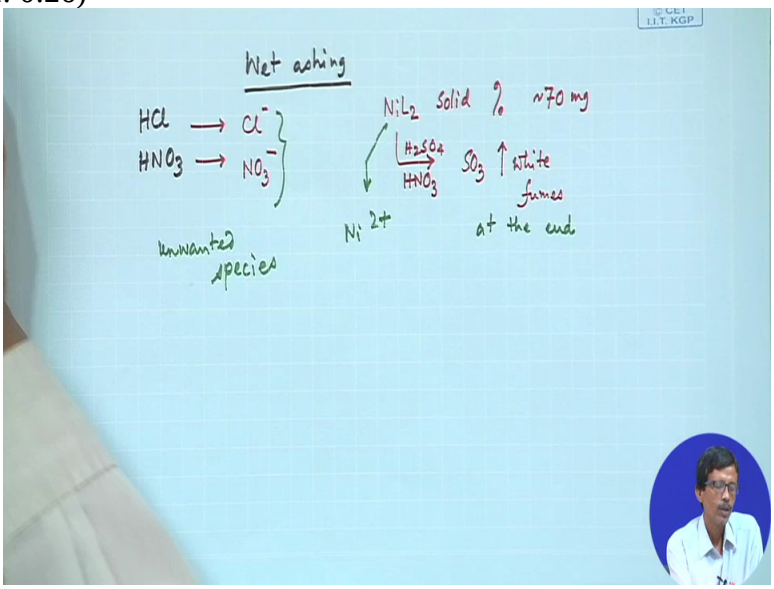
Course on Analytical Chemistry
Professor Debashis Ray
Department of Chemistry
Indian Institute of Technology Kharagpur
Module No 02
Lecture 08: Apparatus and Weighing

(Refer Slide Time: 0:26)



Welcome to this 8th class of analytical chemistry where we are discussing so far during the wet ashing process, that we can dissolve, that means the metal complex can be dissolved by destroying the organic part of the metal complex. And at the same time, we can do something else where we see that during the treatment, suppose we are using hydrochloric acid and nitric acid for the treatment of the any unknown sample, so treatment of these unknown samples and thus can give rise to the solution as contamination of CL - and NO3 -.

So what we find from there that these we are not require, we are not requiring these anions during the analysis. So for a particular type of analysis where we have seen that we are talking about NIL2 and the organic part were trying to destroy by concentrated sulphuric acid and concentrated nitric acid, so during the ashing process, your nitric acid is destroying the organic part and towards the end what can happen?

That is how we will say that how our most of the organic part has been decomposed. So in the beaker what we just seen in our previous class that the covered that particular beaker with a glass

rod inserted in it and we just treat it with the concentrated acid mixture of this complex because this complex is solid. So some weighted percentage, so some percentage of nickel we all know from this complex, so some amount of wet say about say 70 milligram of that sample is weighted out and taken into the beaker and treated with this particular acid.

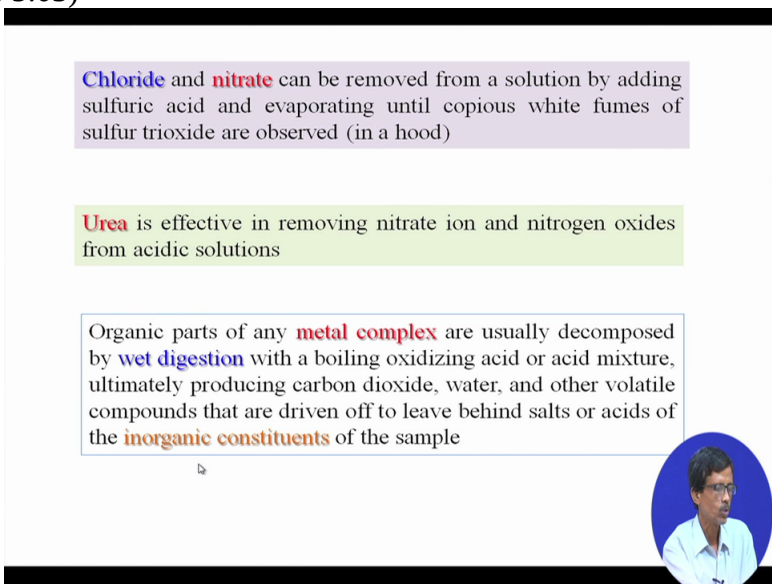
So we keep on hitting with this. So nitric acid is being consumed for the oxidation of the organic part. But at the end what is happening? How do I know that most of the quantitatively, the organic part, all the organic part has been decomposed and destroyed by the formation of water and the carbon dioxide is known at the end by the elimination of white fumes of sulphur trioxide because this white fumes of sulphur trioxide will be coming from this sulphuric acid when the sulphuric acid are no longer used for the destruction of the organic part.

