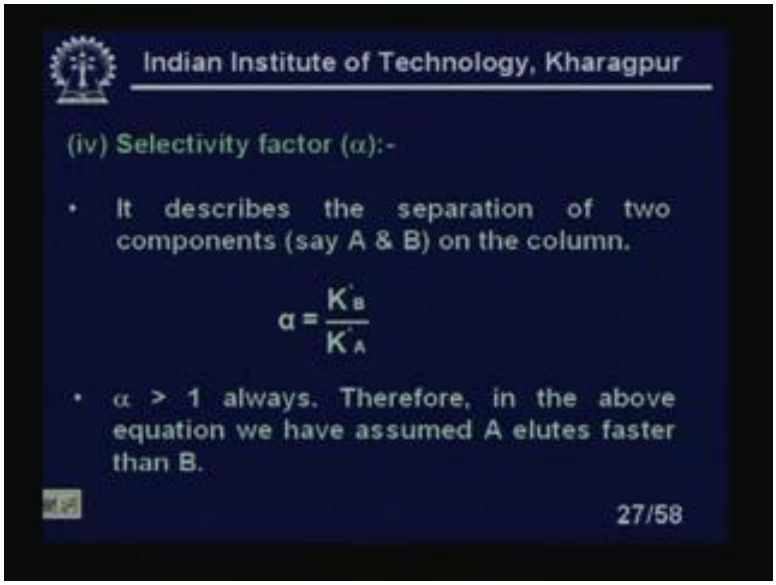

Selectivity factor alpha it describes the separations of the components say A and B on the column. It describes the separation of the 2 components say A and B on the column. Alpha equal to K dash B upon K dash A. 2 components we have K dash B K dash B and K dash A. Alpha is greater than 1 always therefore, in the above equation we have assumed A elutes faster than B. So, A is coming out and faster than B.

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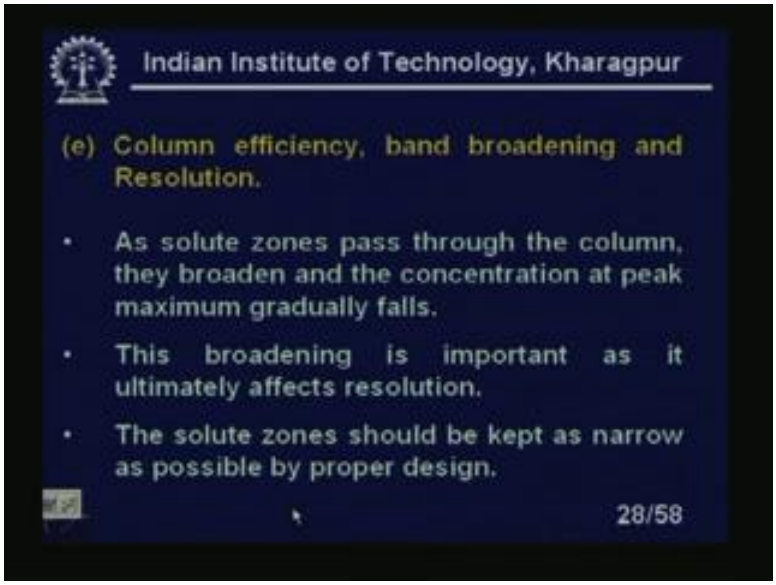

Column efficiency band broadening and the resolution these are the important parameters. As solute zones pass through the column, they broaden and the concentration at peak maximum gradually falls. This will more important. So, whenever we more clear when you show the graph. This broadening is important as it ultimately affects the resolution; that means, how closely how close the characteristics I mean in the case suppose the measurements. Suppose I have a voltmeter, so resolution means what?

What is the minimum voltage of separations it can detect? Whether it can detect 2.77 and 2.776 volt or it will be 2.7 or 2.6 volt. Obviously the voltmeter which can have resolutions of which can measure 0.001 volt so; obviously, that will be preferred. Similarly, in this case in the case of resolution 2 is broadening if it is broadened. So, it is; obviously, we say that is resolution is poor. So, broadening is important as ultimately affects the resolution. If the broadening is higher the resolutions will be poor. The solute zones should be kept as narrow as possible by proper design. So, we should have a very narrow zone, so that the peak will be very sharp.

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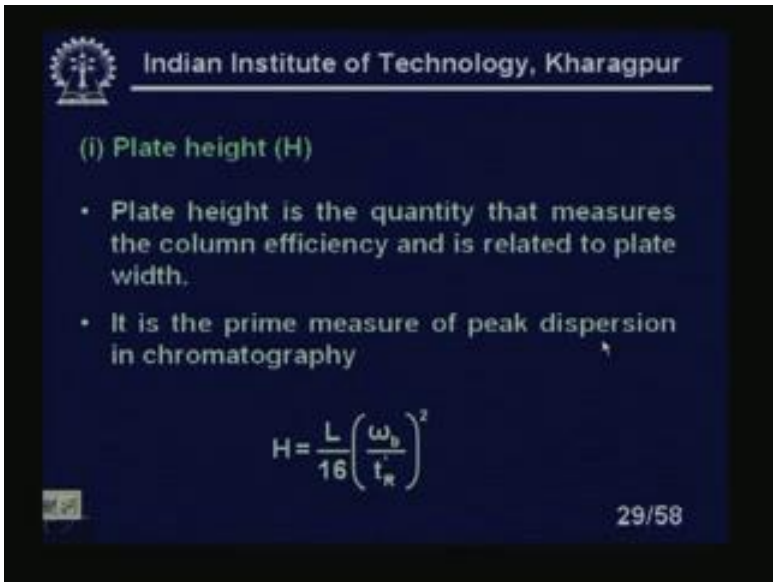

Plate height this is another I mean parameters will include here. Plate height is the quantity that measures the column efficiency and is related to the plate width. We will show what is the plate height and plate width? It is a prime measure of peak dispersion in chromatography. It is a prime measure or peak dispersions in chromatography, which is given by H equal to L by 16 omega B upon tR dash whole square.

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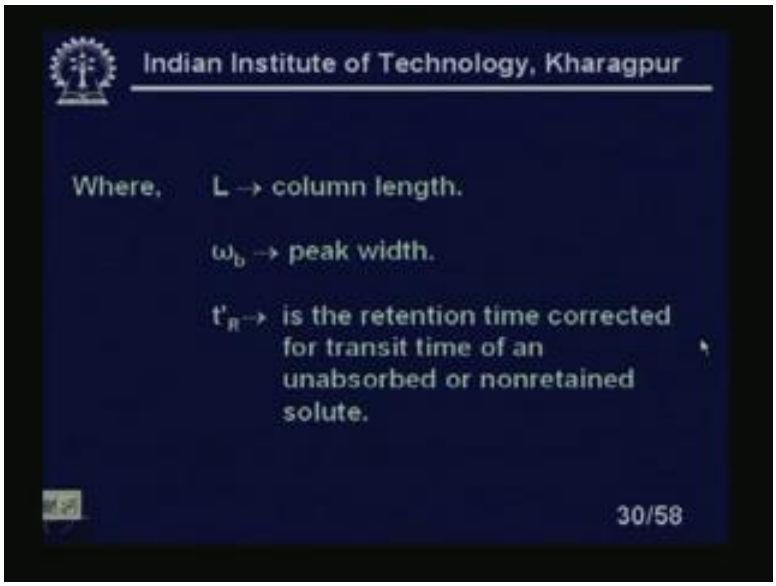

What is this ((Refer Time: 26:38)) let us explain where L is the column length; omega B is the peak width; and t dash R is the retention time corrected for the transit time of an unabsorbed or non retained solute.

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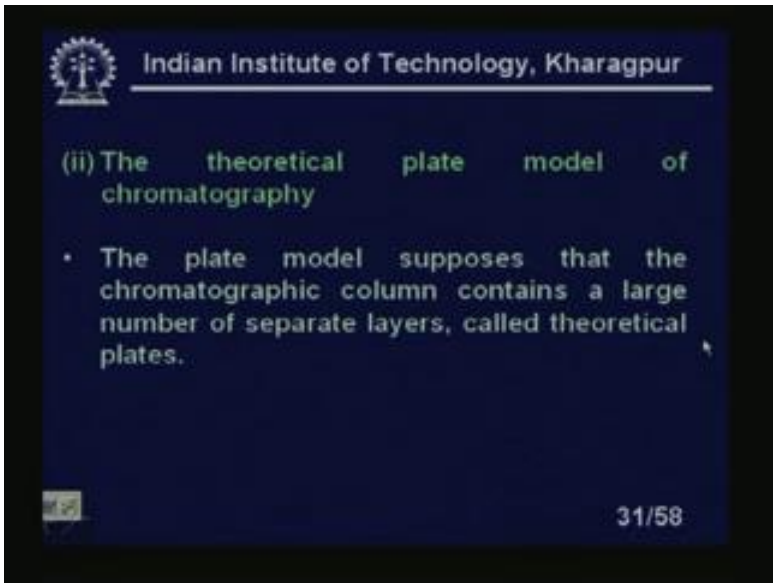

The theoretical plate model of the chromatography we are assuming I mean in a theoretical model, we are assuming there is a separate plate I mean remains there, but actually it is not it is a mixture. So, the plate model supposes that the chromatographic column contains large number of separate layers called the theoretical plates.

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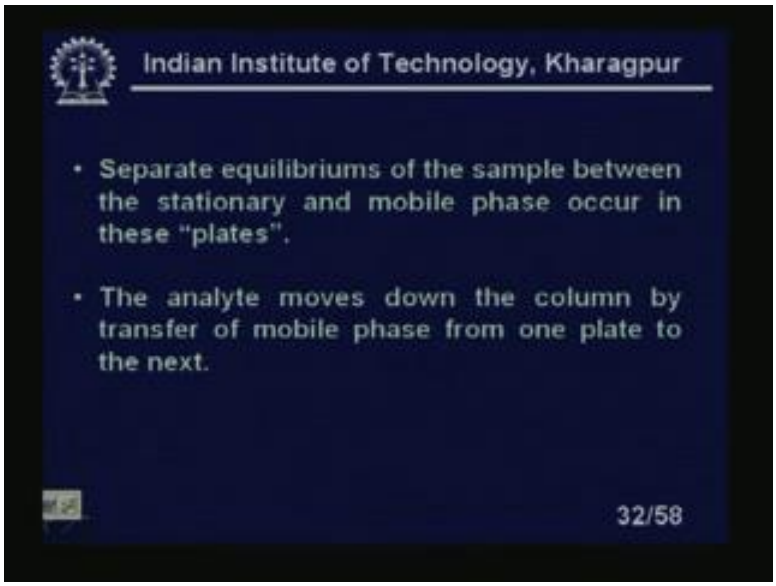

Now, separate equilibrium of the sample between the stationary and the mobile phase occurs in these plates. The separate equilibrium of the sample between the stationary and the mobile phase occurs in these plates. We are assuming there is a separate plate. The analyte moves down the column by transfer of mobile phase from 1 plate to the next. 1 by 1 we have assuming that there are a several plates. So, 1 plate to another plates I mean analyte is moving with a in a presence of the mobile phase.

