

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) for Pollution Monitoring

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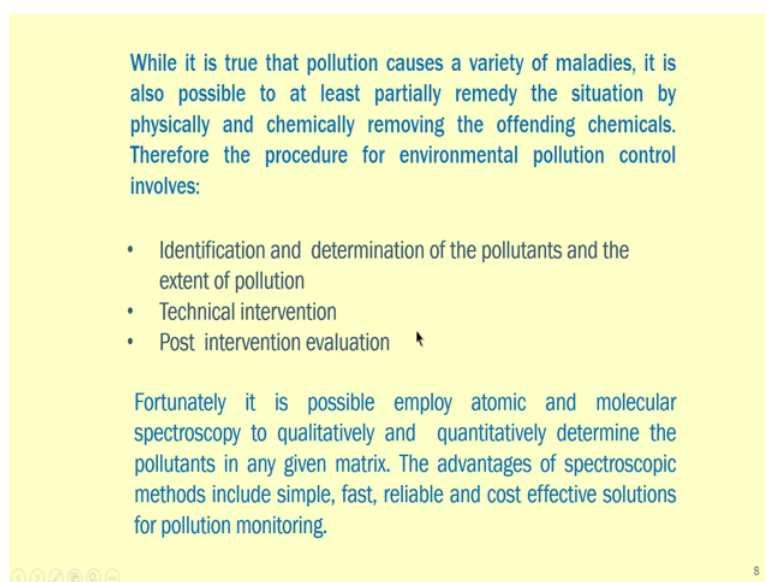
**Department of Chemical Engineering
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Lecture – 02

Course introduction and atomic structure – II

So, in the previous session we have been talking about the analytical science and pollution and we said that the procedure for environmental pollution control involves identification and determination of the pollutants and extent of pollution.

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While it is true that pollution causes a variety of maladies, it is also possible to at least partially remedy the situation by physically and chemically removing the offending chemicals. Therefore the procedure for environmental pollution control involves:

- Identification and determination of the pollutants and the extent of pollution
- Technical intervention
- Post intervention evaluation

Fortunately it is possible employ atomic and molecular spectroscopy to qualitatively and quantitatively determine the pollutants in any given matrix. The advantages of spectroscopic methods include simple, fast, reliable and cost effective solutions for pollution monitoring.

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Second is technical intervention to remedy the situation and post intervention, we need to evaluate. So, in all these things and it is important for us to analyze the chemicals that are responsible for the pollution monitor for pollution.

So, fortunately it is possible for us to employee spectroscopy and to address most of these problems and why spectroscopy means because it is simple, fast, reliable and gives us a cost effective solution for pollution monitoring. Now, in all these activities you need to practice a science known as analytical science and with when it is related to pollution control, we call it environmental analytical science. that is, it aims at developing the methodologies for specific chemical analysis, identification and quantification.

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Environmental analytical Science aims at developing methodologies, instrumentation and/or mathematical correlations and models that predict the environmental fate of new and existing chemical compounds. It presents in a concise form the most important properties relating to the chemical reactions and the amount of substances present. A thorough knowledge of the environmental analytical chemistry greatly helps to measure the extent of environmental pollution monitoring which in turn can be adopted to pollution control .

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And it also involves the learning about the instrumentation or mathematical correlations, algorithms and modeling and the models what do they do? Models will predict the environmental fate of new and existing chemicals in the environment whether it is water air or land whatever it is. It presents in a concise form the most important properties relating to the chemical reactions and the amount of substances present in the environment, a thorough knowledge of the environmental analytical chemistry.

It greatly helps to measure the extent of environmental pollution as well as monitoring which in turn can be adopted for pollution control so.

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Spectrophotometry is one of the earliest instrumental method of analysis. A majority of the chemical compounds are colorless and hence not amenable for spectrophotometry. Fortunately it is possible to convert them into colored compounds by reacting them with suitable chromophores. As of now more than 1,00,000 compounds can be made to undergo chemical reactions to produce colored compounds on an average more than 30 million spectrophotometric measurements are made daily worldwide. Another advantage of spectrophotometry is that the procedures can be easily automated with little or no manual intervention .

