Course on Analytical Chemistry
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Module No 02
Lecture 07: Techniques, Wet Ashing

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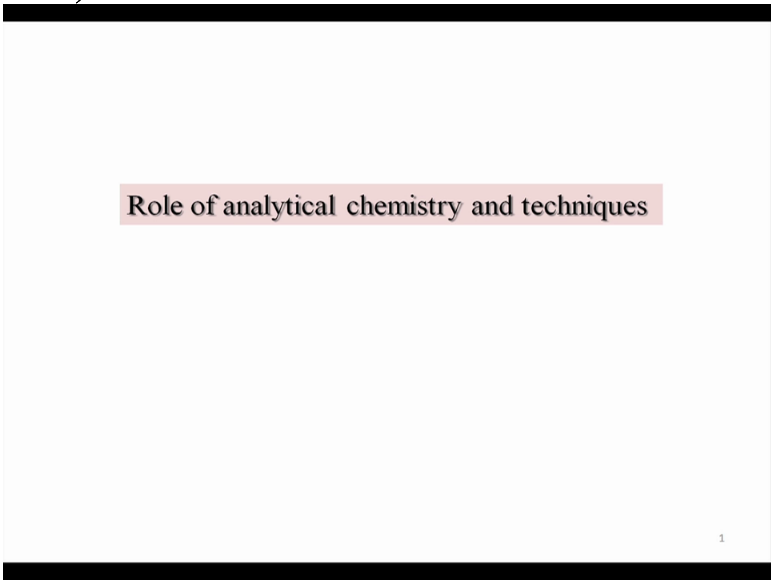
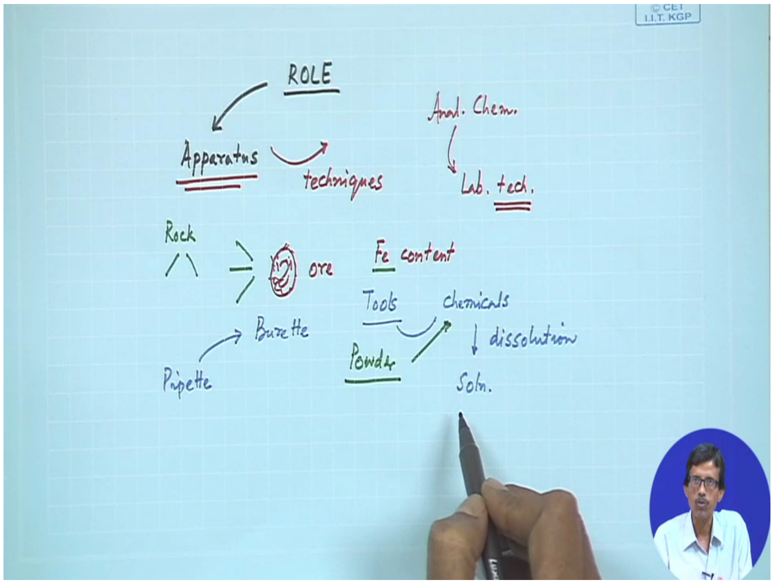


Role of analytical chemistry and techniques



Good evening, welcome to this 7th class of analytical chemistry where we are discussing about the role of analytical chemistry in different spheres of life. So what we are talking about is the role as well as the different techniques what we can use and what we can follow particularly the apparatus because the most important thing always we know that what are the apparatus we can use to follow some analysis because this particular thing what we are talking about the analytical chemistry and the laboratory techniques, so these laboratory techniques are very much dependent on the apparatus what will be used.

So that is why this apparatus is related to the techniques where we know that a typical chemical analysis when a technician can perform, he is relying on this particular technique and the handling of so many apparatus for that particular technique. So slowly we will see that how these tools, the different techniques and the chemicals are important. Suppose we are analysing some ore.

So suppose some ore material so very 1st thing is that a problem we know that is a ore material we have to analyse or say, its RN content. So what are the things we can use or what are the things we can apply for that starting from the different tools to the chemicals, so these are very abstract definitions for the time being to you but what you can feel is that definitely when we do for something where the tools are very simple tools we all know that a bullet.

