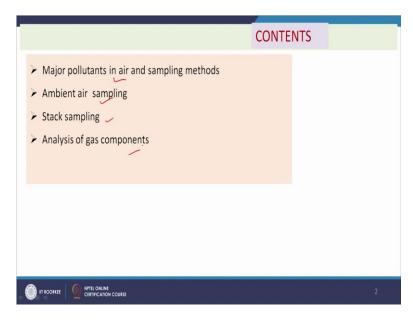
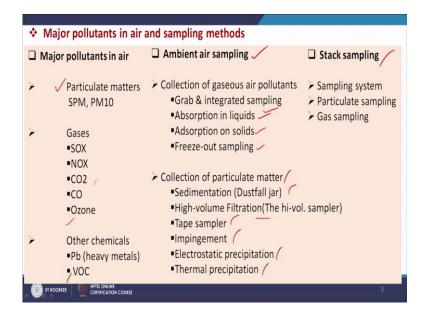
Basic Environmental Engineering and Pollution Abatement Professor Prasenjit Mondal Department of Chemical Engineering Indian Institute of Technology Roorkee Lecture 16 Sampling and Characterization – 1 (Gas, Air, Emission)

Hello everyone, now we will discuss on the topic sampling and characterization part 1, as we have discussed in our previous classes that different types of pollutants are present in air, water and soil and above certain values these pollutants are harmful to our health. Thus there are some standards have been prescribed by CPCB to follow so that environmental quality will be maintained, we have already discussed.

Now we will discuss how to analyze or characterize these quality parameters, analyze these quality parameters or characterize the air, water and soil samples and when we want to detect a particular pollutants in either water, air or soil, sampling is very very important so one way is sampling and another is its characterization or analysis. so those part are very very important and we will be discussing in this class regarding the sampling of gaseous samples and air samples or industrial emissions and also how to characterize those.

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So the contents will be major pollutants in air and sampling methods then ambient air sampling, stack sampling and analysis of gas components. Now as we are concentrated on the sampling of air so some report we have got that this area some pollutants are available so we want to analyze the water quality so we need to take the samples from it. so now we can take ambient air sample when it is needed or we can take industrial air sample that means the waste gas and flue gas which is coming out from a chimney so that may be needed to be collected and the sampling for its analysis.

So exactly what pollutants are coming out from the stacks if we want to know we need to collect the sample from that and then we have to carry it for further analysis. similarly from any environmental that is ambient air in our normal environment if we want to get the concentration of pollutants then we have to again collect the samples but you see that two different cases are different.

So ambient air sampling and stack sampling both we will discuss and we are interested with air quality so certain parameters are necessary already we have discussed those so here particulate matters and SOX, NOX, carbon dioxide, carbon monoxide, ozone and lead and VOC already we have discussed. so now if we think about the ambient air sampling then we have to collect the gas or air and then we have to send it for analysis and there are a number of methods for this purpose one is graph and integrated sampling, next is absorptions in liquids or adsorption on solids or freeze out sampling.

So we are talking about the sampling of ambient air so in that case directly we can take the air from the ambient or the environment or from the atmosphere and or this air can be passed through some liquid for its absorption or it can be passed through some solids for adsorption and then those will be analyzed further. so these are indirect method and somewhere it is possible that we are cooling the air and at lower temperature the pollutants are being separated from the air and we can collect it and analyze so that way different methods have been explored and used for its collection and the sampling process.

Similarly for collection of particulate matter when our major concern is to find out the particulate matter concentrations. So there are some methods specified that is say sedimentation we can use dustfall jars, we can use high volume filtrations or high volume sampler and tape sampler, impingement, electrostatic precipitation and thermal precipitation so these are the different methods which can be used and people have reported this.

