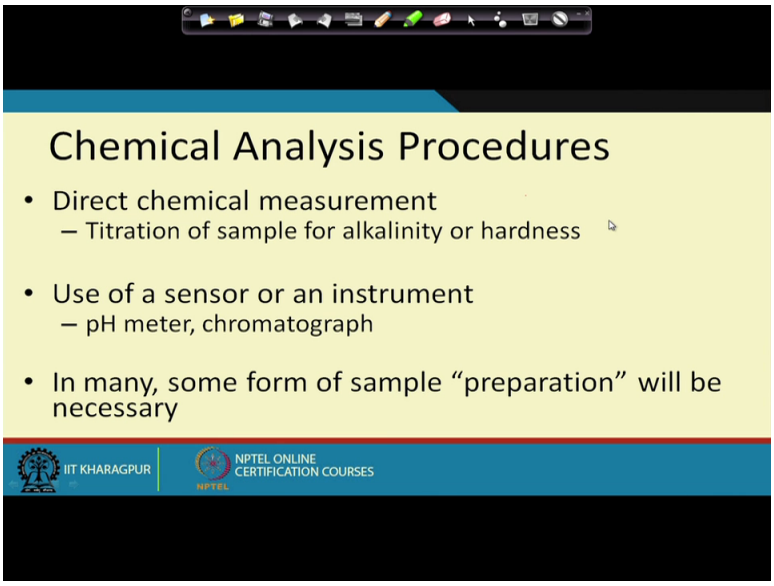
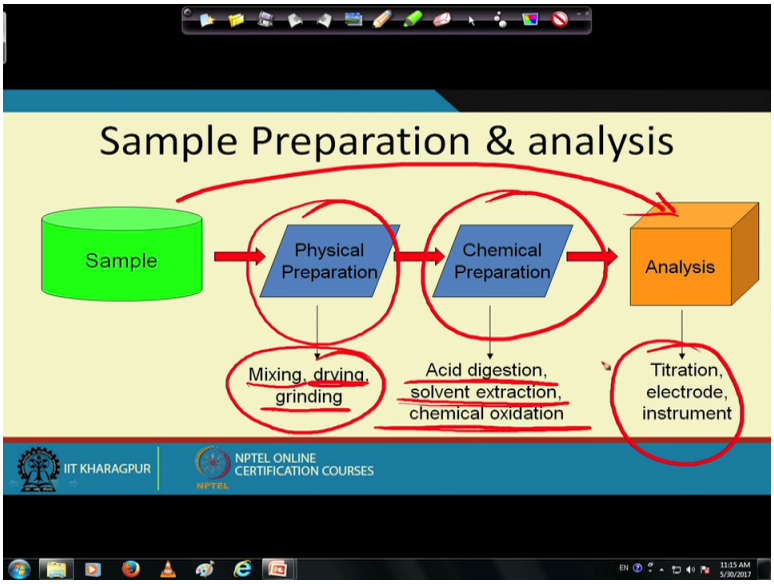
Course on Integrated Waste Management for a Smart City
Professor Brajesh Kumar Dubey
Department of Civil Engineering
Indian Institute of Technology, Kharagpur
Module-03 Lecture-12
Chemical Analysis Procedure

(Refer Slide Time: 0:30)



So let us get started where we left in the previous video. So we were talking about this chemical analysis, we were looking at the chemical analysis part of that. So we will look at chemical analysis procedure which is we talked about this direct chemical measurement, use of a sensor or and then I said that you need to do some sort of sample preparation before you go for at the chemical analysis.

(Refer Slide Time: 0:45)



So when we talk about the sample preparation, we will kind of go over this. What do we mean by that? So when you say, if you go and collect a solid waste sample or even a landfill leachate sample or a soil sample, you do not take that sample and directly put it in that analysis. Say what kind of, whatever analysis that you are going to use, you are not going to just take the sample and just go directly into, you do not kind of, cannot go from here to here, directly put it into the instrument. You have to do some sort of preparation in between.

And that is way it called either some sort of physical preparation or chemical preparation. So when we say physical preparation and the chemical preparation, what does it mean? Physical preparation means you may have to mix it. Say waste is a highly heterogeneous material, so waste, when you take a waste sample, they are not like they are not very, very, I would say they are just not all mixed up very nicely.