We all know what are the bullet and how the bullet is also useful with the pipette. So these are the very simple tools. We are not going for talking about the complicated instruments where the instrumental techniques are available. So those instrumental techniques are also giving us something related to those rules. So a bullet and pipette can be your tool. And what are the chemicals we can use? That means ore and the next immediate problem to that ore is the dissolution, how we can dissolve this or it do some solution such that ultimately what we can have?

We can have the solution where the analytes are present and we can analyse that particular solution for its iron content. So we are talking about so many things because this ore and this is coming from some mining process or it canit can be obtained from the different rock samples as I told you earlier, from the Earth surface or the mars surface or any other surfaces what we can get these also the physical characteristics of these old what we are talking about is also important when we can reduce the size of this solid sample to a powder form.

So the useful form what we can use for the treatment of the different chemicals for its dissolution is also dependent because if you have a big size of ore which we cannot put inside the in some mineral acid such that it can be solubilised.

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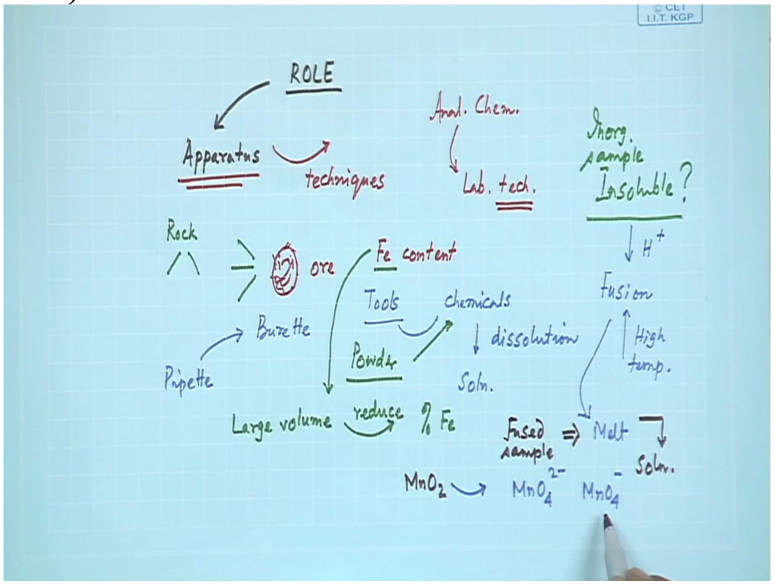

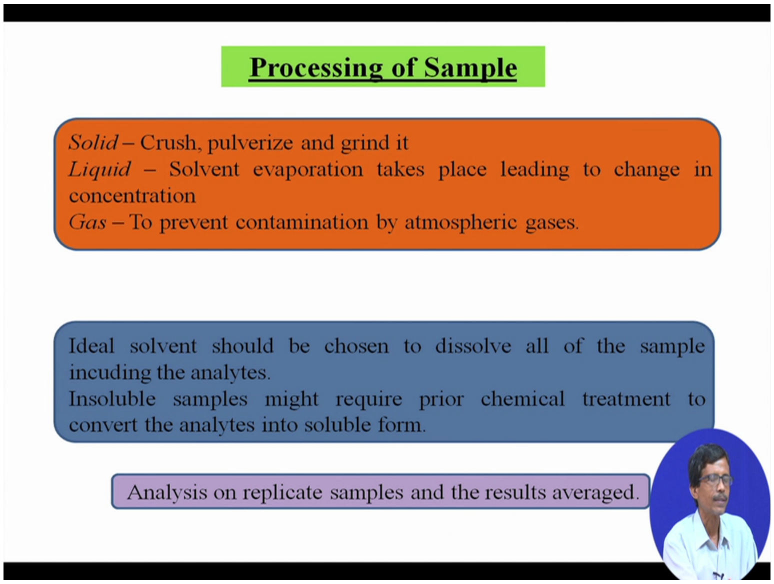

Processing of Sample

Solid – Crush, pulverize and grind it
Liquid – Solvent evaporation takes place leading to change in concentration
Gas – To prevent contamination by atmospheric gases.