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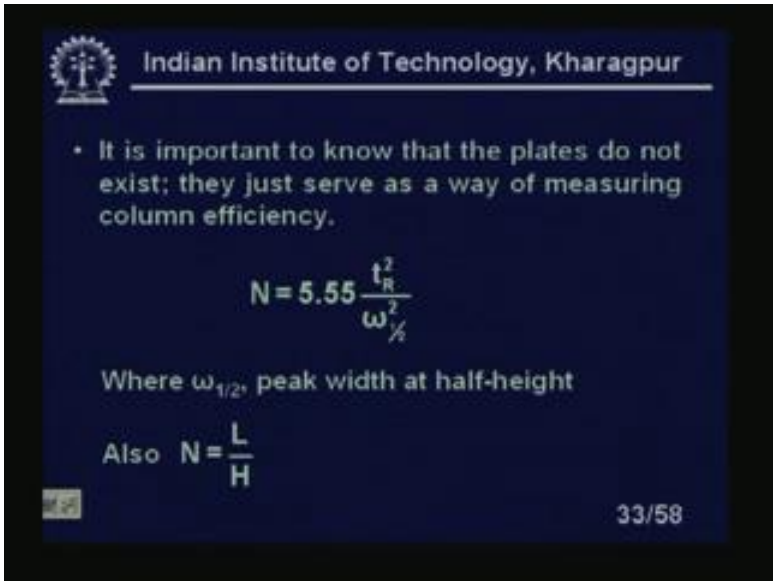

It is important to know that the plates do not exist plates do not exist; they just serve as a way of measuring column efficiency, because the column efficiency is important. N equal to 5.55 tR square upon omega square omega half to the power square. What is this? Where omega half is the peak width at half height. It is the peak width that the half height and N equal to L by H.

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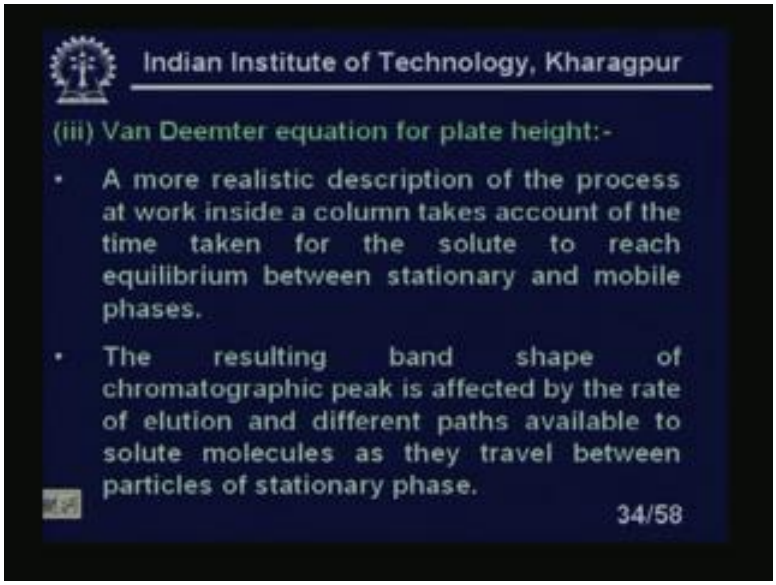

Now, one more equations we will introduce is a Van Deemter equation for plate height A more realistic descriptions of the process at work inside a column takes account of the time taken for the solute to reach the equilibrium between the stationary and the mobile phases. The resulting band shape of the chromatographic peak is affected by the rate of elution and the different paths available to solute molecules as they travel between the particles of stationary phases.

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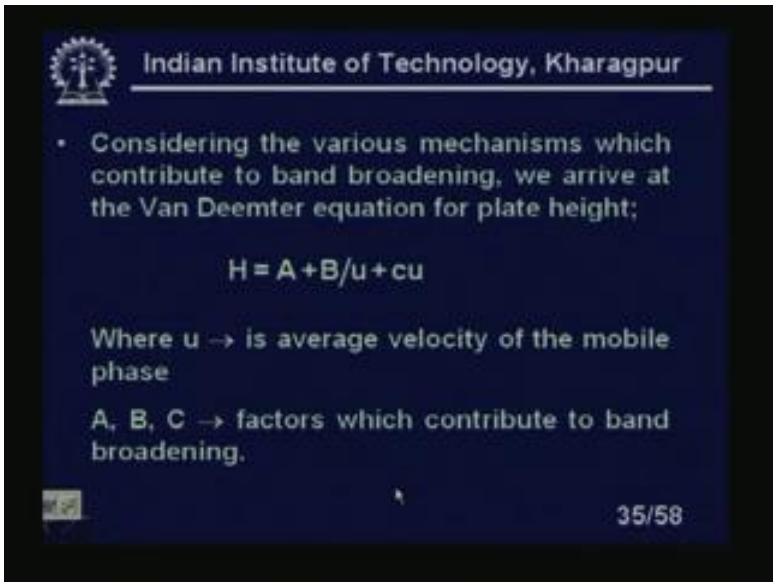

Now, considering the various mechanism which contribute to the band broadening we arrive at the Van Deemter equation for the plate height which is given by A plus B upon u plus cu. This is all the I mean dimensions of length you see. We will explain this a b c in the next where u is the average velocity of the mobile phase. A b and c factors, which contribute to the band broadening what are these a b c? Let us explain. So, these are the factor, because band broadening will control the resolutions. So, how closely the 2 elements are there. So, how close the elements are in characteristics which we can separate. So, this we have discussed here in this particular. So, a b c factors a b a b and c the factors which contribute to the band broadening.

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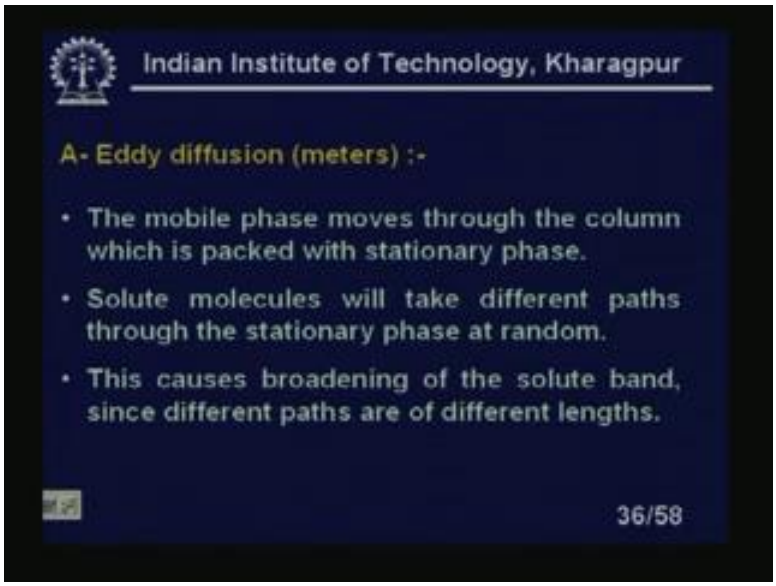

Now, A is actually what are the factors let us three factors we talk about A is the Eddy diffusion it is in meters. It is diffusion in meters. The mobile phase moves through the column, which is packed with stationary phase. Solute molecules will take different paths though the stationary phase goes at random. The causes this causes broadening of the solute band since different paths of different lengths. So, it is actually making different paths we cannot restrict a single path, because is a there is a definite width or definite diameter of the column.

So, this will make a different path. So, it will reach at a different. So, that will make the different broadening of the band. So, the length which we are talking about length in chromatography is not is actually the height if it comes straight away from the bottom to top. So, we can say that it is a x ((Refer Time: 31:03)), but if it comes suppose this way or something like this so; obviously, the length will be define. So, if there are in numeral I mean number of I mean path through which the liquid can moved through the column.

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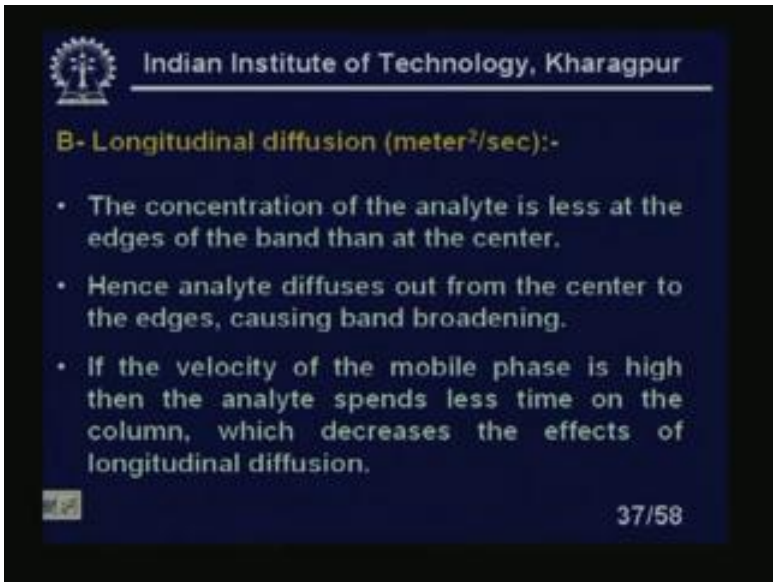

Longitudinal diffusions this again the problem we are always I mean talking about the lateral diffusions with longitudinal diffusion which is meter square per second The concentration of the analyte is less at the edges of the band than at the center at the center. So, it is less so; obviously, if it is not homogenous mixture, because it is a mixture after all it not a compound that is the entire this column that through which in which we are actually putting the all the materials which actually I mean absorbed or adsorbed and elutes the gases or liquids.

So; obviously, it is very difficult to make the concentration we are assuming that the concentration of the analyte is less at the edges and band and at the center hence analyte diffuses out from the center to the edges causing band broadening. So, it is going out to the, so there is band broadening is there. If the velocity of the mobile phase is high then the analyte spends less time on the column which decreases the effects of the longitudinal diffusions is high then analyte spends less time on the column which decreases the effects of the longitudinal distribution diffusion.