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One of the earliest technique spectroscopic technique are to be adopted for pollution monitoring was spectrophotometry, about spectrophotometry I have talked about it in my first mooc course, you can still go through that and if necessary you can do that and it is available online and what does a spectrophotometer do? A majority of the chemical compounds are colorless and hence not amenable for spectrophotometry.

So, what does it mean? It means spectrophotometry measures the color of the samples, it measures the it quantifies concentration and colors, if the substance is colored you just measure the color and say how much of it is there in the environment. Fortunately even though many compounds are colorless we know enough chemistry to convert them by a chemical reactions into another color compound which can be monitored very easily and such compounds are known as chromophores. The chromophore when it reacts with a substance it converts the chemical or it forms another chemical which is colored, sometimes it need not form colored substance. it can even reduce the colored substance also, if it is a substance inherently colored we can remove the color by a chemical reaction. Either way spectrophotometry measures the color of the sample and tries to correlate it to the concentration of the substance, as of now more than 100000 compounds can be made to undergo chemical reactions to produce colored compounds, on an average more than 30 millions spectrophotometric measurements are made daily worldwide. You can imagine how popular it is the spectroscopic technique that is known as spectrophotometry, very very popular and then another advantage.

Of spectrophotometry is that the procedures can be automated with little or no manual intervention also. So, why not use it? So, the, what I am trying to tell is how spectroscopic techniques are useful for monitoring the environmental pollutants. Earliest somewhere around 1930's spectrophotometry was introduced all most about 80, 90 years before our times is still being used more than 100000 compounds being determined. More than 30 million spectrophotometric procedures being made every day and that is a standing example of how a spectroscopy technique can be used for pollution monitoring.

Now, the spectrophotometry can be used for metals and nonmetals, gases etcetera organic compounds in organic compounds all almost all, the only problem is you have to learn a lot about chemistry. So, if you are not a chemist you may not be in a very good position to carry out a spectrophotometric determination, you must be trained for that and learning chemistry is not in everybody's (Refer Time: 07:40), but leading the result is in everybody is requirement. So, spectrophotometry has got its own limitations so to improvement came in fast and wide, now right now it is based.

Spectrophotometry is definitely as I told you it is based on the measurement of colors heterocyclic chemistry and analytical chemistry today are still rapidly changing subjects.

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Spectrophotometry is based on the measurement of colour of the chemicals. Heterocyclic Chemistry and Analytical Chemistry today are rapidly changing subjects whose almost frenetic activities are attested by the countless research papers appearing in established and new journals and by the proliferation of monographs and reviews on all aspects of the fields. The interdependence of these two major branches of chemistry has resulted in the resurgence of UV-visible spectrophotometry due to the enhanced selectivity and sensitivity of the method by choosing appropriate heterocyclics to react with the target species. Consequently, majority of this research has been transferred in the last two decades to the field of Environmental Analytical Chemistry.

Who is almost frenetic activities are attested by countless research papers appearing in established a new journals and by the poly proliferation of monographs, reviews, its books, symposia, conferences, international call all. So, in all aspects of the chemical

spectrophotometry there are, lot of activities going on around. So, the inter dependence of the heterocyclic's and the spectroscopy has resulted in the resurgence of UV visible spectrophotometry due to the enhanced selectivity and sensitivity of the method by choosing appropriate hydro analytical reagents mostly based on heterocyclic's to react with the target species.

Consequently majority of this research has been transferred in the last 2 decades to the field of environmental analytical chemistry, information is available if you can use it well and good and it is beneficial also.

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While spectrophotometry addresses molecular absorption of coloured compounds atomic absorption spectrometry is very useful for the determination of metallic elements. While the theory of atomic absorption was known since the days of Fraunhofer the technique gained wide acceptance only around 1960s.

Walsh, in 1955 developed the first real application of atomic absorption to chemical analysis. In the same year Alkemade and Miatz described a double beam method of spectral selection with two flames, the first being the source and the second as atomizer. Since then the atomic absorption spectrometry is in the forefront of chemical analysis. The first commercial atomic absorption instruments appeared in 1960s.

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So, what are the problems then? The problems is while spectrophotometry addresses molecular absorption of colored compounds it does not absorb, it does not estimate the elements as such.

