Advance Analytical Course

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Module No. # 01
Lecture No. # 40

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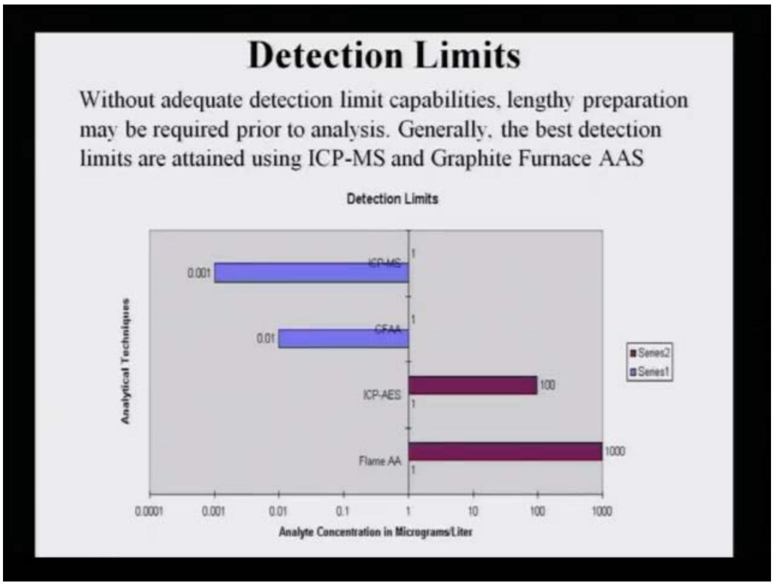

Detection Limits

Without adequate detection limit capabilities, lengthy preparation may be required prior to analysis. Generally, the best detection limits are attained using ICP-MS and Graphite Furnace AAS

Looking at the limits of detection or detection limits, one also needs to understand where to use what. This particular slide will give you an idea not only about the sensitivity of the different methods, of course, we took a look at the fact that what are the factors that need to be taken into consideration, but even the sensitivity factor is of great importance for an analyst. Without adequate understanding of detection limit capabilities, lengthy preparation procedures may be required prior to analysis. Generally, the best detection limits are attained using ICP-MS and graphite furnace AAS.

Now, you will see that GFAA and ICP-MS can go up to 0.01 and 0.001 respectively to the limits of detection, whereas for less sensitive and more frequent analysis, flame AAS and ICP-AES are good enough and that is why there is this available information what to use and when to use.

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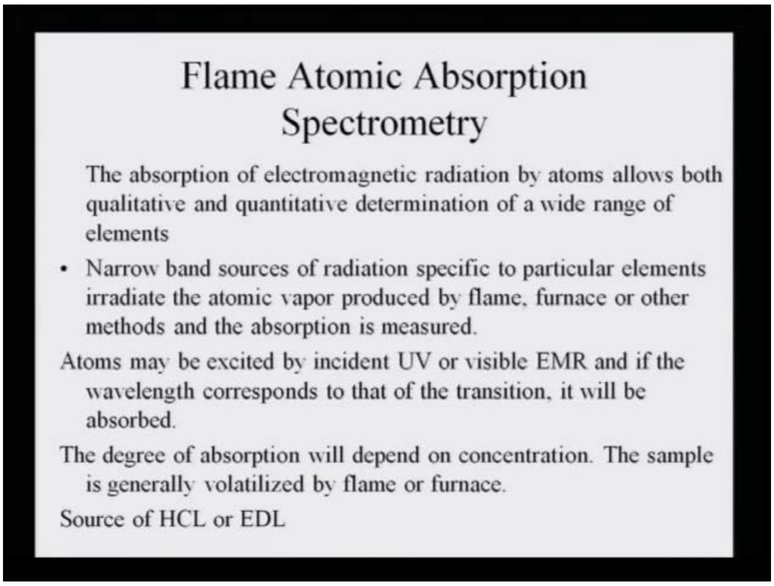

Flame Atomic Absorption Spectrometry

The absorption of electromagnetic radiation by atoms allows both qualitative and quantitative determination of a wide range of elements

- Narrow band sources of radiation specific to particular elements irradiate the atomic vapor produced by flame, furnace or other methods and the absorption is measured.

Atoms may be excited by incident UV or visible EMR and if the wavelength corresponds to that of the transition, it will be absorbed.

The degree of absorption will depend on concentration. The sample is generally volatilized by flame or furnace.

Source of HCL or EDL

Flame atomic absorption spectrometry - the absorption of electromagnetic radiation by atoms allows both qualitative and quantitative determination of a wide range of elements. So, not only qualitative, whether it is present or absent, but also how much of it in concentration is present can be determined for a large variety of elements.

Narrow band sources of radiations specific to a particular element irradiate the atomic vapour produced by flame, furnace or other methods and then absorption is measured and the beauty of this spectroscopic method is that the absorption is directly proportional to the molecular species that is present.

So, it cannot be only under very severe chemical coagulation method or if there are chemical agglomerates, then this can vary. Otherwise, if it is a dilute solution, it will always give correct result because the absorption is directly proportional to the molecular species concentration and that is why it is always recommended that very dilute solutions should be analyzed and then multiplied by the dilution factor. Atoms may be excited by incident UV or visible EMR and if the wavelength corresponds to that of the transition, it will be absorbed.

Now, it is the incident radiation should be matching with the excitation wavelength that is required for that particular molecular species or atom or ion; otherwise, it will not excite. Suppose, if we are analyzing copper metal and particular wave length which is very suited for copper is not shown, some other wave length is shown, it will not give the analysis because it will not excite the copper ions to the vapours and finally, the vapours getting the atomized atom then interacting with that radiation. The degree of absorption will depend on concentration. The sample is generally volatilized by flame or furnace and the source of lamp is hollow cathode lamp or electrode less lamp.

However, when such analysis is being done, it is not always foolproof. There are always interferences and what these interferences are and how to avoid these interferences is also a challenge for the analyst to face.

