Flow through Porous Media
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Lecture – 60
Characterization (Contd.) & Experimental Demonstration of BED Analysis

I welcome you to this lecture of Flow through porous media. We are going to talk very briefly about Characterization in this module and then there would be some laboratory module which you will get to go through. Now, this characterization here we have already talked about some methods like porosity, etcetera.

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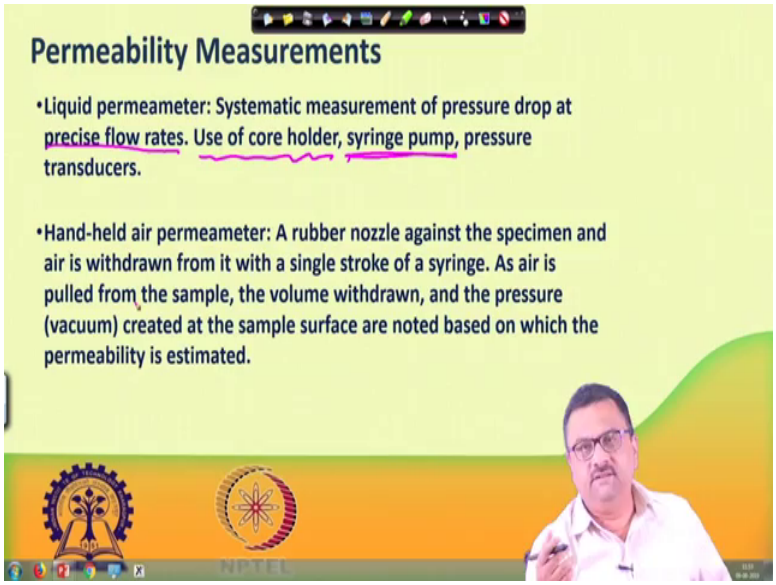

We need to discuss about the permeability measurement quickly; permeability measurement we have already talked about this liquid permeameter; I mean, we have not called this as such liquid permeameter, but the essential theory is already known to you that you need to systematically measure the pressure drop at precise flow rates. So, precise flow rates can be generated by instrument called syringe pump, where a syringe just like an injection syringe; a syringe will be the piston will be moved by a specialized rack and pinion arrangement that is a very precisely controlled.

And, then pressure transducers will be there to measure the pressure drop across the length of that porous medium and one can use core holder if you are if you want to

measure the permeability of the core without disturbing the core one can put it in a core holder and using rubber sleeve; so, that there is no leakage through the side so, there are ways to measure these permeability. And, then also I must point out that there are handheld air permeameter, which is basically a rubber nozzle against the specimen and air.

So, basically if a on a rock face you can hold this and then try to pull out some air and try to see what kind of pressure one has to generate for a particular volume of volume withdrawn and from there you can. So, it is basically from a rock face, see if we want to just get a quick idea how much is the permeability one can use these handheld air permeameter.

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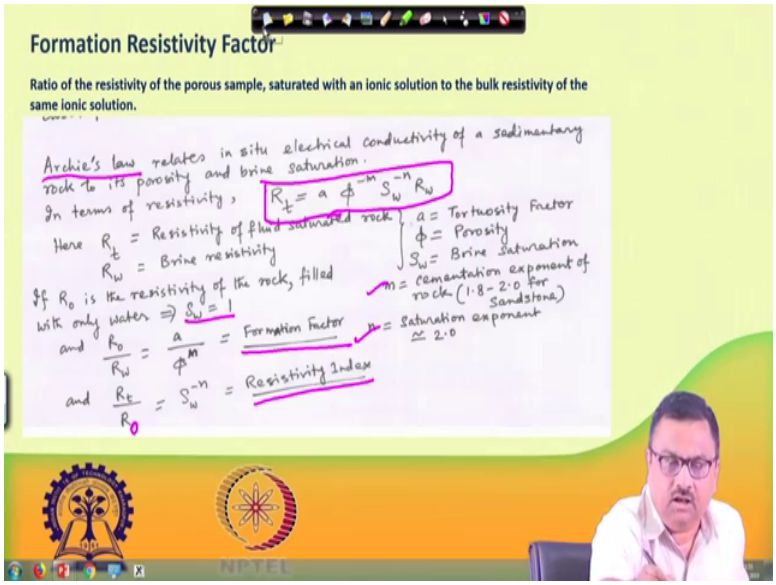

There is also this formation resistivity factor I must point out here; this is the ratio of resistivity of the porous sample saturated with an ionic solution to the bulk resistivity. So, one needs to measure the electrical conductivity and one has to invoke these Archie's law which says, that this relates this electrical conductivity of a sedimentary rock to it is porosity.

