Environmental Soil Chemistry

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Lecture – 57
Analytical Techniques for Assessing Soil Pollution Continued

Welcome friends to this second lecture of week 12 of this NPTEL online certification course of environmental soil chemistry, and in this week, we are talking about analytical techniques for assessing soil pollution.

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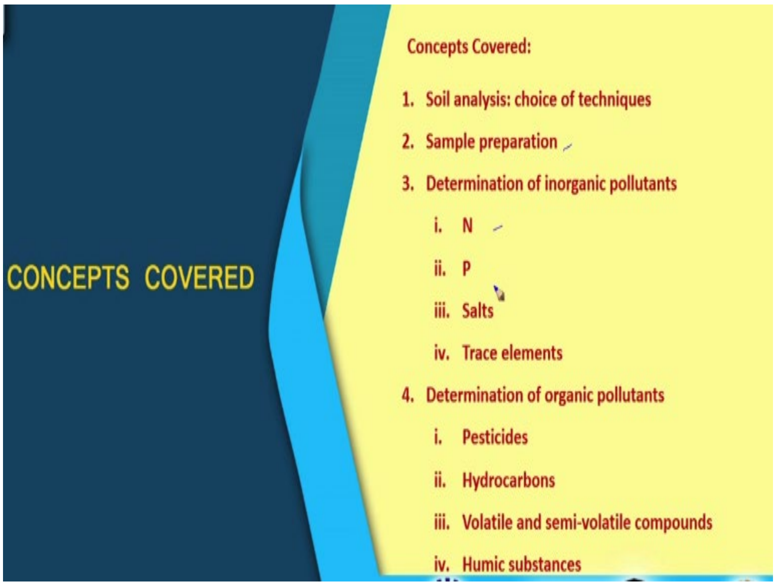

CONCEPTS COVERED

Concepts Covered:

1. Soil analysis: choice of techniques
2. Sample preparation
3. Determination of inorganic pollutants
   i. N
   ii. P
   iii. Salts
   iv. Trace elements
4. Determination of organic pollutants
   i. Pesticides
   ii. Hydrocarbons
   iii. Volatile and semi-volatile compounds
   iv. Humic substances

So, in our previous lecture, we have covered the following concepts. We have started with choice of techniques for soil analysis and why soil analysis important and then we talked about the sample preparation, and then we started with the determination of inorganic pollutants. So, we started with the nitrogen and we will try to finish the nitrogen today and then we will start from phosphorus and salts and so on and so forth.

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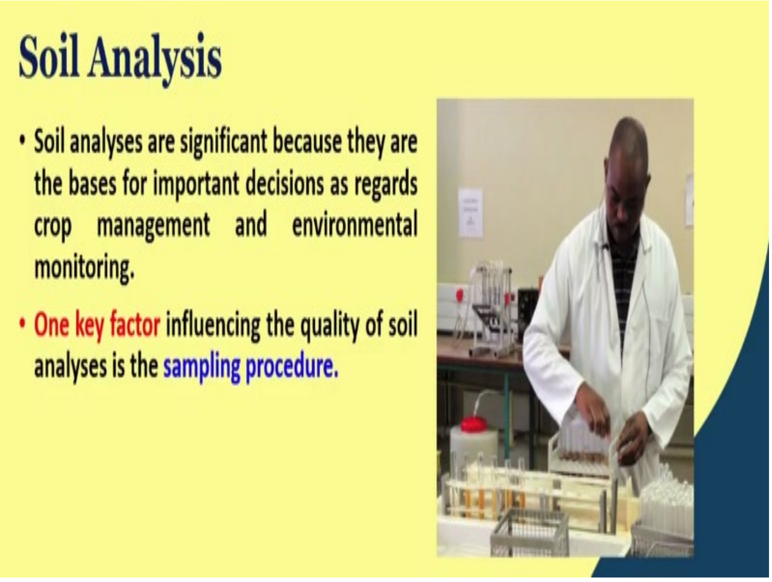

Soil Analysis

- Soil analyses are significant because they are the bases for important decisions as regards crop management and environmental monitoring.
- One key factor influencing the quality of soil analyses is the sampling procedure.

So, in the previous lecture, the first lecture of this module 12, we have covered these basic aspects of soil analysis.

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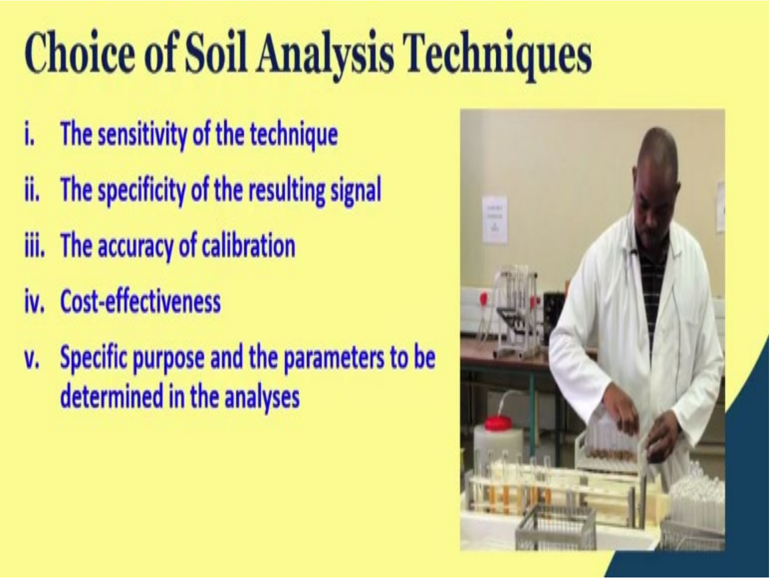

Choice of Soil Analysis Techniques

i. The sensitivity of the technique
ii. The specificity of the resulting signal
iii. The accuracy of calibration
iv. Cost-effectiveness
v. Specific purpose and the parameters to be determined in the analyses

And then what are the major choices of soil analysis technique, what are the major consideration like sensitivity, specificity, accuracy of calibration, cost effectiveness, specific purpose of the parameters to be determined in the analysis. So, we have discussed all these things in details.

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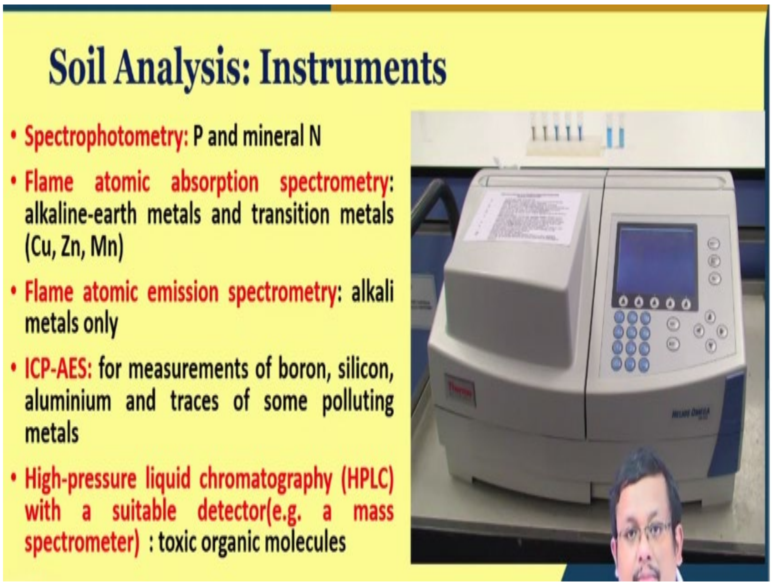

Soil Analysis: Instruments

- Spectrophotometry: P and mineral N
- Flame atomic absorption spectrometry: alkaline-earth metals and transition metals (Cu, Zn, Mn)
- Flame atomic emission spectrometry: alkali metals only
- ICP-AES: for measurements of boron, silicon, aluminium and traces of some polluting metals
- High-pressure liquid chromatography (HPLC) with a suitable detector(e.g. a mass spectrometer) : toxic organic molecules

Then we have talked about some basic instruments like spectrophotometer, flame atomic absorption spectrometry, flame atomic emission spectrometry, ICP-AES, then HPLC and also HPLC-MS like mass spectrometer for measurement of different pollutants.