You may have a piece of cloth somewhere, you may have a piece of paper or some metals and some other things in there. But that is, what is the representative sample then? So we have, if you want to get the representative sample, you may have to do some mixing, you may have to do some grinding, you may have to do some drying as well. Drying for to go on the dry basis that we talked about earlier.

If you want to go for dry basis versus wet basis, but even for if you have to do some grinding, first you have to dry because we do not have wet grinders that much. Usually we use the dry

grinders. So we need to do this kind of some of this mixing, drying and grinding to make the sample really uniform, make it, so that when you do multiple analysis, you are actually measuring the same thing. Otherwise, getting representative sample becomes very difficult.

And towards the end of this, towards the end when we, I think in the ninth and tenth week, when we are talking about electronic waste, again this will come. Like, how to get a representative sample from e-waste, electronic waste. There are so many different components but what is the representative sample? How to use that sample to do any testing? We will talk about that again.

So those are some of this physical preparation, just to get a representative sample, make it a more uniform sample. But even after doing this grinding, mixing and drying, they are not, you cannot take it and inject it in a machine because as if you have walked into any environmental lab, if you have not, I would encourage you to go and see an environmental lab so that you can appreciate the concept that I am trying to explain. Because why it is needed?

If you are using ICPMS or GC-MS, or GC or simple ICP and I will show you some of those pictures of those, there are small like you cannot take the sample and inject it directly there. You have to do some type of extra action. Say solid waste, we have some solid waste sample and we want to measure how much heavy metals are there. So but heavy metals are not just like freely around on the solid waste so that we just take this heavy metal and try to find out the number. These heavy metals are bound up with organic matter, they are in some sort of complex form. So there are certain bonds are there, covalent bond, ionic bond. We need to break those bonds to make these heavy metals free.

So for those things, we need to do some sort of acid digestion because that acid, very low pH, heavy metals will dissolve. So that is what over here in terms of the chemical preparation. We have this acid digestion we have to do. Then there are something called solvent extraction. Certain things are present but they are, they have to be extracted. Especially, the organic compounds, they are, they will be there in the sample, but you have to extract them and you have to extract them using some certain solvent.

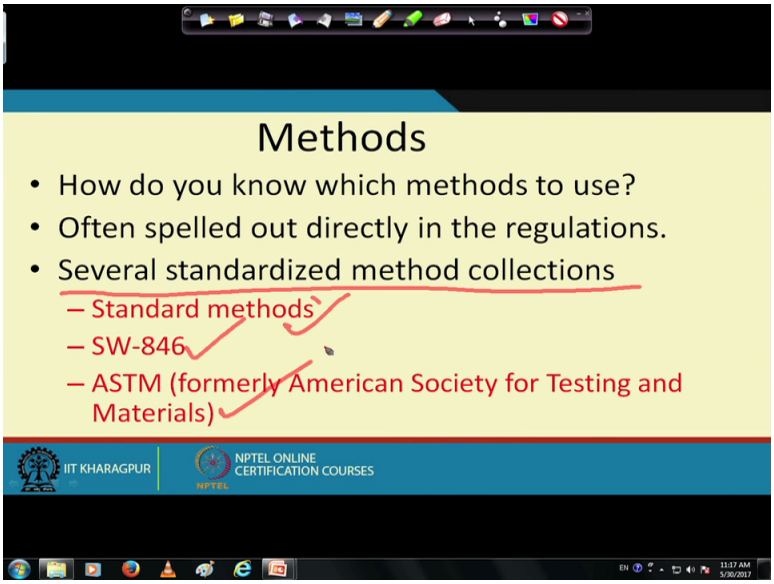
Solvent means something which will solubilize that particular compound that we are interested in. So that is your solvent extraction, then you may have to do some chemical oxidation. Chemical oxidation, what it will do? It will break up all the organic bonds and or even the ionic

bonds. And then it will make things free and then the things will come into solution, and then we can inject that in terms of the analysis. And that could be done using either titration, electrode or instrument. So there are different types of instruments are used.

So this is very, very critical, especially those of you who are, who intend to do lab based work or have, if you have done lab based work, you definitely know this part. But if you have not and you are planning to do some lab based work, and this like doing good lab technique is very, very critical in producing good data. Without good data, good data is kind of the raw material of a good design for waste management system and that is where we are lacking, where we have to have a good database of waste quantity, quality and different types of characterization like physical, chemical, calorific value and those should be the real sample not the sample collected.

