Advance Analytical Course

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Lecture No. # 15

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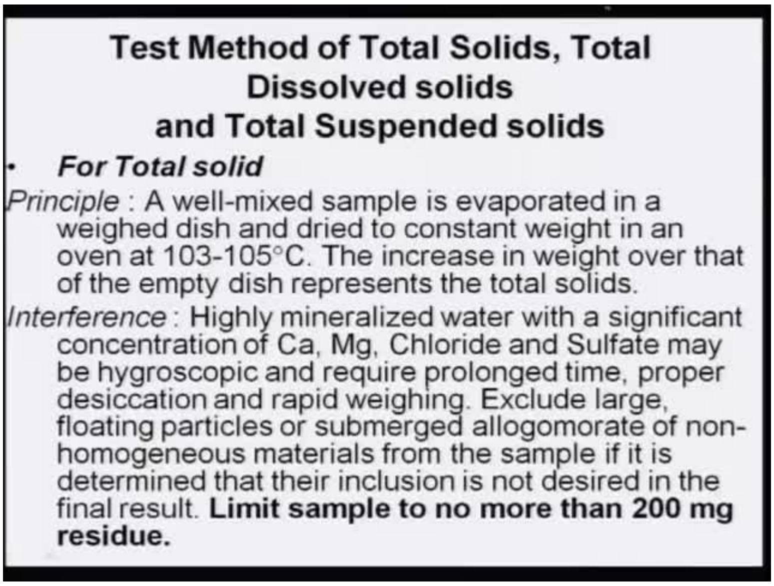

Test Method of Total Solids, Total Dissolved solids and Total Suspended solids

- ***For Total solid***

Principle : A well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103-105°C. The increase in weight over that of the empty dish represents the total solids.

Interference : Highly mineralized water with a significant concentration of Ca, Mg, Chloride and Sulfate may be hygroscopic and require prolonged time, proper desiccation and rapid weighing. Exclude large, floating particles or submerged allogomorate of non-homogeneous materials from the sample if it is determined that their inclusion is not desired in the final result. **Limit sample to no more than 200 mg residue.**

Today’s lecture is based on chemical analysis. It is a very important area, where the aspect of and importance of chemical analysis have been highlighted. Test methods that are used to find out total solids, total dissolved solids and total suspended solids. Now, one would like to understand why is there a necessity? Because when we talk about the hardness of water or the water not being pure or water unfit for drinking, it is mostly because it has some dissolved solids and some suspended solids, and what is the total content of these solids needs to be understood.

The principle - a well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103 to 105 degree centigrade. The increase in weight over that of the empty dish represents the total solid. So, it is simple procedure that any sample is just taken and evaporated on a weighed dish. Interferences - highly mineralized water with a significant concentration of calcium, magnesium, chloride and sulfate may be hygroscopic and require prolonged time, proper desiccation and rapid weighing.

Exclude large, floating particles or submerged agglomerates of non-homogeneous material from the sample, if it is determined that their inclusion is not desired in the final result. Limit sample to no more than 200 milligram residue. So, that means that any sample should not have more than 200 milligram of total solids; otherwise, that sample requires to be rejected.

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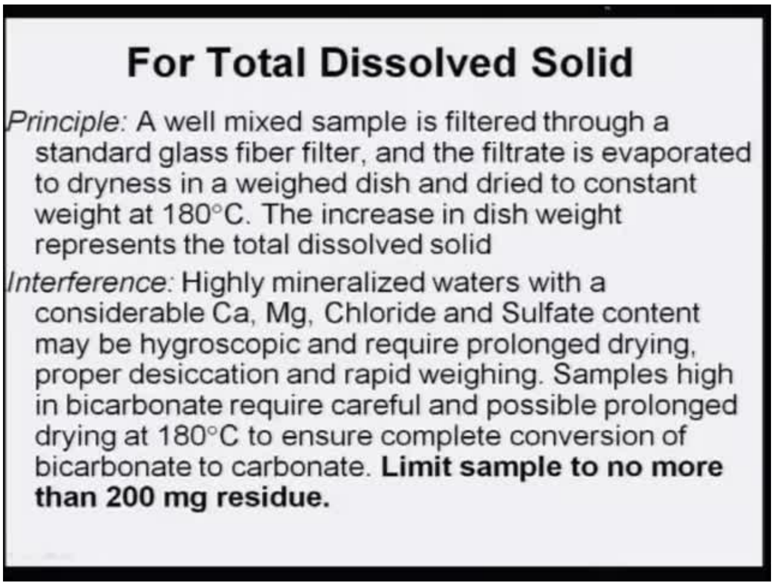

For Total Dissolved Solid

Principle: A well mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180°C. The increase in dish weight represents the total dissolved solid

Interference: Highly mineralized waters with a considerable Ca, Mg, Chloride and Sulfate content may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing. Samples high in bicarbonate require careful and possible prolonged drying at 180°C to ensure complete conversion of bicarbonate to carbonate. **Limit sample to no more than 200 mg residue.**

For total dissolved solids, the principle again is that a well-mixed sample is filtered through a standard glass fiber filter and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180 degrees. The increase in dish weight represents the total dissolved solids. Dissolved solids are the ones which we cannot see through our eyes, but they are still in the water and they need to be identified and that is why the temperature is kept at a higher temperature. For total solids, it was only 103 to 105 degrees, but for the total dissolved solids, the temperature is 180 centigrade.

The interferences that are generally to be avoided are highly mineralized water with a considerable amount of calcium, magnesium, chloride and sulfate content may be hygroscopic and require prolonged dryness and proper desiccation and rapid weighing, and here also, the residue must not be more than 200 milligram; otherwise, the sample needs to be rejected.

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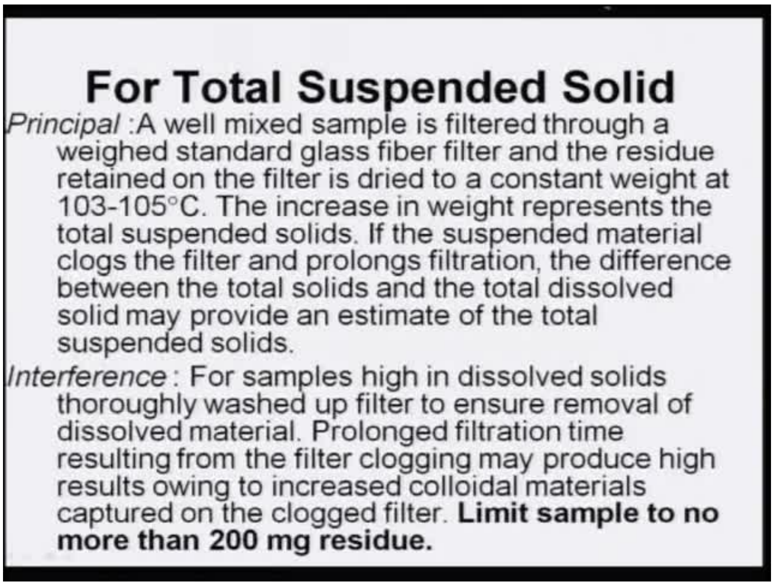

For Total Suspended Solid

Principal :A well mixed sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 103-105°C. The increase in weight represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and the total dissolved solid may provide an estimate of the total suspended solids.

Interference : For samples high in dissolved solids thoroughly washed up filter to ensure removal of dissolved material. Prolonged filtration time resulting from the filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter. **Limit sample to no more than 200 mg residue.**

For total suspended solid, the protocol for testing is that a well-mixed sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 103 to 105 degree centigrade. The increase in weight represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solid and the total dissolved solid may provide an estimate of the total suspended solids.

Here the interferences are, for sample high in dissolved solids, thoroughly washed up filter to ensure removal of dissolved material. Prolong filtration time resulting from the filter clogging may produce high results owing to increased colloidal material captured on the clogged filter. Here also, the limit should be that it should not exceed 200 milligram in the residue. Now, these are different parameters in water which needs to be tested for its purity content.

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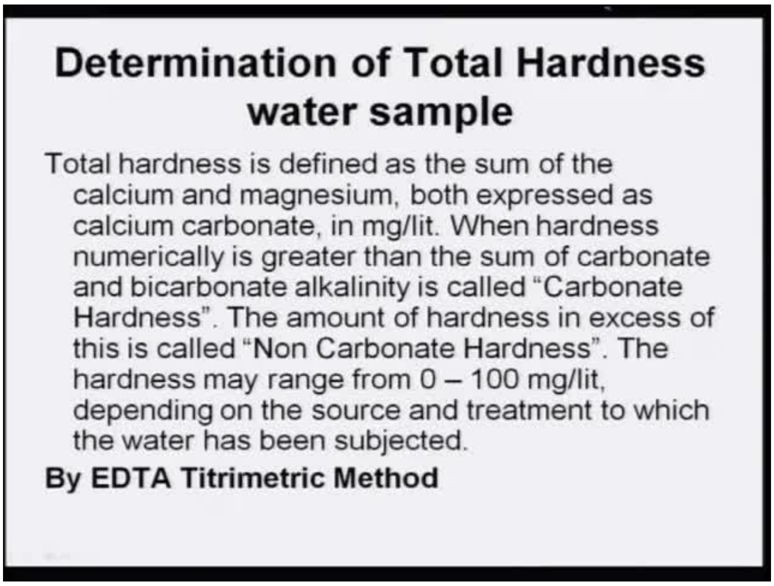

Determination of Total Hardness water sample

Total hardness is defined as the sum of the calcium and magnesium, both expressed as calcium carbonate, in mg/lit. When hardness numerically is greater than the sum of carbonate and bicarbonate alkalinity is called “Carbonate Hardness”. The amount of hardness in excess of this is called “Non Carbonate Hardness”. The hardness may range from 0 – 100 mg/lit, depending on the source and treatment to which the water has been subjected.

By EDTA Titrimetric Method

Then the next parameter for the purity content of water is the determination of total hardness in water sample. Total hardness, you must have often heard that this water is soft and this water is hard, and if the water is hard, it cannot do a proper washing. What does this term hardness or softness of water actually mean? Total hardness is defined as the sum of the calcium and magnesium, both expressed as calcium carbonate in milligram per litre. When hardness numerically is greater than the sum of carbonates and bicarbonates alkalinity, it is called carbonate hardness.

The amount of hardness in excess is called non-carbonate hardness. The hardness may range from 0 to 100 milligram per litre depending on the source and treatment to which the water has to be subjected. It can also be determined by EDTA titrimetric method.

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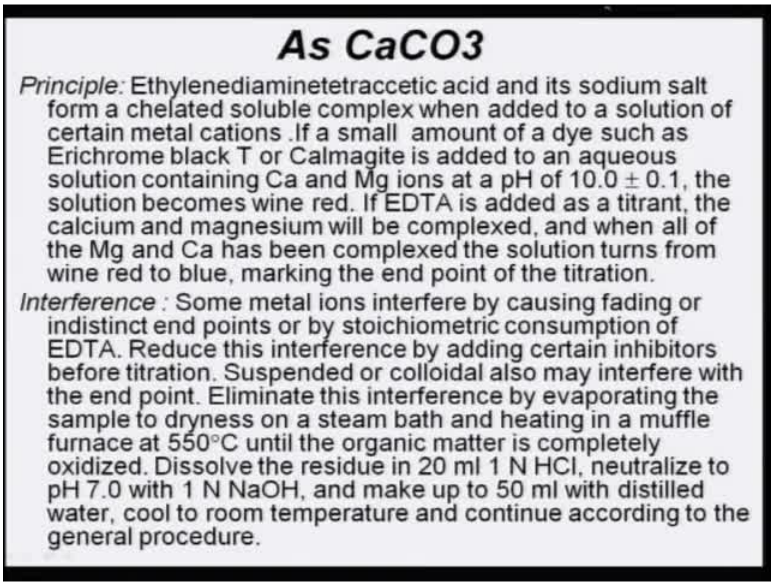

As CaCO3

Principle: Ethylenediaminetetraccetic acid and its sodium salt form a chelated soluble complex when added to a solution of certain metal cations .If a small amount of a dye such as Erichrome black T or Calmagite is added to an aqueous solution containing Ca and Mg ions at a pH of 10.0 ± 0.1, the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the Mg and Ca has been complexed the solution turns from wine red to blue, marking the end point of the titration.

Interference : Some metal ions interfere by causing fading or indistinct end points or by stoichiometric consumption of EDTA. Reduce this interference by adding certain inhibitors before titration. Suspended or colloidal also may interfere with the end point. Eliminate this interference by evaporating the sample to dryness on a steam bath and heating in a muffle furnace at 550°C until the organic matter is completely oxidized. Dissolve the residue in 20 ml 1 N HCl, neutralize to pH 7.0 with 1 N NaOH, and make up to 50 ml with distilled water, cool to room temperature and continue according to the general procedure.

As calcium carbonate - if it has to be estimated as calcium carbonate, the methodology uses EDTA or ethylenediaminetetraccetic acid and its sodium salt form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Erichrome black T or Calmagite is added to an aqueous solution containing calcium and magnesium ions at a pH of 10 plus minus 0.1, the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed and will all and when all of the magnesium and calcium has been complexed, the solution turns from wine red to blue marking the end point of the titration.

