

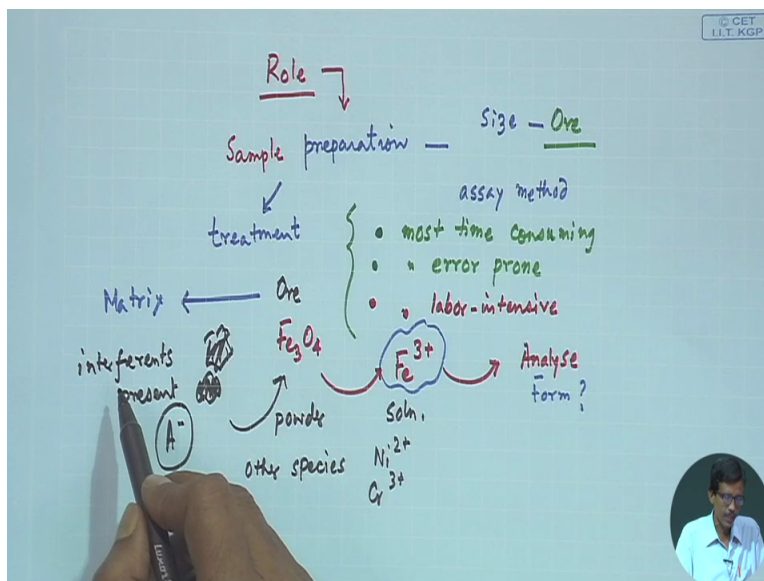
Course on Analytical Chemistry
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Module No 02
Lecture 06: Role of Analytical Chemistry

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Role of analytical chemistry and techniques

Introduction of the tools, the techniques used, and chemicals that are needed

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Welcome to this class of analytical chemistry where we will be discussing about the individual role of this particular subject. That means where we try to understand the role of analytical chemistry and the different techniques because if we try to find out a particular technique which

we can apply for any real-life problem-solving affair that this particular technique should be comfortable with that particular chemistry what can be utilised to solve this analytical process.

So there we get the different tools basically, the different techniques and the different types of chemicals that will be needed for a particular type of analysis. So 1st thing or weekly we should discuss this particular role in terms of the sample. And already we were discussing about the corresponding sample and the sample preparation from the real-life examples what we have seen, the starting from the ore to deer kidneys, how we get the corresponding samples for this particular analysis.

So the different sizes, that means if we have the ore or any other thing that we all know that the sizes are different. So we choose that particular size that size is important and in which particular form those species are available that we should take. So this particular sample preparation should be associated with something which is known as treatment or sometimes it is pre-treatment, how we can utilise the knowledge of chemistry for the development of a particular assay or the assay method we call because these are the most tedious process because to get this sample starting from say some sample is if it is ore, very big sizes, so we have to break them, we have to pulverise them, we have to make as the powder.

So during this particular process, definitely for the sample preparation, we can find out this as the most time-consuming step. So most time-consuming step is not that you get it in the final form and you determine by some titrametric method or some gravimetric method or some spectrophotometric method, the typical unknown concentration. So the sample preparation where the analytical chemist should intervene nicely for this corresponding preparation for the direct or be ore sample and it is also the corresponding thing where we get that it is most error prone also.

That means if we are not careful enough to take the required precautions, so something can happen such that our desired form of the analyte coming from the raw sample is not possible to achieve it and the 3rd one is also it is most labour intensive. That means we must have to pay some time as well as some labour for that. This is a typical or a tedious process.

That means if we can have something, that means this ore, suppose we are handling something where we have the Fe_3O_4 and our ideas should be there, we should use this, how we can convert

this as Fe^{3+} which we can analyse and which we can determine the corresponding unknown concentration. So this particular thing, that means but if we can get these that is a bigger chunk of these ores are there, so bigger chunk of these ores.

So how to get this initially in the powder form and those powder forms, you have to grind them and then we can take into some solution state. So this Fe^{3+} will be in solution. So during this process when we are looking for Fe^{3+} but this particular ore sample can have other species present. And sometimes some valuable components are also present.