So, this is the equation where R t is the resistivity of fluid saturated rock and R W is the brine resistivity. So, brine is 1 percent basically a salt solution, it has very small amount of sodium chloride or I mean, one can simulate a brine by adding 1 percent NaCl in

water. Now, R o is the resistivity of the rock filled with only water; that means, when S W is equal to 1.

So, then this R o by R W that is by this equation R o by R W would be a divided by phi to the power m, because S W is equal to 1 and this is known as formation factor. And, similarly, R t by R o; R t by R o would be in that case S W to the power minus n and this is known as the resistivity index. So, one can measure this formation factor and resistivity index and from this one can get some idea of how much of fluid is there inside a porous medium.

So, these are the numbers given a is the tortuosity factor and phi S w, etcetera they are given and m and n these numbers for rock they are given. What is the use of this formation resistivity factor?

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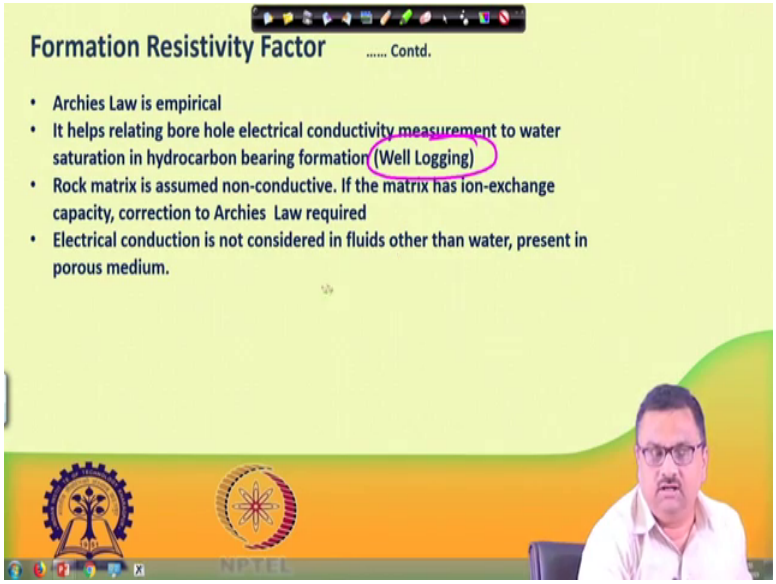

This is these are some of the things you must note this law is empirical, it helps relating bore hole electrical conductivity measurement to water saturation in hydrocarbon bearing formation. This is well logging those who are in petroleum engineering or geo or measuring the subsurface, who are into subsurface measurements these well logging is a very important technique, where one can find out how much of liquid is there in subsurface and therefore that these resistivity factor is very important.

So, here you assume rock matrix is non-conductive, if the matrix is ion exchange capacity then some correction is required and electrical conduction is not considered in fluids other than water. So, if you have oil and water you will assume only electrical conduction is in water. So, these are some of the points to be noted in this context.

