

Modern Instrumental Methods of Analysis
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Lecture No. # 41

Gas Chromatography-1 Instrumentation

Welcome to our next class on a gas chromatography, we discussed a few aspects of gas chromatography in the earlier class. What I had told you was that gas chromatography differs from other forms for chromatography in that the mobile phase is a gas and the components are separated as vapors, the separation is accomplished by partitioning the sample between the stationary phase and the mobile phase. And the stationary phase is usually a liquid coated onto a solid which is filled in a column and the sample containing the solutes is usually injected into a heated block, where it immediately vaporizes the sample that is the analyte and then it is swept away by the carrier gas in to the column inlet.



The solutes are absorbed by the stationary phase and there will be equilibration process between the stationary phase and the mobile phase and after the separation the components will emerge out one by one into different zones. So, when they come out of the column they meet the registrar; that means, they enter the detectors and register a series of signals resulting from the concentration changes or on the mass rate change, based on the mass rate change and the rates of illusion on the recorder is usually a plot of time versus the composition of the carrier gas stream.

So, the appearance time, height, width, area of all these peaks can be measured to yield the quantitative data use of longer of columns and higher velocity carrier gas permits, the vast separation of the components within a matter of few minutes. So, higher working temperatures also permits the possibility of converting into a volatile component making gas chromatography one of the most versatile techniques for the separation of the components.

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Gas chromatography is composed of six parts:

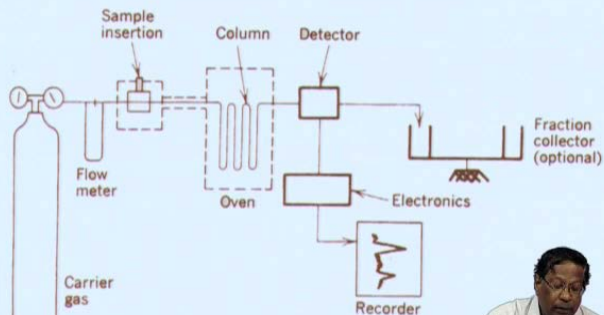
- Supply of carrier gas in a high pressure cylinder with attendant pressure regulators and flow meters
- Sample injection system
- The separation column
- Detector
- Recorder
- Separate thermostated compartments for columns and detectors





So, what are the components of a gas chromatograph? You can see here, on the slide that it consists of about six parts, six components; one component is supply of carrier gas in a high pressure cylinder with attendant pressure, regulator flow meter, needle, valves etcetera and the second component important; component is sample injection system through which the samples are injected into the column and third and the most important is the separation column that we can say is the heart of the chromatograph and the fourth one is the detector and recorder and the last one is the compartments for columns and detectors.

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SCHEMATIC OF GAS CHROMATOGRAPH



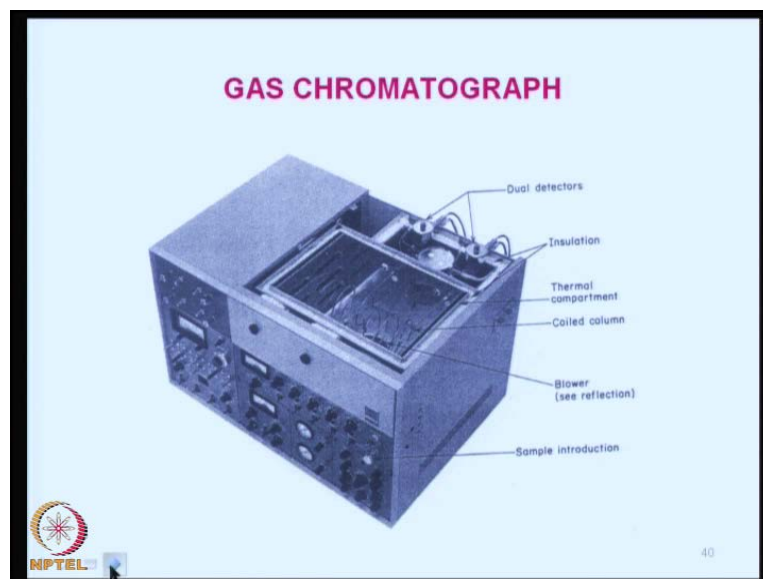
The diagram illustrates the components and flow of a gas chromatograph. It starts with a 'Carrier gas' cylinder on the left, connected to a 'Flow meter'. The gas then passes through a 'Sample insertion' point into a 'Column' housed within an 'Oven'. The output from the column goes to a 'Detector', which is connected to 'Electronics' and a 'Recorder' (showing a chromatogram). An optional 'Fraction collector' is also shown at the end of the line.



Now, what we will do? Are we will take a look at each of these components in a slightly detailed fashion? Now, you can see the schematic of the gas chromatograph like this **this** is the first part that is the carrier gas cylinder through which the gas is allowed to pass through a flow meter and then it enters a sample insertion area that is the second component and as the sample comes here it goes into takes the sample as a plug of vapor and then into the column separation column and then you need a detector when it emerges out of the column followed by a recorder and then a fraction collector if you are interested in collecting the sample.

So, what is important to notice in these things is, the sample injection, column sample, injector and the column and the detector and all these things are housed in thermostated compartments; that mean, they are all maintained at particular temperature usually high enough to keep the components into the gaseous phase or vapor phase.

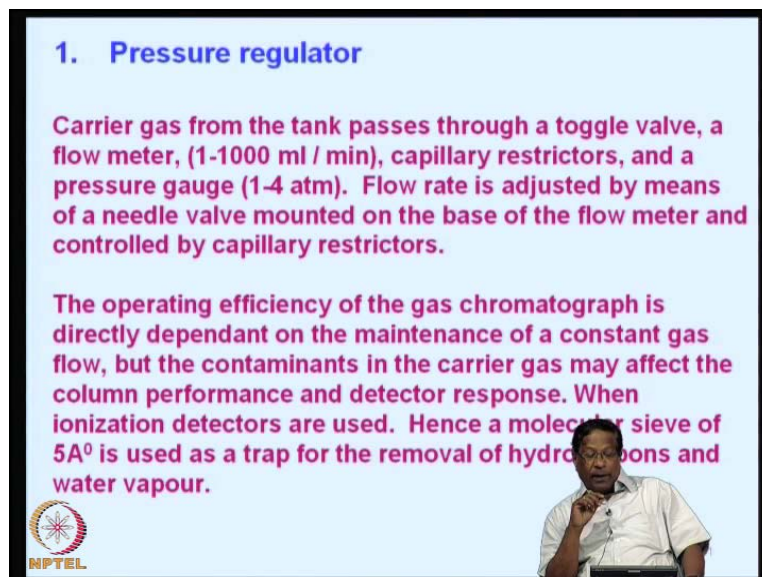
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So, now we will take look at the components in a more detailed fashion. This is usually a typical this is how a typical gas chromatograph looks you what you will be seeing is actual a big nice box with several with several controls at the back and then you will see most of the there are boxes inside the box which represent thermostated compartments and here you can see the columns. So, the detectors are placed outside the column as they emerged out of the system and then insulation is a very important component then thermal compartments are there and then columns are there, a blower is there and then

sample introduction system is here. So, the sample will be injected through this septum using a syringe. Now, we will take a look at the each component in detail..


