Modern Instrumental Methods of Analysis

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Lecture No. # 01

Introduction to the Modern Instrumental Methods of Analysis

Hello! Greetings to you. I am Doctor J. R. Mudakavi, a faculty in the chemical engineering department of Indian Institute of Science, Bangalore. I am giving a course on the modern instrumental methods of analysis. This course is designed for undergraduate and postgraduate students of chemical engineering, civil engineering, metallurgy, instrumentation and environmental engineering. The course is also useful for postgraduate students of chemistry, environmental science, botany, agricultural sciences, and material science.

In this course, I will be discussing about the modern instrumental methods of analysis useful for the determination of traits and ultra traits and quantities of substances. The course also includes topics on the characterization of materials such as thermogravimetric analysis, differential thermal analysis, x-ray techniques, EDAC, SEM, etcetera. And, chromatographic techniques such as iron chromatography, gas chromatography, high pressure liquid chromatography, etcetera. The course consists of about forty lectures. At the end of the course, you will be able to feel the familiarity with various instrumental methods; and, you will be able to decide on the appropriate modern instrumental methods of methods of analysis to carry out analytical determinations. You will also be able to decide on the determination of technique – what you want to use for the instruments for the method of analysis.

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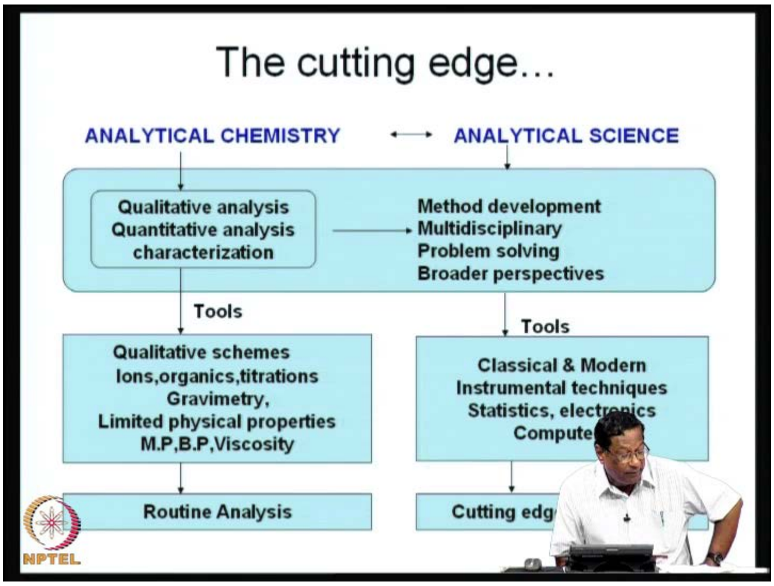

Traditionally, the study of chemistry refers to the composition structure, physical properties and reactivity of the matter. For convenience, it is divided into organic chemistry, inorganic chemistry, physical chemistry, biochemical and analytical sciences. This division nowadays is approximately only of academic interest as several interdisciplinary areas, such as organ metallic chemistry, environmental chemistry, climate science, bioanalytical chemistry, etcetera have developed over the years and are providing a unique perspective to the study of chemistry.

Tremendous developments and advances in these areas including peripheral technologies, such as electronics, computers, sensors, optics and mechanical engineering have necessitated a revolutionary change of perception on the understanding of chemistry and analytical chemistry in particular. This brings us to the question what is analytical chemistry? As you can see in this (Refer Slide Time: 03:39) slide, qualitative analytical chemistry and analytical sciences are slightly different in their approach to the problem. For example, in the slide, you can see that qualitative analysis, quantitative analysis characterization is in the domain of analytical chemistry. The tools they employ are qualitative schemes for ions, organics, titrations, gravimetry and some limited physical properties, such as melting point, boiling point, viscosity, etcetera. These are all things coming in the domain routine analysis; whereas, analytical science comes in the other area, such as method development and multidisciplinary. You can see that we have the interaction coming from analytical chemistry also and then problem solving. And, analytical science as such has got broader perspectives. So, the tools what we employ for analytical science are classical and modern instrumental methods of analysis, statistics, electronics and computers. Cutting edge technology also is the domain of analytical sciences.