So during this particular process, we should not lose any analyte or this particular analysis because if some other metal ions, Ni^{2+} is there or chromium $3+$ is there and if we focus our attention that we should only get this particular Fe^{3+} nicely but during this particular process, that means the dissolution, evaporation and all this that some amount of Ni^{2+} or some amount of chromium $3+$ can also be unavailable from that particular solution medium.

So once we get that and which particular form, means if Fe^{3+} is present, then we can directly determine Fe^{3+} in the solution most of that means, the form of the analyte is also important. Also sometimes that we had some reagents and the reagent can attack the particular species, that means Fe^{3+} or As^{3+} or any other species to form some coloured species or some insoluble precipitate.

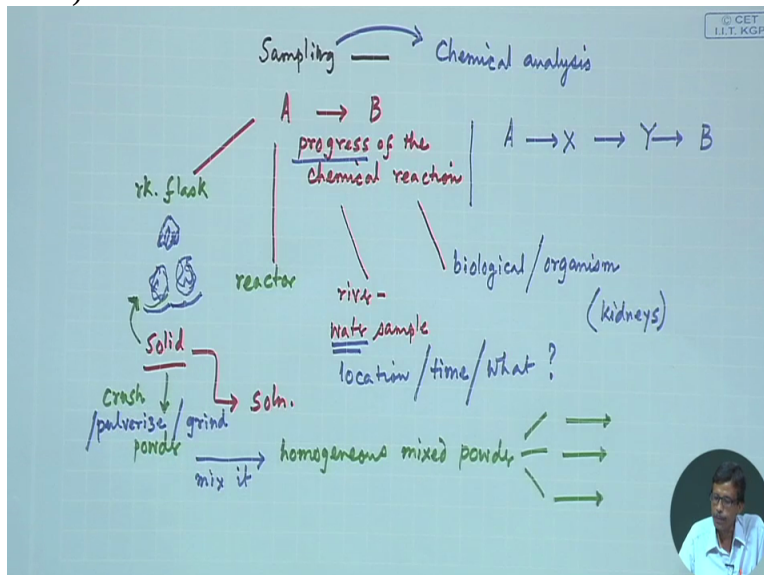
So this particular analysis, that means the analyte what we get that? So the form of this particular analyte is important for a typical assay process. So if we get that, a complex ore and that complex ore if we know that sometimes is very complex, so we call it as the matrix. So in that particular matrix, so this matrix we want to solubilise it such that we get it into the solution. So there will be some interferences and the interference present in the matrix can also make our life difficult to get this in the solution.

And already if we have the interference, interference present, so sometimes due to the treatment by the acid, so if we have the ore matrix is being treated by the acid, so all these acids can have typical anions like chloride, like nitrate, like sulphate and all this. So these were not originally present between this sample. So due to that particular process, this addition of these acids will all be there and this will be the new anions available in these solutions because this particular oxide

when treated by acid, it is not forming any anionic anions over there because all will be converted into water molecule.

But if we treat this with some inorganic acids, so anions will be there so they will also interference. So already we have some amount of interference present over there any bit difference can come over there to the process of this sample preparation.

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So what we see there that this sample preparation can have something how we do the sampling process, the sampling? So this sampling is also important when we take the order sample for the analysis of iron say of any ore and this sampling is also important if we try to monitor a particular progress of the reaction. That means some conversion is taking place in some land or in some other process that a chemical reaction A is converting to B.

So the progress of the chemical reaction, so progress of the chemical reaction can be monitored which is a typical challenge. So sampling is a real challenge for any chemical analysis. So if we try to do any chemical analysis, we should have the sample in our hand and how to get these samples from any biological source, for any ore or any rock sample or any soil sample or any water sample or the air sample? So this will further be complicated if we tried to monitor the progress of a chemical reaction/

So this progress what we see that this particular progress and how it is converting A to B, so A is not directly converting to B, so if there are some intermediate species like X and Y. So if this X and Y is forming and those we are not able to monitor, their concentration but we are directly converting these to the corresponding final form of B and this final form of B we try to find out by looking at the corresponding concentration of B which is forming from A.

