Inductive Couple Plasma Atomic Emission Spectrometry (ICP-AES) for Pollution Monitoring

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Lecture – 10
Instrumentation for ICP AES – I

So, continuing our discussion with respect to ICP, Inductive Couple Plasma Atomic Emission Spectrometry. Now we are going to talk only about ICP-AES; no more of the general subjects which are essential for the course, but you already are familiar with the details of what I wanted to teach you. Now it will become very simple for you to understand when I talk about the instrumentation for ICP etcetera.

So, now I am going to talk about the specific instrumentation for atomic emission spectroscopy that is using modules, that is in the module 4. We are going to start; this contains this also contains a number of slides. I beg your patience with respect to the number, but I think most of them are essential for our understanding of ICP. And the beauty is, the more you understand about the fundamentals, the fundamentals of instrumentation and general background etcetera, the more you will be familiar with the technique also.

So, that is the big advantage of learning going through a course. Now, if you are studying in a postgraduate degree college or something like that, most of it you would have studied and you may find it a little repetitive. But you also may, I also have students who has stopped studying since so many years; they are all in a job, in a profession and they want to learn this subject because it is a brand new this thing that is the in thing now. Everybody wants to buy an atomic emission spectrometer. For them it becomes essential to know a little bit about the fundamentals and to just to refresh the memory.

So, if now that refreshing memory part is all over, now we are going to concentrate on the instrumentation for atomic emission. So, here goes the first slide.

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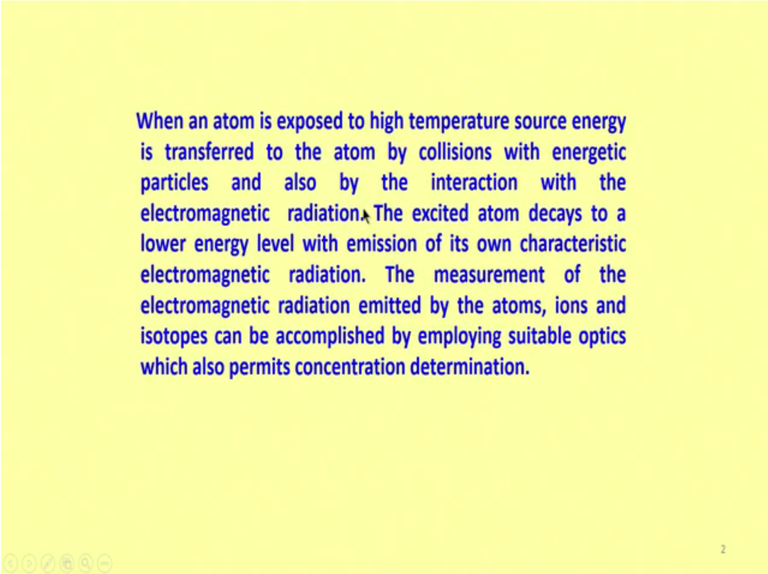

And so when, basic slide is very important, very simple; we have discussed it again and again. So, when an atom is exposed to high temperature, the energy is transferred from the atom by collisions with energetic particles and also by interaction with the electromagnetic radiation. So, the excited atom decays into a lower energy level with the emission of it is own characteristic e m radiation.

The measurement of the electromagnetic radiation emitted by the atoms, ions or isotopes, it can be accomplished by employing suitable optics. So, that we are going to study now which also permits concentration, determination. See the basically, the whole idea of this course is to use an instrument called as ICP for routine analysis as well as for pollution monitoring, you understand! So, if I do not correlate the changes in the electromagnetic radiation with respect to the sample to the technique, the purpose is not served. And once you finish your studies are getting to a job, you may not find enough time to go through all these details.

But then you may find it extremely challenging to learn about the technique. So, the basic requirement of any analytical instrument is to correlate the response to the concentration. So, that is what we are trying to achieve in this course now. So, the principle is measurement of the electromagnetic radiation emitted by the sample that is, it may contain atoms ions and la isotopes and all those things to concentration of the sample. The sample you may get it from anywhere. So, it becomes a challenge all the time.