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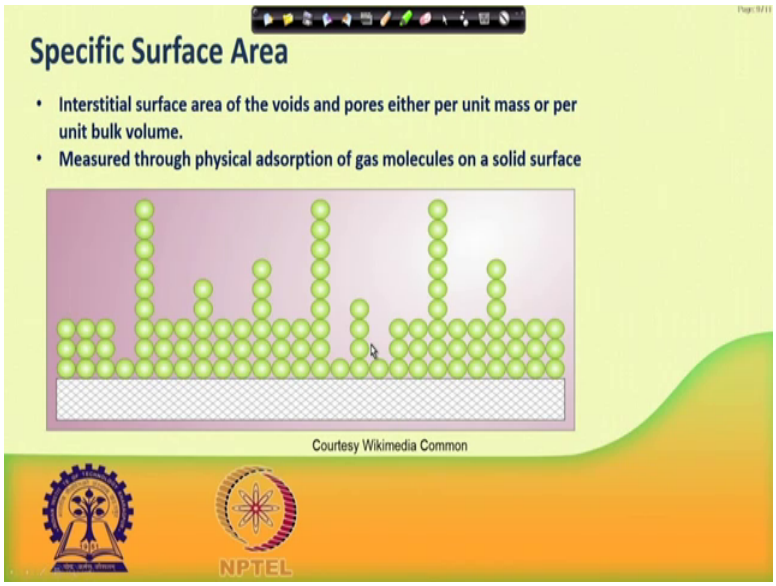

There is this other the last point that we must note here which is the specific surface area. How much of surface area is there inside a porous medium and generally this nitrogen adsorption desorption experiment is performed for this purpose? So, there interstitial surface area of voids and pores either per unit mass or per unit bulk volume can be measured.

This measurement is through physical adsorption of gas molecules on a solid surface. So, gas molecules can form mono layer; that means, it is a single layer. So, this is mono layer coverage, this particular portion is only a mono layer coverage you can see, but here then you can have some R 2 layers, some R 3 layers, some are multiple layers.

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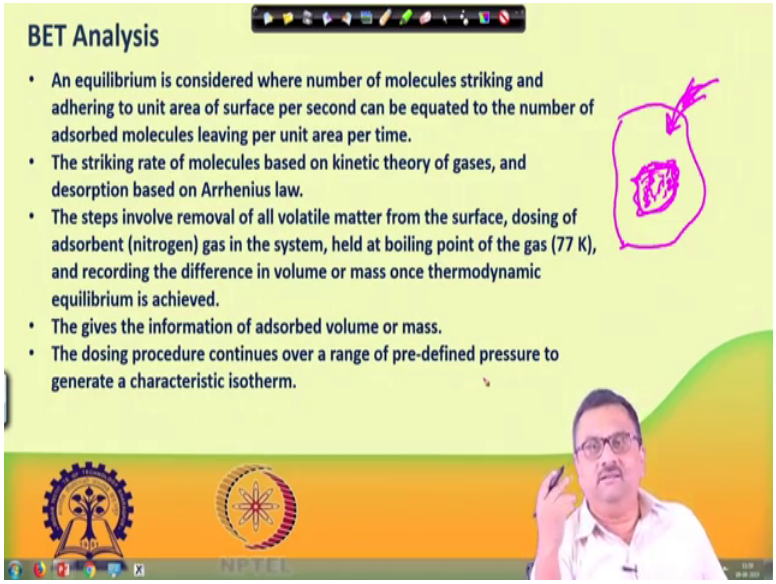

So, this is something which you have. Now, one can perform something called a BET analysis. We have a very good experimental description of this immediately after this within this lecture module itself. One assumes an equilibrium on the number of molecules striking and adhering to unit area of surface per second and that is equated to the number of adsorbed molecules leaving per unit area per time.

So, this is basically the essential equation that is solved here and the striking rate of molecules are based on kinetic theory of gases and desorption based on Arrhenius law. The steps involved removal; so, when per somebody performs a BET experiment the steps involved as first remove all volatile matter from the surface by applying vacuum, then dozing of adsorbent which is mostly nitrogen gas, because it is inert it will not react with the system. So, adsorbent gas is introduced in the system which is held at the boiling point of the gas which is 77 Kelvin.

So, one is the elaborate cooling there and then recording the difference in volume or mass once thermodynamic equilibrium is achieved. That means, you have these let us say I have this much of solid material placed in a chamber and then I bring in nitrogen gas. And I see that as the pressure changes, how much of nitrogen is adsorbed on the surface, because there are porous there is internal surface available inside this porous medium. So, I want to see as the pressure changes as I increase the pressure then, as I

increase the pressure how much of nitrogen extra nitrogen goes into this enclosed chamber.