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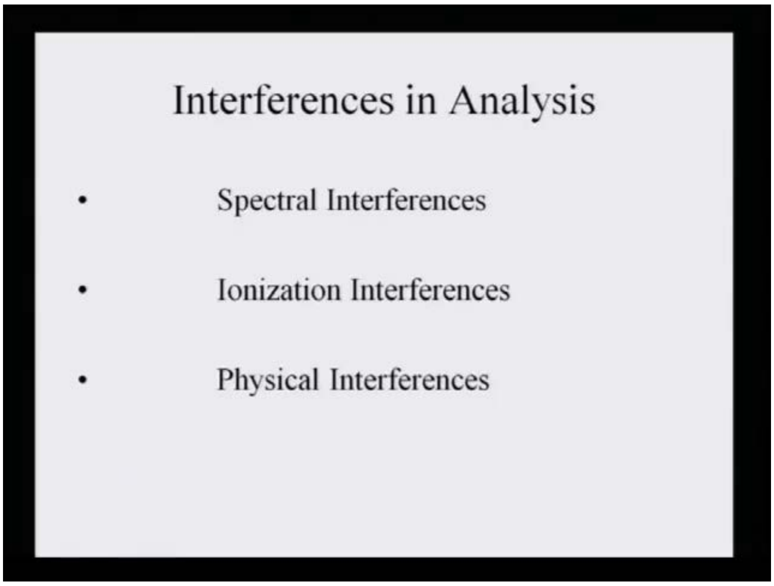

Interferences in Analysis

- Spectral Interferences
- Ionization Interferences
- Physical Interferences

The interferences in such analysis are spectral interferences, ionization interferences and physical interferences. As the name suggests, spectral interferences come in to play when we are taking into account ICP. In multi element analysis, where the spectral lines of excitations are very close, one has to choose the higher range of spectral lines, if they are coming very close.

Similarly, if there are not many ionized situation, the metal has not completely dissolved, it causes ionization interferences and of course, there could be some foreign bodies which can cause physical interferences. So, the samples should be very dilute, should be clear and should be filtered carefully through filter paper because otherwise the nebulizer will get clogged and the aerosol will not form.

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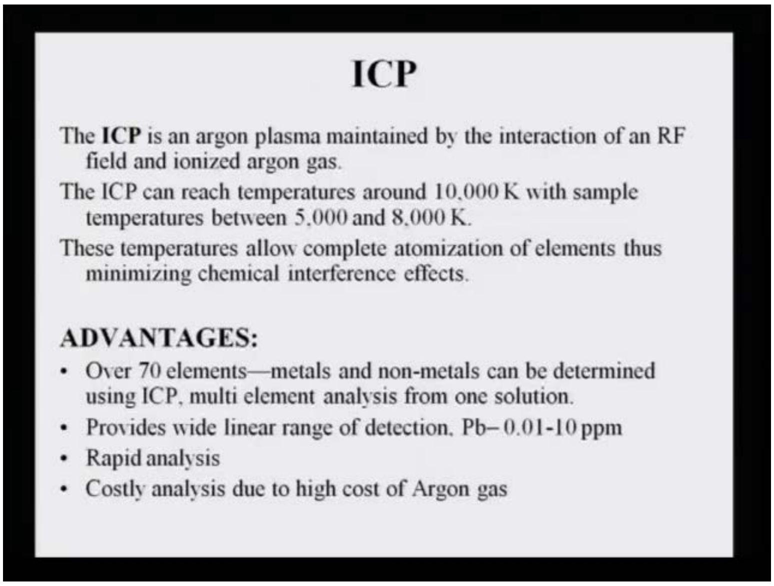

ICP

The **ICP** is an argon plasma maintained by the interaction of an RF field and ionized argon gas.

The ICP can reach temperatures around 10,000 K with sample temperatures between 5,000 and 8,000 K.

These temperatures allow complete atomization of elements thus minimizing chemical interference effects.

ADVANTAGES:

- Over 70 elements—metals and non-metals can be determined using ICP, multi element analysis from one solution.
- Provides wide linear range of detection. Pb– 0.01-10 ppm
- Rapid analysis
- Costly analysis due to high cost of Argon gas

Taking into more account and detailed account of ICP, the ICP is argon plasma maintained by the interaction of the RF field and the ionization argon gas. Now, this reflective field is there outside the flame. The ICP can reach temperatures as around 10000 kelvin with sample temperatures between 5000 and 8000 kelvin. So, you see that the flame has very high temperature and therefore, the ionization and the excitation takes place very readily. These temperatures allow complete atomization of elements, thus minimizing any chemical interferences effects.

The advantages of using ICP - there are several, several advantages. Over 70 elements, metals and non-metals can be determined using ICP, multi element analysis from one solution. You see, I told you in case of AAS, one has to go one by one. Suppose, if I want to analyze 9 heavy metals, I will have to do the experiment 9 times by changing the lamp every time and for every analysis, there will be 3 times the experiment will be carried - first, the standard, then the blank and then the sample.

So, 27 experiments need to be carried out, whereas if we are using ICP, just by one nebulization, the all the 9 elements can be programmed and analyze simultaneously. Provides wide linear range of detection - that means from a very low level to a fairly high level, it has a wide range of linearity. That means when the calibration graph is made, the calibration graph shows a very linear response and lead for example, can show linearity or you know range of detection from 0.01 to 10 ppm. It is very rapid analysis.

Although it is costly due to high cost of argon gas, but when it comes to sensitivity, to be able to analyze trace quantities versus to be able to analyze in a more accurate manner, then it is definitely, ICP has an edge over AAS.

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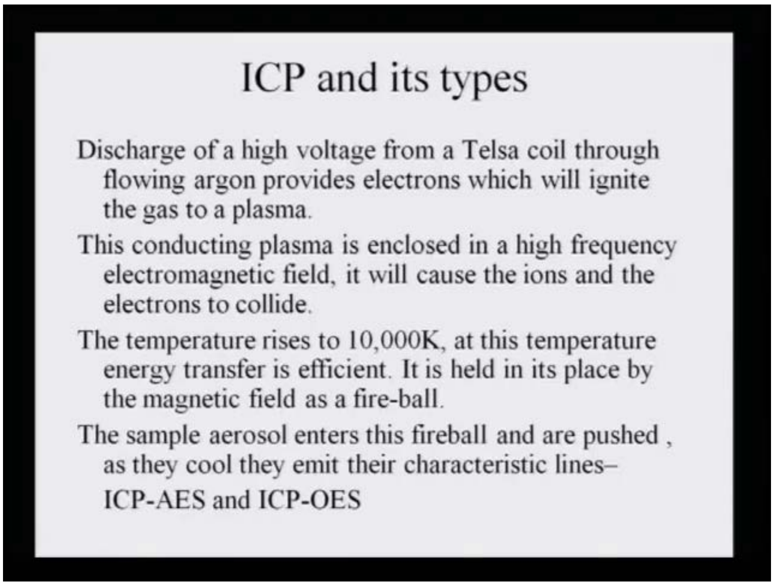

ICP and its types

Discharge of a high voltage from a Telsa coil through flowing argon provides electrons which will ignite the gas to a plasma.

This conducting plasma is enclosed in a high frequency electromagnetic field, it will cause the ions and the electrons to collide.

The temperature rises to 10,000K, at this temperature energy transfer is efficient. It is held in its place by the magnetic field as a fire-ball.

