INDIAN INSTITUTE OF TECHNOLOGY ROORKEE
NPTEL
NPTEL ONLINE CERTIFICATION COURSE
Structural Analysis of Nanomaterials
Lecture- 20
Other Characterization Techniques
With
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Hello today we are going to discuss about the other characterization techniques. This is the last lecture from this particular course. So, in our last two lectures we have discussed about the different types of characterization techniques.

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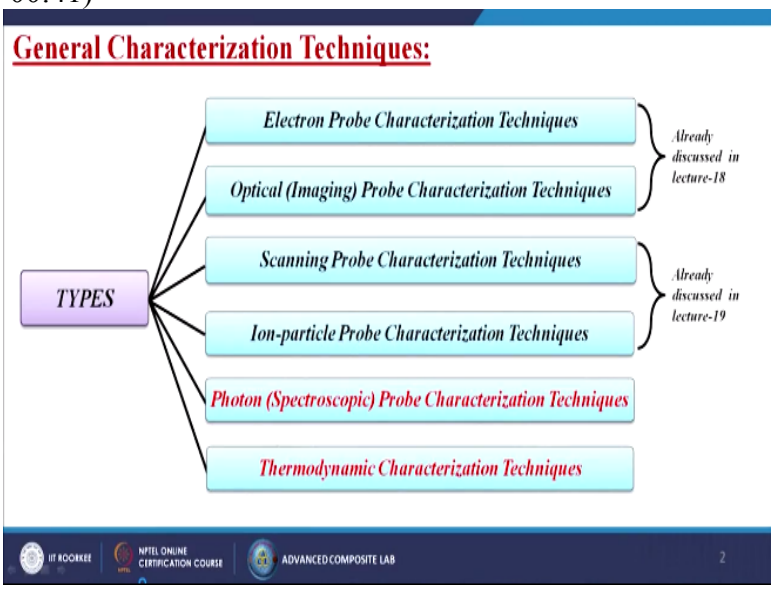

Mainly, electron probe characterization techniques, optical imaging probe characterization techniques, scanning probe characterization techniques, ion particle probe characterization techniques. In this particular lecture we are going to discuss about the photon spectroscopic probe characterization techniques and the thermodynamic characterization techniques.

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Photon (Spectroscopic) Probe Characterization Techniques:

Acronym	Technique	Utility
UV-Vis	UV-Visible Spectroscopy	Chemical Analysis
FS	Fluorescence Spectroscopy	Elemental Analysis
AAS	Atomic Absorption Spectroscopy	Chemical Analysis

UV-Visible Spectroscopy (UV-Vis):

- It is also known as electronic spectroscopy.
- In this, the amount of light absorbed at each wavelength of UV and visible regions of electromagnetic spectrum is measured.
- This absorption of electromagnetic radiations by the molecules leads to molecular excitation.
- It is usually applied to molecules and inorganic ions in solution.

So, first when we are going to discuss about the photon spectroscopic probe characterization techniques: So, generally the three characterization technique is coming under these particular techniques, one is called the UV-Vis that is the UV-Visible spectroscopic. So, generally we are using it for the chemical analysis. Another one is called FS or maybe the fluorescence spectroscopy, we are using it for the elemental analysis another one is called the AAS that is atomic absorption spectroscopy that is for the chemical analysis of the materials. So, first let us know that what is UV-Visible spectroscopy?

So, it is also known as the electronic spectroscopy. In this, the amount of light absorbed at each wavelength of UV and visible regions of electromagnetic spectrum is measured. This absorption of electromagnetic radiations by the molecules leads to molecular excitation. It is usually applied to molecules and inorganic ions in solution.

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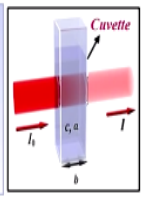
Beer-Lambert Law:

- Analyte concentration from absorbance at one wavelength can be determine using *Beer-Lambert law;*

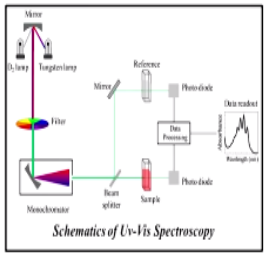
$$A = -\log\left(I/I_0\right) = a * b * c$$

where; A = Absorbance, a = Absorption coefficient, b = Path length, and c = Concentration, I_0 and I = Intensity of incident and transmitted light respectively.



Instrumentation:

- ✓ They are dual-beam spectrometers where first channel contains sample and second channel holds control (buffer) for correction.
- ✓ Light source is tungsten filament bulb for visible part of spectrum, and deuterium bulb for UV region.
- ✓ Monochromator, consists of prism or a rotating metal grid of high precision called grating, is placed between light source and sample.
- ✓ Prism splits the incoming light into its components by refraction and refraction occurs because radiation of different wavelengths travels along different paths in medium of higher density.
- ✓ For UV measurements, quartz cuvettes are used.



Schematics of Uv-Vis Spectroscopy

Now we are going to discuss about the Beer-Lambert law. This means analyte concentration from absorbance at one wavelength can be determined using the Beer-Lambert law. What is that?

That is capital A = -log (I/I_0) = A * B * C. So, where capital A is the absorbance, small A is the absorption coefficient, B is the path length, C is the concentration, I_0 and I is equal to intensity of incident and transmitted light respectively.

So, from this image you can easily understand about the Beer-Lambert law. So, how we are going to measure this one? So, generally they are dual-beam spectrometers where first channel contains sample and second channel holds control for corrections. So, generally it is for the buffer. Light source is tungsten filament bulb for visible part of spectrum and deuterium bulb for the UV region.

Monochromator consists of a prism or a rotating metal grid of high precision called grating is placed between the light source and the sample itself. So, here in this case we are having D2 lamp and the tungsten lamp and we are having the mirror. Then we are having the filter, so we are using the Monochromator then we are having the beam splitter then it is going to the mirror, then it is falling to the reference and then photodiode and data processing and from here another one it is going directly to the sample and here also we are using another photodiode then data processing and we are getting the results.

So, prism splits the incoming light into its components by refractions and refraction occurs because radiation of different wavelengths travels along the different paths in medium of higher density. For UV measurements, quarts cuvettes are used. Procedures in the lab prepare a solution of the compound

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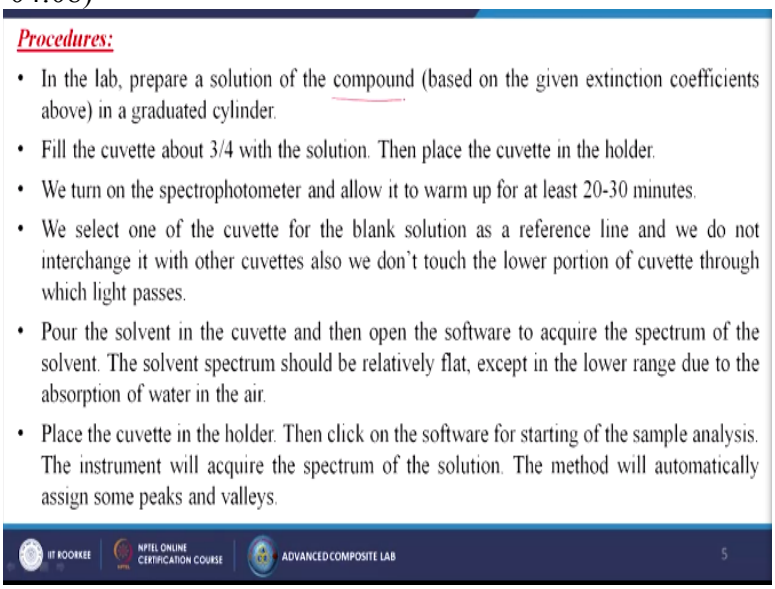

Based on the given extinction coefficients above in a graduated cylinder, fill the cuvette about ¾ with the solutions then place the cuvette in the holder itself. We turn on the spectrophotometer and allow it to warm up for at least 20-30 minutes. We select one of the cuvette for the blank solution as a reference line and we do not interchange it with other cuvettes also we do not touch the lower portion of the cuvettes through which the light passes, otherwise maybe something a paint or maybe something may come.