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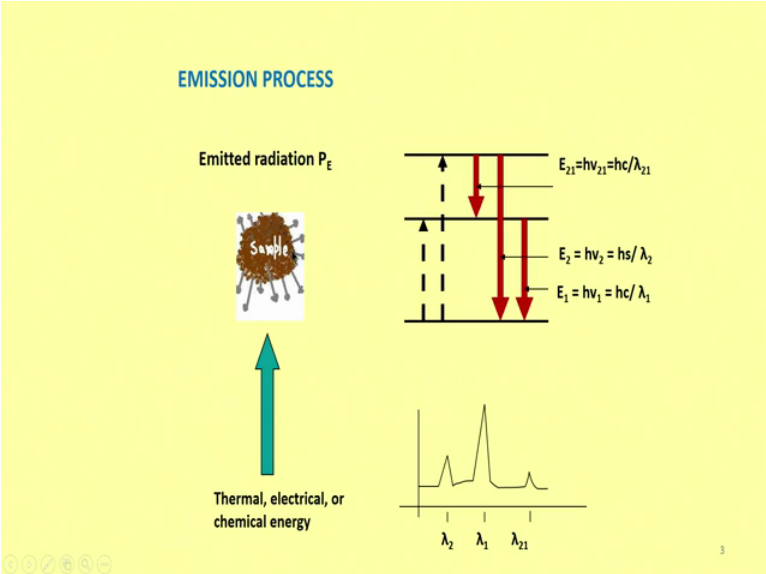

So, this is the emission process. This we have already seen here seen earlier, thermal electrical sample and this is absorption, this is emission and this is how the responses.

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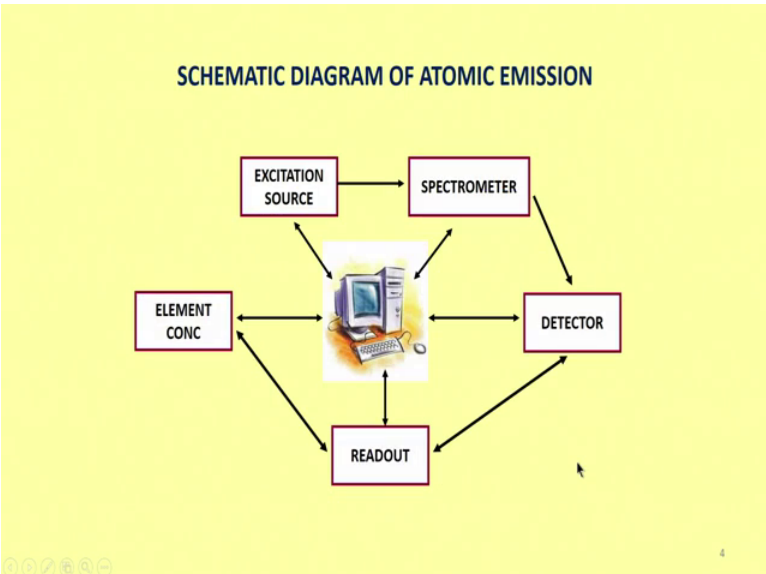

Now, if I have to make a spectrophotometer out of this, simple previous arrangement, what all things I need? So, these are the things I need. What are the things first is I need an excitation source ok.

Now, from now onwards we are going to talk only about atomic emission, a ICP, atomic emission spectrometry among there among those ICP only we are talking about it much more. So, excitation source I need. So, what is an excitation source? I need to provide a space where high temperature is required to excite the sample atoms. So, that is my excitation source that is inductively coupled plasma ICP.

So, the excitation source is called as ICP, inductively coupled plasma that is all, not no further spectroscopy or something it is ICP, it is a plasma. As you know many of the substances exist in solid, gaseous and liquid state. So, the temperatures, high temperatures are obtained in different areas. So, I have to put my sample in a an area where high temperature is required to excite the atoms.

Now, I can heat it, but it is any heating, what we know conventionally is not going to excite the atoms into the, excite the sample into atoms. We all, we are all familiar with molecules. Sometimes, elements also as solids metals, but if I have a sample of air or sample of a liquid or an oversample for that matter, I need to convert that oversample into atoms; for that the only way I know is, heat it. How do I heat it? I have to put it in a zone where high temperature is available.

For that, I need to dissolve it, convert it into small droplet us and then convert those droplet us into small atoms; and all this process needs energy. So, conventional energies with energy available to us in the form of a gas stove or electrical stove or a furnace or arc or something like that is not going to help us in producing the sample into atoms. We had already said that the temperature required is of the order of about 6 to 8000 degree kelvin.

Now, the solar suns temperature at the corona is around 6 to 8 degree 8000 degree kelvin corona. That is outrage, now inside edge it is several million degrees. Now the, that kind of temperature is available to us in the form of technology that is known as plasma. So, the excitation source is nothing but a plasma where I generate. Plasma is nothing but gas molecules present at higher temperatures. Sometimes they are luminous and they get some they are visible to your eyes as a small dull flame or they are not visible also ok.