The interferences some that needs to be checked in this case are some metal ions interfere by causing fading or indistinct end points or by stoichiometric consumption of EDTA. Reduce these interferences by adding certain inhibitors before titration. Suspended or colloidal also may interfere with the end point. Eliminate this interference by evaporating the sample to dryness on a steam bath and heating in a muffle furnace at 550 degree centigrade until the organic matter is completely oxidized.

Dissolve the residue in 20 ml of 0.1 HCl, neutralize to pH 7 with 1 normal NaOH and make up to 50 ml with distilled water. Cool to room temperature and continue according to the general procedure. So, that is how the EDTA is used as an in the estimation of hardness, particularly in terms of calcium carbonate.

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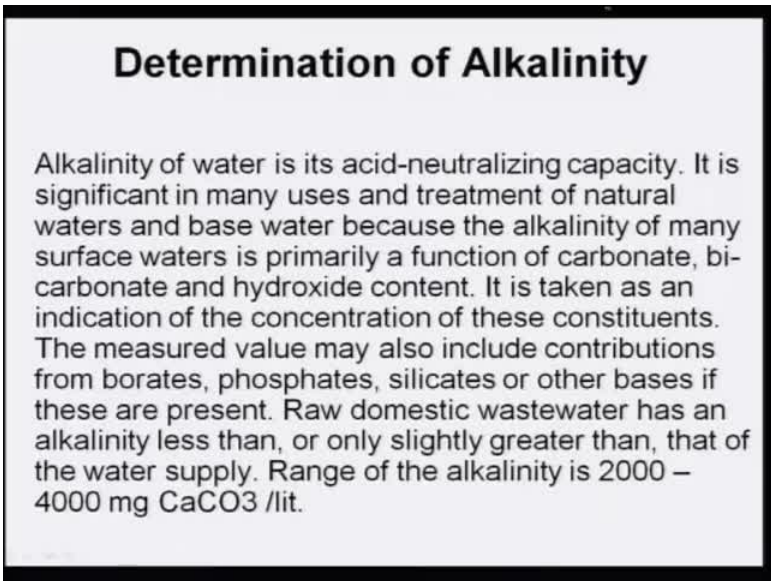

Determination of Alkalinity

Alkalinity of water is its acid-neutralizing capacity. It is significant in many uses and treatment of natural waters and base water because the alkalinity of many surface waters is primarily a function of carbonate, bi-carbonate and hydroxide content. It is taken as an indication of the concentration of these constituents. The measured value may also include contributions from borates, phosphates, silicates or other bases if these are present. Raw domestic wastewater has an alkalinity less than, or only slightly greater than, that of the water supply. Range of the alkalinity is 2000 – 4000 mg $CaCO_3$ /lit.

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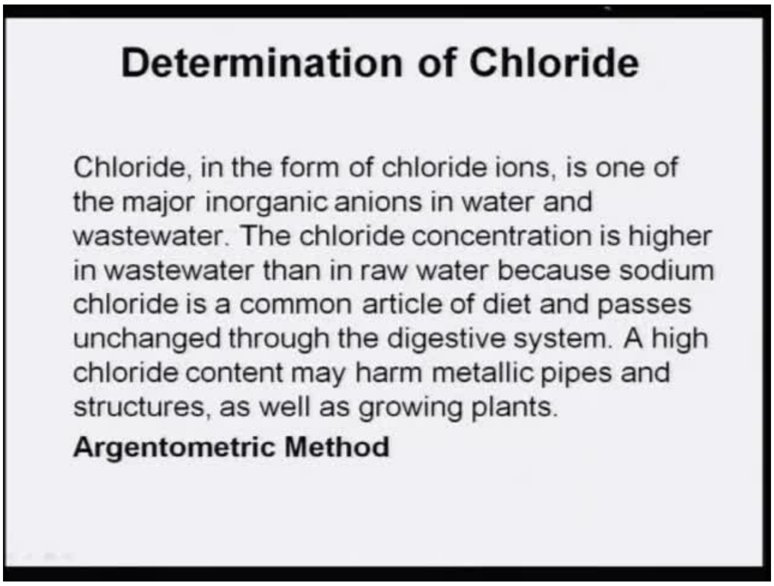

Determination of Chloride

Chloride, in the form of chloride ions, is one of the major inorganic anions in water and wastewater. The chloride concentration is higher in wastewater than in raw water because sodium chloride is a common article of diet and passes unchanged through the digestive system. A high chloride content may harm metallic pipes and structures, as well as growing plants.

Argentometric Method

Determination of chloride - we often say that there is chloride present in water. Now, what does that mean? Chloride in the form of chloride ion is one of the major inorganic anions in water and waste water. The chloride concentration is higher in waste water than in raw water because sodium chloride is a common article of diet and passes unchanged through the digestive system. A high chloride content may harm metallic pipes and structures, as well as growing plants, and the method for determination is the Argentometric method.

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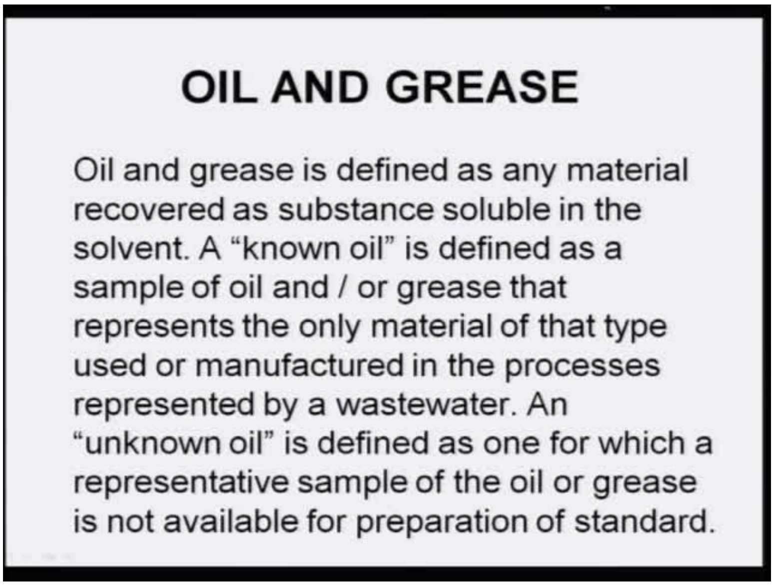

OIL AND GREASE

Oil and grease is defined as any material recovered as substance soluble in the solvent. A “known oil” is defined as a sample of oil and / or grease that represents the only material of that type used or manufactured in the processes represented by a wastewater. An “unknown oil” is defined as one for which a representative sample of the oil or grease is not available for preparation of standard.

Oil and grease - how they are determined in oil in water sample because some time water does have some oil particles floating on the top. Oil and grease is defined as any material recovered as substance soluble substances in the solvent. A “known oil” is defined as a sample of oil or grease that represents the only material of that type used or manufactured in the processes represented by waste water. An “unknown oil” is defined as one for which a representative sample of the oil or grease is not available for preparation of standard.

So, sometimes it could be a known oil; that means, there is an oil spill where you know that this type of oil was floating on the water and sometimes it is unknown, but there is a protocol for testing of these oils and grease in water.

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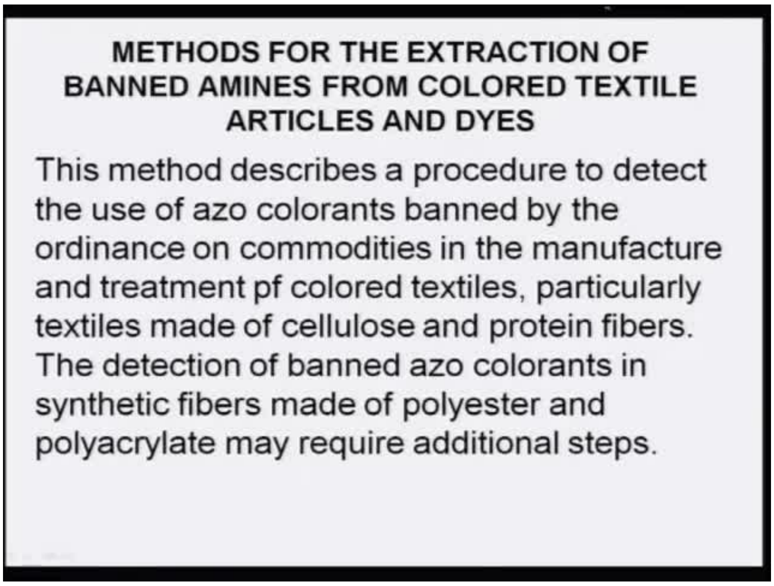

METHODS FOR THE EXTRACTION OF BANNED AMINES FROM COLORED TEXTILE ARTICLES AND DYES

This method describes a procedure to detect the use of azo colorants banned by the ordinance on commodities in the manufacture and treatment pf colored textiles, particularly textiles made of cellulose and protein fibers. The detection of banned azo colorants in synthetic fibers made of polyester and polyacrylate may require additional steps.

Method for extraction of banned amines from coloured textile articles and dyes - this method describes a procedure to detect the use of azo colourants banned by the ordinance on commodities in the manufacture and treatment of coloured textiles, particularly textiles made of cellulose and protein fibers.

The detection of banned azo colorants in synthetic fibers made of polyester and polyacrylate may require still additional steps. So, there are testing protocols. What I am trying to draw your attention is that these protocols have to be strictly followed. The chemical analysis cannot be done at one’s whim and fancy. One has to follow the procedure and for every banned chemical, there is a testing protocol prescribed by the government or the ordinance.

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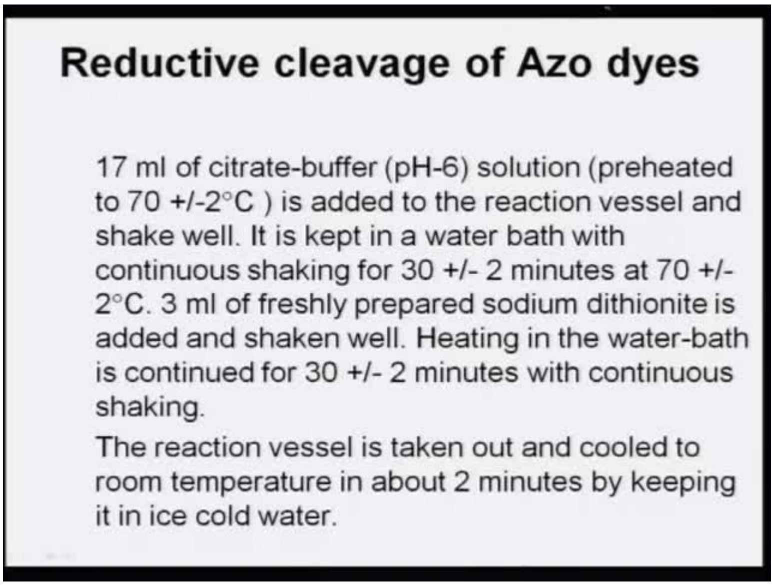

Reductive cleavage of Azo dyes

17 ml of citrate-buffer (pH-6) solution (preheated to 70 +/-2°C) is added to the reaction vessel and shake well. It is kept in a water bath with continuous shaking for 30 +/- 2 minutes at 70 +/-2°C. 3 ml of freshly prepared sodium dithionite is added and shaken well. Heating in the water-bath is continued for 30 +/- 2 minutes with continuous shaking.

The reaction vessel is taken out and cooled to room temperature in about 2 minutes by keeping it in ice cold water.

Reductive cleavage of Azo dyes - 17 ml of citrate-buffer which is at pH-6 solution preheated to 70 degrees approximately is added to the reaction vessel and shaken well. It is kept in a water bath with continuous shaking for 30 minutes or so, at 70 degrees. 3 ml of freshly prepared sodium dithionite is added and shaken well. Heating in the water bath is continued for 30 minutes approximately with continuous shaking. The reaction vessel is then taken out and cooled to room temperature in about 2 minutes by keeping it in ice cold water and thus, the reduction of the azo dye to amines takes place.

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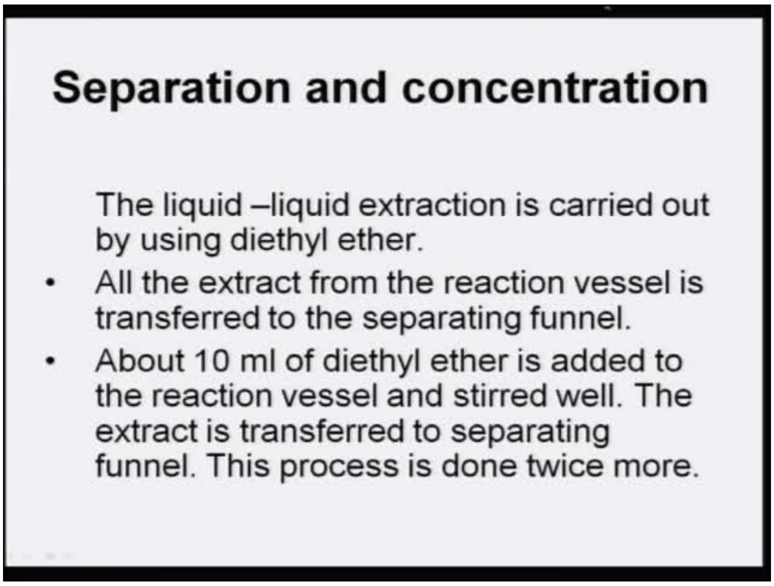

Separation and concentration

The liquid –liquid extraction is carried out by using diethyl ether.