If it is being collected from the houses but there are changes in between, you should track the changes in between. So we should in fact collect sample at the house level, at the primary collection center, at the secondary collection center and all the way to the disposal site, the dump site so that we can see how the calorific value is changing, how the biodegradability fraction is changing. So all those, then we can make a good judgment in terms of what kind of systems will work in our city. So this sample preparation analysis is very, very critical and again this is not only for solid waste, it is true for waste water, it is true for water sample, it is true for soil. And all these different types of samples can be used, for similar concept can be used.

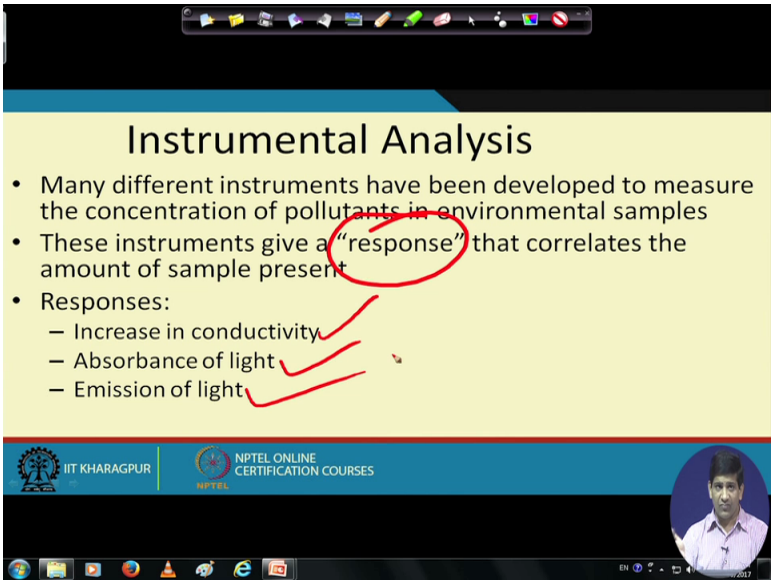
(Refer Slide Time: 6:12)



So when we, once you do the sample preparation, you will go for, you have to use certain methods for the analysis and then when we talk about these methods, it is, there is a standard method we talked about. How do you know which method to use? Often, it is a typically in the regulatory world, they will tell you what methods to be done. And you can use this standard methods book to find out which methods are being done. As a researcher or a student if you are working on your masters theses, PhD theses, even for a BTech project, you will do some literature review.

So you will know how, what other people have used the methods so that you can use similar methods where you can compare your information that you gathered from their information, their data that the they had. So those things needs to be done. And so there are several standard methods which is out there, which one of them we have like standard methods that we talked about. Like if you go to any lab, you will find that. Then SW-846, I mentioned to you about that as well. Then there is, there are some ASTM method, formerly American Society for Testing and Material. So those are the different methods which is like a source where you can get those details and use it for your analysis.

(Refer Slide Time: 7:32)



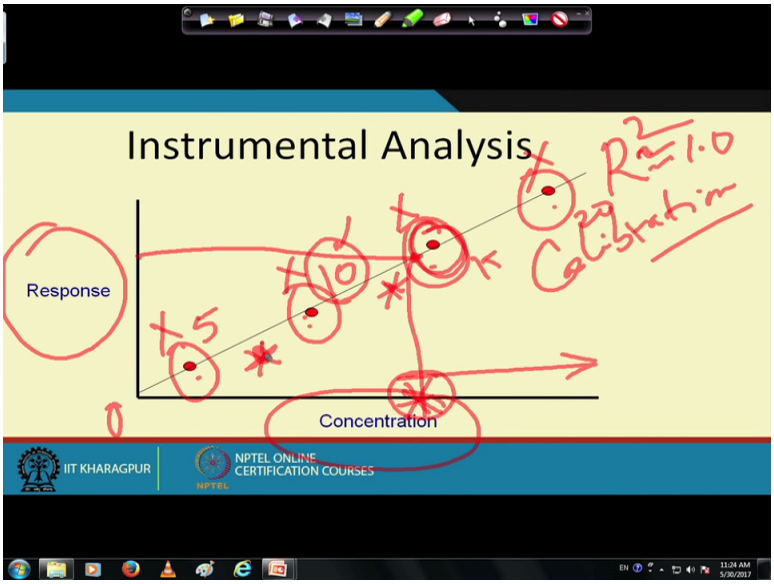
So after you have the methods, you have selected the methods, you will try to go for some sort of instrumental analysis. So there are different types of instruments are used, have been developed. In the environmental samples, there are the instruments. All these instruments what they do, they give a response. So we get a response from these instruments and we use this response in terms of the analysis. And that response correlates the amount of sample present, so that response could be increase in conductivity, the response could be absorbance of light, response could be emission of light.

