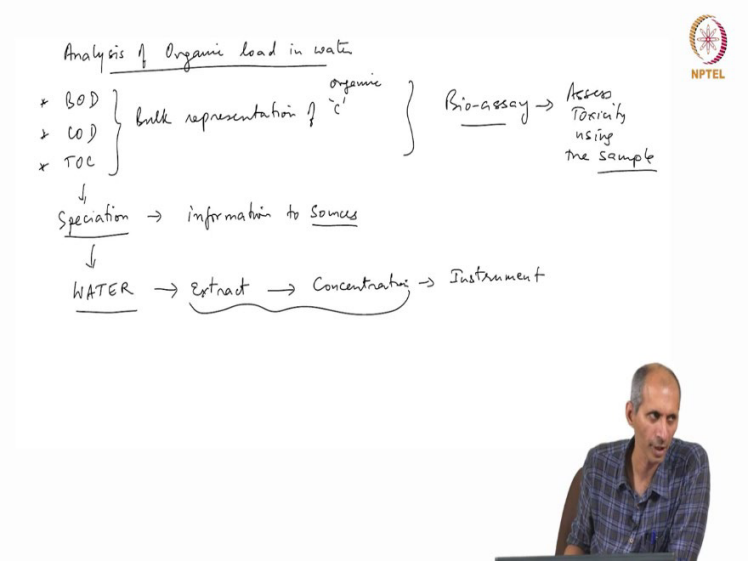
Environmental Quality:
Monitoring and Analysis
Prof. Ravi Krishna
Department of Chemical Engineering
Indian Institute of Technology-Madras

Lecture No. 21
Analysis Methods – Review of Standard Methods

(Refer Slide Time: 00:13)



Today will just briefly look at 1 small thing and then we will move on to analysis of organic chemicals and inorganic chemicals in water trace speciation of that looked at the BOD, COD and TOC last class so all these are bulk representation of carbon organic carbon. So, if you want to do speciation what we mean by speciation is individually find out what is there and what organic chemical is there and also the BOD, COD and TOC are good representation of state of the water what is clean not clean.

You can just give a very simple cut off for regulatory purposes for where the volumes are very large you are looking at very large areas and all that but once you cross that you need the specific information about toxicity and all that you need to know what is it what is the chemical you can also analyze toxicity of particular water body but taking that water sample and doing what is called as a Bio Assay.

You can do the toxicity directly by doing what is called as a bio assay we would not talk much about it what bio assay, means is you assess the toxicity using the sample. For example, if I want to save a particular lake is contaminated, I take that water, bring it my lab and I will put 1 test organism there and see how many of them survive or if they are having a problem. So, this is called as a bio assay. And this can be done with any number of organisms there are some indicator organisms that people use to look at bio assay, this is done for that.

And there are various number of the other usually the indicator organism is something that is local. You cannot use an indicator organisms that is used in Europe, bring it to India and use it will not survive for other reasons. So something that would normally survive in clean water. Something happened to it in the presence of the organic load. So you know, the organic load cod is 250 milligrams per liter is that 250 milligrams is toxic or not, then you do the bio assay to determine that that can be done.

So, you directly take the COD or BOD value and go to the toxicity analysis by doing this is one approach for doing it. This is done by biologists know ecological scientists, they do that there is a big section of that. So we are not going to discuss that here. There are also groups in which people just either look at the fatalities of the organism or this specific change in the organism. So, if something is happening to this, for example, if you use fish, something is happening to this fish, what is happening is they are losing their sense of direction or they are losing some function.

So they will assess that and then they will go deep and see what part of the body is being affected because of this and also it is depending on how you and that is a field by itself bio assay and automated bio assay so you did not have to take anything out to this that is a very large field which is one approach to it. The other approach is to find out what is there in the water and then you know toxicology information about that perspective, you can directly find out if the toxicology of that compound.