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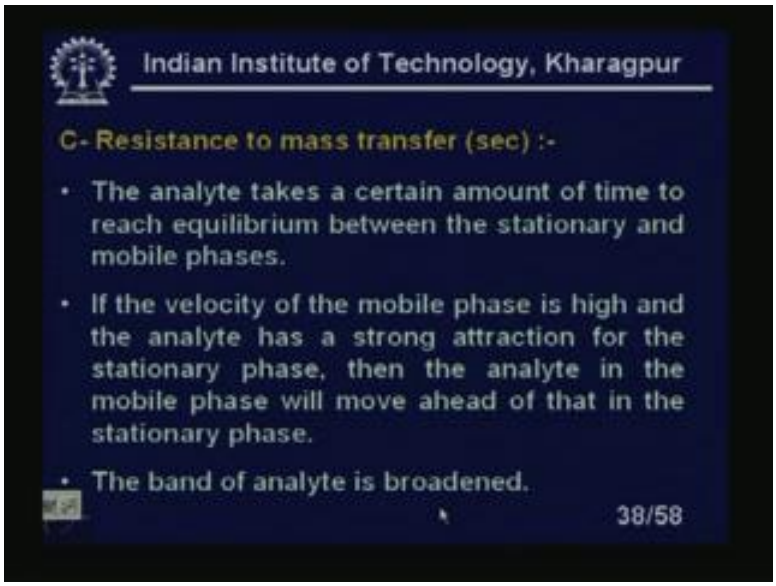

Now, resistance to mass transfer which is I mean component C. The analyte takes a certain amount of time to reach equilibrium between the stationary and the mobile phases. It is takes certain amount of time to reach equilibrium between the stationary and mobile phase. If the velocity of the mobile phase is high the analyte has a strong attraction for the stationary phase, then the analyte in the mobile phase will move ahead of that in the stationary phase. The band of analyte is broadened now again.

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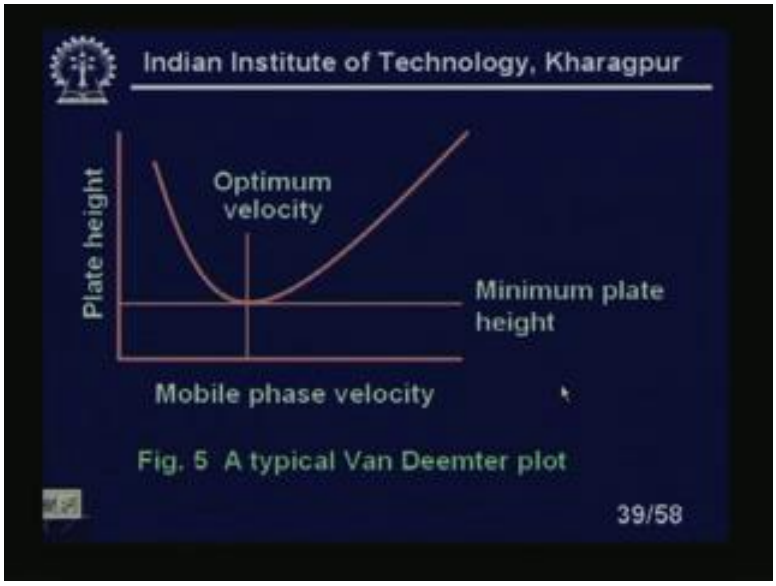

So, you see here, so a typical Van Deemter plot. So, minimum plate height is like this one. So, we are seeing assuming the plate height is like this 1 optimum velocity will give you this 1 right which will make them minimum plate height.

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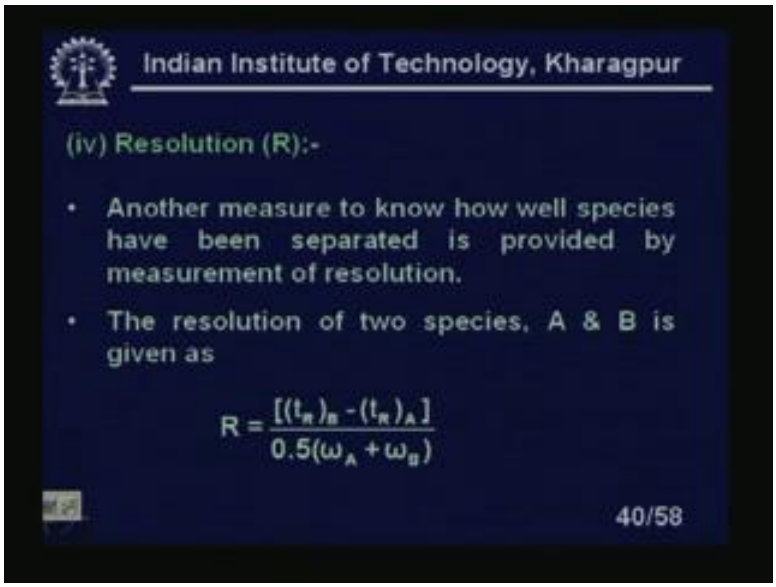

Resolution is another important factors another measure to know how well the species have been separated is provided by the measurement of resolution. The resolution of the 2 species A and B is given by R equal to tR B retention time of the component B minus retention type of the component A upon point five plus omega A plus omega B.

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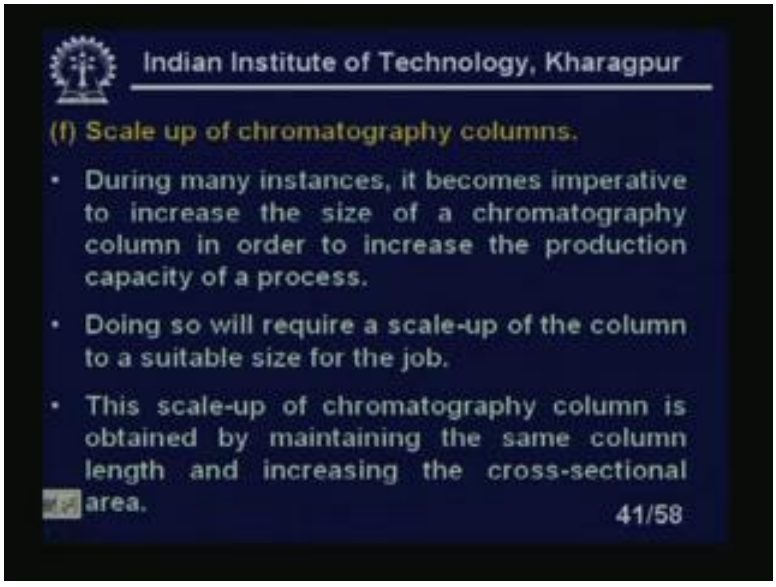

Scale up of chromatographic columns, because depending on the different dimensions of the volume of the liquid you have to scale up. Now, there is some typical formula how we can scale up the, because I have the existing column laboratory column I mean laboratory model if I want to use it industrially with a large analytical I mean a quantity and volume flow of the gases. So; obviously, I have to increase the dimensions, so I have to think of dimension; however, I choose this diameter of the diameter and the length of the column that is to be discussed now.

During many instances it becomes imperative to increase the size of the chromatography column in order to increase the production capacity of a process doing, so will require a scale-up of the column to suitable size for the job. The scale-up of chromatography column is obtained by maintaining the same column length and increasing the cross-sectional area. So, we are changing the cross sectional area increasing the cross sectional area, so the length remains same. So, I am changing the cross section area by a formula let us look at.

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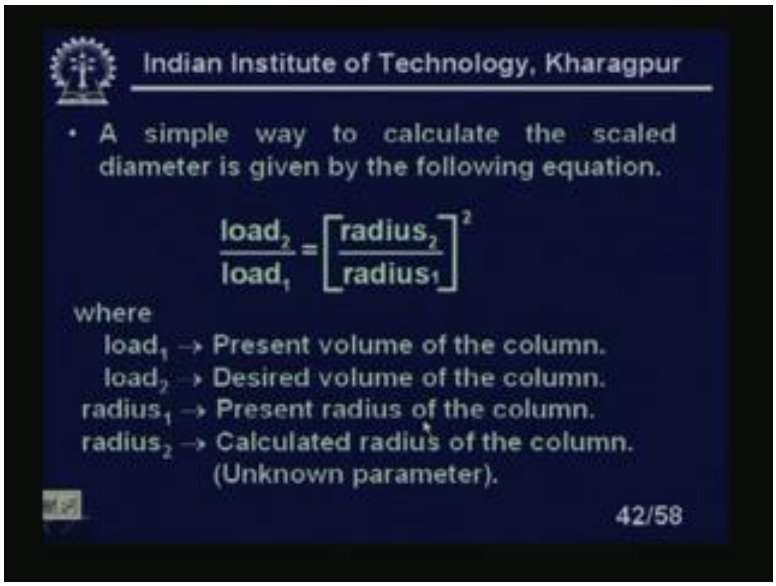

A simple way to calculate the scaled diameter is given by the following equations. We have a load 2 upon load 1 equal to radius 2 upon radius 1 to the power square. What is load 1 load 2, let us look at where load 1 is the present volume of the column it is the present volume of the column. And load 2 is the desired volume of the column and if it is the present volume of the column and desired volume of the column radius1 is a ((Refer

Time: 35:41)) present radius of the column. Obviously 3 is in the equation 4 are unknown 4 are four parameters 3 are known so; obviously, radius 2 can be calculated is the calculated radius of the column. So, we can calculate an accordingly I can change very simple formula though I mean with some limitations obviously.

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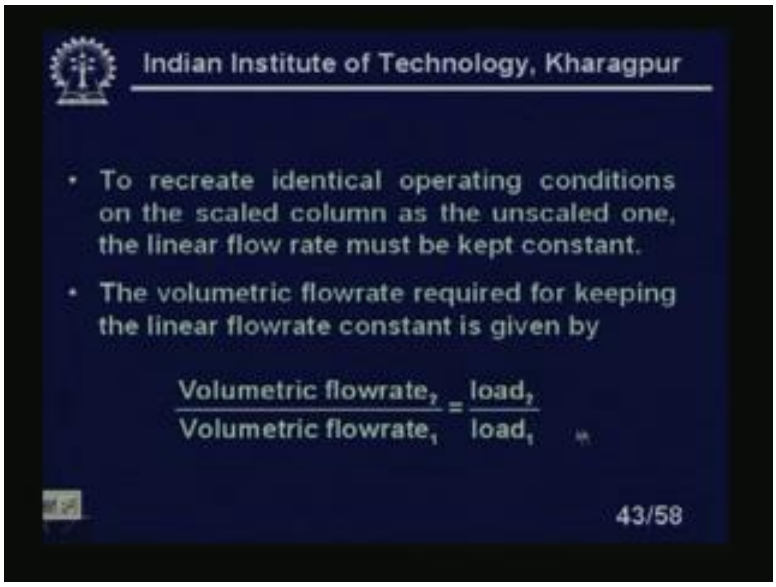

To recreate identical operating conditions on the scaled column as the unscaled 1 the linear flow rate must be kept constant. The flow rate must kept constant we should have flow meters to know that because the feed mobile phase is moving through this 1. The volumetric flow rate required for keeping the linear flow rate constant is given by volumetric flow rate 2 upon volumetric flow rate 1 equal to load 2 by load 1. So; obviously, we know that the here in this case we have volumetric load 2 from these we can calculate the parameter.