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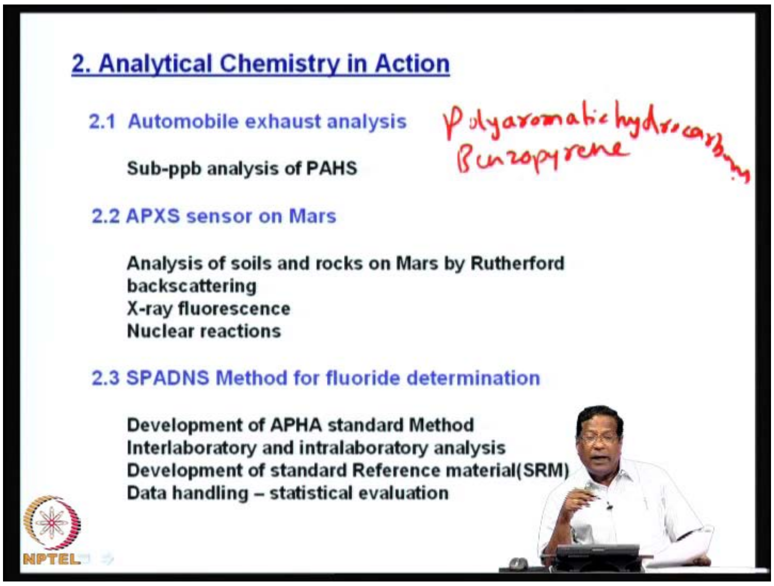

Analytical chemistry deals with the identification, characterization and measurement of chemical species present in a sample. It also deals with the characterization of a sample through measurement of its physical properties, such as viscosity and melting point, boiling point, crystal structure of a mineral, particle size measurement, etcetera; distribution of a pesticide and their degradation products on agricultural lands, etcetera. These are some of the problems associated with the analytical science, where analytical scientists are called upon to contribute in different areas. For example, if you want to determine the trace elements and gases in the stratosphere, moon, planets, interstellar space and characterization of paints in old priceless paintings, traces of chlorofluro carbons in the environment, what we have to do is just to learn analytical science. Then, we will be in a position to contribute to the solution of all these problems, which I had already stated.

The scope of modern analytical chemistry is thus extremely broad and it continues to expand as society and technology impose new demands on it. To cope with such demands, analytical chemists are expected to keep abreast of new developments in several other scientific disciplines and technologies including physics, mathematics, engineering, optics, electrical, mechanical, computer and communication engineering, etcetera. Therefore, it may not be appropriate if we call analytical chemistry as analytical science.

Analytical science is basically a dynamic changing field. It is called upon to solve several kinds of problems mentioned above with the technology that constantly provides new measurement tools. For example, the challenges extend from the identification and measurement of parts per billion levels of impurities in the environmental contaminants, such as water, air, soil, semiconductors, etcetera. And, metal ions in bloodstreams in the hospital patients or as indicators of health or specific disorders, drug metabolites in human body parts, and several other related problems associated with modern day living.

Take for example, analytical challenges presented by automobile exhaust analysis. For example, you take a look at this (Refer Slide Time: 08:52). In general, more than 150 polyaromatic compounds are detected in petrol and diesel engine emissions. Almost all the carcinogenic effect of the exhaust condensate comes from the polyaromatic hydrocarbons containing 4 to 7 rings, which is the only 4 percent of the total weight of the condensates. Benzopyrene alone (Refer Slide Time: 09:27) accounts for 4 percent of the total weight of the condensates and about 10 percent of the condensates containing carbon monoxide; 2 to 5 percent also sometimes. And, other things constitute the remaining other PAHS – what we call polyaromatic hydrocarbons (Refer Slide Time: 10:08). Out of this, 4 percent alone is benzopyrene; whereas, 10 percent is the total polyaromatic hydrocarbons.

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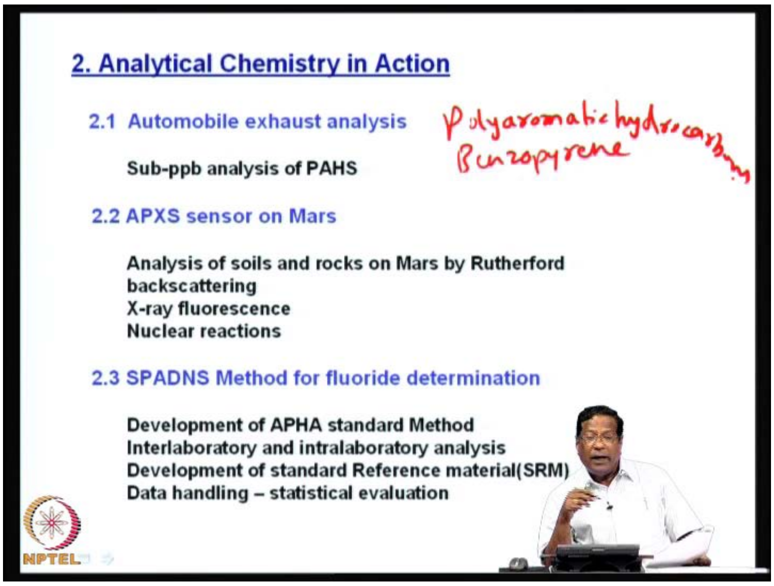

Now, you can imagine for testing the automobile exhaust gases on experimental animals for example, it provides the best stimulation model on the animals. However, this approach is not practical, because basically, 2 to 5 percent of the carbon monoxide already present in the exhaust itself is highly carcinogenic and highly poisonous for direct inhalation. Animals can survive only if the exhaust is considerably diluted to reduce the carbon monoxide concentration to acceptable levels. But, in this process, other polyaromatic hydrocarbons, which are present only in about 4 percent, would also get diluted to still lower levels. Therefore, the problem is dilution results in the concentration of all other pollutants also that do not… And, they results in concentrations that do not provoke carcinogenic effect in the respiratory tracts of the animals; nor, would they be deductable in highly diluted samples even by the most sensitive techniques.