And the competition warrant determination that this is so, the bio assay is good advantage of using a bio assay is suppose there are you will determine that there are 10 organic compounds. Each of them have concentration less than what is the prescribed toxicity level, but combined all of them

together may have a toxic effect on an order so that you cannot find out from getting toxicity information, individual toxicity.

You can only do that by getting something like a Bio-assay, ok, which is really the proof that you want because people do that. So in other in other words, Bio-assay is really the starting point of all our investigation. So we usually have information about receptors having a problem and then we back calculate and see what is there in environment. So we are just closing the loop. In this case, you find out what is there and they say something is there and then you see if this is really what is causing the problem.

But in any case, speciation does give you valuable information as to what is there more speciation, the value of speciation is to find out what is the source of the pollution. So one of the advantages of doing the speciation is to be it will give you information pertaining to sources. If you know the source, you can go and do something about it, ok. You also know; what is the relative contribution of each source what is what we call as source apportionment.

And this relative contribution of sources is very useful so you can target sector for example, if you know that this is a chemical A is coming from one particular industry, you know that that industry is primary contributor. So, you can go and do something about it or it gives more information and you can have useful analysis of that information, ok. So, to do speciation we are we are trying to do we will look at each matrix separately. So, we will look at water first.

So, we discussed the general methodologies what we are going to discuss is a little more specific to the instrumental analysis. So, we looked at water. Now we determine that water we have to extract from water and then we go to an instrument and we go to processing concentration and to the instrument so we will first look at these two things extraction and concentration, ok. The first so before we go here I wanted to show last class we couldn't show this.

The standard method so, this is the website for standard methods for the examination of water and wastewater. I don't think you can do this here, so I'll just point this out that so, this is the you have to buy it is not available for free on internet but I can still show you. When you go to browse you

have different types of analysis. So it says individual organic compounds. Aggregate organic constituent what we mean by aggregator is.

What we did yesterday, BOD and COD they all aggregates you go to aggregate organic constituents, look at biological oxygen demand, chemical oxygen demand and dissolved oxygen, all all of these things, ok. So, if you go to bio biochemical oxygen demand it will ask you, so, there is a chemical number method number is 5210 is the method number, ok, it says you have to buy it but full method you have to buy full text it'll ask for this thing, but gives you a general description of what the method is.

You also see that in all of these you have one there is a quality assurance page everywhere. If you go to the quality assurance page it will say QC important attribute of any laboratory assurance program without QC there is no confidence in results of analytical tests. So, for every method, there is a QA/QC described for every analysis you whether it is aggregate it is individual, it does not matter even if you are just weighing a small piece of paper QA/QC is important and this will give you so they have a lot of things QA/QC a lot more it's it's it's very involved I think what I have given you is only the very basic information, but the philosophy behind QA/QC is the same. So, for if you know what you need to do then you will go find out the tools required for that, so that is the module I have given you some introduction to it. So, these are all this other color taste the standard methods for examination of water and wastewater will give you so, it gives you this one is specifically methane for example, a very specific chemicals here that are individual organic compounds. So, they have a this thing on extraction, yeah. This is one source, yeah, standard order. If you go back to the about so this is published by the American Public Health Association, whatever association this is proprietary to them. For example, you have heard of this thing called ASTM American testing materials I think that, it is an organization. It is not a government agency. So they have spent money and effort to do this, so it belongs to them if you want to buy you have to buy the method. So people have spent money over this. So it is a copyrighted thing. So here this thing IIT has subscription to this you can go to library, there is a book, standard method, there is a book, it is a big book is there in our lab, you can take a look at it if you want. So, there are individual methods for all of this. So if you if you go and look at individual organic compounds, if you look at say phenols let say aldehydes.

So it gives you the method, semi volatiles covered this section may contain different classes but the method is a broad spectrum gas chromatography analysis so it giving you the instrument and it is also giving you what is the processing technique one line, yeah. So, the matrix water this is water obviously this all of this is applicable to water and it is giving you the method number is giving you this is instrument that is used and this is the extraction that is used. So, there are it it gives you a bunch of other information. So, this is one source of this method.