- All the extract from the reaction vessel is transferred to the separating funnel.
- About 10 ml of diethyl ether is added to the reaction vessel and stirred well. The extract is transferred to separating funnel. This process is done twice more.

Separation and concentration - the liquid-liquid extraction is carried out by using diethyl ether as a solvent. All the extract from the reaction vessel is transferred to the separating funnel. About 10 ml of diethyl ether is added to the reaction vessel and stirred well. The extract is transferred to separating funnel. This process is done twice more. Now, I will explain you one thing that extraction is not done just one time. One can take 10 ml or into three times or one can take 30 ml in one time.

Now, you will think that why should we do three times the same thing, when we can do with the larger volume in one time, but the partitioning co-efficient does not separate itself completely, if we do it one time. So, that is why three times with lesser volume of solvent is recommended, but not one time with larger volume of the solvent.

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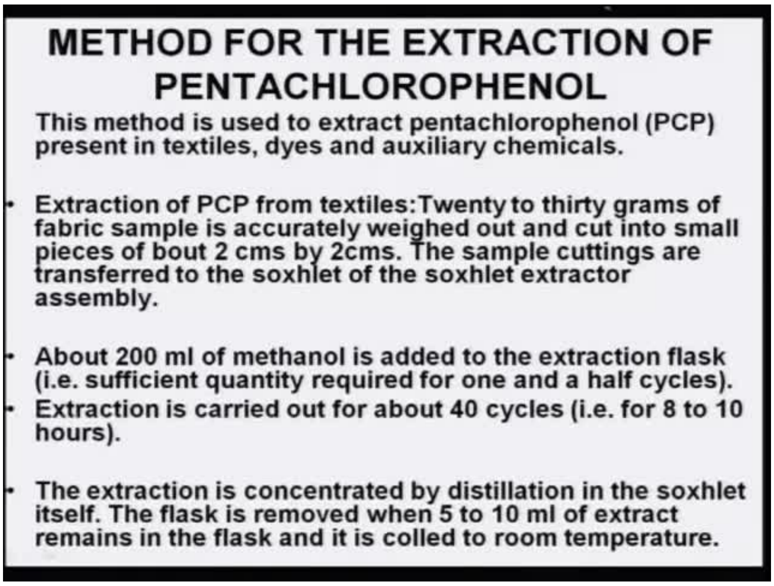

METHOD FOR THE EXTRACTION OF PENTACHLOROPHENOL

This method is used to extract pentachlorophenol (PCP) present in textiles, dyes and auxiliary chemicals.

- Extraction of PCP from textiles:Twenty to thirty grams of fabric sample is accurately weighed out and cut into small pieces of bout 2 cms by 2cms. The sample cuttings are transferred to the soxhlet of the soxhlet extractor assembly.

- About 200 ml of methanol is added to the extraction flask (i.e. sufficient quantity required for one and a half cycles).
- Extraction is carried out for about 40 cycles (i.e. for 8 to 10 hours).

- The extraction is concentrated by distillation in the soxhlet itself. The flask is removed when 5 to 10 ml of extract remains in the flask and it is colled to room temperature.

Method for the extraction of pentachlorophenol - this method is used for the extract of pentachlorophenol or PCP present in textiles, dyes and auxiliary chemicals. Extraction of PCP from textiles - 20 to 30 grams of fabric sample is accurately weighed out and cut into small pieces of about 2 centimetres by 2 centimetres. The sample cuttings are transferred to the soxhlet of the soxhlet extractor assembly. About 200 ml of methanol is added to the extraction flask (sufficient quantity required for one and a half cycles). Extraction is carried out for 40 cycles and it almost takes 8 to 10 hours.

The extraction is concentrated by distillation in the soxhlet itself. The flask is removed when 5 to 10 ml of extract remains. The flask is then cooled to room temperature and analyzed on GC machine for the content of the pentachlorophenol. I would like to tell you at this point of time that pentachlorophenol is used as a preservative and any amount which is remaining as an excess as a residue is injurious.

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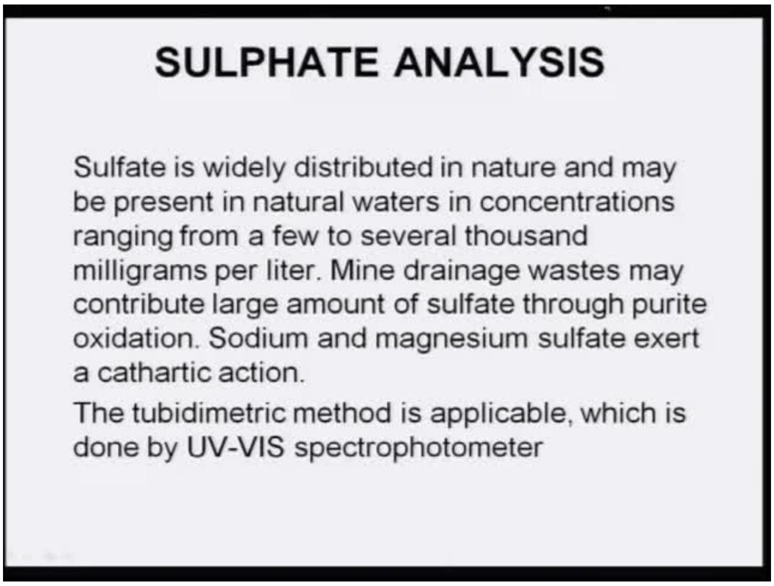

SULPHATE ANALYSIS

Sulfate is widely distributed in nature and may be present in natural waters in concentrations ranging from a few to several thousand milligrams per liter. Mine drainage wastes may contribute large amount of sulfate through purite oxidation. Sodium and magnesium sulfate exert a cathartic action.

The tubidimetric method is applicable, which is done by UV-VIS spectrophotometer

Sulphate analysis - sulphate is widely distributed in nature and may be present in natural water in concentrations ranging from a few to several thousand milligrams per litre. Mine drainage waste may contribute large amounts of sulphate through the purite oxidation. Sodium and magnesium sulphate exert a cathartic action. The tubidimetric method is applicable, which is done by UV-VIS spectrophotometer. The tubidimetric method is applicable, which is done by UV-VIS spectrophotometer.

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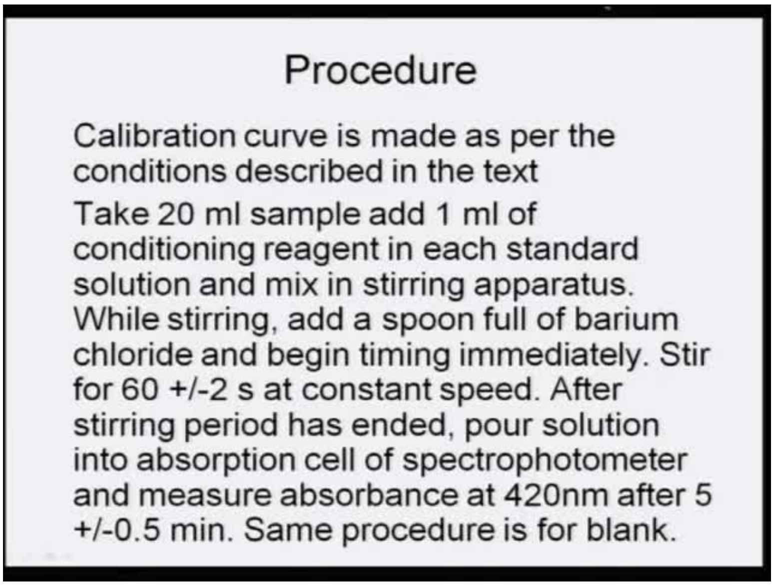

Procedure

Calibration curve is made as per the conditions described in the text

Take 20 ml sample add 1 ml of conditioning reagent in each standard solution and mix in stirring apparatus. While stirring, add a spoon full of barium chloride and begin timing immediately. Stir for 60 +/-2 s at constant speed. After stirring period has ended, pour solution into absorption cell of spectrophotometer and measure absorbance at 420nm after 5 +/-0.5 min. Same procedure is for blank.

How does this procedure take place? Calibration curve is made as per the conditions described in the text. Take 20 ml of the sample, add 1 ml of conditioning reagent in each standard solution and mix in stirring apparatus. While stirring, add a spoon full of barium chloride and begin timing immediately. Stir for 60 seconds at constant speed. After stirring period has ended, pour the solution into absorption cell of the spectrophotometer and measure the absorbance at 420 nanometres after 5 minutes. Same procedure is followed for blank. If sulphate is present, then it would give absorption peak at 420 nanometre.

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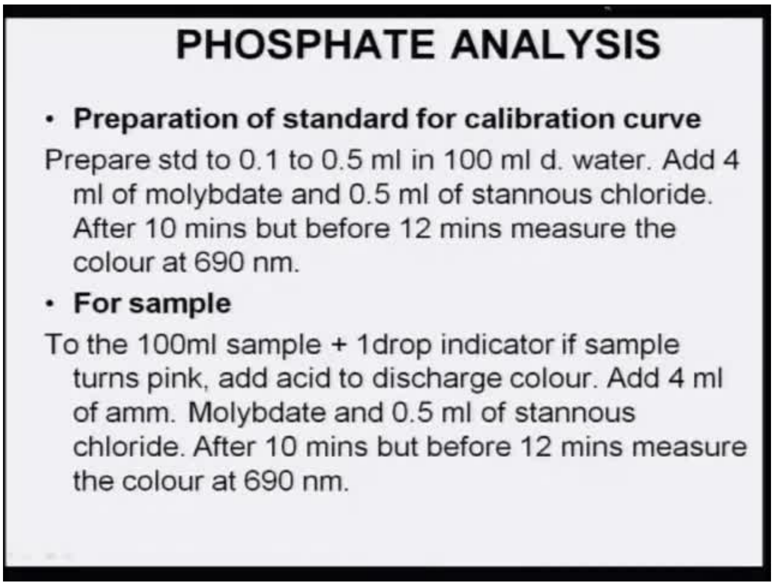

PHOSPHATE ANALYSIS

- **Preparation of standard for calibration curve**

Prepare std to 0.1 to 0.5 ml in 100 ml d. water. Add 4 ml of molybdate and 0.5 ml of stannous chloride. After 10 mins but before 12 mins measure the colour at 690 nm.

- **For sample**

To the 100ml sample + 1drop indicator if sample turns pink, add acid to discharge colour. Add 4 ml of amm. Molybdate and 0.5 ml of stannous chloride. After 10 mins but before 12 mins measure the colour at 690 nm.

Phosphate analysis – Again, whether the water is contaminated with phosphate or not can be found out by this analysis. Preparation of standard for calibration curve - preparation of standard of 0.1 to 0.5 in 100 ml of de-ionized water is prepared. An addition of 4 ml of molybdate and 0.5 ml of stannous chloride is carried out. After 10 minutes, but before 12 minutes, the solution is measured at 690 nanometres in UV visible spectrophotometer.

For sample - again 100 ml of the sample, one drop of indicator, if sample turns pink, add acid to dye, discharge the color. Add 4 ml of ammonium molybdate and 0.5 ml of stannous chloride, wait for 10 minutes and before it ends 12 minutes, the measurements must be taken on the UV visible spectrophotometer at 690 nanometres.

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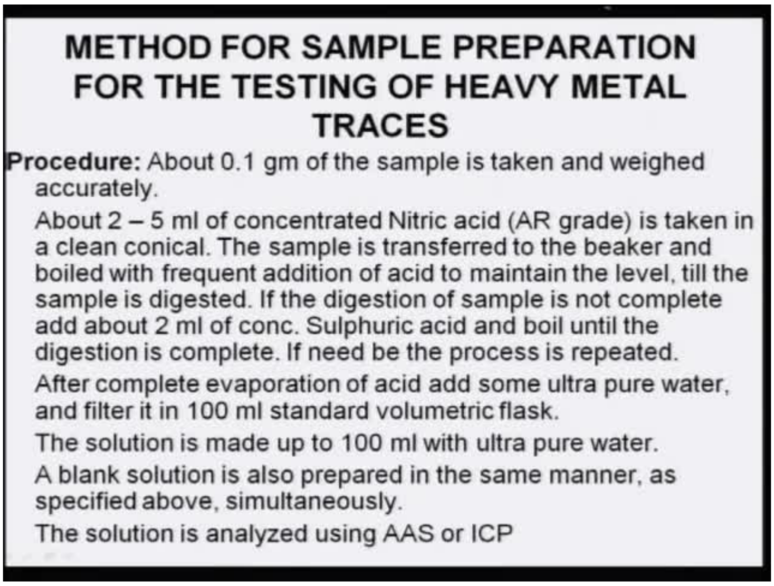

METHOD FOR SAMPLE PREPARATION FOR THE TESTING OF HEAVY METAL TRACES

Procedure: About 0.1 gm of the sample is taken and weighed accurately.

