Course on Analytical Chemistry
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Module No 02
Lecture 10: Crucibles, Filter Papers and their Uses

Hello, welcome to this class where we were discussing in the previous one that how we can use the different crucibles.

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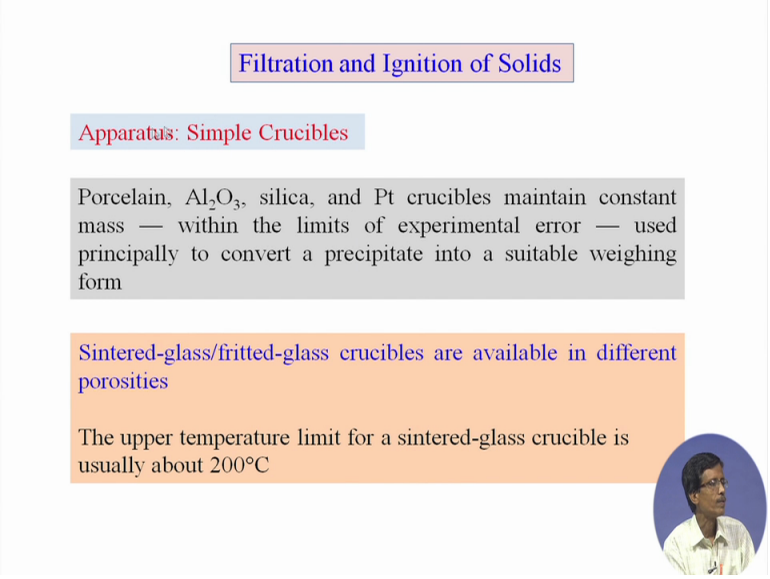

So there we had seen that we can have different types, that means porcelain crucible, the aluminium crucible, silica Crucible and platinum crucible. And we were discussing about the precipitate ferric hydroxide. So for ferric hydroxide, this is all very simple one, the porcelain and within this porcelain crucible, we have to transfer the precipitated ferric hydroxide. So what we can do?

We can do the filtration on filter paper and how to transfer that filter paper within this porcelain crucible? That is the question. And we should see that there are other varieties of those crucibles. So for these if we consider that you should know little bit about the different crucibles, we should know where we use water. So this as I discussed and gave you the example, the sintered glass or the fritted glass where you have the bottom and the in within that particular bottom, you have the corresponding Ggass frit is attached.

So this, if this glass frit is attached, we can consider this as a sintered glass and use of those crucibles, you do not require the use of filter paper for filtration. So that is why, for nickel DNG filtration, we do not use filter paper and then basically you see that it can withstand a good temperature up to 200 degrees centigrade.

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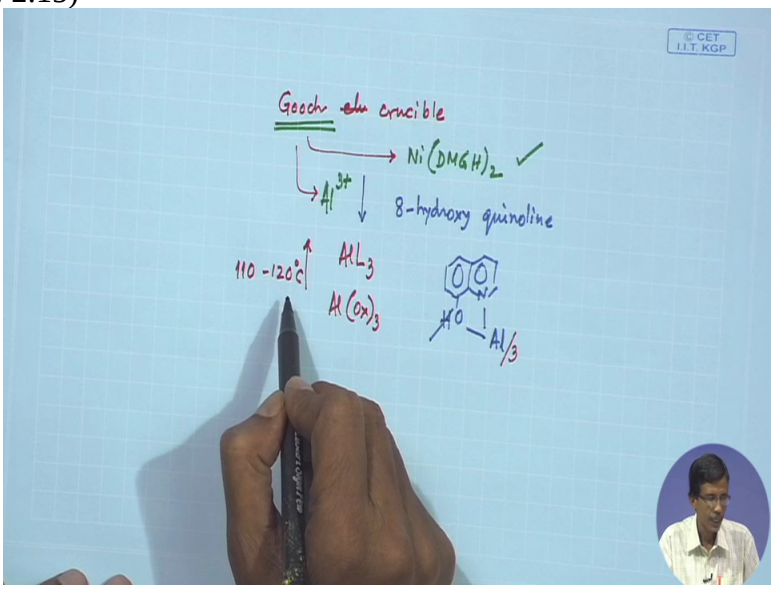

So this glass Crucible or the centred Crucible of the sintered glass Crucible, so which is known as the Gooch Crucible where received that name after Gooch, is somebody's name, scientist's name. So we have NiDNGH2 and we have seen that you can use this at a particular temperature. And this particular gravimetric technique, we will discuss in detail afterwards. But for the time being, you see, it can be precipitated out when aluminium is there in your unknown solution.