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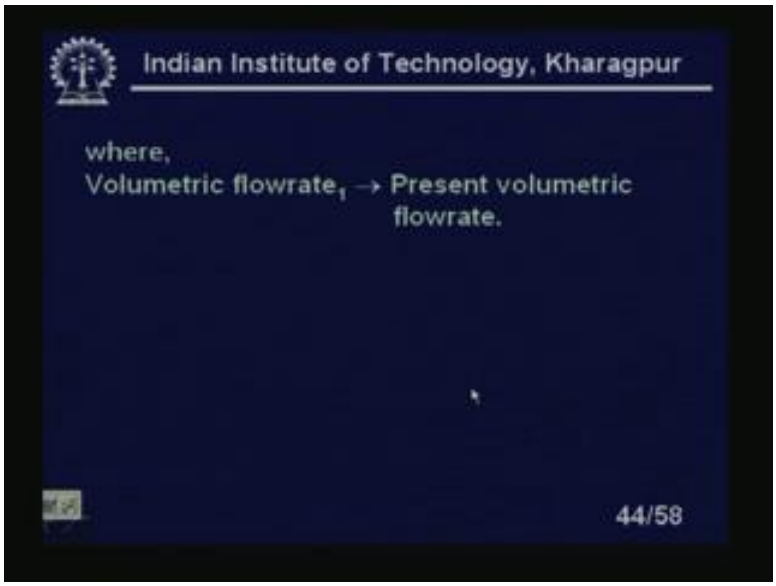

What is the where volumetric flow rate; one is the present volumetric flow rate that is for the laboratory models and volumetric flow rate calculate a volumetric flow rate that is we can calculate.

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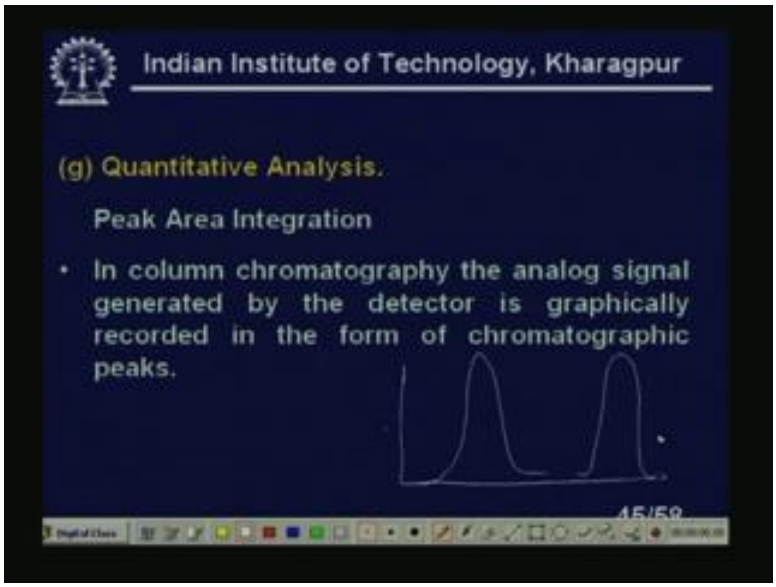

Now, quantitative analysis this is also very important in the case of gas chromatography. Let us look at that peak area integration what is that peak area integration. let us look at… In column chromatography the analog signal generated by the detector is graphically recorded in the form of chromatographic peaks which is called the

chromatogram that we have seen it looks like this. So, it is coming like this one, you have seen it is coming like peak like this 1, peak like this 1, so this is called the chromatograph.

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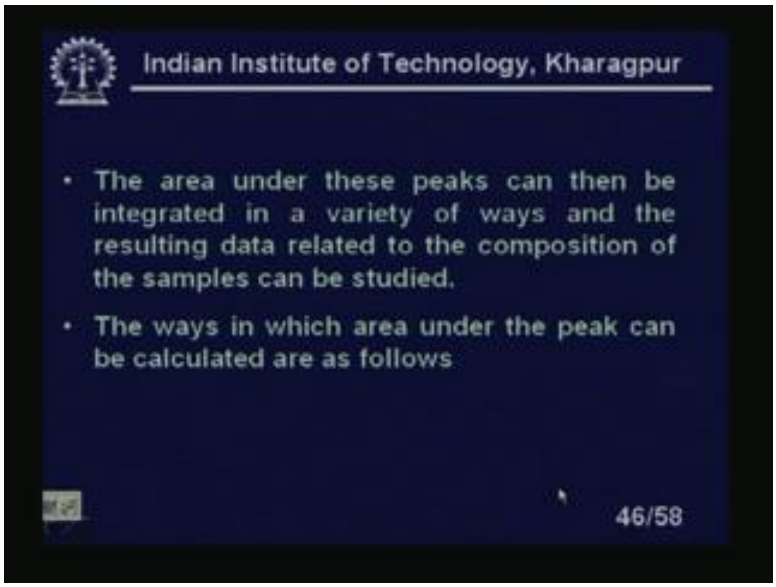

The area under these peaks can then be integrated in a variety of ways. So, we are integrating that areas and the resulting data related to the composition of the samples can be studied this is most important thing. Now, we come to the detector side once you have I mean with some detectors we can collect the analog signals electrical signals now, it will be plotted. So, the area under these peaks can then be integrated in a variety of ways and the resulting data related to the composition of the samples can be studied. Because the area will give you the relative contents of the different compositions the ways in which the area under the peak can be calculated are as follows. There are various methods by which areas can be calculated we will discuss 1 by 1.

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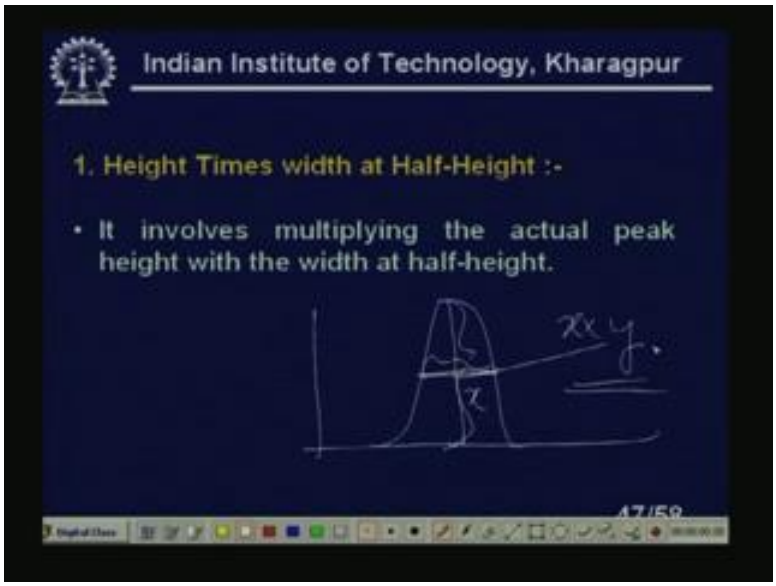

Height times width at the half height. Height multiply by this basically height multiplied by the width at the half height what is that? It involves the multiplying the actual peak height with the width of the width at the half height It looks like this; that means, I will show you actual peak height suppose we have a peak height is this. This is multiplied by the half height width of the half height we multiply by this suppose this is x this is y. So, x in to y is the height time's width. Height times width at the half height will be will be measured of the integration.

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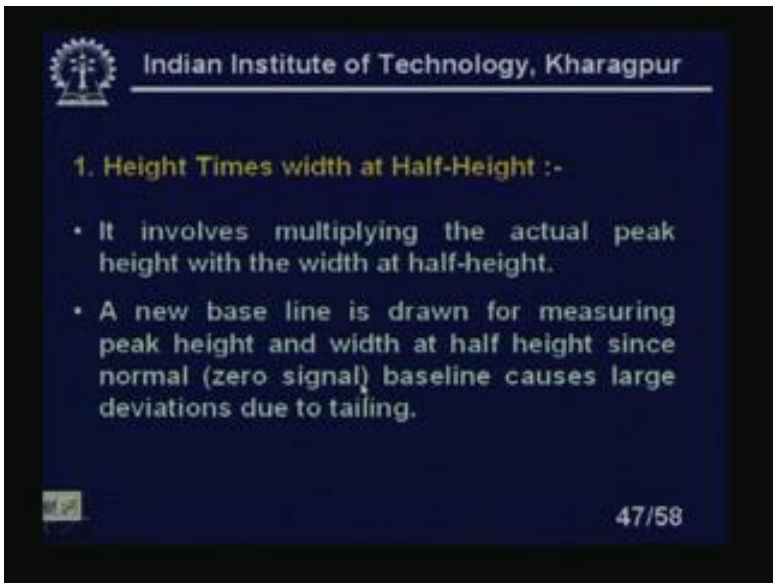

A new baseline is drawn for measuring peak height, and the width at the half height since the normal zero signal baseline causes large deviation due to tailing.

