Course on Analytical Chemistry
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Module No 02
Lecture 09: Filtration, Ignition

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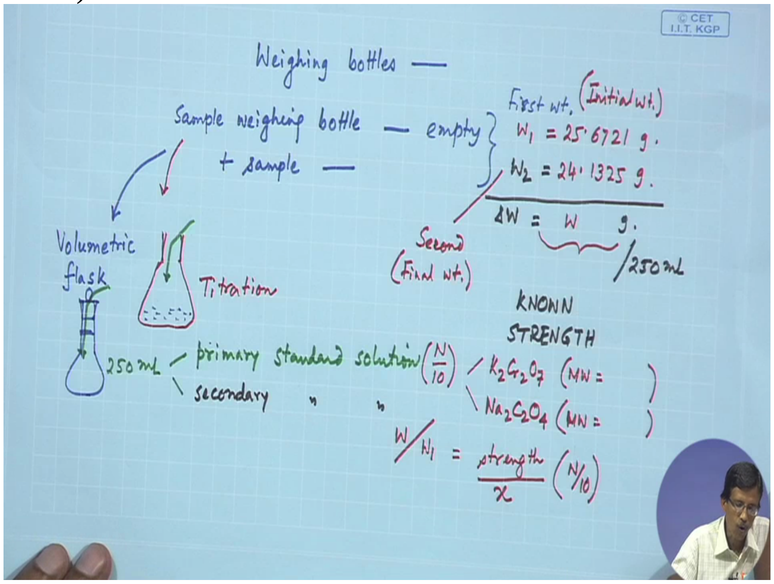

Hello, welcome to this class where we are so far talking about how we can handle the weighing bottles to measure some quantity of the sample to be analysed. So one such technique what we can consider is weighing by difference. So what does it mean basically we will see in this particular class that we have a sample weighing bottle which can be empty okay? Then becomes not sample in it. Therefore we get sample + weighing bottle.

So if we take the initial weight of that, that means W1, what is that? W1 will be the sample + the weighing bottle, the weight of the Sample + weighing bottle. So it is a certain amount of grams, say 25.6721 grams say. Then what we do? We just basically without introducing anything in between, we want to transfer that particular Sample directly to the reaction vessel or their conical flask where we can go for titration when we go for titrametric analysis or we can use that directly for the solution preparation in volumetric flask.

So the problem is, how we can directly transfer this particular Sample to this reaction flask, the Allen wire flask or the conical flask or the corresponding volumetric flask. So it has the marking over here and we all know that it has the stopper also. So without using anything, any filter paper, any watch glass or any other board, aluminium foil board or anything, we directly want to transfer sample from this sample bottle + sample to it.

Then this is the 1st weight. That means, it is the 1st weight of your sample + weighing bottle. Then we slowly transfer some amount in this conical flask or in this volumetric flask to make a volume of say 250 millilitres for making solution. So you all know that we can make primary standard solution or sometimes, secondary standard solution. Once again they will discuss all these in detail when we go into the corresponding chapters where we can discuss about the volumetric analysis or the titrametric analysis of any unknown sample where the analyte can be your mineral or analyte can be metal ion or analyte can be something else.

So what you can see from your is that you have W1, then you get W2 because the difference between these 2 will give you the W, Delta W which is actually being transferred to all these flask. That means weight we should know nicely. Then when we make or measure the volume this up to 250 millilitres, we can say that this much amount of of solute or analyte has been transferred to these containers to make a solution of known strength because this will be of known of known strength.

So after this transfer, what will happen? You have the weight which is less. So say it is 24.1325 grams. So the 1st weight and this can be your 2nd weight or sometime we call it as initial weight and this as the final weight. So this amount of that sample which is W, we know this particular one and that should be very close to the sample what we will be requiring to get some solution when we make some primary standard solutions we all know that we have some idea to make a deci normal solution and by 10 strength.

So to make a deci normal solution, how much weight of these samples a supposed that to concrete examples we can take that weight of say K2CR2O7 or disodium salt of oxalic acid. To make a standard solution of these because this is the primary standard sample, this is also a primary standard sample, depending upon their molecular weight, we know that Hamas is their corresponding equivalent weight and based on that equivalent weight, we can transfer the required amount of that particular sample.

So when we get these, suppose this particular so disodium salt or oxalic acid dihydrate, you know this W and the amount required, that means W1 which should be very close to this W1. So weight taken W by W1 will be your strength of the solution what we are going to make. So if it is in the range of this N by 10, so there will be a factor, we call it as some factor X into N by 10. So that is a very simple technique where we go for the sample preparation or solution preparation by difference.