Pour the solvent in cuvette and then open the software to acquire the spectrum on the solvent. The solvent spectrum should be relatively flat, except in the lower range due to the absorption of

water in the air. Place the cuvette in the holder then click on the software for starting of the sample analysis. The instrument will acquire the spectrum of the solution. The method will automatically assign some peaks and valleys. So, this is the whole procedure generally how we are going to perform UV-Vis analysis for our sample. Sample handling virtually,

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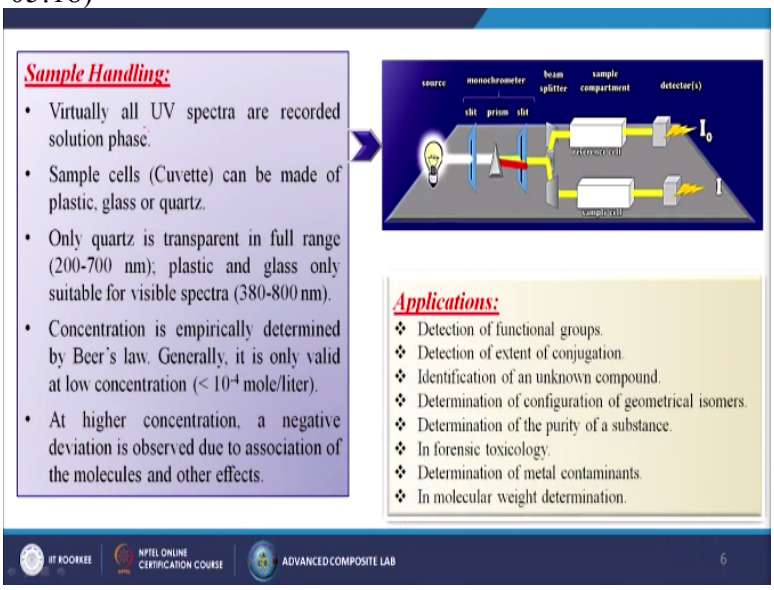

All UV spectra are recorded solution phase. Sample cells whatever I was talking about the cuvette can be made of, maybe plastic, glass or maybe the quartz. Only quartz is transparent in full range. Generally 200-700 nanometer; plastic and glass only suitable for visible spectra generally it is 380-800 nanometer range. Concentration is empirically determined by Beer's law. Generally, it is only valid at low concentration that is < is equal to 10^{-4} mole or liter. At higher concentration, a negative deviation is observed due to association of the molecules and other effects.

So, this is the whole procedure over there. So, now what are the applications means where we are using these UV-Vis spectra? For detection of functional groups, detection of extent of conjugation, identification of an unknown compound, determination of configuration of geometrical isomers. Determination of the purity of the substance, in forensic toxicology, determination of metal contaminants, in molecular weight determinations but still we are using this one for other applications also. Factors affecting UV-Vis absorptions: so generally the biochemical

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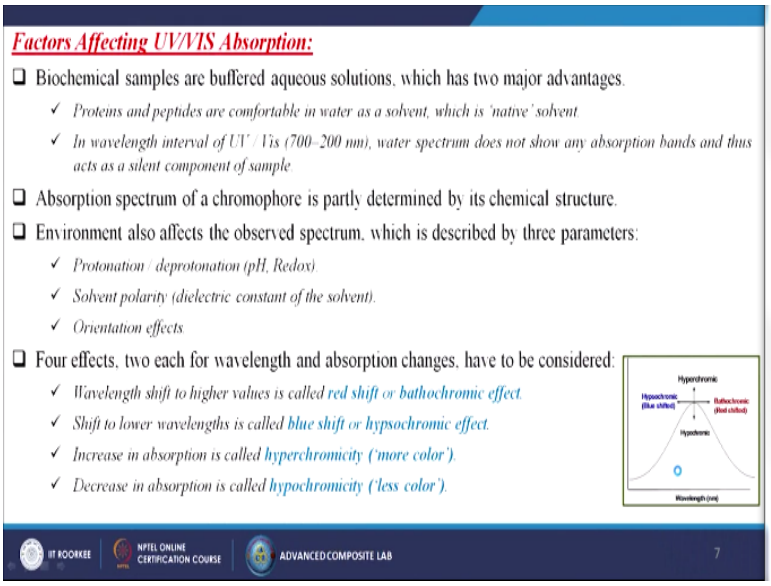

Samples are buffered aqueous solutions, which has two major advantages. One is proteins and peptides are comfortable in water as a solvent which is native solvent. In wavelength interval of UV-Vis, in general in between 700-200 nanometer, water spectrum does not show any absorption bands and thus acts as a silent component of sample. Absorption spectrum of a chromophore is partly determined by its chemical structure.

Environment also affects the observed spectrum which is described by three parameters. First one is called the protonation or maybe the, deprotonation that means either pH level or maybe the Redox reaction. Second solvent polarity, dielectric constant of the solvent and third is the orientation effects. Four effects, two each for wavelength and absorption changes have to be considered. What are those?

Wavelength shift to higher values is called the red shift or maybe the bathochromic effect. Shifts to the lower wavelength, is called the blue shift or maybe the hypsochromic effect. Increase in absorption is called the hyperchromicity, more color. Decrease in absorption is called the hypochromicity that is less color. So from this, if it is going to the left side, it is the blue shifted. If it is going to the right side it is called the red shifted. If it is going to the above that is hypochromic and it is coming down it is known as the hyperchromic.

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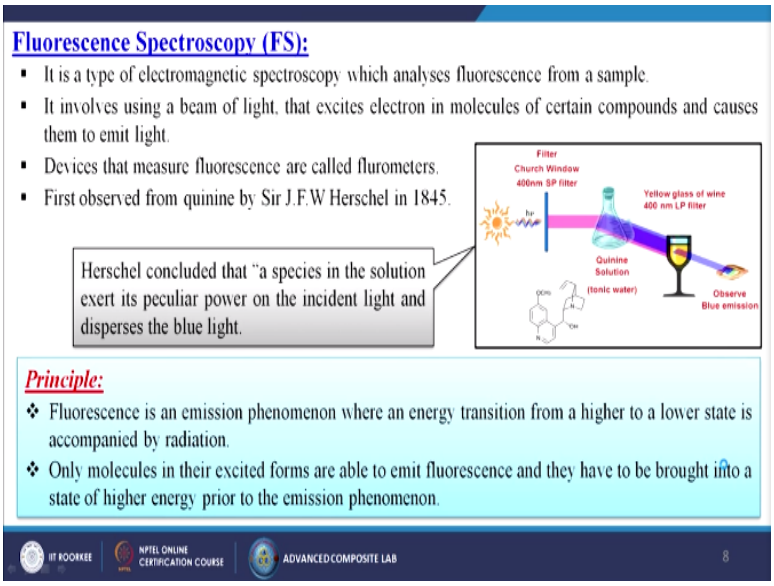

Next technique is called the fluorescence spectroscopy in short generally we are calling it as FS. It is a type of electromagnetic spectroscopy which analyses fluorescence from a sample itself. It involves using a beam of light that excites electron in molecules of certain compounds and causes them to emit light. Devices that measure fluorescence are called flurometers. First, observed from quinine by Sir J.F.W Herschel in 1845.

Herschel concluded that a species in the solution exert its peculiar power on the incident light and disperses the blue light itself. So, here we are having the source and then we are giving the energy each new filter church window generally 400 nanometer SP filter. Then we are having that quinine solutions or maybe the tonic water, is known. The yellow glass of wine 400 nm LP filer, generally we are using then we are observing the blue emission.

Principle: fluorescence is an emission phenomenon where an energy transmission from a higher to a lower state is accompanied by radiations. Only molecules in their excited forms are able to emit fluorescence and they have to be brought into a state of higher energy prior to the emission phenomenon. What are the components of spectro flurometer light source?

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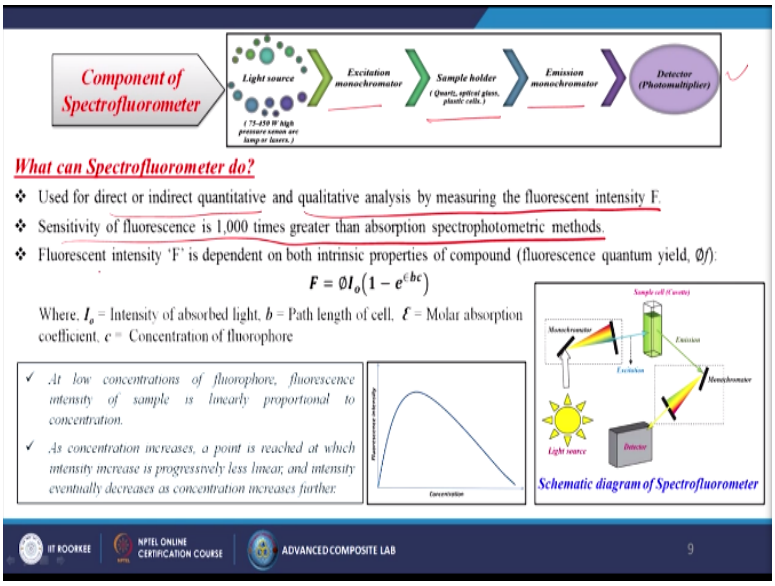

So, generally it is 750, 75-450 watt, high pressure xenon arc lamp or maybe the lasers. Then excitation Monochromator, then sample holder either it may be made by quartz optical glass or maybe the plastic cells. Then emission Monochromator and last we are having the detector or maybe the photomultiplier. What can spectroflurometer do? Used for direct or indirect quantitative and qualitative analysis by measuring the fluorescent intensity F.