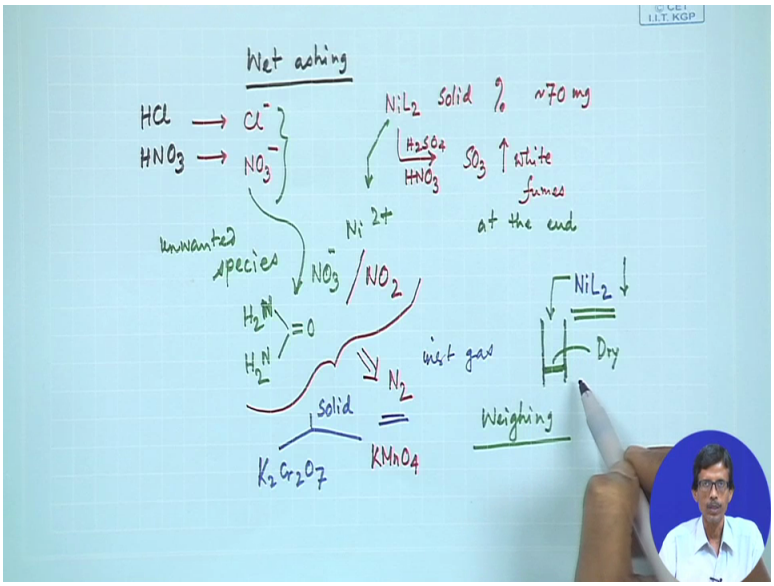
So these white fumes are seen there at the end of the stick will oxidation process which is nothing but the destruction of all these organic part of these complexes. So during this process, the solution what we are trying to get is your NI2 +, so this NI2 + is though forming in the solution but when we are treating this, if your nitrates are there, CL - is there but what we are getting from the sulphate anion, SO3 is going out and escaping out from the medium.

But if we have these anions, so these are unwanted species which we do not like to have in the solution for the analysis. So these unwanted species because most of these anions because this we know that this nitrogen is present in the + 5 oxidising oxidation state, so this is highly oxidising. So it will just interfere the corresponding treatment, the follower treatment for the analysis of the metal ion further. So unwanted species, we should remove it.

So how to remove this because this again we will follow the same technique. That means if we take this sulphuric acid from there, this will react and again, sulphur trioxide fumes will come out and that can destroy both chloride and nitrates.

(Refer Slide Time: 5:03)





So this can be removed. So in a statement what we can say, the chloride ions and the nitrate ions basically can be removed from any solution of this particular process by adding sulphuric acid and evaporating until copious white fumes of sulphur trioxide are observed. What I just told to you that SO3, large amount of SO3 fumes will be forming from there and if it is covered with a lid, so if the whole beaker is full of this sulphur trioxide fume, and this should be done in a toxy because this is toxic for our health, we should do this particular process inside a hood.

So this particular thing can eliminate this chloride and nitrates. So similarly some other reagents can be externally added. Urea which is nothing but NH2 NH2CO. So this is a typical organic species but this can react with NO3 -. So we all know the reaction. Just now I was talking that NO3 - is the oxidising species which can oxidise the urea. So if we try to avoid the addition of sulphuric acid in that particular medium, if we do not try to destroy the nitrates present in the solution by sulphuric acid, we can add some amount of urea and that urea can destroy these or some other nitrogen oxides like nitrogen dioxide also.

