Course on Analytical Chemistry
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Module No 01
Lecture 04: Methods (Contd.)

Hello, good evening. So in this class, what I told you in our previous class that we can have 2 types of analysis, one is the corresponding identification which is known as the qualitative analysis and then we can have the corresponding quantitative analysis.

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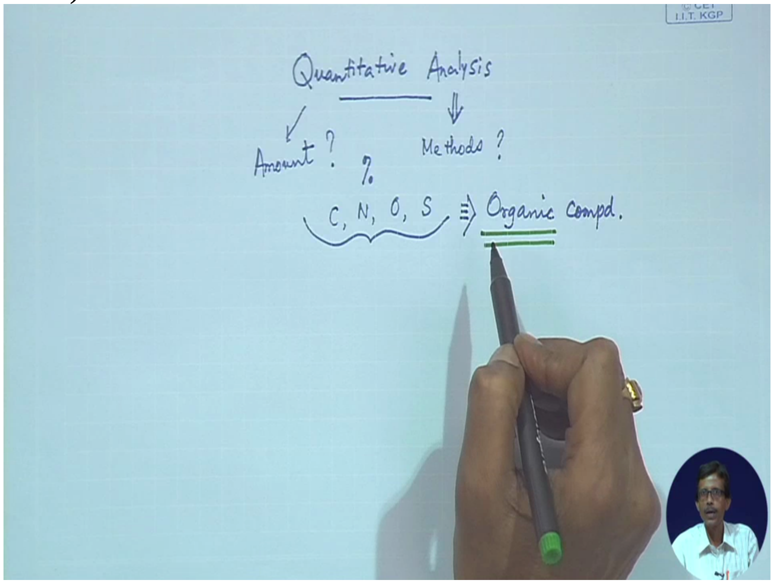

So if we get this particular procedure for any quantitative analysis where we try to know the quantity, so quantitative analysis or quantitative chemical analysis will tell us how much quantity of iron or how much quantity of sulphur or how much quantity of any other material or metal iron is present in this unknown sample. So how you can proceed basically? So you can have some good knowledge of chemistry. It is not that it is not necessarily that you will only expert in inorganic chemistry because some other areas of knowledge is also useful.

So our chemical understanding, the chemical knowledge is definitely useful to go for this corresponding quantitative analysis. So what this, since it is a analytical method or analytical technique, so what are the typical quantitative analytical methods? So how we can develop any method? How to use that particular method? How to apply this method to analyse any unknown sample?

So what are the things we can basically monitor because these methods will ultimately tell us about the amount amount of any of these compositions. Suppose a organic molecule is in our hand and we want to analyse it and the problem, the goal of our analysis is definitely is there. We want to know the percentages of say carbon, of say nitrogen, of say oxygen and of say sulphur. So we all know that these are the corresponding elements which are basically present in some say organic compounds.

It can be a solid or it can be a liquid. So to analyse these organic compounds, so this particular information, we must have that we are handing something which is organic in nature and how we can detect the presence of carbon, the presence of nitrogen, the presence of oxygen and presence of sulphur. So we must have to know that the corresponding method and how we can proceed from one step to another. That means, the flowchart or the flow diagram which can be applied for this particular analytical technique.

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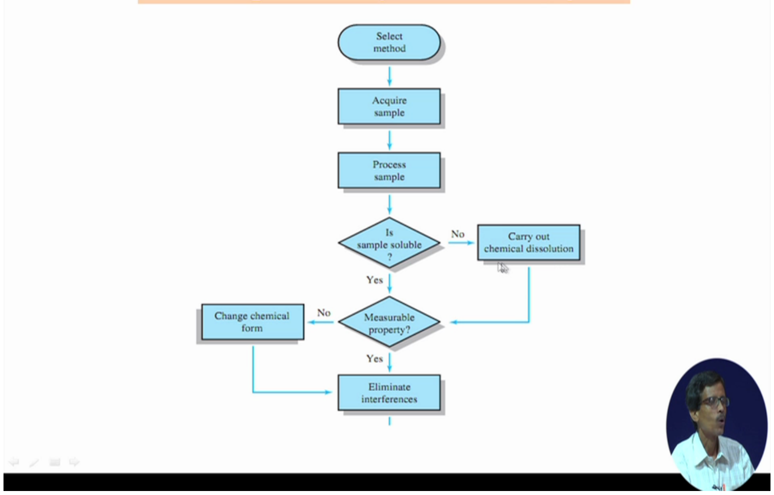