Depending on the type of experiment, increase in conductivity, many times you see that in terms of IC when we use ion chromatographs and everything is, ions means they will conductivity. More the ions, higher should be the conductivity and more and should be higher the TDS as well, Total Dissolved Solids. Absorbance of light, that is what AAS works on. AAS works on the principles of absorbance of light. Emission of light is what, even absorbance of even spectrophotometer, your spectrophotometer that you use quite frequently in our environmental labs, that is again works on the principles of absorbance of light.

And emission of light is ICP which is, use is emission of light. Absorbance of light, we need to be really careful. It is a colorimetric, usually they are colorimetric method. Colorimetric means which uses color and if you, if your sample itself is colored, then using the spectrophotometer gets little bit tricky. You have to be, you have to make sure that you are getting, you are taking

care of the background correction which may be needed. So that is different instruments can be used based on different analysis.

(Refer Slide Time: 9:16)



And whenever we go to do this kind of instrument analysis, first we prepare this graph. And if you are done any lab work, I am pretty sure that you know what it is. It is calibration curve. We call it a calibration curve. Now what is a calibration curve? What we are doing over here? So this is called calibration. We have the calibration curve, so c-a-l-i-b-r-a-t-i-o-n. So that is the calibration curve. And here, we have, x axis is the concentration, y axis is the response. So what we are doing here? We are increasing the concentration. As we are increasing the concentration, we are seeing a different response.

These are our response points that we are getting. And then we are doing a base pit line. And many times you hear this term, R square, you get the R square number which should be closer to 1, it is better. As you go closer to 1, it is better for us in terms of the R square. So this, that is the instrumental analysis. So based on the concentration and its response, so here these concentrations are our, we have prepared these concentrations. And we have passed it through the instrument, we got certain response. So these are the response points.

Now and then we got this base pit line. So this base pit line is kind of the equation that we use for analyzing for sample. So what we will do next? Say if you have, if you have a unknown sample, you will get some response. So from the unknown, when you do the unknown sample,

you get the response. So say if you come over here, that is the response. Then you kind of go down here, so that is the concentration with that particular response. So this is how it works.

So since more and this is there for each and every instrument. So what happens is this basic, this like a basic principle of having this calibration curve and getting this response, that is once you make your calibration curve using the different standards that you use and then you come up with this like a base pit line. Then you go for unknown sample, you get a response and then you have this concentration being given to you.

Most of these, because of this computerization and everything, most of the analysis, this happens in background. You do not really see it which happening. It is a software coming with your instrument does it for you, but you need to understand what is going on. Again, as I was telling you in the previous video, there is a difference between a research student, master student, PhD students and postdoc and a lab technician, although I would expect the lab technician also to try to understand that. But my expectation from a research student is much higher than from a lab technician.

So you need to really understand what is really going on. So do not, if for some reason if you made these standards wrong, if you messed up in these standards, then your calibration curve will be wrong. And I have seen in some of the labs what students were doing. They were making these standards and they were doing this calibration curve. And then they will use one of these standards to check whether the calibration curve is correct or not.

So if you had made the mistake in the beginning, that mistake will be, say two mistakes, since your initial standard you made wrong, later on if you check with the same standard, it will give you the correct number because the input is wrong to start with. So you need to really be careful in terms of, so you should make a newer standard. You should, that is called, we call it a calibration check standard and that should be made from scratch.

You should make a fresh calibration check standard. And then you check whether your calibration was okay or not because if you made first set of standards, you start from, so this is our 0, you start from 5 ppm, 10 ppm, 15 ppm or 20 ppm whatever. And then you got your base fit line for that. But then if you use the same 10 ppm to check it if the 10 ppm was wrong to start with, the next time when you check it will give you the correct because what we are doing in the

beginning is we are telling the machine that if you get this response, that is for 5 ppm. If you get this response, this is for 10 ppm. If you get this response, this is for 15 ppm.