You have to do some mathematics a little bit of calculation from colored compound deduce basically it is a deduction very simple deduction though that how much of the element is there. So, to address these situations atomic absorption spectrometry has made its appearance for the determination of metallic elements specifically, while the theory of atomic absorption was known since the days of Fraunhofer that is somewhere around Fraunhofer that is somewhere around seventeenth century, late seventeenth century the technique of atomic absorption has gained wide acceptance only since 1960's that is spectrophotometry was introduced around 1920's and 30's whereas.

In 1960's atomic absorption made its appearance in the field of analytical chemistry. So, in 1955 Walsh developed the first real application of atomic absorption to chemical analysis. In the same year Alkamade and Miatz described a double beam technique for the determine spectrum selection of with 2 flame, with 2 flames the first being the source and second as the atomizer. So, what is what does it do an atomic absorption spectrometer, atomic absorption spectrometer converts a given sample into vapor with lot of electrons are around it and metal ions.

In the vaporized form picks up the electrons forms atoms, these atoms get excited to next higher energy level and when they fall back to the atomic absorb to the ground level the radiation they absorb quantized radiation to fall back. Now, the if I know how much of radiation I am supplying before the atomization and how much I am getting after the atomization I can correlate it to concentration, that is the basic technique that this course also I have given for about 20 hours in MOOC you can look it up, but for your kind information this is all about atomic absorption, convert the sample not into colored substance, but into atoms.

The technology to converts the sample into atoms is what made atomic absorption the great technique that it is today even now. Since then, since 1960's the atomic absorption spectrometry is in the fore front of chemical analysis, the first commercial atomic absorption appeared in 1960's commercial one.

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Spectacular advances in instrumentation, electronics, automation and computers over the years have made atomic absorption spectrometry, one of the most reliable analytical techniques of modern times perhaps equaled only by atomic emission spectrometry in terms of simplicity, sensitivity, specificity and speed of operation.

The development of electrothermal atomization by L'vov and Massmann pushed the detection limits of atomic absorption technique to nanogram and picogram and sometimes even up to femtogram levels.

So, spectacular advances in instrumentation, electronics, automation and computers over the years have made atomic absorption spectrometry one of the most reliable absolute analytical technique of modern times perhaps equaled only by atomic emission spectrometry which I we are going to study now and in what way does I c p that is atomic emission spectrometry is comparable to atomic absorption spectrometry of the 1960's.

That is in terms of simplicity, sensitivity that is how low you can determine the given pollutant or metal element and then specificity how specifically, how sure you are that what you are giving a result corresponds to the same element. What is the percent of error in the interpretation? There should be 0 percent basically if you are monitoring a particular metal ion element and speed of operation of course, it has to be comparable to atomic absorption if atomic emission spectrometry has to become an acceptable technique.

So, the there are further improvements in atomic m a absorption, the improvements initially where not much compared to spectrophotometry. A spectrophotometry could be determined a parts per million level at its peak and improvements made it up to 10^{-9} that is nanogram level below nanogram or sub below sub ppm. It was difficult to for all elements to be determined now the development of electro thermal atomization by Lvov and massmann pushed the detection limits using atomic absorption as, to the levels of nanograms.

That is quantitative determination in terms of nanograms that is 10^{-9} grams and the picogram 10^{-12} sometimes femptograms, that is 10^{-15} grams. To that level atomic absorption developed absorption atomic absorption not atomic emission, now we are not talking about atomic emission yet.

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Hydride generation atomic absorption spectrometry for arsenic, antimony, bismuth, selenium, tellurium, germanium, lead and cold vapor mercury determination have proved attractive accessories for atomic absorption technique to make it the first choice of analytical chemists throughout the world.

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But, now we are going to do that, but before that a small intervention that is another advantage of atomic absorption is hydride generation, atomic emission atomic absorption spectrometry for arsenic, antimony, bismuth, selenium and tellurium, germanium, lead and a cold vapor mercury determination have proved attractive accessories for atomic absorption technique to make it a first choice of analytical chemists throughout the world.