So, that gives me that additional nitrogen must be getting adsorbed on the surface, first it would be a monolayer coverage and then as you increase the pressure there will be multiple layers and then as you continue to increase there would be condensation inside the pore we have already talked about it Kelvin equation, etcetera. And, when this pressure reaches the saturation vapor pressure when P by P naught becomes 1 then we will see nitrogen forms liquid everywhere on the surface.

So, that is idea so, we are not going to that level of P by P naught, but very small amount of P by P naught as we change the pressure we note how much of mass or how much of volume of nitrogen has to, nitrogen goes into the system. So, that is what we note, this gives the information of adsorbed volume or mass, the dozing procedure continues over a range of predefined pressure to generate a characteristic isotherm.

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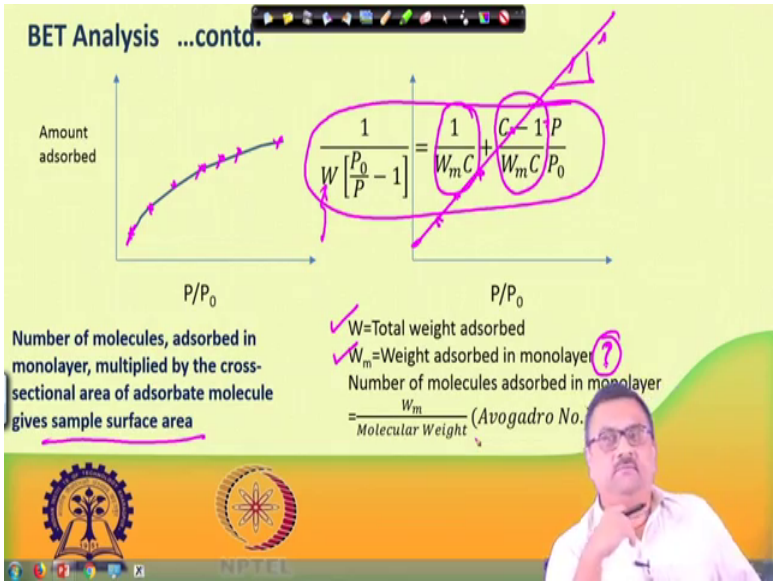

So, what we do here is, we generally this if we look at the amount adsorbed versus P by P naught, we will see that initially this would be a monolayer coverage; initially this is a mono layer coverage and then it takes a turn so, it would be multiple layer.

So, it is this is called isotherm, which is the amount adsorbed as it increases you must remember we have talked about type 1, type 2 isotherm and type 4 isotherm where there

is capillary condensation, we are not going up to that we are just focusing on this part of the adsorption. So, now, here if you the theory that I said if one equates the amount of molecules that are amount adsorbed and the molecules that are leaving the site by desorption one equates them at equilibrium one can arrive at this equation.

So, here W is the total weight adsorbed and W m which is unknown to me, I do not know what is W m, but W I know, because I know how much of extra nitrogen has gone into that end closed chamber; so, I know how much of total weight of nitrogen is adsorbed if I keep a book if I maintain a book how much nitrogen is going in right from the start. So, then I can record I can find out how much nitrogen is adsorbed? So, that is basically W and, that W changes with pressure.

So, now, and W m is something which I want to know I do not know how much is a monolayer coverage, but one thing I know that if I know this weight adsorbed in mono layer then from there I can find out number of molecules adsorbed in mono layer would be simply W m the weight of molecule absorbed as mono layer divided by the molecular weight of nitrogen, this gives me the number of moles of nitrogen adsorbed on the surface. So, number of moles will have how many molecules you have to multiply by Avogadro number 6.02310 to the power 23.

So, you have to multiply that Avogadro number and you will get the, this would be the number of molecules adsorbed in mono layer and then if you go you know one thing for sure that all the surfaces there will be a monolayer coverage some surfaces will have 2 layer, some less surfaces will have 3 layer like this, but at least all surfaces will have mono layer covering. So, number of molecules adsorbed in mono layer, multiplied by the cross sectional area of one molecule.

So, if I know what is the cross sectional area of one nitrogen molecule, we multiply it by this number of molecules that I mentioned then I can get what is the sample surface area? Ok. So, now, this is something which will be done now how to find out W m? So, W m can be found out by the equation that one has generated by considering that equilibrium as I said just now; number of molecules is striking the surface and number of molecules leaving through desorption at equilibrium they are equal.