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1. Pressure regulator

Carrier gas from the tank passes through a toggle valve, a flow meter, (1-1000 ml / min), capillary restrictors, and a pressure gauge (1-4 atm). Flow rate is adjusted by means of a needle valve mounted on the base of the flow meter and controlled by capillary restrictors.

The operating efficiency of the gas chromatograph is directly dependant on the maintenance of a constant gas flow, but the contaminants in the carrier gas may affect the column performance and detector response. When ionization detectors are used. Hence a molecular sieve of 5A⁰ is used as a trap for the removal of hydrocarbons and water vapour.

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So, let us take a look at the pressure regulator that is the carrier gas system; the carrier gas from the tank passes through a toggle valve and it has a flow meter which will give you about 1 to 1000 milliliters per minute and then it will have capillary restrictors and a pressure gauge of about 1 to 4 atmospheres.

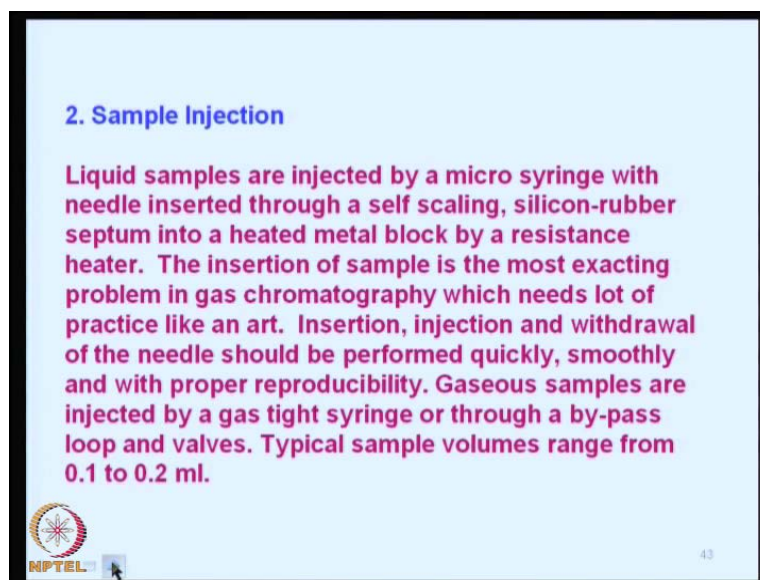
Flow rate is usually adjusted by means of a needle valve mounted on the base of the flow meter and controlled by the capillary restrictors. The operating efficiency of the gas chromatograph is directly dependent on the maintenance of the constant gas pressure; this is very important especially, because the gas pressure is the one which controls the success of a chromatographic separation. So, the contaminants in the carrier gas are also assumed importance. So, the carrier gas should be as pure as possible and then it should be of order of about 99 or 99.9 percent purity and the performance of the detector is also dependent upon the purity of the carrier gas. Therefore, a molecular of about 5 angstroms is usually used as a trap to remove the hydrocarbons and water vapor coming from the carrier gas that is very important.

So, the pressure regulator or the carrier gas is usually taken care of it in this fashion, to make sure that it does not have the moisture as in impure component and it does not have hydrocarbons as the impurities as which will ensure a good performance with respect to

the detector also. Usually, what are the carrier gases we use in chromatography? We can use any gas for that matter, but, helium seems to be the best, but, helium is costly still people use helium and nitrogen is another very common carrier gas you can use hydrogen argon etcetera.


Helium is preferred thermal conductivity detectors, because of its high thermal conductivity relative to that of most of the organic vapors that you are trying to separate in general, I have to tell you at this point that the separation from the gas chromatography is 99 percent restricted to organic compounds. So, with respect to these things organic compound helium is a best carrier gas because of its high thermal conductivity, nitrogen is also preferable it is cheaper.

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2. Sample Injection

Liquid samples are injected by a micro syringe with needle inserted through a self scaling, silicon-rubber septum into a heated metal block by a resistance heater. The insertion of sample is the most exacting problem in gas chromatography which needs lot of practice like an art. Insertion, injection and withdrawal of the needle should be performed quickly, smoothly and with proper reproducibility. Gaseous samples are injected by a gas tight syringe or through a by-pass loop and valves. Typical sample volumes range from 0.1 to 0.2 ml.

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Even large consumption of carrier gas is required then it is better to go for nitrogen. Hydrogen and other things are used as special cases that we will see as we proceed into the more usually liquid samples are injected by a micro syringe you must have seen a several syringes when you have visited a doctor and what he does? He takes the medicine in a syringe and inject you here and it is essentially the same syringe, but, the capacity of the syringe what we use in gas chromatography is of the order of a few micro liters may be 1 or 0.5 milliliters maximum that is about 500 micro liters. People do use 1 ml etcetera, but, it is better to control the liquids to a few milliliters micro liters milliliters, I mean the insertion of the sample is the most exacting problem in gas

chromatography what you should be doing is you are suppose to take the sample through a into a micro syringe. And then push it through that white hole which I had shown as a rubber septum in my previous this thing, this here you have to this is the rubber septum through which you have to inject the needle just like what your family doctor does it for you whenever you have to whenever you go to him.

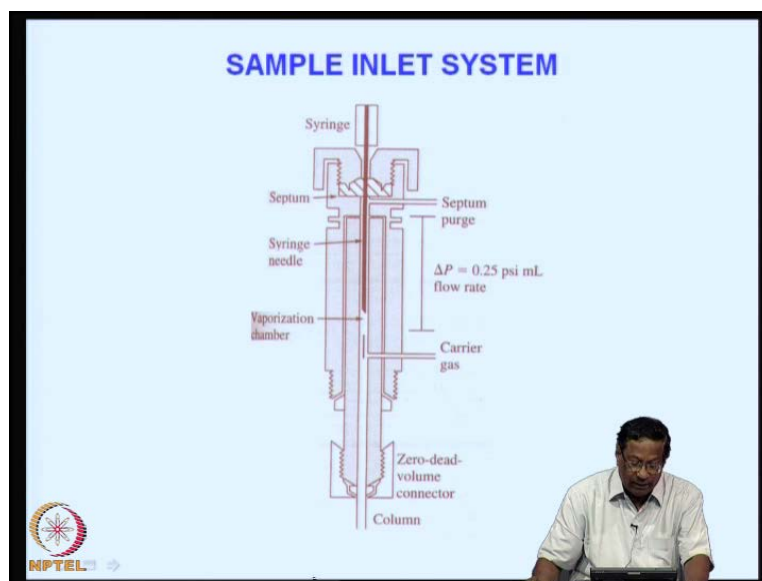
So, the insertion, injection and withdrawal of the needle should be accomplished in such a way that there is no loss of the sample when you take it from the sample inject it into the gas chromatograph as well as when you pull it out. So, the liquid should be drawn out without any loss from the liquid phase and then it should be inserted into a heated column before that there should not be any loss and when you are pulling out there should not be any liquid left out into the syringe.

So, this whole operation sucking injecting and then pulling out that itself forms a fine art usually good gas chromatographers gas chromatographic instrument operators usually perfect this technique otherwise other people would require a little more practice to take out the sample inject it and take it out etcetera **etcetera**.