Ideal solvent should be chosen to dissolve all of the sample incuding the analytes.
Insoluble samples might require prior chemical treatment to convert the analytes into soluble form.

Analysis on replicate samples and the results averaged.

So what we see is that if we have a solid for the processing of the sample, so this we already discussed a little bit. So if we have the solid sample so we can crush it, we can pulverise it and grind it. So these 3 processes for processing of the sample give rise to the powder material which we can ultimately analyse for its content of the analyte. If we have the liquid motor we can do?

The liquid if we have come of the solvent evaporation can take place leading to change in concentration.

So if we have a liquid of large volume and therefore the concentration of this iron such that you have the powder sample of that ore and we make it powder and then the powder is dissolving in the acid. So we are dissolving in that particular acid and after that, if the solubility or the dissolution process is not so easy and we get a very large volume, so immediately what we can do? We can reduce the volume of any liquid sample. So for liquid sample, it is very easy to tell that a large volume can be concentrated such that we can increase the concentration so that means, the percentage of iron in the solution can be increased.

For the gas sample what we see? That to prevent contamination by atmospheric gases, that means we will not use something where the atmospheric gases will come and contaminate the gas sample what we are going to analyse such that we are going to analyse nitrogen and if the nitrogen is contaminated by the oxygen of the air as well as the nitrogen of the air, then our process will be a difficult one, our estimation will be erroneous.

Then to dissolve all these things, an ideal solvent should be chosen resolve all the sample including the analytes. So what we can see that these, including these analytes that acid can be your solvent what we can consider it but if it is some organic matter, some organic solvent we can use to dissolve these analytes and if we have some insoluble material, so which will require some chemical treatment, that means we will discuss afterwards that for some insoluble material we know that for a typical analytical process, the inorganic sample if we can have which is insoluble, what does it mean?

That insoluble inorganic sample, that is it is not soluble in the aqueous medium, is not soluble in the water medium even after we treat with it by the mineral acid. That means if we have the sample containing oxides or sulphides or nitrates or sulphates, this is not going to the solution. So we can go for something which we will discuss afterwards that we can go for fusion, a high-temperature fusion can be useful. When we go for this fusion, what we get? We get a melt.

That means, if you sample which is nothing but your fused this insoluble inorganic sample. So if we have the fused sample which we get as the melt and now if we add pure water, that means acid free water or some alkali or some mineral acid, we get, there is every chance of getting a solution out of that particular melt. We all know that at this point of time we can give you some examples that is a mineral of manganese which is MNO2 which is a black powder is a pyrolusite.

And the solubility of this pyrolusite in acid is not good and in water, it is incompletely insoluble. So we can follow this particular losses, you can use it and if you go for the alkali fusion, that means sodium carbonate and sodium hydroxide or potassium hydroxide on some mike of oil at a high-temperature on the Bunsen burner or any other open flame, what we get is that this can be converted to some melt.

And what is that particular melt? Melt is coming out from MNO2 due to this oxidation and it can give rise to MNO42 - or MNO4 -. That means it can be used to manganate or permanganate anions because the corresponding cations which are sodium or potassium depending upon the use of potassium hydroxide or sodium hydroxide during the fusion process, we get either sodium manganate or sodium permanganate or potassium manganate or potassium permanganate as the melt.

So the chemical treatment that is why is very important. So insoluble samples might require because we have to be very much careful whether the sample is purely insoluble. So we need take Uhh the advantage of this chemical treatment and this chemical treatment in this particular case is nothing but the fusion treatment to convert the analyte into the soluble form where we can solubilise this particular analyte into the solution.

So what we see that analysis on replicate samples and the results are averaged. So we can go for 1, 2, 3 or 4 samples. So 4 samples can be analysed together and we take the average of it which is acceptable range of the corresponding percentage of either manganese or iron in this particular example of ore.