So, then that gives me this equation here these W and P by P naught we already know, because amount adsorbed is W and P by P naught; so, this is generated from the nitrogen

adsorption. These experimental data points are generated by nitrogen adsorption experiment just that I mentioned just now. And, then these I have to be plotted like this and then you if you plot like this you will have data points which would be coming like this.

Now, if you have a least square feet; wherever it is having an intercept that intercept value would be 1 by W m C and the slope would be C minus 1 by W m C, ok. So, by looking at this intercept and slope of this line one can get this idea what how much is W m. And, once we have W m then you find number of molecules adsorbed in mono layer as by this equation and then multiplied by area, cross sectional area of an individual nitrogen molecule one can get the amount of sample surface area.

So, generally high porous when the porous matrix that has high surface area, one can calculate this or you can find out what is the surface area frankly speaking. So, now, what I am going to do is now, next in this lecture as part of this lecture module itself first there will be a laboratory component; where you will be introduced to an experiment of BET or an experimental setup how to do these a nitrogen adsorption experiment. And then we will have some other small experiment on flow through porous media and fluidization bed, ok. So, that is how this lecture module will be organized. So, next we are moving to the experimental demonstration of BET analysis or nitrogen adsorption experiment demonstration.

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For taking the sample there are several types of sample cells are available. So, on that case is if the sample is powdered sample then you can use this type of cell this is called 6 mm cell, ok. Now, if the samples of macroporous nature means, the surface area is very low; say 1 meter square per gram or since low very low, then we have to take a large volume of sample, we have to take a large volume of sample.

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For that we will use this 9 mm bulb cell type, ok. Another types of sample cells are also there pellet cell or your this type of these are called pellet cells. When you are having say some tablet type of materials or beads then you can take the sample in the beads. Actually this sample cells where which type of sample cell we will have to take that depends upon the type of the sample and the if it is a very low surface area material then you have to take more samples, then use this large volume of 9 mm bulk sample and if it is the high surface area sample, then you have to take this 6 mm cell, ok.

Now, whatever sample you have taken now, first to weight in and take it into the cell. Now, this is this has to be degassed at a temperature, at the temperature should be such that it should not change the morphology of the sample.

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So, you can take that carbon sample in that this 6 mm cell and these are the heating mantles, from where you can put the sample or heat the sample; you just put it take the sample first into this way the sample and take the samples into this cell 6 mm cell. Now, put it into this there is a mantle, heating mantle this is called heating mantle.

Now, clamping of this heating mantle should be in this way after taking the sample now, take these are the nuts and some washers, this is one nut and this is another nut these are the two nuts, you just fit it there and a oaring is also there oaring now, oaring is there, ok. Now, this is complete.

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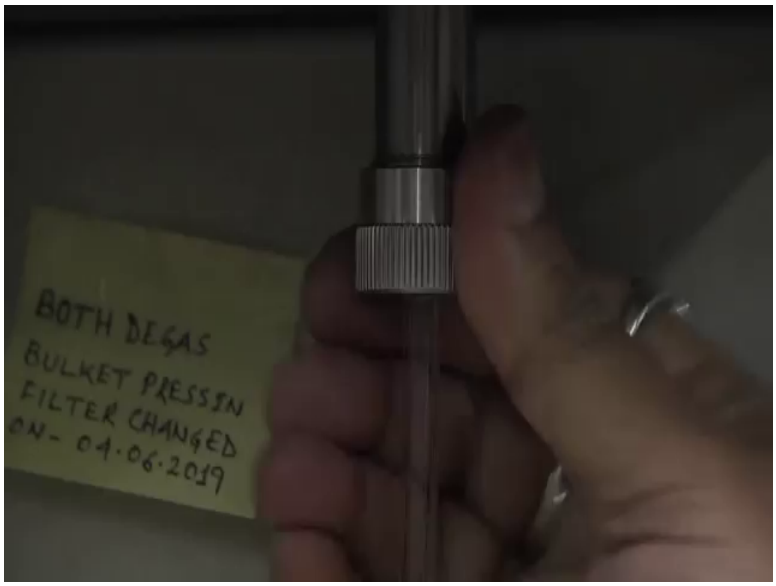

Now, you can fit it over here like this. So, here two do gassing stations are there. This is station 2 and this is station 1; you can use simultaneously both are stations or you can use only one stations also that is up to you.