Since many diseases become clinically evident only after chronic exposure over several years, current environmental assessment of several organic and inorganic compounds are of limited value. Moreover, the actual dose absorbed by a person or an animal is dependent upon its age, its genetic factors, then health of the animal, height, weight and several other factors to such an extent that even when the level of the pollutant is known, it is hard to pinpoint the actual amount received by a particular individual. It is still harder to determine the exact role of each one of the chemical constituents. Herein lies the challenge to analytical scientist, which has been overcome by the ingenuity of the scientist in developing subparts per billion level analysis and parts per trillion level analysis also. And, for this, the chromatographic techniques had to be developed, which would separate the components in parts per billion levels; and, the detectors should be developed for the determination of 10 raised to minus 12, that is, picogram level concentrations of the separated components. Herein lies the challenge to the analytical scientist.

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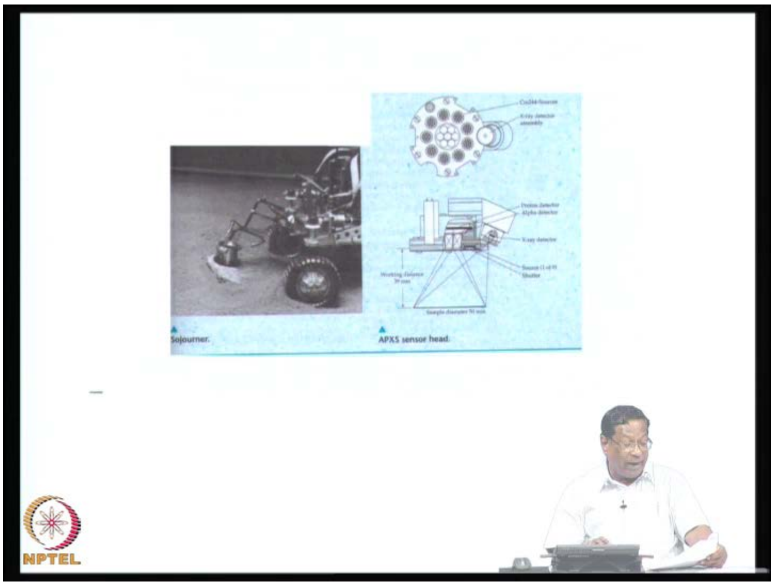

Another challenge successfully overcome by the analytical scientist is by the development of APXS sensor head fixed on the path finder microrover of the moon mission. In July 1977, the moon mission carried out photographic surveys and chemical analysis of the rocks on the martian soil. The chemical analysis was obtained by an alphatron x-ray spectrometer (APXS) – it is here, which provided the complete and the detailed analysis of the chemical elements on the martian soil and rocks near the landing site.

Now, the spectrometer consisted of a sensor head what you see here (Refer Slide Time: 14:36) – mounted on a movable arm, that is, here – on a movable arm, which could be placed on the soil rock samples like this. The APXS sensors spectrometer uses 244 curium as a source; and, when these radiations hit the surface, three types of interactions occur. These include Rutherford backscattering, x-ray fluorescents and nuclear reactions. The three spectrometers are individually inadequate, but highly complementary to each other when used in conjunction together. For example, Rutherford backscattering is useful for light elements, such as carbon, oxygen and other things; while, proton emission spectrometer mainly sensitive to sodium, magnesium, aluminum, silicon, sulphur, etcetera, which are the natural constituents of the soils and rocks. And, x-ray emission spectrometer is more sensitive to heavier elements. A combination of all these three spectrometers enables the quantities of all these elements except hydrogen and helium when their concentrations are greater than 0.2 percent weight by weight.

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A number of such path breaking examples can be given wherein analytical scientists have successfully overcome the challenges imposed by time, space and accessibility constraints; and then, irrespective of their specialization, whether it is in analytical science or in physical science or in chemical sciences and any of these things. In fact, desperate sciences, such as archeology, anthropology, botany, engineering sciences, forensic sciences, geology, material science, medicine, molecular biology, pharmacology, toxicology and the list keeps on increasing. Such sciences are all highly dependent upon the chemical analysis, because they need to answer the questions regarding the content of the materials they work with.