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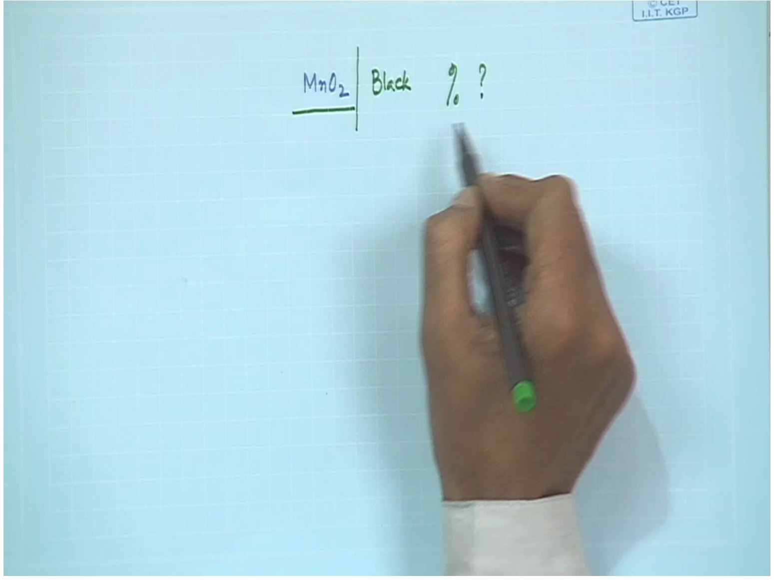

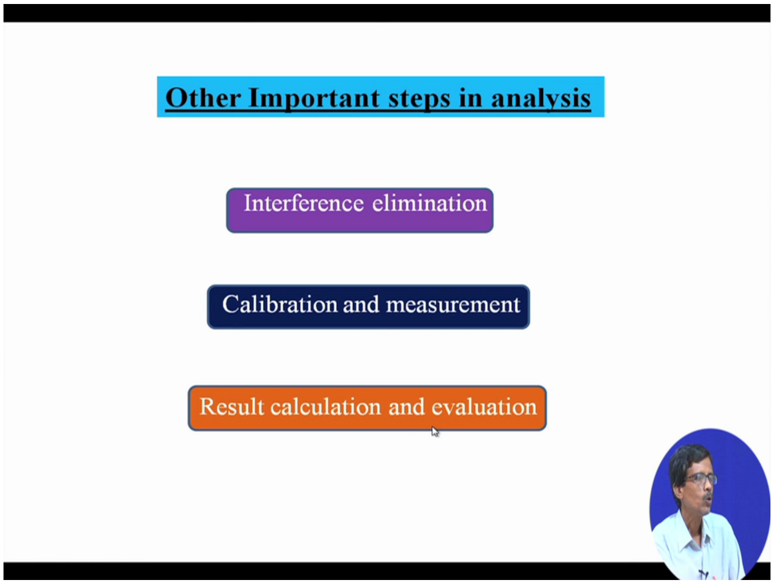
Other Important steps in analysis

Interference elimination

Calibration and measurement

Result calculation and evaluation

Then several other important steps we can follow for this particular analysis where we see that how the other important steps we can follow for this particular analysis where we have interference. So if we have this manganese dioxide, that means the pyrolusite as the ore and this manganese dioxide which is a black powder and depending upon its source, that means wherefrom it is coming, that means which mine is giving this pyrolusite we get this as the black powder.

But what is the percentage of MNO2 that means the pure MNO2, that we do not know. So it can have charcoal, it can have other black material inside. So how to eliminate the interference such that we can go for enrichment. So that is a typical process. Enrichment is that means we want to increase the corresponding concentration of manganese dioxide in pyrolusite. So the typical enrichment can also give rise something that depending upon this percentage of MN02 compared to the percentage of say some charcoal, some charcoal is also present along with this manganese dioxide as pyrolusite.

So this particular enrichment process if we can follow, we can increase the concentration of pure MNO2 in this particular material such that we eliminate the interferences of some other extraneous matter. Then we can go for calibration for a article type of measurement. So calibration and measurement, both will go side-by-side where a particular technique can be liberated.

That means we can take some known sample of manganese, known sample of pyrolusite which is can be a certified sample which we can analyse by this particular technique and the concentration can be tagged with the unknown sample what we are measuring. And finally we give the results for the different calculations and ultimately we can evaluate the corresponding technique how good this particular technique is to Uhh go go for the corresponding analysis of the sample.