Now, this heating mantle contains a temperature control and a cut off temperature control. This heating mantle can go up to 350 degree centigrade of temperature. So, if by chance this temperature exceeds 350 degree centigrade, then there is a thermocouple which will cut the temperature and go to safety level. So, that there should not be any bad effect or harmful to the this instrument, ok.

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So, in this region what actually happens? By heating say we are taking carbon sample, so, we have just, we have clicked the degassing temperature, set the degassing temperature to 300 degree centigrade. Now, it is under high vacuum that pores are being cleared or whatever moistures or volatile matters are present inside that your material surfaces sample that should be taken out, taken out under high vacuum, ok.

So, if this is the carbon sample it may take 3 hours like for completing degaussing. In this software there is a there is a section where you can check whether the your degassing is complete or not, if this is a very important; degassing is a very important for the surface area analysis. Because if you take the incomplete degassing sample and put it into your analysis section then the actual you may not get the actual surface area. Because some of the pores may be blocked by some volatile matters or moisture where the actual adsorbate gas that is nitrogen may not get to it.

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So, after completion of degassing; now, here is the another section this section is called cold trap. Now, what is the function of this cold trap? While degassing say some moistures or volatile matters are coming out or maybe from the there are two pumps are there inside the inside this instrument; one is called dry vacuum pump and another one is called turbo molecular vacuum pump. From that side also if any oil or spillage or comes to this section then this will go directly to this cold trap.

This cold trap is set within a, at a temperature of liquid nitrogen temperatures at that critical very low temperature whatever organic matter or volatile matter comes or moisture comes, may comes through this section or from any other sections of the instrument that will be trapped inside this section and it will not go further to the analysis section. So, that it will not harm to the analysis section.

Now, after completion of degassing; so, while you are doing this degassing you should have to put the cold trap under liquid nitrogen temperature. Means, you have to take liquid nitrogen into this flask.

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Now, after completion of degassing that will be shown by your software whether it is completed or not, degassing is completed or not. Say for carbon it may 3 hours for some other materials it may take up to 5 hours or 6 hours, if it is the organic type of material suppose you have to measure the surface area of some organic matters.

So, you may have to degas the sample at very low temperature say 40 degree centigrade or 50 degree centigrade, you cannot go beyond 50 degree centigrade, because after that the morphology of that sample may for organic samples particularly that may change. So, for that type of low temperature the samples degassing, it may take a higher degassing time; say 12 hours or 14 hours that may take.

So, after degassing is completed as shown by the instance software now, you can cool down the, it will be automatically cool down to the when the degassing is completed it will automatically cut off and cool down, and cool down to room temperature.

So, degassing is done under very high vacuum and elevated temperature; high vacuum and elevated temperature and the motto of degassing is to evacuate the pores or the moistures or the volatile matters whatever within the sample present to clear the pores; so, that the adsorbate gas get into the sample, ok.

Now, after the completion of the degassing, we will take out this sample cell and it will be reweighed; it will be reweighed to get the actual weight of the sample that we have

taken. So, after degassing of the sample you can you put this to this analysis section, this analysis section in this analysis section you can see that there are three ports are there.

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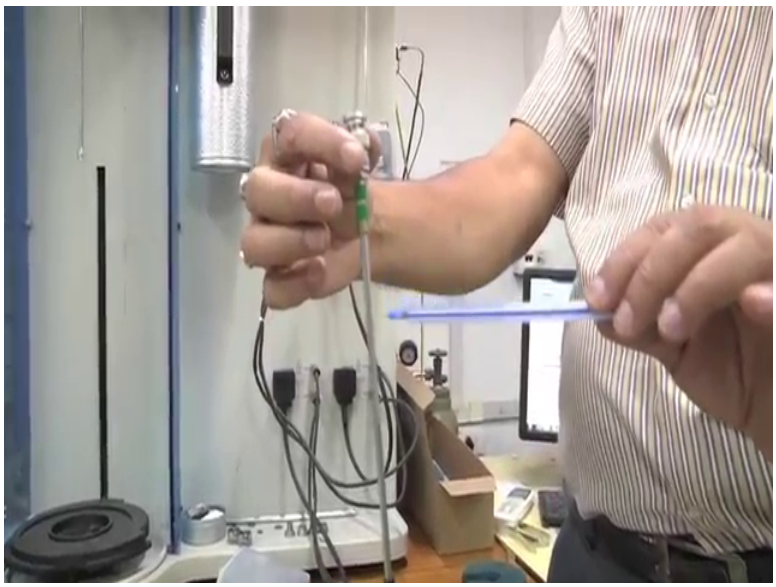