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Ambient air sampling	Grab & integrated sampling
 place within an interval of a few second portion removed from the gross same Since there is no alteration to the same is often referred to as —Whole Air Sa When a series of grab samples are of grab s	ample (pollutant separation by sorption), grab sampling ampling. combined or when a grab sample is conducted at a low period of time, up to 24-48 hours, the sample is said to ted sampling).
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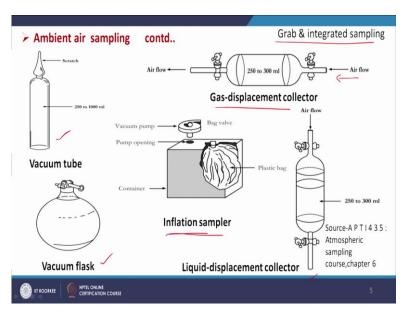
Now we will see grab and integrated sampling, so grab sampling means within very small span of time say few seconds or one or two minutes I am collecting some sample from the environment or atmosphere. so that is grab sampling and integrated means we are taking number of grab samplings and taking the average of it or we are developing a certain method that continuously for certain more duration maybe say 24 hours very small amount of samples we are collecting we have developed a special technique so that way we will get the integrated sampling.

So the term grab sample suggest two concepts a sample taken at a particular time and place within an interval of a few seconds to a minute or two and a small representative portions removed from the gross sample with no alteration. That means if we have a air sample in a bigger volume so we can take small volume from it also so that is also example of grab sample and in this case as such we are taking there is no change like unlike other method like say adsorptions on liquid or adsorption on solid we are not going for directly without any alteration we are collecting the sample.

Since there is no alteration to the sample grab sampling is open referred to as whole air sampling and when a series of grab samples are combined or when a graph sample is conducted at a low sample flow rate over an extended period of time up to 24 to 48 hours the sample is said to be an integrated sample. so we have already discussed. And then grab samples are usually collected in one of the following ways there are number of ways to do the grab sampling.

We can use an evacuated container by purging or water displacement or inflation of a plastic bag or using a syringe so these are the different methods we can apply for the grab sampling. And evacuated containers used for grab sampling may be of different types like say stainless steel canister, vacuum tubes and vacuum flasks.





So here some photographs are shown here for this grab and integrated sampling, so this is your vacuum tube, so vacuum flask and this is gas displacement collector. so when I say this is air flow and when I want to collect the air so air will be getting entry here so this may be 250 to 300 ml volume so after certain time of flow we will be closing this two end and it will collect so that is one way or we can say liquid displacement also if already filled with liquid

and then we will send the water so liquid will be displaced and that volume will be occupied by the air or any gas sample so that way also we can use for the sampling purpose.

And another is your inflation sampler so if we have a just like a, all of us we have seen that the bladder of a football so there we can insert air so similar way this is the inflation sampler a bag, plastic bag is there which can be filled with the gas also through its, this is the bulb through which this can be possible.

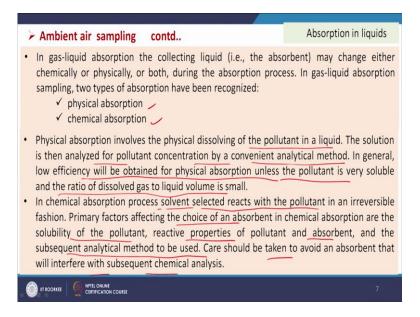
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Storage properties of gases and vapors in plastic bag Gas or vapour Bag type Various Various Vinyl chloride Aluminised Hydrocarbons PVF Vinyl chloride Tedlar Hydrocarbon Saranex Mylar, tedlar Mylar, tedlar Benzene tedlar Chlorinated hydrocarbons tedlar Methanol,acetone; Saran, Teflon Benzene, butadiene; Halar, tedlar Butane, trichloroethylene aluminised	Ambient air sampling	contd	Grab & integrated samplin	
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Butane,trichloroethylene aluminised	Benzene, butadiene;	Hal	ar, tedlar 🦯	
	Butane, trichloroethylene	alui	ninised	

So different types of gas components are present in the gas so if we have some idea so accordingly we can select different types of materials for thus collection of these gas samples like for example say gas or vapor, different various types of gas or vapors we can have various types of bags and like say vinyl chloride we can go for aluminized bag, hydrocarbons PVF polyvinyl fluoride and vinyl chloride Tedlar, hydrocarbon.

So, Saranex, Mylar Tedlar, these are the different types of bag their composition is different and then benzene we can go for tedlar so chlorinated hydrocarbons again tedlar. So methanol, acetone and saran, teflon and benzene butadiene halar tedlar and butane trichloroethylene so aluminized, so this is some typical example.