So, the whole performance should be completed very quickly and smoothly and with reproducibility if there is no reproducibility, what is the sanctity of analysis? So, that itself is a fine art. So, the gaseous samples how do you inject a gaseous sample, if it is a liquid we do it through a micro syringe how do you do a gas sample what you should be doing is you have to take a micro syringe which is gas tight. So, you have to inject suck the gas into a gas tight syringe and then insert it and proceed ahead as usual.

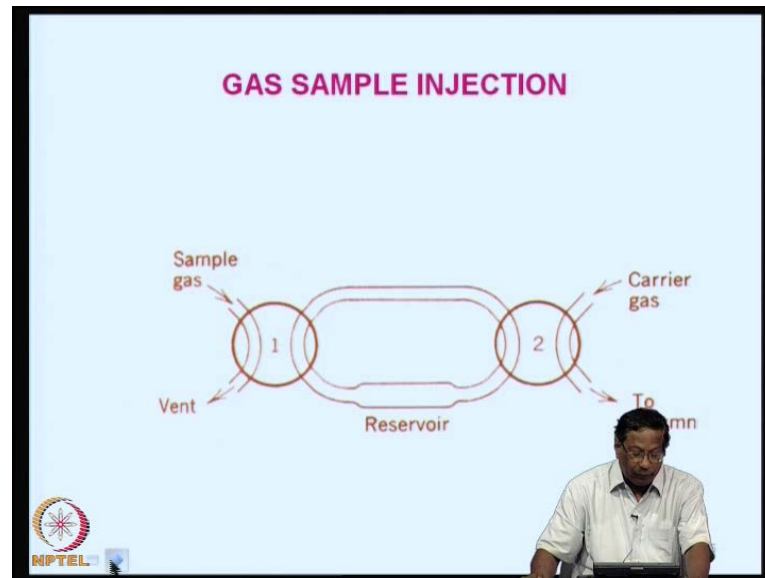
But it is also possible to introduce the gas through a **through a** or arrangement of loops and valves. I will show you the loops and valves arrangement shortly, but, what you should remember is typical sample volumes range from 0.1 to 2 milliliters that is 200 micro liters, this is how? A sample inlet system looks see here this is a syringe and this is a piston and there is vaporization.

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This is a chamber which is heated and carrier gas is coming through this through the side. So, the syringe is almost very near where carrier gas comes in and when you plunge the syringe plunger it goes directly below this carrier gas; and the carrier gas will be taking with it the sample vapor also. And this is the syringe needle septum this is the rubber septum which I had shown as a **as a** white marked piece and then here you can **you can** see this septum purge and other things are there usually the pressure regulator is 0.25 psi per ml and the zero dead volume connector is also connected here. So, what it does is there should not be any volume of the sample that is retained here. So, this is the dead volume basically.

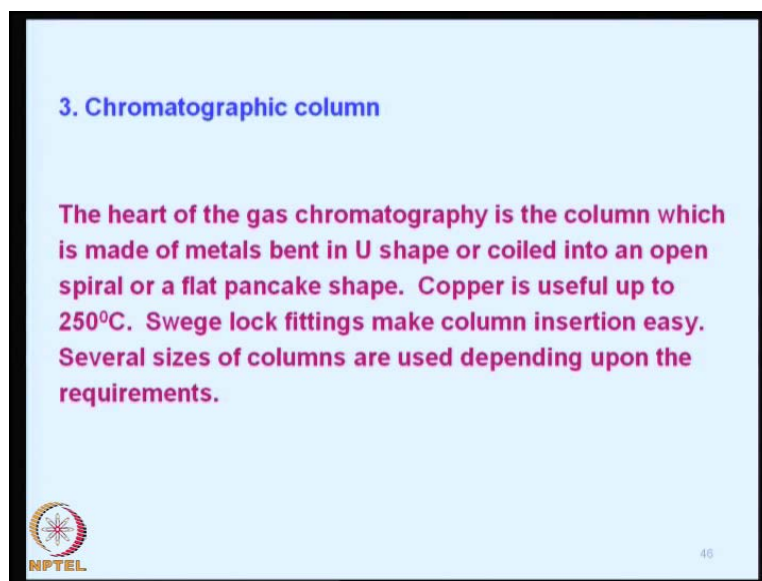
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So, whenever you are injecting a sample you have to make sure that the sample does not remain here and in general, that is how? A liquid sample is connected, injected now; this is a gas sample injection through a valve and loop arrangement.

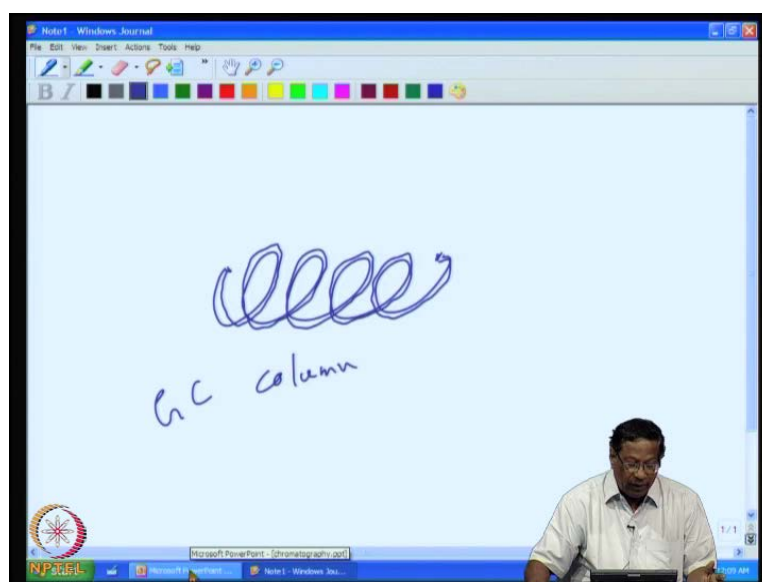
Here, the sample gas is coming through and it is allowed it goes out. What we have? Here is a locking arrangement and this is the carrier gas arrangement and that also is passing through like this. So, whenever the gas is coming through we collect the gas here and then this carrier gas is connected and takes it as a plug and directly through the loop system.

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So the next part, what we want to discuss? Is about the chromatographic column as I told you, the chromatographic column is basically the heart of the gas chromatography it is the column which is made of metals; it is a metal tube basically it is bent in U shape or coiled into an open tube, why do we need this coiling? Is because, it will occupy less space. So, this is how a column will look.

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So, this is the gas chromatography column this can be made of the metal as well as it can be made of glass, we will see how more about this when we discuss this further. So, what

I would like to say at this stage is the gas chromatographic columns are either in u-shape or they are coiled into an open spiral or a flat pan cake type and copper is a useful material for gas chromatography and it is useful up to 250 degree centigrade higher than that is not, so useful.

So, we will try to look for, if your applications are within 250 degree centigrade; that means, if your samples can be separated as vapors up to 250 degree centigrade; that means, there boiling points are below 250 degree centigrade you can use copper columns otherwise you can go for still columns or glass columns and depending upon other applications.