If it is forming locally, so that can also react with because this NO2 is also oxidising, that can destroy this particular urea. So if we see that something can be there which can be oxidised by these anions, these ions like NO3 - or NO2 and this urea can be added but from that particular reaction, what is forming? If nitrogen gas, the N2 is forming out of that particular reaction, it will be good sometimes that instead of oxygen in the medium, we can have N2 in the medium. So the beaker beautiful of now N2 which is a inert gas.

So the analytical technique which we are following on the open beaker, normal state of it is full with sulphur trioxide, it can be filled with nitrogen gas N2 if we use urea for the destruction of the nitrate ion. So this extraneous matter we just removing, there is now I told you what is the same thing in a statement what we can write is that the organic part of any metal complex, the example I gave you as the nickel chloride, nickel DNG complex can be decomposed by wet digestion.

So these are the things which we can solve immediately one after another. That means you have metal complex, you go for wet digestion by boiling with some oxidising acids or so any other oxidising agents like bromine or hydrogen peroxide and we drive out all the gas is what is forming over there and you will be leaving behind what? As I told you, you will be leaving behind with the corresponding metal ions.

Since we are handling the corresponding mental acid, we will not be ending the process person metal ion things. So you have the inorganic constituents. So all inorganic matter will be there. That means the way we are talking about the destruction of the chloride and nitrate ions, so these are all of inorganic origin. So these anions will be there as well as the cations like NI2 + or FE3 + will be there in the solution which we can further take up for our analysis.

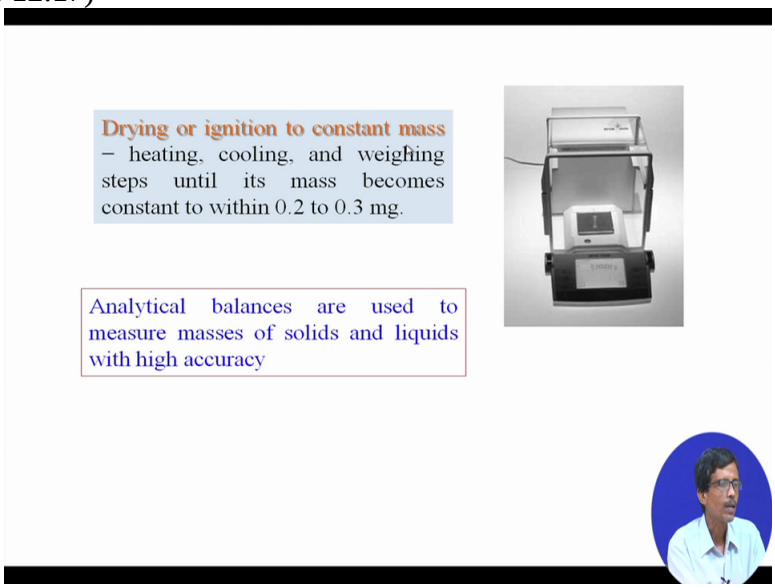
So how we do this this particular analytical thing is that we require analytical balance. So just now what we talking about this NIL2 formation, we can take this example, that means NIL2 is forming, nickel DNG is forming in the solution as the red pink precipitate and we can filter it out on the filter paper and we can dry it in oven, say air oven. So this basically we use, we can filter it in some Gooche Crucible, we call it as a Gooche Crucible. So it has a glass filtering bed. So through this bed, the solution of this nickel DNG we can filter it out.