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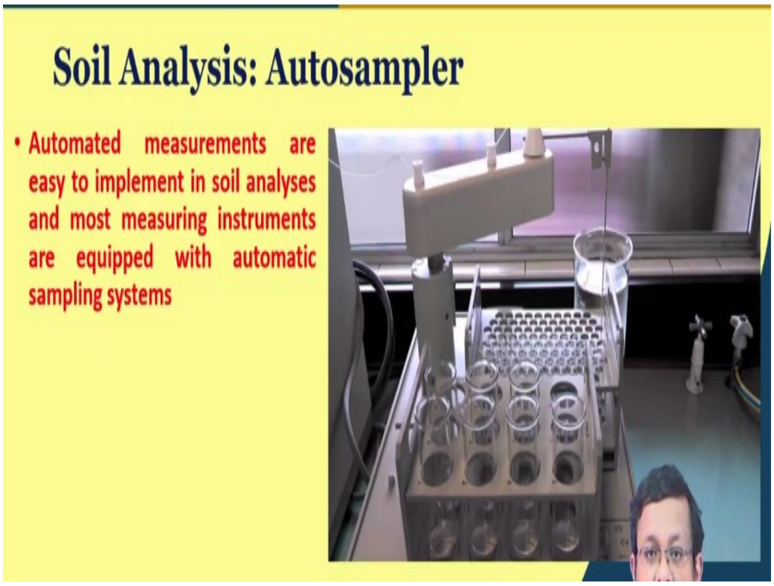

Soil Analysis: Autosampler

- Automated measurements are easy to implement in soil analyses and most measuring instruments are equipped with automatic sampling systems

We also talked about the autosampler which is required for you know automated measurements without human interventions. So, this autosampler is an integral part of different high valued instruments, which generally helps for automatic sampling for subsequent analysis.

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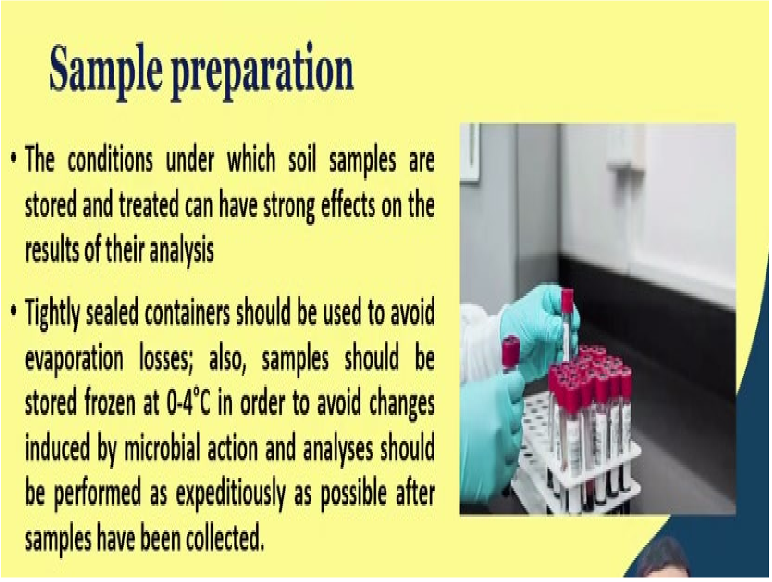

Sample preparation

- The conditions under which soil samples are stored and treated can have strong effects on the results of their analysis
- Tightly sealed containers should be used to avoid evaporation losses; also, samples should be stored frozen at 0-4°C in order to avoid changes induced by microbial action and analyses should be performed as expeditiously as possible after samples have been collected.

Then, we talked about the sample preparation, why it is important.

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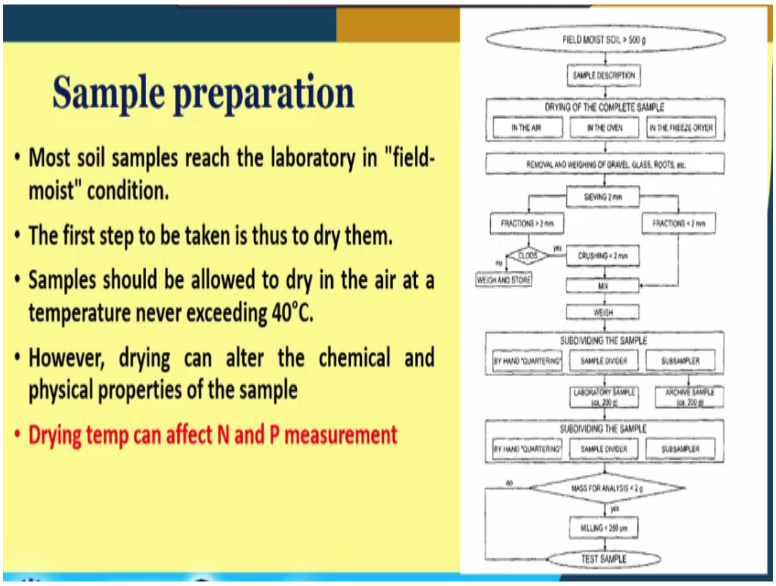

Sample preparation

- Most soil samples reach the laboratory in "field-moist" condition.
- The first step to be taken is thus to dry them.
- Samples should be allowed to dry in the air at a temperature never exceeding 40°C.
- However, drying can alter the chemical and physical properties of the sample
- Drying temp can affect N and P measurement

And we have covered different steps of sample preparation like sample description, drying of the complete samples in the air, oven drying and freeze drying and all these things.

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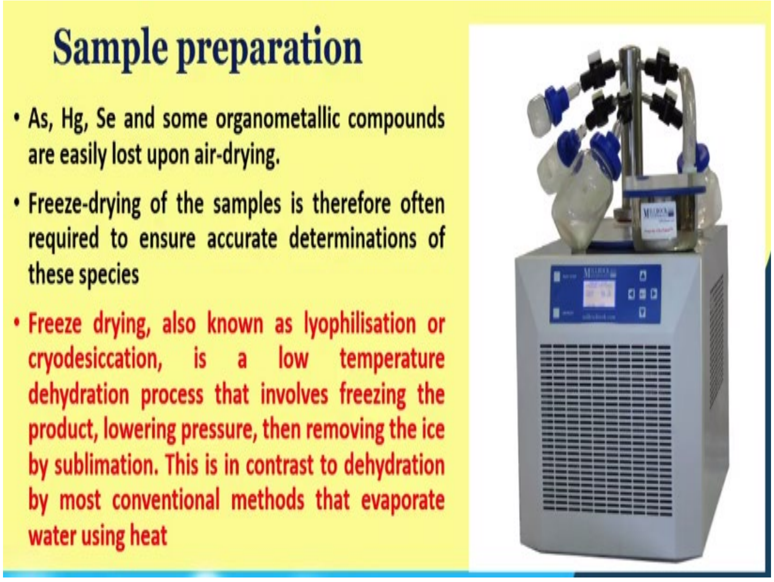

Sample preparation

- As, Hg, Se and some organometallic compounds are easily lost upon air-drying.
- Freeze-drying of the samples is therefore often required to ensure accurate determinations of these species
- Freeze drying, also known as lyophilisation or cryodesiccation, is a low temperature dehydration process that involves freezing the product, lowering pressure, then removing the ice by sublimation. This is in contrast to dehydration by most conventional methods that evaporate water using heat

So, we have discussed the freeze-drying method, why it is required for certain elements like arsenic, like mercury, like selenium, and some other organometallic compounds, which had easily lost upon by air drying. So, that is for these organometallic compounds and these elements, we generally go for freeze drying for sample preparation and keeping those samples for a long period of time.