Now, what do analytical scientist do? In essence, an analytical scientist determines the quantity of a substance in a sample. This is called quantitative analysis. Working in this area includes much more than the familiarity with various methods of analysis. For example, it involves the evaluation of problems associated with the proper acquisition of samples, selection of the analytical methodology, consideration of the possible interfering species present in the sample matrix, data evaluation using statistics based on sound interpretation and the reporting. One should also follow good laboratory practices known as GLP to get the results, which are in good agreement with the modern practices, so that when you are running an experiment, you should not be having difficulty with respect to hazardous nature of the chemicals what you are handling.

If an analytical chemist does only a quantitative analysis, then probably, there is no reason to classify the analytical science as analytical chemistry. After all, every scientist worth is sort does the chemical analysis of some sort or the other either during his college days or in the high school days or in subsequently in his working field. For example, qualitative schemes and for identifying inorganic ions, organic substances and then acid-based titrations, redox reactions, complexation reactions, titrations, etcetera – they are all part of the college chemistry.

Unfortunately, this description of an analytical scientist ignores the unique perspective that an analytical scientist brings to the study of chemistry. Performing a routine analysis on a routine sample needs only a chemist; it does not need an analytical scientist, because everything is there, procedure is there, chemicals are there and you are there to run the analysis; you get the result. But, what does an analytical scientist do? He brings his expertise. And, an analytical scientist therefore does much more; and, he brings into it an establishing method, extending existing methods for newer types of samples and developing new methods for measuring chemical phenomena.

According to Professor C. N. Reilly, who was a professor of chemistry at the university of North Carolina at Chappel Hill in 1977 – what he said is analytical scientist develops new methods for measuring chemical phenomena. And, an example of the distinction between analytical chemistry and analytical chemical analysis is given here. For example, just think about a geologist. What does a geologist do? A geologist does analysis of an ore. His job is to find out how much of ore is there in a given hill. And, he keeps on taking samples from that surface of the hill at different depths.

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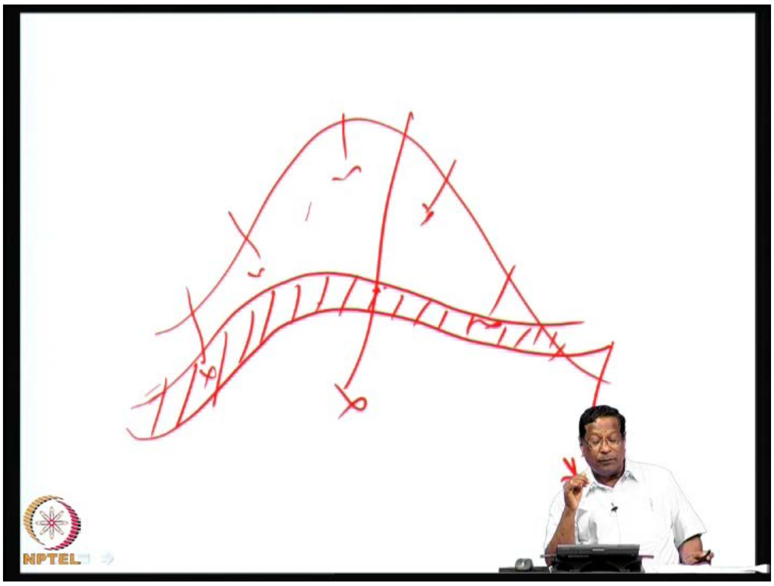

Now, you can imagine a hill like this. Now, the sample – one may be here; one may be here; one may be here; one may be here, etcetera. And, see the problem is the ore may go like this – ore may be concentrated in an area like this; that means, you are interested only in the samples drawn in this area. This is where the value of the ore lies.

Now, a geologist – he is interested only in such a path that is known as way. And therefore, what they do is they take random sample at different depths like this (Refer Slide Time: 21:48). And then, you are supposed to estimate the element in different samples. Therefore, the challenge of developing and validating a method to be analyzed for providing the right kind of information is analytical chemist's responsibility. Now, because we are interested only in this region and the range is very small, it is not visible to the outside naked eye; it is under the surface. And, all these challenges need to be solved by an analytical chemist, but not by a geological scientist. Similarly, we can give number of examples, where an analytical scientist works on different areas related to the development of methods.