Number 1; this port, this port this is called liquid nitrogen sensor LN2 sensor this is called Liquid Nitrogen sensor. This will show you the level of liquid nitrogen in the bath.

Now, this tube this is almost like this sample 6 mm sample cell this tube, this is called P 0 tube. P 0 tube the function of the P 0 tube is to measure the this pressure around and

inside this year. You can use this P 0 tube otherwise you can put the atmospheric pressure at this place, where you are measuring say it is around 760 mm of Ag, ok.

Now, you have to put the sample after degassing into this port, the third port. Now, it is this is the complete section after degassing you have put the sample into samples to analysis station, this is called analysis station, ok.

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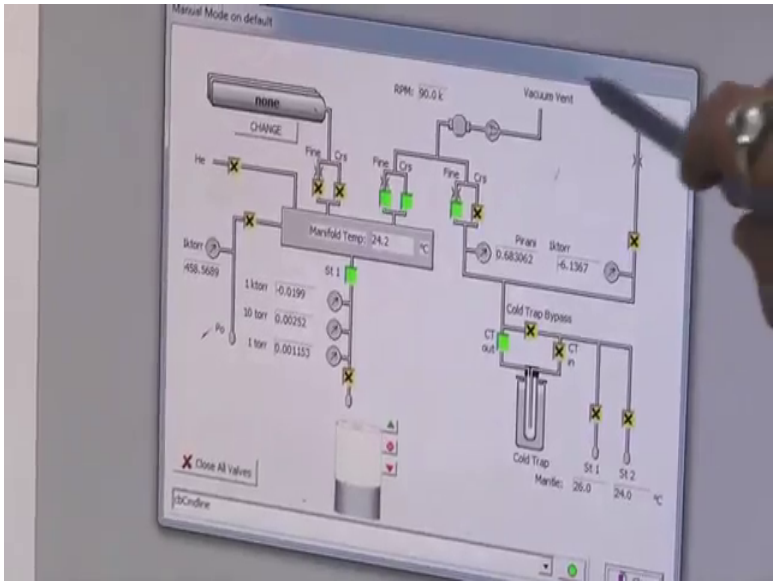

Now, see this particular, this section; this section is the degassing section means, this one this section this is the cold trap these are the two stations degassing stations 1 and 2 when that actually it measures the adsorption of the adsorbate gas. If it is nitrogen at various relative pressure means, P by P 0 that P 0 is constant here that is may be 760 mm of Ag and the P, P is the sample cell pressure.

It is varied, it is varied from say 10 to the power minus 3 torr 210 to the power minus 6 or minus 7 torr, it may go up to 10 to the power minus 7 torr. So, it varies and it comes down up to unity means, so, where P by P 0 is equal to 0.999 like this. So, now, how do this get analysis is once you degassing is completed now, you fit the sample into your sample station, after putting it into the sample station means, here. First of all this section will be clean or purged by a gas called purging gas; that is generally used as helium gas; helium gas is used as a purging gas to clear any other vapors or any other gases inside the cell or that thing.

After purging now, there are two ports are there for adsorbate gases inside. Generally, for BET analysis nitrogen gas is used as a adsorbate. Now, nitrogen gas is dosed at a measured dosed at various relative pressure means, P by P 0 from 10 to the power minus 7 to 10 to the power say unity means, 0.999 it varies relative pressure. Now, the what is the volume of nitrogen gas adsorbed onto the sample, onto the sample or desorbed from the sample that is actually taken and that is the from that volume we can calculate what is the BET or the surface area of that particular sample. Here generally for BET analysis nitrogen gas is adsorbed.