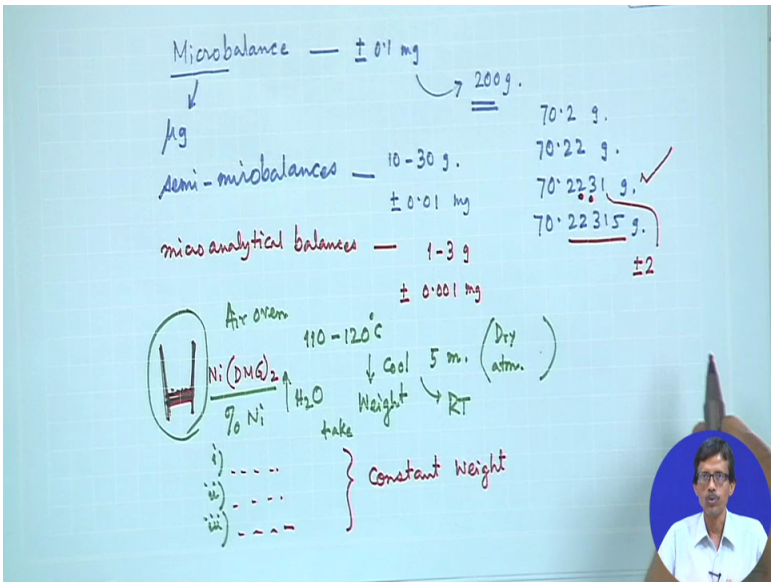
So elaborate procedure for all this filtration we will discuss afterwards but right now what we can give the example of the different amounts what we can, so how much of this NIL2 we can weigh out. So if we have the drive sample on air oven, it can be dried. And if we know the empty weight of this Crucible, we can take two weights, one is the empty Crucible weight and another is the weight of this Crucible + this complex.

And from the difference, we can find out the weight of this NIL2 from this. So for this particular thing, that means the next thing what we can do is the weighing, how we can take the weight of any unknown sample. Suppose we try to get some solution as we all know that weigh and how you can is a very important process in analytical chemistry. Suppose we want to play from standard solutions what we know from our schooldays again that if we can make some solution of potassium permanganate, this we all know that these are the corresponding standard solutions or K2 CO2 O7.

So these standard solutions how we can do that thing, again we will take a help of the corresponding analytical balance. Commercial the corresponding masses or the weight of these cavemen of 4 or K2 CO2 07 because both of these 2 are solid samples. So how to weigh out any solid sample by this particular process, that means the weighing process how you can weight a metal complex over a Gooche is Crucible, that we can do from this particular analytical balances.

(Refer Slide Time: 12:17)





So analytical balances are used to measure the masses of solid and if required, we can use it for measuring the liquids with high accuracy. So there are different types of analytical balances are available and depending upon these balances, if we are able to measure something which we can consider this analytical balances are measuring from very low amount of this weight, that means in some amount of milligrams to upto several grams, we can use a typical analytical balance and those balances are known as a micro balance.

So what is that micro balance? This can go for a corresponding weight which is basically a micro ground. So micro balance we can use for weighing some amount of mass which is from 0.1 milligram up to 200 grams. So the limit, the maximum limit or the weighing capacity of this particular balances are micro balances which are different from the typical laboratory balances where we can use for weighing them for several grams event for the kilo grams.

So these micro balances as the analytical balances can be used for this particular range of weight. Then we can have semi-micro balances. So all these different balances we can have. So this is the very small weight what we can take out such that we can have several milligrams of samples were weighing like that of nickel DNG complex or some ores we are weighing for taking the analysis. That means 5 grams of ore we can weigh out.

So one such micro balance is fine for weighing the ore as well as this particular nickel complex in the milligrams scale but for the semimicro balance because this is the corresponding weight and the accuracy of this particular weighing process in this micro balance is + - 0.1 milligram. So accurately we can weigh out, that means that 3rd place or fourth-place of decimal what we talk about because we can weigh out say 70.2 gram of weight we can measure in one balance, 70.22 gram of weight we can measure, then 70.2231 we can measure or 70.22315 grams.

So these are the things, upto which particular decimal point we can weigh out by using these balances. And depending upon their range of weighing capacity and the range what we can use for this particular balance that the range is higher, that means we can go for several grams of weight and up to this point, 1 milligram range we can use for this particular weighing. Then semimicro balances we can have a range of 20 to 30 gram of weighing and the curiously is 0.01 milligram. It was 0.1 and it is 0.01 milligram.