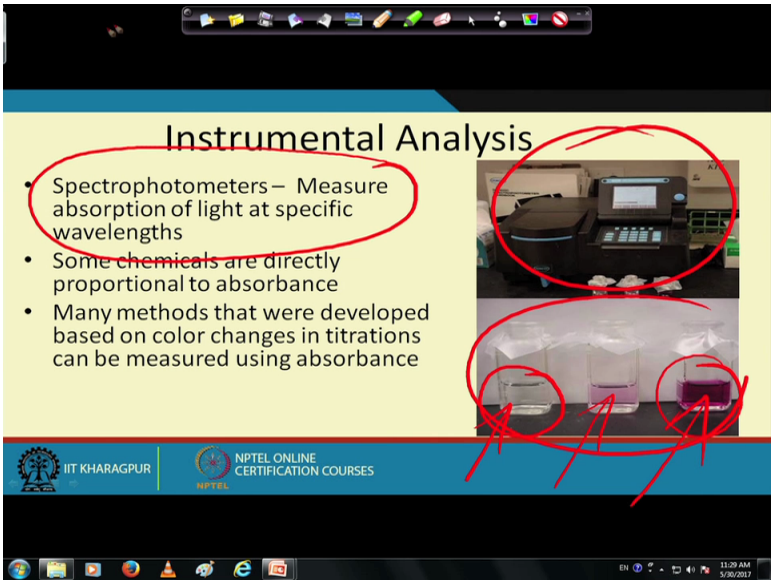
If you get this response, it is for 20 ppm. But if my 10 ppm standard I made it wrong and that can happen. If you are working in the lab, suddenly your phone rang and you lost your concentration and then rather you added up, while you are palpating it, you added maybe a drop more or drop less. And based on your calculation and then your 10 ppm standard has gone bad. So how will we check that is when you do this calibration curve, you come up with a newer set of standard.

So you make a newer data point here. You make a newer data point there and you make this standard as a fresh standard from this standard bottle that you have purchased and you check it whether the numbers are correct or not. So it is, the probability of making the same mistake again is very low. So that is why if you have made mistake in the beginning, that will be checked in the second time. So I hope it is a very, very important point.

And what based on my experience of last two years working in India, before that like I was abroad but in India we are not that careful in terms of quality assurance and quality control. And that is why most of our papers, many times we struggle to get it in a very good quality journal because we do not follow good quality assurance and quality control, we do not. And that is critical in terms of getting our research product, research quality better.

So I strongly encourage you that you, if this part of the video you listen again and again if you have to. If you do not understand, ask me as many questions as you want through the discussion forum. We will be very happy to respond but you definitely understand that this is very, very critical and it is true for any environmental analysis you do. Even whether, not even the environmental, I will say even in any experimental work that you do in any field. That, it is you have to be really careful in terms of the data quality. Your data has to be good. And if and you have to be confident, you have to defend that why this data is good. And we will talk about some of this aspect again in a minute or so. I hope that kind of clears up the importance of this part.

(Refer Slide Time: 16:02)



And then in terms of the some examples of instrument analysis, spectrophotometer which is, it measures absorption of light at a specific wavelength. So we use different wavelengths to measure this light. Some chemicals are directly proportional to absorbents. They are many methods that are developed based on the color change, we use in the titration as well. Can we measure using those things? Can we measure using absorbents? Here you see at the bottom here, we have three beakers. And those beakers, it is essentially for the H2S analysis. These are the H2S analysis beakers. Here this one is my blank, it has no H2S, little bit of H2S and then it has high H2S.

So these are my kind of, you can say one standard. So I am trying to, so if I have a colored sample, again now if you look at here, this is a clear water sample, so it has no background. It is a clear water sample. So the absorbance of light, there should not be any absorbance of light as such. Little bit of background will be there which we can zero it. But if we had a sample to start with which was colored, so if you have a nasty landfill leachate sample which is colored, remember I told you that nasty landfill leachate will be like a dark coffee colored, black coffee.