Flow diagram in a quantitative analysis

So in these introductory classes, we just give some idea about the corresponding flow diagram if we can have in a quantitative analysis. So the technique is that we have a quantitative analytical method and how we can proceed from a particular step to the next? That if we can have a method, so 1st thing that we have to select a particular method. So definitely we have to have a corresponding method. And at that whatever methodology, we must focus our attention that what particular method we will utilising to analyse this particular unknown sample.

It can be a simple titration, a volumetric titration such as that we used in the bullet pipette case using bullet and pipette. In a conical flask, we get the corresponding analyte and some reagent we get in the bullet and we titrate it. So some rough idea we can have to select the method. Then we acquire the sample. That means, these are the very typical steps where an analytical chemist can interfere to go for the typical steps. So acquire the sample.

So if we have the sample, so this sample is there and that sample has to be processed. Suppose we get from any rock sample or any sample, industrial sample, or any fertiliser sample, solid fertiliser sample, we have to process this particular sample. That means, pretty meant we call it. This so we have to treat it was so so these are very important steps. Until and unless we do all these things, we cannot get a sample which can be analysed very nicely.

The in one of our previous class, the way we are talking about that if you have a steel sample and nickel is present and suppose you know that you have some idea that nickel we can estimate from our school days knowledge by a gyrometric technique, a gyrometric method can be utilised for analysing the nickel sample by nickel DNG method. So the nickel DNG method is there some so what we can do?

What we learned also from our usual knowledge that if the nickel is present in some volumetric flask or any other reaction flask or even in the test tube, we can add the reagent at DNG and we can get some precipitate and from there, by gyrometric, that means by owing the precipitate after drying at a certain temperature, we can analyse the nickel content of that particular sample. But in most of these cases, these are very simple techniques.

So that is why the nickel sample directly given from the nickel salt such as nickel sulphate or nickel nitrate or any other nickel compound is given to you and the solution is prepared for you and the solution is supplied to you for analysis. But now the real challenge to an analytical chemist is that you have to develop a process where we can treat the sample, we can analyse the samples. That means if you have the steel sample, think of it that the steel sample which is a solid one and I want to get the nickel out of that steel sample in a solution.

So this particular step that means how you can process the sample and is the sample soluble? That means we have to solublelise if we go for a solution technique only because the solution technique is very useful. We can go for volumetric analysis, we can go for gyrometric analysis. If it is soluble, is very fine. That means we can have some measurable property. So what is that measurable property? That measurable property, we can find out if it is giving some colour reaction with some reagent.

That means, with DNG it gives rise to a colour reaction and if the corresponding product of that reaction that means the nickel DNG complex which is beautifully red pink coloured, so that compound which is should be soluble in some other solvent, we all know that that is soluble in chloroform or any other solvent which is similar to that of our chloroform, like that of our dichloromethane. So if it is soluble, so in solution if we can measure the colour intensity. So what is your measurable property?

Your measurable property is the colour intensity and that colour density will be directly proportional to the corresponding concentration of the nickel percent in the unknown sample. So up to this point is good. That means if you have a measurable property, you are happy and you can go you can proceed further to analyse this thing. But if it is not, that means you the thing what you have tried is not able to solubilise this steel sample.