Sensitivity of fluorescence is 1000 times greater than absorption spectrophotometric methods. Fluorescent intensity F is depend on both intrinsic properties of compound that is fluorescence quantum yield or maybe the Φ F. $F = \Phi I_0 * (1-e^{\varepsilon bc})$ where I_0 is the intensity of absorbed light, B is the path length of cell, ε is the molar absorption coefficient, C is the concentration of fluorophore.

At low concentration of fluorophore, fluorescence intensity of sample is linearly proportional to concentrations. As concentration increases, a point is reached at which the intensity increases is progressively less linear and intensity eventually decrease as the concentration of increase further. So, first it is rapidly increasing after that it will become slow then after that it is going to decrease.

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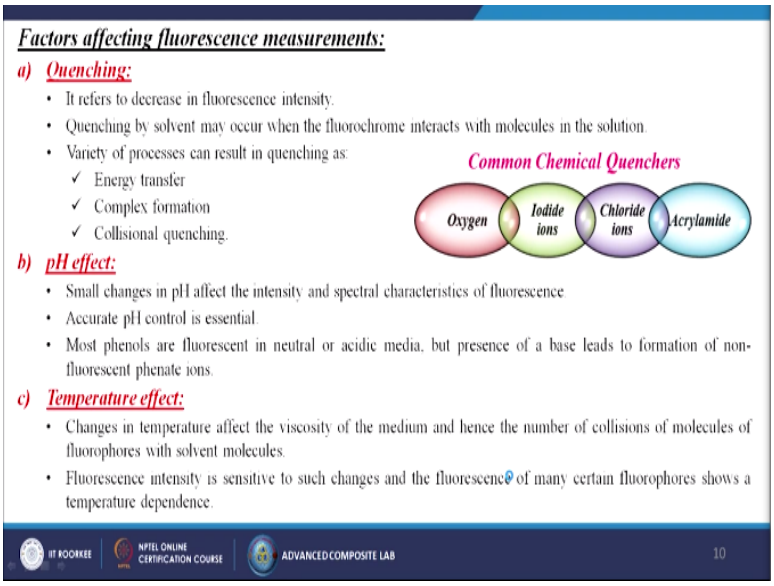

Factors affecting the fluorescence measurements: what are those? First one is called the quenching. It refers to the decrease in fluorescence intensity. Quenching by solvent may occur when the fluorochrome interacts with molecules in the solution. Variety of processes can result in quenching as energy transfer, complex formations and the collisional quenching. So, common chemical quenchers are oxygen, iodide ions, chloride ions and the acrylamide. Next one is the pH effect: small changes in pH affect the intensity and the spectral characteristics of fluorescence.

Accurate pH control is essential. Most phenols are fluorescent in neutral or acidic media, but presence of a base leads to formation of non-fluorescent phenate ions. Then third one is called the temperature effect. So, changes in temperature affect, the viscosity of the medium and hence the number of collisions of molecules of fluorophores with solvent molecules. Fluorescence intensity is sensitive to such changes and the fluorescence of many certain fluorophores shows, a temperature dependence.

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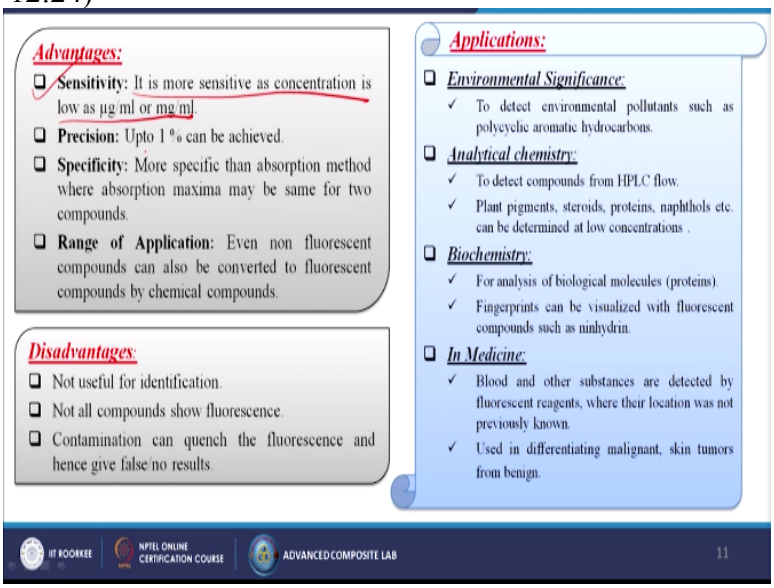

Advantages: so, first one is sensitivity, it is more sensitive as concentration is low as microgram per milliliter or maybe the milligram per milliliter. Second one is called the precision: up to 1% can be achieved. Third one is called the specificity: more specific than absorption method where absorption maxima may be same for two compounds and the last one is called the range of applications, even non-fluorescent compounds can also be converted to fluorescent compounds by chemical compounds.

Then disadvantages, not useful for identifications, not all compounds show fluorescence and third one is the contamination can quench the fluorescence and hence give false or maybe any no results. What are the applications? Generally it can be used for the environmental significance, to detect the environmental pollutants such as polycyclic aromatic hydrocarbons. It can be used for the analytical chemistry to detect compounds from HPLC flow.

Plant pigments, steroids, proteins, naplathols etc can be determined at low concentrations. Biochemistry: for analysis of biological molecules like proteins. Fingerprints can be visualized with fluorescent compounds such as ninhydrin. In medicine: blood and other substances are detected by fluorescent reagents, where their location was not previously known. Used in differentiating malignant, skin tumors from benign.

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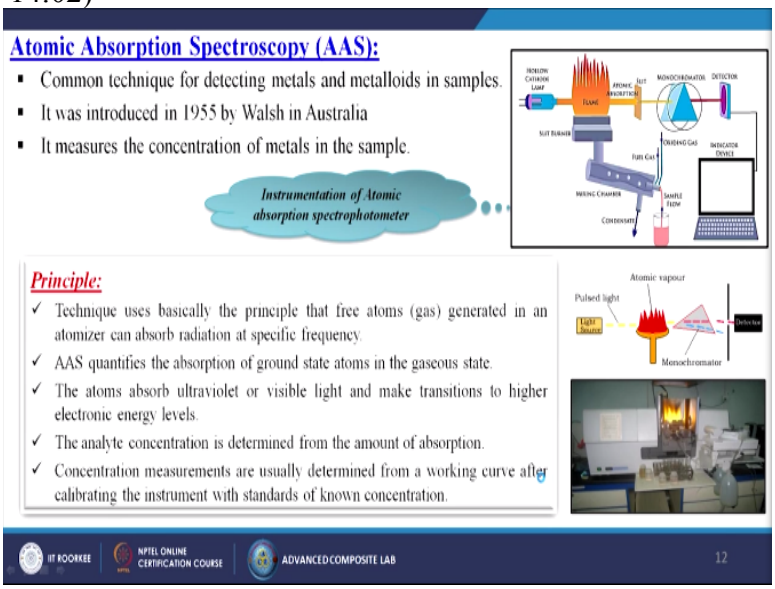

Next one is called the atomic absorption spectroscopy; in short generally we are calling it as AAS. It is the common technique for detecting metals and metalloids in samples. It was introduced in 1955 by Walsh in Australia. It measures the concentration of metals in the sample. So, basically this is the instrumentation of atomic absorption spectrophotometer. So, we are having hollow cathode lamp and then we are having the burner, so just we are burning the sample then atomic absorption is taking place then we are having the slit then we are having the Monochromator then we are having the detector.

So, this is the mixing chamber and condensate and sample flow is taking place. So fuel gas and oxidizing gas is evolving from this particular point and from detector, directly we are getting the data into the computer itself. So, what are the principles? Technique uses basically the principle

that free atoms gas generated in an atomizer can absorb radiation at specific frequency. AAS quantifies the absorption of ground state atoms in the gaseous state.

The atoms absorb ultraviolet or visible light and make transitions to higher electronic energy levels. The analyte concentration is determined from the amount of absorption. Concentration measurements are usually determined from a working curve after calibrating the instrument with standards of known concentrations. So, generally we are having the pulse light or may be the light source then we are having the atomic vapor then Monochromator and the detector. This is the simplest and this is the instrument generally what we are using for the AAS study.