There are of course, other sources where you can look at these methods. One of them is the US EPA. So when we go to the US EPA website, I think it is very illustrative for you to go there, environmental topics. So, we'll go to water will go to water. This is very relevant to the US now, water research methods and models for water research water data, water and wastewater management this is methods for analysis of different things, biological manual development for this thing, microbial methods standard practice of different types of things, see different chemicals we have drinking water treatment and analysis so they have this method number again EPA has different methods. So, EPA is an open source thing. I can go and access the method this is free for everybody, it's a government agency so it is free and there are different government agencies where websites are free so you can go and access methods available.

So, again here determination of sector perfluorinated alkyl acids in drinking water by some method of extraction and the method of instrument this is given method description is there already. So, you can find out if that is useful for you and all that remediation, recovery and all that. So, very specifically I can also look at there also have models will get to models later. When we do when we start the third module we will go to come to the models.

You can very specifically look for EPA say method for analysis of PAHs in water. So you'll get a document like this method for analysis of PAHs in water, in industrial waste method for organic chemical analysis, Appendix to part A method 601 if you look at this method first method, what does it give you it gives you this method is applicable for the analysis of all these chemicals, individual chemicals, these are all poly aromatic hydrocarbons some of you may recognize me from your assignment 1 you will see this is a PAH.

And this is the description this is a chromatographic method we will come to that in a minute everything is given here summary of the method measured volume of whatever whatever is done this is the summary of mathod what is there then we go into interferences whatever we discussed in this matter if you use water what are the interferences how would you how should you handle it, ok? Then safety then apparatus and materials all everything is given listed down as you would write a lab report.

This is how you should write a lab report when you are doing analysis. But many of us don't do it even I don't do it. But this is the method because this is information going to people who are not at all used to it. So I am an experienced analyst, I don't have to strictly follow it. I know exactly what I am doing, but I have to document it this has to be done, then you it will stand in court. So this calibration it says external standard calibration procedure.

And if you remember what we talked about external standard calibration. What we normally do by taking a standard putting it into a solvent making a concentration and making a calibration chart that is called external calibration. There is another thing called internal calibration, we will talk about it in this discussion in the procedure succeeding discussion. Then there is response factor, there's a response factor is the calibration what we get from a calibration is a response factor, the curve then we have quality control.

We are still not talking about the method, we are talking about all this other quality issues and you know, accuracy and all that you can read it, so your sec next assignment is going to be this you have to read I will tell you what the assignment is right away. So, you have 6 chemicals that you have chosen, you pick 1 chemical or you pick the 3 of you are in one team. So, you can you have to you have to do 6 pieces of analysis, you have to pick 2 chemicals ah 3 chemicals and 3 matrices, air water and solids, and you mix and match one chemical at least in each one of them.

So, you pick chemical A in water, chemical B in air, chemical C in solid or some some combination of it. So you have experience of doing different types of chemicals in different matrices, analytical instrument will change because of that, of the so the properties if you notice the 6 chemicals that

you have you have been given, they have a certain order of some of the properties see solubility KOC all of them are increasing toward as you go towards PCB and PHS.

So, the methods will change appropriately and some of and one of them will be a inorganic method is completely different. So, 1 inorganic, 2 organics you pick 1 varying property and do the analysis. So, you have to read the method and give me a summary, a one page summary. So like the summary they have written so you have to understand and extract information as you would give it to a novice who doesn't know what they are doing.

Now, you start section number 8 sample extraction starts then sample analysis start cleanup separation, high per chromatography comes, gas chromatography and then we have all this the what conditions you should you maintain and all that then they've references then there are retention time this will come to all this there's we are getting ahead. This accuracy overall precision is all reported. You look at this table method accuracy and precision we talked about all this this has to be reported.

You must be you must know what it is like this there are other this is for water this method we are we have seen is for water so you can generally search like this in wastewater very specifically says waste water like that you have methods for air vapor phase in the particulate phase and in solids. USEPA is a good source for doing it. So, we don't want to rely on AWWE standard all we usually go with the EPA method because it is easily traceable. Suppose I am using a standard method I don't have access to it.