Then we can have micro analytical balances where we still go to lower range where the maximum weighing capacity for this is 1 to 3 gram but the range, the decimal place what we can go up to 0.001 milligram. So these are the things how accurately we can weigh out all these samples because that is dependent how small sample we are handling for this particular weighing process.

So these analytical balances are therefore useful also when we go for some drying process or some ashing process, dry ashing process to a constant weight. That means just now what I told you that nickel DNG we are weighing in that Gooche Crucible. So which is having the centre bed and we can filter it out. So the bed is typically covered with that NI DNG2. So this NI DNG2 we can have and the whole thing we can put inside a air oven and heat it up to 110 to 120 degrees centigrade.

Because we were filtering this NIL2 or NI DNGs will formed in the solution and we are filtering. So trapped water is there which is initially it is moist but if we heat it at 110 and 120 degrees centigrade, the water vapour or the moistures present in the sample will go out and we will leave behind with the nickel DNG sample of definite composition and we can analyse this particular one for percentage of nickel present in it.

So what we can do? We can heat it at this particular temperature, then we can cool it for say 5 minute. And then take the weight, take weight. So during this particular cooling process where we can cool because we have heated it up because this is is made up of glass surface, so these are glass and is a sintered place, the porous glass bed is there for the filtering process. So we should use some technique which that means some dry environment or a dry atmosphere we should maintain where not much water vapour or moisture is present such that moisture or water absorption can take place on the surface of this nickel DNG disputed or on the surface of this glass Crucible.

How to do that? So we can have a uniform heating and cooling cycle. That means, you heat it initially for a longer period but after that, for a constant time, say 3 minutes or 5 minutes for heating, and then cooling in this particular dry atmosphere maintained by a desiccator. So we will define, we will see what is desiccator and how we can dry that particular one. So once it is cooled to room temperature, so check how far, that means cooling to room temperature, we will lead to room temperature and then we take the weight.

So we have 1st weight by that. Then what we can do? We can put the sample inside the air oven, heat it to 120 degrees centigrade again for say 5 minutes or 2 minutes, then cooled in the desiccator and take the weight, this is your 2nd weight. And then we can have also the 3rd weight. So doing this process, that means we take the weight once, 2 and 3 and this heating and cooling

process and cooling in the dry environment, that means within the desiccator, we take these weights and this is the weight of the Crucible + your precipitate.

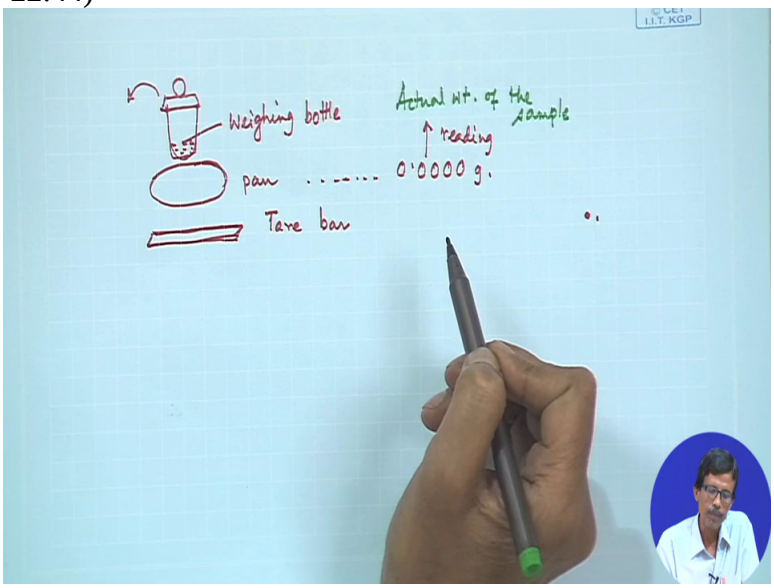
We take these weights and this is the weight of the Crucible + your precipitate and we take suppose we are using a balance of this type where we can have this weight, so we get this and initially we will see the variation in this particular place. That means the 2nd place of decimal. Then as we keep on heating and cooling cycle more and more, we see that this particular point is also constant.