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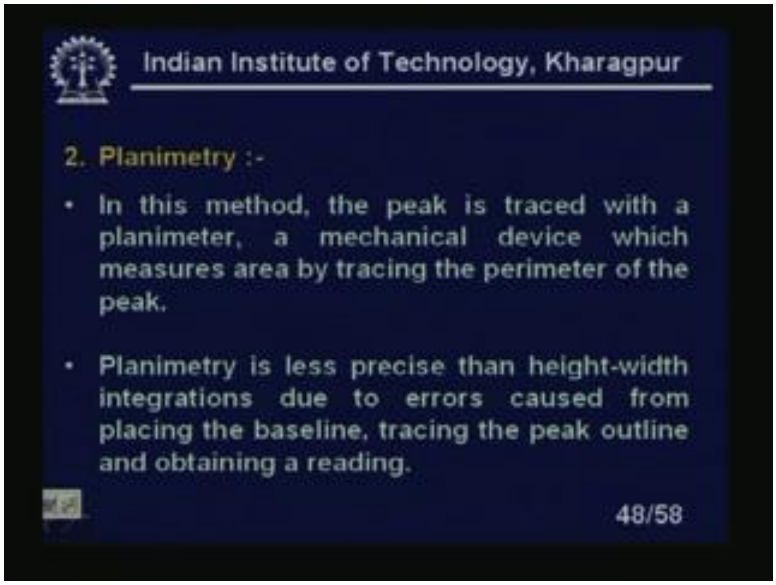

Planimetry; this is another method of integration. In this method the peak is traced with a planimeter a mechanical device which measures area by tracing the perimeter of the peak. In this method the peak is traced with a planimeter a mechanical device, which measures the area by tracing the perimeter of the peak. Planimetry is less precise than the height width integrations due to errors caused from the placing the baseline tracing the peak outline and obtaining a reading.

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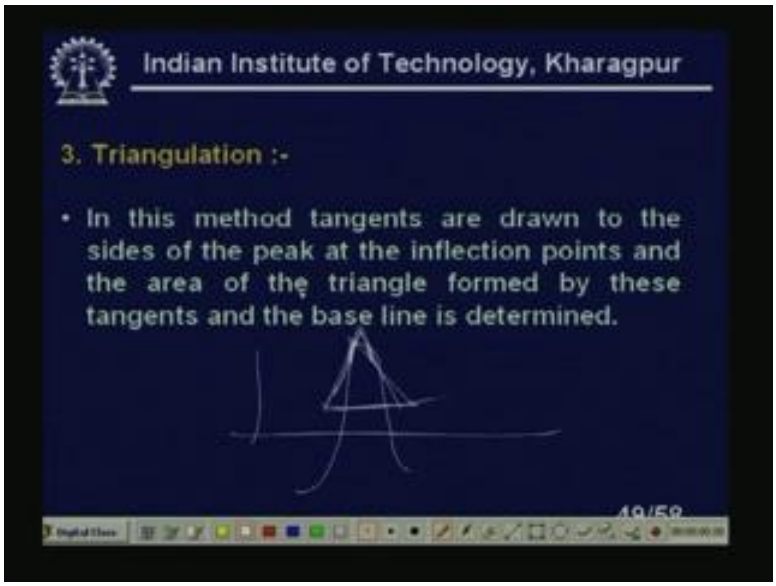

Triangulation; in this method tangents are drawn to the sides of the peak at the inflection points and area of the triangle formed by these tangents and the baseline is determined. So, it is looks like this I have a peak I have a peak here I will show here. So, I am drawing a tangent here tangent here. So, we will give you the area of the triangle will form this and the baseline is determined right. So, tangents we have drawn one at the inflection point inflection point like this one inflection points rather. So, we are trying to tangents where it meets. So, we are making one triangle. So, that area of the triangle formed by this tangents baseline is determined.

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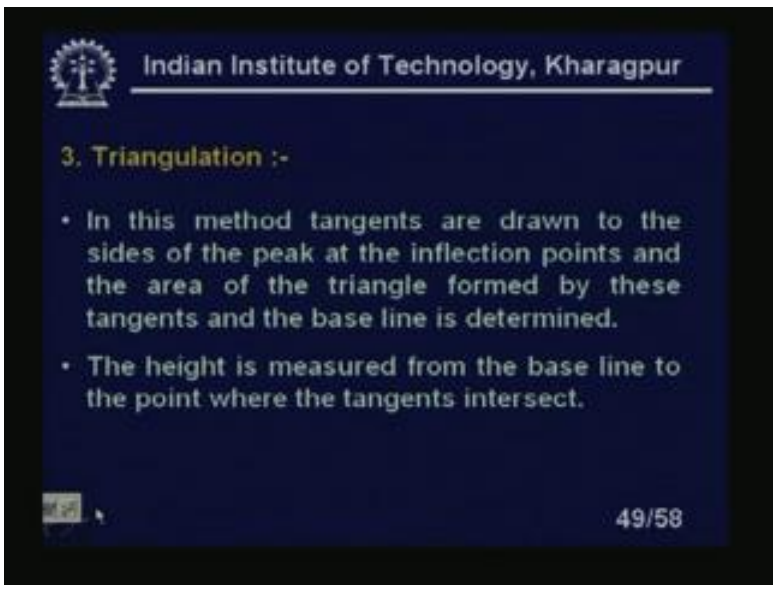

The height is measured from the baseline to point where the tangents intersect right.

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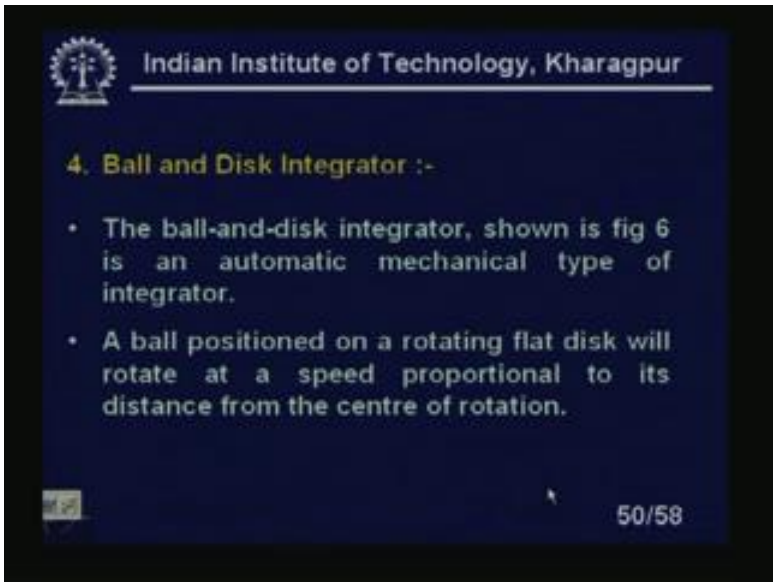

Ball and disk integrators; this is very mechanical integrator is useful the ball and disk integrator shown in figure 6, in the next figure in an automatic mechanical type of integrator. A ball positioned on a rotating flat disk will rotate at a speed proportional to the distance from the centre of rotation.

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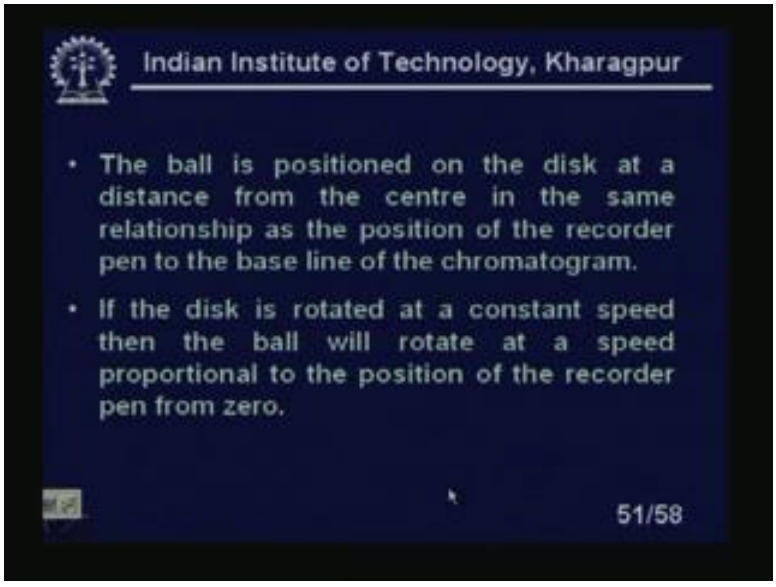

The ball is positioned on the disk at a distance from the centre in the same relationship as the position of the recorder pen to the baseline of the chromatogram. If the disk is rotated

at the constant speed then the ball will rotate at a speed proportional to the position of the recorder pen from zero right.

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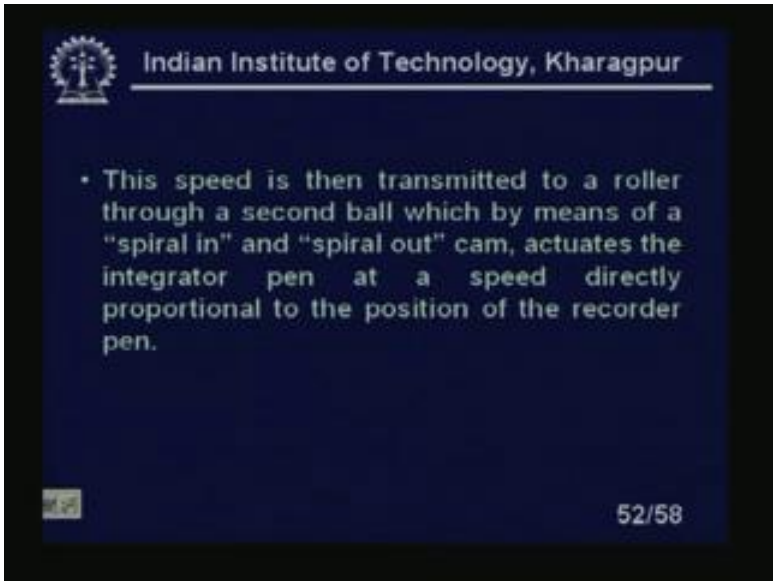

The speed is then transmitted to a roller through a second ball, which by means of a spiral in and spiral out cam actuates the integrator pen at a speed directly proportional the position of the recorder pen clear? We use more clear once we show the diagram

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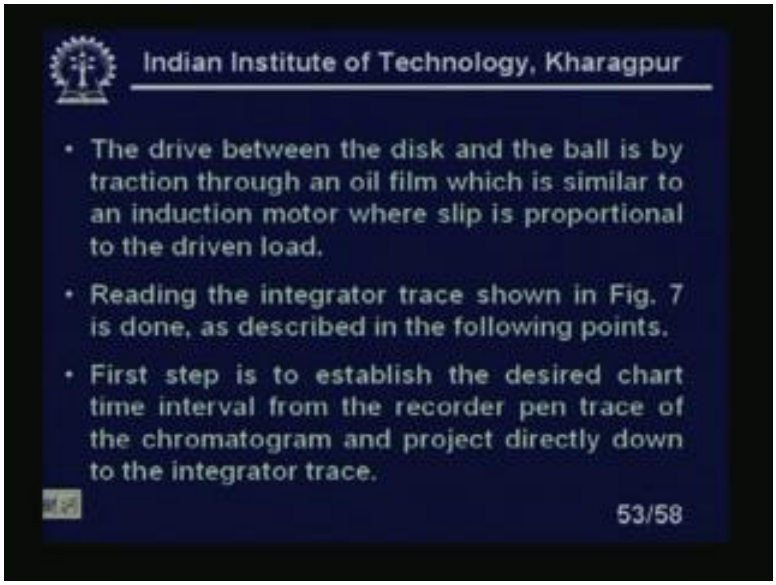

The drive between the disk and the ball is by traction through an oil film, which is similar to an induction motor where slip is proportional to the driven load. Reading the

integrator trace shown in figure seven is done as described in the following points. First step is to establish the desired chart time interval from the recorder pen trace of the chromatogram, and the project directly down to the integrator trace.