So it is the 8 hydroxy quinonine 8 hydroxy quinonine which is again yours is a chelating ligand. So this ligand can bind to this particular aluminium centre so this will go out and 3 of these can form, so like your NIL2 this particular case my your aluminium will form ALL3. And again you can dry it out and use this particular one as the weight of the aluminium oxinate , tris aluminium oxinate and that can be useful to filter through Gooch Crucible and dry at a particular temperature of say 100 and 120 degree because at that particular temperature, your entire moisture trapped in this particular aluminium oxinate is is very nice to write in this fashion that is oxinate is the abbreviation for the oxinate ion.

So this again at 110 4to 120 degrees centigrade is fine for your drying. So you get two examples for use of Gooch Crucible for gravimetry. So for aluminium estimation and for the nickel estimation but this is the normal procedure because you are only dying at 110 and 120 degrees but that is not your ignition gravimetry or ignition use of crucible for the ignition purpose. So this particular sintered glass Crucible, what is very useful up to 200 degrees centigrade, so when we use this drying for this nickel DNG or aluminium oxinate, we use it till 120 degree but it for some other cases, even we can have some other solvent also.

Suppose the medium is not your water medium only, so if we try to heat that at a very high temperature in this particular range, that means 150 degrees centigrade or 160 degrees centigrade, we can use that particular sintered glass Crucible for this particular purpose.

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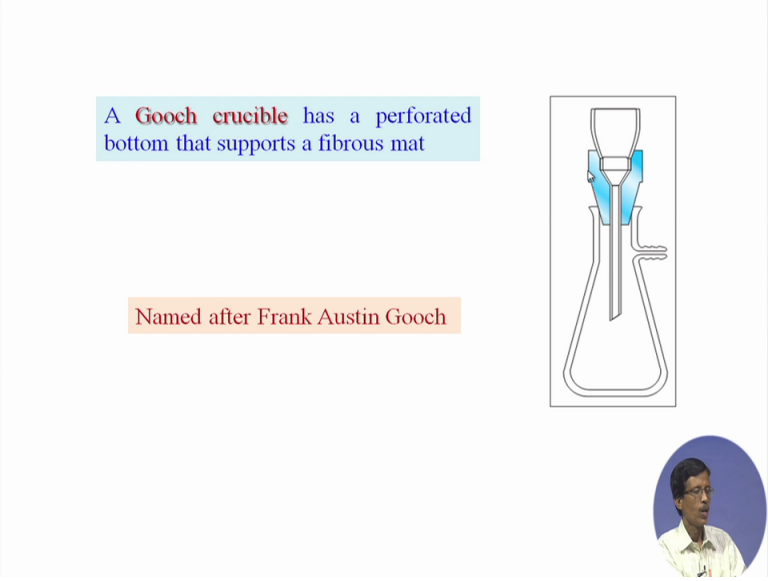

So it has a perforated button as I told you and support on a fibrous mat because you require a fibrous mat, that means the porous mat on it which can pass the corresponding liquid and how to use this as the corresponding device for filtration. So we all have some good idea about the corresponding process of using funnel and filter paper, again we just recall back, how to fold the filter paper and how to put in the funnel because everytime we use this and it has some relationship with the filtration of ferric hydroxide.

So as I told you that particular Crucible which is there and that Crucible as it named after Frank Austin Gooch. So this Gooch Crucible so this is nothing but your filtering flask, this the pipe adapter, the rubber tubing adaptor is attached to it this particular point. And it can be used with water suction in the water tap or it can be used to some pump. So such that you can have suction unit, so this is the corresponding base and you have the adaptor, so this is the adaptor.

So on top of it, you can put this Gooch Crucible over it and you transfer the precipitate on it and you just keep on filtering over it. So this is the arrangement. So this sort of arrangement you should all know that what are the things basically you require for filtration using Gooch Crucible which is different for the filtration using your corresponding filter paper.