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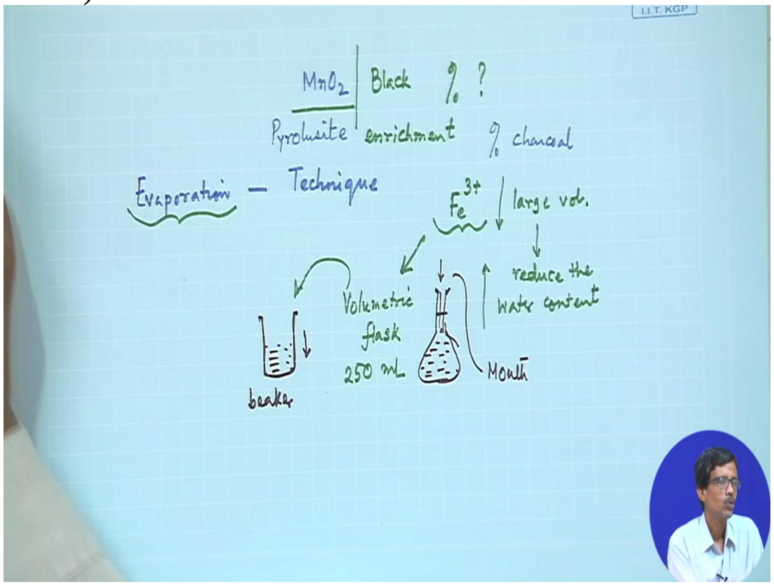

Then one particular thing we can follow now is the evaporation. So evaporation is a very simple and useful technique because we were discussing about the different techniques. So if we consider a very simple thing that means which is evaporation, so how will you define and how will you go for the evaporation? Just now we are talking about a very dilute solution of any sample which contains a FE3 +.

So a ferric solution, a ferric iron solution is there, so this ferric iron solution in a large volume of water, so volume of water is very large. So we have to reduce the water content. How we can do? Because we all know that it we want to remove this water, it is a simple process of evaporation. So how to do that evaporation? So this is there say suppose this particular ferric solution is there in a volumetric flask and we know that volumetric flasks are coming of different volumes, say 100 millilitres, 256 millilitres, 500 millilitre or a litre or 2 litres, et cetera.

So from this volumetric flask, we can have some volume of this solution. Suppose we are having some 250 millilitre of this ferric solution and the concentration of this ferric present in this particular flask is very less. So we can go for evaporation. So how to evaporate with that? So that is the next question what we can ask that how to evaporate this particular solution so that there will be very rapid and quick evaporation of the water molecules from the solution.

So we cannot do? So we all know that a very simple volumetric flask has a shape of this type so which is graduated so we can have this volumetric flask. So what is this? This is the mouth and the mom is very small. And we cannot heat the volumetric flask because we can damage the volumetric flask if we heat it. So what we can do, we just transfer this to a beaker brother mouth is large. So we just transfer the whole solution of this 250 millilitre in a beaker.

So we have the beaker. Then we put this over a Bunsen flame and we go for the evaporation. So what will happen? That evaporation can take place and as this volume is reducing and your FE3 + concentration is increasing, what to do? That we have to be very much careful that this open beaker may not be very much useful when the volume is reduced to a small amount.

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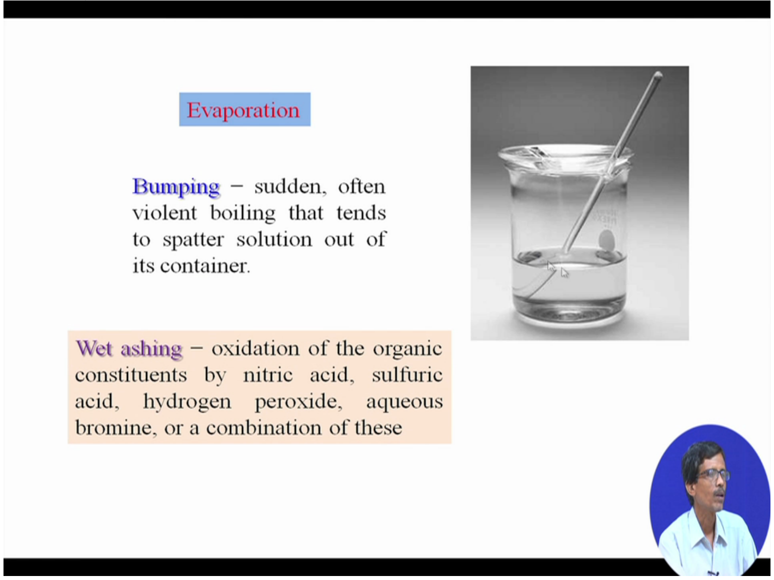