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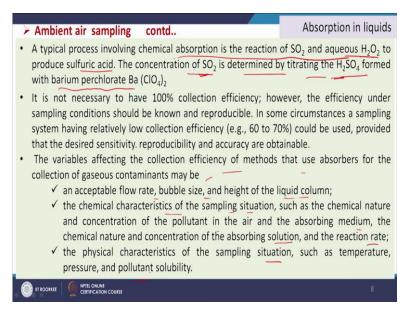


Now we will see the absorption in liquids so those are the grab sampling is direct sampling from the atmosphere but in case of this that is absorption in liquids the air will be passed through a liquids or a solution when the air components will be absorbed in the liquid and then those will be analyzed by some instrumental methods. This capturing of this components in the liquid phase may be by physical or maybe of chemical absorption types there may be physical absorption or maybe chemical absorption.

And physical absorptions involves the physical dissolving of the pollutant in a liquid the solutions is then analyzed for pollutant concentrations by a convenient analytical method, in general low efficiency will be obtained for physical absorption unless the pollutant is very soluble and the ratio of dissolved gas to liquid volume is small.

In chemical absorption process solvent selected reacts with the pollutant in an irreversible fashion so primary factors affecting the choice of an absorbent in chemical absorptions are the solubility of the pollutant and then reactive properties of the pollutant and the absorbent and the subsequent analytical methods to be used. care should be taken to avoid an absorbent that will interfere with the subsequent chemical analysis. So we cannot select any absorbent that will hamper the analysis in the next step.

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One example is given here that is a typical process involving chemical absorptions is the reaction of SO_2 and aqueous H_2O_2 to produce sulfuric acid so the concentration of SO_2 is determined by titrating the H_2SO_4 formed with barium perchlorate. so this is one example so SO_2 is converted to sulphuric acid by this dissolution or absorption in H_2O_2 plus water solution and then that is titrated against this and there is some relationship between the SO_2 presence and H_2SO_4 formed so that way it is determined.

And in this case it is not necessary that all pollutants is absorbed by the absorbent but it is very essential that it must be repetitive. so if we perform the same experiments again and again under the same conditions so then the result should be very near with it, repetitive that means the error limits will be within very narrow range so this is the basic requirement for the gas sampling through the absorption in liquid phase.

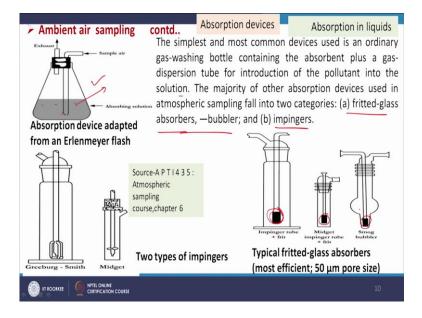
The variables affecting the collection efficiency of methods that use absorbers for the collections of gaseous contaminants may be an acceptable flow rate, bubble size and height of the liquid columns. The chemical characteristics of the sampling solution such as the chemical nature and the concentrations of the pollutant in the air and the absorbing medium, the chemical nature and the concentration of the absorbing solutions and the reaction rate. the physical characteristics of the sampling situation such as the temperature, pressure and pollutant solubility, so that will also play a role for the sampling.

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Ambient air sampling	contd	Absorption in liquids
	Absorption devices	
Type of absorber	Absorbent capacity(ml)	Sample rate (ml/min)
Simple gas	5-100	5-3000
Washing bottle		
Spiral and helical	10-100	40-500
Fritted bubblers	1-100	500-100 000
Glass- bead columns	5-50	500-2000
Viscosity of sorbent is ve	ry high	
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So main objective of the sampling is to give representative data that means which is available the quality which is available in reality when we are taking the sample that sample will represent the bulk properties basically so that is the main objective. Now here we will see the some example type of different type of absorber and different absorbent capacity and sample rate. so simple gas washing bottle, may be spiral and helical absorber, it may be fritted bubblers or may be glass bead columns so in this case they are having different absorbent capacity and different sample rate.