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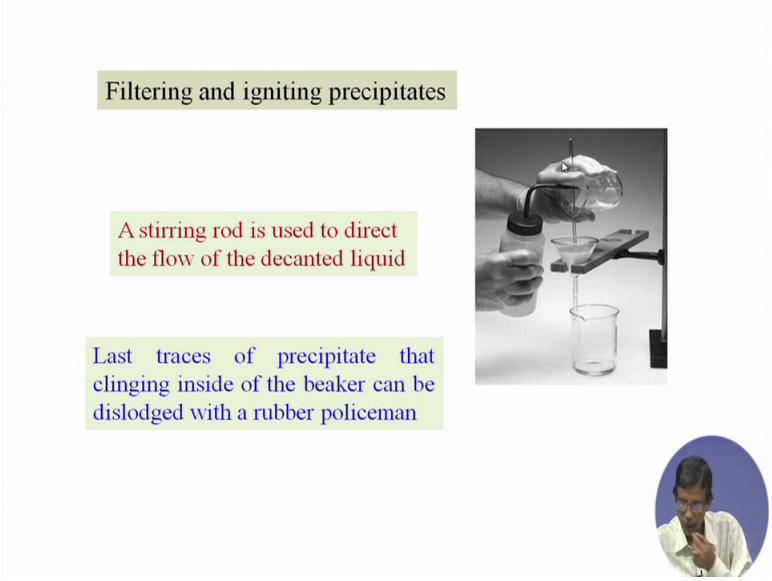

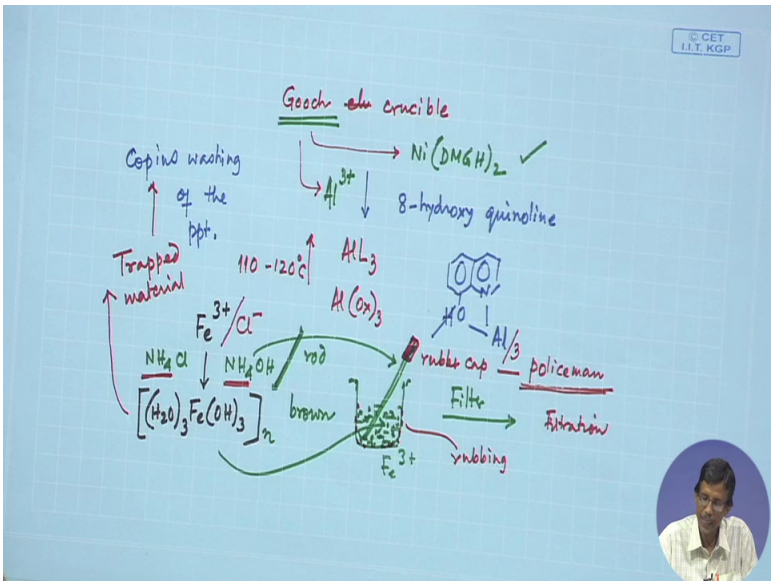

So when we go for this filtering and ignition of the precipitating what we just now see that filtration we can do and you have to burn or ignite that particular filter to give the corresponding FE2O3 from ferric hydroxide, hydrated ferric hydroxide. So in this particular beaker, what you do? You just go for the corresponding reagent. So is this vehicle, what you do? You have FE3 + and this FE3 + is precipitated as a ferric hydroxide.

So FE3 + was there which has been precipitated as ferric hydroxide. As I told you, it is wise to write this in this form. So this is your form of ferric hydroxide and it is basically a three-dimensional structure, is a polymeric network because these hydroxide groups are bleaching more than one iron Centre, this is not a monomorphic structure. So this yellow brown floppy material so which is voluminous also, you get it in a beaker.

So you have the beaker in your hands. So you have the precipitate of this in it and you have the aqueous solution over it. Now how to filter it? You have to filter it. So once you, so initially what you do? Initially you transfer the solid. So when most of the solid has been transferred, this is basically last step, so we are just giving the example because in all these cases, one important thing what we use all the time when we consider in many of our class, the bumping thing, what is called bumping when we go for evaporation of the liquid from a corresponding beaker?