So you have to go for something which can be termed as carry out some other processes for techniques which can go for chemical dissolution. So there are large number of steps or large number of processes available for that chemical dissolution. We will slowly see that how we can process the thing. So chemical dissolution because in this particular case if it is a steel sample, if we treat it with some acid or some mixed acid, say concentrated sulphuric acid or concentrated nitric acid, if this acid can carry the dissolution process, that means steel can be attacked by this as its, you get the solution.

But if it is not, we can have to have some other technique. That means, the fusion technique, alkali fusion technique or any harsh conditions or high-temperature conditions can be applied. So all these things can come under this chemical dissolution step. So if the chemical dissolution step is satisfied, we can reach over here, that means in the solution state and in the solution itself is coloured, we can proceed directly from there and if it is not coloured, you can have have, we have to have add some reagent and that reagent can give rise to some colouration and that we can measure.

So if it is yes, then we can eliminate the interferences. What is that? That if nickel can interact with nickel ions, Ni2 + can interact with the DNG giving life to the corresponding colour sample or the coloured complex which we can measure. But if some other metal ions are also present, we all know that the steel sample can have some other metal ions. It can have the chromium or any other metal ion. If those are also can interfere, those can also interfere the corresponding estimation, that means it can react with the DNG.

It may not give the same colour but it can consume some amount of DNG. So that will interfere our measurement. So these, all these interferences we have to eliminate. Then only you can determine that only nickel DNG can be estimated for our analysis. But if we cannot have a measurable property like my simple addition of DNG, if it is no in this flow diagram, we change the corresponding chemical form.

Suppose we are analysing not nickel and some other element like that of our aluminium is present in some other source. So your analyte contains some aluminium. So if it is no for DNG, so we change the chemical form. So aluminium will take some other reagent which is 8 hydroxyquinonin. That we will discuss afterwards.

So you have to go for some other reagent. That reagent can give rise to something which can interact with the species and we can get something which is a measurable quantity, it may not be coloured, it is insoluble precipitate and if it is insoluble precipitate, that can be a way out and go for the corresponding estimation of aluminium.

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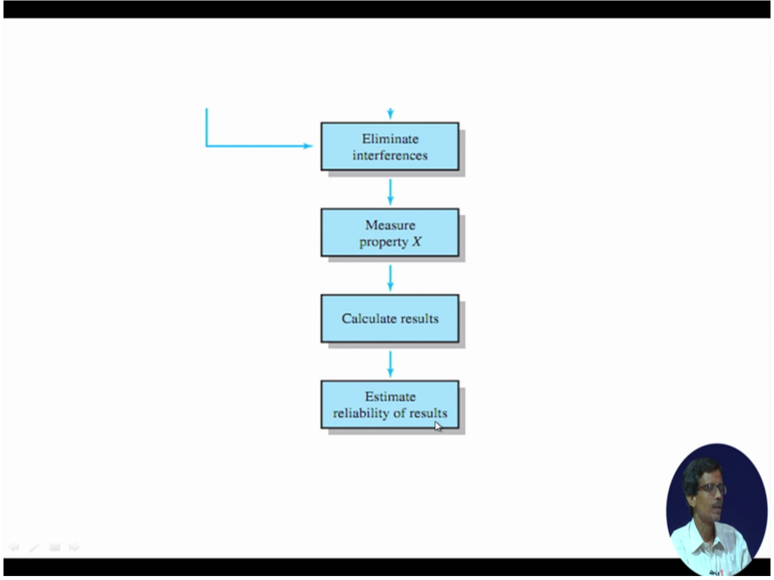

So after elimination of these interferences, what we get there that once we eliminate that interferences, we measure the property X. So measurement of this property is very unique technique for this and this can be your colour, this can be your weight, or this can be any other thing. So once we measure the property, the intensity of the colour will be related to something which can be considered as the corresponding photometric data as the absorbance.