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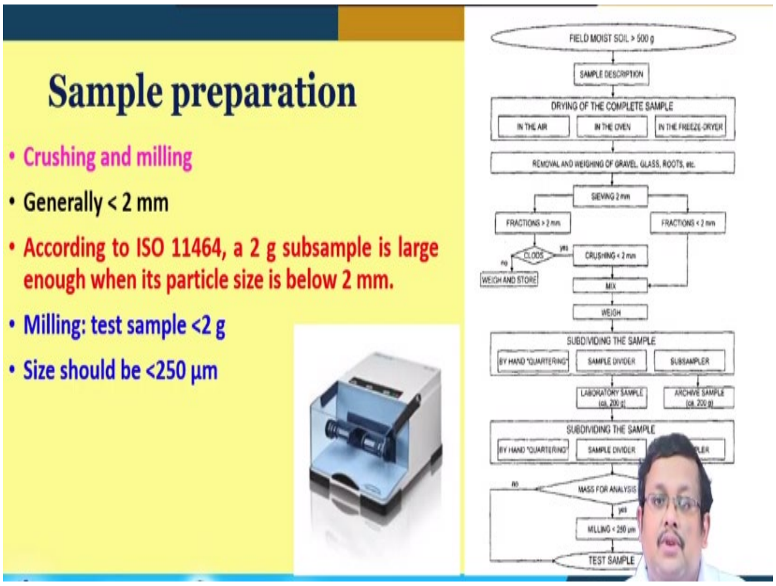

Sample preparation

- Crushing and milling
- Generally < 2 mm
- According to ISO 11464, a 2 g subsample is large enough when its particle size is below 2 mm.
- Milling: test sample <2 g
- Size should be <250 μm

Then, we have talked about the sample crushing, sample homogenization, milling process, crushing process, why it is required, for which condition we will go with the crushing. When the test sample is less than 2 gram, we will go for the milling process and the size should be less than 250 microns for this case.

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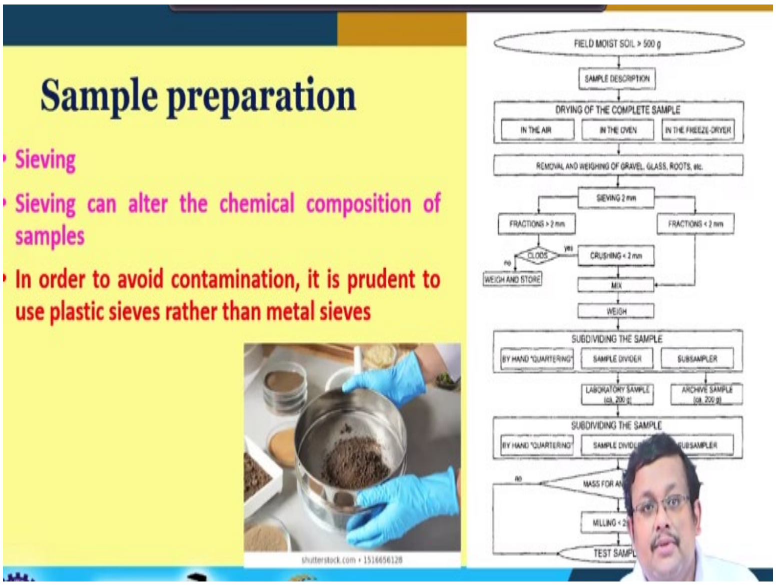

Sample preparation

- Sieving
- Sieving can alter the chemical composition of samples
- In order to avoid contamination, it is prudent to use plastic sieves rather than metal sieves

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After the milling, we have talked about the sieving and why it is required, why it is preferable to have plastic sieve instead of metallic sieve we have also discussed.

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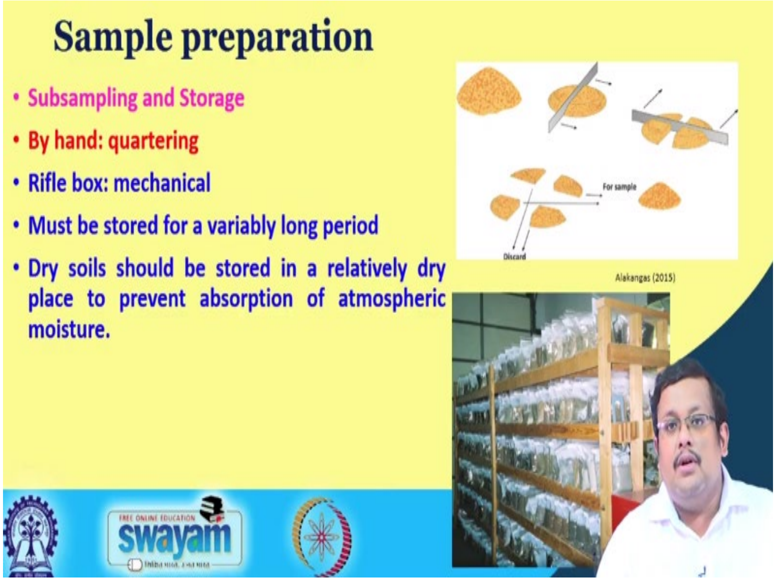

Sample preparation

- Subsampling and Storage
- By hand: quartering
- Rifle box: mechanical
- Must be stored for a variably long period
- Dry soils should be stored in a relatively dry place to prevent absorption of atmospheric moisture.

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Then we have talked about the subsampling and storage. What is the quartering method I have discussed and then what are the consideration for keeping the samples for a long period of time and what is storage we have also covered.

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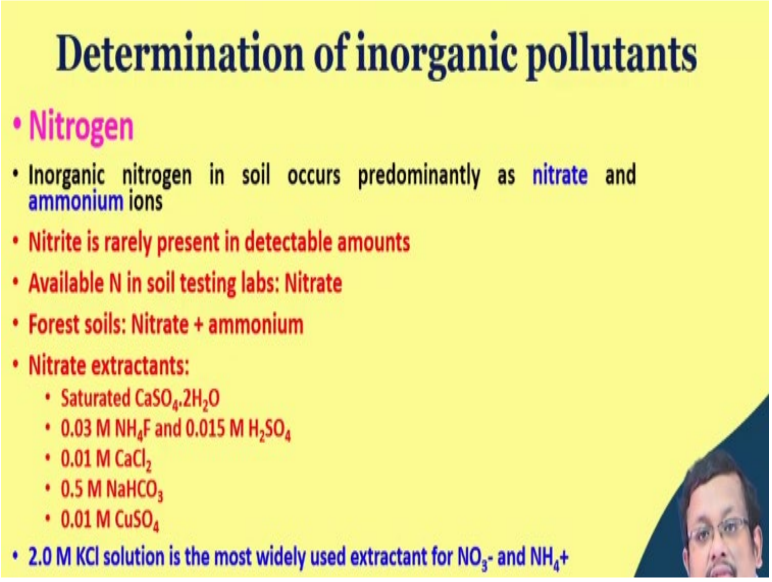

Determination of inorganic pollutants

- **Nitrogen**
- Inorganic nitrogen in soil occurs predominantly as nitrate and ammonium ions
- Nitrite is rarely present in detectable amounts
- Available N in soil testing labs: Nitrate
- Forest soils: Nitrate + ammonium
- Nitrate extractants:
  - Saturated $CaSO_4.2H_2O$
  - 0.03 M NH_4F and 0.015 M H_2SO_4
  - 0.01 M $CaCl_2$
  - 0.5 M $NaHCO_3$
  - 0.01 M $CuSO_4$
- 2.0 M KCl solution is the most widely used extractant for NO_3- and NH_4+

Then we started with the determination inorganic pollutants like nitrogen and we have talked about different extractant like saturated calcium sulfate 2H2O, then calcium chloride, sodium bicarbonate, copper sulfate and so on and so forth.

