

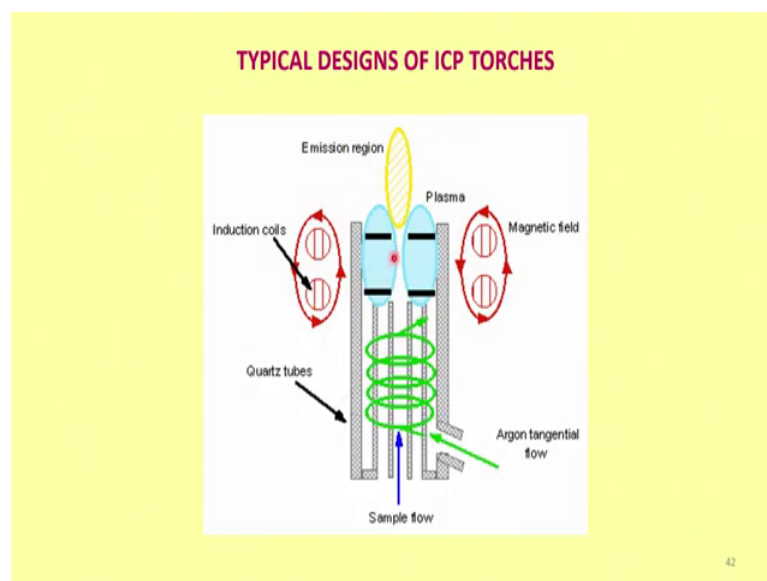
# **Inductive Couple Plasma Atomic Emission Spectrometry (ICP-AES) for Pollution Monitoring**

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## **Lecture – 14** **Instrumentation for ICP AES-VI-ICP Torches**

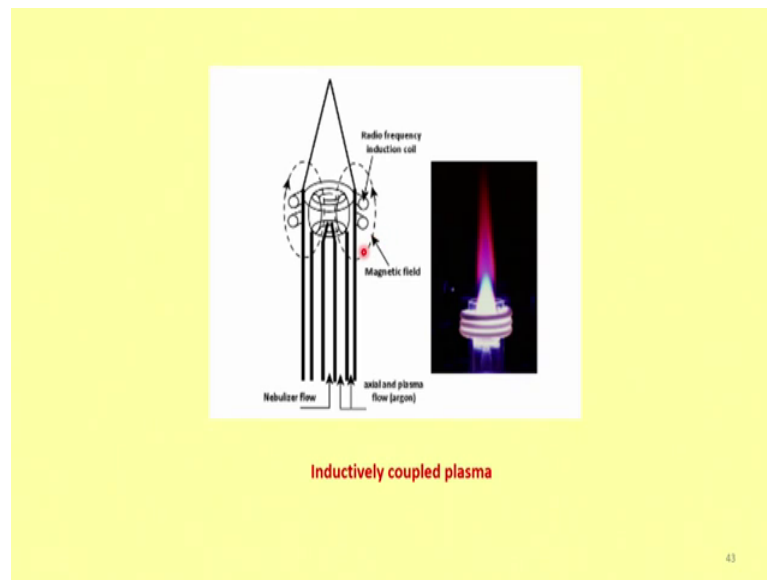
This is the torch we were discussing.

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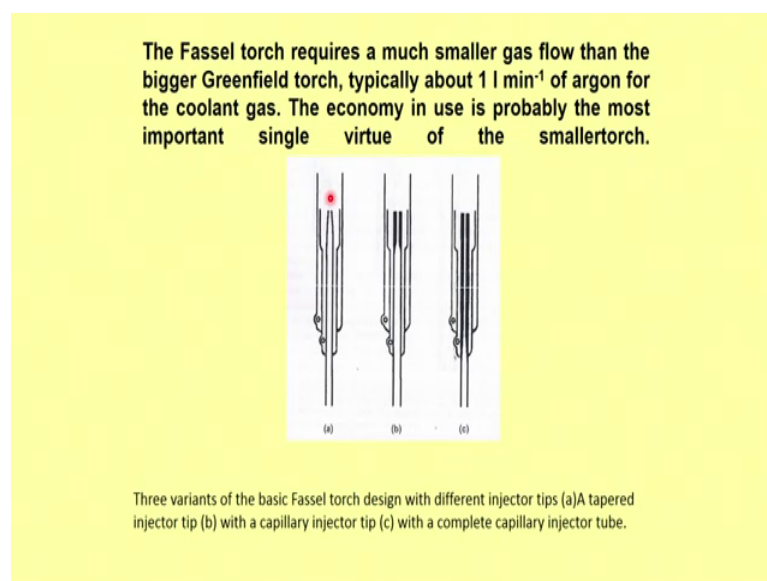
That 3 concentric circles in which the Argon gas is flowing, it is flowing round and round also, its sample is picked up like this, and then it enters the system.