Usually, how do you fit a column to the injection tube and to the injector, sample injector and then you have to also to fit the column to a detector at the end. So, at all these joints there should not be gas leakage. So, usually the arrangements are made in such a way that stage lock is fittings are used to make the column insertion easy and stage lock fittings also make sure that the gas is not leaking.

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COLUMN SPECIFICATIONS					
Parameters	Open tubular	1/16 *	1/8 *	1/4 *	3/8 *
ID (mm)	0.25 0.50	1.2	1.65	3.94	8
Length (m)	100	20	20	20	30
Plates	3,00,000	60,000	48,000	30,000	15,000
Plates / m	3000	3000	2400	1500	750-500
Liq.phase	3	5	10	20
Film thickness	1	5	5	10	20
Sample size (μl)	0.01	1.0	2.0	20	1000

NPTEL All dimensions in inches 47

So, several sizes of columns are used depending upon the requirements. You can see here, I have listed a few column specifications in the table you can see that I have written here parameters and these are all open tubular this is one inch this is 1 by 18, this is 1 by 4 and this is 3 by 8 and all these are in dimensions are in inches. So, the internal diameter

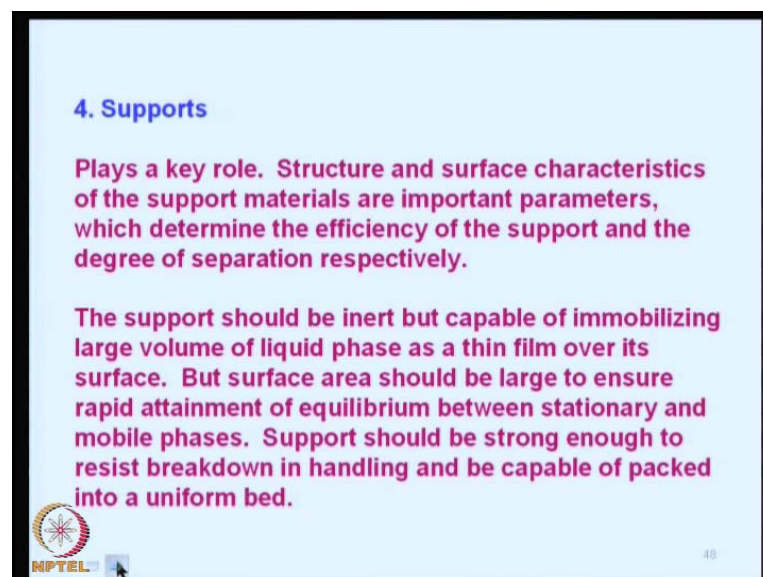
is of the order of a few millimeters and open tubular columns it is 0.25 to 0.5 millimeter and 1 by 16 it is about 1.2, 1.65, 3.94, 8 etcetera that is for bigger diameters columns.

And then lengths typical lengths of a column are of the order of 100 meters. So, you will imagine that if, big column is to be used it should better be in the spiral form otherwise how are you going to fix a 100 meter tube into a gas chromatograph without making it look huge and its functions remain the same. So, it is better to use spiral columns or u-tube columns to make sure that such long lengths are accommodated in the small sample in the small equipment and 1 by 16 inch are of the order of about 20, 20, 20, 30 etcetera.

And the theoretical number of plates this is a very important concept and this plates are of the order of about 300000 for open tubular 60000 for 1 by 16 48000 for 1 by 8, 30 and 15 you can see that as the diameter increases plate **plate** numbers number of plates will keep on decreasing. So, this is very logical. So, plates per meter are of the order of about 3000 to 750.

So, the liquid phase we will not discuss it for the time being and this is a approximate loading that is 3, 5, 10 and 20 percent; that means, the liquid component in the stationary column is of the order now film thickness it is in **it is in** millimeter 1, 5, 10 it could be microns **sorry** they are **they are** in 1 to 20 microns and then sample size useful for open tubular column is 0.01 micro liters and 1 by 16 it is 1 to 10 etcetera micro liter and maximum you can handle is approximately around 1000 micro liter.



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4. Supports

Plays a key role. Structure and surface characteristics of the support materials are important parameters, which determine the efficiency of the support and the degree of separation respectively.

The support should be inert but capable of immobilizing large volume of liquid phase as a thin film over its surface. But surface area should be large to ensure rapid attainment of equilibrium between stationary and mobile phases. Support should be strong enough to resist breakdown in handling and be capable of packed into a uniform bed.

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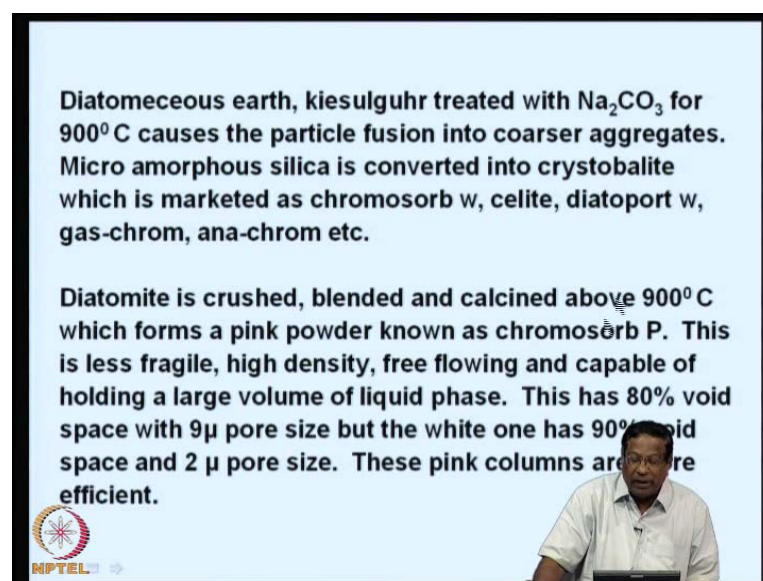
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Now, the column cannot be metal tube alone, because metal tube only acts as a support now the column should have a solid phase on which the solid phase is to be coated. So, the support is filled into the column after coating the support. So, the support also plays a very big role in the separation. Therefore, the structure and surface characteristics of the support materials are all the important parameters in the selection of the columns. In fact, these things determine the efficiency of the support and the degree of separation respectively.

The support should be inert it should not react with the carrier gas nor the components that is one of the requirement anyway and it is capable of immobilizing large volume of liquid phase; because, larger the liquid phase volume the better would be the number of plates, but, the thickness should be as small as possible to get more number of theoretical plates which will ensure higher degree of separation better separation.



Then the surface area should be large to ensure rapid attainment of the equilibrium between the stationary phase and the mobile phase that is also very important. So, the support should be strong enough to withstand the breakdown in handling and be capable of being packed in a small tube to a good consistency into. So, what we are essentially looking at is the good packing of the support material in a coat support material coated with the liquid and put into a tube and this tube should be capable of being coiled and put into the column it should be put not into the column, but, in the oven.

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Diatomeaceous earth, kiesulguhr treated with Na_2CO_3 for 900°C causes the particle fusion into coarser aggregates. Micro amorphous silica is converted into cristobalite which is marketed as chromosorb w, celite, diatoport w, gas-chrom, ana-chrom etc.