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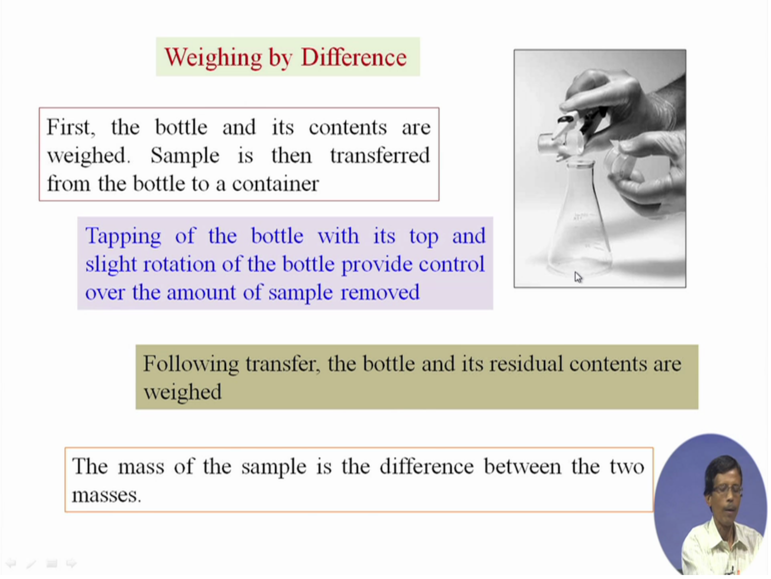


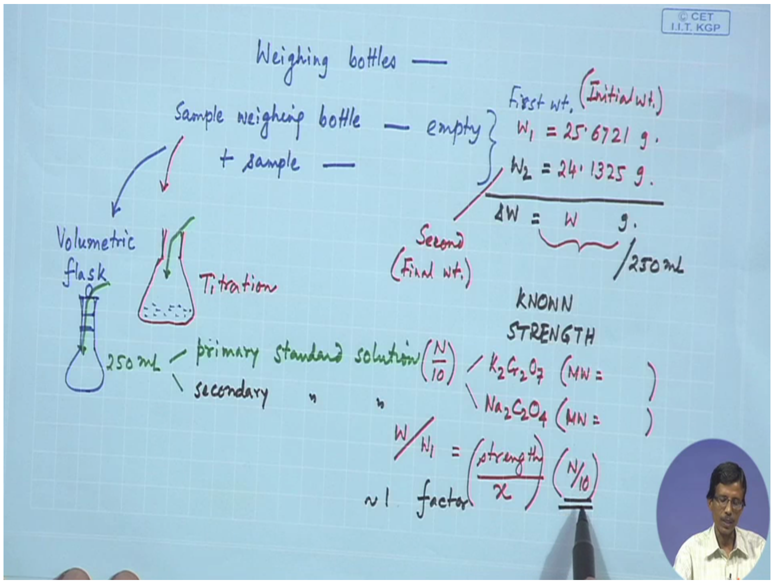
Weighing by Difference

First, the bottle and its contents are weighed. Sample is then transferred from the bottle to a container

Tapping of the bottle with its top and slight rotation of the bottle provide control over the amount of sample removed

Following transfer, the bottle and its residual contents are weighed

The mass of the sample is the difference between the two masses.



So this difference basically tells us something where we get that 1st the bottle and its contents what just now I told you, so this is in the liquid form so that you can good nice note of it, so Sample is then transferred from the bottle to the container. So after taking the weight of the sample with that of your weighing bottle, you transfer some amount to those flasks. So this is the typical technique, so this is taken again from the book of scoogs. So Scoogs book is the photograph is there.

How we can transfer? We have the glove protected hands and we have some faucet and this is the sample which is given to you in a weighing bottle. So in a previous plasma what we have seen that how we use the weighing bottle and after that particular weight, that means after the initial weight, that means the Sample and the sample vial or the weighing bottle, you transfer this directly to this Alden Meyer flask.

So this you do 1st, then how to transfer this? So this particular terminology because we have some terminology which is associated for any other technique of this class. We find that capping of the bottle with its top and slight rotation of the bottle, how to control of transferring the solid powder matter. If it is your potassium dichromate or oxalic acid or sodium oxalate, what we see that slowly we can transfer to this particular sample container and where we know that if we want to say 1.2258 gram of transferring the sample, slowly we should transfer these.