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So, this is Inductively coupled plasma, actual photograph of the plasma torch. Here you can see the torch, plasma torch. And then the plasma is shown in the red one, the sample comes through this tube from the plasma torch, and then enters some sort of a flame, and then there is a space here, violet colored inside the red one and that is where the plasma temperatures are reached. And basically the structure is very simple, just like, what I had explained to you, this is the nebulizer flow that is the sample in the innermost circle and in the circle outside, there is axial flow of the Argon. And these are the magnetic field, radio frequency, detection coil, then there will be plasma here, plasma you have to start it like a button, you have to start it using a switch that is known as tesla coil, and once the plasma starts it has to stabilize.

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So, there are different types of plasma torches, normally people use. Earlier plasma torches, for, something like this, they all used to use Argon flow quite a lot, that is about 20 liters per minute, total, in all the 3 put together, but then people thought that, the Argon consumption is too much, is there any way we can 20 liters per minute means, in a day you will consume 1 big cylinder of the Argon, if you work for about 8 hours, 1 cylinder is required per day, for ICP AES work, and out of that productive work would be about 5 hours, because plasma has to start, stabilized, and in between you may not have the sample plasma moves so, maintenance and all that it will take more time. So, people thought, why not we try to reduce the Argon consumption.

So, new torches have been developed and that is known as Fassel torch, another variant, and in that variant, the Argon flow is approximately about 1 liter per minute of the Argon for the coolant gas. So, the plasma torch which I had shown you earlier, that is this torch, this torch is known as Greenfield torch, it is the name of the scientist, in which all the 3 gases flow to the same extent, and that is known as Greenfield star torch. Earlier most of the instruments, ICP instruments used to come with a Greenfield torch, and then the improvements are in the form of, we make this a little sharper here, the sample this thing. And Greenfield torch is the most the important aspect of Fassel torches economy of the gas. So, economy in use, is probably the most single virtue of a smaller torch, there nothing much to say about it except for the design. Here you can see, I am showing you 3 types of torches, 1 is a, b, and c.

The 3 variants of the, they differ only near the injection tip, that is the innermost concentric tube, you can see in all a, b, c. Here, they are, in a, that is here the innermost tube, if you come up and take a look at it, it is a constricted or tapered injector tip for the sample ok.

And second one is, it has become a little thicker here near the top. So, it becomes a capillary, sort of capillary injector tip, only the tip is sort of made thicker, so that the sample comes out of a capillary; that means, smaller, sample quantity becomes smaller here. So, there, that is an improvement in this. And third one is, I simply use a capillary here for the sample, total, right. From this point onwards, see, to the delivery of the sample, there is only a capillary, the full tube is a capillary. So, it can be even in a Teflon capillary or quartz capillary. Now you will see, in all these designs that the outermost tube is about 1 centimeter taller than or than the inner tubes, this is a very characteristic of the Fassel torch, because if you see the previous Greenfield torch, you can see that all of them are same size, same level, and here also it is almost same,

It is just about 2 millimeters difference between the top and bottom of the this thing, even in Greenfield torch, you may feel there is too much difference, but this is only about 2mm difference, this between this plate and this plate, 2 black plates inside the green system that is hardly about 2 millimeters whereas, in this thing, Fassel torch it is about 2 centimeter, so that the sample get cooled before it enters the plasma toroidal space ok.

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The geometry of the torch ensures that a vertical tunnel is blown through the flat base of the plasma by the central flow of argon which carries the sample material. Thus the hottest zone of the ICP is toroidal in form. The material for excitation passes through the central tunnel and reaches a temperature of 8000K. At this temperature atomization is virtually complete and the atoms become highly excited and partially ionized. The spectrum emitted is observed just above the very bright plasma where the atomic emissions can be measured against a low background. It is the special geometry of the plasma torch which endows the ICP with many of its unique spectroscopic properties.

So, the geometry of this torch ensures that a vertical tunnel is blown through. Vertical tunnel is blown through the plasma, that toroidal system, and the sample is introduced forcibly. So, the central flow of the Argon is the one, which carries the sample. So, if the Argon flow is low, smaller quantity of sample can be sprayed into the plasma and it can be analyzed.