So beaker is in your hand and you go for the corresponding evaporation, putting one glass rod inside it. Similarly, this glass rod is useful for transferring the reagent. How? When we use solid

ammonium chloride, because you can use solid ammonium chloride, but ammonium hydroxide what you can use which is a liquid.

So drop by drop addition of ammonium hydroxide can be done through the side of the stirring rod so which is your rod, stirring rod inside the beaker because this beaker always we see that you can have the corresponding stirring rod in it and you transfer this particular one drop by drop within the ferric solution FE3 + and you see the corresponding lump formation, that means the precipitate formation within this particular beaker.

So initially the whole bulk is transferred over the funnel fitted the filter paper. We will see how we can fold the filter paper also. And this is the original glass rod we have used for transferring the reagent. So at the end when most of the bulk of the solid compound of ferric hydroxide is being transferred to this particular filter report, you have to wash or you have to clean this particular beaker which is having the ferric hydroxide because whatever ferric hydroxide precipitate you have, you have to quantitatively transfer the entire precipitating on this funnel.

So this particular transfer will take place on this funnel. So then we use this as the waterjet we call, we consider as the wash bottle. In our schooldays we usually used to make this particular class wash bottle from the glass bottles also with waterjet but this is a plastic bottle. When you place it, the waterjet is coming. So waterjet is pointing towards this particular side and it is basically cleaning all the beaker.

Basically is a cleaning, washing and transferring process where he last trace of ferric hydroxide if it is added to the side of the beaker, can be transferred to this particular filter paper, on the filter paper okay? So this particular stirring rod, it has functioned during the filtration also because these are the typical techniques. Most of the time, we do not know and we are unable to follow it. If you do not follow it nicely, the errors will come, your results will not be very good.

So everytime you have to take the nice precaution, you have to take the precautions. So these techniques, not only handling this apparatus but your technique must be very much useful for this particular process. So this technique is very unique one. So you have to put this jet and you transfer the last precipitate or the last part of the precipitate to this particular filter paper. So the stirring rod is used to direct the flow of the decanted liquid initially.

And then at the end when you go for this waterjet, the waterjet is then there and you see this water will come through this rod. So by the side of this rod, your water will come and again fall on this filter paper such that this water is also washing your ferric hydroxide because this ferric hydroxide would just know we have seen that this ferric hydroxide whatever we were using, your ammonium ion is there. This is also ammonium ion.

So it can have some trapped material. What is that? You can have ammonium ion, you can have the chloride ion because if you have you are handling only that solution taken from the ferric chloride, you can have the chloride ions. You can have the excess hydroxide ion which has not been used for the precipitate. So all these anions base you have to wash it out. So this trapped material can be removed by copious washing of the precipitate.

So when you transfer this by this waterjet, you are at the same time washing your precipitate and your washing liquid, that means what is passing through this precipitate is coming to this particular beaker, this is your filtrate and your residue will be on this filter paper. So the last trace, that is why I was just telling you now the last trace of precipitate that is adhering or attaching to your beaker, this beaker because this is the beaker where you precipitated your ferric hydroxide can be dislodged with a rubber police man.

So this is another use of this thing because this is not a very simple glass rod sometimes because if you have this glass rod and if this glass rod when we go for the precipitation you have a glass rubber tubing over it, so basically is like a cap of of a police man. So this is a rubber cap which is your corresponding police man.

So during the filtration, you take this side down and you have rubber cap on the top but during filtration, at the end basically, towards the end of the filtration, you take this rubber cap inside this and whatever ferric hydroxide attached or adhered to this beaker, you just love it.

So roubing this police man with the rubber cap is required for rubbing the attached ferric hydroxide or lodging this ferric hydroxide which is basically attached on the beaker surface because only glass surface is not very much useful to remove all of them because if you rub it with the only this end of this glass rod, we will find that some amount of yellow stain is still there on the beaker surface.

So you use this police man which is a nice technique because the rubber tubing, you put the rubber tubing and the glass rod inside, you make the police man yourself. So that is police man is basically utilised for this particular attached ferric hydroxide from the beaker. So you should know that what is police man, how you can make it and how we use this for removing the last trace of ferric hydroxide from the beaker. So dislodge the last trace of the ferric hydroxide from there.