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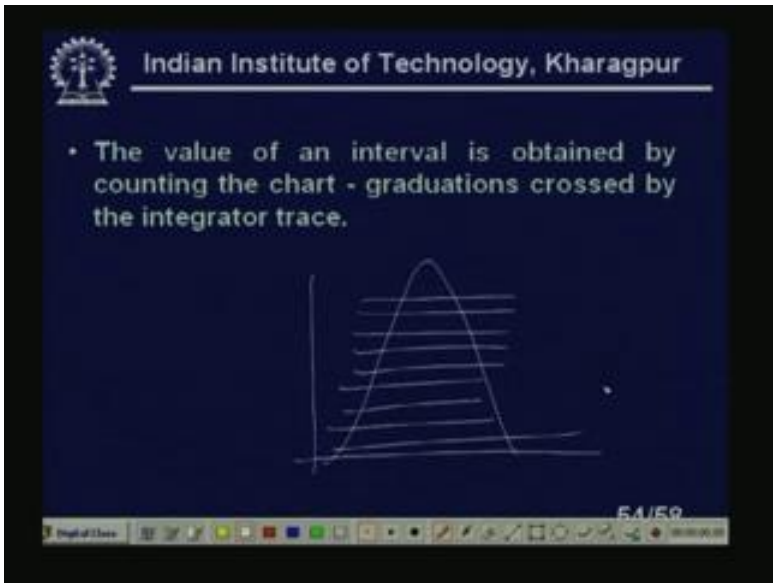

The value of an interval is obtained by counting the chart graduations crossed by the integrator trace. So, it will be a like this one we will see it will be like this one. So, it will be graduation crossed by integrator trace. So, interval is obtained by counting the chart like this one.

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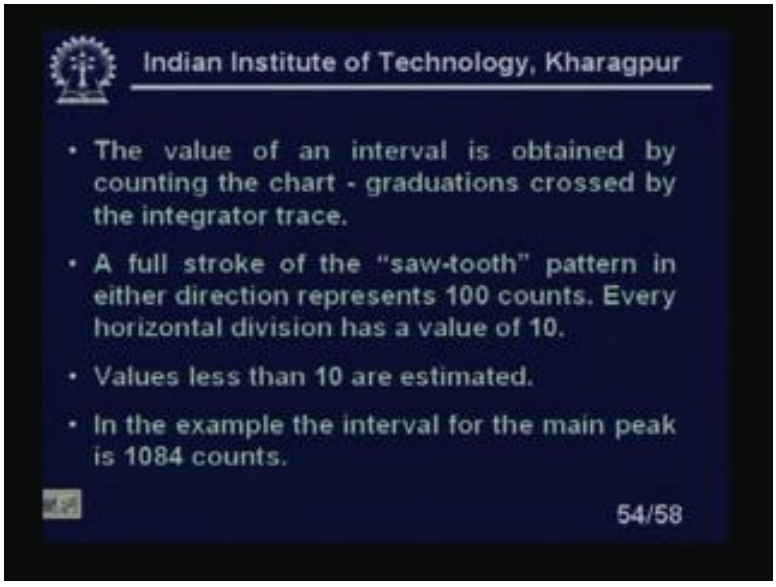

A full stroke of the saw-tooth pattern in the either direction represents hundred counts and every horizontal division has a value of 10. So, full stroke of the saw tooth pattern in either direction represents the 100 counts. So, may be the horizontal division has a value of 10. Values less than 10 are estimated; that means, only 10 is minimum resolutions we can get. So if in between comes anything that needs to be estimated in the example the interval for the main peak is 1084 counts.

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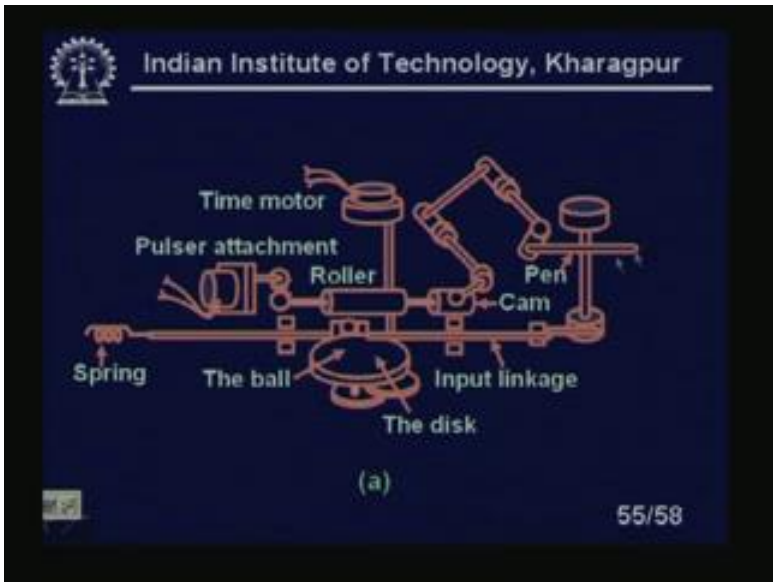

You see this is the disk and we are this pen is going to a recorder that I am not showing. Spring ((Refer Time: 43:46)) a valve and the disk this input linkage is here. This is a pulsar, this is a roller and this is the time motors. So, it is going to the pen in the next graph we will show.

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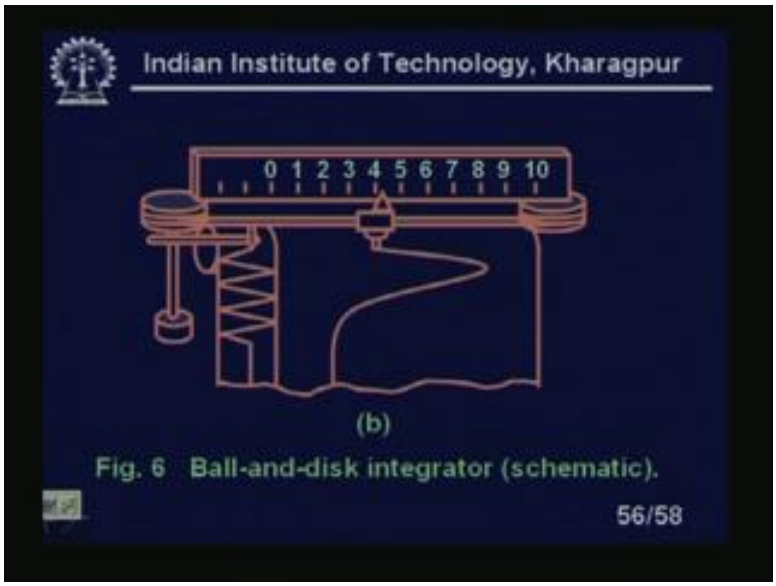

You see here this pen goes here which is recording on a chart right. So, ball and disk integrated symmetric. So, this is about the about our, the gas chromatography basic recorder. Let us look at the plot now.

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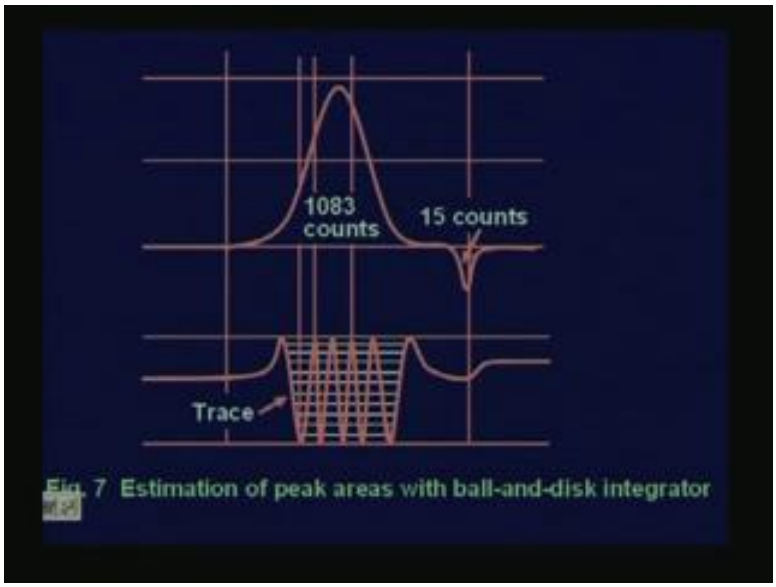

You see there are total 1084 counts 10218 1083. So, the estimation of the peak areas with ball and disk integrators, so we have making ten divisions here. So, each has a 10 counts here. So, we will get 1084 counts here. So, with this we can estimate the peak areas of the gas chromatography peak areas of the systems. Now, gas chromatography as

you know it is extensively used not only for analytical. Because as the beginning we say that the initially it is not only for the making the analysis of the gas mixtures which is coming out in this industrial process.

But also to know if suppose a some other companies making some particular mixture of gases what is the components of the gases. It is very vague though it is very difficult to tell. Now, once you separated the gas components; obviously, you can make the analysis you can say will the other method to know what is the component? But the basic separation is the most important thing where you have to wait one by one it will come. So, basically I can say that the this chromatography is something like that if I pass the gas mixtures through a column whether it is open channel open tubular form or a flat bed reactor or flat bed column.

So, it will absorbs or adsorbs for sometime it will retained the gast for sometime then it will slowly all the gas will not come out immediately or not it will come after certain amount of time. It will come one by one from the packed bed column. So, these actually makes our gas chromatography flexible right. Now, in these case one thing we should know that this is the just a tremendous not only in the chemistries. It has a tremendous use in the industrial operations in many situations you know we have to trace the presence of gases of the gas mixtures and complete the process.