You show it somebody they say I don't have a license to it I don't know I can't see it done, we we are stuck. So, methods are generally similar, unless it is a very specific method for something very special for a very specific industry, we don't go to at least in this class we will not go to a closed method we will go to an open method like this. So, this this opens up a whole big chunk of information that is available to you, ok, just the way we did the search now, to get this method you just do that write the keyword and analysis I n the sequence I had given analysis of this using this by this you don't even have to give them methodology you say this, you will something will show up like this, but usually they have a method number, ok. So, the method number is is the reference.

So, when you say, you say I analyzed the concentration of phenon3 in this this water sample, using method 610, USEPA, 610 done, you don't have to describe anything else.

Because the references here people can go and check this method. Now, if you are modified this whether you say I have analyzed it based on methods 610 the deviations are the following. I did not use these these this so I changed the method slightly. So it is a very subjective thing, sometimes something is not available if you can't stop the analysis just because you don't have everything that they are telling you, they are putting everything here because the completion they want everything covered.

But you don't have it does not mean you cannot do it, you do it, but then you find out what the error is and say the deviations are these possibly then therefore, extraction efficiencies are different from what is reported in the method or the analysis calibration is different because I we use something else depending on it, ok, any questions on this? This is how a method is presented to you and if you are making a method you have to present all his information.

So, in the exercise, you have to go through at least 5 or 6 of these methods in detail. And so this is one source for water. The other source that we have is I'm I'm giving it to here so that for air, EPA a lot of information about air everything that we are doing in this class you can go there and learn there are videos extensive videos of this you can go and type it will give you whatever you need. So, air monitoring methods, ok.

This quality assurance is very expensive this some of these things you so air monitoring methods you have air toxics inorganic and organic they classified nicely straight away. So, this word organic, air toxic organic so it is it is telling the determination of VOC volatile organic compounds in air which means it is a vapor phase compound measurement, yeah, vapor phase. You go to 2 I think there's a method description summary somewhere, so here we are yeah, method TO1 method of the VOC method of VOC is using this in ambient method of uses using this and I am going to which means it is vapor phase.

Now, at some point somewhere you will see there is a this thing for filters I think it is included in the analysis itself. So, if you go to TO TO1 itself, ok will come back to this and there is inorganic. So this method determination of PM PM is here, filter suspended particulate matter all all of that is here. So, PM comes under inorganic because predominantly it is inorganic but then there may be organic constituents in the PM so, that that part needs to be resolved.

So, for that we will discuss separately you also have something for criteria pollutants, criteria pollutants are things like CO there are 5 things that we said PM is one class of it. The other things are the sulfur dioxide, nitrogen. The equivalent list of designated and equivalent methods PDF just a equivalent methods, this thing, ok. What I am going to do is that I will I have shown you this there is a there is a third class this is for air.

The third test set of methods which is called hazardous waste test methods. Series short solid waste SW stands for solid waste so we have one set of methods for water, one for air and one for solid waste solid samples. So this is for soil, sludge, sediment, anything, all of those things and there is a bunch of methods for that all all kind of thing which what is SW, how is it organized? SW basics, what is the methods? Methods themselves so 5 sample preparations 8000 chromatographic methods so on to various methods.

So we will just go to one of these will go to 8000 8270 this is a method for semi volatile organic compound using GC/MS gas chromatography ways the same thing, the the looks just like what we saw in the other EPA method there is a description summary what is it used for, application notes so on. All this you can see all this information is given there a full set of information what is they are all used for what can you analyze using this and it also gives you information about how to run it, ok.

So, these are the 3 sources that you can directly go to even irrespective of what I do in this class whenever you want to get any analysis method, ok. What we will do in this class will help you understand is to interpret whatever is is there in the method little more. Little more fundamentally you know what we are why are we selecting certain methods okay.