So absorbance A can be measured and that absorbance A is your data related to that of your concentration and we get the corresponding results in terms of the absorbances and in terms of the corresponding concentration of the nickel or any other element. So we calculate the results. Then we go for the estimation in terms of the reliability of the results. That means the repeatability of these results.

We should not be happy by doing someone measurement because errors are always there if we do not have a good calibration is also. So we will also learn slowly that what is called calibration. We can have the corresponding calibration curve and how we can utilise this calibration to estimate the corresponding unknown quantity. So reliability of the results will also tell us the corresponding estimation. So we can go simply for 3 different measurements.

So we take 3 samples of this, 3 steals samples. We go for the dissolution, we get in solution, we estimate is corresponding colour and the colour intensity for these 3 samples we can measure and we take the average. If the average in are good matching range, then we can have some reliable data for these 3 measurements and we can consider that we have reached some estimation which is some which is giving some good results for our corresponding estimation of our unknown quantity.

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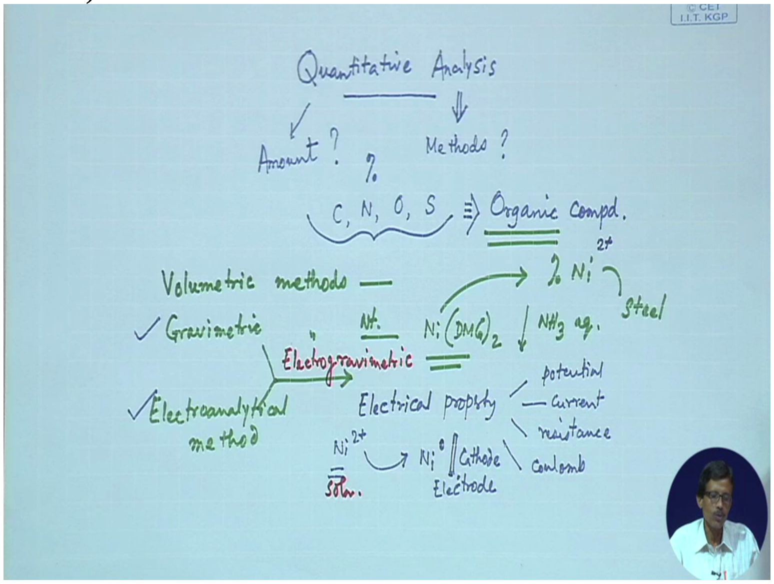

So this quantitative analysis is therefore very much important for our volumetric methods and then we can have the gravimetric method. Just just now as I told that we can have the nickel DNG complex which is precipitating out from inaqueous medium in ammonia callacous medium because ammonia solution aqueous ammonia we have to add. So nickel DNG will be precipitated. We take the weight.

So weight of this nickel DNG precipitate can tell us about the corresponding percentage of nickel in the unknown sample which is our steel sample or any stainless steel sample. So when we talk about the weight, when we talk about the volume of something which is directly proportional to the unknown concentration of nickel, we get the volumetric analysis and if we can tag this, that means this particular technique with some electrochemical method, we can give rise to something which is known as electro analytical method electro analytical method.

And in that particular electro analytical method, so mass of the analyte we can analyse. So this is typically a simple gravimetric method from the addition of DNG to nickel 2 + and as a percentage of nickel we can analyse it but in this electrochemical method, what will be measuring? That means some electrical property we will be measuring. So this is a new quantity. So we can measure the electrical charge or electric current.

So the potential we can measure or we can measure the current, we can measure the resistance or we can go for the coulomb count. That means, the correspondent quantity of electric charge which is being carried out by some metal ions and all these things. So if we have a cation, that means this NI2 +, the same sample NI2 + is there and we all know, it can be reduced by some electrochemical cell if we can have some electrochemical cell.