So, that type of situation we can collect a sample gas and inject through this one through the feed, because we have seen that we have a stationary phase and the mobile phase. So, that, so this will go through the feed as a feed through the systems or the carrier through the systems. So, slowly it will separate and it will tell the composition. Now, different I mean compositions I mean determination of the percentage and all this that is that is coming next that is not I mean we are coming lateral. But the basic difference I mean basic finding that is presence of particular gas or composition detecting the particular gas or the components of the gas is most important.

Later on you can use the spectroscopy and all those things to find what are the elements with the gases are there mixture of the gases the first is separation. So, this is only method of the mankind. So, far developed this is a very excellent method as. So, far you know to find the making a separations. Before that as I told you this is very unreliable method this is a quite reliable method, and is not that I mean sensitive to temperatures.

And all though in some cases, we have to maintain a particular temperature, while you are calibrating, but most of the cases the, I mean temperature I mean maintaining the temperature is not very crucial. That is the reason we were saying it is not necessary it is not very harsh. It is not very severe environment while we are... So, many cases suppose there is some gasses, suppose it is I mean getting decomposed or in some other particular temperature that type of cases also we can we can use the separation.

In other method, you can have high temperatures and all this lower low temperature those things are not necessary in the case of gas chromatography. So, with this I come to the end of the gas chromatography 1 and the lesson 36 we will continue with this particular. That mean the after chromatography which is a chromatography 2. Preview of next lecture; welcome to the lesson 36 of industrial instrumentation. This lesson is a continuation of the lesson 36 where we have discussed the chromatography 1. In this lesson, we will discuss chromatography 2. So, let us look at the contents. It is a chromatography 2 as I told you.

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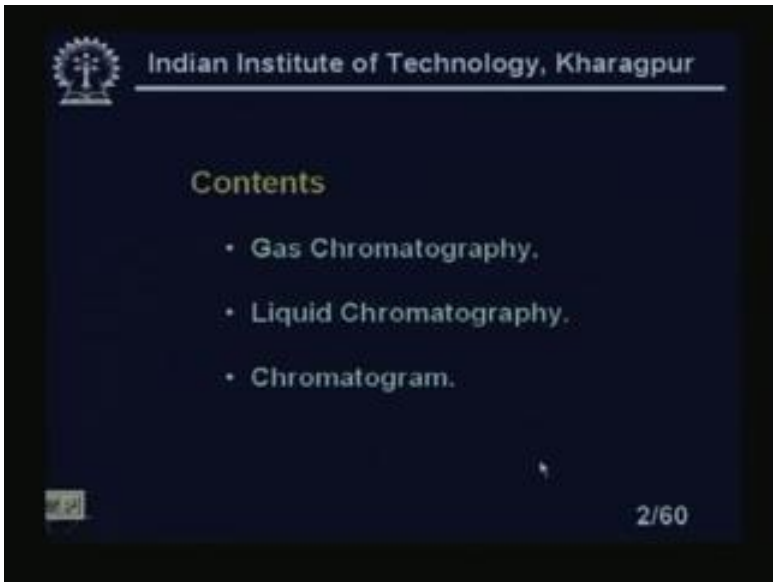

The contents are gas chromatography, liquid chromatography, chromatogram. We will see that in this particular lessons. So, we discuss the actual the how that chromatogram should like and what are the, if the multiple picks comes and what are the problems what exactly I mean the shape of the chromatogram should be. I mean that depends actual the finding the concentrations of the different components of the gas and liquids precisely.

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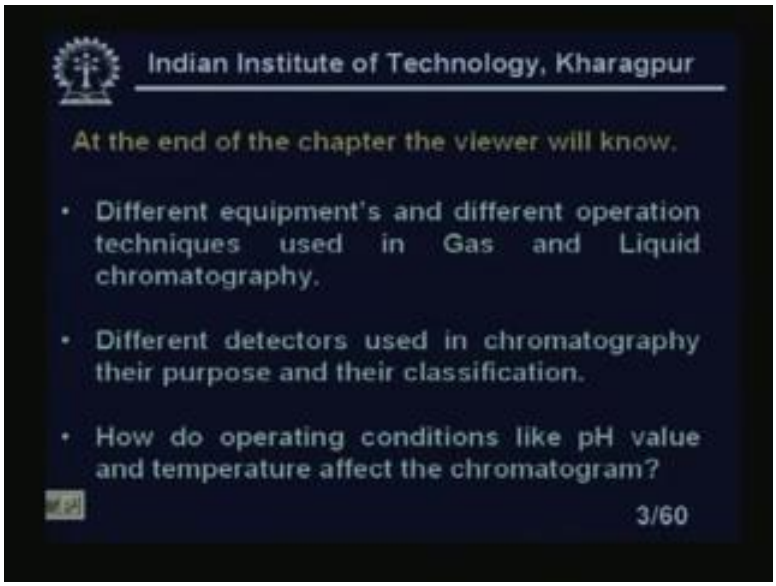

At the end of the lesson or end of the chapter the viewer will know different equipment's, and different operation techniques used in gas and liquid chromatography. Different detectors used in the chromatography and their purpose and their classification. We will find there are different types of detectors available. There are thermal conductivity detector there are flame ionization detector there are electron caption detect capture detector.

So, there are different I mean the principles are I mean somewhat different in though the thermal conductivity detectors are the oldest one, and most widely used detectors in the gas chromatography. But we will discuss the other detectors also where you will find that still making the derivatives of the particular element of the mixtures. So, that to find the concentration accurately, how do operating conditions like pH value temperature affect the chromatogram? So, we will find these things also the pH value though I mean its I mean not very importance in the case of gas chromatography. This is very important in the case of liquid chromatography.

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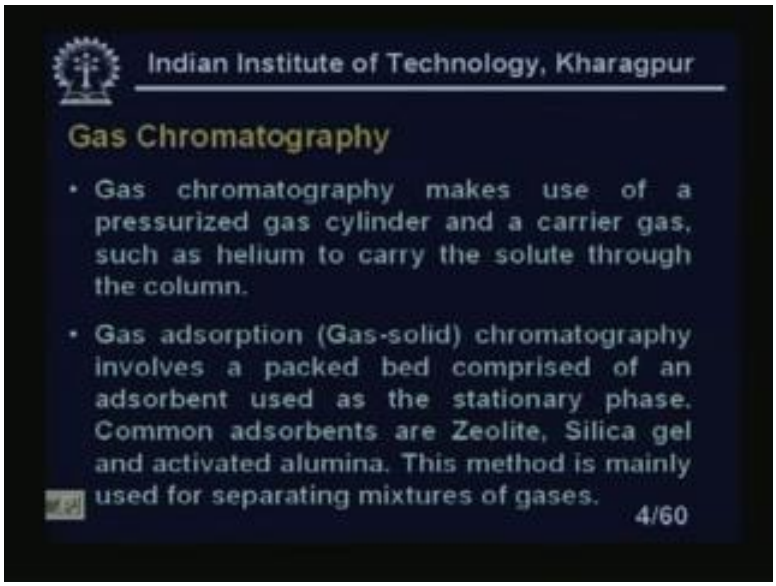

Gas chromatography; already that we have discussed that gas chromatography makes the use of a pressurized gas cylinder and a carrier gas such as helium which is inert nature to carry the solute through the column. Because we will find we have discussed these things that there is a column and through column these the gas would flow right and can and there is a packing material inside the column. So which will adsorb or absorb and elute it after sometime one by one. Gas adsorptions that is gas solid chromatography involves a packed bed comprised of an adsorbent used as the stationary phase. Common adsorbents are zeolites silica gel and activated alumina these are the most common sort of I mean adsorbents are used in a chromatography. These method is mainly used for separating mixture of gases we will find this is basically used for mixture of gases, right.

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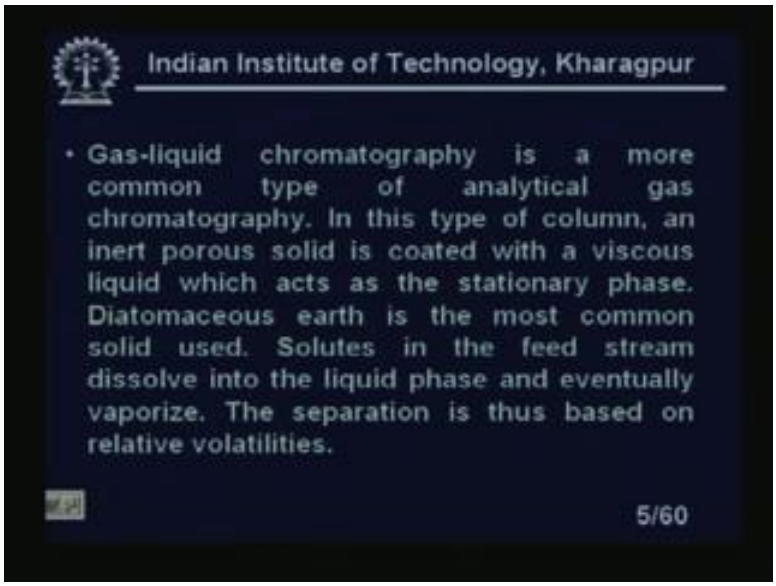

Gas liquid chromatography is a more common type of analytical gas chromatography. In this type of column an inert porous solid is coated with a viscous liquid which acts as a stationary phase. And diatomaceous earth is the most common solid used and solutes in the feed stream dissolve into the liquid phase and eventually vaporize.

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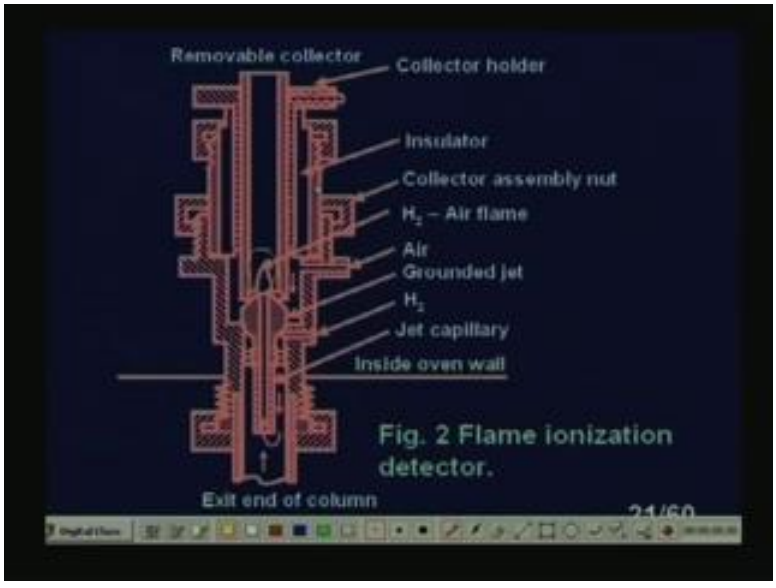

Current flow across the external resistor is sensed with a voltage drop and it is amplified and displayed on a recorder. Now, hydrocarbon groups are introduced into a flame and a complex process takes place in which positively charged carbons spaces and electrons

are formed. Now, the current is greatly increased. Now, this frame ionization detector response only to the substance that produce charged ions when bond in hydrogen air flame that is most important thing. In an organic compound the response is proportional to the number of oxidizable carbon atoms. So, this is basic principles of the flame ionization detector.