The sample aerosol enters this fireball and are pushed , as they cool they emit their characteristic lines–
ICP-AES and ICP-OES

ICP and its types - discharge of high voltage from a Tesla coil through flowing argon provides electrons which ignite the gas to a plasma. This conducting plasma is enclosed in a high frequency electromagnetic field; it will cause the ions and the electrons to collide. The temperature rises to 10000 kelvin. At this temperature, energy transfer is efficient. It is held in its place by the magnetic field as a fire-ball. The sample aerosol enters this fireball and thus pushed. As they cool, they emit their characteristic lines. That is what is shown in ICP-AES and in ICP-OES; both are emission spectroscopic methods.

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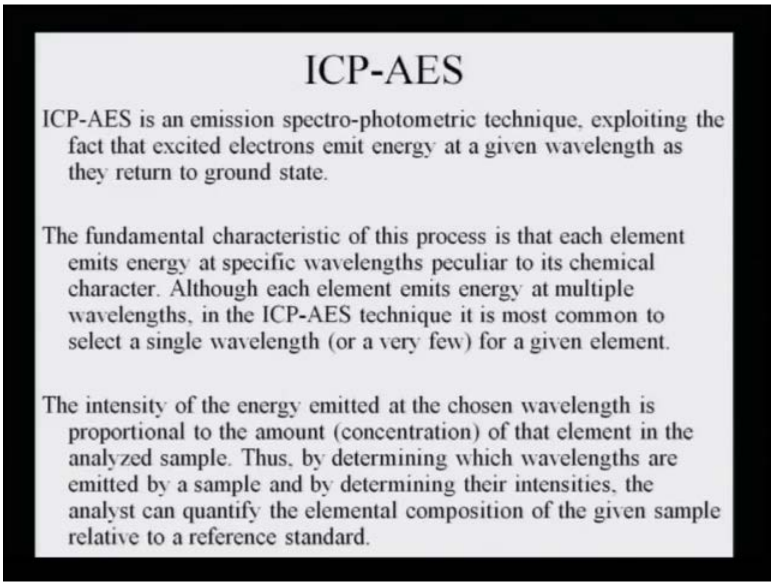

ICP-AES

ICP-AES is an emission spectro-photometric technique, exploiting the fact that excited electrons emit energy at a given wavelength as they return to ground state.

The fundamental characteristic of this process is that each element emits energy at specific wavelengths peculiar to its chemical character. Although each element emits energy at multiple wavelengths, in the ICP-AES technique it is most common to select a single wavelength (or a very few) for a given element.

The intensity of the energy emitted at the chosen wavelength is proportional to the amount (concentration) of that element in the analyzed sample. Thus, by determining which wavelengths are emitted by a sample and by determining their intensities, the analyst can quantify the elemental composition of the given sample relative to a reference standard.

Coming to ICP-AES, the ICP-AES is an emission spectroscopic photometric technique exploiting the fact that excited electrons emit energy at a given wavelength as they return to the ground state. It is of common understanding that suppose, if an atom is getting excited by observing light, it will go and reside on a higher excitation state, but it cannot remain there for too long. So, it will then come back to its ground state by emitting this light and it is this emitted light, which is actually the amount that it has absorbed and the amount would depend on the molecular species that is present. So, everything is related to each other.

The fundamental characteristic of this process is that each element emits energy at specific wavelength, particularly to its chemical character. That means it is very, very peculiar and particular to its chemical character only. Although each element emits energy at multiple wave lengths, in the ICP-AES technique, it is most common to select a single wave length or very few for a given element. So, as I said there are variable and multiple wavelengths available for every element.

Now, if we are making a program of analyzing copper, cadmium, arsenic, mercury at the same time and if by chance the copper and the cadmium have very close wave lengths, then for one of them, we will use the first wave length and in the case of cadmium, we will use a higher wavelength so that it can be identified correctly. Otherwise, if we choose the same wave length or the closer wave length, we would not know whether it is referring to cadmium’s concentration or copper’s concentration. So, that is a little tricky job; otherwise, analyzing an ICP is not much of a challenge.

The intensity of energy emitted at a chosen wavelength is proportional to the amount, that is, the concentration of that element in the analyzed sample. So, the crux of the analysis of this particular spectroscopic method is, and even other spectroscopic methods, where we do quantitative analysis is that whatever is the intensity of the energy that is emitted of a chosen wavelength is directly proportional to the amount or the concentration of the analyte of that element in that sample. Thus, by determining which wavelengths are emitted by a sample and by determining their intensities, the analyst can quantify the elemental composition of a given sample relative to the reference standard. So, we have a standard say, of 4 ppm. Now, in the sample, when we analyze we find out, whether it is more than 4 ppm or less than 4 ppm or close to 4 ppm; because of the determination of their intensities, it is possible to find out the concentration of the analyte very easily.

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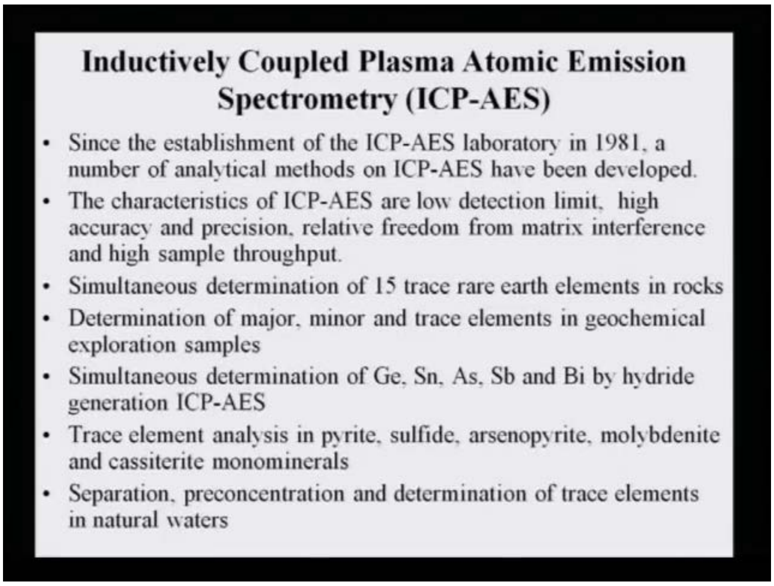

Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

- Since the establishment of the ICP-AES laboratory in 1981, a number of analytical methods on ICP-AES have been developed.
- The characteristics of ICP-AES are low detection limit, high accuracy and precision, relative freedom from matrix interference and high sample throughput.
- Simultaneous determination of 15 trace rare earth elements in rocks
- Determination of major, minor and trace elements in geochemical exploration samples
- Simultaneous determination of Ge, Sn, As, Sb and Bi by hydride generation ICP-AES
- Trace element analysis in pyrite, sulfide, arsenopyrite, molybdenite and cassiterite monominerals
- Separation, preconcentration and determination of trace elements in natural waters

Inductively coupled plasma atomic emission spectrometry, that is ICP-AES - Since the establishment of the ICP-AES laboratory in 1981, it is quite recent that means, a number of analytical methods on ICP-AES have been developed. The characteristics of ICP-AES are low detection limit, high accuracy and precision, relative freedom from matrix interferences and high sample throughput.