Diatomite is crushed, blended and calcined above 900°C which forms a pink powder known as chromosorb P. This is less fragile, high density, free flowing and capable of holding a large volume of liquid phase. This has 80% void space with 9μ pore size but the white one has 90% void space and 2μ pore size. These pink columns are more efficient.



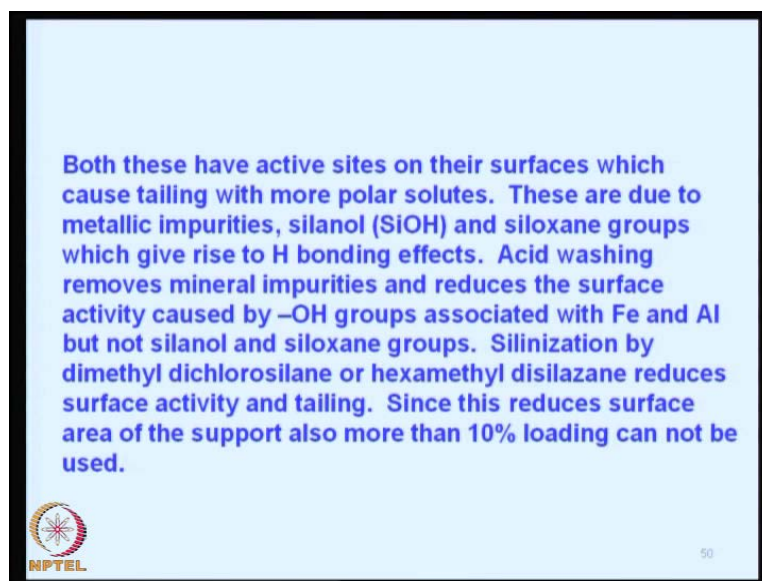
So, what are support materials you can choose the number of them; one is diatomaceous earth, one is kieselguhr treated with sodium carbonate kieselguhr is another type of soil that is available and it is treated with sodium carbonate with about for 900 degree centigrade and when you take this kind of soil particles and mix it with sodium carbonate for about 900 centigrade it causes the particles to fuse together into coarser aggregates and this aggregates you will have to powder them and then it is your support material.

So, more micro amorphous silica is usually converted into cristobalite that is another form of the silica silica structure which is marketed as Chromosorb W, Celite, Diatoport W, Gas-chrom, Ana-chrom etcetera. What I am trying to tell here is the support material is marketed separately and you can buy these things from the vendors, who are supplying chromatographic material? They sell you sir I have a Chromosorb W and another will say sir, I have Celite, sir I sell only Diatoport W like that there are different trade names and all these trade names of the support materials actually refers to the treated silica with sodium carbonate at around 900 degree centigrade and powdered into different particles sizes.

So, diatomite is usually crushed blended and calcined about 900 degrees centigrade which forms a pink powder known as Chromosorb P; Chromosorb P is the trade name. Now, you would have seen this diatomite earth and this is just a type of ordinary bricks just like we use for construction in our houses. So, there are so many brick making factories and what is important is? Many of those things you would have seen after heating they get converted into pinkish bricks these things are powdered and then marketed as Chromosorb P.

So, there is no need to panic the moment you hear Chromosorb P or something like that being marketed at it could be something special no it is nothing very special it is just the soil heated at 900 degree centigrade and made into bricks and powdered. So, this is less fragile and it is got high density and when you powder it and pour it from the top it is free flowing; that means, it does not stick to your hands or the container and it is capable of holding a large volume of liquid phase. This has about 80 percent void space with about 9 micron pore size and the one which is marketed as white white one has about 90 percent void space and 2 micron pore size. These pink columns are more efficient among the white and pink materials pink columns are usually more efficient.

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So, both these activities both these substances have active sites on their surfaces that is both the chromosorb support materials have active silica and OH groups on their surfaces which cause usually tailing with molar more polar solutes; that means, if you have the more active sites in the support material your chromatograph that is coming out at the end will have more tailing, because the more is the active site, more is the liquid that a sample that is held. So, it will come out slightly more slowly.

These are due to metallic impurities and SiOH group, silanol groups and siloxane groups which give rise to hydrogen bonding effects. So, acid washing usually removes acid washing removes mineral impurities and reduces the surface activity caused by the OH groups associated with iron and aluminum and other several other metals that are there in the silanol.

Silinization by dimethyl, dichlorosilane or hexamethyl disilazane you can do silinization; that means, you make the converted it into silanol group silinization by using dichlorosilane or hexamethyl, disilzane this reduces the surface activity; that means, they convert most of them into silanol group and then there is the active sites are reduced; that means, there will not be any tailing they no tailing means the peak will be sharper.

So, this silinization reduces the surface area of the support also more than by about 10 percent and the loading can be approximately 10 percent loading cannot be used; that means, about 10 percent is lost activity is lost during these processes. So, coating of the

support that is now that you have the brick powder white and pink that is chromosorb P's you have the support and we can use stearic acid to coat on the brick powder or chromosorb it is incorporated stearic acid incorporated in silicone oil we use them to eliminate tailing in the separation of fatty acids, because stearic acid is also an acid hydrocarbon compound which is a solid compound which melts at high temperature you can incorporate stearic acid in silicone oil and then use it on the chromosorb that helps in eliminating the tailing in the separation of fatty acids.


For amides what do we do for amines we can use potassium hydroxide treatments. So, different treatment systems are available for the support material to be treated to avoid tailing in the end sometimes what we do? Is apart from this chromosorb we also use glass beads. So, glass beads with a low surface area and a low porosity they can also be used to coat you can use up to 3 percent of the stationary phase and to coat and then usually about 0.05 to 0.2 percent loadings permit the analysis of high boiling substances at fairly low temperatures.

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Coating of the support with stearic acid incorporated in silicone oil eliminates tailing in the separation of fatty acids. For amines KOH may be used for coating.

Glass beads with a low surface area and low porosity can be used to coat up to 3% stationary phases. 0.05 – 0.2% loadings permit the analysis of high boiling substances at fairly low temperatures.

Porous polymer beads differing in the degree of cross linking of styrene with alkyl-vinyl benzene are also used which are stable up to 250^o C. These are marketed as Porapak P,Q,R,S etc.

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Now, you can see that apart from the chlorine apart from the glass beads you can use porous polymer beads you would have seen number of porous polymer beads they look like ion exchange resins small **small** white transparent beads or you would have seen polished iron beads which are used in the exhibitions. And other places you can use polished iron also such beads differing in the degree of cross linking of styrene with

alkyl, vinyl benzene are used which are stable up to 250 degree centigrade usually polymer beads even though they are used as supports you cannot use them at very high temperature, because they themselves will start disintegrating at higher temperature we do not want that to happen.