That is also from our school days knowledge that if we have some electrochemical cell, we have the cathode, we have the anode we all know. So to estimate NI2 +, how you think of some process or some methodology which is can be useful for our quantitative analysis that NI2 + is there and this NI2 + we have to reduce therefore to get nickel 0 and this particular nickel 0 in its metallic form can be deposited on some electrode surface.

So if it is cathode, so on the cathode surface, if it is there, so if we have the cathode surface, so on the cathode surface, so it is electrode. So electrode is your cathode and this nickel can be deposited on the cathode. Now how we can think of it as the combined, these 2? That means, a gravimetric analysis or an electrochemical analysis or electro analytical method which can be combined. So this thing is there.

That means we utilise the corresponding passage of electric current on some electrochemical cell and we can have some monitor of these, how much are than how much current and all these things are there. But ultimately if we focus our attention on the corresponding reduction of nickel 2 + to nickel 0, what we see that this nickel 0 is there and which is deposited on this thing. That means, the cathode is gaining its weight.

So before deposition and after deposition, there will be a change in the weight of this cathode material. So if we can get the corresponding weight difference, that means if we go for the corresponding weight change during this electrolysis process, that means the nickel 0 is forming from NI2 + from the solution. It is there in our solution and which is on the surface. So this change in weight basically gives rise to a combine from which can be termed as a corresponding electro gravimetric method.

So electro gravy metric is the combined form. So its realising the technique or the knowledge of gravy metric analysis to that of our electroanalytical method to give rise to some electro gravy metric method. So a new methodology we can follow which is known as electro gravy metric method. So that gives rise to some understanding that how we can utilise this particular thing. That means, the sample where we can have the corresponding sample. So now we can give rise to something where we get the analysis of a typical real-life example because we are trying understand the nature. So we will take some example, so definitely it is from textbook also.

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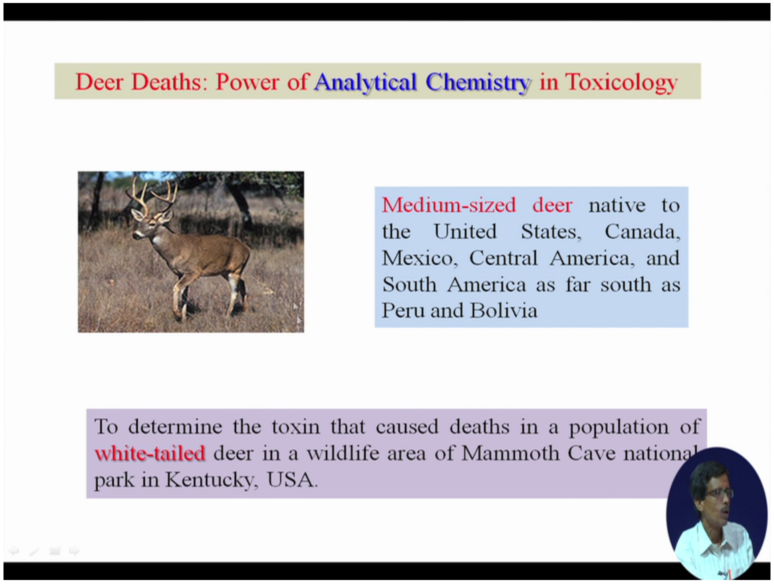

Deer Deaths: Power of Analytical Chemistry in Toxicology

Medium-sized deer native to the United States, Canada, Mexico, Central America, and South America as far south as Peru and Bolivia

To determine the toxin that caused deaths in a population of white-tailed deer in a wildlife area of Mammoth Cave national park in Kentucky, USA.

So it is there in the book that all these applications that some dear, so is the animal, death of some animal is cost by some pathogens. So is the toxic effect of some material. So basically we call these as the typical environmental disaster. So this particular thing has been has happened in US, in Kentucky. So there, people found that some deers we basically if we consider that a deer park is there and by taking or grazing grass, the deer death is occurring.