So, you see, it has four very advantageous qualities; that means it can go up to low levels of detection. Even if it is in trace quantities, ICP-AES is the answer. Then it has very high accuracy and precision. The results will always been correct. There is a repeatability of the result and precision. Relative freedom from the matrix interferences - that means the matrix does not interfere in the analytical method and there large number of high number of samples can be analyzed; that is why it has high sample throughput.

Simultaneous determination of 15 trace earth elements in rocks could be carried out. Now, 15 elements, rare trace elements could be analyzed. Determination of major, minor and trace elements in geochemical exploration samples could also be carried out. These are the few examples to show you the versatility of the method ICP-AES.

How many types of different situations, different samples, yet ICP-AES is the answer. Simultaneous determination of germanium, tin, arsenic, antimony and bismuth by hydride generation of ICP-AES. Trace element analysis in pyrite, sulfide, arsenopyrite, molybdenite and cassiterite, monominerals separation, pre-concentration and determination of trace elements in natural waters.

Now, you see, so many varieties have been carried out by using ICP-AES. I had mentioned something about hydride generation, whether it is atomic absorption or it is ICP, the molecular species of such transition metals like germanium, antimony, tin, arsenic and bismuth need to be converted to 1 oxidation state. Normally, they would exist in either 2 and 4 oxidation state or 3 and 5 oxidation state in the solution.

Now, to be able to analyze, whether it is in 3 oxidation state or 5 oxidation state or whether it is the metal is in 2 oxidation state or 4 oxidation state, they must be brought down to one particular oxidation state and that is what is done. The higher oxidation state is reduced to the lower. So, then there is only one type of molecular species per element and that is done by the method of hydride generation method. It is applicable to atomic absorption as well as to ICP, both.

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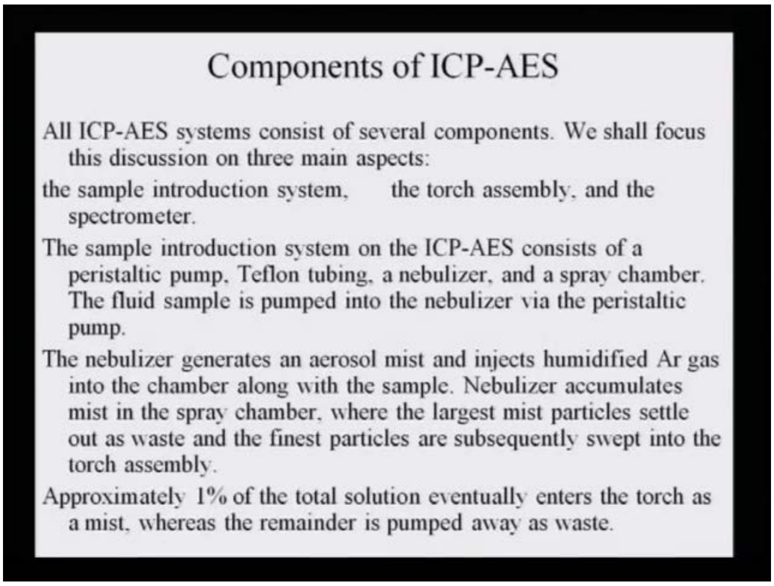

Components of ICP-AES

All ICP-AES systems consist of several components. We shall focus this discussion on three main aspects:

the sample introduction system, the torch assembly, and the spectrometer.

The sample introduction system on the ICP-AES consists of a peristaltic pump, Teflon tubing, a nebulizer, and a spray chamber. The fluid sample is pumped into the nebulizer via the peristaltic pump.

The nebulizer generates an aerosol mist and injects humidified Ar gas into the chamber along with the sample. Nebulizer accumulates mist in the spray chamber, where the largest mist particles settle out as waste and the finest particles are subsequently swept into the torch assembly.

Approximately 1% of the total solution eventually enters the torch as a mist, whereas the remainder is pumped away as waste.

Components of ICP and AES - all ICP-AES system consist of several components. We shall focus this discussion on three main aspects: the sample introduction system, the torch assembly and the spectrometer. So, if we look at any ICP machine, how does it differ from one machine to any another machine are only on three aspects. The sample introduction technique may be different or the plasma torch assembly may be slightly different or the spectrometer analytical part or the detector part can be different. The sample introduction system on ICP-AES consists of a peristaltic pump, teflon tubing, a nebulizer and a spray chamber. The fluid sample is pumped into the nebulizer via the peristaltic pump.

So, there is a very specialized method of sample introduction up to the nebulizer. There is a peristaltic pump which pushes and then it comes to the nebulizer where the aerosol is formed. this was not the situation when we looked at an ASS. so the sample introduction system is slightly modified and is more advanced and it uses teflon tubing. why because teflon is most un-reactive to most of the acidified solutions.

The nebulizer then generates an aerosol or mist and injects humidified argon gas into the chamber along with the sample. The nebulizer accumulates mist in the spray chamber, where the largest mist particles settle out as waste and the finest particles are subsequently swept into the torch assembly.

So, the role of nebulizer is to first form the aerosol mist, and that with the humidified argon gas are put into the chamber of with the sample. Then the nebulizer actually accumulates in that spray chamber and then the larger particles are going to the wall of the spray chamber, but the finer particles actually gets swept into the torch assembly.

Approximately, 1 percent of the total solution eventually enters the torch as mist, whereas the remainder is pumped away as waste. So, you see that how sensitive it is that even with 1 percent of the solution entering the torch, it is able to analyze as many as the analyte has as many elements as the analyte has in its composition.

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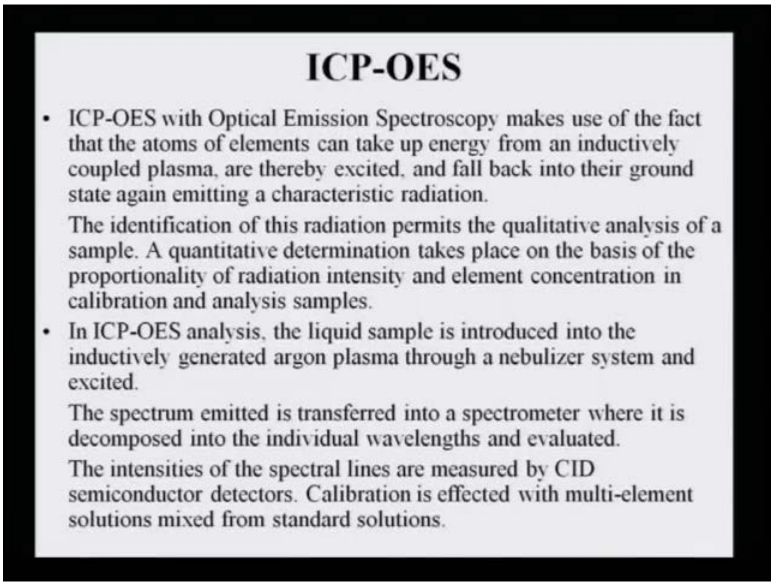

ICP-OES

- ICP-OES with Optical Emission Spectroscopy makes use of the fact that the atoms of elements can take up energy from an inductively coupled plasma, are thereby excited, and fall back into their ground state again emitting a characteristic radiation.