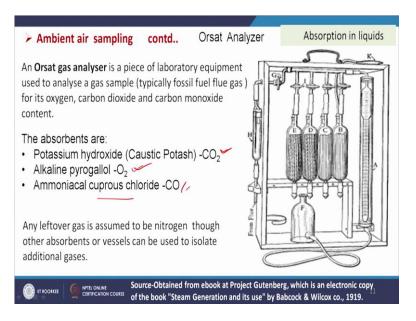
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So some photographs are shown where the absorptions in liquids is performed, this is very simple one the simplest and the most common device used is an ordinary gas washing bottle containing the absorbent and a gas dispersion tube for introductions of the pollutant into the solution. so from this the gas will be coming we will be having some liquid and then the pollutants or the elements will be absorbed here the rest gas will be going out. so then we will be collecting this one and will analyze for this so that way we will be able to get the sample and further analysis we will do.

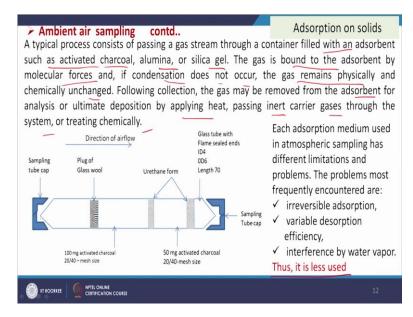
Other types of devices which are used for this purpose may be into maybe a fritted glass absorber and bubbler and a impingers. So as shown here so these are impingers, and here impingers but fritted glass so this part is in addition so if we compare this so this part is fritted part so that is called fritted glass absorber and impingers so these are the different equipment or devices which are used for this purpose.

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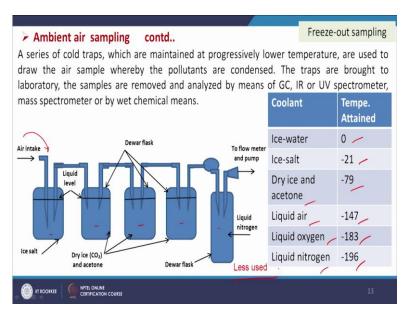
Another process for the analysis is your orsat analyzer so in that case basically flue gas from the fossil fuels is targeted to measure the concentration of CO_2 , O_2 and CO present in it. So this is your set analyzer you see different columns are there in these we have different solvents so selective absorption is possible like say potassium hydroxide if we use in any one of it so carbon dioxide will be selected selectively absorbed and then alkaline pyrogallol will selectively absorb oxygen and ammoniacal cuprous chloride will absorb carbon monoxide so we have more here. So other solvents can be used for the determination of other gas components expected otherwise we will be considering as the nitrogen, so this is one way we can do the analysis of the gas but this accuracy is not that high in that case and then we are going to adsorptions on solids.

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So similar way so if we pass the gas samples on a solid then solid, there will be some adsorption so maybe physical or chemical adsorption so that can also be collected and further it can be taken out from it and then analyzed. So a typical process consists of passing a gas stream through a container filled with an adsorbent such as activated charcoal, alumina or silica gel, the gas is bound to the adsorbent by molecular forces and if condensation does not occur the gas remains physically and chemically unchanged. Following collection the gas may be removed from the adsorbent for analysis or ultimate deposition by applying heat, passing inert carrier gases through the system or treating chemically.

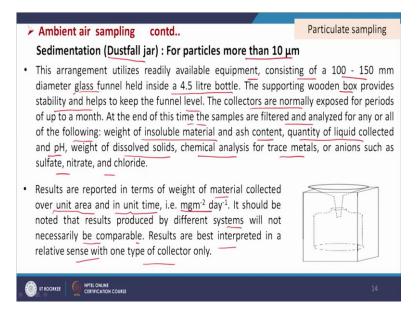
So these are the next phase of process through which the analysis will be done but basically say if we have some sample tube it will coming and then there are some adsorbents so then will be adsorbed and it will be taken out and then further treatment will be done. But this method it is less used, this method is not widely used but this can be used for very specific purposes but normally not that use. (Refer Slide Time: 18:28)



Another is freeze out sampling so as I mentioned that if we reduce the temperature so that component may be selectively separated for example here say ice water if we use the temperature, we can get 0, ice salt -21, dry ice and acetone -79, liquid air -147, liquid oxygen -183 and liquid nitrogen -196 we can get.

So, different different coolants we can use in different chambers and then if we pass the air so the pollutants may be collected, different pollutants may be collected in different chambers. So that way the freeze out sampling is also can be one options for this collection and analysis but this is also less used, it is not used very larger extent.