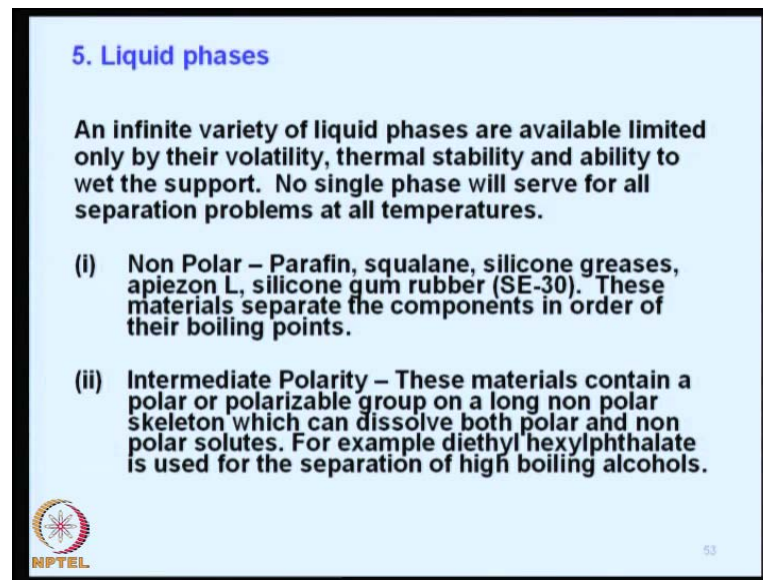
So, there is a limit which we can use glass beads also or plastic beads, polymer beads and such polymer beads are marketed with **with** trade name starting from P that is they are marketed as a porapak **porapak** P, porapak Q, porapak R, porapak S, etcetera which represent different types of loading of the liquids on to the polymer resins. So, the mesh size of the support that is the particle size of the support determines the average particle diameter which in turn determines the H E T P; that is the theoretical number of plates, height of the effective theoretical numbers of the plate.

Therefore theoretically smallest particle should be used because that will give you better efficiency more number of plates. So, that it causes the permeability which is essentially a product of the particle which is proportional to the particle diameter square of the particle diameter and pressure drop it is the permeability also will reduce the pressure drop in longer columns.

Coarser particles can be used to reduce the pressure drop, best columns are those with 80 to 100 meshes now the 80 to 100 mesh particle sizes you cannot use in very small columns. So, the optimum size of the column also is important. So, 80 by 100 mesh size of the chromosorb or a support material or whatever it is you can fill comfortably in 1 by 8th inch and higher columns. So, 1 by 8th inch is a diatomaceous earth type supports they are usually very comfortable.

So, for effective packing of any column the internal diameter should be about 8 times, the width of the 8 times the diameter of the supports this is a general empirical rule that is the internal diameter should be at least 8 times the diameter of the supports **supports** material. Now, let us discuss about the liquid phases.


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5. Liquid phases

An infinite variety of liquid phases are available limited only by their volatility, thermal stability and ability to wet the support. No single phase will serve for all separation problems at all temperatures.

- (i) **Non Polar** – Paraffin, squalane, silicone greases, apiezon L, silicone gum rubber (SE-30). These materials separate the components in order of their boiling points.
- (ii) **Intermediate Polarity** – These materials contain a polar or polarizable group on a long non polar skeleton which can dissolve both polar and non polar solutes. For example diethyl hexylphthalate is used for the separation of high boiling alcohols.

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So, the liquid phases are usually can have a number of liquid phases they are readily available and there use is only limited by their volatility, thermal stability and ability to get the support it is a very important parameter, the support must also be wetted by the liquid to form a thin film membrane over the material.

So, no single phase actually will serve for all separation column there is no (()) you have to try to understand, what kind of materials you are separate? You you want to separate and there is a requirement which will satisfy the column material, column support etcetera. And the liquid phase there are a number of them which will satisfy, but, there is no single phase that will serve for all separation problems at all the temperature we have to understand this in a very clear manner, because this will tell you that you will have to buy a number of columns or variety of columns to achieve the separation of a variety of substances.

That means if your samples are varied if they if they have compositions which are different from from known from known composition you have to have more number of columns with different liquid supports. So, what we can do? Is we can classify them into 2 or 3 2 or 3 classes; one is non polar and the liquid phase non polar means you can the examples are like this paraffin, squalane, silicon greases, apiezon L and silicon gum rubber that is known as marketed by a trade name marketed by a trade name known as

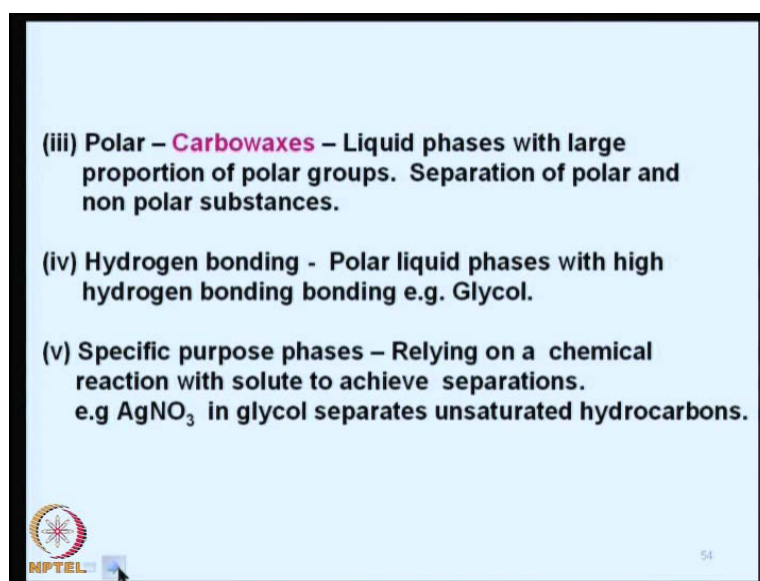
SE 30 and all these things are basically non polar liquid substances of very high molecular weights.

These materials separate the separate the components in the order of their boiling points; that means, if you have hydrocarbons you want to separate them and their boiling points are different then what you should do is? You should chose a column with a support material coated with paraffins, squalane, silicon greases, apiezon L, silicon gum rubber, etcetera one of these things will do.

So, if you want to separate materials which are partly polar and partly non polar that is intermediate polarity then what you should do is? You should dissolve a liquid known diethyl hexylphthalate on to the you should coat these substances diethyl hexylphthalate on to the liquid support and then that can be used for the separation of higher boiling alcohols; usually, alcohols are having higher polarity compared to hydrocarbons.

So, if you want to separate the alcohols you can go for these a such liquids and then you can have the polar substances you want to separate only the polar substances then in that case the carbowaxes are required and liquid phases with large proportion of polar groups and separation of which will help in the separation of polar as well as non polar substances that is also possible..



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(iii) Polar – **Carbowaxes** – Liquid phases with large proportion of polar groups. Separation of polar and non polar substances.

(iv) Hydrogen bonding - Polar liquid phases with high hydrogen bonding bonding e.g. Glycol.