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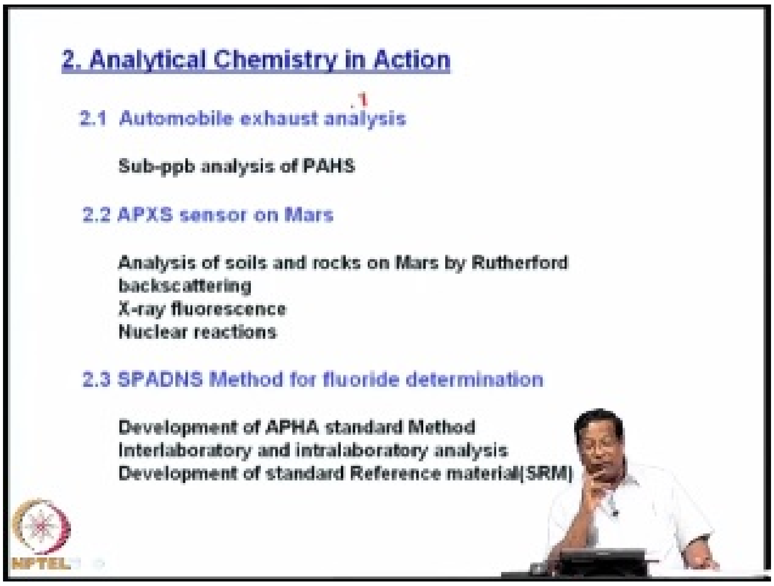

Now, let us go back to the slide what we have shown earlier. Let us take a look at this SPADNS method for fluoride determination. Development of the method for fluoride – you are all aware that fluoride is implicated in number of health related problems; there is fluoride in the drinking water, fluoride in the lakes, fluoride in the rivers, fluoride in your toothpaste and fluoride in several others commercial samples in what you use for talcum powder, etcetera. And, all these things have necessitated that a substance like fluoride needs to be monitored in various kinds of samples; and, we need a dependable method, because one characteristic of a chemical analysis is wherever one follows the method, the result should be the same whether it is in Bangalore or Bombay or Madras or Timbuktu or America or Russia, anywhere. Wherever it is there, an analytical method must give the same result for the same sample; then only, it is considered accurate. Therefore, we need a method, which is acceptable all over the world.

However, before this method – SPADNS method was accepted as a standard method of analysis, APHA, that is, American Public Health Association analyzed a number of standard samples, natural samples spiked with fluoride samples in a number of laboratories spread all over the world, collected their analysis, collated the data, determined the bias of the results and standard deviation, relative error, percent error, etcetera, and ensured that the method works for various types of samples. Then only, it was accepted and published as a standard method of analysis. This process – you will not believe, it has taken a number of years before it was accepted as a standard method. And, American Public Health Association has undertaken most of the responsibility for all the parameters involved in the examination of water and waste water. This is a shining example of how a simple analysis like fluoride should be adopted for the chemical analysis in different substances, different matrices and different circumstances, so that the results can be correlated anywhere in the world. That is the challenge of analytical scientists.

I will give you another example. In 1905, dimethylglyoxime was first used for the analysis of nickel and palladium till 1970. Remember, from 1905 till 1970, this method was employed for the determination of nickel when it was replaced by flame analysis, atomic absorption. Till recently, this method was employed until inductively coupled plasma atomic absorption spectrometry is currently the choice of analysis. That has resulted in the considerable savings of time and cost. A careful look into the examples cited so far, in fact, there are quite a few more. But, due to fear of digressing and becoming too lengthy, I am shortening the number of examples. But, the fact remains that the examples what I have given exemplify the fact that the scope of analytical chemistry needs to be redefined.

Probably, a most acceptable definition of analytical science could be the science of inventing and applying the concepts, principles and drawing up of strategies for measuring the characteristics of chemical systems and species. And, analytical chemistry usually operates at the extreme edges of the determination levels let us say; that means, either the sample could be very small, so that it does not permit you to reduce; that it is difficult to get another sample. For example, if somebody wants to analyze the body fluid, he cannot take 100 ml of the body fluid and do the analysis. You will get only 5 ml, maybe 1 ml or 2 ml on which samples needs to be analyzed for tricky chemicals. And, that is the challenge.

The science of the inventing concepts – you need to invent a concept how to do this analysis. Usually, the challenge may be in finding out the quantity of a substance, which means in a sense it is like finding a needle in the hay stack. But, an analytical scientist is challenged to find how many needles are there in the hay stack, instead of just needles. So, that is the challenge of an analytical scientist. An analytical chemist usually… That is why I keep on saying that they operate at the extreme edges of the detection level. It may be extreme edges of the sample detection or in the instrumental detection level or it may be in the conversion of the concentration and reporting the standard deviation at the extreme edges. That is what it makes the analytical science as most exacting and exciting also to the people who work on such problems. There are number of problems, number of samples, complex samples, shorter time intervals and on species present at lower concentration in a variety of matrices fostering multidisciplinary result at the cutting edge of the science; never forget that word – cutting edge of the science is where the challenge lies. This does not mean that the traditional methods are given a go-by; no, not at all.