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And then we are going for particulate sampling so we are talking about the gas sampling when our main concern is to know the composition of gases present in the air like say SOX, NOX, hydrocarbon etc. But here when we are concentrated with the particulate sampling that means we are more concerned with the particulate matter, particulate matter like say PM 10, PM 2.5, PM 4 like this so in that case other type of devices are normally used so here we are discussing those.

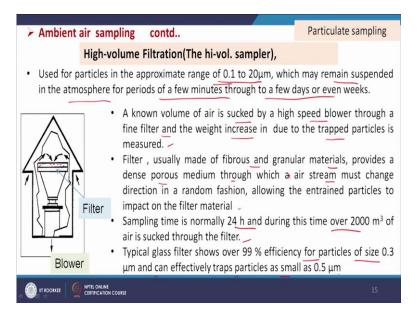
So one is sedimentations or dustfall jar for particles more than 10 mm. we can use the dust fall jar as shown here, so we have here one funnel so then it is put in this box so we will put it for certain time so the particulates etcetera which is available in the air will also be come in contact with this surface area of this and it will be collected.

So after certain time interval we will collect it and we will see how much solid is collected so that per area per time how much material is collected so that we can calculate. so you see this arrangement utilizes readily available equipment consisting of a 100 to 150 mm diameter glass funnel held inside a 4.5 liter bottle the supporting wooden box provides stability and helps to keep the funnel level, the collectors are normally exposed for periods of up to a month at the end of this time the samples are filtered and analyzed for any or all of the following.

Maybe depending upon our situation we may go to analyze any one or all of those, so that weight of the insoluble material and ash content quantity of liquid collected and pH, weight of dissolved solids, chemical analysis for trace metals or anions such as sulphate, nitrate chloride, etc.

And results are reported in terms of weight of the material collected over unit area and in unit time that is mg/m^2day . Because it is for a longer period the sample is taken so that way the results are presented, it should be noted that results produced by different systems will not necessarily be comparable so results are best interpreted in a relative sense with one type of collector only. So which we can conclude this is an option but this may not give us an accurate information.

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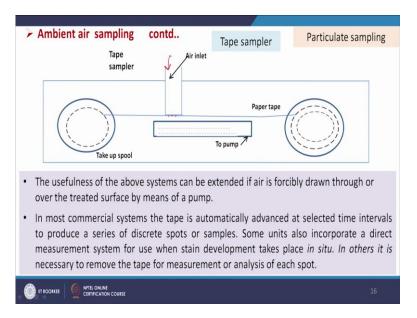
So another option is high volume filtration, so here you see this is high volume filter so we have here one filter cloth and then there is one blower so there will be suction of ambient air through this device and if the particles are present so those will be deposited here. So after certain time of operation we will be collecting this filter cloth and we will see the mass changes so we will get the mass of the particles which were present in the total volume of air suction of which was made through this device.

So used for particles in the approximate range of 0.1 to 20 μ m which may remain suspended in the atmosphere for a period of few minutes through to a few days or even weeks. That is very very small particles can also be separated, so a known volume of air is sucked by a high speed blower through a fine filter and the weight increase in due to the trapped particle is measured just we have discussed.

And filter usually made of fibrous and granular materials provides a dense porous medium through which a airstream must change directions in a random fashion allowing the entrained particles to impact on the filter material and sampling time is normally 24 hour. And during this time over 2000 m^3 of air is sucked through the filter.

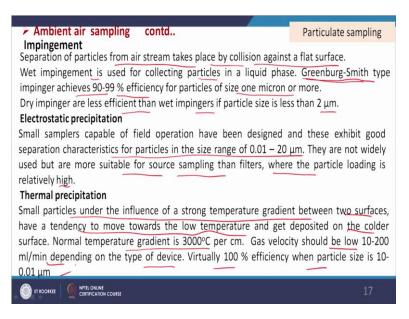
So relatively large amount of air is sucked because the collection will be sufficient for analysis. And typical glass filter shows over 99 % efficiency for particles of size 0.3 μ m and can effectively traps particles as small as 0.5 μ m.

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Next another type is your tape sampler, so tape sampler so then we know this is our air inlet so there is a tape which will be moving and then if it is a particulate is present and from this we have a pump. So here will be passing through this and particulates are there so that will be arrested here on the tape and this step is moving so after certain time we can collect or we can measure how much particles have been deposited on the tape and how much volume we have collected so that way it can be done.