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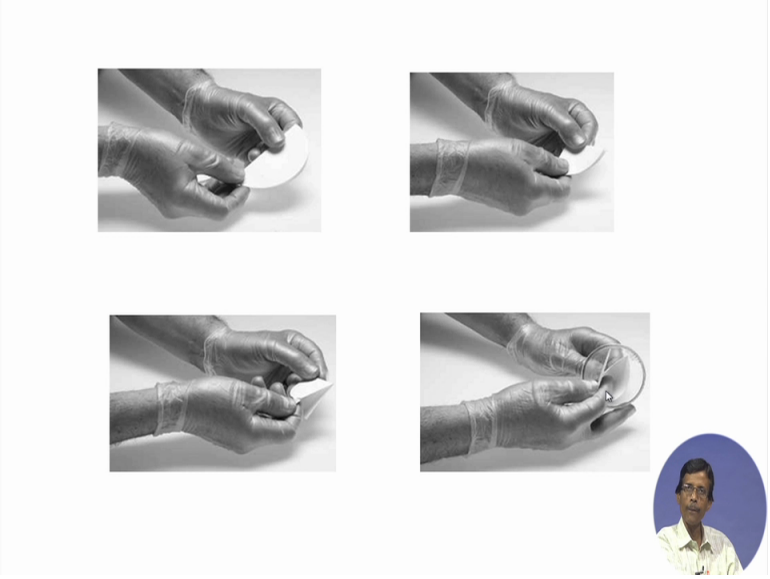

Now we see how we use the corresponding folding of the filter paper, this you all know from your schooldays but again we just recapitulation, you recall back how you quickly make yes this is one type of folding of the filter paper, is not the fluted filter paper. That is another one where we recall from the 1st filtration because you can have different type of filter paper because the pore sizes are different depending upon the particle size what you are filtering.

If you are filtering ferric hydroxide, you require one particular pore size and if you filter calcium oxalate, you require a different pore size. So there is a thread numbering for all these, so and company name is Wortman and Wortman filter paper 41, 40 and 42. So these are there. And these porosities are comparable to that of our good crucible porosities. That we will discuss in detail when we talk about that gravimetry related to this particular crucibles and the filter paper.

So this is the technique for the technique, so this is the 1st fold of the circular filter paper, is a circular strip of filter paper. This is the 1st fold when you make half half of it and this is the 2nd fold. So 2nd fold is making you some angular one. So when you open up three fourth of this on one side and one fourth on the other side, you just basically open up this particular one. And this opening up will give you this particular thing, that means three fourth of this folded part is one side and one on the other side such that it will nicely fit inside the funnel.

What else we can do? You are filter paper is dry, you can put some amount of water in it and you allow the water to drain out from the funnel as well as the filter paper such that you filter paper can fit nicely on the funnel side, on the side of the funnel such that there is no gap, air gap by the side of this filter paper. Otherwise, your rate of filtration will be affected because if air is air passed, if air we can pass through the side of this filter paper, your rate of filtration will be less and it is very difficult to filter it out within a timeframe of any quantitative laboratory experiment because most of the time that is why we can have not a three-hour lab class, can have a 6 hour lab class because this basically takes a lot of time for your filtration process.

But care should be taken, you must be careful to fit the filter paper nicely within this funnel such as that your rate of filtration will be enhanced because when you put because initially we will not transfer the entire ferric hydroxide, the water will pass 1st. Then slowly slowly, we put the entire ferric hydroxide ones, the whole ferric hydroxide is transferred to this filter paper. Your filter paper pores will be further clogged which will also decrease again your rate of filtration.

