

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

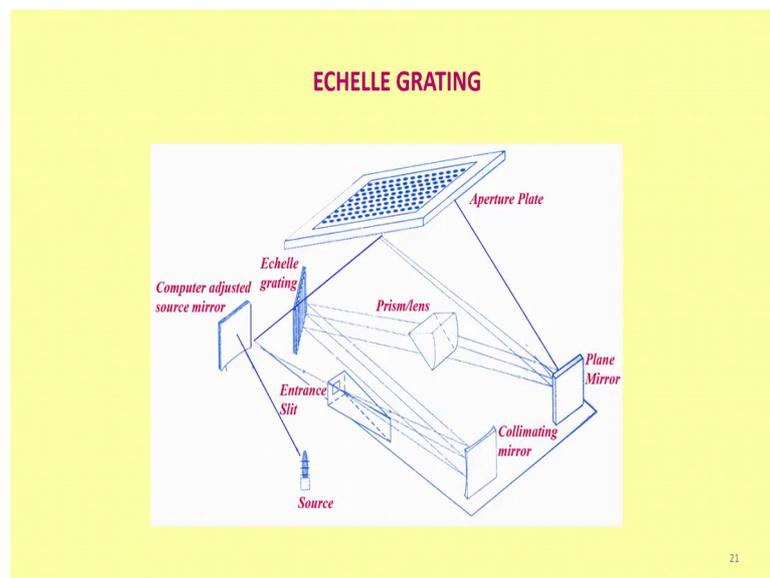
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Lecture – 12

Instrumentation for ICP AES- Optical mountings

So, friends, we have been discussing about the optical mountings in ICP. It is used for the separation of electromagnetic radiation coming from the elements we want to analyze. So, I had said the electromagnetic radiation can be separated, that means, we can choose the emission wavelength corresponding to each element using gratings, prisms etcetera.

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And, here I am showing you a grating arrangement that is known as Echelle grating. Echelle is the name of the scientist who has developed this. I have already taught you the principles of grating, how a grating works or a prism works etcetera. We will assume that we have already learned. Basically, what a grating does is it contains a number of prisms, you take a glass plate like this and then keep on diamond tool and keep on ruling them and for per centimetre you can rule about 30000 rulings per centimetre; that means, each cut is a dip in the on the surface. So, a radiation falling perpendicularly on that will be hitting a small prism and then it will be the radiation coming out will be going in they reflect getting reflected because the bottom portion is mirrored. If the bottom is mirrored

it will fall here and then get reflected according to the laws of reflection and also refraction because if the grating material has to pass through the grating material, it will act like a prism.

So, depending upon the prism angle where I collect the radiation different wavelengths I can pick up that is the fundamental principle of a grating; that means, instead of using one prism one big prism I am using 30000 prisms per centimetre. One big glass like this mobile on this I keep on ruling cutting a diamond tool and then I let the radiation for come like this and it goes off like this and I put a slit here, a small opening, it will collect only the radiation coming through that opening I move that slit another wavelength will come I move that I am slit another place another wavelength will come. So, this is how the grating works.

Now, the Echelle grating is a simple grating with about 70 to 80 prisms per centimetre, 70 to 80 lines. So, here the arrangement is very simple I have a source light here this is my plasma from the plasma excited atoms generate radiations. So, those radiations I collect all of them on a mirror, this is the mirror. So, this mirror is a concave mirror. So, what does a concave mirror do? It concentrates and makes an image look smaller. You imagine your car window outside the car window there is a small mirror that shows things which are behind in very sharp image, that is a concave mirror. So, a concave mirror is bulging inside and mirror mirrored outside. So, that all the radiations are collected in this and then reflected in a parallel beam of radiation. So, it may be going here and there etcetera, but once they fall all of them will give you a parallel beam of light.