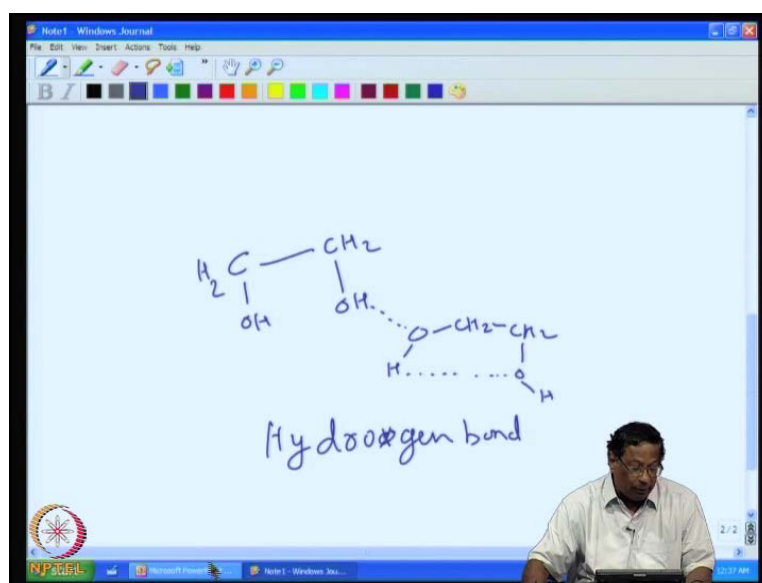
(v) Specific purpose phases – Relying on a chemical reaction with solute to achieve separations.
e.g AgNO_3 in glycol separates unsaturated hydrocarbons.

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So, depending upon the polarity and the boiling point you can choose different kinds of liquid support, liquid **liquid** phase and the support also, sometimes you may have substances which will form hydrogen bonds. For example, glycol; the glycols have a possibility for forming hydrogen bonds and these things will have this hydrogen will form a weak bond with oxygen and then this will be hydrogen this will be another molecule $\text{CH}_2\text{CH}_2\text{OH}$ and this hydrogen can also form a very weak bond with another oxygen like that several substances are capable of forming these hydrogen bonds.

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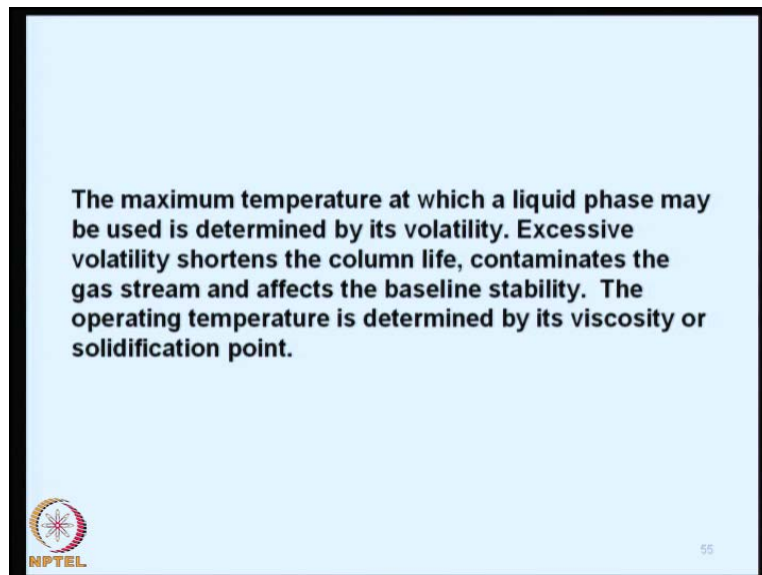


These are hydrogen bonds. Similarly; alcohols, water and so many other substances will form hydrogen bonds. So, you can go back and see whether your substances you want to separate are capable of forming the hydrogen bonds or not. So, in such cases polar liquid phases with higher hydrogen bonding usually for go for glycol this should not have been there bonding, but, we will correct them if possible.

So, specific purpose phases we can have apart from the polar, non polar and then intermediate polar as well as hydrogen bonding etcetera. So, these things reliable rely on a chemical reaction with the solute to achieve separations. For example, I can use silver nitrate in glycol to separate the unsaturated hydrocarbons from a given mixture of the sample, suppose you have hydrocarbons and you want to mix with unsaturated

hydrocarbons we can simply pass them through a column containing silver nitrate in glycol and then they will retain the unsaturated hydrocarbons.

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The maximum temperature at which a liquid phase may be used is determined by its volatility. Now, again we are coming back to the generalities. So, the maximum temperature at which a liquid phase may be used is determined by volatile; if, it is volatile you need not raise the temperature of the column much higher than its volatilization temperature. So, excessive volatility supposes you raise the column temperature to very high level then the liquid will decompose to some extent. So, the excessive volatility usually shortens the column life it contaminates the gas stream and then it affects the base line, stability also.

The operating temperature is usually determined by its viscosity and solidification point that is what I want to convey to you that whenever you want to make any separation in the gas chromatography column do not raise the column temperature much higher than the boiling points of the components what you want to use. So, the maximum temperature should be just about 10 or 15 degrees higher than the maximum boiling temperature component of your sample.



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6. Column loading

15% loading means 15% liquid phase and 85% support. The lower the amount of the solid phase, the smaller is the sample that can be handled.

7. Open tubular columns (capillary)


An open glass capillary tube coated with liquid phase on the interior of the tubing. This permits use of very long columns (30-100 meters). Because of the low pressure drop, such columns give a very high overall column efficiency. Theoretical plates of 100,000 are possible. The diameter could be 0.07 – 0.25 mm. But the sample capacity is only about 0.1 to 0.3% of normal samples.

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So, another aspect that is column loading, how much you want to, how much you would like to load your columns with the liquid that is mobile phase **no sorry** stationary phase. Usually, 15 percent loading we have an expression whenever you want to buy a column for gas chromatograph we will say, the vendor will tell you sir, I have a prop a column with 15 percent loading. So, what does it mean it means the column has got 15 percent liquid phase and 85 percent of it is support. The lower the amount of the solid phase the smaller is the sample that can be handled. So, higher loading more is the sample that can be separated. So, this point I want to convey to you; that it is important information whenever you want to buy a column.

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Parameters	Open tubular	1/16 *	1/8 *	1/4 *	3/8 *
ID (mm)	0.25 0.50	1.2	1.65	3.94	8
Length (m)	100	20	20	20	30
Plates	3,00,000	60,000	48,000	30,000	15,000
Plates / m	3000	3000	2400	1500	750
Liq.phase	3	5	10	20
Film thickness	1	5	5	10	20
Sample size (μ l)	0.01	1.0	2.0	20	1000

 All dimensions in inches 47

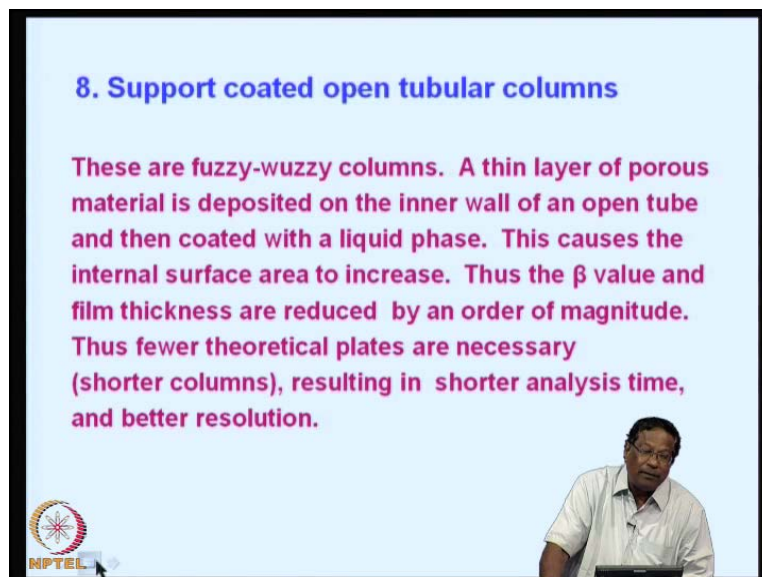
So, there are other kinds of column which are known as open tubular columns. If you remember my previous one of the previous slides in which I showed the characteristics of the columns you can see that, I have put open tubular columns here and the first one is open tubular columns and its ID is 2.25 to 0.5 mm and it shows highest number of theoretical plates. And, now, I am going to discuss little bit about this, just to give you an idea about the open tubular columns. So, what is an open tubular column it is basically a capillary column that is a glass tube with 0.5 mm diameter. So, the diameter of the tube is about 0.5 and you have to lower the liquid, coat the liquid inside the capillary and this permits the use of very long columns almost about 30 to 100 meters.