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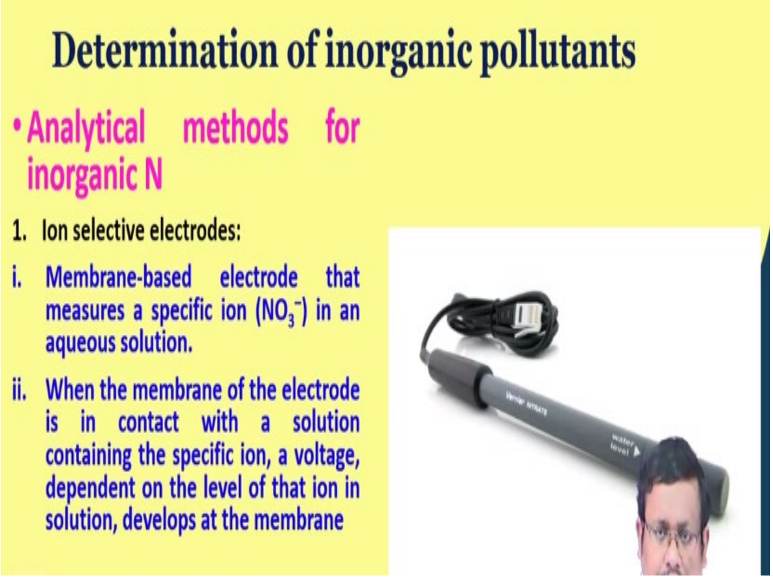

Determination of inorganic pollutants

- **Analytical methods for inorganic N**

1. Ion selective electrodes:

i. Membrane-based electrode that measures a specific ion (NO_3^-) in an aqueous solution.

ii. When the membrane of the electrode is in contact with a solution containing the specific ion, a voltage, dependent on the level of that ion in solution, develops at the membrane

And then we have talked about some analytical methods or instruments or tools which we require for inorganic measurement of inorganic nitrogen. So, we started with ion selective electrodes. These ion selective electrodes are basically membrane-based electrodes that are sensitive to a specific ion in aqueous solution in this case is nitrate ion.

So, when the membrane of the electrode is in contact with the solution containing this specific ion voltage difference or voltage gradient is developed across the membrane and that is basically measured in this ion specific electrode or ISE.

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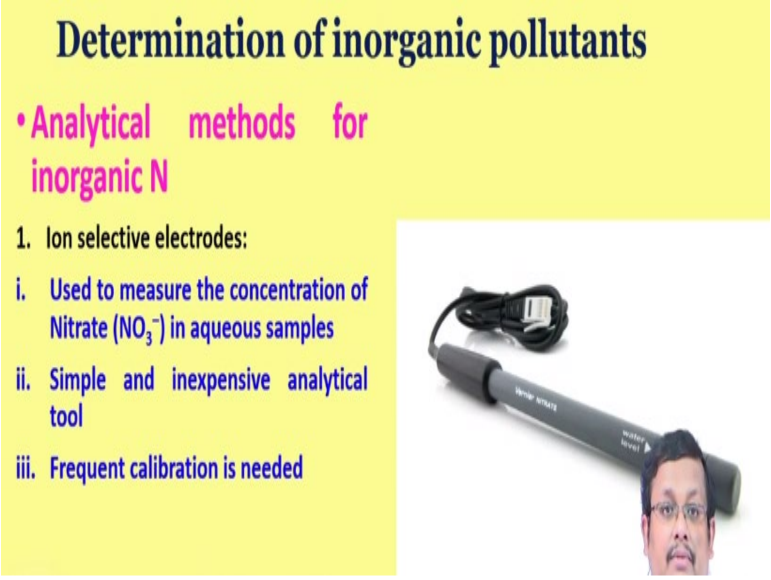

Determination of inorganic pollutants

- Analytical methods for inorganic N

1. Ion selective electrodes:
   - i. Used to measure the concentration of Nitrate (NO_3^-) in aqueous samples
   - ii. Simple and inexpensive analytical tool
   - iii. Frequent calibration is needed

Then you know one of the major drawbacks of the ion selective electrode that sometimes interferences are there. So, frequent calibration is required for these types of electrodes.

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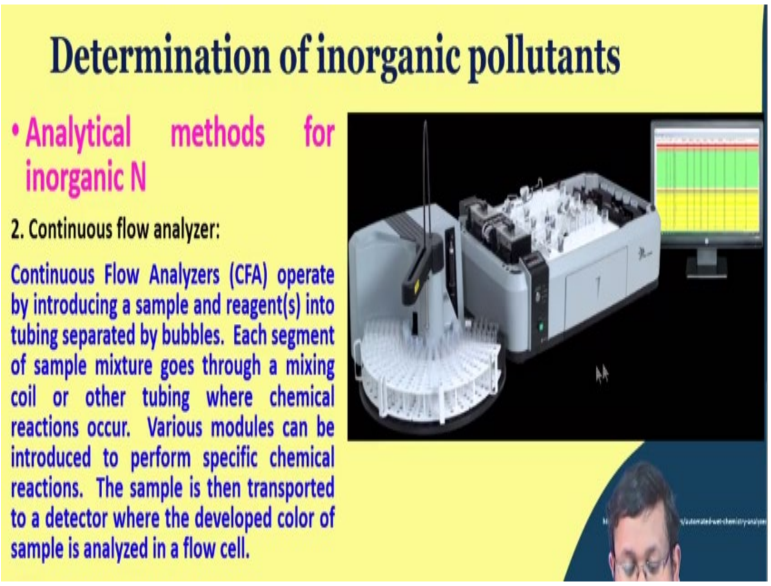

Determination of inorganic pollutants

- Analytical methods for inorganic N

2. Continuous flow analyzer:

Continuous Flow Analyzers (CFA) operate by introducing a sample and reagent(s) into tubing separated by bubbles. Each segment of sample mixture goes through a mixing coil or other tubing where chemical reactions occur. Various modules can be introduced to perform specific chemical reactions. The sample is then transported to a detector where the developed color of sample is analyzed in a flow cell.

So, today we are going to start with this new instrument, I mean this is another instrument which you use for measurement of inorganic nitrogen, we call it continuous flow analyzer or in short form CFA. Now, this continuous flow analyzers basically operates on the basis of continuous flow analysis. Now, this continuous flow analysis or continuous flow analyzer operate by introducing a sample and reagents into tubing separated by bubbles.

I will show you one picture. Now, each segment of sample mixture goes through a mixing coil or other tubing where chemical reactions basically occur. So, various modules can be introduced to perform specific chemical reactions and the sample is then transported to a detector where they develop color. So, there is a color development and this developed color is basically measured.

This developed color is measured and then sample is analyzed in a flow cell. So, this is basically the principle of this continuous flow analyzer. Again in this continuous flow analyzer, the segment of sample mixture goes through a mixing coil and other tubing where chemical reagents basically mix with them. So, these samples are separated by bubbles.