Otherwise, the whole lamp will come out of the weighing bottle and will fall on this conical flask. So control over the amount of sample to be removed. So it is with practice you can do it because in the very 1st time, you cannot do that but you have to do that direct transfer of the sample from the weighing bottle to the conical flask. So then we see, after this, the bottle and the residual contents are weighed.

That means what I just now told you that this is your 2nd or final weight. You have the initial weight., That is your 1st weight and after transfer, you have the 2nd weight. Then the mass of the sample is your Delta W. So mass of the sample is the difference between the 2 masses. So these 2 masses, that means W between the 1st weight and the 2nd weight is the actual amount of the solid sample what you can transfer to this particular conical flask.

So in the same way, if your sample is not a solid one, if it is a liquid one, liquid which can be nonvolatile also because otherwise, it can volatilise in air, so that can also be transferred by this particular method but only thing is that how to dispense that in a drop oil fashion. But it is most convenient way of doing is that you can transfer everything from the solid and it should be very fine in powdery form because the grain size should be less.

Otherwise, a big grain can change your weight difference much and you your factor would be above 1 because what we are looking for here, in this particular factor what we are getting over here, this we can consider as factor, it should be close to 1. So in by mistake, you take less amount, you can control your transfer and such that you can reach to do this particular value of 1. Otherwise you can have a factor of 1.1, 1.12, 1.15 or 1.2 such that.

So because what we are looking for because when we will be titrating that, this particular thing, your Sample strength, that means the analyte and the titer should have a very similar strength. Suppose we want to analyse something, that means your sample concentration within this conical flask is in the decimolar uhh deci normal solution so your reagent concentration or the titer concentration should also be of the deci normal strength.

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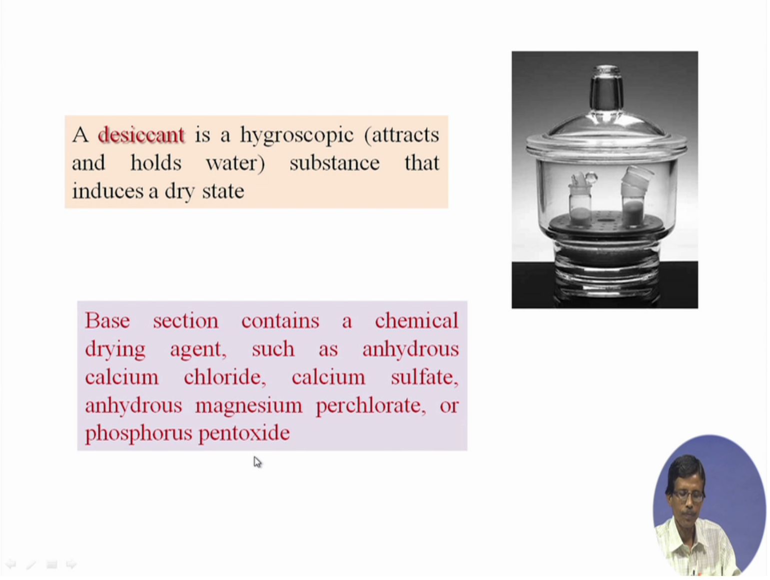
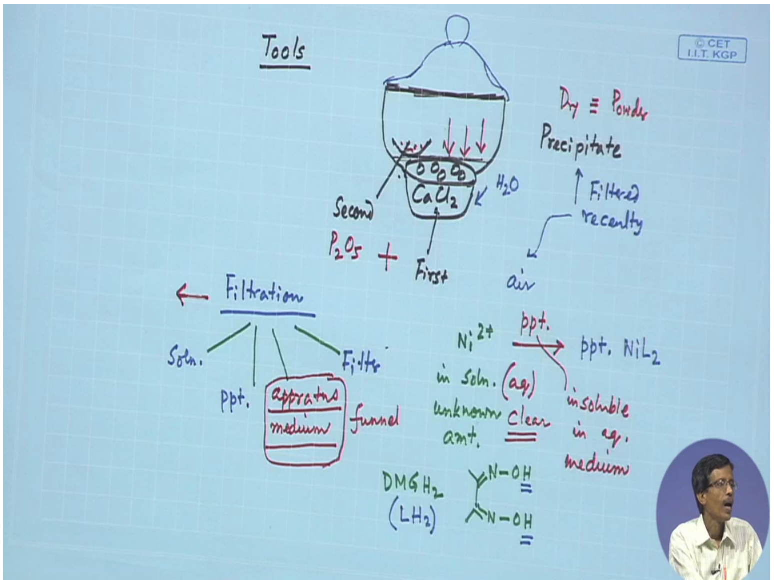


A desiccant is a hygroscopic (attracts and holds water) substance that induces a dry state

Base section contains a chemical drying agent, such as anhydrous calcium chloride, calcium sulfate, anhydrous magnesium perchlorate, or phosphorus pentoxide



So this weight by difference once we do, we can also use something because this we are discussing about the different tools and where we see that for the different tools, how we basically make the solution. We have the volumetric flask and we have the conical flask but if we have a desiccator, so that we are basically looking for, that how to keep your sample inside a desiccator. That means within a dry atmosphere.