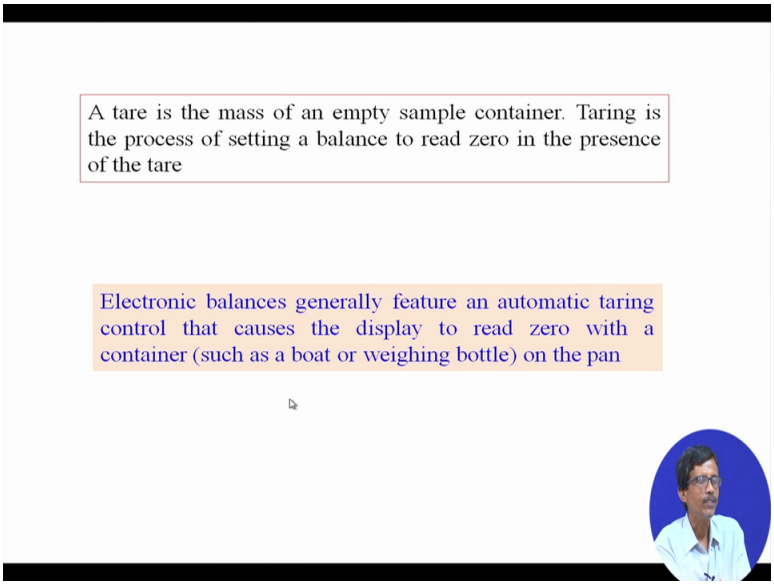
That means constant in the third-place of decimal and finally we should be happy enough that okay we do not go beyond this number 3 measurement, we should not go for the 4th measurement this can vary between this 4th place, this 4th place is varying between + - 2 or 3. We can take that as a corresponding constant weight. So this is known as the corresponding constant weight.

So we should know what is known as the constant weight. So this particular one, so when it is constant to a particular range of weight, we can go for this particular analysis. That means we have what we see that the heating, cooling and weighing steps. So we can have 3 steps, one is heating, then cooling inside the desiccator and weighing steps until it masses become constant to within 0.2 to 0.3 milligram.

So the error limit or the range what we can just tolerate is only 0.2 to 0.3 milligram. Otherwise the weight is constant and that constant weight we can use for taking the corresponding wait for your difference and the difference in weight for the Gooche Crucible + precipitate and the Gooche Crucible will give you the corresponding wait of the nickel and from that, we can found that we can find the corresponding percentage of nickel. So this has all been there in the school's book.

(Refer Slide Time: 22:44)





So we can have a look of that particular book. So another particular terminology what we will be seeing over here is how we can use the taring process. So taring of this when we use a weighing bottle for this particular weighing process, so mass of an empty sample container. What is that sample container? How we know that sample container?

So it is the weighing bottle. So that particular weighing bottle we find there that taring is the process of setting the balance to read 0 in the presence of the tar. So we can have now a glass vial

type of thing. So this is the glass vial which we consider as weighing bottle. So you can have the initial weight of this bottle. Then we take some sample in it, then sample + the weighing bottle.

When we put this balance because the balance what we are talking about so far all these analytical balances are your single pan balance. You put this corresponding weighing bottle or any other container or boat, some weighing boat we can use, the metallic boat or paper boat for this weighing purpose. So when we get that, so initially when we put on the pan, this weighing of this thing will give the weight of the empty weighing bottle.