So, this is how this continuous flow analyzer basically operates. You can see here, this is a continuous flow analyzer and this is an autosampler for this continuous flow analyzer.

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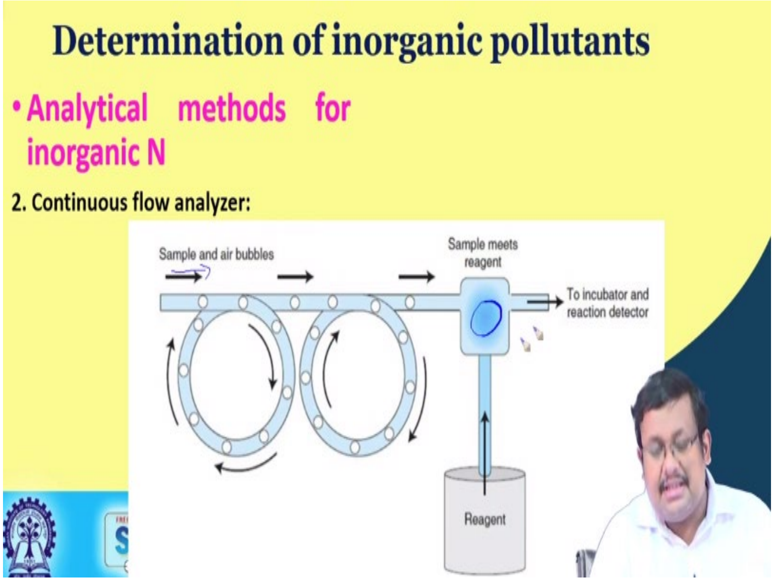

Determination of inorganic pollutants

- Analytical methods for inorganic N

2. Continuous flow analyzer:

Now, as I told you that the principle of continuous flow analyzer says that through this tubing the sample and air bubbles are basically circulated. So, as you can see samples are separated by these air bubbles. So, the samples are moving through these tubing's and the reagent is also pumped in this tubing in the certain parts of the tubing. So, here in this point there is a mixing between the sample and the reagent.

And obviously you know after the mixing, there are some chemical reactions and after this chemical reaction, they move to the incubator and reaction detector where they are being detected by the development of certain colors. So, this is how this continuous flow analyzer basically operates. It is basically the operating principle of this continuous flow analyzer.

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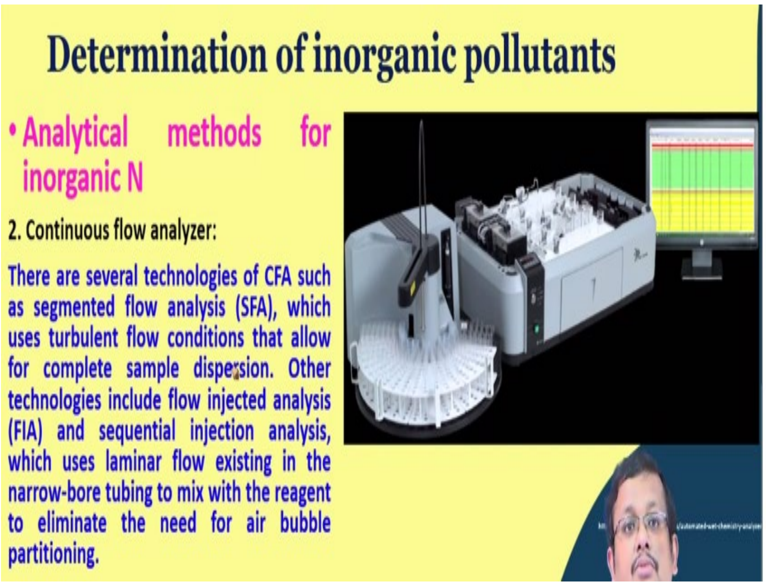

Determination of inorganic pollutants

- Analytical methods for inorganic N

2. Continuous flow analyzer:

There are several technologies of CFA such as segmented flow analysis (SFA), which uses turbulent flow conditions that allow for complete sample dispersion. Other technologies include flow injected analysis (FIA) and sequential injection analysis, which uses laminar flow existing in the narrow-bore tubing to mix with the reagent to eliminate the need for air bubble partitioning.

Now, there are several techniques of continuous flow analyzers such as segmented flow method or segmented flow analysis, in short we call it SFA, which basically use turbulent flow conditions that allow for complete sample dispersion. So, this SFA or segmented flow analysis basically deals with the turbulent flow condition that allow for complete sample dispersion.

Other technique for this continuous flow analysis is known as flow injection analysis, in short form we call it FIA. Now, this flow injection analysis and sequential injection analysis uses laminar flow existing in the narrow bore tube to mix the reagent to eliminate the need of air bubble partitioning. So, there is no need for air bubble partitioning. So, there is a laminar flow existing in the narrow bore tubing basically to mix the reagent and there is no need for air bubbles partitioning.

So, this is called continuous flow analysis and also the instrument is known as, I am sorry this is called the flow injection analysis and the instrument is known as flow injection analyzer. So, this is one type of continuous flow analysis okay.

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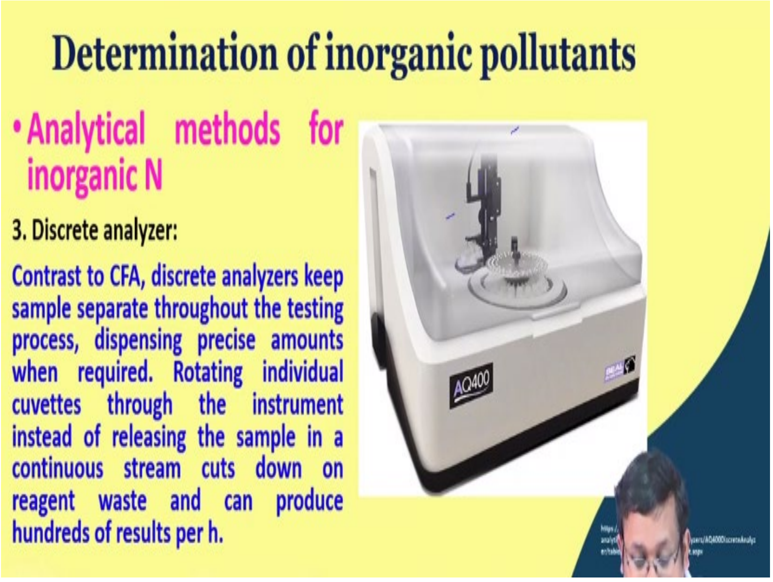

Determination of inorganic pollutants

- Analytical methods for inorganic N

3. Discrete analyzer:

Contrast to CFA, discrete analyzers keep sample separate throughout the testing process, dispensing precise amounts when required. Rotating individual cuvettes through the instrument instead of releasing the sample in a continuous stream cuts down on reagent waste and can produce hundreds of results per h.

So, another instrument is there for measurement of inorganic nitrogen that is called discrete analyzer. Now, what is this discrete analyzer? Now, it is contrast to this continuous flow analyzer, this discrete analyzer basically keeps samples separate throughout the testing process. So, in the continuous flow analyzer you are seeing that the instrument basically flows the samples continuously.

However, in case of discrete analyzer, they keep the sample separate throughout the testing procedure and dispensing precise amount when is required. Now, rotating individual cuvettes through the instrument instead of releasing the sample in a continuous stream, so basically when they basically rotate the individual cuvettes through the instrument instead of releasing the sample in a continuous stream that cuts down the reagent waste and can produce hundreds of results per hour.

So, this instrument is also very precise and it requires a specific amount of specific chemical and without going for the continuous flow, it basically keeps the sample separate. So, this is called discrete analyzer and as you can see here, this is a picture of a discrete analyzer.