So, the hottest zone of the ICP is always toroidal in the form. So, the material for excitation passes through the central tunnel and reaches a temperature of about 8000K. At this temperature atomization is virtually complete; that means, whatever sample you introduce, it just gets converted into vapor and then atoms, but if the sample is not converted into a fine droplet, what happens is bigger droplet is remain, they fall down back into the system through gravity, that is not required, because the efficiency of analysis always depends upon the efficiency of conversion of the sample into atom atomized material. So, whatever sample you carry, it must be in the mist form. So, that is where the efficiency of atomic emission spectroscopy comes into the picture.

So, the spectrum emitted is observed just above the very bright plasma where atomic emissions can be measured against the low background. Again, I will take you back to Greenfield torch figure. Here, this is the emission region near the top, and plasma is here and the high temperatures are reached here, but the atomization is virtually complete and they start emitting radiation in all the directions, top, vertical, angular, east, west, to the sides everywhere. So, near the edge, the emission, the temperature is also very high. So, if the temperature is very high, normal emissions would be very less, that is, noise, noise of the emission, will, emission noise is very less near the edge, that is why we go back to this figure, where there is red one, and near the tip is where, here, in this region I place my optics. You go back 3, 4 figures, and you will see that optics arrangement should be placed somewhere near here so that it can, there will not be much change in the emission of the background ok.

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#### SAMPLE INTRODUCTION

The most common means of sample introduction is through the Bernoulli effect. High velocity moving gas flowing around a capillary sucks the sample and delivers it at the tip where the liquid breaks up into fine droplets before entering the plasma.

Another way of introducing the sample is to pump it through the capillary using a peristaltic pump. At the tip, high velocity argon flows across at right angles causing the same Bernoulli effect.

It is also possible to atomize the sample in a graphite tube and introduce the vapor into the plasma.

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So, this is how, we do the sampling and sample introduction. So, the most common way of sample introduction, is through the Bernoulli effect. So, what is a Bernoulli effect? it is a very simple system that, if you imagine a tornado, a tornado goes in a particular direction, but whatever is there, inside that also gets pulled up. I think you must have seen in many films and other means, if you look up even the YouTube, you will see that tornadoes how they pick up everything. So, initially there will be a low-pressure region and then the air will rush in, through that, during that process soils, and mud, this, that, trees, everything will get sucked into that, that sucking effect is basically a Bernoulli effect. And the idea is, high velocity moving gas flowing around a capillary, sucks the sample, that is the fundamental rule that is Bernoulli effect.

Any gas that is moving very fast, will suck along with it, whatever is the non-moving part or whatever is not to be moved. So, once the sample is sucked, inside the tornado, it goes, it gets pulled up to the top and then delivers at the height of the tornado, that is why, there will be lot of casualties in a tornado, because tables, chairs, this, that, hands, trees, and everything will get sucked up and then thrown out of the tornado at very high heights, and then they fall to the ground due to gravity; obviously, the damage will be more, if somebody gets stuck in the sucked into the tornado. Here, same thing happens here, in ICP, here what happens is, the sample gets sucked, and then there it gets delivered, it gets delivered into a fine mist, and near the tip, and near the tip, the sample breaks into very fine droplets, before it enters into the plasma.

So, it is fairly simple, all you got to do is, put the Greenfield torch or Fassel torch, and then dip the innermost tube into the sample, and start the Argon gas, that is Bernoulli effect. It will just suck the sample, on its own.

And then, there are other ways of doing it for example, the sample can be pumped. So, the pumping sample is a very tricky thing, because no amount of, no material is safe enough to put a sample directly into the plasma. So, the liquid needs to be carried up to the end of the plasma torch, and then it has to deliver. So, how do you carry the liquid through a ICP sampling system. So, gas also needs to be there, but liquid also needs to be transported. So, for that we use what is known as peristaltic pump, in the peristaltic pump, the advantage of a peristaltic pump is that, it will suck the sample, but the sample will not come into contact with any of the machine parts. So, it has to be sucked through the tube without touching any of the material parts, and connected to the Greenfield torch. We will see, how we can do that. And let me see whether I can show you here.

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Assume this is the sample. And then sample gets sucked, and I have bearings here, I have the sample coming in, and then it goes into a tube like this. So, the sample moves like this. So, the, without, the sample without touching into these red things are called as bearings, ball these are ball bearings. The sample moves along with that, along the, inside the tube, but as the bearings move the sample will start moving.

So, it will move from here and all the way, and comes out into the plasma torch. So, this is known as peristaltic pump.