So these we are seeing from our previous class, these are a weighing bottle, so weighing bottle we have seen that we can have 2 types of weighing bottles, one is capped from above and another is a normal lid. So this is a solid sample, you consider that you can have potassium dichromate within it and another one is oxalic acid and you will see that this cover has been opened, this cover is also opened up because your inside of this weighing bottle should be in the environment of this desiccator.

So this particular container we call it as a desiccator where we put some desiccant also that this particular environment within this particular and it is dry and moisture free. So we have the lid. This is the lid which which we can take out and on the rim basically, this is the ground glass joining, these all are ground glass joining and it is basically nicely greased such that no air can air and moisture can go inside.

So when we are not performing the weighing operation, what you can do? You can do this sample, you can put this sample inside the desiccator such that your potassium dichromate or oxalic acid can be kept in a dry condition because otherwise we can we know something depending on the environment, if there is a high moisture content in the atmosphere, the relative humidity of that particular day is very high, this particular thing can trap some amount of moisture.

So this moisture weight will also be taken into account if we are not having some dry sample. So how we get this desiccator, so desiccator having some desiccant is a hygroscopic substance that induces a dry state. That means if you have a hygroscopic matter which put inside so there there is some disc over it, so porcelain disc is there and above that we give the sample but you have holes created on this porcelain disc.

But the desiccant we can put such as calcium chloride. So calcium chloride desiccant we can use over that and that particular one we all know that is a nice material which can trap moisture very easily from the environment. So the base section, what is that base section? Basically the base section contains the chemical drying agent. So this is the base such that as I told you, it can be your anhydrous calcium chloride, it can be calcium sulphate or anhydrous magnesium perchlorate or phosphorus pentoxide.

So when we do, that means we can have some amount of calcium chloride in this particular base. So solid calcium chloride and above that we have this particular porcelain disc with some holes over it and this is the base basically and where we put this particular sample. So this is keeping something really dry atmosphere inside the desiccator but what we find? That sometimes we need further drying of this particular environment such that a precipitate you can have and which has been filtered recently.

So this precipitate which is filtered recently, initially you dry it in air. So some amount of moisture or some amount of water which is being trapped inside the corresponding precipitate, is dried, then you can put inside this particular desiccator Uhh. So this desiccator you can have this cover. So you open up this and you put this particular material over there and what we find that if this is not enough, that means calcium chloride have some capacity to trap moisture, so it is basically trapping the moisture.

But if we can use some other desiccant which is superior than that of this calcium chloride, what we can put? We have this base basically end there we can put some watch glass or (())(17:51) where we put a 2nd desiccant. So this is the 1st desiccant, your calcium chloride is your 1st desiccant and the 2nd one which is more hygroscopic such that white powder of phosphorus pentoxide okay? So this white powder you can put inside this and you can keep sample over here, you can keep sample over here and you can keep sample over here.

So within few hours or within overnight, your sample, that means the recipe to it is dry enough. So you make it dry. You get the powder sample which is pretty dry. So this particular sample, the powder or are very dry sample you can get. So when you have, along with calcium chloride, you can also use a secondary or a 2nd desiccant which is your P2O5 okay? So this magnesium perchlorate or phosphorus pentoxide is not that separately were using phosphorus pentoxide.

So along with calcium chloride, we can use this particular phosphorus pentoxide for a very good Uhh Uhh Uhh dry atmosphere within the desiccator. Next we can do something where we can know the about the tools that how you go for filtration. So filtration we all know that there are several things. The 1st thing, how we can explain the process of filtration okay?

So we are discussing all we are talking to you from the very beginning of our class that you can have Ni2 + in solution which is unknown amount and we want to determine the corresponding concentration of this Ni2 +. So we give some reagent. So if you have this solution which is aqueous solution of certain concentration of Ni2 + and this is giving you a very clear solution. So this solution is definitely a clear solution but to know this, how you know this filtration basically?