Traditional methods of routine analysis are still valued and required in special circumstances. For example, no jeweler would accept analytical results of gold and platinum until he sees the gold; otherwise, he cannot value a substance. When you want to sell your gold, he has to see the gold. Similarly, in an ore, the gold sample must be seen to appreciate its economic value. Therefore, at the same time, traditional methods are being valued, being used, but we should be open to newer methods of analysis, such as modern instrumental methods of analysis, which this course is all about.

An essential feature of such an adaption is to develop an analytical perspective. Now, what is analytical perspective? An analytical perspective is approximately equivalent to analytical approach to solving problems; while, the exact definition of the analytical approach varies from person to person; every scientist can have a different definition of the analytical perspective. But, an analytical perspective does consist of some basic steps.

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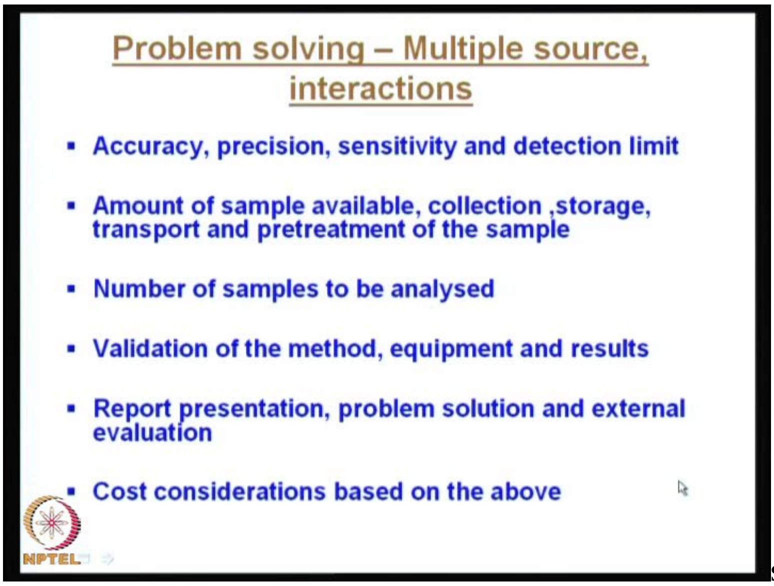

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The analytical perspective involves some basic steps. For example, identification and defining the problem; that is one part. Then, design and experimental procedure including sampling, pretreatment and chemical analysis; then, you have to carry out the experiment and gather the data. Next part is analysis of the collected experimental data; then finally, presenting the solution to the problem. So, an analytical approach contains these five steps; and, steps 1 and 5 for example, identifying the problem involves different kinds of people with whom an analytical scientist interacts.

For example, suppose there is a sportsman; he has taken steroids; he have got gold medal. Now, you want to know whether a sportsman has taken a steroid or not. Now, who comes to the analytical scientist? A sports official; for example, the Olympic sports official or somebody comes to IISc; calls me Doctor Mudakavi, I have a sportsman here; he has won a gold medal, but I want the analysis of his urine, so that we can find out whether he has taken steroids or not, so that we can decide whether to give him a medal or not. Now, the problem is and me Doctor Mudakavi as an analytical scientist, I have to give him the results within half a minute or something like that; run the analysis and tell the sportsman that yes, he has taken or he has not taken, because after winning the sports event, sportsman will not wait indefinitely to get his medal. So, all these things we need to interact with people, who are having the problem; and, they are not the specialist in analytical science; whereas, we are the specialist. So, the problem is like this.

Now, there are several problems like this; steps 2, 3 and 4, for example, what I said about carry out the experiment and the experimental data, presenting the analytical data – all these things an analytical chemist will do. But, when you present the data, you are going to present it to the person – sports official; he does not want to know how you have done the analysis, but he wants to know that what you have analyzed is a presentable form, so that he can understand – yes, he has taken or not taken; he is guilty or not guilty. So, an analytical scientist is supposed to solve problem of variety of types. And, some of them I can tell you; very simple things, but very difficult to imagine. For example, this I have already told you about the sportsman having taking steroids; then, in Kerala, there are people who are using pesticides Endosulfan. And, there are several NGOs and government officials and world health organization – all these people want to analyze Endosulfan in pregnant ladies, boys, children and old people, etcetera how much Endosulfan they have consumed when they eat cashew nuts and other foods, because they are all sprayed all over the soil and it has already gone into their bodies. Now, this needs the chemical analysis of Endosulfan in the soil, in the water, in the air, and in the body fluids, and in vegetables, and in fishes, and in chicken, etcetera. That is how the challenge becomes complicated.