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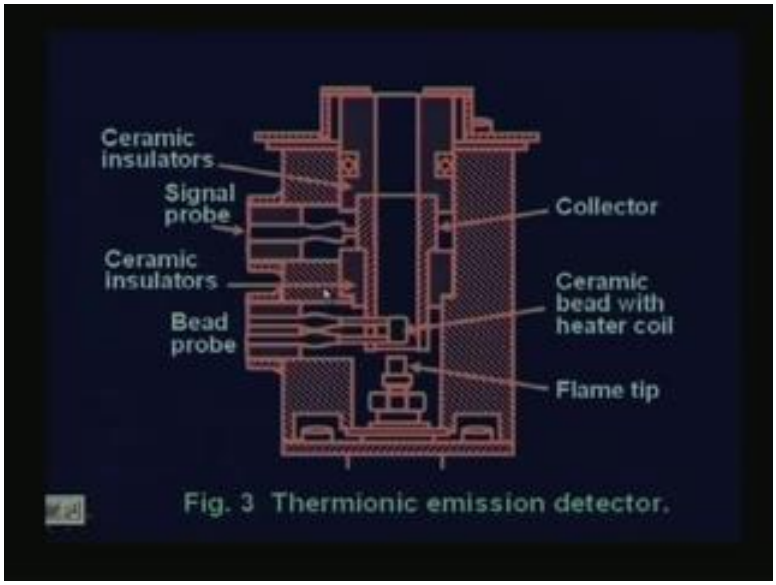

Now, if I look at the thermionic emission detector you see this is thermionic emission detector it is principle is something different. It employed a fuel poor hydrogen plasma. These low temperature source suppresses the normal flame ionization response of a compounds not containing hydrogen and phosphorous although the response to carbon is not entirely eliminated. A non-volatile rubidium silicate bead here you see here non-volatile rubidium silicate bead centered about 1.2 centimeter above the plasma jet is electrically heated by a variable current supply to between 600 to 800 degree centigrade.

These arrangement permits the fine adjustments of the beads temperature and independent of the plasma as a source of thermal energy. With a very small hydrogen flow the detector responds to both nitrogen and phosphorous compounds enlarging the plasma. The changing polarity between the plasma and the detector responds only to the phosphorous compounds. So, that I can make it nitrogen I can make it sensitive to the phosphorous compounds also. Now, compared to the compared with the flame ionization

detector the thermal emission detector, thermionic emission detector is our 50 times more sensitive for nitrogen and about 500 times more sensitive for phosphorous.

So, any traces of phosphorous any traces of nitrogens can be better detectors in the case of. I mean thermionic emission detector compared to the flame ionization detectors though flame ionization is also comparatively relatively new compare to the thermal conductivity detector. The minimum detectable limit is around point zero six pg I mean per second for nitrogen. So, this is the thing you can see here that there is a flame tip which is one point around one point per 2 second above the plasma jet. So, this is our, I mean thermionic emission detector. Last, we will discuss the electron capture detector.

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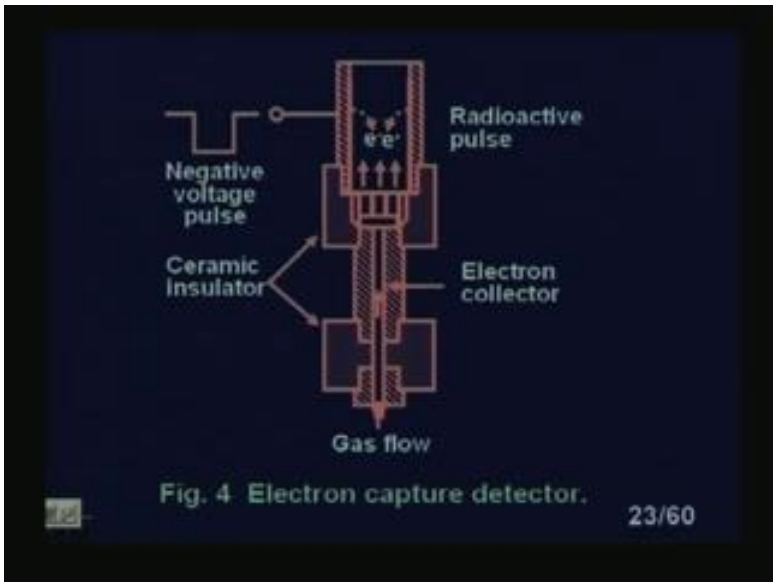

You see this is a electron capture detector. Now, electron capture detector has two electrodes within the, with the column affluent passing between one of the electrodes is treated with radio isotopes. That emits high energy electrons as it decays and this emitted electrons produce. I mean amounts of large amount of low energy thermal secondary electrons in the gas chromatography carrier gas all of which are collected by other positively polarized electrode either of the positive polarized electrode. Molecules that have an affinity for thermal electrons captured electrons as they pass between the electrodes and deduce the steady state current thus providing a electrical reproductions. Or the gas chromatography peak of the two general designs; the plane parallel and the

concentric and the later design is preferred since it is easier to construct a small volume cell in this form.

Now, particular radioactive sources which are used that the titanium adsorbed in the titanium or scandium and nickel 63 as a foil or platter on the interior of the cathode chamber. So, your chromatograph will change; obviously if the chromatogram change efficiency selectivity everything will change. So, this is to be, so whatever the desired value of the chromatogram that is to be maintained right.

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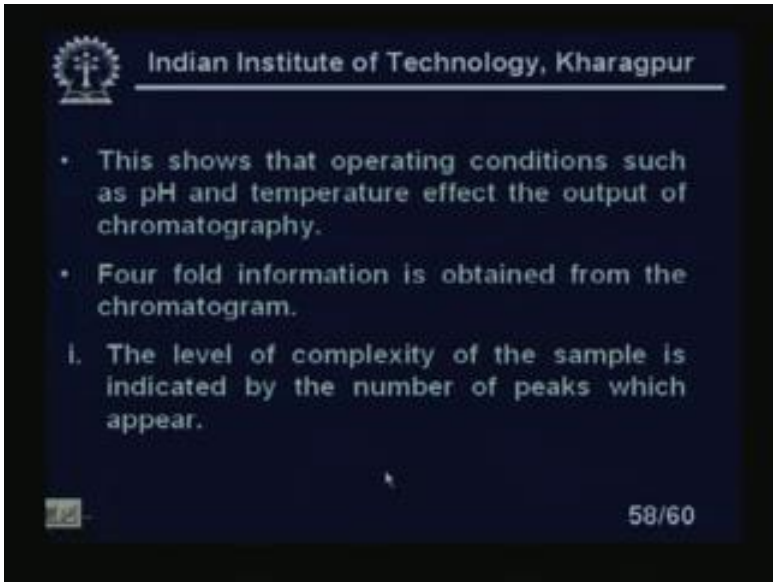

Four fold information is obtained from the chromatogram. What are the different I mean components I am getting from the chromatogram, what are the different I mean informations I am getting from this chromatogram? So, you know already we have discussed let us downright. What one by one, the level of complexity of the sample is indicated by the number of peaks, how many what is the complex mixtures I mean that we can detect from this one? So, level of complexity of the sample is indicated by the number of peaks, which appear.

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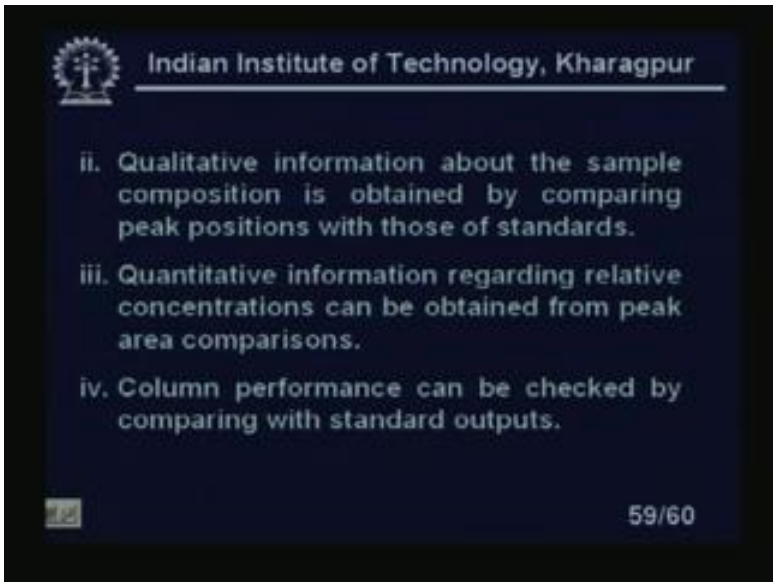

Qualitative information about the sample of composition is obtained by comparing peak positions with those of the standards. Qualitative information about the sample composition is obtained by comparing the peak positions with those of standards. Quantitative information regarding the relative concentrations this is most important we are measuring the relative, because of the sample. If the we are absolutes measuring we are making the relative comparison relative concentrations actually we are measuring the gas chromatography. Because otherwise we cannot measure it, because we need the injections at the, it is only ten micro liter that is the best other samples betrays the detection. Quantitative information regarding the relative concentration can be obtained from the peak and area comparisons. Column performance can be checked by comparing with the standard outputs.

This column performance also, because if I have if I knew if I knew the particular suppose if I want to calibrate the column performance I know the particular gas with which actually we are injecting. So, the, that time when an unknown gas is coming, if I want to calibrate the how the column actually working? So, the column efficiency can also be known I mean I mean can be known by looking at the output. So, these are the 4r I mean features I mean which will get from the gas chromatography. The most important thing is the relative I mean first of all the, what are the different components of the gas present in the mixture? Second thing is the relative this is the most prime most importance. Second thing is the relative concentrations of the gas or liquid or whatever it

may be in the mixture. So, with this I come to the end of the lesson thirty-six on industrial instrumentation.