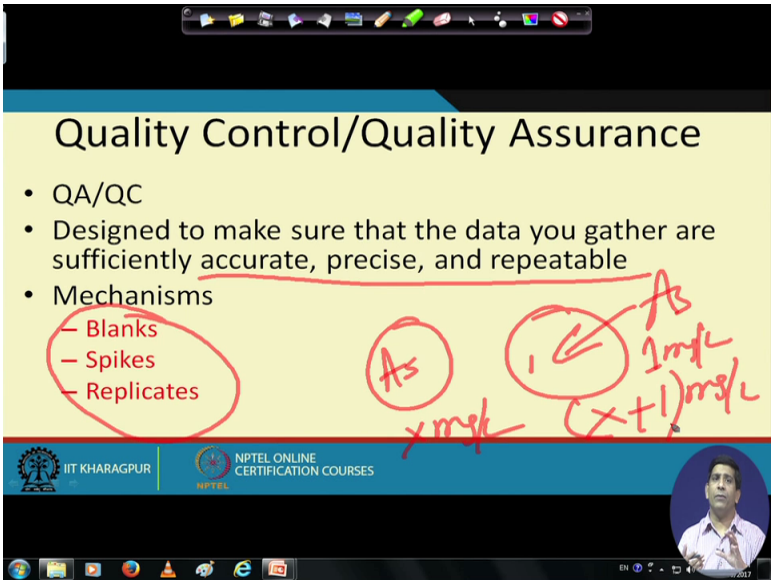
So if we had that sample and we had to use a spectrophotometer, since the so much of color is already there, it will be difficult to use a spectrophotometer for those kind of sample. Luckily what happens is since the landfill leachate has so much, the concentration is so high, even if we

take like 1 ml or even like 0.5 ml and dilute it, that concentration is good enough to be above the detection limit of the machine.

So we can still use it and when it is diluted, mixed with the DI water, it is the color goes away, color is almost gone or it is a very faint color and that spectrophotometer can be used for that. But there are, it is, spectrophotometer, what it does? It measure the absorbance of light at a specific wavelength. So here again we will do, we will have a certain sort of standards going on where these standards will have certain absorbents number and that absorbents number will be used.

So based on certain concentration, there is certain absorbents. And then when you do this unknown sample, whatever is the absorbents value you get for this unknown sample and it will calculate back the concentration. Similarly, as very similar to what we are talking about in that calibration check diagram just in the previous slide. So those things happens already inside this machine right there. So this machine has a computer, it does it for you. You do not, may not realize that but this is exactly what happens in terms of the instrument analysis for that part.

(Refer Slide Time: 18:51)



So that is in terms of, so there are concept of quality assurance, quality control, that is a QA/QC. It is designed to make sure the data you gather are sufficiently accurate, precise and repeatable. So again data quality is very, very important. As I said earlier, that is one area where I see we

lack. As in general, some people might be doing great but in general observation that I have, our data quality, we do not really pay very good attention to our data quality.

Some of them is because of as many of our labs are not that good equipped but labs are getting better. We are getting good instruments, we are getting good lab especially in good universities and good institutes like IIT. But still our data quality, the culture of having this quality control, quality assurance, making sure that the quality is good is, it is missing. And that is, so how we make sure this quality is good and what are the things that typically done in a good lab practice?

So what is, when we say quality control, quality assurance, what really we mean? We would, it is designed to make sure that data that you are gathering is accurate so that you can, it is sufficiently accurate, precise and repeatable. When we say accurate, means it is real data. And how to make sure it is a precise? So it is, and then repeatable. So if you do it again and again, you should be able to get the same numbers. So there are mechanisms of doing that. We can use, we use blank sample, we can use a spike sample, we use replicate sample and I will try to explain each one of them a little bit.

When we say blanks, why we use blank samples? What is the usage of blank? What we really mean by blank? Blank means we will use DI water or deionized water or DDI water, whatever you call it, Nanopure water. And so since you are using certain beakers, certain lab instruments, lab equipments that you are using, those lab equipments, those beakers maybe contaminated. Say somebody else has used it.

For example, you are trying to use some solid waste sample but previously I had used it for electronic waste. I had those beakers which I had some e-waste going on in there. Typically e-waste means lot of heavy metals, especially lead, cadmium and those things will be very high. So and I did not clean it properly or I was, I kept it for cleaning but you had, you rushed in and you had to use it, so you took those beakers not realizing that they are not really clean.

Because you do not, you will really, when you do this digestion of electronic waste sample or anything, you do not see any color or anything in there, or any like a dirt or gray material to tell you, “Oh, this is dirty. I should not touch it.” They look pretty, they look white, they look, it is a glass beaker but it is not really clean because there is a cleaning procedure. We have to clean it

using some dilute acid. We keep it in a dilute acid bath overnight and then we clean it and then we pass it through some Nanopure water or DI water to make it really clean.