  The identification of this radiation permits the qualitative analysis of a sample. A quantitative determination takes place on the basis of the proportionality of radiation intensity and element concentration in calibration and analysis samples.
- In ICP-OES analysis, the liquid sample is introduced into the inductively generated argon plasma through a nebulizer system and excited.

  The spectrum emitted is transferred into a spectrometer where it is decomposed into the individual wavelengths and evaluated.

  The intensities of the spectral lines are measured by CID semiconductor detectors. Calibration is effected with multi-element solutions mixed from standard solutions.

ICP-OES - ICP-OES is optical emission spectroscopy, which makes use of the fact that the atoms of elements can take up energy from an inductively coupled plasma, are thereby excited and fall back into their ground state again, emitting a characteristic radiation.

So, here, it is the optically sensitive emission which is trapped and which is then correlated with the amount of analyte that has been present, which has caused this and it is done with the help of inductively coupled plasma. So, thereby, it excites and then falls back to the ground state and it emits a characteristic radiation.

So, it is as simple as that. Although the names are slightly different, but their functioning remains the same. The identification of this radiation permits the qualitative analysis of a sample. The quantitative determination takes place on the basis of proportionality of radiation intensity and element concentration in calibration and analysis sample.

So, one can do qualitative analysis as well as quantitative analysis and for doing quantitative analysis, calibration curve must be required because you need to have reference standard against which the sample will be analyzed, whether this much concentration is present or this much concentration is present. If there is no reference, if there is no scale, an absolute value cannot be given to the sample.

In ICP-OES analysis, the liquid sample is introduced into the inductively generated argon plasma through a nebulizer system and excited. So, here also, there is a use of nebulizer and there is this argon that produces the plasma flame and so, all other things remain the same. The spectrum emitted is transferred into a spectrometer, where it is decomposed into the individual wave lengths and evaluated. The intensities of the spectral lines are measured by CID, that is, the semiconductor detectors. The calibration is effected with multi-element solutions mixed with standard solutions.

So, you see that multi-element analysis can be carried out, but you need to have multi element standards for that. All the standards are mixed and a multi-element standard is prepared and then with the help of semiconductor detectors, it can be these spectral lines can be detected very efficiently.

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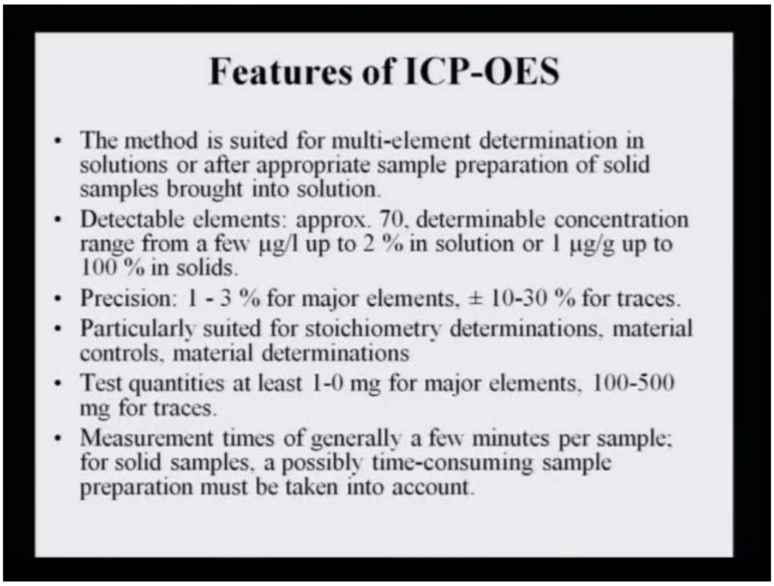

Features of ICP-OES

- The method is suited for multi-element determination in solutions or after appropriate sample preparation of solid samples brought into solution.
- Detectable elements: approx. 70, determinable concentration range from a few µg/l up to 2 % in solution or 1 µg/g up to 100 % in solids.
- Precision: 1 - 3 % for major elements, ± 10-30 % for traces.
- Particularly suited for stoichiometry determinations, material controls, material determinations
- Test quantities at least 1-0 mg for major elements, 100-500 mg for traces.
- Measurement times of generally a few minutes per sample; for solid samples, a possibly time-consuming sample preparation must be taken into account.

The features of ICP-OES - the method is suited for multi-element determination in solution or after appropriate sample preparation of solid samples brought into solution. So, it is both applicable to solid samples as well as appropriately dissolved samples can be prepared and multi-element determination can be carried out using ICP optical emission spectroscopy, that is, ICP-OES.

Detectable elements - approximately 70 determinable concentration range from a few microgram per litre to 2 percent in solution or 1 microgram per gram up to 100 percent in solids can be carried out very effectively. The precision - 1 to 3 percent for major elements, plus minus 10 to 30 percent for trace elements is the precision rate.

Particularly suited for stoichiometric determination, material controls, and material determinations. Tests quantities at least range of 1 to 0 milligram for major elements and 100 to 500 milligram for trace. So, these are the test sample range; that, even if you have very small quantities, major elements can be identified. If there are 100 to 500 milligram samples, even trace quantities can be identified. Measurement times of generally a few minutes per sample; for solid samples, a possibly time consuming sample preparation must be taken into account. Although in some cases the sample preparation time is a little longer, but that does not deter because the accuracy, the precision, the kind of range of 70 possibility of doing 70 element analysis in one go is the attraction for using ICP.