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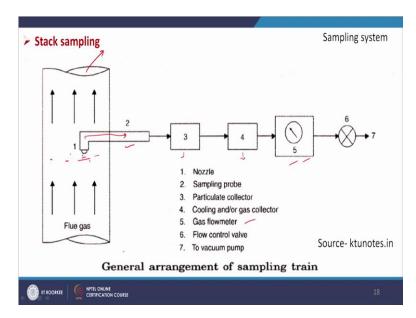
Now impingement, so what is this separation of particles from air stream takes place by collision against a flat surface. so wet impingement is used for collecting particles in a liquid

phase. And one example Greenberg Smith type just we have seen in the previous slides that type of impingers. so Greenberg Smith type impinger achieves 90 to 99 % efficiency for particles of size 1 micron or more and dry impinger are less efficient than wet impingers if particle size is less than $2 \mu m$.

And electrostatic precipitation this technique can also be used small samplers capable of field operation have been designed and these exhibit good separation characteristics for particles in the size range of 0.01 to 20 μ m. They are not widely used but are more suitable for source sampling than filters like say high ball filters so where the particle loading is relatively high.

And thermal precipitation so here the small particles under the influence of strong temperature gradient between 2 surfaces have a tendency to move towards the low temperature and get deposited on the colders surface. So normal temperature gradient is 3000 $^{\circ}$ C/cm. So gas velocity should be low 10 to 200 ml/min depending on the type of device and virtually 100 percent efficiency when particle size is 10 to 0.1 µm. so these are some technically available devices or these are the techniques available but not widely used in application.

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Now we are going to discuss on the sampling systems related with stack samples. So stack samplings so this is the diagram which shows that so this is our stack so flue gas is generated and it is going out say so we want to get the sample from this stack. So how we will do it so you, these are the different parts for this so 1 is your nozzle and 2 is your sampling probe so

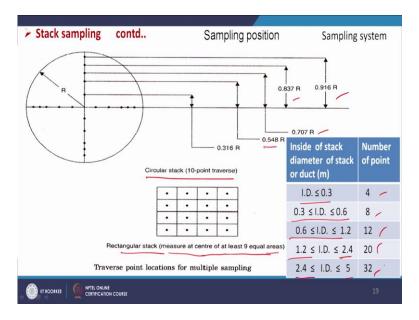
we will be inserting one probe inside the stack attached with the nozzle and then so pressure is higher so gas will be coming out this side and then we have particulate collector.

So it is coming in particles are there then that will be collected here then it is coming to cooling or gas collector so here it may be high temperature if it is it may need some cooling so that our gas collector and then it will be going to gas flow meter so what is the gas is going out that we know so here if we are interested to get the particles so that can be available here we can get the gas components for gas components we can make some devices here so that different gas components can be collected or the whole gas can be collected.

And then we are going for flow control valve and then 6 is your flow control valve and 5 is your gas flow meter. So we know how much gas is going out and how much particulates are collected so that way we can calculate the particulate matter concentration and we can directly get sample here or we can take apply any methods which we have discussed for the collection of gas samples may be absorbed in liquid or adsorbed in solid or directly sampling we can take and then we will get the volumetric flow rate of the outlet gas and one will be the control valve that will maintain the flow rate, so this is the devices or the process which is used for stack sampling.

Now one question, so what will be the position of this nozzle. So if the stack is circular or stack may also be your rectangular so different shape different diameter so stacks may be so how many samples we will take from this one and what will be the location those are very very important so that we can get the representative sample.

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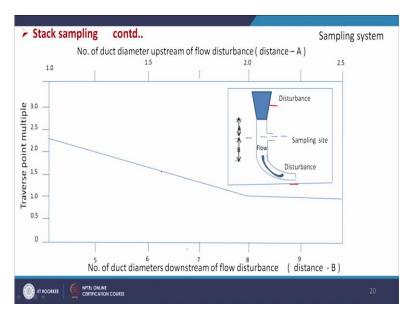


So that is why one guideline is here so circular stack there 10 point traverse is shown here so 1 2 3 4 and 5, 10 point traverse and here it is say rectangular stack at least you know measure at center of at least 9 equal areas so will can get anyway see if we have a cross sectional area so we will be knowing the dimension of it and make a diagram and then we will identify what will be the position for the sampling so that way and you see here see if we go for consider for this 10 point.