So there are several things what can happen for this particular reaction. One is that if we can have a reaction flask and within this particular reaction flask, the reaction is taking place and this particular reaction we will not handle in such a way that A is directly converting to B without going through the intermediate X and Y. So we can monitor a particular reaction in a flask in a lab.

Then if there is a reactor in the industry so or in a process plant in that particular industry, so reactor is also responsible for any conversion, chemical conversion of A to B. So we can monitor that and in between, that means when the reaction is taking place for this particular process, suppose a metallurgical process, a furnace and all other reactions are taking place within that particular furnace or the reactor, so intermittently or continuously we can monitor the progress of the reaction where A is not fully converting to B or A along with X and along with Y, they all these are present in a mixture.

So typically A is not present, the entire concentration of A is not there. So concentration of A is decreasing but we are not able to get the right concentration of B whatever amount of A is being lost. So these intermediate concentrations are there. If we are unable to detect the concentration of X and Y, we will find that A is being destroyed but is not happening over there. So continuously we can also monitor the decrease in concentration of A and increase in concentration of B.

So this particular sampling process for this chemical analysis is also important on an open field. Suppose we are taking the river water, the riverbed is our environment and from there we try to get the water sample. So from the river, we can collect the water samples but this is directly related to the environment. So the location is very important, the time is also important that when we are taking this particular sample throughout the year, throughout, in the month or a particular day, in a particular time.

So where we are taking, when we are taking and what we are taking along with the water sample. So if it is contaminated with some species, we will be trying to look at that particular species from those river samples. So sampling for the river is also very important. We have to go in the field, in the riverbed and we gather the corresponding river samples for the analysis.

Then lastly, the other sample which we have seen the example in case of the death of the deers that dialogical samples, so biological samples in a particular organism can be utilised the way we have seen in case of the kidneys of dead deers. So this particular case, that means when we get the ore sample also the nature of these samples are important. That means we are handling some solid samples and the solid samples will be taken as the solution and also we will see when we go for a gravimetric estimation, this particular solid sample in between it convert to a solution.

Again we go for some precipitation. So solid we will be getting and that solid can also be analysed for the presence of that particular species such as the ferric ion or any other metal ion in the original solid sample. So what we do? We just get this particular solid and one other important factor for the solid material of different sizes, the chunks and all these if we are getting those as from the real-life samples, so these results chunks or the bigger size of the solid material are not homogeneous in nature.

So to bring homogeneity into this particular sample, how to make this particular homogeneous sample because we converted this particular solid sample of different sizes to a powdered sample, so this powdered sample making this as a powdered sample is important, so we go for crushing. So crushing is there. So we crush that, then we can pulverise it or finally we can grind them.

So to make these in powdered form so how to get this powder basically? So what are the instruments and what are the techniques we should follow to get a powdered sample from a very real life solid sample of different ores. That means the physical characteristics of this particular solid sample is different from the sample which is in the powder form. So we are changing the form.

So crushing, pulverising and grinding, so these are the 3 steps what we can apply on this and we get this powder and then we mix it nicely. So mixing of these, that means if one part of the solid,

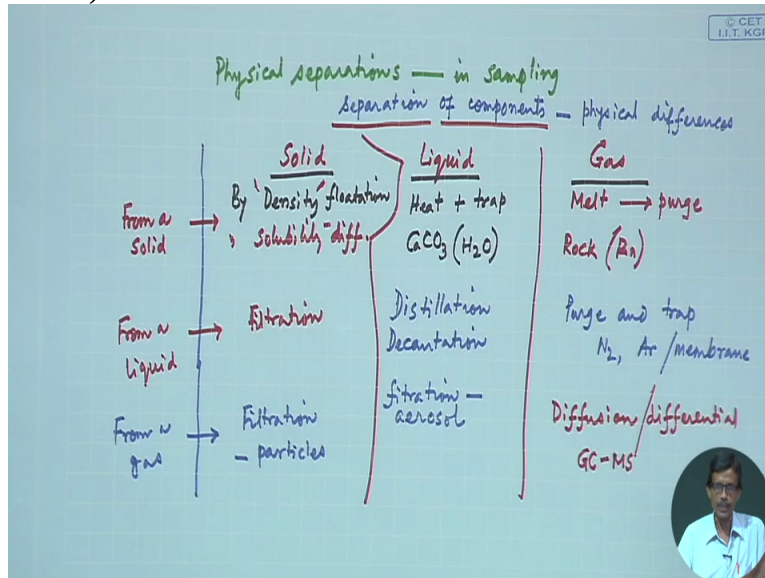
so if, if we have one part of the solid and all this because the geologists what find all these samples so they are not homogeneous. So in homogeneity is there.