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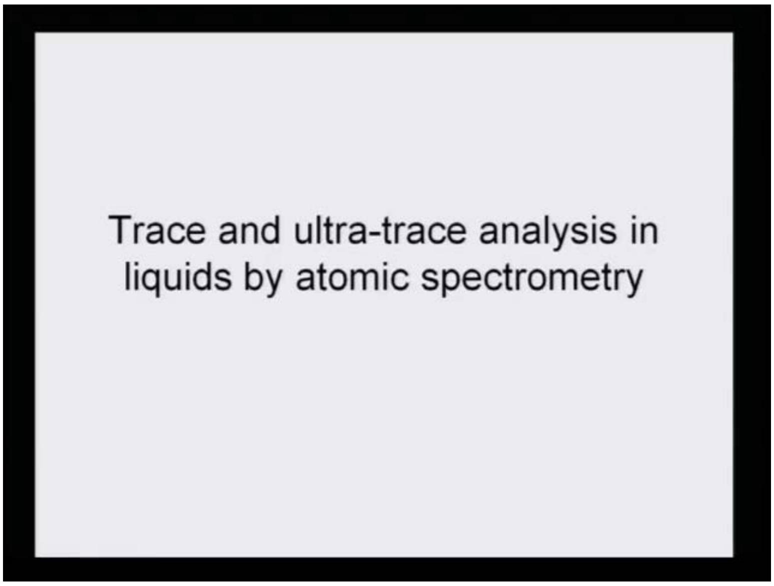

Trace and ultra-trace analysis in liquids by atomic spectrometry

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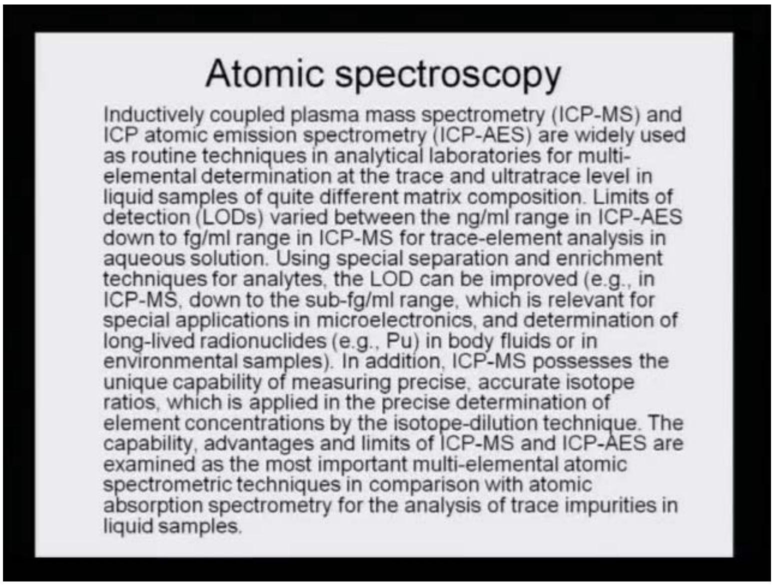

Atomic spectroscopy

Inductively coupled plasma mass spectrometry (ICP-MS) and ICP atomic emission spectrometry (ICP-AES) are widely used as routine techniques in analytical laboratories for multi-elemental determination at the trace and ultratrace level in liquid samples of quite different matrix composition. Limits of detection (LODs) varied between the ng/ml range in ICP-AES down to fg/ml range in ICP-MS for trace-element analysis in aqueous solution. Using special separation and enrichment techniques for analytes, the LOD can be improved (e.g., in ICP-MS, down to the sub-fg/ml range, which is relevant for special applications in microelectronics, and determination of long-lived radionuclides (e.g., Pu) in body fluids or in environmental samples). In addition, ICP-MS possesses the unique capability of measuring precise, accurate isotope ratios, which is applied in the precise determination of element concentrations by the isotope-dilution technique. The capability, advantages and limits of ICP-MS and ICP-AES are examined as the most important multi-elemental atomic spectrometric techniques in comparison with atomic absorption spectrometry for the analysis of trace impurities in liquid samples.

Coming to yet another very important topic is the trace and ultra-trace analysis in liquids by atomic spectrometry. So far, we have seen the advantages of using AAS or ICP and at what point of time, the analyst must make this choice is a very crucial decision. Atomic spectroscopy, whether it is inductively coupled plasma mass spectrometry, that is, ICP-MS or ICP atomic emission spectrometry, that is, ICP-AES are widely used as routine techniques in analytical laboratories for multi-elemental determination at trace and ultra-trace levels in liquid samples of quite different matrix composition.

Limits of detection, that is, the LODs varied between the nanogram per millilitre range in ICP-AES down to fentogram per ml range in ICP-MS. For trace element analysis in aqueous solution, using special separation and enrichment techniques for analytes, the limit of detection can also be further improved. That is in techniques for analytes which like ICP-MS, it can go down to even sub fentogram per ml range, which is relevant for special applications in microelectronics and determination of long radio nuclides in body fluids or in environmental samples.

So, you see as and when the importance and the relevance of analyzing trace and ultra-trace, you see sub fentogram in 1 ml is a very, very small quantity of analyte and when there was a necessity to analyze, even at that range, there is an answer by ICP-MS or ICP-AES method which can do so.

In addition, ICP-MS possesses the unique capability of measuring precise, accurate isotope ratios, which is applied in the precise determination of element concentrations by isotope dilution technique. The capabilities, advantages and limit of ICP-MS and ICP-AES are examined as most important multi-elemental atomic spectrometric techniques in comparison with atomic absorption spectrometry for the analysis of trace impurities in liquid samples.

You see, that is the reason why I went on to explain to you that what to use where because ideally a flame absorption spectrometer is well suited for routine analysis at ppm level, but when fentogram or nanograms have to be analyzed in a liquid sample, there has to be certain modification in the machine and the analytical method, otherwise they cannot be analyzed quantitatively, although they may be analyzed qualitatively. Presence or absence is not a big deal to find out, but to find out exact concentration of its presence in a sample is a big challenge for the analyst.

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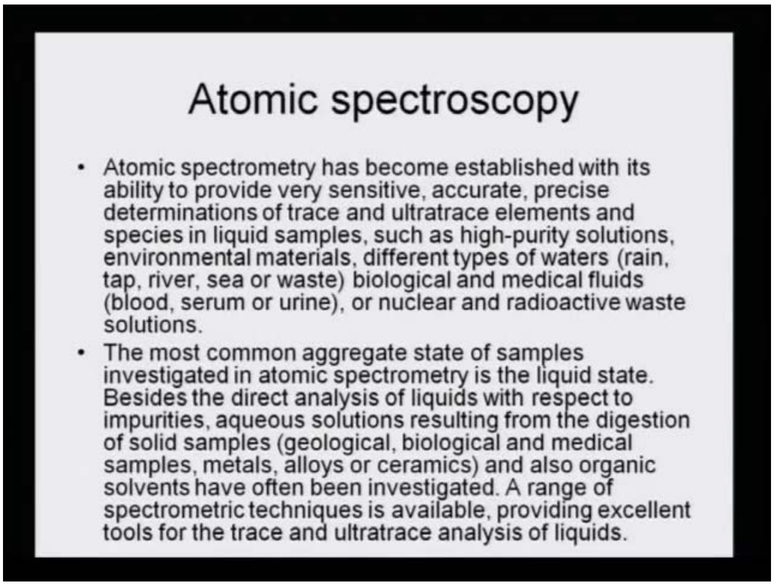

Atomic spectroscopy

- Atomic spectrometry has become established with its ability to provide very sensitive, accurate, precise determinations of trace and ultratrace elements and species in liquid samples, such as high-purity solutions, environmental materials, different types of waters (rain, tap, river, sea or waste) biological and medical fluids (blood, serum or urine), or nuclear and radioactive waste solutions.
- The most common aggregate state of samples investigated in atomic spectrometry is the liquid state. Besides the direct analysis of liquids with respect to impurities, aqueous solutions resulting from the digestion of solid samples (geological, biological and medical samples, metals, alloys or ceramics) and also organic solvents have often been investigated. A range of spectrometric techniques is available, providing excellent tools for the trace and ultratrace analysis of liquids.