Evaporation

Bumping – sudden, often violent boiling that tends to spatter solution out of its container.

Wet ashing – oxidation of the organic constituents by nitric acid, sulfuric acid, hydrogen peroxide, aqueous bromine, or a combination of these

So what we can do? This evaporation process by doing this, so taking the help of this beaker with a glass of water and this weekend on the Bunsen burner and we cover it with a lid. So is a typical lid of glass. So we consider this as a corresponding lid of glass and cover it said that you have this vacant side on this particularly this tip side where the rod is being inserted and we keep on heating this solution.

So solution original volume was say up to this point say and it is reducing and we can reduce further. And during this process, through the sides when the volume is less if we keep on evaporating this from the solution that this vapour, water vapours will come out from the side of this lid, the glass lid and the corresponding thing, that means if we do not allow is open evaporation is covered with a lid and therefore the evaporation rate is restricted, and some of the water vapour is coming and touching this lid and getting condensed and will go back to the solution again.

So the rate of evaporation when it is covered is less. So initially when it was open, the rate of evaporation was very high and a very fast reduction of this volume was taking place but remember that the concentration of ferric iron was also less. But when the concentration is increasing, we should be very much careful in covering the solution and we should allow slow evaporation. So the rate of evaporation is being reduced.

But one important thing, one difficult thing can happen at this particular point that is in the solution is getting concentrated, so when we are handling a concentrated solution and in the concentrated solution, what can happen that local heating can take place and if there is a sudden and violent boiling that tends to spatter solution out of the container, that is known as bumping. So at this point, bumping can take place such that this because what is going out from the solution is a dissolved air or the dissolved oxygen as bubbles.

So after some time, the amount of dissolved oxygen or air is less and if this glass rod is not sufficient to allow the escape of these vapours, that means the water or air vapours by this side of this glass rod, what can happen? There is the local heating can take place over here and this water vapour from here can go out as bumping the solution. So when this is bumping, it can take out some of the solution out of the beaker.

So we will be losing some of the solution. So care should be taken to avoid that particular type of bumping such that no such solution or even a single drop of solution can go out from the beaker which will reduce the actual concentration of the solution. So this particular process because everything is related because we are talking about the dissolution.

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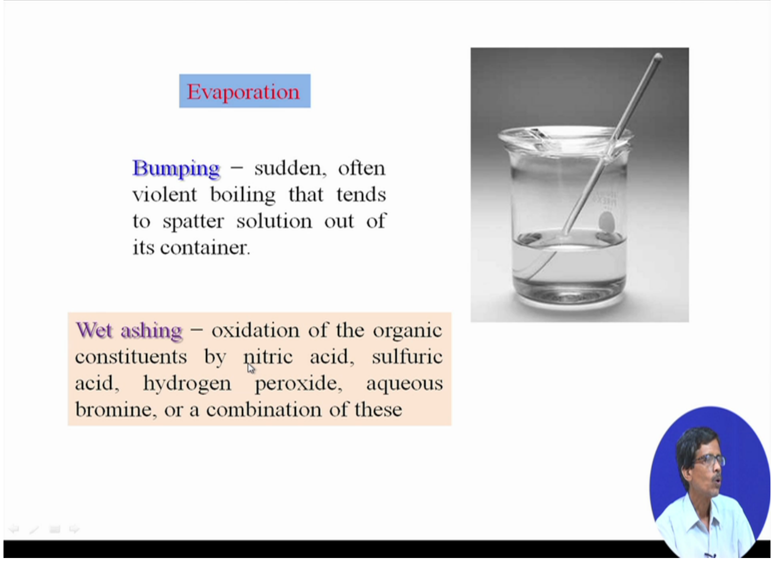