So how analytical chemistry can be useful in terms of understanding the toxic effect of something. So toxicology as I told you in our previous class that how analytical chemistry is helpful in understanding the toxicological effect or the toxicology. So power of this analytical chemistry we can utilise to understand these. So these are the corresponding deer which is available in this part which is a medium sized deer which is native to United States and some other countries.

So they have is white tailed deer and the death of this white tailed deer is due to consuming these grasses where something has been added. So we all should be very much careful what we are consuming. So is related to everything, is related to environmental chemistry, is related to that of these things and when particularly we are studying the toxic effects, something some unknown quantity or unknown compound, so this is coming under the purview of toxicology.

So the toxic effect of this compound what we can utilise for analysing this things. So the toxin, why this is causing? Why the death of deer is causing? So how we can determine the toxic effect of something which is the uhh which is given to this particular part. So white tailed deer in a wildlife area of Mammoth Cave National Park in Kentucky, US is happened. So this is related to something and people tried to understand it in a different way.

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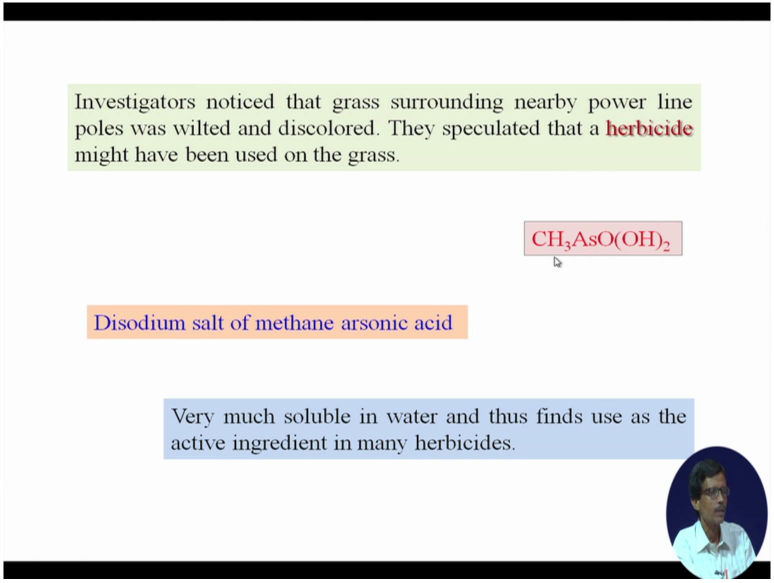

Investigators noticed that grass surrounding nearby power line poles was wilted and discolored. They speculated that a **herbicide** might have been used on the grass.

$CH_3AsO(OH)_2$

Disodium salt of methane arsonic acid

Very much soluble in water and thus finds use as the active ingredient in many herbicides.

That means they called the investigators. So investigators are asked to understand how this grass surrounding the corresponding power poles basically the grass is there and if we try to monitor the corresponding growth of grass, we can put some herbicides. So they basically suspected that some herbicide has been sprayed on these grasses and this grass is contaminated with this herbicide and this herbicide concentration or the nature of this herbicide can be lethal to the deer.

So deer is consuming something which is getting contaminated from this herbicide though we are utilising this herbicides to reduce the growth of these grass and some point and there are other types of herbicides we all know. So if we can have something basically this has been sprayed over there which is an arsenic-based herbicide.

So there comes the knowledge of typical chemical knowledge, that means understanding this particular compound because occasionally we are adding for something we are utilising this for, spraying this on the grass surface but the compound whether it is deadly to the human being or whether it is deadly to the deer, that we all do not know. So this is very interesting that we are talking something is arsenic because we all know that our environment, nowadays we talk about the arsenic contamination or arsenic pollution in the groundwater also.

That we will discuss at some point of time. That is why I have chosen this example which is a arsenic bass compound and this compound is nothing but some arsonic acid. So it is not a typical arsonic acid because it is not a typical inorganic compound. Some group is attached to wait. So is a methane arsenic acid where a methyl group is attached to arsenic and is not that free acid is or (())(24:51) disodium salt. So unless S double bonded O and OH.