So, the excitation source is one part of the component of the inductively coupled plasma spectrometer, spectrometer should have it is excitation source and then I need to power put the sample in the excitation source and then pass the electromagnetic radiation through that sample and that equipment is known as spectrometer. Now, once the electrons, once the atoms, once the molecules get excited in the plasma and they receive the radiation, they emit they also emit the radiation that is characteristic of the element. That radiation needs to be detected and quantified. For that we need photoelectric multiplier or photodiode array detector.

So, the radiation coming out through is connect and collected through the detector. And this detector comes out through the conversion of photons into electric impulse and this electrical impulse is converted into a measurable format of photons or resistance or conductance or voltage etcetera and that comes out as a read out; some reading will come, measurement and this measurement is related to the concentration of the sample ok.

So, that is how the different parts of the spectrometer ICP-AES are functioning; these are their functions. And all these functions are called connected or correlated using a computer, centralized computer. Nowadays, a desktop computer will do that job. Earlier there they were all used to be dedicated microscopic instruments, something like a black box. Nowadays everything is becoming simpler. So, this centralized computer will control the excitation source, it will control the excitation temperature, it will control the moderation or continuance of the spectrum plasma so that excitation becomes a continuous system.

And it will control the spectrometer, what radiation should go into that etcetera and it will control the detector, and detector how much current comes out, how it should be amplified and how it should be presented etcetera and the same computer will also give you the readout and this readout is fed to the computer again and from the computer again it calculates the concentration of the sample. So, the whole systematic arrangement of the atomic emission is done here in 1 system.

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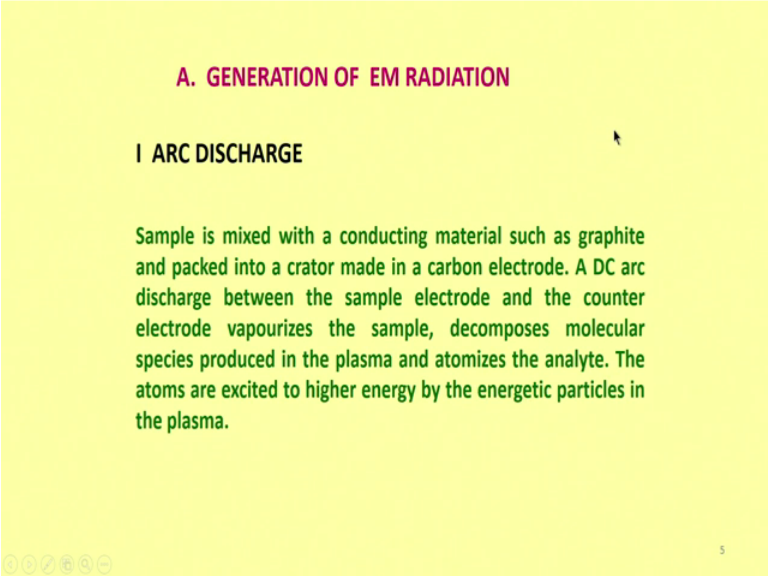

So, now we go in to different parts of the spectrometer. First one is we are going to talk about is Excitation Source. So, what are we going to talk about, how to generate plasma temperatures, high temperature where we can put the sample inside? So, there are different ways of doing it and the first one I am going to discuss is the Arc Discharge. So, Arc Discharge, usually how we do it is that, we take the sample mix it with the conducting materials and we take 2 electrodes containing, then put the sample in a hole inside this and then bring the 2 electrodes nearer and then strike an arc, pass the electric current and the moment the 2 electrodes strike, there will be flash of fire and the arc is generated; we call it arc is discharged.

So, as long as the current passes through the, from one arc from one electrode to another, the arc is active. So, the sample is mixed up or the, at the high temperature that is obtained, it decomposes the molecular species is produced in the plasma and atomizes the analyte; that is what we want. We want the molecules to be to decompose and they become atoms, ions and isotopes. So, you put them in the electrode itself and strike the arc. The atoms are excited to higher energy by the energetic particles in the plasma.

So, the arc discharge is nothing but a space where plasma is there. Plasma means practically for all practical purposes, you can take it as high energy zone with very high temperatures ranging from 8000 to 1000 degree kelvin.