So when we are grinding, so one particular part because these are all the crystallised form so one particular part can have some elements or some chemical elements of high concentration than the other form. So when we grind this particular part, the concentration of one particular species is high over here than the concentration of this particular part. So if we entirely make a powder and then the whole powder from this part, from the spot and this part, we mix it, we get a mixed powder form and this mixed powder form will be homogeneous in the solid state.

What we will be looking for? We will be looking for something where we get homogeneous and which is mixed powder. So this powder we can store basically from the different sites, like the collection of sample from the riverbed, wherefrom we are collecting the particular sample which river, when, where and at what particular time. Similarly, the source of the solid samples of these ores are also important and we make these, so we can have this powder sample.

So if we have 3 different samples from 3 different origin, so we make them powder and we can store it because in the next step we have to set the corresponding process how we can analyse this powder form for their different analyte, the presence of these different analytes. So before going for this grinding of this powder, so solids we can have. Now we can bring two other forms, physical forms of these samples.

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That means if we have these, that means during the sampling process, whether it is possible to go for the physical separations because ultimately what we can do that physical separations in sampling we are still with that particular topic of sampling. So in sampling, the physical separation because ultimately what we do is now known to us, is very much clear to us is that we can go for some chemical treatment and chemically we try to identify the unknown species.

But the physical separation this can be very much useful if we can have all these things. That means if we can have solid, if we can have the liquid or if we can have gas how these are there? Because if we can have one solid and if we try to separate from other solid, that means the separation, so what is that? So it is separation. So separation of different components. So if there are component A, component B and component C and all of them are solid, then how to separate these components?

So separation of these components which are present within the samples due to the corresponding differences in the physical characteristics, that means the physical differences are there. So what about this? That means if we have the difference, that means if solid is mixed with liquid, and liquid is mixed with gas or gas is mixed with solid and gas is mixed with liquid we can separate it out. But if there are some solid and we try to separate it from a solid, how to do that?

So separation of solid from a solid, that means if we see that the different solids are present and the colour of those changes and their corresponding nature is also different, that a particular scientist who is devoting with the geology or geophysics, so geologists can see the thing and sometimes making this particular thing with some typical observation, by microscopes and all.

So looking at this material, they can separate out some particular part from the other. But in actual practice, the science behind that particular separation is something else. If we just look at the density, density of one solid part from the other is different. That means a particular part they will be occurring in same area some solid matter containing iron, some solid matter containing copper and some solid matter containing more precious metal ions say like Palladium or platinum.

So these are present in the ores but their density will be different. So by density difference, using the corresponding parameter is the density difference. So what we can do? We can do this separation by density flotation. So what is that? So density flotation is that if we choose some liquid, and on that particular liquid, the depending on its corresponding density whether it can float or it can submerge in that particular liquid, we can differentiate it out.

So if we have a particular density column where we have a density differences in the liquid over, so using that particular thing, we can use that thing like that of our liquid but solid as a solid. So solid vs solid separation we just simply utilise the corresponding density of the solid material, the difference in the density of the solid material or by solubility difference. So one particular part is soluble in one particular solvent, other part is not.

So if there is any solubility difference, we just separate it out from a typical solubility difference for one solid from the other. So next is that how we can separate a liquid from a solid also? So liquid is being, suppose it is being trapped, say a sample like calcium carbonate is in our hand and that calcium carbonate has some trapped water. We all know that this we call is as the water of hydration.