So then 1 2 3 4 5 so if this is R so this positions may be 0.316 R so then equal to this one and the next one is equal to 0.707 R and this distance may be this one and this distance may be this so that way some guidelines are available. And depending upon the diameter how many sampling points will fix that is also some guidelines are given that is this ID less than 0.3 m so 4 number of point and if it is 0.3 to 0.6 m so then 8 number of points 0.6 to 1.2 it is 12 number of points so 1.2 to 2.4 that is 20 number and 2.4 to 5 then equal to 32, so these are the guidelines.

So, certainly the experience also will help to collect the samples. And another important is that in the stack where we are collecting the samples there may be different bends. So if we have a bend or some disturbance of to the flow inside the stack then where we will collect the sample that is also a matter of question.

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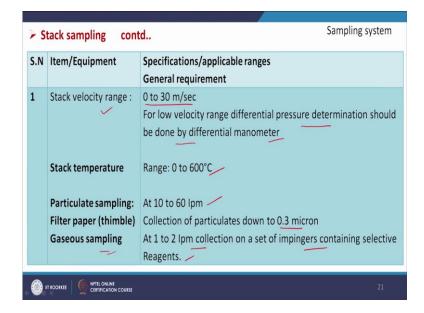


And there are some guidelines like this say if this is one disturbance and this is one disturbance, this disturbance means so this is suddenly the diameter is changing that means the flow will change here also there is a bend. That means flow will change disturbance means that these will help to change the flow inside the stack. So in such cases our objective will be to collect samples where the isokinetic conditions is maintained so when the gas is generated and it is going towards the stack the same velocity also be available in the sampling point so that is the main concern.

And some guideline are also there say you see here so this is for example say our sample point so A and B so what is A, A is your number of duct diameter upstream of flow disturbance so this is one flow disturbance, so this flow disturbance this is your upstream and number of duct diameters downstream of flow disturbance so for this respect so this is our, for this disturbance this is our downstream disturbance. So then so if this is a bend is there so after bend certain, after certain distance we can take this one, what is that, that is b.

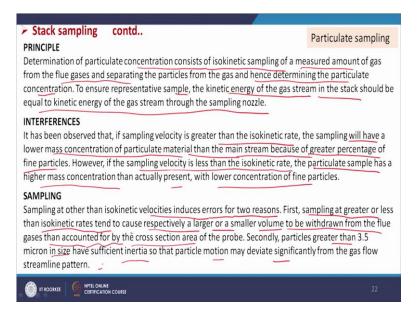
So here 5 6 7 8 9 these are nothing but the number of duct diameter. So what is the duct diameter that will be multiplied by that so 5*R*2 like this so that way and A will be the number of duck diameter upstream of flow disturbance, so this distance will be the A that is 1, 1.5, 1 point so for any point we can consider, so what will be the number of duct diameter upstream, what will the number of duct diameter downstream so accordingly we can calculate.

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And there are some conditions which are also maintained for the stack sampling like say stack velocity range what will be the stack velocity that is 0 to 30 m/s and for low velocity range differential pressure determination should be done by differential manometer and stack temperature may be 0 to 600 °C. And particulate sampling that is filter paper or gaseous sampling so both sampling we can do at 10 to 60 lpm. And this is for particulate sampling and collection of particulates down to 0.3 microns we can collect up to this and then for gas sampling at 1 to 2 lpm collection on a set of impingers containing selective reagents that we have discussed in the previous slide.

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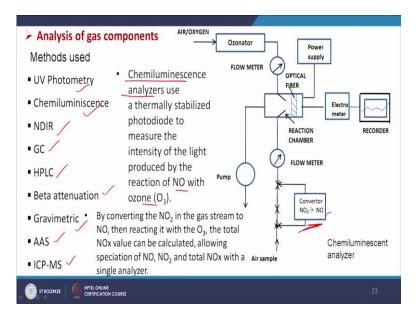
So these are the different consideration or different aspects we should consider. And now we will see the principle for particulate sampling as I already have mentioned. The determination of particulate concentration consists of isokinetic sampling of a measured amount of gas from the flue gases and separating the particles from the gas and hence determining the particulate concentration. To ensure representative sample the kinetic energy of the gas stream in the stack should be equal to kinetic energy of the gas stream through the sampling nozzle. So that is very very important consideration for the gas sampling and that is why different guidelines have been provided.