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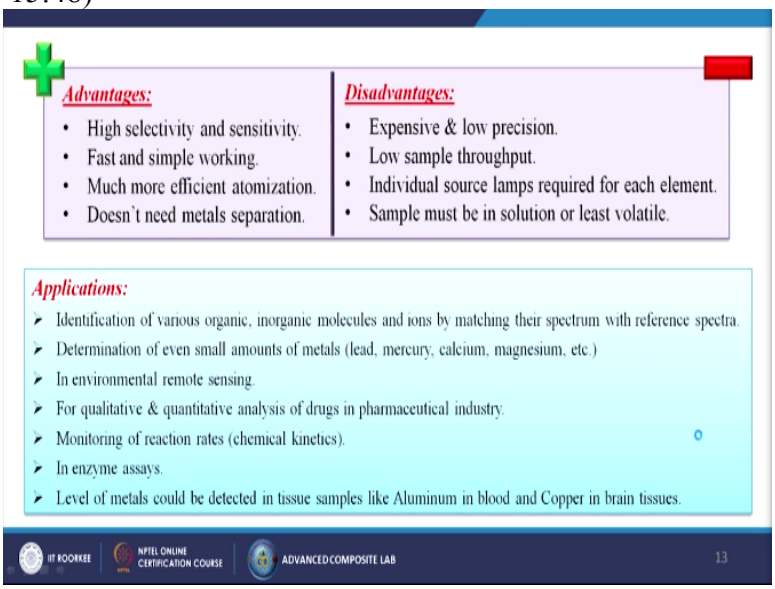

What are the advantages? High selectivity and sensitivity, fast and simple working, much more efficient atomization, does not need metals separation. Of course there are certain disadvantages also. What are those? First one is the expensive and low precision. Second is that low sample throughput. Third is the individual source lamps required for each element. Fourth is the sample must be in solution or least volatile.

What are the applications of AAS? Identification of various organic, inorganic molecules and ions by matching their spectrum with reference spectra, determination of even small amounts of metals like lead, mercury, calcium, magnesium etc in environmental remote sensing. For quantitative and qualitative analysis of drugs in pharmaceutical industry, monitoring of reaction rates like chemical kinetics.

In enzyme assays, level of metals could be detected in tissue samples like aluminium in blood and copper in brain tissues. Now we are going to discuss about the thermodynamic characterization techniques. Of course there are some other sub techniques of this particular group. What are those? First one is called the TGA, TGA the full form is thermal gravimetric analysis.

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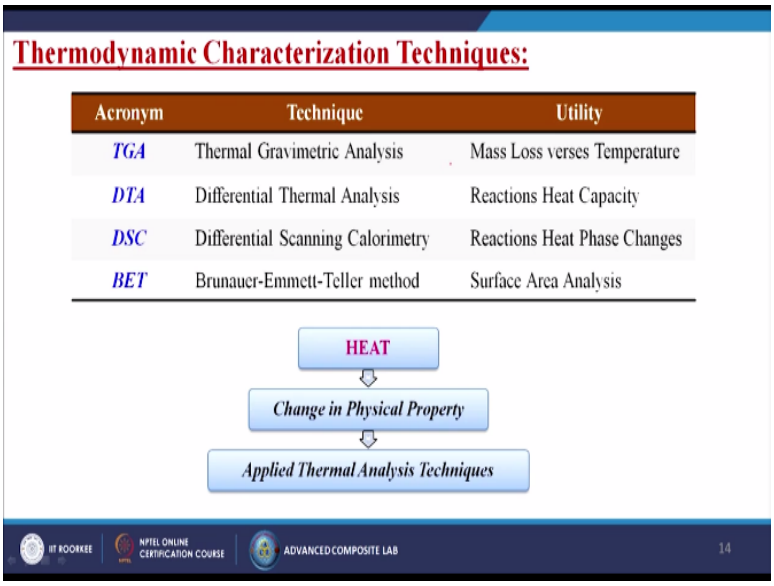

Must loss verses temperature generally we are getting. Next one is called the DTA, differential thermal analysis that is reactions heat capacity we are getting. Next one is called the DSC, differential scanning calorimetry that is reaction heat phase changes generally we are getting and the last one is called the BET that is brunauer-Emmett-teller method. Generally the surface area analysis we are doing. So, how we are doing? We are giving the heart to the samples, then change in the physical property and applied thermal analysis techniques over there.

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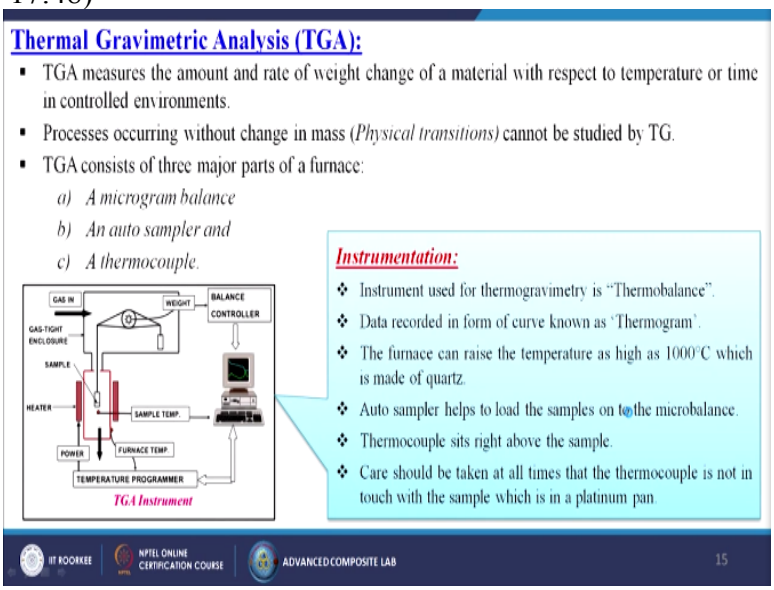

So, first we are going to do the thermal gravimetric analysis or maybe the TGA. Sometimes it is called the thermal gravimetric analysis also. So, TGA measures the amount and rate of weight change of a material with respect to temperature or time in controlled environments. Processes occurring without change in mass (physical transitions) cannot be studied by the TG. TGA consists of three major parts of furnace, first one is called the, a microgram balance, second an auto sampler and third one a thermocouple.

The instrumentations are looks like this. So, generally we are having that heater and then we are putting our samples, then we are giving the samples, a particular temperature from a minimum to maximum with a constant heating or maybe sometimes it maybe the repudiating rate or maybe sometimes it maybe the discontinuous heating rate. So, what are the instrumentations? Instrument used for thermogarvimetry is the thermobalance.

Data recorded in form of curve known as thermogram. The furnace can raise the temperature as high as 1000 ° C which is made of quartz. Auto sampler helps to load the samples on to the microbalance. Thermocouple sits right above the sample. Care should be taken at all times that the thermocouple is not in touch with the sample which is in a platinum pan.

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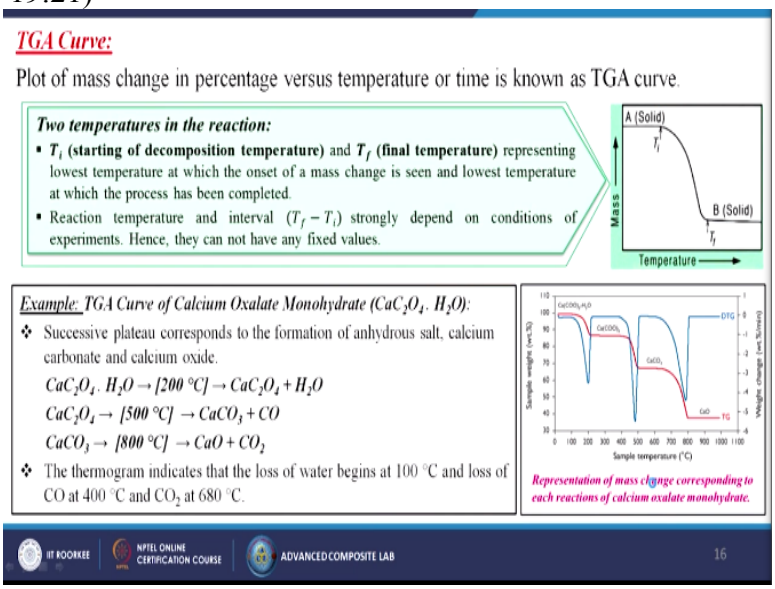

Generally the TGA curve is looks like this. So, plot of mass change in percentage versus temperature of time is known as the TGA curve. So, here in this case we are giving the mass in maybe generally into the weight percent and in this case generally we are giving the temperature in maybe ° C. So, two temperatures in the reactions in this particular case T_i that is the starting of decomposition temperature and T_f that is the final temperature.