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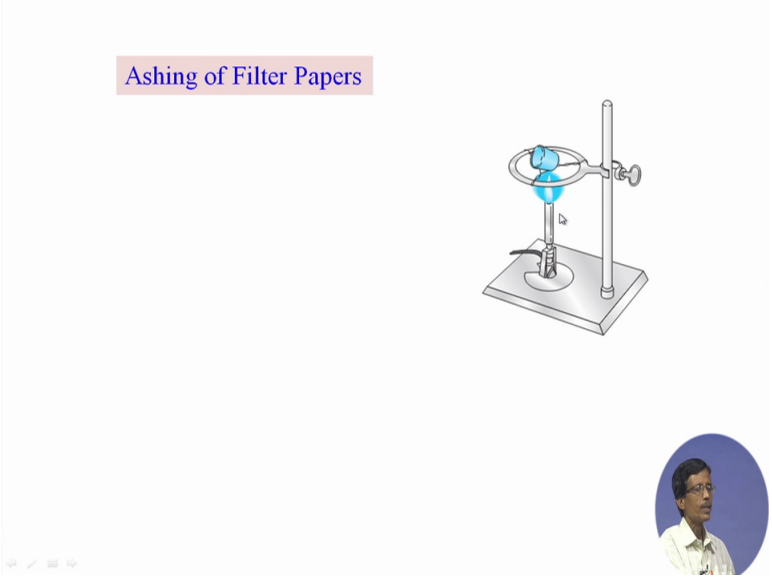

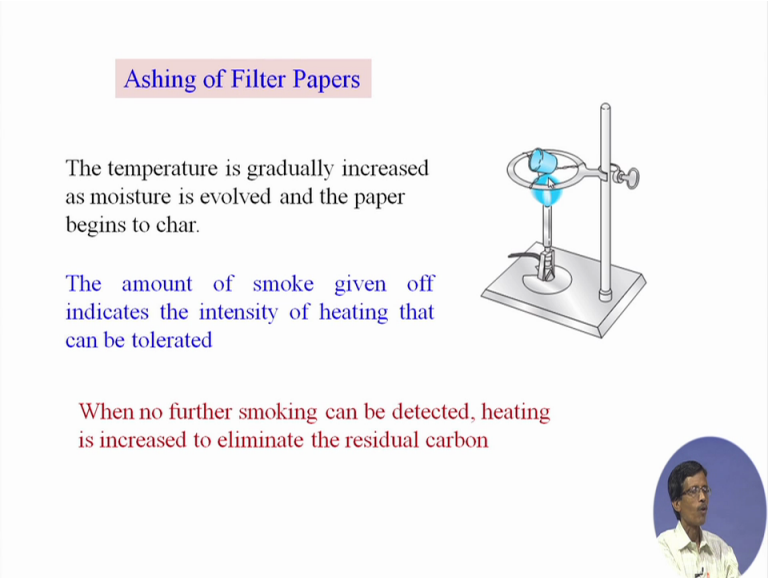

So this is all about this particular filter paper. Now you how you go for ashing of the filter papers? Because we were talking about the conversion of your ferric hydroxide to FE2O3 which is there on the filter paper, so your conical shaped filter paper which is there in the funnel, so this particular mass, mass of ferric hydroxide is on the filter paper. So how to get that? So this is this is mass of your so-called FEOH whole 3 hydroxide, the Brown ferric hydroxide.

And we want to go for this conversion through heat. So everything we have to transfer now because this ferric hydroxide along this paper, we have to transfer to one of your porcelain

crucible. So now you think for a while that what are the things are there because within the porcelain crucible, if this is your crucible, you have to particular, you have to have this particular filter paper which is basically the water is also there.

So its weight, so is weight filter paper along with your ferric hydroxide. So you just fold it. So this filter paper, you fold it and you cover it with this open face and you this folded form, so this folded form basically in this form, so you transfer this within this particular porcelain crucible. So now if you put burner on it, you have to burn everything. So what initially you have to remove this corresponding water what is present in the filtrate that means some of them has trapped in the ferric hydroxide also.

So what we see that this trapped water molecules will go. So during 100 and 120 degrees centigrade of heating, so your water vapour and water moisture will go. Then this, you get the dry filter paper and slowly the water vapour will also come from inside of this, that means the trapped water molecules inside the ferric hydroxide bulk. So that will also go and that is basically then drying up.

Now thing is that your conversion of ferric hydroxide to FE2O3 will take at a high temperature and that particular high temperature also what you see that you can have also the chance of burning this particular carbonious matter of the filter paper. So that means you have to burn the filter paper. So we will completely burn the filter paper. That is why it is known as ignition gravimetry or the corresponding corresponding burning process for the filter paper.