About 2 – 5 ml of concentrated Nitric acid (AR grade) is taken in a clean conical. The sample is transferred to the beaker and boiled with frequent addition of acid to maintain the level, till the sample is digested. If the digestion of sample is not complete add about 2 ml of conc. Sulphuric acid and boil until the digestion is complete. If need be the process is repeated.

After complete evaporation of acid add some ultra pure water, and filter it in 100 ml standard volumetric flask.

The solution is made up to 100 ml with ultra pure water.

A blank solution is also prepared in the same manner, as specified above, simultaneously.

The solution is analyzed using AAS or ICP

Methods for sample preparation for the testing of heavy metal traces - about 0.1 gram of the sample is taken and weighed accurately. About 2 to 5 ml of concentrated nitric acid is taken in a clean conical. The sample is transferred to the beaker and boiled with frequent addition of acid to maintain the level, till the sample is digested. If the digestion of sample is not complete, add about 2 ml concentrated sulphuric acid and boil until the digestion is complete.

If needed be, the process is repeated. After complete evaporation of the acid, add some ultra-pure water and filter it in 100 ml standard volumetric flask. The solution is made up to 100 ml with ultra-pure water. A blank solution is also prepared in the same manner as specified above and simultaneously, the solutions are analyzed on atomic absorption spectrophotometer or ICP - that is, inductively coupled plasma spectrometry.

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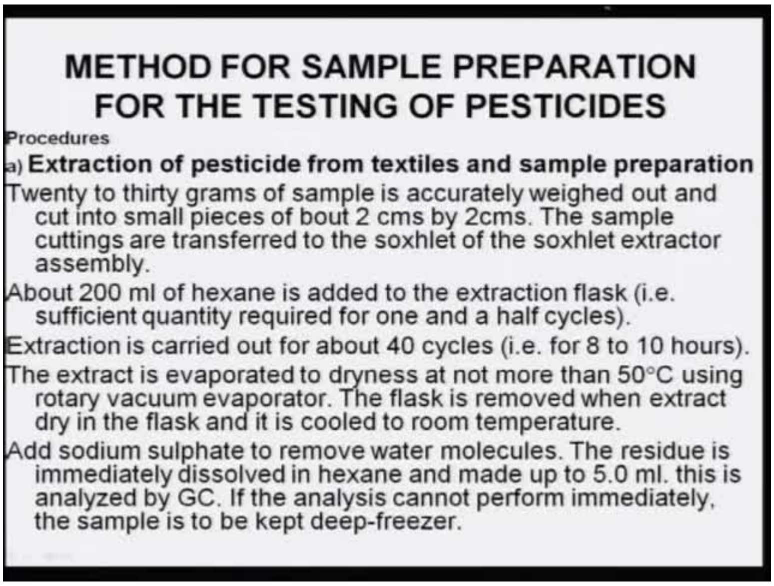

METHOD FOR SAMPLE PREPARATION FOR THE TESTING OF PESTICIDES

Procedures

a) **Extraction of pesticide from textiles and sample preparation**

Twenty to thirty grams of sample is accurately weighed out and cut into small pieces of bout 2 cms by 2cms. The sample cuttings are transferred to the soxhlet of the soxhlet extractor assembly.

About 200 ml of hexane is added to the extraction flask (i.e. sufficient quantity required for one and a half cycles).

Extraction is carried out for about 40 cycles (i.e. for 8 to 10 hours).

The extract is evaporated to dryness at not more than 50°C using rotary vacuum evaporator. The flask is removed when extract dry in the flask and it is cooled to room temperature.

Add sodium sulphate to remove water molecules. The residue is immediately dissolved in hexane and made up to 5.0 ml. this is analyzed by GC. If the analysis cannot perform immediately, the sample is to be kept deep-freezer.

Method for sample preparation for the testing of pesticides - we just had learnt that GC is the ideal machine for testing of pesticides. Extraction is a very important step when we are analyzing pesticides. Time and again I have emphasized that the extraction plays a very crucial role. Extraction of pesticides from textiles and sample preparation is very important and a little tricky. 20 to 30 grams of the sample is accurately weighed out and cut into small pieces of about 2 centimetres to 2 centimetres.

The sample cuttings are transferred to the soxhlet and put into the soxhlet extractor assembly. About 200 ml of hexane is added to the extraction flask and this is sufficient quantity required for one and a half cycles to run. Extraction is carried out for about 40 cycles which takes about 8 to 10 hours. The extract is evaporated to dryness at not more than 50 degrees centigrade using rotary vacuum evaporator. Why, because this is a hexane solvent; in earlier case, it was methanol solvent.

The flask is removed when extract is dried in the flask and it is cooled at room temperature. Sodium sulphate is added and is to remove water samples that may be present in the sample. During the process of extraction, some water may have come into it. The residue is immediately dissolved in hexane and made up to 5 ml. This is analyzed by GC. If the analysis cannot perform immediately, the sample must be kept in deep freezer because pesticides are liable to get decomposed at higher room temperature. So, they need to be preserved. Having done the extraction, it is important that if the analysis is not taking place at that particular time or for some reason, the analyst is busy, the sample must be preserved carefully.

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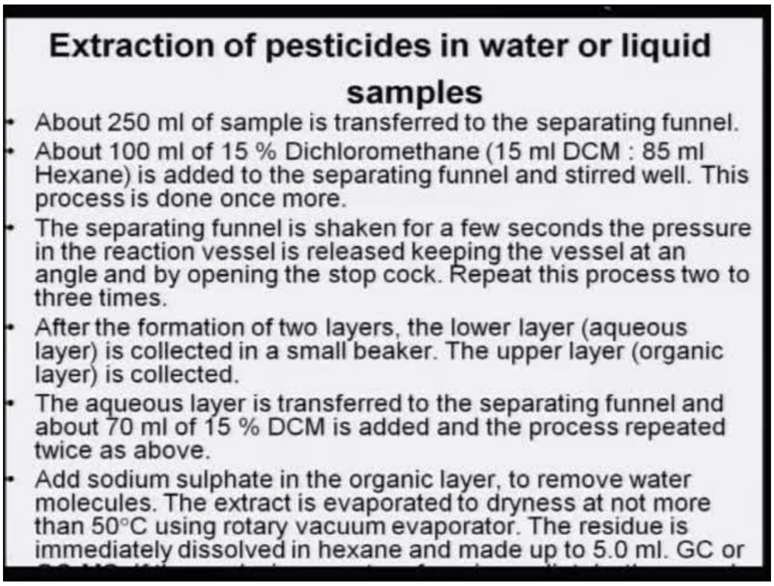

Extraction of pesticides in water or liquid samples

- About 250 ml of sample is transferred to the separating funnel.
- About 100 ml of 15 % Dichloromethane (15 ml DCM : 85 ml Hexane) is added to the separating funnel and stirred well. This process is done once more.
- The separating funnel is shaken for a few seconds the pressure in the reaction vessel is released keeping the vessel at an angle and by opening the stop cock. Repeat this process two to three times.
- After the formation of two layers, the lower layer (aqueous layer) is collected in a small beaker. The upper layer (organic layer) is collected.
- The aqueous layer is transferred to the separating funnel and about 70 ml of 15 % DCM is added and the process repeated twice as above.
- Add sodium sulphate in the organic layer, to remove water molecules. The extract is evaporated to dryness at not more than 50°C using rotary vacuum evaporator. The residue is immediately dissolved in hexane and made up to 5.0 ml. GC or

Extraction of pesticides in water or liquid sample - that was from the solid sample and this particular method is from the liquid sample. About 200 ml of the sample is transferred to the separating funnel. About 100 ml of 15 percent dichloromethane having 15 ml of DCM and 85 of hexane is added to the separating funnel and stirred well or rather shaken well. This process is done twice. This process is done once more. The separating funnel is shaken for a few seconds. The pressure in the reaction vessel is released keeping the vessel at an angle and by opening the stop cock. Repeat this process two to three times. After the formation of two layers, the lower layer, which is the aqueous layer, is collected in a small beaker.

The upper layer which is the organic layer is collected. The aqueous layer is transferred to the separating funnel and about 70 ml of 15 percent DCM is again added and the process is repeated two times again. To this, Sodium sulphate is added to the organic layer to remove any traces of water molecules. The extract is evaporated to dryness at not more than 50 degrees using rotary vacuum evaporator.

The residue is immediately dissolved in hexane and made up to 5 ml and then it is analyzed on GC or GCMS for its detection. So, you saw that for different band chemicals, there were different methodologies that were adapted and yet, we have not completed the whole story of analysis. There are different types of analyses, as I said which are equally important.

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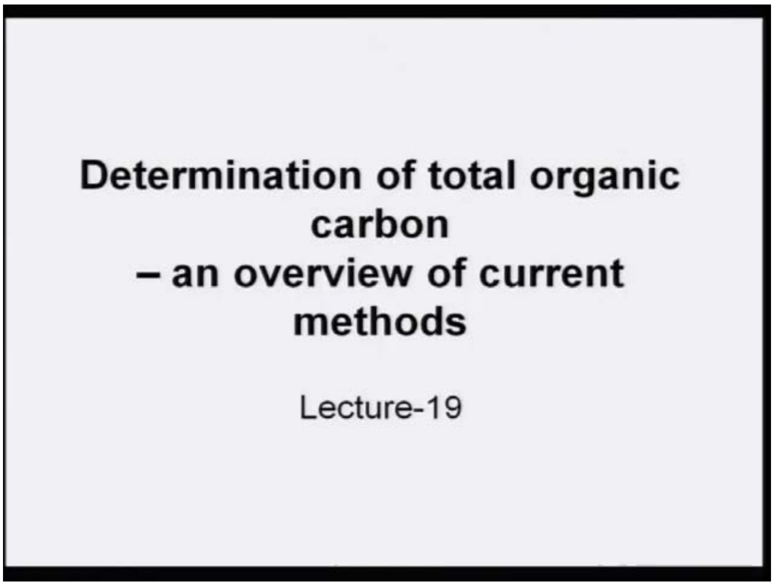

Determination of total organic carbon
– an overview of current methods

Lecture-19

Now, another aspect to find out determination of total organic carbon and how this method is viewed in the whole scenario is also very, very important for a chemical analyst. So, I thought I should dedicate a part of the lecture for explaining you, what is total organic carbon and how it can be determined.

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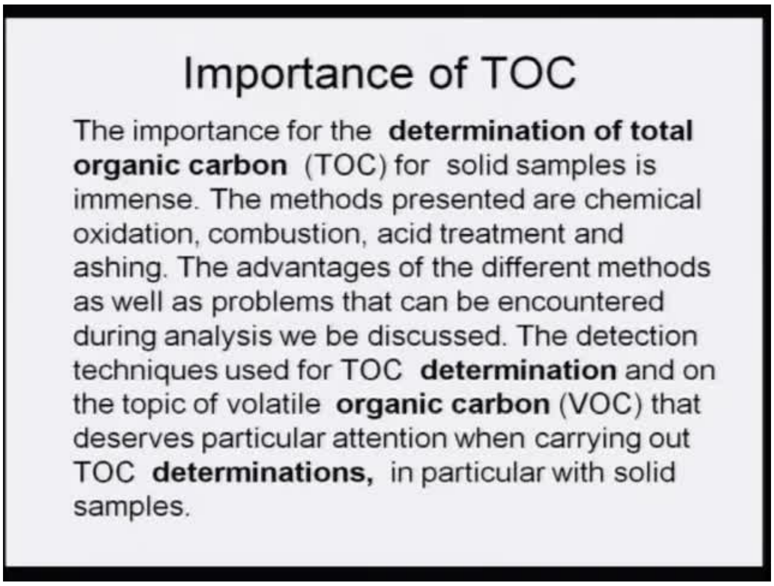

Importance of TOC

The importance for the **determination of total organic carbon** (TOC) for solid samples is immense. The methods presented are chemical oxidation, combustion, acid treatment and ashing. The advantages of the different methods as well as problems that can be encountered during analysis we be discussed. The detection techniques used for TOC **determination** and on the topic of volatile **organic carbon** (VOC) that deserves particular attention when carrying out TOC **determinations,** in particular with solid samples.

Importance of TOC - we will refer total organic carbon as TOC. The importance for determination of total organic carbon for solid samples is immense. The methods represent are chemical oxidation, combustion, acid treatment and ashing. The advantages of the different methods as well as problems that can be encountered during analysis will all be discussed here. The detection techniques used for TOC determination and on the topic of volatile organic carbon, that is, the VOC that deserves particular attention when carrying out the TOC determination, in particular with solid samples.