Now, similarly, you want to think about chemical warfare. In chemical warfare, what happens? Suddenly, some chemical is sprayed in chemical warfare and you want to know what it is; otherwise, you will not survive. Therefore, the challenge of finding the chemicals used in warfare is again another cutting edge technology that has to be found out before the enemy is ready to launch, so that we can survive the attack when the attack comes. Similarly, developing multi-element miniaturized sensors for real time, air quality monitoring – all these things are there. And, such solving problems analytical chemist does not solve on his own; he has to interact with people, who are associated with several disciplines. And, collaboration between an analytical chemist and the persons interested in solving the problem is mandatory. So, in the first case, they interact with officials, organizations and persons, who have to deal with the problem. For example, just imagine, very recently, they had an oil spill in Bombay; and, that oil spill port authorities have to handle. So, the port authority does not know what oil it is, how much has been spilled, how much is to be analyzed. And, all these things an analytical scientist has to interact with port authorities and find out, go through the steps what I have stated earlier.

Now, if this is all the thing, analytical chemist is not needed. An analytical chemist has to bring his own expertise in solving the problem. For example, look at this slide (Refer Slide Time: 38:20). What is required? Accuracy, precision – if the your result is not accurate, nobody is going to believe you; if the result is not precise, you say it may be 10 to 20 percent; nobody is liking; nobody will like it. And, sensitivity – it must be deductible in very small quantities. And, the detection limit of the method – that is important. Amount of sample available, collection – now, who will do the collection? analytical scientist or that port authority?

Now, I will never believe a sampling done by somebody without my presence. For example, if I ask somebody to take a water sample from a river, how do I know that he goes to the river and collects, instead of just getting it from the well in his house or from the tap in his house? It is crazy. So, I have to be there when he takes the sample. Now, assume that he goes to the river. Now, where does he take the sample? Near the edge or near the middle or near another edge or in the middle or at what depth? Now, how do I know that the depth of the river is exactly, where the sample is representative? Problem. So, this is where an analytical scientist brings his expertise. He says find out what is the average depth of the river and go there; and probably, there is a road bridge; and, take a sample from the road bridge somewhere in the middle of the stream, so that you get a representative sample.

Now, the analytical chemist also has to deal with how many samples… Third one – number of samples to be… How many number of samples to be analyzed? See theoretically, you will never get a correct value. So, you will get most appropriate value. Now, this most appropriate value will be accurate the more number of samples you do. Now, you cannot keep on doing hundred and thousands of sample, same analysis, because it is question of waste time, money, cost; somebody has to pay for that. Now, validation of the method, equipment and results, etcetera are also important. Report presentation, problem solution and external evaluation – this is another problem. Cost considerations based on the above are also important now.

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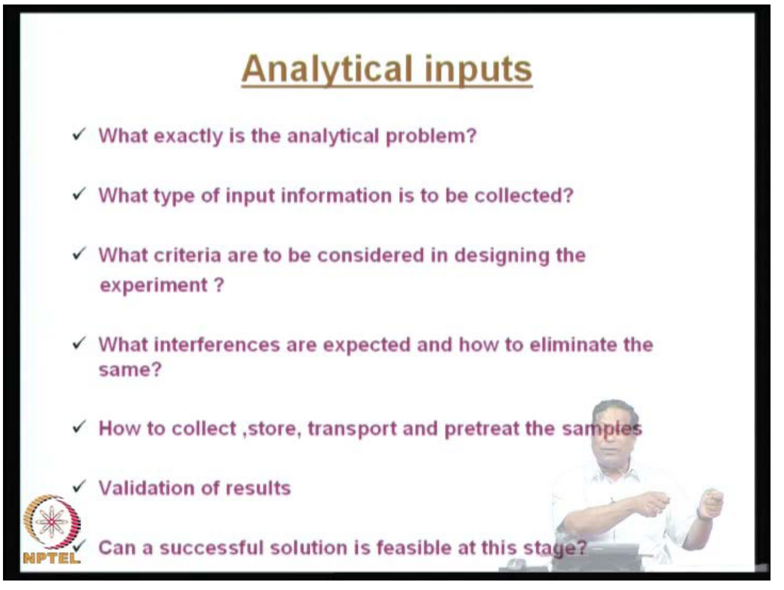

Now, apart from this, an analytical scientist brings his own expertise in these areas. For example, what exactly is the analytical problem? What type of input information is to be collected? This analytical scientist will tell. He will say like what I told – go to the river; go to the centre of the river; go to the depth of the river; check the river and then collect the sample. What criteria are to be considered in designing an experiment? What type of interferences are expected and how to eliminate the same? How to collect a sample, store transport and pretreat the sample? You cannot have a gas sample analysis and bring it in a bladder; it may leak. When you bring the sample to the lab, there may be nothing left. So, you have to make sure that how the sample is transported. And, validation of the results – if you do not validate, no amount of analysis makes any sense. Can a successful solution is feasible at this stage?