Now, this total analysis, this total analysis is carried out at a critical temperature of nitrogen that is 77 Kelvin. Now, how do this analysis is taken into that temperature for that this instrument or we are having Dewar, ok.

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This is this is called liquid nitrogen containing Dewar. Liquid nitrogen is filled into this Dewar and placed here during the analysis.

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Raise this Dewar will automatically raise and it will maintain the temperature of the sample cell at a temperature 77 Kelvin, ok. So, while taking liquid nitrogen into this Dewar you should have to be very causes, because it may cause you frost burn or cold burned.

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So, for that you can use some protective gloves like, gloves or your protective this gloves or your protective goggles, you should have to use. Now, this Dewar rises like this one and the liquid LN2 sensor liquid nitrogen sensor it will detect the level of the liquid

nitrogen inside the Dewar and where it will detect the level of the liquid nitrogen then it will stop.

And, as the time goes on the liquid nitrogen will evaporate. So, as it evaporates so, liquid nitrogen level will goes down then that Dewar will goes up slightly. So, for this type of Dewar it may take can be used for 90 hours.

So, this is an experiment on flow through packed bed.

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Here what we see is this is this red colored cylindrical object that you see this is the packed bed inside we have packed medium.

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So, the flow takes place from that side r or rather flow takes place through this end, flow comes that there is an underground pipe from which the flow comes through this white line. And then flow comes through this white line and continues, and then enters into this packed bed; this red colored object that you see and flow takes place through this packed bed. And, then it goes out through that white colored line out from there and then out from the pipe can you start the flow from the that side.

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So, now, you can see that the water is flowing through this packed bed and is connected at the effluent end. So, the flow is taking place from this side towards that side and then flow is continuing and flow is collected from the outlet.

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Now, at the same time we are making two types of measurements; one is the flow that is collected from that end that can be measured in a graduated measuring cylinder or in a beaker and that can be weighed, and that weight is collected at regular intervals and, the interval is calculated or estimated from a by using a stopwatch.

So, one can find out what is the flow rate? That is the flow rate of water that is coming out from the outlet. And at the same time we have a manometer, you can see one manometer port on this side and another manometer port on that side of the porous medium. So, the pressure difference between these two ports these are measured using these manometer; you can see one side of the manometer is the manometer the mercury level is higher and this side the mercury level is lower.

So, what that means, is pressure on this limb is higher and the pressure on this limb is lower. So, this side this pressure is higher, because this is connected to the upstream side and this other one is connected to the downstream side. These pressure difference; that means, these h the delta h into density of mercury which is 13.6 gram per cc multiplied by acceleration due to gravity, that gives me the pressure drop. So, that pressure drop is actually occurring between these two ports across this porous medium.

So, this delta P is monitored using the manometer, flow rate is measured from whatever liquid flow we get from the outlet and then these two measurements are complete, these two measurements are taken to compute what is the permeability of this porous medium? One can use Darcy's law; one can use other types of equations that are available for flow through porous media to arrive at the flow resistance offered by the porous medium inside this red tube.

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This is an experiment on fluidized bed. As you can see here we have these packing which will this is a packed bed and then there is a liquid column above it. So, what we are going to have is flow from below that there will be flow and then gradually we will see that as the flow continues as the velocity is increased these particles this packed bed will get suspended and this bed height will continue to rise.

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We have a Rota meter on the right side to measure the flow rate of water you can see this float movement of the float there. And then on the right side we have a manometer so, as the as the flow takes place we can measure the pressure drop over a height of this bed.

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Now, can we start this flow now to see how these, how the bed height grows as the flow of liquid continues. As you can see in this picture the flow has started already and the bed height continues to grow. So, you can see the flow out on the right hand side the

Rota meter the float has already moved. So, that gives the flow rate and on the right side there you have manometer which gives the pressure drop across this bed.

So, here the bed is in fluidized condition. So, this is not a packed bed here the superficial velocity has increased beyond the incipient fluidization velocity, so, the fluidization has started, that is all.

Thank you very much.