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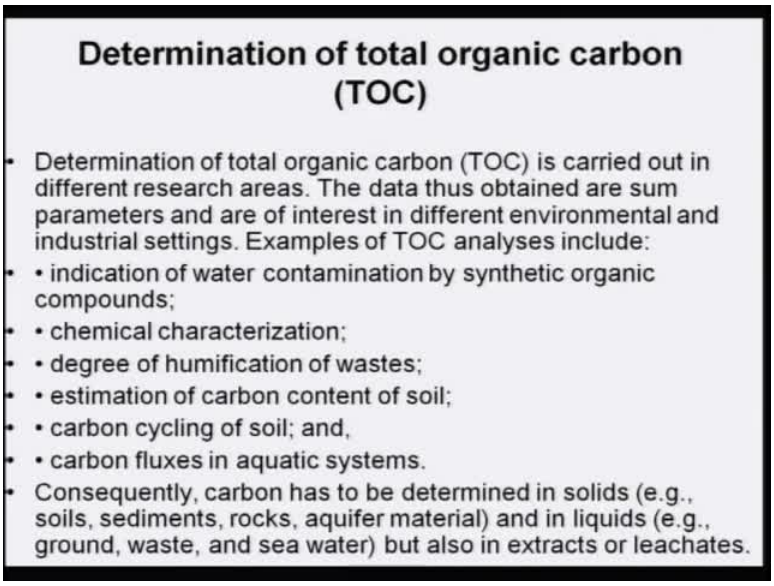

Determination of total organic carbon (TOC)

- Determination of total organic carbon (TOC) is carried out in different research areas. The data thus obtained are sum parameters and are of interest in different environmental and industrial settings. Examples of TOC analyses include:
- • indication of water contamination by synthetic organic compounds;
- • chemical characterization;
- • degree of humification of wastes;
- • estimation of carbon content of soil;
- • carbon cycling of soil; and,
- • carbon fluxes in aquatic systems.
- Consequently, carbon has to be determined in solids (e.g., soils, sediments, rocks, aquifer material) and in liquids (e.g., ground, waste, and sea water) but also in extracts or leachates.

Determination of total organic carbon, that is, TOC - determination of total organic carbon is carried out in different research areas. The data thus obtained are sum of parameters and are of interest in different environmental and industrial settings. Examples of TOC analysis include indication of water contamination by synthetic organic compounds - if we want to find out whether water has any organic contaminant or not, this is the best method to find out, chemical characterization, degree of humification of wastes, estimation of carbon content of soil, carbon cycling of soil and carbon fluxes in aquatic system. Consequently, carbon has to be determined in solids (soils, sediments, rocks, aquifer material) and in liquid (ground water, waste water, sea water), but also in extracts and leachates.

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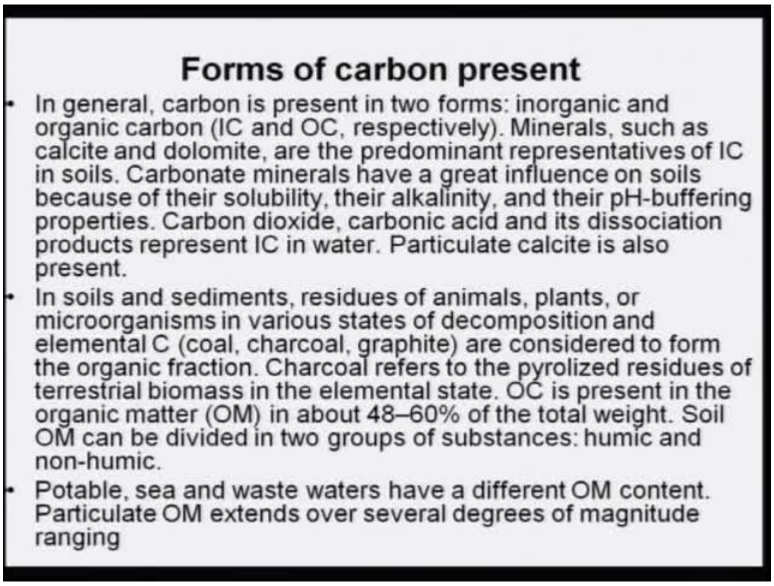

Forms of carbon present

- In general, carbon is present in two forms: inorganic and organic carbon (IC and OC, respectively). Minerals, such as calcite and dolomite, are the predominant representatives of IC in soils. Carbonate minerals have a great influence on soils because of their solubility, their alkalinity, and their pH-buffering properties. Carbon dioxide, carbonic acid and its dissociation products represent IC in water. Particulate calcite is also present.
- In soils and sediments, residues of animals, plants, or microorganisms in various states of decomposition and elemental C (coal, charcoal, graphite) are considered to form the organic fraction. Charcoal refers to the pyrolized residues of terrestrial biomass in the elemental state. OC is present in the organic matter (OM) in about 48–60% of the total weight. Soil OM can be divided in two groups of substances: humic and non-humic.
- Potable, sea and waste waters have a different OM content. Particulate OM extends over several degrees of magnitude ranging

Forms of carbon that are present - in general, carbon is present in two forms: inorganic and organic. So, we will refer it as IC and OC respectively. Minerals such as calcite, dolomite are the predominant representatives of IC in soils. Carbonate minerals have a great influence on soils because of their solubilities, their alkalinities and their pH buffering properties. Carbon dioxide, carbonic acid and its dissociation products represent OC in water. Particulate calcite is also present.

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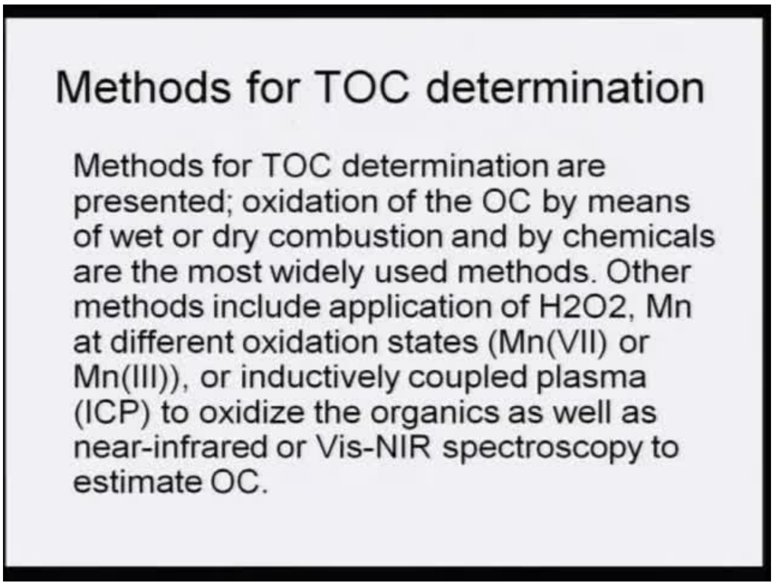

Methods for TOC determination

Methods for TOC determination are presented; oxidation of the OC by means of wet or dry combustion and by chemicals are the most widely used methods. Other methods include application of H2O2, Mn at different oxidation states (Mn(VII) or Mn(III)), or inductively coupled plasma (ICP) to oxidize the organics as well as near-infrared or Vis-NIR spectroscopy to estimate OC.

Methods of TOC determination - methods for TOC determination are presented. Oxidation of OC - that is, organic carbon, by means of wet or dry combustion, or by chemicals are the most widely used methods. Other methods include application of hydrogen peroxide, manganese at different oxidation state, that is, manganese seven or manganese three, or inductively coupled plasma, that is, ICP to oxidize the organics as well as near-infrared or visible infrared spectroscopy to estimate the organic content or the organic carbon.

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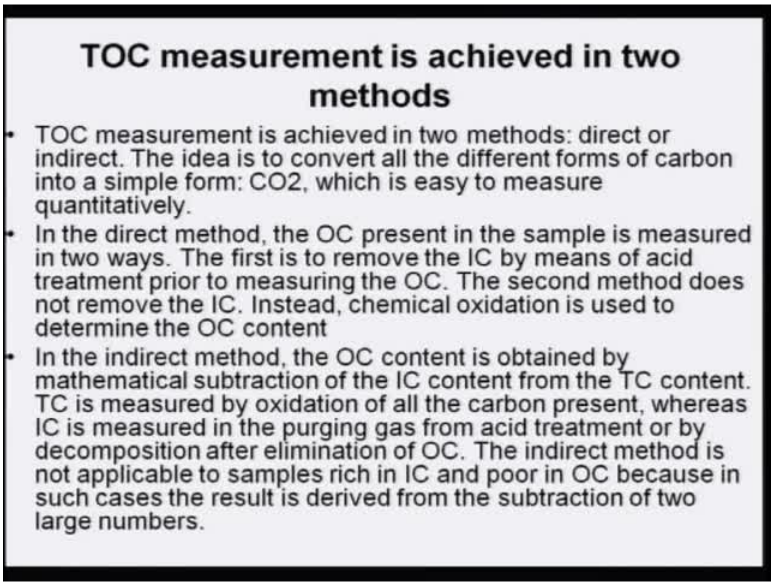

TOC measurement is achieved in two methods

- TOC measurement is achieved in two methods: direct or indirect. The idea is to convert all the different forms of carbon into a simple form: CO2, which is easy to measure quantitatively.
- In the direct method, the OC present in the sample is measured in two ways. The first is to remove the IC by means of acid treatment prior to measuring the OC. The second method does not remove the IC. Instead, chemical oxidation is used to determine the OC content
- In the indirect method, the OC content is obtained by mathematical subtraction of the IC content from the TC content. TC is measured by oxidation of all the carbon present, whereas IC is measured in the purging gas from acid treatment or by decomposition after elimination of OC. The indirect method is not applicable to samples rich in IC and poor in OC because in such cases the result is derived from the subtraction of two large numbers.

TOC measurement is achieved in two methods. TOC measurement is achieved in two methods, either by direct method or indirect method. The idea is to convert all the different forms of carbon into simple form, carbon dioxide, which is easy to measure quantitatively. In the direct method, the organic carbon present in the sample is measured in two ways. The first is to remove the inorganic carbon, that is, the IC by means of acid treatment prior to measuring the OC. The second method does not remove the IC; instead, chemical oxidation is used to determine the OC directly.

In the indirect method, the OC content is obtained by mathematical subtraction of the IC content from the TC content. TC is measured by oxidation of all the carbon present, whereas IC is measured in the purging gas from acid treatment or by decomposition after elimination of organic carbon. The indirect method is not applicable to samples rich in IC and poor in OC because in such cases, the result is derived from the subtraction of the two large numbers.

So, obviously, if the inorganic carbon content is very high, the latter method will not be applicable. It is only applicable when they are in equal quantities or if the organic content is much higher. Then from the total carbon content, the organic carbon is derived by subtracting the inorganic carbon.

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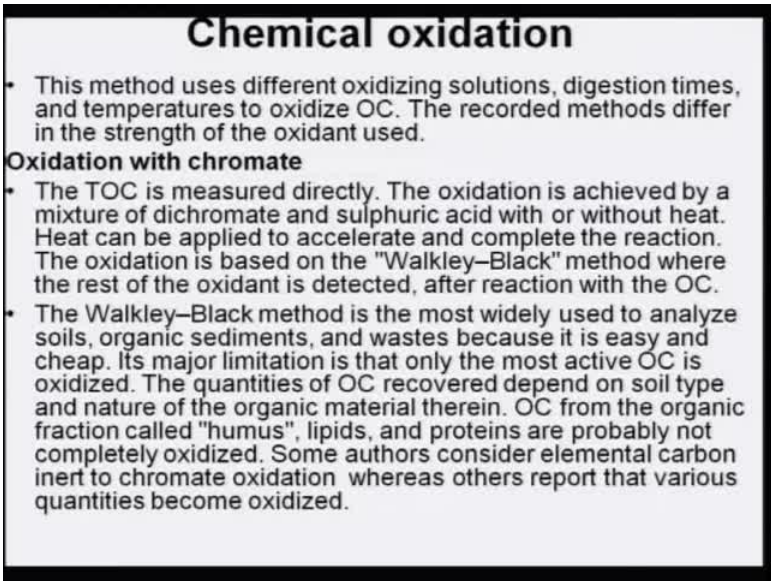

Chemical oxidation

- This method uses different oxidizing solutions, digestion times, and temperatures to oxidize OC. The recorded methods differ in the strength of the oxidant used.

Oxidation with chromate

- The TOC is measured directly. The oxidation is achieved by a mixture of dichromate and sulphuric acid with or without heat. Heat can be applied to accelerate and complete the reaction. The oxidation is based on the "Walkley–Black" method where the rest of the oxidant is detected, after reaction with the OC.
- The Walkley–Black method is the most widely used to analyze soils, organic sediments, and wastes because it is easy and cheap. Its major limitation is that only the most active OC is oxidized. The quantities of OC recovered depend on soil type and nature of the organic material therein. OC from the organic fraction called "humus", lipids, and proteins are probably not completely oxidized. Some authors consider elemental carbon inert to chromate oxidation whereas others report that various quantities become oxidized.

Chemical oxidation - this method uses different oxidizing solution, digestion times and temperature to oxidize the organic carbon. The recorded methods differ in the strength of oxidant used.

Oxidation with chromate - the TOC is measured directly. The oxidation is achieved by a mixture of dichromate and sulphuric acid with or without heat. Heat can be applied to accelerate and complete the reaction. The oxidation is based on the Walkley-Black method, where the rest of the oxidant is detected after reaction with the organic carbon. The Walkley-Black method is most widely used to analyse soils, organic sediments and wastes because it is easy and cheap method.

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So, there can be a little debate whether the chromate method is the best method or not, but however, it gives it does give good results. So, with this we have come to an end of the analysis of total organic carbon and how the inorganic carbon and the organic carbon can be estimated from these results. Then we switch on to the next topic which is of equal importance. We have been talking in the past about heavy metal toxicity. How injurious it is to our body, to us is what we should need to understand first in order to be able to analyse.