So, what is the situation now? The situation now is the atomic absorption spectrometry is one of the most preferred technique as far as the analysis of metal elements are concerned. We do not want to do it by spectrophotometry because it involves lot of chemistry work and the determination limits are not that good, they are cumbersome, that atomic absorption as of now it is preferred technique. Now, I am going to talk to you about atomic emission spectrometry.

Why should we do it? If you are already if we already have the capability to determine the chemical elements at picogram levels, femtogram levels by atomic absorption why should we learn one more technique? Why should we have one more technique? To do the same analysis unfortunately the science does not work like that, science always gives you an alternative that is the beauty of science. So, the advent of atomic emission spectroscopy has also been equally in spectacular and it is simpler than atomic absorption it is faster than atomic absorption.

And with the advent of electronics computer this that etcetera everything is as good as atomic absorption. So, now, you have a choice of 2 techniques, one is atomic absorption another is atomic emission technique. So, what is the atomic emission? Since last 30 years it is there and commercial instruments have been available in the market, the story of atomic emission spectroscopy is an interesting one.

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Since last 30 years atomic emission spectroscopy has gained wide popularity as an alternate technology for AAS. The story of atomic emission spectroscopy (AES) is an interesting one. From 1925, photographic atomic emission spectroscopy (spectrography) was accepted as a quantitative multi-element analytical technique. In the 1960s, AES declined as atomic absorption spectroscopy (AAS) took hold, but there was a resurgence of interest in the emission spectroscopy in 1970s with the advent of modern excitation sources, such as the inductively coupled plasma (ICP), and the parallel development of improved electronic detection methods and microprocessor technology.

Today, atomic emission spectroscopy is a well-established analytical technique that no-one involved or interested in chemical analysis can afford to ignore.

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From 1925 we know the theory of atomic emission, photographic atomic emission spectroscopy was known as spectrography, the principal was known and then applications were known.

The chemical system was being practiced in almost all boundaries and alloy factories, alloy manufacturing factories and it was accepted spectrography was expected, accepted as a quantitative multi element technique, multi element analytical technique, with atomic absorption I never said anything about multi element analysis. Now, here is the advantage of atomic emission spectroscopy that is you can simultaneously determine many elements using one equipment, one source, one instrumentation and one computer as compared to single element with respect to atomic absorption spectrometry.

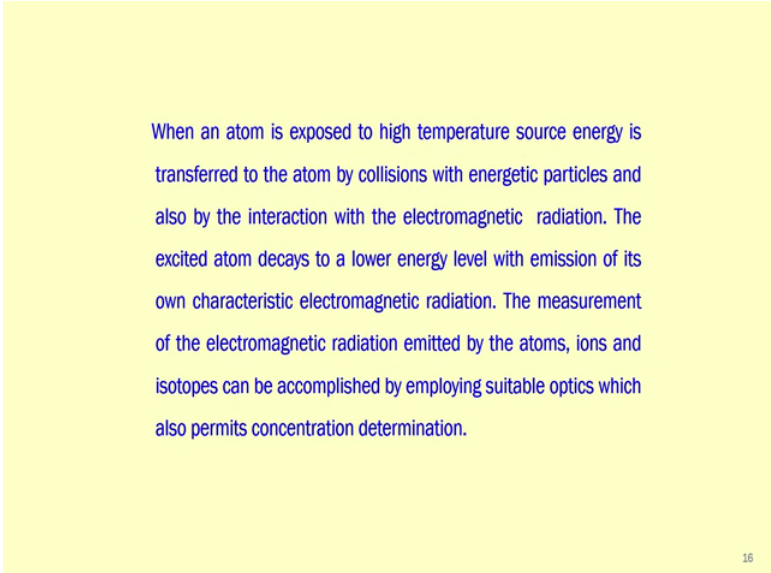
Which is better? Obviously, atomic emission spectroscopy, now the catch is it was available only to foundries, metal manufacturing industries and it was not known as the atomic emission spectrometry it was known as spectrography and basically it was a technique to determine the elements in by measuring the density of the spectrographic

lines, more dark more is the concentration, less dark less is the concentration. So, in 1960's atomic emission declined actually decline even though the technique is known since 1925 around 1960's because of the advent of atomic absorption the practice of atomic emission declined.