Representing lowest temperature at which the onset of mass change is seen and lowest temperature at which the process has been completed. Reaction temperature and interval, that means $T_f - T_i$ strongly depends on conditions of experiments. Hence, they cannot have any fixed values. So, slowly I am heating the materials and the temperature is going up. So, for example: TGA curve of calcium oxalate monohydrate ($CaC_2O_4 . H_2O$): successive plateau corresponds to the formation of anhydrous salt, calcium carbonate and calcium oxide.

So, $CaC_2O_4.H2O$ if I heat up to 200 ° C it will form $CaC_2O_4+H_2O$. Then if I heat it up to 500° C then it will form the calcium carbonate + carbon monoxide and if we heat it up to 800 ° C, it will form the calcium oxide + carbon dioxide. So, in this particular case you can see that the first formation is this one then second this one, third is calcium carbonate and fourth is the calcium oxide.

So, the thermogram indicates that the loss of water begins at 100 ° C. So, 100 ° C after that we are not getting any H_2 over there and loss of carbon monoxide at 400 ° C and carbon dioxide at 680 ° C. Factors affecting the TG curve: first one is called the

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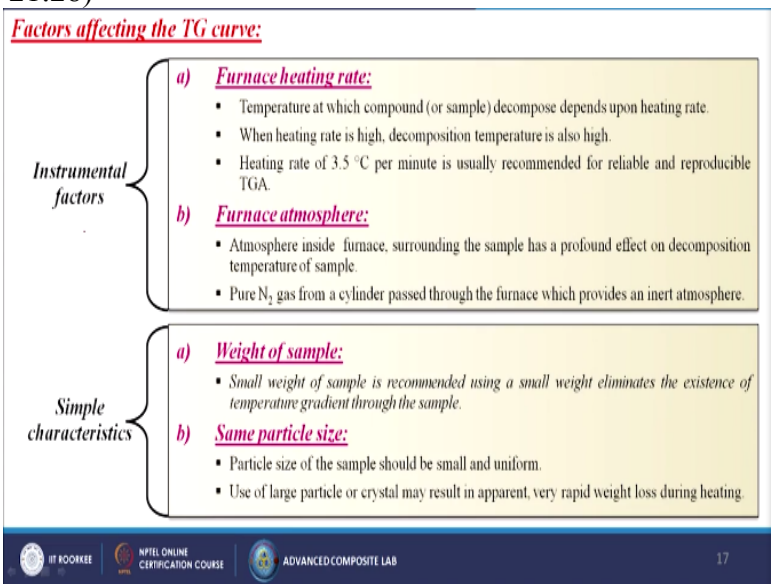

Instrumental factors, what are those? Furnace heating rate: temperature at which compound or maybe the sample decompose, depends upon heating rate. When heating rate is high, decomposition temperature is also high. Heating rate of generally 3-3.5 ° C per minute is usually recommended for reliable and reproducible TGA. Second one is called the furnace atmosphere: Atmosphere inside furnace surrounding the sample has a profound effect on decomposition temperature of sample.

Pure nitrogen gas from a cylinder passed through the furnace which provides an inert atmosphere. Simple characteristics generally weight of sample: small weight of sample is recommended using a small weight eliminates the existence of temperature gradient through the sample and same particle size: particle size of the sample should be small and uniform. Use of large particle or crystal may result in apparent, very rapid weight loss during heating.

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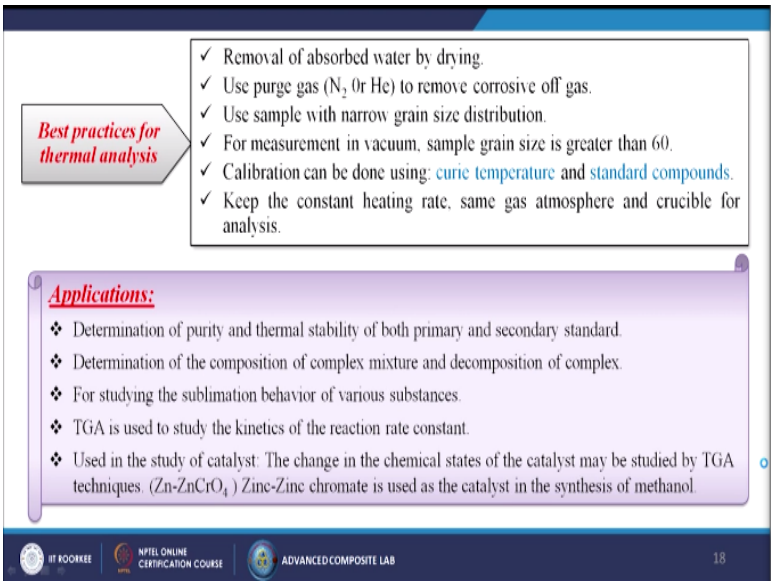

Best practice for thermal analysis, generally removal of absorbed water by drying. Use purge gas like nitrogen or maybe the helium to remove the corrosive off gas. Use sample with narrow grain size distribution. For measurement in vacuum, sample grain size is greater than 60. Calibration can be done using Curie temperature and the standard compounds. Keep the constant heating rate, same gas atmosphere and crucible for analysis.

What are the applications determination of purity and thermal stability of both primary and secondary standard determination of the composition of complex mixture and decomposition of complex for studying the sublimation behavior of various substances? TGA is used to study the kinetics of the reaction rate constant. Used in the study of catalyst, the change in the chemical states of the catalyst may be studied by the TGA techniques. That is (zinc $ZnCrO_4$) zinc and zinc chromate is used as the catalyst in the synthesis of methanol.

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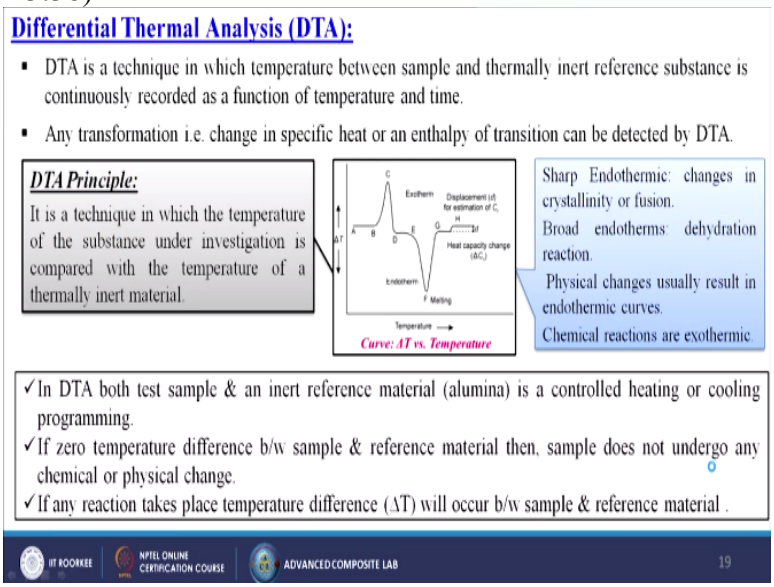

Then next one is called the differential thermal analysis or maybe the DTA. So, DTA is a technique in which temperature between sample and thermally inert reference substance is

continuously recorded as a function of temperature and time. Any transformations, that is change in specific heat or an enthalpy of transition can be detected by DTA. What is the DTA principle? It is the technique in which the temperature of the substance under investigation is compared with the temperature of a thermally inert material.

So, sharp endothermic changes in the crystallinity of fusion, so we are getting exotherm and endotherm we are getting the melting temperature and broad endotherm that is the dehydration reactions. Physical changes usually result in endothermic curves. Chemical reactions are exothermic. In DTA both test sample and inert reference material like alumina is a controlled heating or cooling programming.