Sometimes what happens is that, the liquid is either very precious, or it should not be contaminated, or it should be very difficult to handle, in such cases we use peristaltic pumps. So, the beauty of peristaltic pumps is that, the liquid does not come into any material part of the pump for example, if one has a problem with kidney, you know, dialysis, lot of people undergo dialysis with the diabetes and other elements, there what happens is, they take out the blood, pass it through dialysis unit, and then purify the blood, and put it back into the human system so, that is a peristaltic, job of the peristaltic pump. A similar peristaltic pump is used in such systems also, in ICP AES and.

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Sample material for excitation has to be carried into the plasma by means of the central gas flow, "the injector gas flow". In the most usual arrangement the sample in the form of an aqueous solution, is partially converted into fine droplets by a nebulizer. It is an aerosol of these fine droplets in argon which is injected. In practice any particles finer than about 10  $\mu\text{m}$  can be transported in a stream of gas without much deposition on tubing etc., so that aerosols of solid particles can be injected. In addition, material for analysis can be injected in the gas phase so long as suitable volatile compounds can be generated.

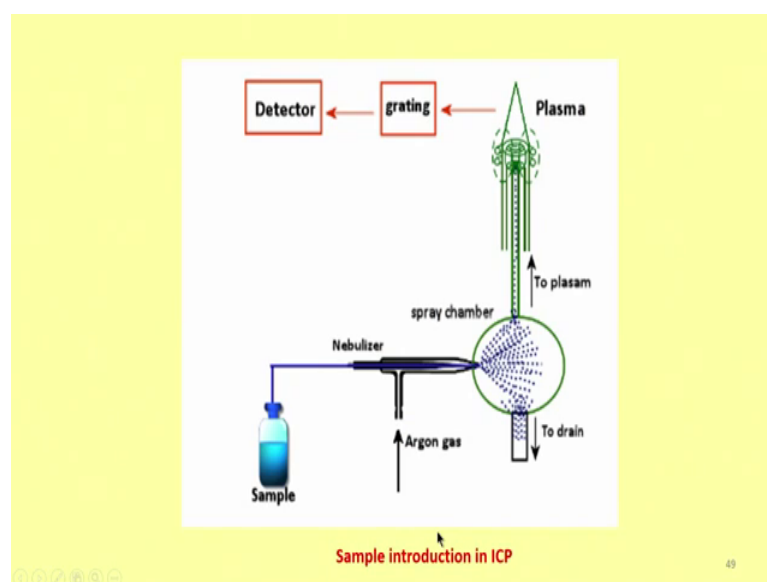
That is how the sample is carried into the plasma. So, one way is, for the sample, to get sucked into the system, using Argon flow itself, or use a peristaltic pump to introduce into the plasma, that is plasma torch. Once it enters the plasma torch, Argon gas will take over, and pass it on to the plasma, but till it is, it reaches the plasma torch, you need a peristaltic pump, through which the sample is sucked, and without getting into contact with any of the machine parts, must be carried into the plasma torch. So, peristaltic pumps are an essential component of the ICP AES, if you want to handle liquid samples.

So, the liquid samples must be carried into the plasma by means of the central gas flow so that is known as injector gas flow.



In most, in the most usual arrangement, the sample is in the form of aqueous solution, in 99 percent of the ICP AES applications. So, that is partially converted into fine droplets by the nebulizer. So, it is an aerosol of these fine droplets in Argon which is injected; so in practice, any particles finer than about 10 micrometer can be transported in a stream of gas without much deposition on tubing etc. Basically the particles of less than 10 micrometer should be carried into the plasma, otherwise plasma will extinguish. So, the aerosols of solid particles we can introduce, if the particle size is less than 10 micrometers. So, in addition material for analysis can also be injected into the gas phase in the gas phase so long as suitable volatile compounds can be generated. So, that is very important because sometimes hydride generation is etcetera we have already talked about it.

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And then, we have, there are ways of introducing the sample. So, what I have here is, I have the sample, a sort of a peristaltic pump, it gets sucked here, and then into the nebulizer, Argon gas is there, a fine mist is generated, bigger droplet us are collected back into the drain, and then the final droplets of the order of about 10 micrometers get carried into the plasma torch, which enter the, into the plasma, and then the emitted radiation must pass through the grating and detector.

So, what I have conveyed today, is the introduction of the sample into the ICP AES along with the optical mounting of the gratings, mirrors etcetera etcetera. We will continue our discussion in the next class.

Thank you very much.