Because AAS spectro advances in atomic absorption was tremendous, but there was a resurgence of interest, in the emission spectroscopy in the 1970's why because the parallel advances in instrumentation computer automation and everything got transferred to atomic emission spectroscopy and commercial instruments made their first appearance in the 1970's and 80's. So, with the advent of modern excitation sources such as inductively coupled plasma ICP that is ICP, inductively coupled plasma which produces very high temperatures to convert any element in to atoms.

So, that is why we call it ICP AES inductively coupled plasma atomic emission spectroscopy, the parallel development of improved electronic detection limits and microprocessors that lead the way to the development of ICP AES, today atomic emission spectroscopy is a well established analytical technique that no one involved or interested in chemical analysis can afford to ignore, that is why we study ICP AES.

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When an atom is exposed to high temperature source energy is transferred to the atom by collisions with energetic particles and also by the interaction with the electromagnetic radiation. The excited atom decays to a lower energy level with emission of its own characteristic electromagnetic radiation. The measurement of the electromagnetic radiation emitted by the atoms, ions and isotopes can be accomplished by employing suitable optics which also permits concentration determination.

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So, how does it work when an atom is exposed to high temperature the energy is transferred to the atom by collisions with energetic particles and also by interaction with the electromagnetic radiation ok.

Now, look at me, the what I want to do these I take my sample supply a high energy convert everything into atoms. Now, the high temperature makes the metal melt boiled, convert it into vapor collide with each other and then the energetic particles will react with the electromagnetic radiation. I pass electromagnetic radiation through a mass of highly excited atoms produced by ICP the excited atom will not stay in the excited state for long time I think every 10th standard student would be aware of this phenomena.

So, I take energy supply it to the atom, goes to next higher energy state it comes back to the ground state after some spending sometime in the excited state and this is accompanied with the emission of its own characteristic electromagnetic radiation. That is apart from the radiation that we are supplying it also emits its own characteristic radiation depending upon what element it contains. So, if I am able to isolate that element, if I am able to isolate that radiation corresponding to the element of my interest I should be able to determine how much of that element is there as simple as that. Of course, it is not as simple as what I am trying to make, but the basic theory and principle remains the same.

So the measurement of electromagnetic radiation emitted by the atoms ions and isotopes etcetera can be accomplished by employing suitable optics which also permits concentration determination. So, what does it mean? We have so far talked about science environmental pollution and spectroscopic techniques, among the spectroscopic techniques we have talked about spectrophotometry and atomic absorption and we have also talked about why and how of inductive couple plasma, that is only in the outlines. So, far we have talked only about the outlines not more than that.

So, you are going to learn about it correct. So, if you are going to learn about it, it should now what you are going to learn about.

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This course has therefore been designed to give you an introduction to the technique of inductively coupled plasma-atomic emission spectroscopy (ICP-AES) and insight into this aspect of pollution control by incorporating several scientific disciplines, which includes:

- Atomic and molecular structure.
- Basics of atomic and molecular spectroscopy.
- Instrumentation for ICP-AES.
- Practice of ICP-AES.
- ICP-AES for metal ion determination and pollution monitoring.

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So, this course has been designed to give you an introduction to the technique of inductively coupled plasma, atomic emission spectroscopy that is ICP AES, it will give you an insight into this aspect of pollution control by incorporating several scientific disciplines. See no atomic emission spectroscopy can come on its own, it has to you have to know about the atomic structure that is what we talked initially the moment I started this course and then we have to know about basics of atomic and molecular spectroscopy, we have to know about the instrumentation.

We have to know about the practice of ICP AES and we also have to know about the determination of metal ion and pulley with respective environmental pollution monitoring. So, you may ask why should I learn about atomic structure, atomic and molecular structure is my first this thing no here, why should I learn? I have to I just want to know the technique, the problem is if you most of the spectroscopic techniques are based on atomic and molecular structure. Basics of atomic and molecular spectroscopy this also you should know, now if you do not know this whenever I am talking about atomic emission spectroscopy you may not be able to follow me at all.