So OH will be substituted by ONL like your sodium salt for acetic acid we all know, sodium acetate from the acetic acid. So if we have the methyl arsenic acid which is CH 3 As OOH whole twice. So disodium salt is there. So this herbicide is nothing but is corresponding disodium salt or methane arsenic acid. So we see that this particular herbicide has been converted to its corresponding acid form to its disodium salt form.

So this disodium salt is very much soluble in water and that is why it is an active ingredient to these herbicides because we put in water and we spray it. So on the grass surface, it is sticking. So the sticked grass surface is there. After evaporation of water, these compounds forming some very minute layer on the grass surface and deer is basically consuming this particular compound. So deer is consuming this compound and that is why after some time after some days, the death is taking place for the consumption of this herbicide.

So basically the toxic effect of this compound on the health of the deer is the corresponding contamination and how to identify this particular arsenic? So arsenic is going to the body of this deer. So you have to analyse it. So is a very typical task because you get a sample of dead deer. So dead deer is in your hand. And how to analyse the arsenic which has been given to this particular deer?

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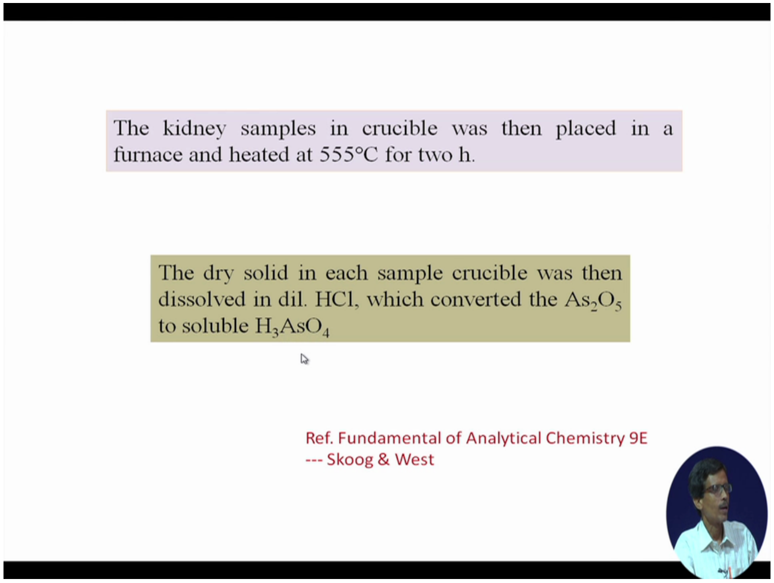

The kidney samples in crucible was then placed in a furnace and heated at 555°C for two h.

The dry solid in each sample crucible was then dissolved in dil. HCl, which converted the As_2O_5 to soluble H_3AsO_4

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--- Skoog & West

That means unknowingly, it has consumed this particular arsenic and this particular thing, how we can monitor is that a very simple chemical technique that means the we will be taking the corresponding kidney samples of this dead deer because anything we consume, we all know that is going out from our body. So it is going through kidneys. So kidneys of these deer are also contaminated due to these arsenic samples.

So the chopped kidney samples so the kidney samples which is organic matter, basically a fleshy matter if we can take it in a crucible and is placed in a furnace and hinted at 550 degrees centigrade for 2 hours. So this is the recipe, this is the methodolgy, this is the technique what we can use to identify this particular stuff a very high temperature. So basically idea of is that that you have the disodium salt of arsenic acid.

So arsenic acid will be converted to arsenic-based oxides. So at high temperature until and unless we do that, so very high temperature, these also give rise to some idea that any biological sample, any living sample which has been taken as the kidney of the deer if we burn at a very high temperature on a crucible because the crucible can sustain, it can be your porcelain crucible, it can be your vitriosil crucible which can sustain a very high temperature.