Initially the filter paper will be charred and the black mass will come and slowly you have to burn this carbon. So you must have the patience of burning this particular process. That means the whole carbon particles whatever carbon as ash is forming due to the burning of this filter paper should be removed because carbon can also contribute the weight of these because in the final stage, what we are looking for, we will be only looking for the weight of the pure FE2O3.

Nothing should be there, no trapped water, no other extraneous matter or no carbon particle from the filter paper. So that will also take some time, several hours, sometime also to burn this and to get this as the corresponding FE2O3 from the ferric ion what we are handling for the estimation of iron. So by knowing the weight of these, we can estimate the amount of FE3 + originally

present in it. So this is the technique what we can do that you have to ash the filter paper 1st before ashing your precipitate.

So this is the technique, basically it is a common textbook thing, anywhere you can get it. This has been taken from your school's book. So this is the burner and how you put it. So you see, this is that corresponding we call, it can be clamped over here. And clamping of this ring and then we have another triangle where triangle is there when some 3 portion support is there, so this basically how you get, so you have this some ware thing.

So this is the ware and this ware is basically some porcelain tubes we put because you have to withstand a high temperature. So this is the triangle. So you have this one, you basically get the support of that ring. So this this angle is basically on the ring. So you have the ring, so basically this goes like this. So this is basically the support system because we are going to burn it to a very high temperature. So this burning process, so this is a made where where this is made up of iron.

So this iron ring is there. So with this ring, you have this triangle, so this is the porcelain one where you have the ware inside hand that you put that and over that, because you have the typical size of this Crucible, and crucible along with your filter paper, you put inside and heat it with the burner. So this is some this is some in the tilted form but initially you can put as a vertical position and for the vertical position, you heat it up, you will be drying slowly and then you can char the filter paper and then you must have a patience for that because it will take some time and sometimes you hold the burner and you put the (())(26:21) from all other sides of this particular crucible.

So the temperature is gradually increased as I told you, so if the moisture is evolved and the paper begins to char, so charring will take place slowly as the temperature is increasing and the amount of smoke given of indicates the intensity of heating that can be tolerated by the system. Because if you heat it very quickly, the filter paper can catch fire. So we should avoid the catching of that particular filter paper.

So you should see that smokes will only come and that particular smoke whatever you get it due to the corresponding burning of that filter paper. So when no further smoke is there, so is we

cannot you do not see that smoke is there, heating is now intensified is this such that you can have the corresponding removal of the residual carbon which is coming from the filter paper because the filter paper has already charred and a black mass is already there with ashes and slowly slowly you heat just increase the heating process and thoughts that you are all the carbon of the filter paper will be converted to carbon dioxide.

So is open air heating so the upper side is open. So the heating process is such that your thing can go for or the entire filter paper will be removed from there as carbon dioxide living behind the corresponding formation of FE2O3 which is slowly forming from your ferric hydroxide.

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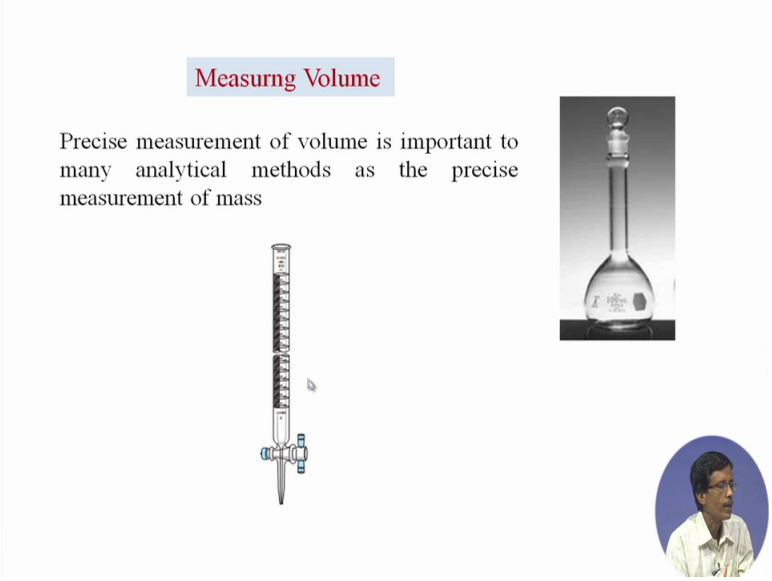