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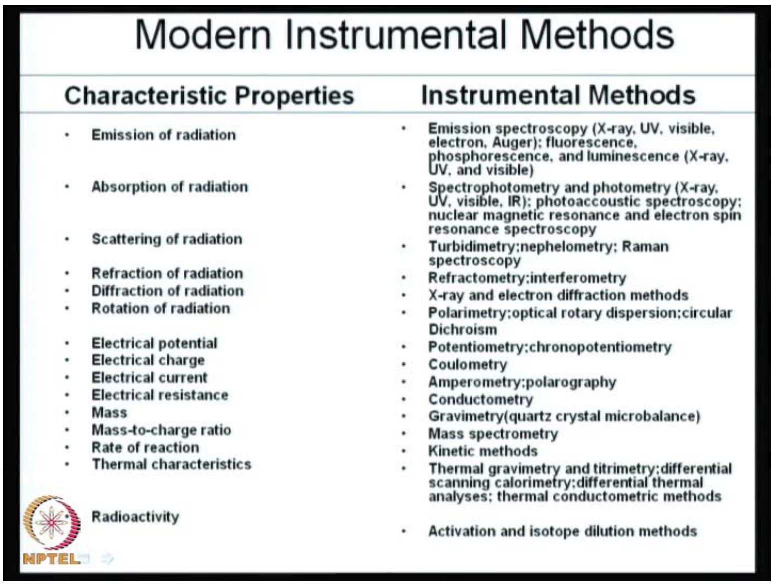

Now, I wanted to give you some inputs regarding the definitions. But, in general, what I want to do now is, I want to list some of the methods, what we analytical scientist employ. So, as outlined earlier, a majority of the problems on which analytical chemist work ultimately involve a qualitative or a quantitative analysis – characterization of a sample or a development of new method. Much of the quantitative analysis is still taught and performed are based on classical methods. Some quantitative test on chemical reactions are also performed in the same manner based on chemical reactions. However, most of the qualitative tests being performed nowadays are based on instrumental methods of analysis.

Now, without much this thing, there are two types of chemical analysis: one is classical; another is modern instrumental. Classical – you have already learnt or you would be learning anytime in your course now. The modern instrumental methods of analysis give you the same kind of information at better accuracy and good quality. Now, here are some… In the classical (Refer Slide Time: 43:30) methods, most analysis were carried out by separating the analyte from the sample either by precipitation, extraction or distillation and subsequently treated with reagents, which would give you a measurable quantity either by titration or by weighing or something like that; or, some analysis could be confirmed by checking the color of the reactions, melting point, boiling point, a Polymetric surfaces, etcetera. In gravimetry, you would make the analyte undergo a chemical reaction and then weigh the substance, which you will see just like what I told you in jewelers.

Now, even though classical methods are for the separation and determination of analytes are still useful and quite often employed, the extent of their application is decreasing with the passage of time and advent of instrumental methods of analysis, because it saves you the time, it saves the cost, it saves you several other things. Now, what are the instrumental methods? Early in the twentieth century, scientists began to experiment with physical properties of materials for solving analytical problems. The advent of modern atomic theory and discovery of fundamental particles and associated phenomena, such as light absorption or emission, charge to mass ratio, fluorescence, electrode potentials, conductivity, etcetera – all provided an impetus to the development of instrumental methods of analysis. Chromatographic and electrophoretic techniques have replaced the traditional distillation, extraction, precipitation etcetera. Collectively, these methods are known as modern instrumental methods of analysis. Now, they have become extremely reliable. And, simple instrumentation and development of electronics and application of microprocessors, computers and other things have revolutionized the analytical science.

This table (Refer Slide Time: 45:32) shows you some of the characteristic property on which instrumental methods have been developed. For example, emission of radiation – we have X-ray, UV-visible, Auger, fluorescence, phosphorescence, luminescence, etcetera. Absorption of radiation – we have Spectrophotometry, photoaccoustic spectroscopy, nuclear magnetic resonance and electron spin resonance spectroscopy. Scattering of radiation – turbidimetry, nephelometry and Raman spectroscopy. We have refraction of radiation – two instrumental methods are based on this; that is, refractometry and interferometry. Diffraction – X-ray. And, rotation of radiation – that is, polarimetry. Electrical potential – potentiometry, chronopotentiometry. Electrical charge corresponds to coulometry. Electrical current – amperometry. Electrical resistance is conductometry.

Suppose you want to do mass to charge ratio, we have a mass spectrometer, which will tell you what kind of fragments are coming out of a chemical reaction. And then, rate of reaction has lead to the development of kinetic methods. Thermal characteristics – thermal gravimetry, titrimetry and calorimetry, differential thermal analysis, etcetera.

Radioactivity – of course, everyone knows that activation and isotope dilution methods are there. So, since so many of the modern instrumental methods of analysis involve atomic properties and interaction of electromagnetic radiation, etcetera, let us study the structure of the atom and nucleus, so that we can have a better grasp of the principles and other procedural methods that are involved with the instrumental methods of analysis. That we will continue in the next class.

Thank you very much.