I want to talk to you about how an atomic structure is represented, you might have studied most of it in your high school or you might have studied in your college level, PUC level or even at BSc level. So, for many of you it may be a repetition, but I know several instances where engineers and other people have forgotten what is an electron,

what is a proton, what is the nucleus, what is a transition, what is k, k shell l shell, what is sub shell? So, the idea is to refresh your memory, with respect to the terminology of atomic structure and then I want to refresh your memory of physics with the optics that is, what is a prism? What is a lens? What are the laws of physics? What is grating? All these things enter into are there in your ICP AES atomic emission spectrometry in instrumentation. So, if for any chance, if by any chance you have to you are in a position to operate the atomic emission spectrometry and if it is like a black box in front of you, know it is just shows you the reading, you will never be able to do the justice for your work. Therefore, you must learn about the fundamental principles of atomic and molecular spectroscopy and the interaction of electromagnetic radiation with the matter with matter means glasses, lenses, mirrors, gratings several other components optical components that are present in atomic emission spectrometer.

And then the instrumentation, how are we going to generate the high temperature? How are we going to generate the atoms? What happens to that etcetera to the atoms, what is the fate? How they are interpreted and all those things. So, these are all basically essential concepts of instrumentation learning if you have taken the course on spectrophotometry or absorption you may find that there is certain amount of repetition. So, if there if you find that there is a certain amount of repetition we due to your previous training, make sure that you are going to learn something slightly different to that level you are going to add to your knowledge about the modern concepts of all these things and then we will go about ICP aes.

So, for about 5 to 6 hours we are going to study their related techniques, then we are going to graduate into ICP AES last 4 or 5 lectures and then let us see how best we can make use of because it is the technique of future. Now, you may not know much about it you may be just hearing about it, so you may have to practice about it or you may have to interpret the results of somebody who is using ICP AES for you. So, all these things you can do a better job as a technologist if you know about this thing, that is why I have included most of these topics for you.

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MOOC – (Syllabus Template)	
Intended audience	Chemists and Chemical Engineers, Environmental Engineers, Environmental Scientists, Civil Engineers, Pollution Control Administrators.
Is it a core/elective course?	Elective Course
Is it a UG/PG course?	PG Course, (Useful for UG also)
Which degrees would it apply to? (BE/ME/MS/BSc/MSc/PhD etc)	BE/ ME / M.Sc /M.Tec/ MS
Pre-requisites in terms of educational qualification of participants, if any other courses should be done before this can be done	10 +2 + 3 years of BE / BSC Basic knowledge of differential calculus and integration
Industry support – List of companies/industry that will recognize/value this online course	Chemical industries, Pollution Control
The time frame of when you would want to offer the course: Jan-Mar 2016 July-Nov 2015	Jan – June 2018
Will the final certification exam be online or offline?	Online.
Weightage of assignments and final exam score to get certificate score	Assignment 25 Examination 75

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Now, this I have a slide I have already shown you as the first slide because now you are in a position to appreciate what I am going to tell you, that is regarding it is for chemist, chemical engineers, environmental engineer, environmental scientist, civil engineers, pollution control administrators and it can be an elective courses. It can be part of your curriculum if you are a student at PG level or under graduate level, it will be useful for you and the beneficiaries would be the students of BE, ME, M.Sc, M.Tech and MS in universities and technological institutions, but the prerequisites I still retain as 10 plus 2 plus 3 and industry wants it, it Is almost all chemical industries dealing with metals.

They need it for pollution control every industry that is having a pollution that is letting out it Is the affluent or solid waste or emission or they need it and the time frame and other things I have already covered and with 25, 75 assignment and examination content.

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Course Title	INDUCTIVE COUPLE PLASMA ATOMIC EMISSION SPECTROMETRY (ICP - AES) FOR POLLUTION MONITORING
Discipline	CHEMICAL ENGINEERING
Number of hours of course 10 hrs/ 20hrs/ 40hrs	10 HRS

Instructors of the course – Please attach a scanned photograph of each instructor						
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You are most welcome to go through most of these courses and any information you need, we will probably be glad to provide you and be feel free to contact us for any problem you wish to discuss with respect to atomic absorption, spectra atomic emission and spectrophotometry or any other chemical technology we are always there for you. So, I wish you all a good learning time for the next 9 hours or 10 hours. So, all the best.