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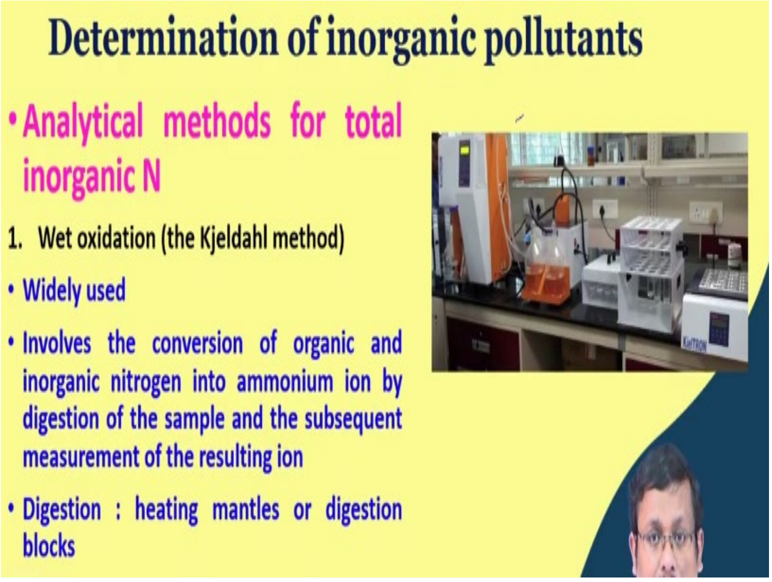

Determination of inorganic pollutants

- Analytical methods for total inorganic N

1. Wet oxidation (the Kjeldahl method)

- Widely used
- Involves the conversion of organic and inorganic nitrogen into ammonium ion by digestion of the sample and the subsequent measurement of the resulting ion
- Digestion : heating mantles or digestion blocks

Now, other method for measurement of inorganic nitrogen. Now we are talking about the total inorganic nitrogen. Now, in the total inorganic nitrogen generally we follow 2 methods, one is the wet oxidation method, another is Dumas method. Now, let us consider first, let us first discuss the wet oxidation methods or Kjeldahl method, it is also known as the Kjeldahl method.

So, this wet oxidation method or Kjeldahl method is a widely used technology and it involves the conversion of organic and inorganic nitrogen into ammonium ion by digestion of the samples and the subsequent measurement of the resulting ion. So, in all most of the soil chemistry laboratory, you will see that this instrument is present and this Kjeldahl apparatus is you know sometimes it is automated, sometimes it is manual.

So, this Kjeldahl analysis or Kjeldahl analyzer involves the conversion of organic and inorganic nitrogen into ammonium ion. So, they basically convert all the organic form of nitrogen which is present in the proteinaceous material and also inorganic nitrogen into ammonium ion by digestion of the samples and then subsequent measurement of the resulting ions.

And for digestion, it has also auxiliary heating mantles or digestion block as you can see in this picture, this is the distillation unit of this Kjeldahl apparatus and this is the digestion unit where block digesters, it is basically block digestion process. So, there the digestions of the samples are occurring in this block digestion chamber. So, this is how this wet oxidation by Kjeldahl method is performed.

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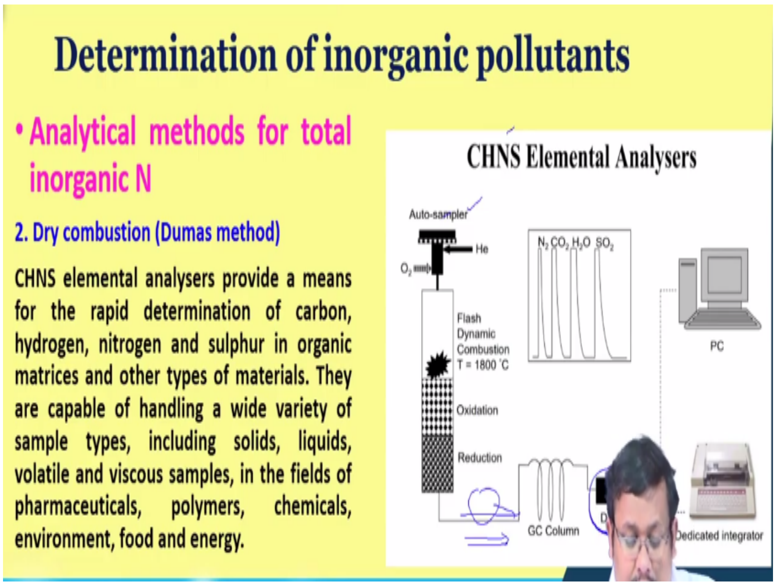

Determination of inorganic pollutants

- Analytical methods for total inorganic N

2. Dry combustion (Dumas method)

CHNS elemental analysers provide a means for the rapid determination of carbon, hydrogen, nitrogen and sulphur in organic matrices and other types of materials. They are capable of handling a wide variety of sample types, including solids, liquids, volatile and viscous samples, in the fields of pharmaceuticals, polymers, chemicals, environment, food and energy.

CHNS Elemental Analysers

Now, the second method is known as the dry combustion and this method is based on Dumas method. Now, the instrument which we generally use for this dry combustion is known as CHNS analyzer or Elemental analyzers. Now, the CHNS stands for carbon, hydrogen, nitrogen and sulphur elemental analyzers. Now, this is a very sophisticated and accurate instruments.

This CHNS analyzer provides a means for rapid determination of carbon, hydrogen, nitrogen and sulphur in organic metrices and other types of materials. So, basically it gives you the total concentration a total content of carbon, hydrogen, nitrogen and sulphur in organic metrices and other types of materials. Now, they are also capable of handling a wide variety of sample types including solids, liquids, volatiles and viscous samples in the field of pharmaceuticals, polymers, chemicals environment, food and energy.

So, this instrument has a widespread use in several industries, not only in agricultural environment discipline, but also in polymers, chemicals, pharmaceuticals, food and energy and so on and so forth. So, this instrument basically has several parts, as you can see here. It has autosampler, then it has sample inlet system, and there where they basically introduce helium and oxygen gas.

And there is a combustion chamber where there is a flat dynamic combustion occurs at temperature of 1800 degrees centigrade where they oxidize, and after they oxidize this nitrogen, carbon, hydrogen and sulphur they basically convert into carbon dioxide and then water and water vapor and also the sulfur dioxide. Then these mixture of gases are circulated through a GC column or gas chromatography column.

When this gas mixture moves through this gas chromatography column, they separate out and being detected by this detector at the end of the GC column to measure the concentration of the total carbon, total hydrogen, total nitrogen, total sulfur. So, this is how this instrument basically works. So, the next slide will give you more information about the CHNS analyzer. The CHNS analyzer is a very important instrument and it is very accurate.

So, if you are interested to measure the total carbon and total nitrogen in the soil, you need this instrument and this is a very sophisticated instrument too.