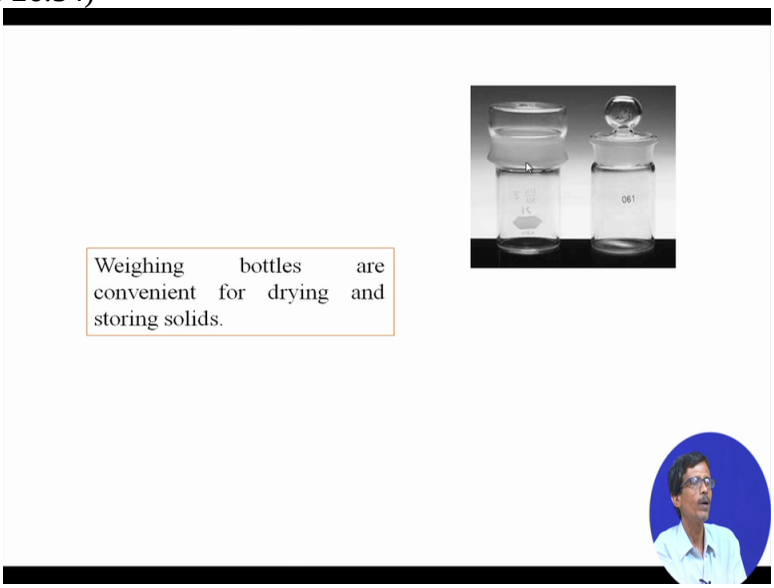
We can take out or remove the lid also, only the bottom part of the weighing bottle became used for weighing process. So this particular fan is there and we can have some reading on the corresponding display of the analytical balance. Then we can have this in front of this balance, we have a bar which is your tar bar. So this particular weight is there and if we tar it, tar means your initial reading will be then 0.0000 gram.

That means it is removing the weight, so far we can have for the weighing bottle. Then this particular process, that means the taring process is important is that we do not have to bother about the corresponding weight of the weighing bottle because everytime you may not use the same weighing bottle for the weighing business. So whatever weight we can take for the sample, it is thus directly we are getting the sample weight.

Now we put the sample inside the weighing bottle and following this tar, after this tar, whatever reading we get on the balance is your actual weight of the sample weight of the sample. So that means without the weight of the weighing bottle. So this is the process. That is why very much useful that how we can go for the taring of a particular sample with the sample inside the weighing bottle.

So electronic balances have automatic tarring control because the bar just now I have drawn that causes the display to read 0 what I just told you with a container, that means the weighing bottle. Container is nothing but your weighing bottle such that the boat it can be aluminium foil boat, it can be paper boat or it can be weighing bottle on the pan. So language wise what we can see is that what is tar and how the taring process can help us to get a 0 reading for the weighing bottle over the pan.

(Refer Slide Time: 26:54)