And so if your glassware is not clean and you did some analysis using that dirty glassware, then what is going to happen? You will have the problem of like this lead and cadmium showing up in your solid waste sample, because it was there the leftover from the analysis that I did few days before on my electronic waste. So how we will check that whether my glasswares were clean? Whether there is no contamination coming from the lab itself in our sample? For that, we use these blanks. So this is the importance of the blank sample. It is very, very important if you think about that. It is because there could be contamination coming in, you never know.

Because it, there is and especially when we are looking at these newer types of contaminants which has very low standards, they are supposed to be, their research is suggesting, the toxicity information is suggesting that they are bad at low concentration. And when we are trying to put certain regulations on them, we have to really have this data. When we classify something as a hazardous waste from a non-hazardous waste, the cost of managing that hazardous waste goes up. Cost of managing something which is, which we think is bad, it goes up.

So if, but the company can go to the court and we have to defend our data, so think about that. There is, there could be litigation, there could be lot of financial implications. So that is why we need to really make sure that there is the data that we have collected is accurate, there is no background contamination coming from the lab environment. So for that, the blank samples are used.

Many times, we also know what is known as field blank, we will carry DI water with us to the field and bring that DI water back from the field. And just to makes sure there is nothing happen along the way, especially if you are looking at some of these organic contaminants. Even the exhaust from your car that you use for, say you took the backside of the bonnet out and there was a car was running, there was some exhaust going on and some of these organic chemicals from the exhaust can get dissolved into the your sample.

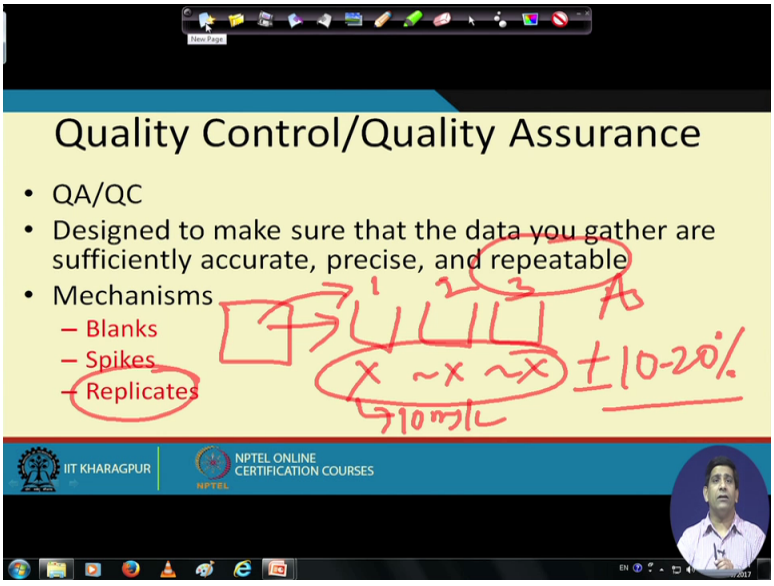
So whether those things are happening? We use a field blank and that is also done. So those are very, very important stuff. Unfortunately, again as I said, in Indian context we do not really pay much attention to this. These are very critical and we need to pay attention to this aspect. Then

we do some spikes. Spikes, what is the spike sample? Say if you are trying to use, you are trying to analyze a certain solid waste for say arsenic, lead or cadmium or certain organic chemicals. So whether my machine is working properly in terms of analyzing these elements?

Say if I am analyzing for arsenic, so whether my machine is reading arsenic properly or not? How will I know? So I will take a sample. So this is my sample. I will take a sample and I am measuring arsenic in that. Now I will take the same sample and what I will do is I will spike some arsenic, I will add some arsenic. Say 1 milligram per liter of arsenic, I will add it. And why, so this is called spiking. I am adding some arsenic to it. And so here when I do the analysis, I got x milligram per liter. Now here I should get x plus+ 1 milligram per liter because I added 1 milligram per liter to this. If I do not get x plus+ 1 milligram per liter, what does it mean?

That means my machine is not reading arsenic properly. Machine is having some problem reading the arsenic. So to do that, this is known as the spike and this is the called a spike sample. When you add certain concentration to get the, to get your, so to do the like spiking of the sample to find out whether you get, whether the machine is reading that particular element correct or not. So that is called the spiking.