So all other organic matter will be burnt away as if we burn in air because this is if this is the air furnace or some special type of furnace where airflow is there, it can be utilised for oxidation of the organic part So carbon will be oxidized to carbon dioxide, hydrogen will be converted to water. So we will be leaving behind with only the arsenic. So that arsenic will be converted to its corresponding arsenic oxide and this arsenic oxide which is getting as AS2O5. So what we will be getting?

So arsenic in the pentavalent form so pentavalent arsenic is forming over this due to this particular heating procedure. So a mass is available. So after heating for this crucible for 2 hour, we will leave behind with some amount of dry solid at the bottom of the crucible. So what we do? We cool the crucible and we dissolve that particular sample with dilute HCL. So dilute hydrochloric acid will react directly with this oxide form to give our soluble H3ASO4, the corresponding acid related to arsenic in the pentavalent state.

So we know that this anion ASO43 - is the corresponding arsenate ion. So is the corresponding arsenic acid which is giving us that arsenic which is present in the pentavalent form. So this is there. So we get something where we have this. So arsenic was present. So what are the changes? So chemical changes try to mind, keep in mind that the what are the chemical changes are taking place.

We were having the corresponding disodium salt of the corresponding acid, arsenic acid. We were now converting this particular form and now we are having this H3ASO4 which is coming from the kidney sample of the dead deer. So how to analyse this? So this can be a simple salt which we get it because this is coming from a corresponding reference of the book which is Shoog & West, the book is the fundamental analytical chemistry book.

You can go through this particular book for this particular example. So we have reached over here is also up to this point.

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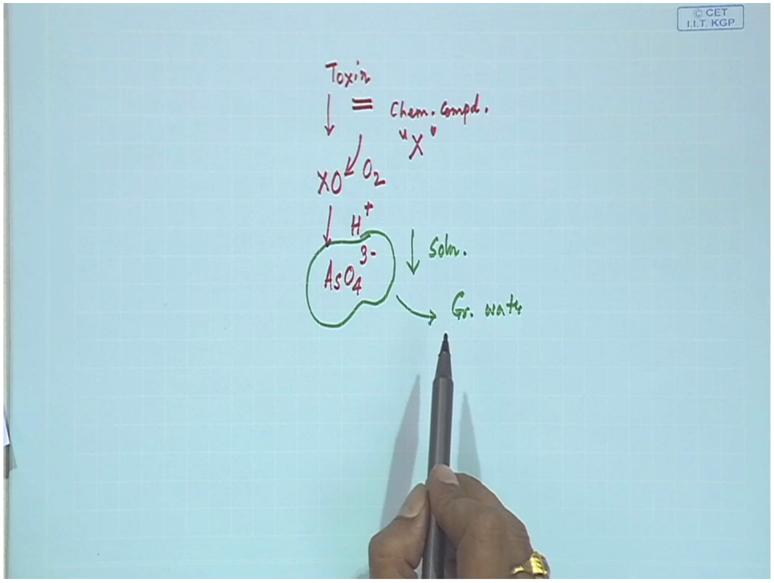

So that we will discuss in our next class that we have some toxin and we try to analyse this particular toxin. And toxin as a typical chemical compound, so we have the chemical compound and idea is that chemical compound if it is some element based, the element X is arsenic over here. So we make some oxide because we are heating in air. So as is there, O2 is there.

Then oxide is there and we all know that if it is dissolved in acid, we get something where the corresponding oxo acids are forming. So we will having some ASO43 -. So our now challenge is that how we can analyse the form AS03 - in solution which is very simple to go for it but we will choose some special techniques for this. So this is related to nothing which is the contamination of arsenic in our ground water also.

So that we will discuss in our some other classes that how the groundwater if it is contaminated with arsenate can be analysed by using this particular technique. Okay? Thank you very much.