So, all the capillary columns must be you must be in a position to coil them round and **round and** round which I had shown you in my previous one of the window journal programmes and the whole thing should be fit able to the sample injection unit as well as to the detector outside and the whole thing should be put in the oven that is thermal compartment. So, this permits the use of very long column and because of the **because of the** tube is open there is nothing inside except the smalls of the capillary are coated with the liquid.

In generally, it is an empty tube. So, because of the low they will not have present much pressure drop, because of the pressure drop such columns give a high over all column efficiency. So, theoretical plates of 100000 are also possible in such cases. The diameter



could be 0.07 to 0.25 mm, but, the sample capacity is only about 0.1 to 0.3 percent of the normal samples then we can discuss a little about support coated open tubular columns.

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8. Support coated open tubular columns

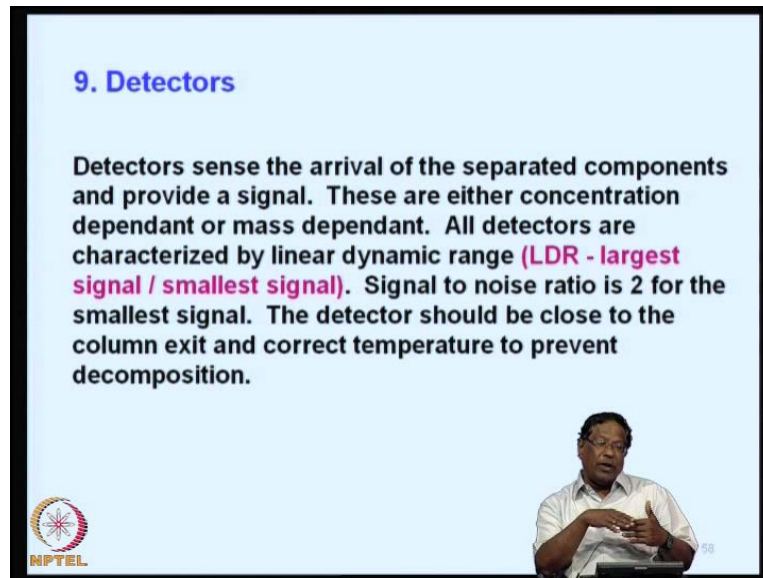
These are fuzzy-wuzzy columns. A thin layer of porous material is deposited on the inner wall of an open tube and then coated with a liquid phase. This causes the internal surface area to increase. Thus the β value and film thickness are reduced by an order of magnitude. Thus fewer theoretical plates are necessary (shorter columns), resulting in shorter analysis time, and better resolution.

These are known as fuzzy-wuzzy columns. A thin layer of porous material is usually deposited on the inner wall of the open tube and then coated with a liquid phase. This causes the internal surface area also to increase that is a very important concept in fuzzy-wuzzy columns, because of the coating of the liquid inside the capillary the surface area increases.

Therefore, the beta value and the film thickness are reduced by an order of the magnitude; beta is the incompressibility of the liquid. So, fewer theoretical plates are necessary; that means, you can use shorter columns, fewer theoretical columns are plates are necessary and resulting in the shorter analysis time. Shorter column means shorter they come out faster. So, the analysis time gets shortened and then if you **you** will get a better resolution also, because basically you are increasing the surface area which will result in better separation.

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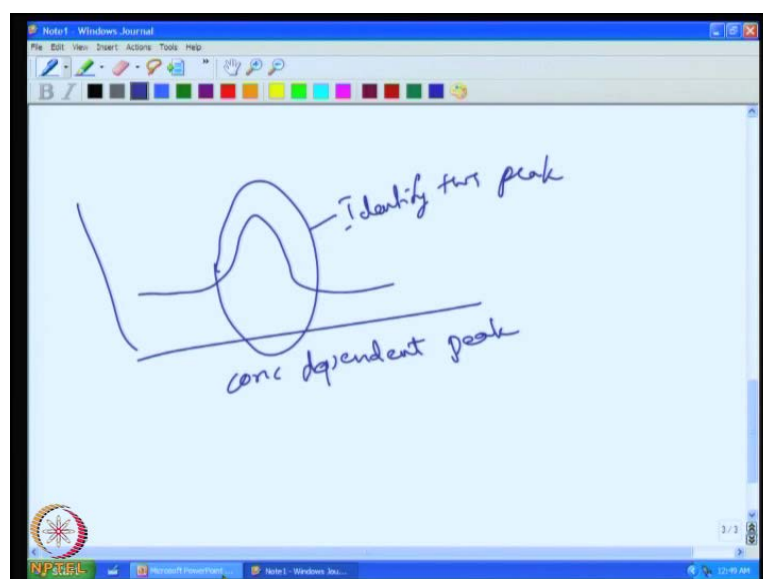
9. Detectors

Detectors sense the arrival of the separated components and provide a signal. These are either concentration dependant or mass dependant. All detectors are characterized by linear dynamic range (LDR - largest signal / smallest signal). Signal to noise ratio is 2 for the smallest signal. The detector should be close to the column exit and correct temperature to prevent decomposition.

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So, now let us stop our discussion on the columns, but, go on to the detectors, I would like to say at this stage that the detectors usually sense the arrival of the separated components and provide a signal, the job of the detector is to sense the arrival of the separated components and it has to give a signal. So, they can be either concentration dependant or they can be mass dependant; that means, when the sample is coming out of the column, the detector should sense as the column, as the concentration of the out coming component increases it should give you a peak something like this **this** is a concentration dependant peak.

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Identify this peak

conc dependent peak

So, this is your basically your carrier gas. So, as the carrier gas as the sample increases, the signal will increase reach its maximum and as it reduces it will come down again to the carrier gas level. So, the job of the detector is to identify this peak; that is, you can identify this peak either of the sample by its specific properties related to concentration or you can use simply the carrier gas.

Take the carrier gas and as the sample increases as the sample comes out the mass of the material reaching the detector will increase. So, long as the component is not separated it will be showing you a steady concentration or steady base line, the moment a sample component comes out **the moment a sample component comes out** the signal will be higher.

So, this is known as mass dependant detector. So, they you can use either a concentration detector like this **like this** or a mass detector, either way the detector can be characterized. So, the signal to noise ratio is twice for the smallest signal, the detector should be close to the column exit that is a very important concept and the correct temperature also it must show to prevent the composition. What we will do is? We will discuss more about the detectors and the applications of gas chromatography in the next class. .