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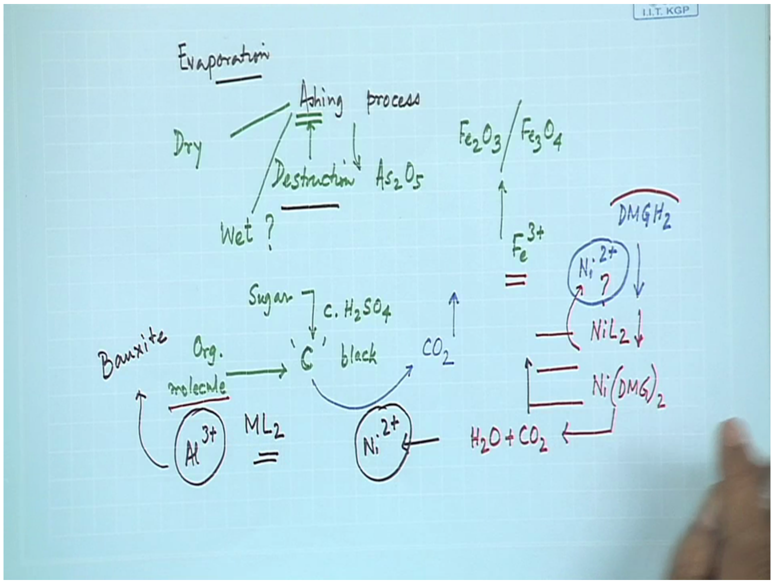

And after that, that means when we talk about the dissolution of any ore, we can have something which is also related to evaporation for process, that means the dissolution process or some ashing process. This we have seen in of anyone of our previous class where we were talking about the arsenic poisoning of the deer family where we are going for some ashing process which we term as the dry ashing because whatever arsenic present over there has been converted to arsenic pentoxide as the corresponding oxide on a Crucible.

So heating any sample and forming the corresponding oxides by heating in air or plenty oxygen of that air giving rise to the corresponding oxides is known as the corresponding dry ashing process. So ashing is such that we are getting the different ashes. Sometimes ashes are not containing any active ingredient, that means active, these oxides and all these because it can so happen that we will see that you can get FE2O3 or FE3O4 from the solution of FE3 +.

So this is a typical process where we can go for a dry ashing of FE3 + solution what we are just seeing for our beaker evaporation, open beaker evaporation of a solution containing FE3 + but ultimately we can convert these as the corresponding FE3O4 or FE3O4 for the ashing process. Now we will see. So these ashing processes can be of 2 types, so this what we discussed earlier is the dry ashing process.

Now we will see something what is the wet ashing process. Ashing process is wet what we are talking about. Dry is fine, we get some white powder. It may or may not contain some oxides of arsenic or any other material. It is simply some S. So carbon when you burn, carbon the we burn, we all know that some ashes are forming. That means those ashes can have some oxides of the material water is coming out from the coal.

But what about this wet ashing Uhh because this is not going with the language that ashing, we are doing for the ashing with the bed. That means, we are doing something or we will be doing something such that we can do some destruction, destruction of some part of the material to go some ash and that ash can be taken into the solution. So one such example is our wet ashing. This wet ashing is nothing but if we have some organic constituent enjoy material what we are handling, so what we can do?

We have to oxidise that particular organic material because the organic material we all know is forming from carbon, hydrogen and oxygen. And if we are able to oxidise by nitric acid, it can be converted to carbon dioxide and water. So this particular thing when we know that initially what we can get if we treat it with acid, sometimes we treat with concentrated sulphuric acid, so we all know that from our schooldays example that Sugar we all know, if that Sugar is treated with concentrated sulphuric acid, we know that we are leaving behind with carbon.

The charcoal type carbon we are leaving behind. So that is a black material, so that black material is leaving behind and that black material if we are not able to treat it, so this carbon from sugar, this carbon can come also from some organic molecule. So from that organic molecule, I have this carbon is coming out as black articles, so we cannot have this black particles inside a solution. So within the solution, the solution the entire solution will be black in colour with floating carbon particles and will be contaminated by all these carbon particles.