If zero temperature difference between sample and reference material then sample does not undergo any chemical or physical change. If any reaction takes place, temperature difference ΔT will occur between the sample and the reference material. Next one is called the components of DTA instruments. So, first the

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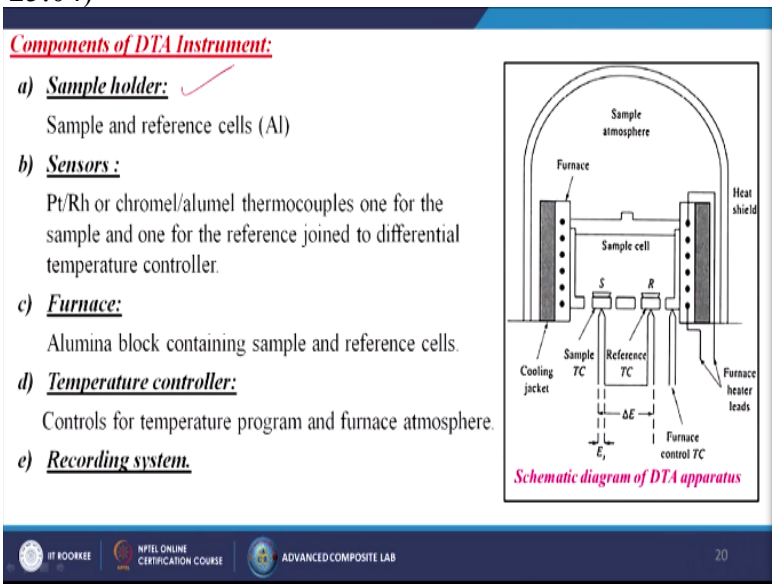

Sample holder: sample and reference cells. So, in this case this is the sample cell over there and this is the sample atmosphere we are having that furnace, we are having the cooling jacket also. So, we are putting the sample TC over here and this is the reference. So, sensors either maybe platinum or maybe the Ruthenium or maybe the chromel or maybe the alumel thermocouples one for the sample and one for the reference joined to differential temperature controller. Furnace: alumina block containing sample and reference cells. Then temperature controller: controls for temperature programs and furnace atmosphere. And last one is called the recording system.

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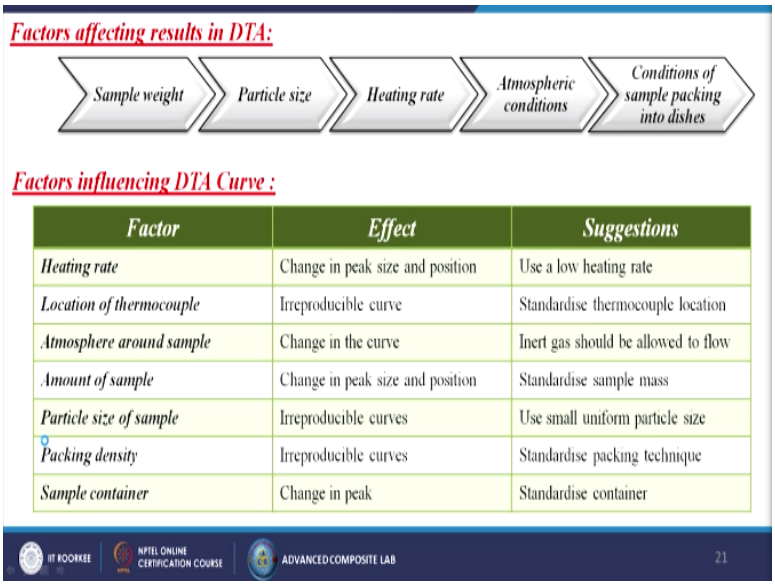

Factors affecting results in DTA: so, generally the sample weight, particle size, heating rate, atmospheric conditions and the conditions of sample packing into dishes. Factors influencing DTA curve, so generally the heating rate, effect change in peak size and positions. Suggestions use a low heating rate. Location of thermocouple, irreproducible curve, standardized thermocouple location. Atmosphere around sample, change in the curve, inert gas should be allowed to flow.

Amount of sample, change in peak size and positions, standardize sample mass. Particle size of sample irreproducible curves, use small uniform particle size. Packing density, irreproducible curves, standardize packing techniques and the sample container change in peak, standardize container.

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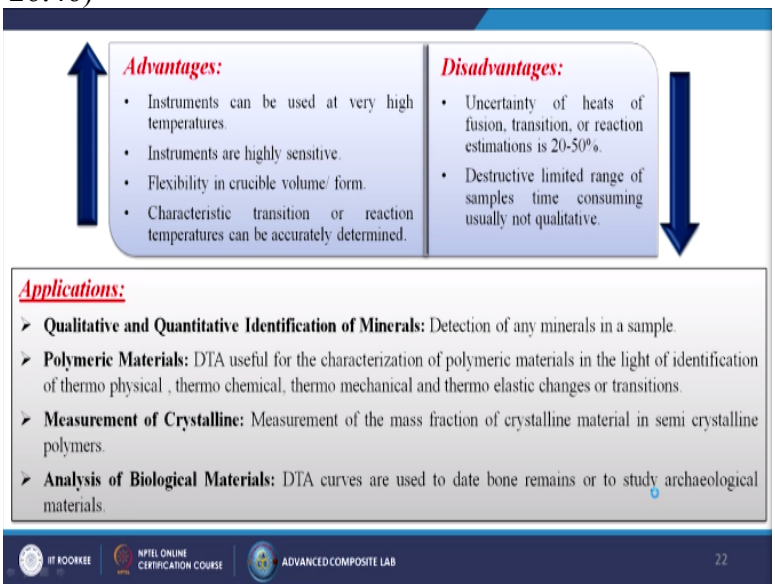

Then what are the advantages? Instruments can be used at very high temperatures. Instruments are highly sensitive. Flexibility in crucible volume or maybe the form, characteristic transition or reaction temperatures can be accurately determined. Of course there are certain disadvantages

also. What are those? Uncertainly of heats of fusion, transition or reaction estimations, is 20-50%.

Destructive limited range of samples time consuming, usually not qualitative. Applications: qualitative and quantitative identification of minerals: detection of any minerals in as sample. Polymeric materials: DTA useful for the characterization of polymeric materials in the light of identification of thermo physical, thermo chemical, thermo mechanical and thermo elastic changes of transitions.

Measurement of crystalline: measurement of the mass fraction of crystalline material in semi crystalline polymers and the last one analysis of biological materials: DTA curves are used to date bone remains or to study archaeological materials.

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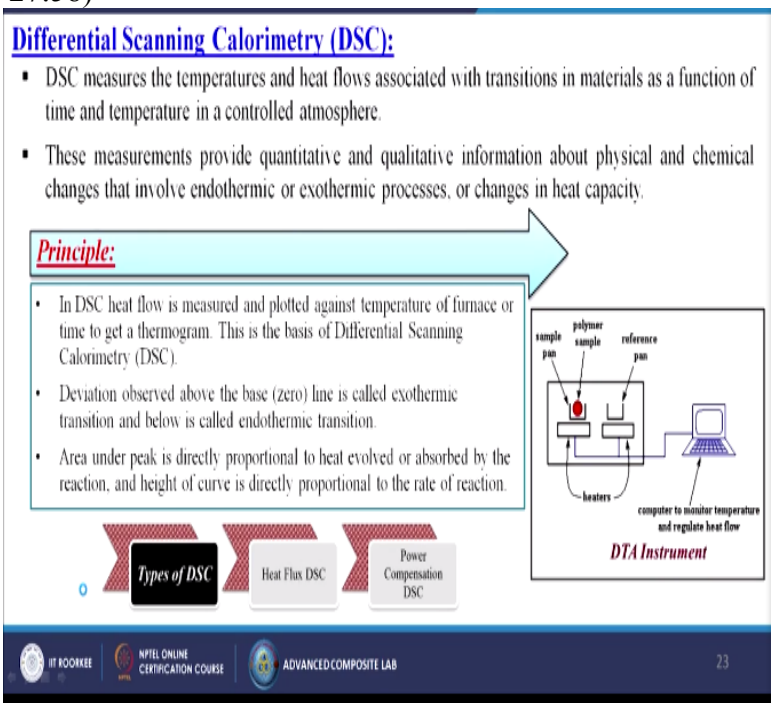

Next one is called the differential scanning calorimetry or maybe the DSC. So, DSC measures the temperatures and heat flows associated with transitions in materials as a function of time and temperature in a controlled atmosphere. These measurements provide quantitative and qualitative information about physical and chemical changes that involve endothermic or maybe the exothermic process or change in heat capacity.

So, what is the principle? In DSC heat flow is measured and plotted against temperature of furnace or time to get a thermogram. This is the basis of differential scanning calorimetry. Deviation observed above the base line is called exothermic transitions and below is called the endothermic transitions. Area under peak is directly proportional to heat evolved or absorbed by the reaction and height of curve is directly proportional to the rate of reactions. So, generally the types of DSC, one is heat flux DSC another one is called the power compensation DSC.