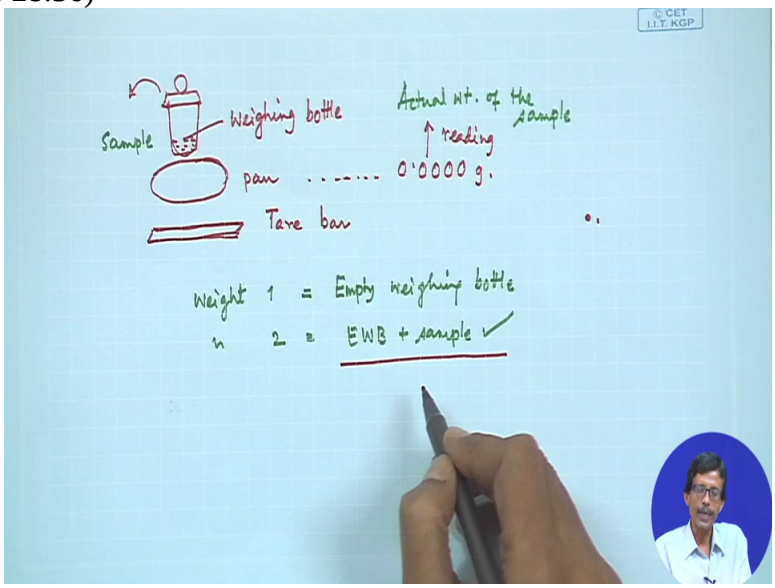


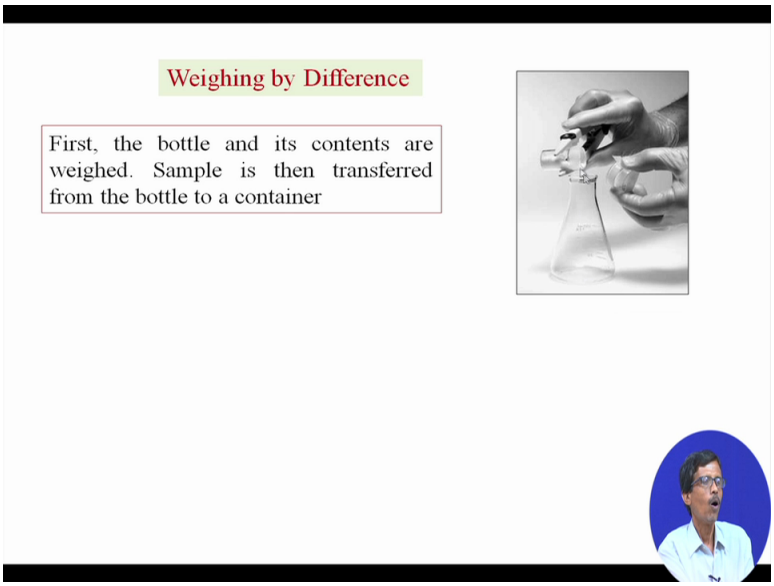
So if you have these weighing bottles and these weighing bottles are very much convenient for drying and storing samples because the entire weighing bottle which is made up of glass like these, these are the 2 examples, the actual examples taken from the school's book again that these are the things and we have to different design. But if you look at minutely, what are the difference between these 2, you have this particular one is the lid is inside this ground glass joint.

So you have to open up this lid and transfer the sample inside it and you can transfer this particular sample for weighing in the volumetric flask also. That people discuss what is known as the weighing by difference. But other one is that you have another cover, this particular cover is of different type where you have the ground joint from outside such that if you use this particular one, so if this particular, so other part can also function as a open weighing bottle.

So this other part can also be used for weighing some amount which is already stored in this particular weighing bottle. So we can store say large amount of the sample inside say potassium dichromate or permanganate what you can take out some of these with this one. So if you open up this and take this as the corresponding another open weighing bottle for your weighing purpose.

(Refer Slide Time: 28:36)





So these we will use particularly we will discuss and we will follow this particular thing to next day that how we can use this particular weighing business by difference. Initially what we can do? We can have 1st the bottle and its constituents. So this bottle is there and the constituents means you have the sample. So weight 1 what we get is equal to your empty bottle and then weight 2 is your empty weighing bottle + sample.

And from the difference, we get the weight of our sample. So in language what we can say that the bottle and its contents or weight if we take this as the 1st weight, and sample is then

transferred from the bottle to a container, this will discuss next day that how we can use all these things, that means initially we can have 2 weight, do not get confused over here. But here what we are seeing?

The 1st, the bottle and its contents, that means whatever I am seeing is that is the 2nd weight, that means the empty bottle + sample, that means the contents of the weighing bottle + sample is taken 1st. And then if we want to remove some of the sample for making a solution, standard solution such as that of our potassium dichromate, we slowly we can remove that and we can take the weight by difference. So this is the technique what we can use that we can transfer this some of these.

So sample + weighing bottle, the 1st weight we retake and this is transferred to this particular conical flask such that some known amount of sample can be transferred to this conical flask and we dissolve it in some known volume of the water or any other solvent to make a solution such that we know the volume of the solution as well as the amount of sample transferred from the weighing bottle to conical flask directly.

We are not using any other container or any other beaker or any other watch glass to transfer this sample from this weighing bottle to this conical flask. So this is a typical direct transfer of sample from the sample container or the weighing bottle to this conical flask where we can make this solution. So next day we will continue this in thing that the 1st thing what I was telling you that you can have only the weight of this empty container, then weight of the container + sample, then if we consider the 2nd weight as your 1st weight, then we can transfer some amount of the sample to make a solution. Okay, thank you very much.