Atomic spectrometry has become established with its ability to provide very sensitive, accurate, precise determination of trace and ultra-trace elements, and species in liquid samples such as high purity solutions, environmental materials, different types of water that is rain water, tap water, river water, sea water and waste water, biological and medical fluids like blood serum or urine, or nuclear and radioactive waste solutions. So, you see there is a huge variety where these ICPs can be used very effectively, very accurately, very sensitively and precise determination can be brought about.

The most common aggregate state of samples investigated in atomic spectrometry in liquid state. Besides the direct analysis of liquids with respect to impurities, aqueous solutions resulting from digestion of solid samples, that is geological samples, biological samples, medical samples, metal alloys and ceramics and also organic solvents have often been investigated. A range of spectrometric techniques is available providing excellent tool in the trace and ultra-trace analysis of the liquids. So, it has covered the entire range of very sensitive areas like the medical, the geological, the biological, metal alloys, ceramics and all can be analyzed by these ICPs very effectively.

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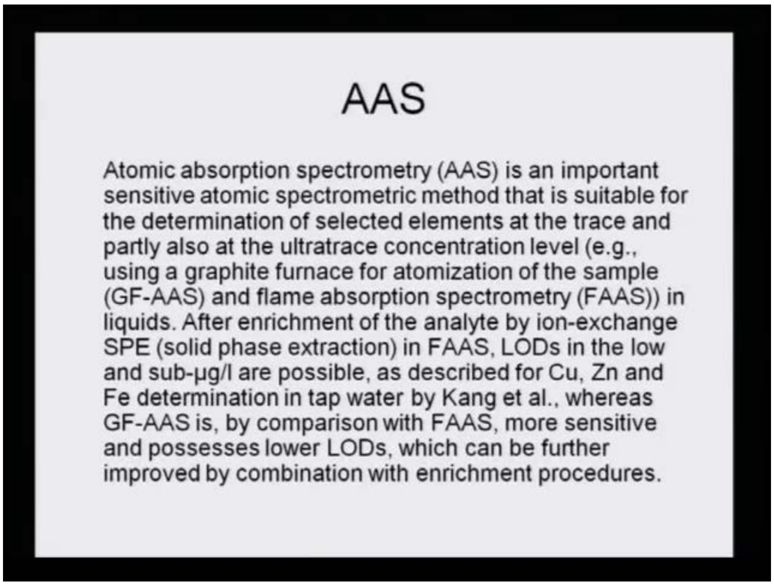

AAS

Atomic absorption spectrometry (AAS) is an important sensitive atomic spectrometric method that is suitable for the determination of selected elements at the trace and partly also at the ultratrace concentration level (e.g., using a graphite furnace for atomization of the sample (GF-AAS) and flame absorption spectrometry (FAAS)) in liquids. After enrichment of the analyte by ion-exchange SPE (solid phase extraction) in FAAS, LODs in the low and sub-μg/l are possible, as described for Cu, Zn and Fe determination in tap water by Kang et al., whereas GF-AAS is, by comparison with FAAS, more sensitive and possesses lower LODs, which can be further improved by combination with enrichment procedures.

AAS of course, you have seen that the flame AAS is not very sensitive method. However, it is sensitive enough to analyze that is suited for the determination of selected elements at trace and partly also, at the ultra-trace concentration, if we are using graphite furnace. So, even the class of AAS, the GF-AAS, that is, the graphite furnace atomic absorption spectroscopy is the answer, but flame atomic absorption, that is, the FAAS is not really suited for trace and ultra-trace.

After enrichment of the analyte by ion exchanger, that is, the solid phase extraction, although the flame AAS also can be upgraded or the sample is enriched. So, it can come within the limits of detection of the FAA. Limits of detection in the low and sub microgram per litre are possible as described in the determination of copper, zinc and iron in tap water by Kang et al. He has shown that this is possible, whereas graphite AAS is by comparison with AF-AAS, more sensitive and possesses lower limit of detections, which can be further improved by combination with enrichment procedures.

So, there is a possibility to be able to use flame AAS, if the sample has gone through an enrichment procedure and what is that enrichment procedure? The enrichment procedure requires an ion exchange solid phase extraction to be added on to the analytical method.

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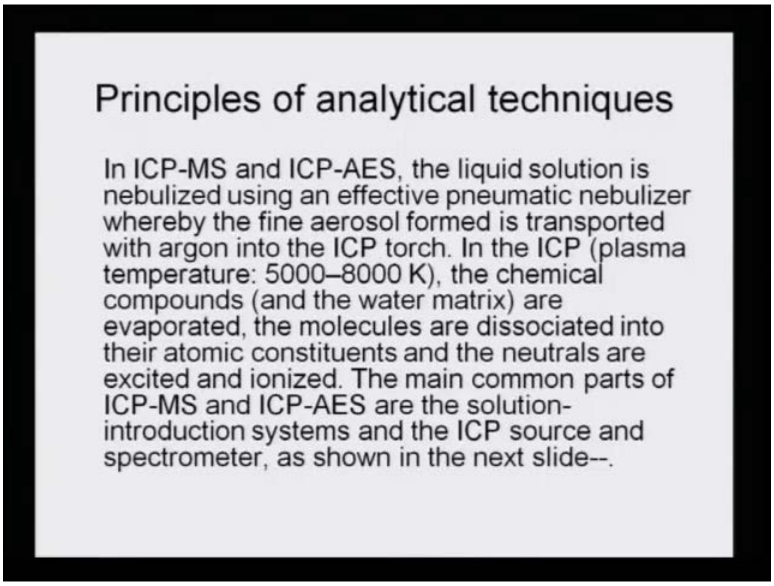

Principles of analytical techniques

In ICP-MS and ICP-AES, the liquid solution is nebulized using an effective pneumatic nebulizer whereby the fine aerosol formed is transported with argon into the ICP torch. In the ICP (plasma temperature: 5000–8000 K), the chemical compounds (and the water matrix) are evaporated, the molecules are dissociated into their atomic constituents and the neutrals are excited and ionized. The main common parts of ICP-MS and ICP-AES are the solution-introduction systems and the ICP source and spectrometer, as shown in the next slide--.

