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## Lecture No. 21 Analysis Methods – Review of Standard Methods

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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 bioassay which is really the proof that you want people do that so you know, 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 in an environment. So we are just closing the loop. In this case, you find out what is there and say something is there and then you see 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. You also know; what is the relative contribution of each source and what is what we call a 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 as 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 the information. So, to do speciation we are trying to do we will look at each matrix separately. So, we will look at water first.

So, we discussed the general methodology is what we are going to discuss a little more specific to the instrumental analysis. So, we looked at water. Now we determined that water we have to extract from water and then we go to an instrument and we go to processing concentration and with the instrument so we will first look at the these 2 things extraction and concentration. The first so before we go here I wanted to show last class we could not show this.

(Video Starts: 07:10) The standard method so, this is the website for standard methods for the examination of water and wastewater. Do this here let us point this out that so, this is 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 let us say individual organic compounds. Aggregate organic constituent what we mean by aggregators.

What we did BOD and COD they all aggregate you go to aggregate organic constituents, look at biological oxygen demand, chemical oxygen demand and dissolved oxygen, all of these things. So, if you go to buy biochemical oxygen demand it last queue, so, there is a chemical number method number is 5210 is the method number it is just you have to buy it but full method you have to buy full text it last for it but gives you a general description of what the method is.

You also see that in all of these you have 1 there is a quality assurance page everywhere. If you go to the college and phase 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 describe every analysis you whether it is aggregate individual, it does not matter 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 get QA, QC a lot more it is very involved and that 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 and you will go find out the tools required for that, that is the modules I have given you some introduction to it. So, these are all this other color just the standard methods for examination of water and wastewater will give you so, it gives you this one is specifically methane.

For example, I am a very specific chemicals here that are individual organic compounds. So, they have this thing on extraction. This is 1 source standard order. This is back to the so this is published by the American Public Health Association, whatever comes in this is proprietary to them. For example, you have heard of this thing called ASTM American testing materials. It is an organization. It is not a government agency.

So they have spent money and effort loses. So it belongs to them if you want to buy or buy the method. So people have been money. So it is copyrighted thing. So here is an IT has subscription

to this you can go to library, there is a book, standard method, there is a book, it is a big book 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 aldehydes.

So it gives you the method, semi volatile compounds, you can condent different classes, but the method is a broad spectrum gas chromatography analysis with giving an instrument and it is also giving you what is the processing technique 1 line. So, the matrix water this is what our obviously is 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 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. Go to the US EPA website, I think it is very illustrative for you to go there, environmental topics. So, we will go to water this is very relevant to the US now, water research methods and models for water data, water and wastewater management says methods for analysis of different things, biological manual development.

For this thing, microbial methods standard practice of different types of things, see different chemicals you have drinking water treatment and as so they have this method number again up as different methods. So, EPA is an open source thing. I can go and access the method is free for everybody to 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 and drinking water by some method of extraction and the method of instrument is given method description is there already. So, you can find out if there is useful for you and all that remediation, recording and all that. So, very specifically I can also look at their also models will get the 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 US EPA method for analysis of PAHs in water. To get a document like the method for analysis of PAHs in water, industrial waste method for organic chemical analysis, Appendix to part A method 601 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 see this is a pH.

And this description is a chromatography method we will come to that in a minute everything is given here summary of the metal measured volume or whatever is done this is a summary of matter 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? Then safety then apparatus and materials all everything is here list it down as you write a lab report.

This is how you should write a lab report when you are doing analysis. But many of us did not do it you and I did not do it. But this is a method because this is information going to people who are not at all used to it. So I am an experienced analyst, I did not have to strictly follow it. I know exactly what I am doing, but I have to documented this has to be done, then you can 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 and the perceived succeeding discussion. Then there is response factor, the response factor is the calibration what we get from a calibration is a response factor and we have quality control.

We are not talking about the method, we are talking about all this other quality issues and accuracy and all that you can read it for your 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 1 team. So, you can you have to do 6 pieces

of analysis, you have to pick 2 chemicals are 3 chemicals and 3 matrices, air water and solids, and you mix and match 1 chemical at least in each one of them.

So, you pick chemical A in water, chemical B in air, chemical C in solid or from 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 for the properties if you notice the 6 chemicals that we have been given, they have a certain order of some of the properties across solubility KOC all of them are increasing toward as you go towards PCB and BHS.

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

Now, you will start section number 8 sample extraction starts then sample analysis start cleanup separation, I chromatography comes, gas chromatography and then we have all this the what conditions you should you maintain and all that then references then there are retention time this is will come to all this getting ahead. Accuracy overall session is all reported. You look at this table method accuracy interesting 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 this for what so you can generally search like this in wastewater really specifically says waste water like that you have methods for air vapor phase in the particular phase. And in solids, UCPS is a good source for doing it. So, we did not want to rely on a WWE standard all we usually go with the EPA methods because it is easily traceable. So well I am using a standard method and I did not have access to it.

You show it somebody said I did not have a license to it. I did not know I cannot see it done, 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 did not 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 opens up a whole big chunk of information that is available to you just the way we did the search now, to get this method you just do that write the keyword analysis.

In the sequence I had given analysis of this using this by this you did not even have to give them methodology you say this, you will something will show up like this, but usually they have a method number. So, the method number is the reference. So, when you say, I analyzed the concentration of 3 in this water sample, using method 610, UACP, 610 done, you did not 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 deviations are the following. I did not use these so I changed the method slightly. So it is a very subjective thing, sometimes something is not available if you cannot stop the analysis just because you did not have everything that they are telling you, they are putting everything here because the completion they want everything covered.

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

So, in the exercise, we have to go through at least 5 or 6 of these methods in detail. And so this is 1 source for water. The other source that we have is giving it to here for 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.

This quality is very expensive this some of these things you to add monitoring whether you have air toxics inorganic and organic is classified nicely straight away. So, this word organic, toxic organic so it is telling the determination of VOC volatile organic compounds in air which means it is a vapor phase compound measurement vapor phase. Go to 2 nothing as a method description summary somewhere, so here we go method to 1 method of the VOC is using this 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 scene for filters I think is included in the analysis itself. So, if you go to one itself, then come back to this and inorganic this method determination of PM is here, filter suspended particulate metal all of that is here. So, PM comes under inorganic because predominantly 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 it pollutants, criteria pollutants are things like co there is 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 colon methods, this thing. What I am going to do 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 we have got one solid waste for water, one for air and one for solid which solid samples. So this is for soil, sludge, sediment, anything, all of those things and there is a bunch of methods for that all kind of thing which what is instead of always organized SW basics, what is the methods? Methods themselves so 5 template progressions 8000 chromatographic methods so on to various methods.

So we will just go to one of these you go to 8270 this method for semi volatile organic compound using GC/MS gas chromatography which same thing, the method looks less like what we saw in the other EPM there is a description summary what is it used for application notes so on all this you can see all this information is given their 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.

So, these are the 3 sources that you can directly go to you in irrespective of what I do in this class whenever you want to get any analysis method. What we will do in this class will help you understand is to interpret whatever is there in the methodology more. Little more fundamentally what we are why are we selecting certain methods okay. (Video Ends: 26:49)