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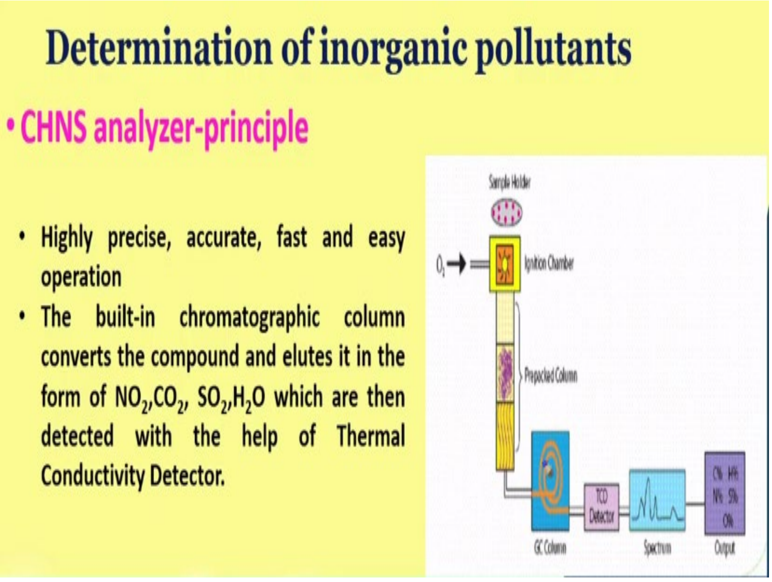

Determination of inorganic pollutants

- CHNS analyzer-principle
  - Highly precise, accurate, fast and easy operation
  - The built-in chromatographic column converts the compound and elutes it in the form of NO_2,CO_2, SO_2,H_2O which are then detected with the help of Thermal Conductivity Detector.

Now this instrument as I told you is highly precise, accurate, fast and shows the easy operation and this built-in chromatographic column converts the compound and elutes it in the form of nitrogen dioxide, carbon dioxide, sulfur dioxide and water vapor which are then detected with the help of thermal conductivity detector. So, this detector which is present is thermal conductivity detector.

So, in this diagram it is more clear. So, there are sample holder or autosampler and then after that there is an ignition chamber and in this ignition chamber the samples are being ignited with the help of oxygen which are being introduced and then they convert into the sulfur dioxide, carbon dioxide, nitrogen dioxide and water vapor and then they move through a prepacked column.

After they move this gas mixture through this prepacked column, they further move through this GC column or gas chromatography column and finally they are being detected by this TCD detector. So, the TCD detector is a short form of thermal conductivity detector and based on the individual compound this spectrum will be obtained.

And from analyzing this spectrum, they can calculate the total carbon percentage, total hydrogen percentage, total nitrogen percentage, and total sulfur percentage also and oxygen percentage also. So, this is how this CHNS analyzer basically operates.

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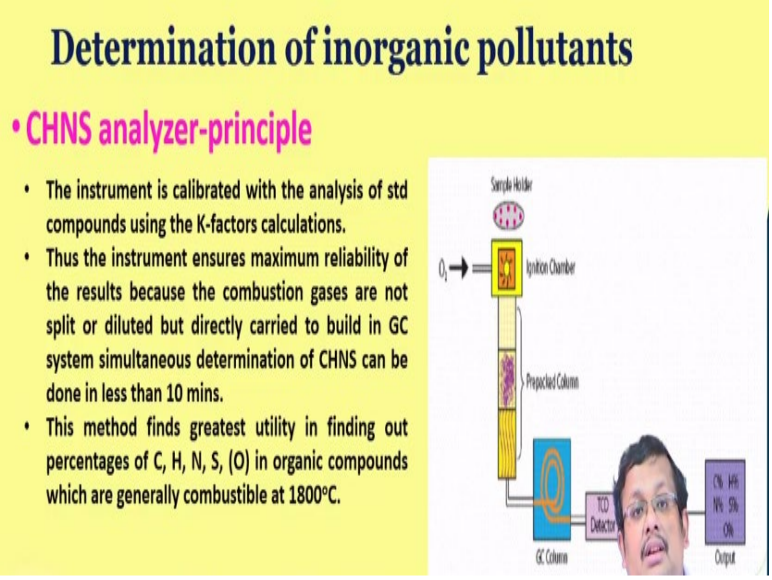

Determination of inorganic pollutants

- CHNS analyzer-principle
  - The instrument is calibrated with the analysis of std compounds using the K-factors calculations.
  - Thus the instrument ensures maximum reliability of the results because the combustion gases are not split or diluted but directly carried to build in GC system simultaneous determination of CHNS can be done in less than 10 mins.
  - This method finds greatest utility in finding out percentages of C, H, N, S, (O) in organic compounds which are generally combustible at 1800°C.

Now, this instrument basically is calibrated with the analysis of standard compounds using the K-factor calculations. Now, thus the instrument ensures maximum reliability of the results because the combustion gases are not split or diluted but directly carried to built-in GC system. So, they are not diverted anywhere, they directly carried to the GC system for simultaneous the detection of the CHNS in less than 10 minutes, so it is very fast also.

Now, this method finds greatest utility in finding out the percentage of carbon, hydrogen, nitrogen, sulfur and oxygen in organic compounds, which are generally combustible at 1800 degree centigrade.

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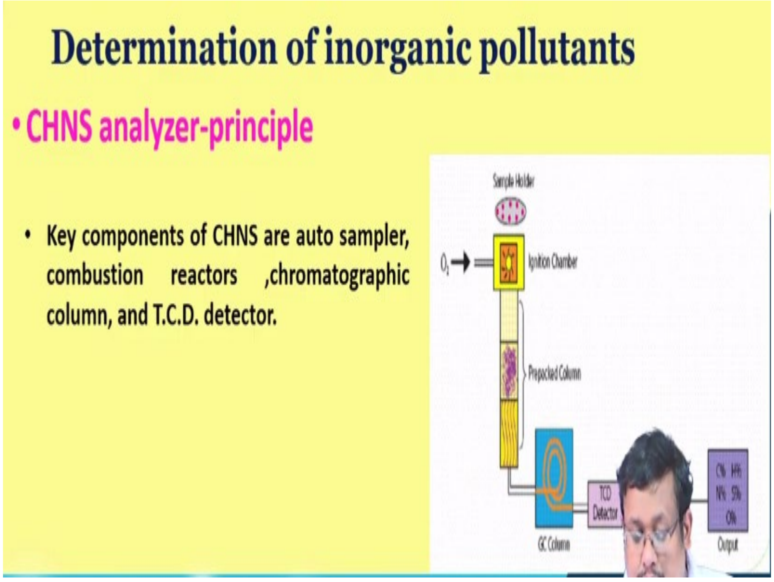

Determination of inorganic pollutants

- CHNS analyzer-principle
  - Key components of CHNS are auto sampler, combustion reactors ,chromatographic column, and T.C.D. detector.

So, guys, the key components of CHNS analyzers are autosampler, combustion reactors, chromatographic columns and TCD detector. So, we can see here first obviously the auto sampler, as you can see here so the autosampler is here and then combustion reactors and then chromatographic columns and then TCD detector. These are the 4 major parts of this CHNS analyzer.

So, guys, I hope that you have got a basic understanding of the principle of CHNS analyzer. So, we have started with the unspecific electrode for determination of inorganic nitrogen and then we talked about the continuous flow analyzer and also we talked about the discrete analyzer for measurement of inorganic nitrogen. And then for measurement of total nitrogen we talked about the Kjeldahl methods which is known for the wet digestion.

And also for dry combustion we have used Dumas method and this method is basically performed with this CHNS analyzer instrument. So, these all instruments are required when you are going to measure the concentration of different forms of nitrogen in different environmental samples, and thus these instruments are integral part for any sophisticated environmental soil chemistry lab, you will see them very often.

Among these all these 4 different instruments which we have talked about, the CHNS analyzer and also these continuous flow analyzer and discrete analyzer are somewhat costly. However, this ion selective electrode is comparatively cheaper and also this Kjeldahl apparatus is also comparatively cheaper than the CHNS analyzers. So, if there is a budgetary constraint, people generally go with the Kjeldahl method instead of CHNS analysis because CHNS analysis is costly.

Although it is accurate, although it is precise and requires less amount of time, however, it is costly and also the consumables specifically the gases are very costly in nature.