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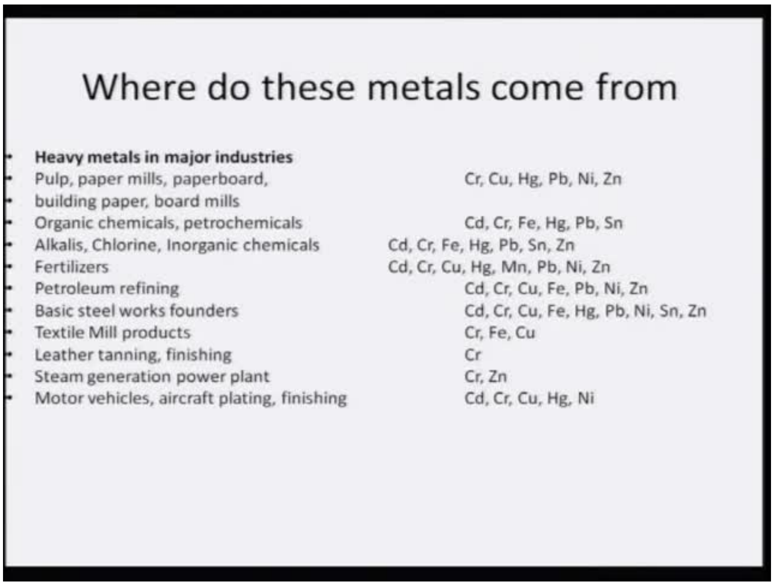

Where do these metals come from

- **Heavy metals in major industries**

Pulp, paper mills, paperboard,	Cr, Cu, Hg, Pb, Ni, Zn
building paper, board mills	
Organic chemicals, petrochemicals	Cd, Cr, Fe, Hg, Pb, Sn
Alkalis, Chlorine, Inorganic chemicals	Cd, Cr, Fe, Hg, Pb, Sn, Zn
Fertilizers	Cd, Cr, Cu, Hg, Mn, Pb, Ni, Zn
Petroleum refining	Cd, Cr, Cu, Fe, Pb, Ni, Zn
Basic steel works founders	Cd, Cr, Cu, Fe, Hg, Pb, Ni, Sn, Zn
Textile Mill products	Cr, Fe, Cu
Leather tanning, finishing	Cr
Steam generation power plant	Cr, Zn
Motor vehicles, aircraft plating, finishing	Cd, Cr, Cu, Hg, Ni

Where do these metals come from? Heavy metals comes from major industries like paper pulp, mills, paper boards and metals like chromium, copper, mercury, lead, nickel, zinc come from there; then building paper, paper boards also add on to metals; organic chemicals, petrochemicals add on to cadmium, chromium, iron, mercury, lead and tin contamination; alkalis, chlorine, inorganic chemicals, fertilizers, petroleum refining, basic steel work foundries, you find that everywhere cadmium, chromium, copper, iron - these are common metals that are contributed from the industries.

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9 Heavy metals

Arsenic
Cadmium
Chromium
Cobalt
Copper
Nickel
Mercury
Lead
Zinc

But there are 9 notorious heavy metals that need to be protected and that is why so much of emphasis has been given - Arsenic, Cadmium, Chromium, Cobalt, Copper, Nickel, Mercury, Lead and Zinc.

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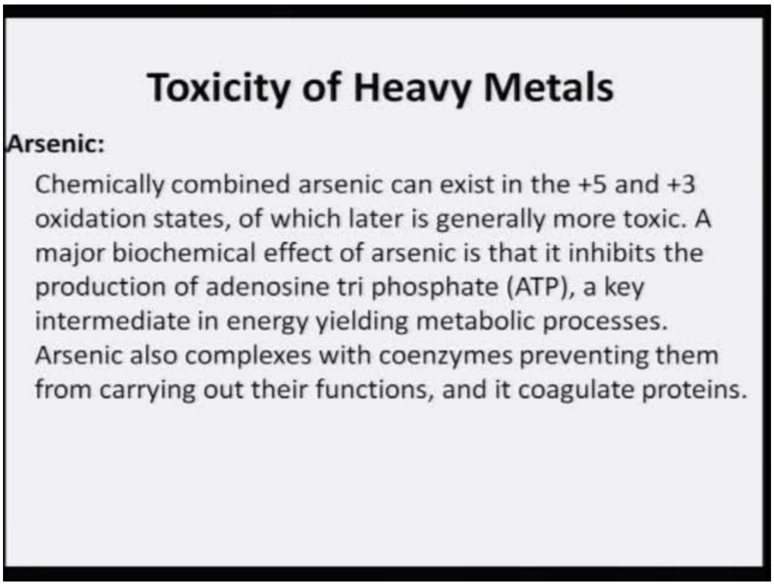

Toxicity of Heavy Metals

Arsenic:

Chemically combined arsenic can exist in the +5 and +3 oxidation states, of which later is generally more toxic. A major biochemical effect of arsenic is that it inhibits the production of adenosine tri phosphate (ATP), a key intermediate in energy yielding metabolic processes. Arsenic also complexes with coenzymes preventing them from carrying out their functions, and it coagulate proteins.

Toxicity of heavy metals - each one has its own very toxic effect and I have just highlighted the toxic effect of these metals in this particular lecture. Arsenic - chemically combined arsenic can exist in the plus 5 oxidation state and plus 3 oxidation state of which later is generally more toxic.

A major biochemical effect of arsenic is that it inhibits the production of adenosine tri phosphate (ATP), a key intermediate in energy yielding metabolic processes. Arsenic also complexes with coenzymes preventing them from carrying out their function and is coagulating is responsible for coagulating proteins.

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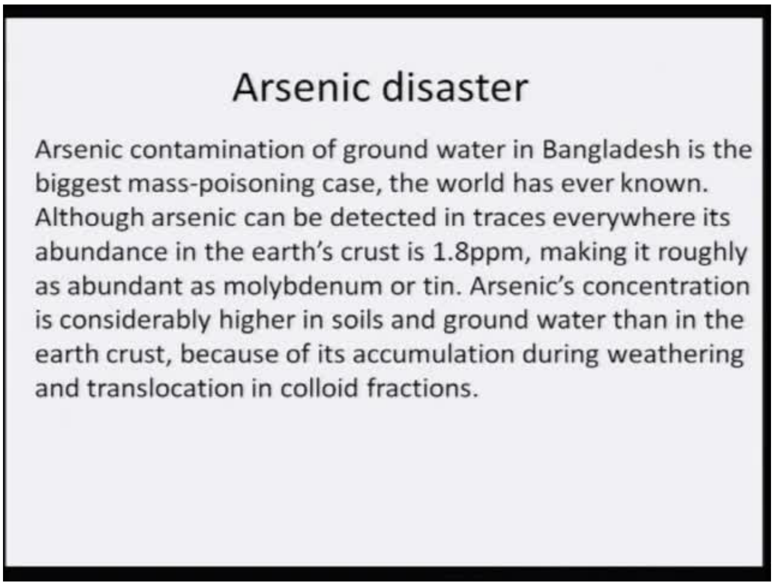

Arsenic disaster

Arsenic contamination of ground water in Bangladesh is the biggest mass-poisoning case, the world has ever known. Although arsenic can be detected in traces everywhere its abundance in the earth's crust is 1.8ppm, making it roughly as abundant as molybdenum or tin. Arsenic's concentration is considerably higher in soils and ground water than in the earth crust, because of its accumulation during weathering and translocation in colloid fractions.

Arsenic disaster - arsenic contamination of ground water in Bangladesh is the biggest mass poisoning case, the world has ever known. Although arsenic can be detected in trace quantities everywhere, its abundance in the earth's crust is about 1.8 ppm (parts per million) making it roughly as abundant as molybdenum or tin. Arsenic's concentration is considerably high in soils and ground water than in the earth crust because of its accumulation during weathering and translocation in colloid fractions.

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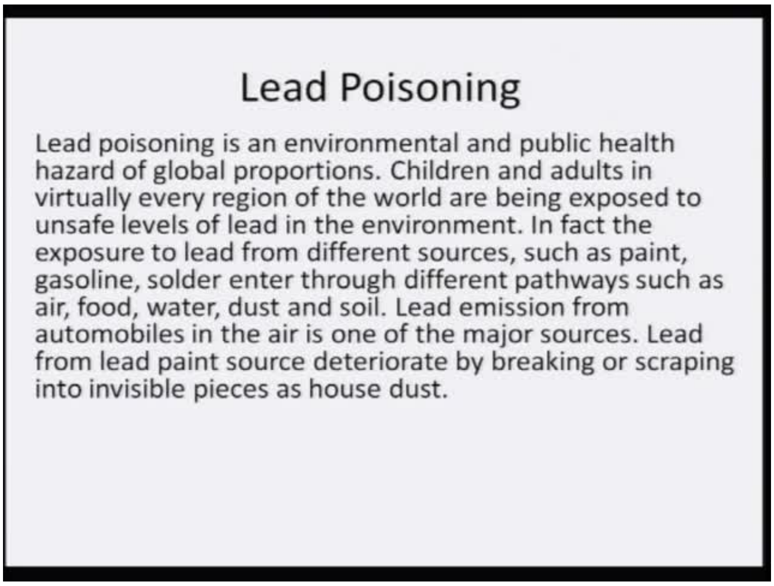

Lead Poisoning

Lead poisoning is an environmental and public health hazard of global proportions. Children and adults in virtually every region of the world are being exposed to unsafe levels of lead in the environment. In fact the exposure to lead from different sources, such as paint, gasoline, solder enter through different pathways such as air, food, water, dust and soil. Lead emission from automobiles in the air is one of the major sources. Lead from lead paint source deteriorate by breaking or scraping into invisible pieces as house dust.

Then comes the lead poisoning. Lead poisoning is an environmental and public health hazard of global proportions. Children and adults in virtually every region of the world are being exposed to unsafe levels of lead in the environment. In fact, the exposure to lead from different sources such as paint, gasoline, solder enters through different pathways such as air, food, water, dust and soil. Lead emission from the automobiles in the air is one of the major sources.

Lead from lead paint sources deteriorate by breaking or scraping its invisible pieces as house dust. Now, let me tell you that is the reason why the unleaded petrol has come into existence because there was so much of lead pollution that was being ejected from these combustion machines.

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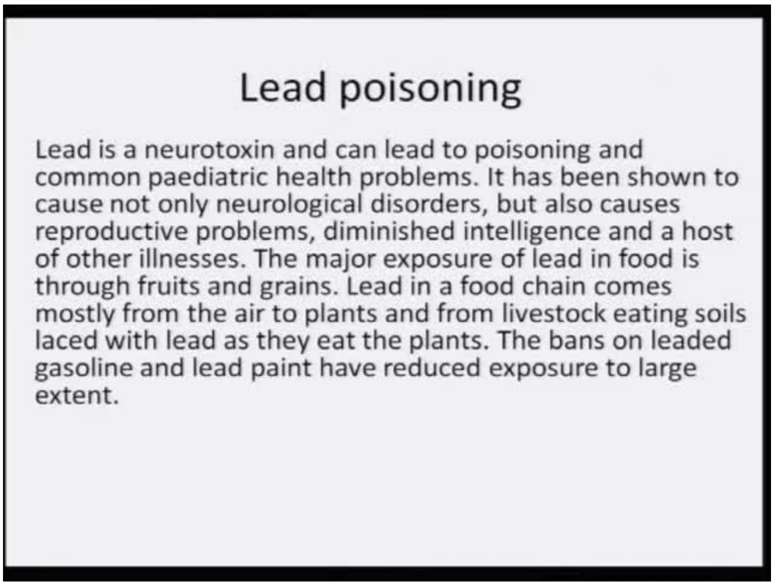

Lead poisoning

Lead is a neurotoxin and can lead to poisoning and common paediatric health problems. It has been shown to cause not only neurological disorders, but also causes reproductive problems, diminished intelligence and a host of other illnesses. The major exposure of lead in food is through fruits and grains. Lead in a food chain comes mostly from the air to plants and from livestock eating soils laced with lead as they eat the plants. The bans on leaded gasoline and lead paint have reduced exposure to large extent.