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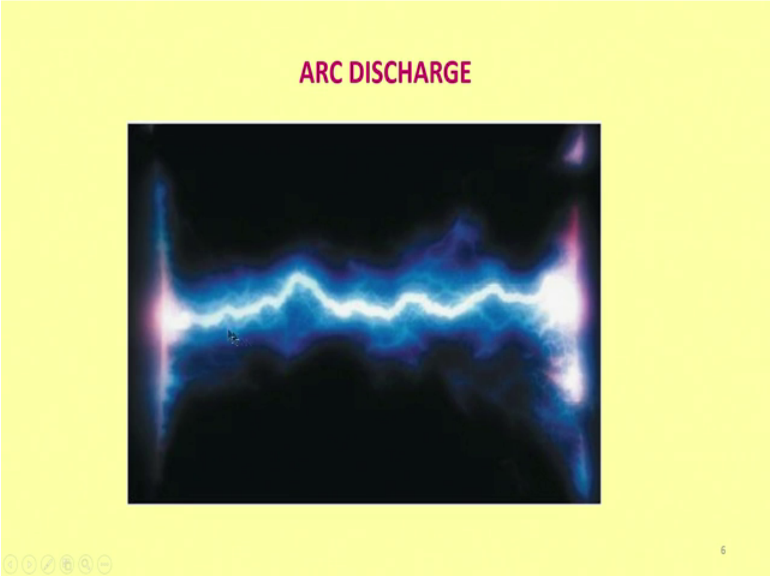

This is how it happens; here is one electrode, here is another electrode and then they come nearer and then the arc will electric current will pass through and this is how the arc discharge will take place.

So, you bring them farther, keep them farther away. Do you keep them farther away; there will not be any interaction. You bring them nearer, nearer, nearer, at some stage there will be attraction between the 2 electrodes and a spark will a zone will be generated and the electric current will pass through this range. And this is this you must have seen in your day to day life especially during rainy season in the skies ok.

So, it is a nothing new it is like lightning. So, you can see, it comes out with a very high sound like trrr and fantastic sound it makes when the arc is being produced.

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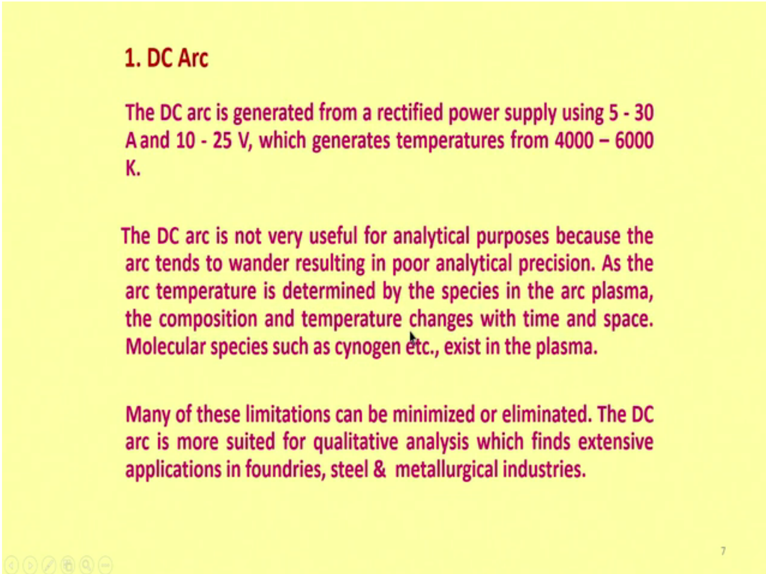

So, there are different types of arcs where we use the generation, which we use for generation. One is known as DC Arc. So, the dc arc is generated from a rectified power supply using 5 to 30 angstroms and 10 to 25 volts that generates temperature from 4000 to 6000 degree kelvin. This is not sufficient for all elements, but for few elements like sodium, potassium etcetera it is good enough, 6000 degree kelvin.

So, but the DC Arc is not very useful for analytical purposes. Why? Because, the direct current arc as it comes nearer and nearer, it becomes very violent. So, the for good analytical work we need a steady space, steady arc yeah. When we put the sample, it should not wander; the energy change should be smooth absorption or emission. So, unfortunately DC Arc is not very useful because the arc is very strong and it tends to wander as the electrode, eat away each other they melt and eat away each other, the arc quality also suffers.

So, the distance between the 2 as they melt away and fall down distance will keep on increasing; the quality of the arc will come down, because if the curve arced, if the electrodes remain at the same point, then the arc will be steady. So, as the arc temperature is determined by the species in the arc plasma, the composition and temperature changes with time and space. So, molecular species also exists in plasma, these are known as cynogens, CN, CO and many a molecular radicals.