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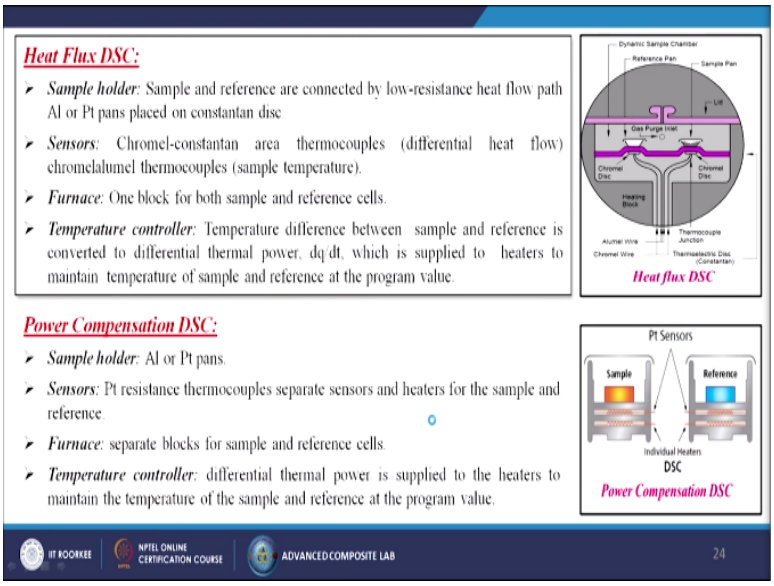

Heat flux DSC: sample holder: sample and reference are connected by low-resistance heat flow path, aluminium or maybe the platinum pans placed on constantan disc. Sensors: chromel or maybe the constantan area thermocouples differential heat flow, chromelalumel thermocouples or maybe the sample temperature. Furnace: one blocks for both sample and reference cells. Temperature controller: temperature differences between, sample and reference is converted to differential thermal power Dq by Dt which is supplied to heater to maintain temperature of sample and reference at the program value if we talk about the power compensation DSC.

So in this case sample holder it generally made by aluminum or maybe the platinum pans. Sensors: platinum resistance thermocouples separate sensors and heaters for the sample and reference. Furnace: separate block for sample and reference cell. Temperature controller: differential thermal power is supplied to the heater to maintain the temperature of the sample and reference at the program value. What are the advantages?

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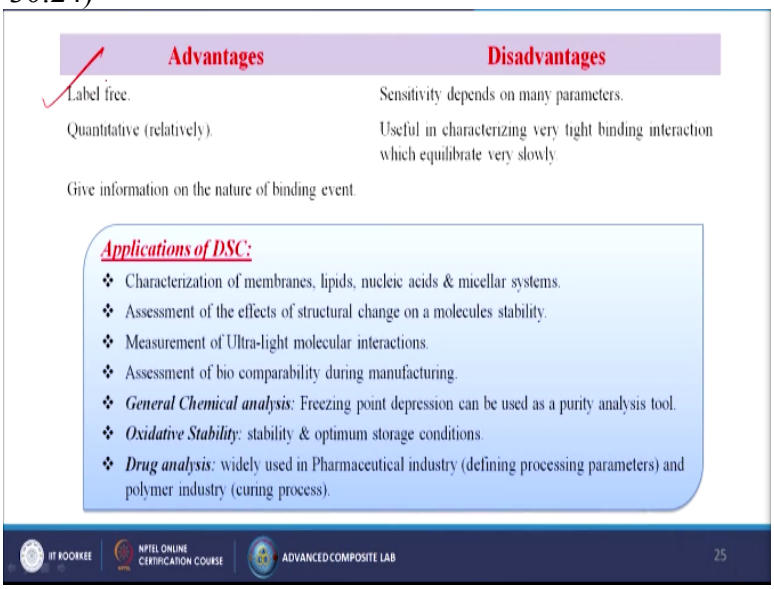

It is label free and quantitative relatively. Disadvantages sensitivity depends on many parameters useful in characterizing very tight binding interaction which equilibrate very slowly. Give information on the nature of binding event. Applications of DSC: characterization of membranes, lipids, nucleic acids or maybe the micellar systems, assessment of the effects of the structural change on a molecules stability, measurement of ultra-light molecular interactions, assessment of bio comparability during manufacturing.

General chemical analysis: freezing point depression can be used as a purity analysis tool. Oxidative stability: stability and optimum storage conditions. Drug analysis: widely used in pharmaceutical industry defining processing parameters and polymer industry that is the curing process. Then compression between TGA, DTA and DSC, so for TGA

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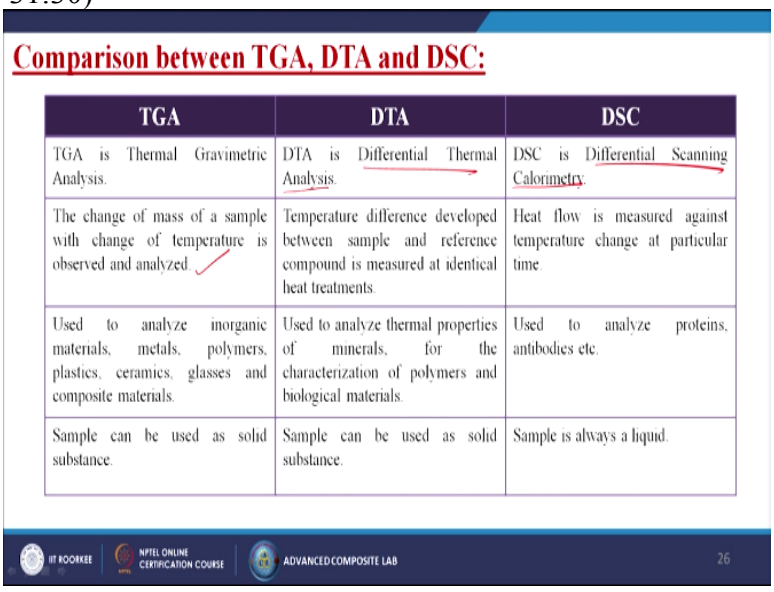

It is the thermal gravimetric analysis. DTA is the differential thermal analysis. DSC is the differential scanning calorimetry. For TGA the change of mass of a sample with change of temperature is observed and analyzed. For DTA temperature difference developed between sample and reference compound is measured at identical heat treatments. For DSC heat flow is measured against temperature change at particular time.

For TGA used to analyze inorganic materials, metals, polymers, plastics, ceramics, glasses and composite materials. DTA used to analyze thermal properties of minerals for the characterization of polymers and biological materials. DSC used to analyze proteins or maybe the antibodies. For TGA sample can be used as a solid substance. DTA sample can be used as solid substance. DSC sample is always a liquid.

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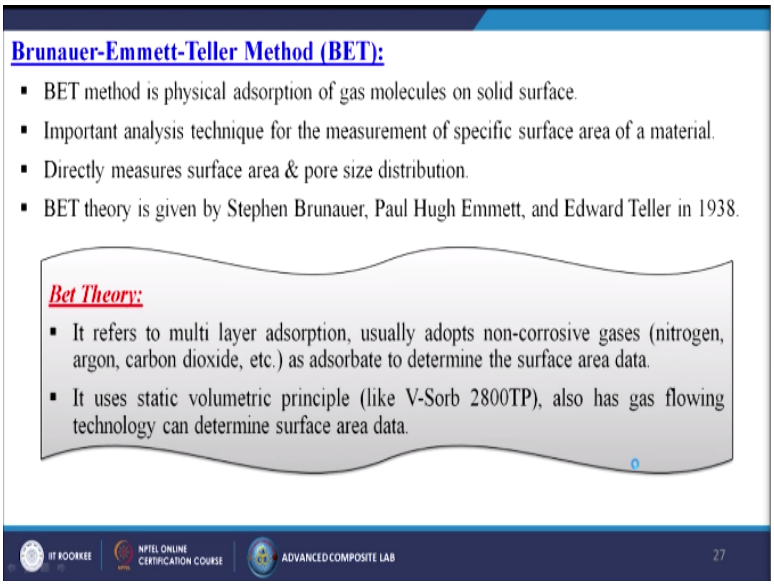

Next one is called the brunauer-Emmett-teller method or maybe the BET. So, BET method is physical absorption of gas molecules on solid surface, important analysis technique for the measurement of specific surface area of a material, directly measures surface area or maybe the pore size distribution. BET theory is given by Stephen Brunauer, Paul Hugh Emmett and Edward teller in the year of 1938. So, what is BET theory?