But in a regular fashion or in a slow way, if we oxidise this particular carboneous matter or any organic sample by addition of sulphuric acid or nitric acid, we can oxidise this carbon, without forming this carbon as the carbon particle we can convert this as the carbon dioxide and those carbon dioxide can escape from the medium. So we can take not only concentrated sulphuric acid, we can take nitric acid, we can have sulphuric acid, all are oxidising agents.

Then hydrogen peroxide, aqueous bromine and sometimes the combination of these because we do not know what are the species will be useful for destroying any such organic matter for this particular oxidation process. So one such example we can take as we will be talking afterwards once again that if we are growing for the analysis of NI2 + and this NI2 + can be complexed with some reagents which is dimethlyglyxine which is abbreviated as DNGH2 and that DNGH2 when we use is forming a complex of stoichometry NIL2.

One nickel is bound to L2, we know these are known as coordination complex. So what it has? So we are talking about something that how we can analyse the concentration of nickel in a solution which is like our this iron solution is unknown solution of nickel. What we can do? We can use this organic molecule, that is your reagents, that is organic chelating agent, we call it as the DNG as the organic chelating agent which bind nicely with NICL2 salt or NI2 + to give this and this is a corresponding pink red precipitate coming out of the solution which is insoluble in water.

So this can give rise to several opportunities for analysis of nickel by this particular technique. So this we will discuss that how we can use by taking the weight of this nickel salt of nickel complex of DNG by gravimetric estimation. If we are able to solubilise this in some organic solvent, we can go for some spectrometric method of analysis of nickel. But for this example that how we can get back this nickel 2 + in Aqueous solution the NIL2 complex, how to get back like the getting the corresponding irons ion or any other metal ion from the ore sample, so this was also another example how we get the nickel ion from this NIL2 species which is a coordination complex.

So there basically we can destroy the organic part. We should be able to destroy this organic part by treating this nickel DNG by mixed acid of concentrated sulphuric and concentrated nitric acid. Though oxidise it, that means this organic molecule, the DNG H2 when it is bound to NIL2, it is the form which is DNG, one proton is lost, so it is one negatively charged which is balancing the corresponding di positive charge of the nickel.

So you have to destroy this entire DNG sample by treating with those combination of acids to oxidise it for CO2 and H2O. And in that particular way, what we get? We just get this in the aqueous solution. Carbon dioxide will be present in this uhh solvent medium, that means aqueous medium and ultimately it will escape or from the medium that what we can have now in the solution? Within the solution, we get NI2 + in that particular solution.

So this is a typical examples of wet ashing, how we destroy the ligand which is bound to our nickel 2 + and how we can handle this particular thing for analysis back. So this is the technique. So these 2 are all interrelated to this particular analysis that we have NI 2 + and that NI 2 + were trying to bind it with some organic reagents and we get this and once it is warm, so this is a particular composition and a definite sample of metal ion complex of nickel 2 + which we can analyse by 2 different techniques, one is the gravimetric technique and another is the volumetric technique.

But at the same time, the process of this wet ashing what we are talking about this wet ashing inside a beaker because we can take the sample of this nickel DNG inside this beaker and we can destroy the organic part, that means the DNG part by treating it with a mixed combination of his nitric acid and sulphuric acid and we will be leaving behind with NI 2 +. So this is also true for any general example of metal complex.

This can be any of these metal ions. It can be AL3 + also and this can be any useful ligand which binds to this metal ion to give you this corresponding complex. So this wet ashing process is always we can follow that we get the corresponding metal complex and we describe the metal complex. So this is the destruction process, the ashing process. So wet ashing process is the destructive, destruction of the corresponding ligand part and if we destroy the ligand part in the aqueous solution, we will again have the corresponding metal ion, that means the aluminium 3 + in the solution.

So this is a particular technique how we get this from say some aluminium ore also which is your bauxite. So something we can get as aluminium 3 + which can be complexed with some ligand and Uhh again we can get back the aluminium 3 + from this complex. Okay? Thank you very much.