Now it has been observed that if sampling velocity is greater than the isokinetic rate the sampling will have a lower mass concentration of particulate material than the main stream because of greater percentage of fine particles. However if the sampling velocity is less than the isokinetic rate the particulate sample has a higher mass concentration than actually present with lower concentration of fine particles and then why so, let us see.

So sampling at other than isokinetic velocities induces errors for two reasons what are those, the sampling at greater or less than isokinetic rates tend to cause respectively larger or a smaller volume to be withdrawn from the flue gases than accounted for by the cross section area of the probe. And secondly the particles greater than 3.5 micron in size have sufficient inertia so that particle motion may be deviated significantly from the gas flow streamline pattern.

So these are the concern regarding the sampling or stack sampling. Now we have covered how to do the ambient air sampling and stack sampling, next another is characterization also how we will characterize the gas samples which we have collected. So different instrumental methods can be used for this already we have discussed in the previous class and we will also make some discussion here.

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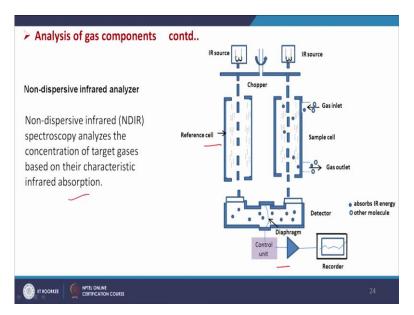


So for analysis of gas components different methods are used that is UV photometry, chemiluminescence, NDIR non dispersive infrared, gas chromatography, high performance liquid chromatography, Beta attenuation, gravimetric analysis, atomic absorption spectroscopy, inductively coupled plasma mass spectroscopy, so we have discussed some of these in the previous classes now we will discuss on chemiluminescence.

So here you see in this chemiluminescence NO reacts with ozone and then that produces some intensity of light so that intensity of light is proportional to the concentration of NO. So this chemiluminescence analysis or analyzer is used to measure the NO concentrations in the gas sample. And if you want to get the NOx say $NO_2 + NO$ then NO_2 can be converted to NO as shown in this figure here NO_2 is converted to NO, so NO is going there and ozone is provided so NO will react with NO_3 and give the light so when we will not make this reaction here so NO and NO_2 both are going so in that case only NO will give the intensity of light.

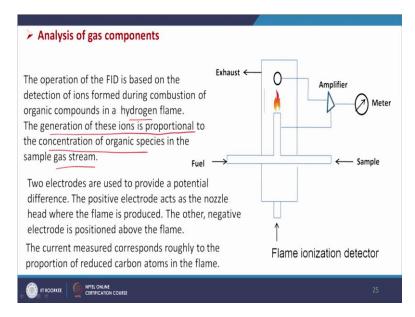
But when we are doing this reduction here so all NOx present in it now in present in the NO form and will give more intensity here. So in this case we will get total NOx and if we do not do this reaction here then that will give only NO so by the the difference will give us NO_2 . So that way this chemiluminescence method can help to determine the NOx concentration in the gas sample both the NO and NO_2 as well as the total, so like the speciation of NOx can also be possible.

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Non dispersive infrared analyzer, non dispersive infrared spectroscopy analyzes the concentration of target gases based on their characteristic infrared absorption. Like for example say so this is our IR source when it is coming through it so this is our reference cell there is no gas component and here the gas components are coming so this gas components will absorb this IR ray and will be heated up so when it is coming here so this diap, this volume will be increased so this diaphragm will be shifted that way and it will give some signal and the control unit will help to determine the concentration of the gas component here.

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And next another is FID is also used to detect the gas component present in the gas stream. So here the like organic compounds are combusted in a hydrogen flame so then the generations of these ions is proportional to the concentration of organic species in the sample gas stream.

So, here two electrodes are there so the ions are generated due to the combustion that is the flame ionization, the hydrogen flame so organic compounds are converted into ions and how much electricity is produced that is amplified and then it is metered and this is proportional to the concentration of the pollutants of the components present in it, so that way also we can measure the gas component present in the sample. So upto this in this class, thank you very much for your patience.