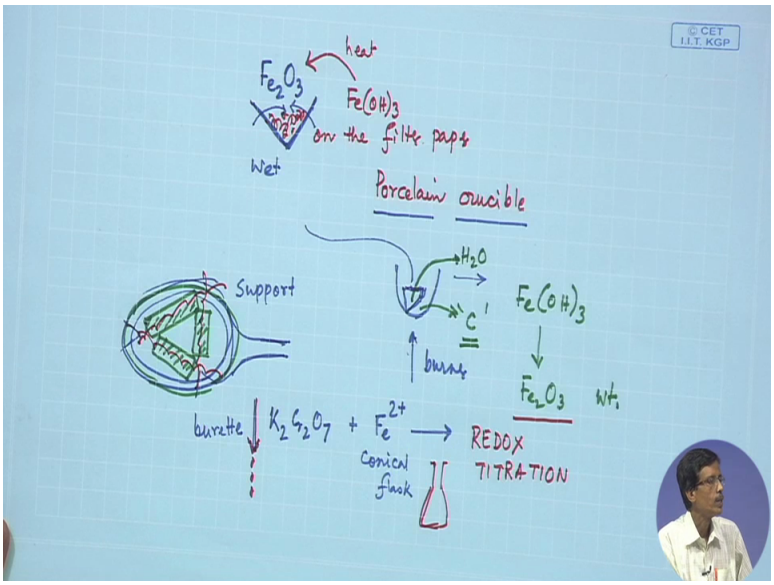

So the next one what we can see quickly that how you go for measuring the volume that already I told you that direct weighing using the weighing bottle to that particular form this is the example of your corresponding volumetric flask and you open up the volumetric flask and from the weighing bottle, you can directly transfer the corresponding sample inside it and there is a mark of some volume. Suppose this is a 250 millilitres volumetric flask and once you transfer the solid, and you then transfer the water from the water jet of the wash bottle, then you slowly dissolve the material.

It can be or oxalic acid or sodium oxalate or potassium dichromate and once the when the flask is half filled, and when all the solid has been dissolved, then only you try to put extra water volume to make the volume, to make up the volume. So when this is there, so you tilt the whole volumetric flask upside down and shake it and such that you have the corresponding uniform solution of this particular potassium dichromate.

So once you do it, you know the weight how much you can take for making a N by 10 solution of potassium dichromate, we require 1.2258 gram of that particular solid sample which can be transferred. So nearby that weight from the weighing bottle or the direct weight, you get one typical factor for that and you know the exact strength. So when we get that, so the volume is known by the mark on this particular bottle.

So precise measurement of volume which is very much important for our analytical technique for all the different analytical method because the precise measurement of mass because whatever we are doing, we should know the amount of mass by knowing the volume of this. So when we make a standard solution of potassium dichromate, we also know the amount of chromium present in it. So it can be a standard solution for your chromium estimation for any other experiment.

So how judiciously you can use that, that means the volume of this as your chromium concentration from this volumetric flask, so this can be used or some redox titration. We, using this particular potassium dichromate for titration of FE2 + solution. So redox titration we will discuss in detail afterwards that you can have your redox titration where the FE2 + can be oxidised by the use of your this potassium dichromate, the K2Cr2O7 the standard technique you know from your school days and this is the thing, that means what you require?

You require the burette. So this 2 things, that means the volumetric flask and the burette, both of them are used for your volume measurement and this basically gives you the corresponding solution of potassium potassium dichromate K2Cr2O7 and that K2Cr2O7 we transfer, we take in this particular burette and this particular burette, that means how much you require that means this is your oxidising agent. So next day what we will see that how you go for this titration of K2Cr2O7 for the titration of any unknown solution of FE2 +.

So this we will do, so this we will take in burette, this we will take in conical flask. So we should have some clear idea how we can do this because this will give you something, that means we from the burette, we drop by drop we had this particular solution to this iron solution taken in the conical flask. So this is the apparatus, this is the solution for what? For your redox titration.

So next day we will see how this measurement of these volumes are important because volumes are directly proportional to the mass of that particular solid what you have taken in this volumetric flask as well as in the burette and how we can go for the corresponding volumetric analysis by redox titration. Okay, thank you very much.