It refers to multi layer adsorption, usually adopts non-corrosive gases like nitrogen, argon, carbon dioxide as adsorbate to determine the surface area data. It uses static volumetric principle like V-Sorb 2800TP also has gas flowing technology can determine surface area data. What is the principle? BET is an extension of Langmuir model

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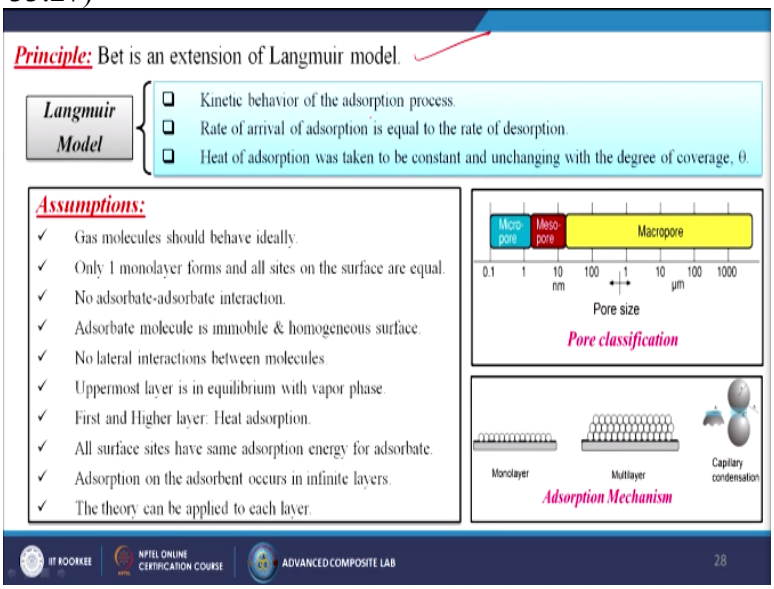

Kinetic behavior of the adsorption process, rate of arrival of adsorption is equal to the rate of adsorption rate of desorption. Heat of adsorption was taken to be constant and unchanging with the ° of coverage θ. What are the assumptions? Gas molecules should behave ideally. Only one

monolayer forms and all sites on the surface are equal. No adsorbate-adsorbate interactions. Adsorbate molecules are immobile and homogeneous surface.
No lateral interactions between molecules. Uppermost layer is in equilibrium with vapor phase. First and higher layer, heat adsorption, all surface sites have same adsorption energy for adsorbate. Adsorption on the adsorbent occurs in infinite layers. The theory can be applied to each layer. So, like this way we can get the whole classifications like micro pore which is f4rom 0.1- 1 nanometers. Mesa pore 1-10 nanometers and then we are having the macro pores from more than 10 nanometer to maybe 1000 micrometer.
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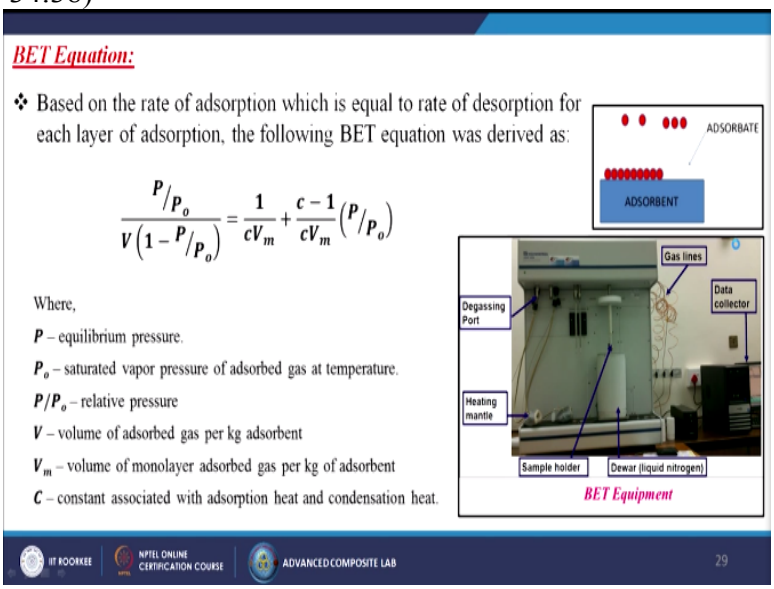

BET equations: based on the rate of adsorption which is equal to rate of desorption for each layer of adsorption, the following BET equations was derived as P/P_0/ V*1-P/P_0=$1/_cV_m$+c-$1/_cV_m$ *P/P_0. Where P is the equilibrium pressure, P_0 saturated vapor pressure of adsorbed gas at temperature, P\P_0 is the relative pressure, V is the volume of adsorbed gas per kg of adsorbent. V_m volume of monolayer adsorbed gas per kg of adsorbent.
C is the constant associated with adsorption heat and condensation heat. So, this is the, generally the BET equipment whatever we are using. Now we have come to the last, not last second last slide of this particular lecture that I have to summarize this particular lecture. So, in this particular lecture
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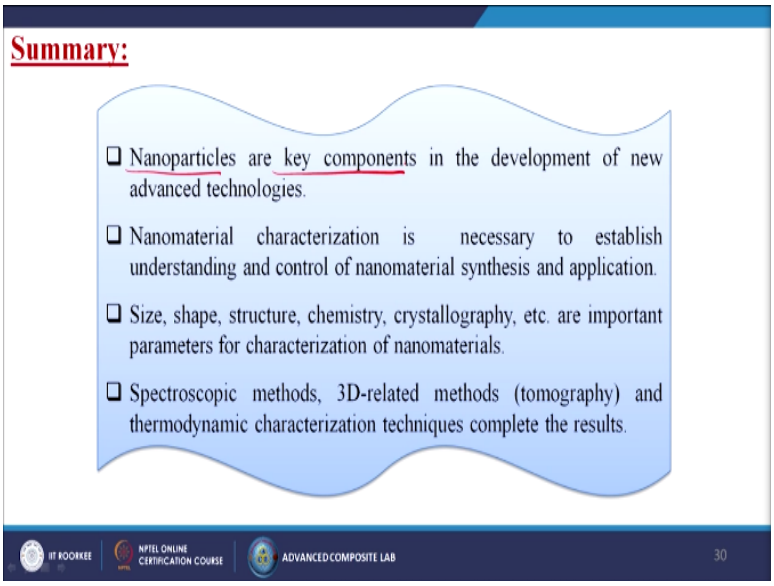

We have discussed about the nano-particles which are the key components in the development of new advanced technologies. Nano-material characterization is necessary to establish understanding and control of Nano-material synthesis and applications. Size, shape, structure, chemistry, crystallography etc are important parameters for characterization of Nano-materials. Spectroscopic methods, 3D related methods like tomography and thermodynamic characterization techniques complete the results.

So, now I am going to give a brief outline of this particular course, as this is the last lecture of this particular course analysis of Nanomaterials. So, generally the structural analysis of Nano-materials is an

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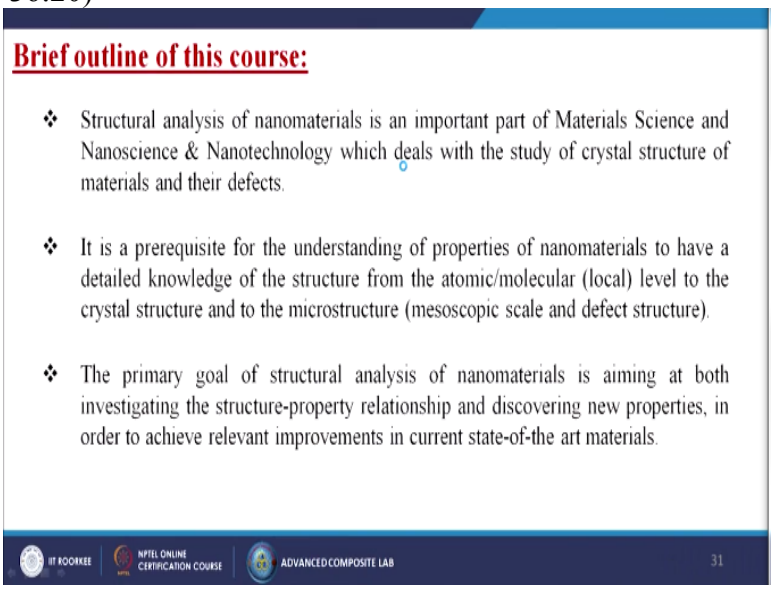

Important part of materials science and nano-science and nano-technology which deals with the study of crystal structure of materials and their defects, it is a prerequisite for the understanding of properties of Nanomaterials to have a detailed knowledge of the structure from the atomic or

maybe the molecular level to the crystal structure and to the microstructure either it is maybe the mesoscopic scale or maybe the defect structure.

The primary goal of structural analysis of Nanomaterials is aiming at both investigating the structure-property relationship and discovering new properties in order to achieve relevant improvements in current state-of-the art materials. So, this is a very useful subject for the material scientist or maybe the, those who are working for inventions of the new materials or maybe the new Nanomaterials. Thank you.

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