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Determination of inorganic pollutants

- CHNS analyzer

So, this is a picture of the CHNS analyzer.

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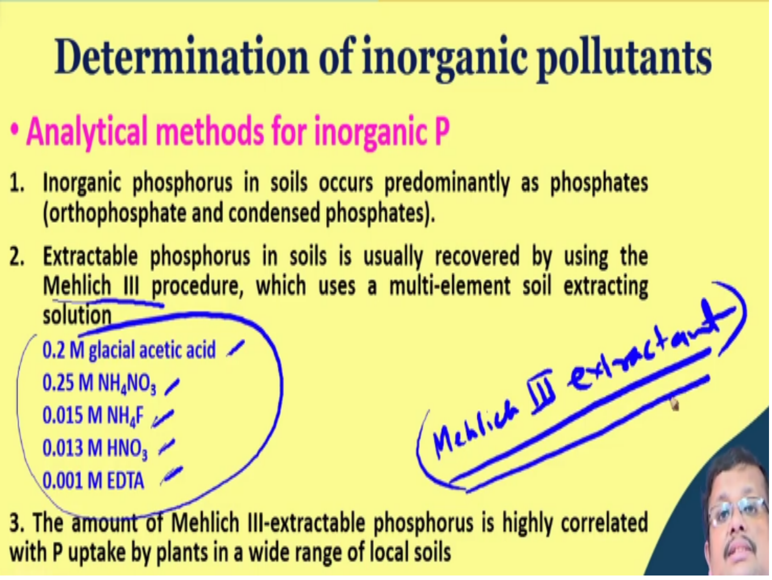

Determination of inorganic pollutants

- Analytical methods for inorganic P

1. Inorganic phosphorus in soils occurs predominantly as phosphates (orthophosphate and condensed phosphates).
2. Extractable phosphorus in soils is usually recovered by using the Mehlich III procedure, which uses a multi-element soil extracting solution
   - 0.2 M glacial acetic acid
   - 0.25 M NH_4NO_3
   - 0.015 M NH_4F
   - 0.013 M HNO_3
   - 0.001 M EDTA
3. The amount of Mehlich III-extractable phosphorus is highly correlated with P uptake by plants in a wide range of local soils

(Mehlich III extractant)

So let us move ahead and see the determination of inorganic phosphorus. So, we have covered the nitrogen. Now let us see how we can measure the inorganic forms of phosphorus in the soil. Now, you know that inorganic phosphorus in soil occurs predominantly as phosphates and this phosphate basically encompasses orthophosphate and condensed phosphates.

And the extractable phosphate in soils is usually recovered by using a specific extractant we call it Mehlich III extractant and the procedure is known as the Mehlich III procedure. This Mehlich III extractant is basically a multielement extractant and it is basically used for extracting the soil solution. Now, this Mehlich III soil extractant is basically composed of these components.

So, it has 0.2 molar glacial acetic acid, it has 0.25 molar of ammonium nitrate, it consists of 0.015 molar of ammonium fluoride, it also has 0.013 molar of nitric acid and 0.001 molar of EDTA or ethylenediaminetetraacetic acid, which is a chelating agent. So, these all comprise in the Mehlich III extractant. So, this Mehlich III extractant contains all these and remember the reason for containing all these individual components has their specific activity.

And that is why the Mehlich III extractant is known for extracting not only phosphorus but a wide range of other elements also from the soil and each of these components is known to extract more than one element. So, apart from nitrogen, these all other important trace elements as well as the primary elements can be extracted by this Mehlich III extractant. Now, the amount of Mehlich III extractable phosphorus.

So let us again come back to phosphorus, so the amount of Mehlich III extractable phosphorus is highly correlated with the phosphorous uptake by plants in a wide range of local soils. So, the Mehlich III extractable phosphates basically encompass the plant available phosphorus. So for soil fertility based measurements also, people generally go for this Mehlich III extraction. So, this is the Mehlich III extraction method.

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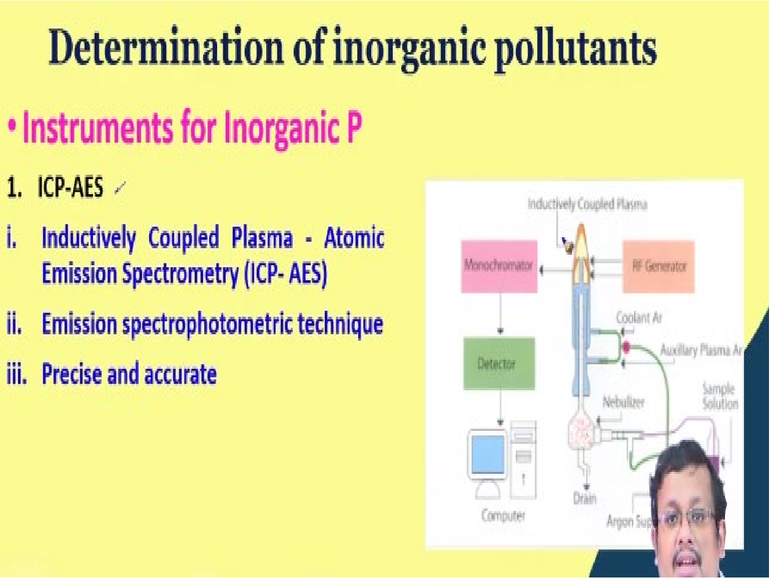

Determination of inorganic pollutants

- Instruments for Inorganic P

1. ICP-AES
   - i. Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP- AES)
   - ii. Emission spectrophotometric technique
   - iii. Precise and accurate

So, let us see; what are the important instruments for measurement of this inorganic phosphorus. So, the most important instrument for measurement of this inorganic phosphorus is ICP-AES. So, after you extract the solution through Mehlich III extractant, this extracted solution is injected into ICP-AES to measure individual ions. Now, this ICP-AES is a short form of Inductively Coupled Plasma - Atomic Emission Spectrometry or ICP-AES.

Now, this is basically an emission spectrophotometric technique and it is highly precise and accurate. So, this is basically the schematics of different components of the ICP-AES. So, basically here the argon gas is supplied for generation of the plasma and why plasma is required we will discuss and also there is a nebulizer to help injecting the samples into the plasma torch.

So, there is a plasma torch as you can see here. This plasma torch generates the inductively coupled plasma which is the source of energy and as a result of that the sample get atomized and further they are being detected, the wavelength which is generated from these excited atoms are basically analyzed by monochromatic, then detector and computer, further analyzed by a computer.

So, there is a sample solution as you can see, we are injecting the sample solution here and there is a nebulizer which helps in introducing this sample into the plasma torch and in this plasma torch to generate the plasma the argon is being supplied and as a result of that there is a plasma formation and this plasma is very high temperature and this high temperature of the plasma helps the atoms of the samples to excite and emit the characteristic wavelengths of light.

Which are being subsequently analyzed by monochromator and detector. So, this is the basic construction of an ICP-AES spectrometer. Let us wrap up here and in the next lecture, we will discuss what is the principle of this ICP-AES and how does this plasma torch basically works, why we call it inductively coupled plasma. So, in details, we will be discussing the working principle as well as the construction of this ICP- AES spectrometer in our next lecture.

So, guys, I hope that you have learned something new in this lecture. We have discussed some important instruments for nitrogen and we have started discussing about the ICP-AES. Please, stay tuned and let us meet in our next lecture to discuss more details about ICP-AES and why it is called a superior instrument, what are the benefits of using the ICP-AES. After we complete ICP-AES, we will also discuss some other sophisticated instruments.

We will be also discussing the spectrophotometry. Thank you very much. Let us meet in our third lecture of week 12.