Principles of analytical techniques - in ICP-MS and ICP-AES, the liquid solution is nebulized using an effective pneumatic nebulizer whereby the fine aerosol forms is transported with argon into the ICP torch. So, instead of a peristaltic pump an on ordinary nebulizer, here there is a pneumatic nebulizer; the rest remains the same.

The formation of the aerosol and getting transported to the argon torch and all that remains the same. The ICP at the plasma temperature is at 5000 to 8000 kelvin. The chemical compounds and the water matrix are all evaporated at this temperature and then the molecules are dissociated into their atomic constituents and the neutrals are excited and ionized. The main common parts of ICP-MS and ICP-AES are the solution introduction systems and the ICP source and the spectrometer and that we will see in a little while in this slide.

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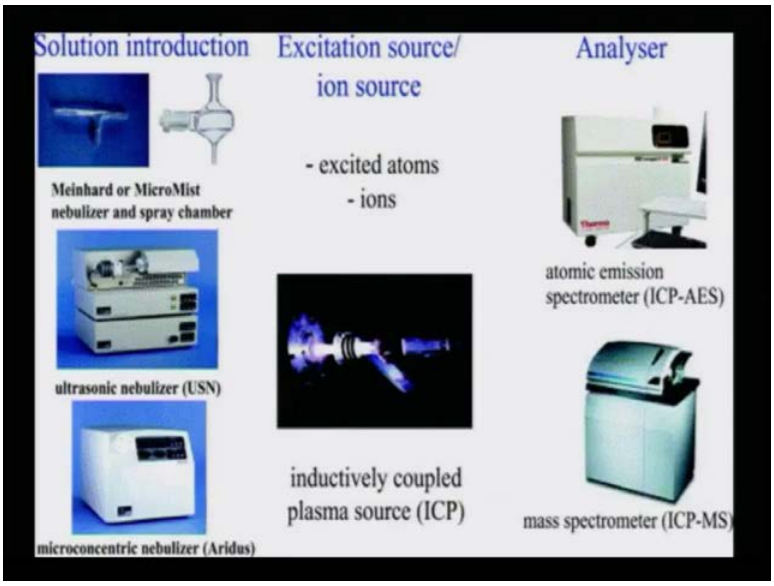

Solution introduction

Meinhard or MicroMist nebulizer and spray chamber

ultrasonic nebulizer (USN)

microconcentric nebulizer (Aridus)

Excitation source/ ion source

- excited atoms
- ions

inductively coupled plasma source (ICP)

Analyser

atomic emission spectrometer (ICP-AES)

mass spectrometer (ICP-MS)

Different types of models and different types of its adaptation are required and therefore, the machine can then be used. You see, this is an ICP-MS machine; this is a simple ICP. When you have a microconcentric nebulizer, how it looks like; if it has ultrasonic nebulizer, how the machine looks like and so on and so forth.

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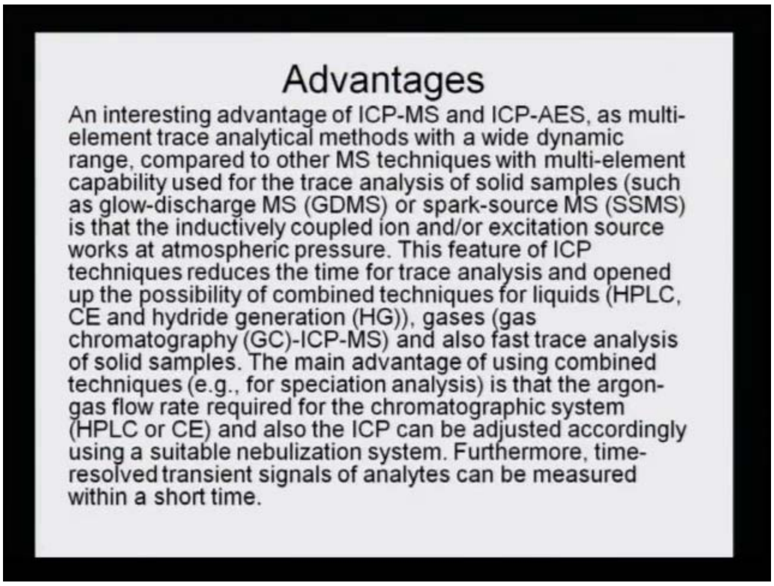

Advantages

An interesting advantage of ICP-MS and ICP-AES, as multi-element trace analytical methods with a wide dynamic range, compared to other MS techniques with multi-element capability used for the trace analysis of solid samples (such as glow-discharge MS (GDMS) or spark-source MS (SSMS) is that the inductively coupled ion and/or excitation source works at atmospheric pressure. This feature of ICP techniques reduces the time for trace analysis and opened up the possibility of combined techniques for liquids (HPLC, CE and hydride generation (HG)), gases (gas chromatography (GC)-ICP-MS) and also fast trace analysis of solid samples. The main advantage of using combined techniques (e.g., for speciation analysis) is that the argon-gas flow rate required for the chromatographic system (HPLC or CE) and also the ICP can be adjusted accordingly using a suitable nebulization system. Furthermore, time-resolved transient signals of analytes can be measured within a short time.

Advantages - an interesting advantage of ICP-MS and ICP-AES, as multi-element trace analytical method with a wide dynamic range compared to other MS techniques with multi-element capabilities used for the trace analysis of solid samples such as glow discharge MS, that is GDMS or spark-source MS (SSMS) is that the inductively coupled ion and/or excitation source works at atmospheric pressure. So, these work at a very ambient atmospheric pressure.

This feature of ICP technique reduces the time of trace analysis and opened up the possibility of combined techniques for liquids, that is HPLC capillary electrophoresis and hydride generation, gases, that is gas chromatography can be coupled to ICP-MS and also, fast trace analysis of solid samples. The main advantage of using combined techniques for speciation analysis is that the argon gas flow rate required for the chromatographic system, that is HPLC or capillary electrophoresis and also, the ICP, can be adjusted accordingly using a suitable nebulization system. Furthermore, time resolved transient signals of analytes can be measured within a short time.

Now, it is also possible that there is, you know a hyphenated machine, where the HPLC or capillary electrophoresis or hydride generation can be coupled with the ICP-MS or ICP-AES; that adds on to further more advantage. It can also be coupled with GC and so, if the gas sample is gaseous, then the gaseous samples can be handled very easily. Therefore, it is one of the best methods of analysis of both solids and liquids and as well as gaseous samples.