So this filtration technique what we can see that you must have a precipitate in it which is insoluble in aqueous medium. So how to do that? You just add the known reagent, everyday I am just talking about when we try to give some example, now just you see what is that? So which is nothing but DMGH2, dimethylglyxine. So this will bind to this nickel 2 + and will give you a reddish pink precipitate.

So you get the precipitate of NIL2 where this is your LH2. You have 2 protons attached to this oxine function which can be removed. So once you get this precipitate, now your challenge is coming. So you have the clear solution, then you have the PPT, then we want to do the filtration, that means filter we can do. Before that what you can see? That what is in your hand? That means, the apparatus you can see, what are the apparatus and the medium through which you can filter it out and how to do that?

Because somebody if he does not know, if he or she does not know how to filter something by this particular thing, so this is a very important part. That means, you require some apparatus and the medium through which you can perform the filtration because there are several things starting from your filter paper, what you know from your school days and you know that we must use the funnel.

And the funnel and the filter paper is one combination, then we can have the filtering crucibles, the Gooche crucibles where the bed, the filtering bed is already there within the funnel that made of glass. We can directly filter through that particular bed. That means we consider this as a special name and how we can use that particular thing. So 2 things are there. Just we will be slowly discussing, they will just go into that that once you have the filtration.

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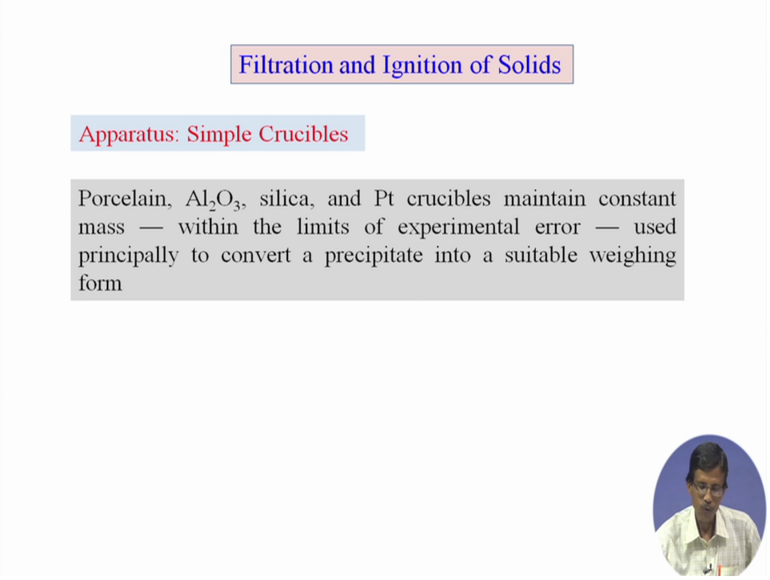

Filtration and Ignition of Solids

Apparatus: Simple Crucibles

Porcelain, Al_2O_3, silica, and Pt crucibles maintain constant mass — within the limits of experimental error — used principally to convert a precipitate into a suitable weighing form

And in this particular slide, we have written something else also, that ignition of solids. So that, I will come there that how we can have some solid which is being filtered. So the filtered solid you can burn to get some information related to your analysis. So as I told you, the apparatus what you can have, the simple crucibles you can have if we just go for this ignition process. So these simple crucibles are of different types. You can have porcelain, you can have alumina, you can have silica, you can have platinum crucibles. What they do?

They maintain a constant mass, that means constant mass platinum crucibles taking in a constant mass within the limits of experimental error. Definitely you can have the error bar when you go for the weighing process used principally to convert a precipitate into a suitable weighing form. So what does it mean basically and how it is related to filtration? So what we are looking for?

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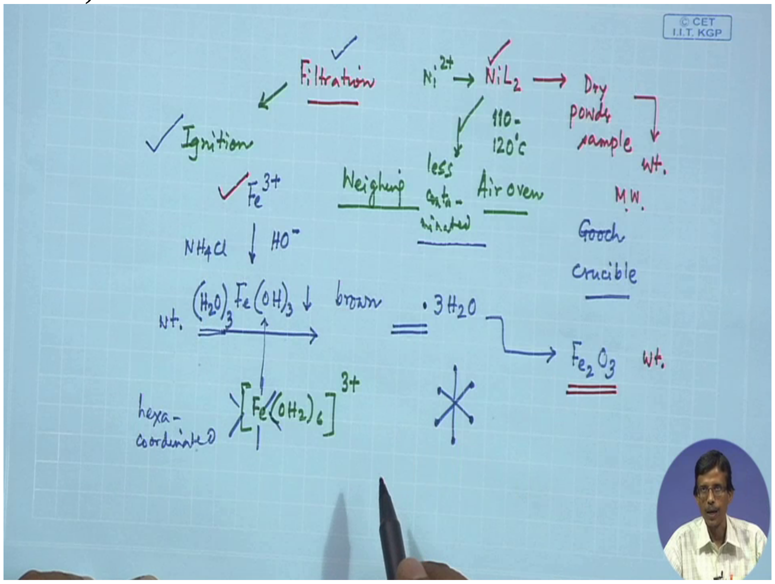