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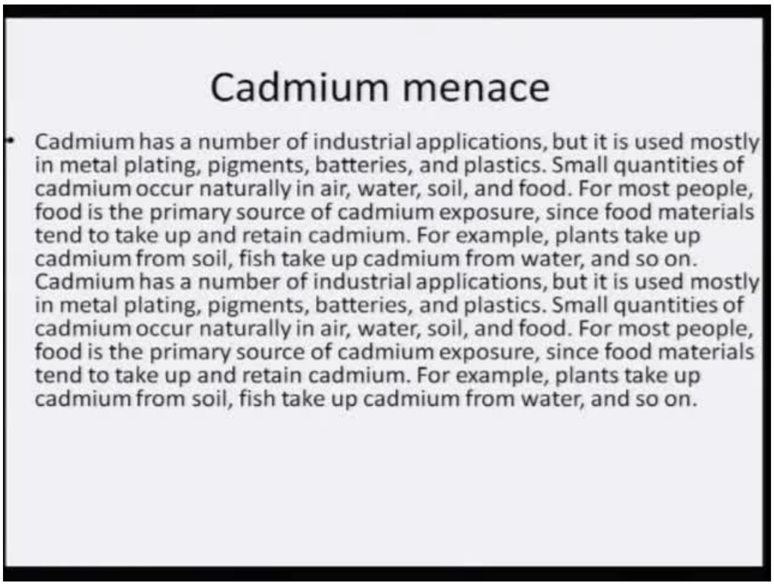

Cadmium menace

- Cadmium has a number of industrial applications, but it is used mostly in metal plating, pigments, batteries, and plastics. Small quantities of cadmium occur naturally in air, water, soil, and food. For most people, food is the primary source of cadmium exposure, since food materials tend to take up and retain cadmium. For example, plants take up cadmium from soil, fish take up cadmium from water, and so on. Cadmium has a number of industrial applications, but it is used mostly in metal plating, pigments, batteries, and plastics. Small quantities of cadmium occur naturally in air, water, soil, and food. For most people, food is the primary source of cadmium exposure, since food materials tend to take up and retain cadmium. For example, plants take up cadmium from soil, fish take up cadmium from water, and so on.

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Cadmium has a number of industrial applications, but it is used mostly in metal plating, pigments, batteries and plastics. Small quantities of cadmium occur naturally in air, water, soil and food. For most people, food is the primary source of exposure and that is why it can go on. Cadmium can get accumulated in the biotic system.

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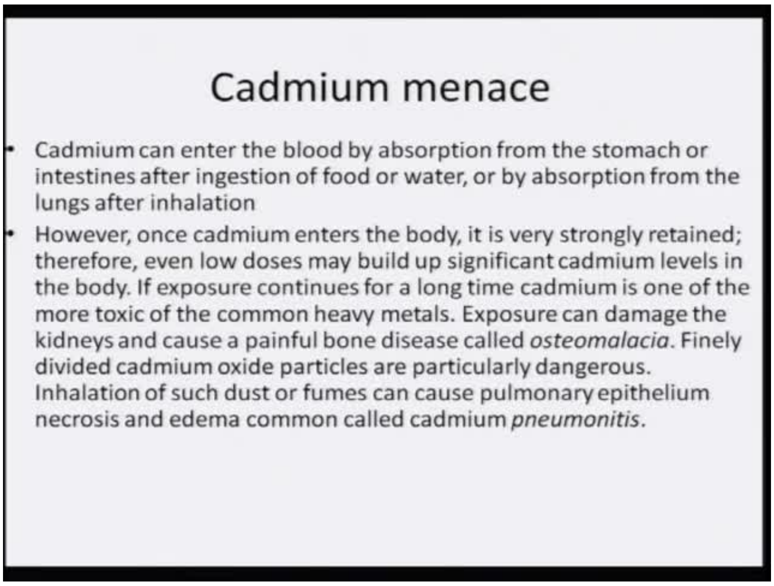

Cadmium menace

- Cadmium can enter the blood by absorption from the stomach or intestines after ingestion of food or water, or by absorption from the lungs after inhalation
- However, once cadmium enters the body, it is very strongly retained; therefore, even low doses may build up significant cadmium levels in the body. If exposure continues for a long time cadmium is one of the more toxic of the common heavy metals. Exposure can damage the kidneys and cause a painful bone disease called *osteomalacia*. Finely divided cadmium oxide particles are particularly dangerous. Inhalation of such dust or fumes can cause pulmonary epithelium necrosis and edema common called cadmium *pneumonitis*.

Cadmium menace - cadmium can enter the body through blood by absorption from the stomach or intestines after indigestion of food or water, or by absorption from the lungs after inhalation. However, once the cadmium enters the body, it is very strongly retained. Therefore, even low doses may build up significant cadmium levels in the body. If exposure continues for a long time, cadmium is one of the more toxic of the common heavy metals. Exposure can damage the kidneys and cause a pain full bone disease called osteomalacia. Finely, divided cadmium oxide particles are particularly dangerous. Inhalation of such dust or fumes can cause pulmonary epithelium necrosis and edema commonly called as cadmium pneumonitis.

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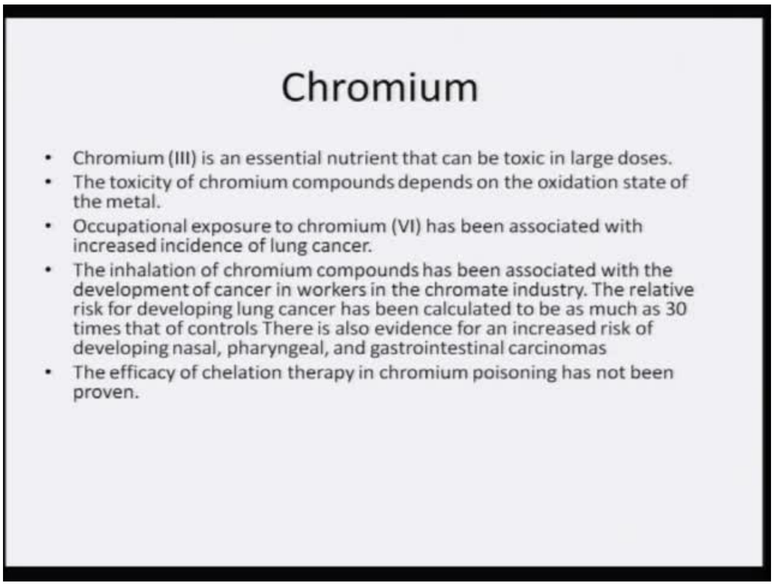

Chromium

- Chromium (III) is an essential nutrient that can be toxic in large doses.
- The toxicity of chromium compounds depends on the oxidation state of the metal.
- Occupational exposure to chromium (VI) has been associated with increased incidence of lung cancer.
- The inhalation of chromium compounds has been associated with the development of cancer in workers in the chromate industry. The relative risk for developing lung cancer has been calculated to be as much as 30 times that of controls There is also evidence for an increased risk of developing nasal, pharyngeal, and gastrointestinal carcinomas
- The efficacy of chelation therapy in chromium poisoning has not been proven.

Chromium - chromium III is an essential nutrient that cannot be toxic, unless it is in large doses. The toxicity of chromium compounds depends on the oxidation state of the metal. Occupational exposure to chromium VI has been associated with increased incidence of lung cancer. The inhalation of chromium compounds has been associated with the development of cancer in workers in the chromate industry. The relative risk for developing lung cancer has been calculated to be as much as 30 times that of controls. There is also evidence for an increased risk of developing nasal, pharyngeal and gastrointestinal carcinomas. The efficacy of chelation therapy in chromium poisoning has also been proven.

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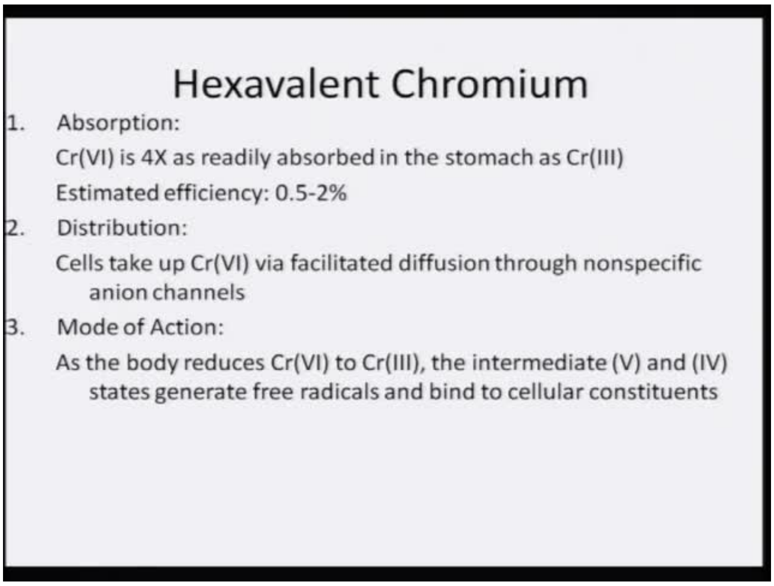

Hexavalent Chromium

1. Absorption:
   Cr(VI) is 4X as readily absorbed in the stomach as Cr(III)
   Estimated efficiency: 0.5-2%
2. Distribution:
   Cells take up Cr(VI) via facilitated diffusion through nonspecific anion channels
3. Mode of Action:
   As the body reduces Cr(VI) to Cr(III), the intermediate (V) and (IV) states generate free radicals and bind to cellular constituents

Hexavalent chromium - absorption - chromium VI is about 4 times readily absorbed in the stomach as compared to chromium III. Estimated efficiency is 0.5 to 2 percent. It is distributed in the cells and they take up chromium VI via facilitating diffusion through non-specific ionic channels and the mode of action as the body reduces chromium VI to chromium III, the intermediate chromium V and IV states generate free radicals and bind to cellular constituents. So, this is the kind of mechanism, where the toxicity and the effect of hexavalent chromium can be understood.

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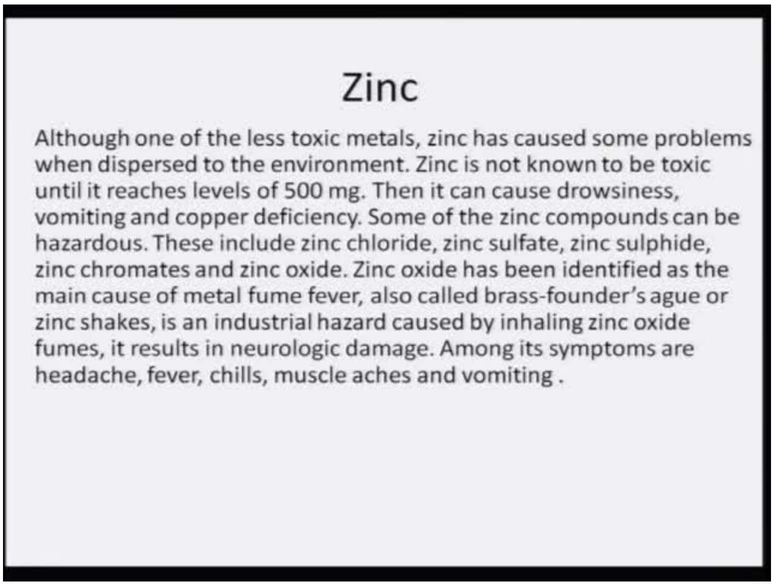

Zinc

Although one of the less toxic metals, zinc has caused some problems when dispersed to the environment. Zinc is not known to be toxic until it reaches levels of 500 mg. Then it can cause drowsiness, vomiting and copper deficiency. Some of the zinc compounds can be hazardous. These include zinc chloride, zinc sulfate, zinc sulphide, zinc chromates and zinc oxide. Zinc oxide has been identified as the main cause of metal fume fever, also called brass-founder's ague or zinc shakes, is an industrial hazard caused by inhaling zinc oxide fumes, it results in neurologic damage. Among its symptoms are headache, fever, chills, muscle aches and vomiting .

Coming to zinc, which is another heavy metal toxic and notorious. Although one of the less toxic metals, zinc has caused some problems when dispersed to the environment. Zinc is not known to be toxic until it reaches levels of 500 milligram. Then it can cause drowsiness, vomiting and copper deficiency. Some of the zinc compounds can be hazardous. These include zinc chloride, zinc sulphate, zinc sulphide, zinc chromates and zinc oxide.

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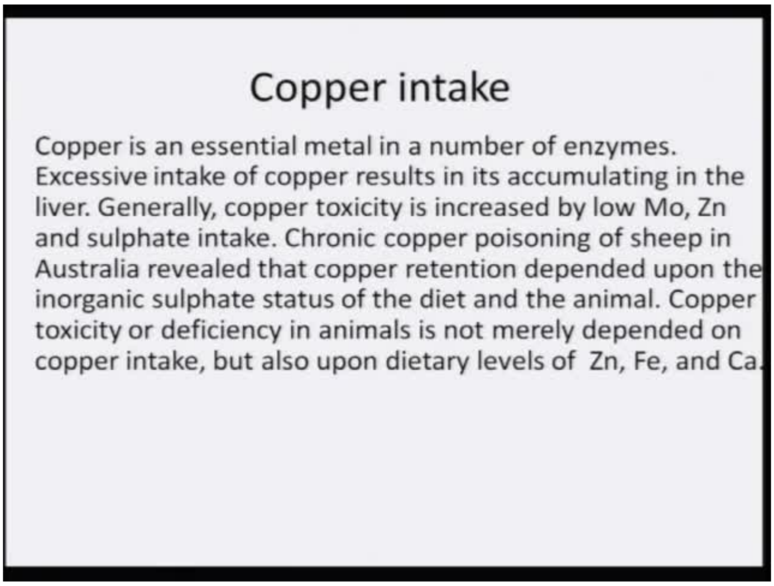

Copper intake

Copper is an essential metal in a number of enzymes. Excessive intake of copper results in its accumulating in the liver. Generally, copper toxicity is increased by low Mo, Zn and sulphate intake. Chronic copper poisoning of sheep in Australia revealed that copper retention depended upon the inorganic sulphate status of the diet and the animal. Copper toxicity or deficiency in animals is not merely depended on copper intake, but also upon dietary levels of Zn, Fe, and Ca.