So this trapped water we can take out, so this as the corresponding liquid. That means the water vapour can be liquid from the solid of calcium carbonate. So we simply heat the material. So heat the material and trap the gas, so this is the usual technique of getting this particular liquid

form of water from the solid calcium carbonate. Now if we have gas and we want to separate gas from the solid material, what we can do?

That if there is a rock sample and this rock sample can have some gas, say we all know that the gas radon is trapped within some rock, similarly we all know nowadays that the corresponding gas available from the cell, so cell gas we call, then we know that the methane is available from the coalbed. So coalbed is trapping the methane gas. So this is some particular physical technique is utilised to get that.

But in this particular case, if the rock is if we get some bore of that rock, and the if the gas is coming out from those bores, we can collect those gases very nicely. Otherwise, we have to melt the solid. Once we melt the solid, and then the gas is available and then we purge that particular gas by some inert gas from there. So next is that how we separate the solid from a liquid? So the 1st thing is then, the solid we have to separate from liquid is the filtration, the way we remove the precipitate from the solution.

Suppose silver nitrate is added to a chloride solution, so silver chloride is being precipitated as the precipitate. And if we filter it out from that aqueous medium, we get the corresponding solid as silver chloride from the aqueous medium which is the water medium. Then liquid from the liquid. That means two liquids are there and if they are miscible and if they are immiscible, we can have something which is called as distillation because they will have the difference in boiling points.

So distillation is one particular technique and sometimes we find that if there are layers, so we can remove them by using say separating funnel we all know which we can consider as decantation of the 2 immiscible liquids using a separating funnel also. Then gas from the liquid is also like that of our solid, from the solid.

Then we have to purge then and trap technique, how we bubble some inert gas, see nitrogen or argon over there and the gas what is coming out will be the gas which is we are looking for for this narration and that is if we cool those gases we just get if they are cold enough because we can liquify them, otherwise this gas can be separated from those inert gases. Sometimes, the

membrane, the gas permeable membranes can be used which can selectively permit one gas to pass from the other gas.

So this membrane will be utilised for the separation of the gas from the liquid also. And the last one is the separation from a gas sample. So what we are doing for the sampling process? We want to know separate the solid from the gas sample. So again, a different type of filtration technique will be used. So solid we can have the suspended particles. So suspended particles can be filtered out from the gas.

So the only thing that like that of our filter paper what we are using for separation of liquid for the gas, a different type of filtration mechanism we can use such that gas can be as the corresponding medium where from the solid can be separated out. And similarly from gas, liquid can be separated out such as our aerosol, aerosol separation. So liquid aerosol can be removed by filtration again, again a typical full narration technique.

So this is a very useful one. So we will discuss in detail also about how filtration is useful for all these different complicated types of separations. So what we are looking over here is our separation of the components. So we should not forget throughout this tabulation, that means what we are tabulating over here is the corresponding separations of all these things. So filtration of the aerosol and lastly the gas from the gas is by diffusion.

So the process what can be utilised is the diffusion technique and this diffusion is different from one type of gas to the other is the differential diffusion. So this differential diffusion can be utilised the way we use it for a particular technique, instrumental technique. We know that this particular diffusion for gases is also possible if we look at the corresponding chromatographic technique, we know that this is gas chromatographic technique.

So this gas chromatographic is the separation of these through some diffusion of these different gases where we can have a carrier gas and carrier gas is purging the other gases. Then mas wise, the individual gas can be separated. So if this is coupled with a mass spectrometer, the corresponding instrumental technique is the GC MS technique and that GC MS technique is based on the contribution of one particular gas from the other gas from which we want to separate this particular gas by this particular differential diffusion technique.

So this differential diffusion technique we do very much useful for separation of one gas from the other because ultimately what we will be looking for, we will apply knowledge of analytical chemistry to identify the separated gases. So we just separate all these out and then we apply this particular technique or one other particular technique to identify this gas on the gas the gas mixtures were present.

So the next day we just follow up these by utilising of the different thing, that means the different technique, that means the different tools and the chemicals if we can use for this particular technique of analytical chemistry, thank you very much.