That we should go for filtration and as I told you this now that we can have NIL2, the nickel dimethylglyxine in your hand and we want to get a dry powder sample of these and if we are able to take the weight in particular form, from that particular weight, by knowing the molecular weight of the complex, you can find out the amount of nickel present in the complex or amount of nickel present in the original sample what we are analysing.

But this is something because filtration we are now tagging with ignition. And when we will detailed discussion we will make for some technique where we can do that particular analysis by taking the weight. So weighing is a very important thing in all these cases because by weight, we are trying to determine the corresponding concentration of the metal ion in a precipitate which is your metal complex.

So this particular weighing process is related to something else also because I have not told you that how you dry it. Is it, we are just keeping in the desiccator or you dry it or heat it at a particular temperature? So in actual practice, I told you in one of my previous class that you can dry it at 110 to 120 degree in air oven. Then you take the weight once it get give you the corresponding weight, that also I told you that what the constant weight means.

So in this particular form, if something is not readily available in this crystalline form because this nickel DNG is a very crystalline form and a very powder form and it is not contaminated. So it is less contaminated. It is less contaminated by bear the species which is present in the solution. But if we can go for estimation of FE3 +, what we can do? We can provide then sufficient amount of hydroxide ions in say ammonium chloride, you know the common ion effect because this is a particular reagent for group separation during the identification of FE3 + in qualitative analysis.

So in quantitative analysis of FE3 +, if we put ammonium chloride and ammonia hydroxide make sure, we will be getting ferric hydroxide precipitate, the Brown precipitate of ferric hydroxide. So that precipitate, you can have now the opportunity to filter it out and once you filter it out, if it has a very definite composition like that of NIL2, you can take the direct weight by taking it some filter paper or in Gooch Crucible because here we will use something which is known as Gooch Crucible because we are talking about the Crucibles.

We will see what is known as Gooch Crucible which is a sintered base Crucible. There, we filter it out and take the direct weight. So weight of this particular form but it is not so easy to get because this is basically forming what we should know here that initially when ferric ion is present in the solution, we have hexa aqu ion, hexa aqu ferric 3 ion was present in it. And slowly one after another, is removing from here as hydroxide ion.

So what is precipitating out is not a monomeric form, is a polymeric and condensed form where this iron because this is the iron what you see and it is interesting to know here also because we do not know most of the time that when you have the 6 water molecules are bound, so basically we get a centre and 6 water molecules, these are the oxygen of water which are bound to the iron Centre giving a corresponding hex aqu species.

So what about these? This is basically precipitating out. So this actual composition of this ferric hydroxide is not bad even if we consider only the corresponding number of water molecules present in A. So what we get? We get this particular from because we cannot disturb the corresponding fixed positions around this iron because iron will always be hexa coordinated. So it will be hexa coordinated.

So what we should know? That whatever we write in the book or whatever we see in the book, and whatever we write in the is the ferric hydroxide but it is not the actual form. It has some water molecules attached to it even if you write something as $3H_2O$, it is very difficult to write the exact platform. That means the exact formula and exact formula weight like your nickel DNG complex is not known for your ferric hydroxide.

It may have several number of H_2O molecules and also is not that always you have 3 hydroxide groups because the 3 hydroxide groups are required to precipitate it out. But you can have 2 + 4 water molecules also. So is basically is not a very pure form you get. So this we can do then that you apply ignition and you convert this particular species to FE_2O_3 at a high temperature.

So this is the corresponding procedure where you have the different formula of this FE_2O_3 we know and it is also a typical gravimetric technique and how we can use the different types of crucibles that we can see and what type of Crucible we can select for this, that means weighing of the precipitate and its ultimate conversion to FE_3O_4 which you can weigh out for its percentage of iron present in the original sample of FE^{3+}. Okay thank you very much...