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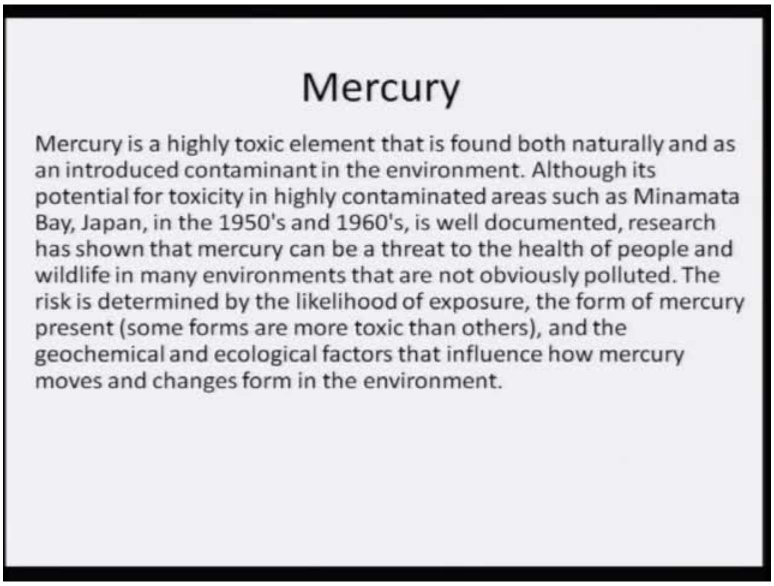

Mercury

Mercury is a highly toxic element that is found both naturally and as an introduced contaminant in the environment. Although its potential for toxicity in highly contaminated areas such as Minamata Bay, Japan, in the 1950's and 1960's, is well documented, research has shown that mercury can be a threat to the health of people and wildlife in many environments that are not obviously polluted. The risk is determined by the likelihood of exposure, the form of mercury present (some forms are more toxic than others), and the geochemical and ecological factors that influence how mercury moves and changes form in the environment.

Mercury is yet another, but very highly toxic element that is found both naturally as an introduced contaminant in the environment. Although its potential for toxicity in highly contaminated areas such as Minamata bay in Japan and in 1950’s and 1960’s it is well documented, that research has shown that mercury can be a threat to the health of people and wildlife in many environments that are not obviously polluted.

The risk is determined by the likelihood of exposure, the form of mercury present. Some forms are more toxic than the others as what we saw in the case of chromium as well. Chromium III is less very less toxic as compared to chromium VI. Similarly, mercury also exists in various forms and some of the forms are more toxic than the others and the geochemical and ecological factors that influence how mercury moves and changes form in the environment is also very well understood. So, from one form to the other form, the mercury somehow reaches the biotech system and the food chain, and that is how it reaches to human beings.

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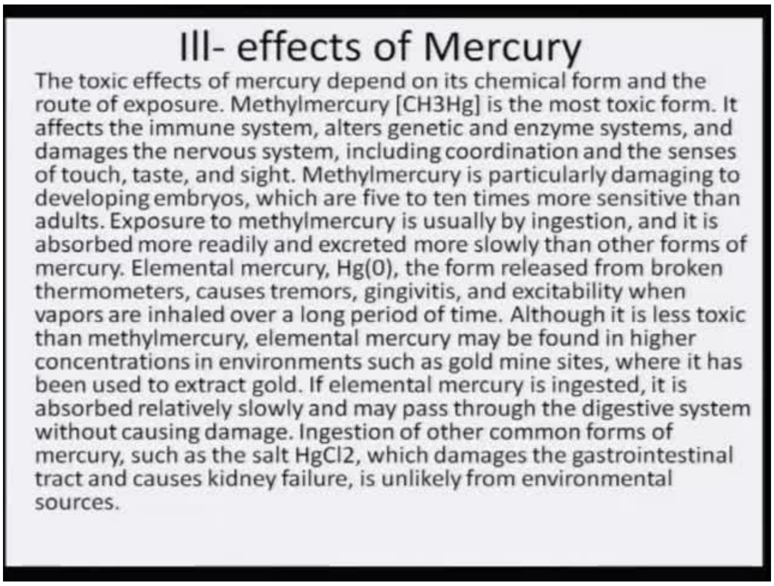

Ill- effects of Mercury

The toxic effects of mercury depend on its chemical form and the route of exposure. Methylmercury [CH3Hg] is the most toxic form. It affects the immune system, alters genetic and enzyme systems, and damages the nervous system, including coordination and the senses of touch, taste, and sight. Methylmercury is particularly damaging to developing embryos, which are five to ten times more sensitive than adults. Exposure to methylmercury is usually by ingestion, and it is absorbed more readily and excreted more slowly than other forms of mercury. Elemental mercury, Hg(0), the form released from broken thermometers, causes tremors, gingivitis, and excitability when vapors are inhaled over a long period of time. Although it is less toxic than methylmercury, elemental mercury may be found in higher concentrations in environments such as gold mine sites, where it has been used to extract gold. If elemental mercury is ingested, it is absorbed relatively slowly and may pass through the digestive system without causing damage. Ingestion of other common forms of mercury, such as the salt HgCl2, which damages the gastrointestinal tract and causes kidney failure, is unlikely from environmental sources.

Ill effects of mercury - the toxic effects of mercury depend on its chemical form and the route of exposure. Methyl mercury, that is, CH3Hg is the most toxic form. It affects the immune system, alters genetic and enzyme system and damages the nervous system, including coordination and the senses of touch, taste and sight.

Methyl mercury is particularly damaging to developing embryos, which are 5 to 10 times more sensitive than adults. So, for foetus it is highly, highly dangerous. Exposure to methyl mercury is usually by ingestion and it is absorbed more readily and excreted more slowly than other forms of mercury. Elemental mercury, that is, Hg in the zero oxidation state, the form released from broken thermometers causes tremors, gingivitis and excitability when vapours are inhaled over a long period of time.

Although it is less toxic than methyl mercury, elementary mercury may be found in higher concentrations in environment such as gold mine sites, where it has been used to extract gold. If elementary mercury is ingested, it is absorbed relatively slowly and may pass through the digestive system without causing much damage. Ingestion of other common forms of mercury such as salts like mercury chloride, which damages the gastrointestinal tract and causes kidney failure, is likely from the environmental source.

So, you see that how dangerous and how toxic these metals are, when particularly these 9 metals that I mentioned and that is why it is important to understand what are the poisoning effect, what is the chemistry behind their reactivity towards the biological system and the human system, and how they should be analyzed and prevented so that they do not spread over in the environment and do not reach the human.

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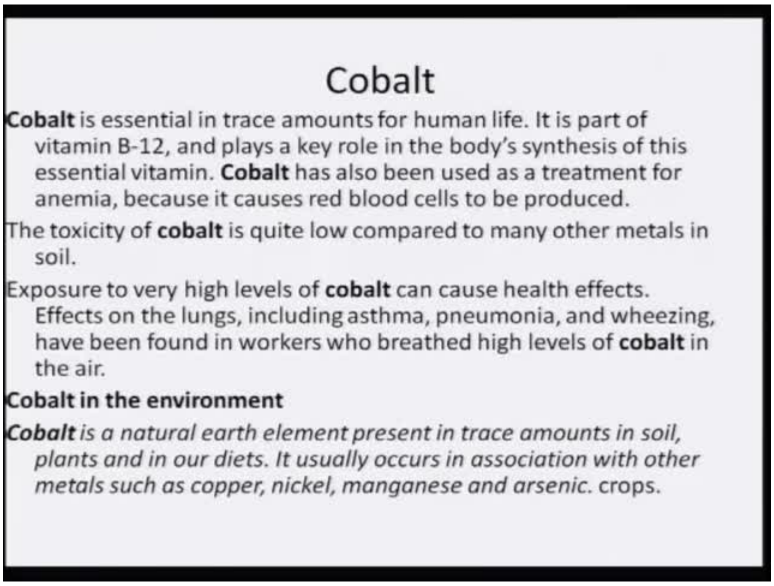

Cobalt

Cobalt is essential in trace amounts for human life. It is part of vitamin B-12, and plays a key role in the body's synthesis of this essential vitamin. **Cobalt** has also been used as a treatment for anemia, because it causes red blood cells to be produced.

The toxicity of **cobalt** is quite low compared to many other metals in soil.

Exposure to very high levels of **cobalt** can cause health effects. Effects on the lungs, including asthma, pneumonia, and wheezing, have been found in workers who breathed high levels of **cobalt** in the air.

Cobalt in the environment

Cobalt *is a natural earth element present in trace amounts in soil, plants and in our diets. It usually occurs in association with other metals such as copper, nickel, manganese and arsenic.* crops.

Similarly, cobalt is also one of the 9 culprits of the heavy metal series. Cobalt is essential in trace amounts for human life. It is part of the vitamin B-12 and plays a key role in the body's synthesis of this essential vitamin. Cobalt has also been used as a treatment for anemia because it causes red blood cells to be produced. The toxicity of cobalt is quite low compared to many other metals that I mentioned a while ago, in soil. Exposure to very high levels of cobalt can cause health effects. Effects on the lung including asthma, pneumonia and wheezing have been found in workers, who breathed high levels of cobalt in the air.

Cobalt in the environment - how much of cobalt is present in the environment? Cobalt is a natural earth element present in trace amounts in soil, plants and in our diets. It usually occurs in association with other metals such as copper, nickel, manganese and arsenic crops.

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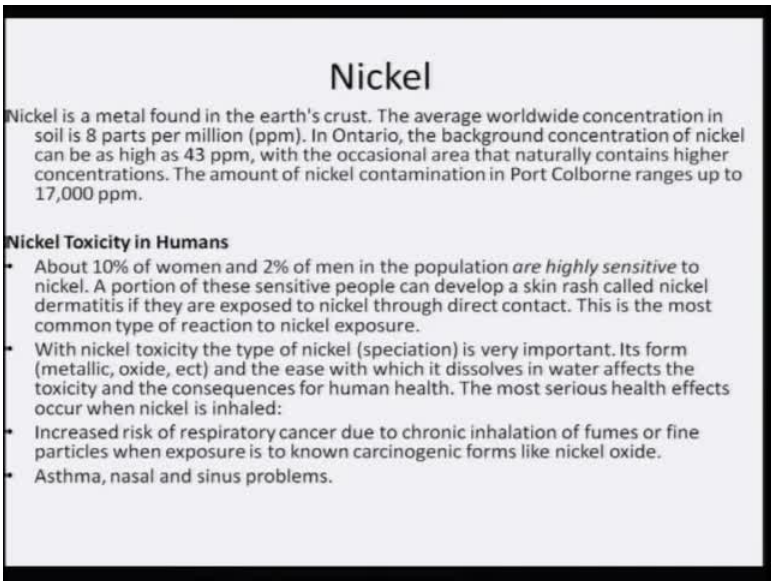

Nickel

Nickel is a metal found in the earth's crust. The average worldwide concentration in soil is 8 parts per million (ppm). In Ontario, the background concentration of nickel can be as high as 43 ppm, with the occasional area that naturally contains higher concentrations. The amount of nickel contamination in Port Colborne ranges up to 17,000 ppm.

Nickel Toxicity in Humans

- About 10% of women and 2% of men in the population *are highly sensitive* to nickel. A portion of these sensitive people can develop a skin rash called nickel dermatitis if they are exposed to nickel through direct contact. This is the most common type of reaction to nickel exposure.
- With nickel toxicity the type of nickel (speciation) is very important. Its form (metallic, oxide, ect) and the ease with which it dissolves in water affects the toxicity and the consequences for human health. The most serious health effects occur when nickel is inhaled:
- Increased risk of respiratory cancer due to chronic inhalation of fumes or fine particles when exposure is to known carcinogenic forms like nickel oxide.
- Asthma, nasal and sinus problems.

Coming to yet another next metal is nickel. It is also not very toxic, but still any over doses of nickel can be in the toxic limit. Nickel is a metal found in the earth’s crust. The average worldwide concentration in soil is 8 parts per million (ppm). In Ontario, that is, Canada, the background concentration of nickel can be as high as 43 ppm with the occasional area that naturally contains higher concentrations. The amount of nickel contamination in Port Colborne ranges up to 17000 ppm. So, there it reaches the toxic limit.

Nickel toxicity in humans - about 10 percent of women and 2 percent of men in the population are highly sensitive to nickel. A portion of these sensitive people can develop skin rash called nickel dermatitis, if they are exposed to nickel through direct contact. This is the most common type of reaction to nickel exposure. With nickel toxicity, the type of nickel (speciation) is very important. Its form, of either in the form of metallic form or oxide form or any other form and the ease with which it dissolves in water affects the toxicity and the consequences to human health. The most serious health effects occur when nickel in inhaled. Increased risk of respiratory cancer due to chronic inhalation of fumes or fine particles when exposed is to be known as carcinogenic form of nickel oxide. Asthma, nasal and sinus problems are usual symptoms that nickel can cause.

So, you have seen that out of these 9 metals not all were very dangerous. However, they are equally dangerous when they are in very high quantity and at the same time, they need to be analyzed properly so that they can be extracted from the matrix and the level of contamination or pollution can be estimated correctly.