Many of these limitations can be minimized or eliminated and the DC Arc is more suited for qualitative analysis. You see, there are always uses for different kinds of arcs and DC Arc has been used since several, since several decades for the qualitative analysis which I for that you do not need a steady arc. You just want to know how many elements are there in a given sample. So, bring them together and strike and record the spectrum straight away and then of all the elements that are there, will be producing a line spectra and you identify the lines and say this element is there or not; pass or fail, pass or fail. So, for quality control, that is fine but not for analytical purposes where quantitative analysis is to be done.

So, where do we need qualitative analysis? We need it in foundries, steel and metallurgical industries etcetera when they make the alloy, they just want to know whether those elements are there or not because before that again they had to analyze and when during the manufacture, many of the metals will vaporize and go away; They do not form the alloys. So, after the formation of the alloy, we need to determine how many elements are there etcetera. So, for that we need the qualitative analysis and these are the industries where they need; foundry, steel and metallurgical industry. So, that is there is a use for DC Arc several instruments are available for the DC Arc spectrographs, the instruments are known as spectrometer or spectrographs etcetera.

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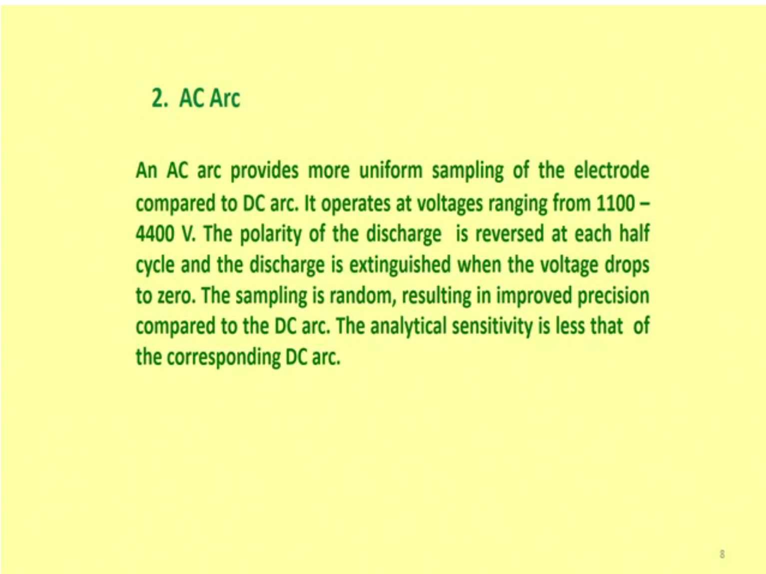

Now, we talk about AC Arc. So, AC Arc provides more uniform sampling procedure, sampling of the electrode compared to DC Arc. It operates voltages ranging from 1100 volts to 4400 volts. The polarity of the discharge is reversed at each half cycle and the discharge is extinguished when the voltage drops to 0. So, the sampling is random, results in improved precision compared to the DC Arc.

So, the analytical sensitivity unfortunately because becomes half of DC Arc because, a half of the time the current is not there, they keeps on switching the polarity also 0 to 1, 1 to 0, 0 to 1, 1 to 0 like that. So, the 50 percent of the time, the analytical sensitivity is lost.

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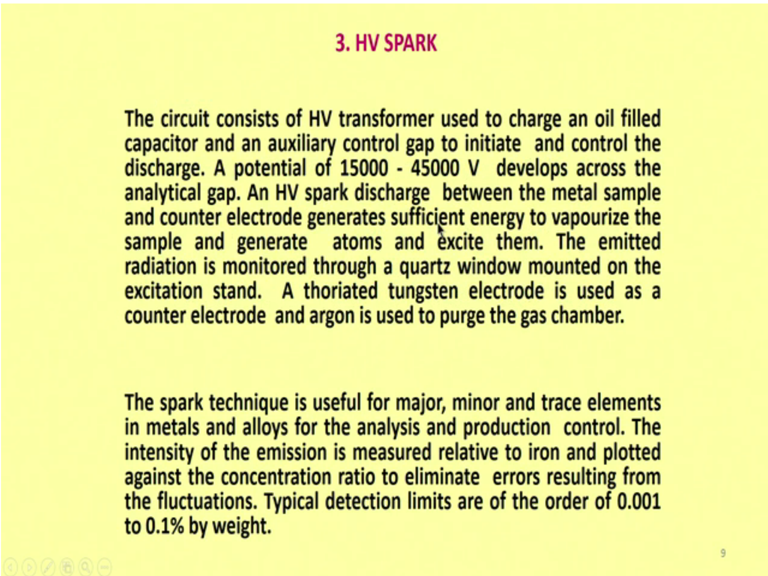

So the detection limits become very less. I think we should continue our discussion in the next class.