(Refer Slide Time: 25:51)



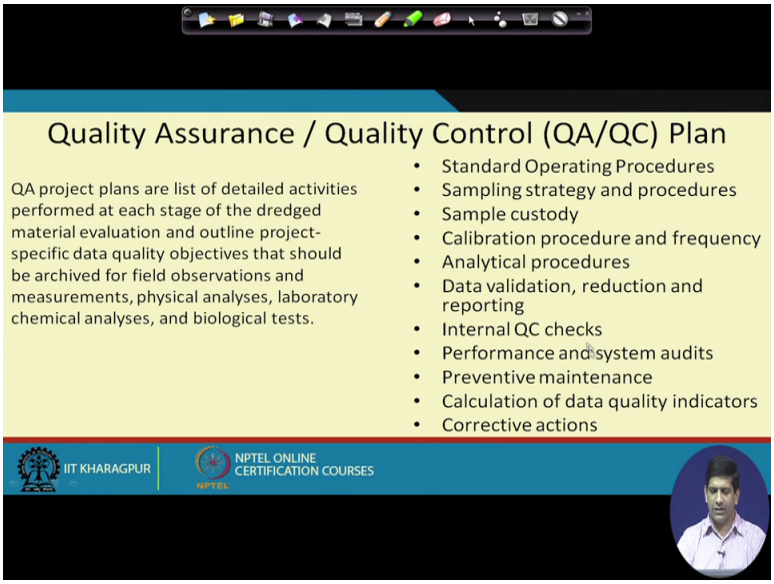
And then replicates, replicates means whether the machine is giving you the same. So this in terms of the replicates, if you, if I collected a sample from the field, now I am trying to analyze it. From the same beaker, I take a sample. Then I take another sample and I take another sample.

So from the same beaker, I took three samples, 1, 2 and 3. Now if I am analyzing, again say if we are analyzing for arsenic, if I get it for x here, I should get something very closer to x and something also very closer to x over here too.

They will not be exactly x because like there are some human errors, could be there. But usually we try to go for plus minus 10 to 20 percent, especially for organic, there for inorganics. So it should be within plus minus 10 percent. So in this if I get x as maybe around 10 milligrams per liter, so here in these two samples or like in this I should get something around 8 to 10 milligram or like 8 to 12. 8 to 12 milligrams per liter what should be in these three data. If it is not, that means there is some problem. My replicates are not showing up the same number, they should because things should be repeatable. That is what we said earlier, things should be precise that we talked about earlier, accurate, precise and repeatable.

To make sure they are accurate, they are precise and repeatable, we do this quality control and quality assurance and it is very, very critical in terms of the good data. And if you do that, I promise you that if you do this in your research projects in your research labs, definitely you will be able to publish your paper in a good journal which is very, very, and we need to get good papers coming out of India in good journals in future so that we can like in other we have to compete. When we say we have to compete globally, we need to produce good quality paper. And only paper is not important but paper is one of the yardstick is there.

(Refer Slide Time: 27:36)



And then there are this quality assurance, quality assurance plan. I will not read through that. It is essentially whatever I have been telling you, this is what it is. It is a QA project, it is a detailed activity, you do that. And there are standard operating procedure, sampling strategy, you do the sample custody, calibration procedure, internal QC checks and all those things. You can read through this. This material will be provided to you as a PDF. It is, so these are different things, we do it.

And if you are interested in that quality assurance, quality control, I would encourage you, go on Google and look at that. There are lot of examples out there. You will see them, QA/QC plan and you can download some reports and you can read those reports as well over there. So with that, let us kind of, we will stop here in terms of this particular. And this, in terms of one of this, this video is one of the very, very important video I would say in terms of environmental, in general for environmental engineers or environmental scientist in terms of understanding of good lab practices. And we will continue that discussion in the next video as well.

And then at the same time, try to understand having this good data quality. Data quality is very, very important in terms of coming up with your good, in terms of good data. Good data in terms of whether you are trying to go for design of the waste management system, waste water system, water treatment plant, air pollution. Anything that you do, soil pollution, soil remediation, data is needed. Data is the raw material for design and that data quality is important.

If the data quality is bad, you may be a great designer but if you are working with bad data, your design will come out to be bad. So with that, I would conclude this video and we will continue our discussion in the next. Thank you.