So, this concave mirror adjusts it is computer move your movements slight moment is possible by computer servo mechanical computers are there. So, the radiation coming from the source is collected onto this mirror, from there it comes passes through a mechanical block a small piece of metal with a small hole, that is, what here I have doing it now this is a ball this is a metal plate with a small hole in that, so that means, I am not collecting all the radiation that comes out only a small portion, but that is parallel beam. So, this is the job of this slit entrance slit. So, from the entrance slit it comes to another mirror that is also concave and then it renders the reflected beam parallel you can see different lines I am showing you 3 lines which are parallel. So, this is known as Echelle grating.

So, from the concave mirror it comes here through the entrance slit and then to collimating mirror onto the Echelle grating and from the grating I take it through a prism or a lens and collect the radiation coming out from onto a plane mirror and from this plane mirror I have an aperture plate again by choosing this end I get one wavelength this end, another wavelength an external slot another wavelength like that I get different wavelengths. So, if I have copper I need to separate 324.7 nanometre. So, this slot if I move this mirror aperture plate a little bit so that this line matches with that 324, I get only 324, nothing else. All other things are blocked.

Suppose, I need 550, I just have to move this plate a little bit this side that side I get 550, like that Echelle grating is one of the most important optical arrangement in a spectrometer any spectrometer whether it is a spectrophotometer or IR spectrometer and so many other places Echelle grating is used nowadays. So, this is one way of arranging the radiation.

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CONCAVE GRATINGS

In Rowland mounting, the grating and the detector are attached to a rigid bar that moves relative to a fixed entrance slit. The angle of diffraction remains constant and the angle of incidence varies. The detector is always kept in a position normal to the grating. Under these conditions, the dispersion is linear over a broad wavelength range.

In Abney mounting, grating and detector are fixed and the slit moves along the axis of the Rowland circle. This is slightly cumbersome because light source and external optics also need to be repositioned when the angle of incidence is changed.

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Then, I have also said that we use concave gratings correct. So, in concave grating the grating and aperture needs to be adjusted, because the aperture is where I get different wavelengths correct. The grating will give you different wavelengths the aperture has to be moved to get what I want. So, that is known as Rowland mounting. So, why I need to collect this radiation coming through the grating because I outside that I want to put a hole in the aperture across that I want to put my detector. So, the radiation falling on the

detector will generate current. So, the detector also has to move along with the aperture, only if I move the aperture only radiation I will get I do not know how much of it is coming out, but if I put a detector along with the aperture I can move it slowly get 550, 450, 350 like that different wavelengths has to be coupled with the detector also.

So, the for a given grating the aperture has to move at the focal length because that is where all the radiations are coming out and that focal length along the focal length I get different wavelengths. That focal length in general describes a circle, then different along the circle if I move the aperture and a detector I get different wavelengths. So, this circle which gives me different wavelengths is known as Rowland circle, that is, the name of the scientist who devised this method for collecting the radiation coming out from the excited atoms emissions.

So, in Rowland mounting that is known as Rowland circle and Rowland mounting aperture and detector the grating and the detector are attached to a rigid bar. You must have seen in railway engines when the railway moves in the engine there is a axle which moves like this like that, but the train moves further isn't it. So, you go and observe the railway engine when it is moving it does not describe the engine piston does not axle does not describe a circular motion. It just gives a repeated motion, but that is converted into a rolling motion. So, the train whole train moves.

Similarly, here I put a bar along the mountain, the bar moves the aperture as well as detector will keep on moving, I keep on moving the bar they will also move. So, the bar moves relative to the fixed entrance slit. So, the angle of diffraction remains constant and the angle of incidence varies say radiation is coming like this if I move it like this it will hit.

Here if I move it like this it will hit here because this is fixed from where the source is coming and I change it another wavelength will come, like that I get different wavelength by moving the grating do you understand. So, the angle of incidence varies the detector is always kept in position normal to the grating, aperture grating everything is fixed. So, under these conditions dispersion is linear over a broad wavelength range. This is how we get different wavelengths in an ICP spectrometer optical bench that is Rowland circle.

So, there is another mounting called as Abney mounting that is name of another scientist. Here, what happens grating and detector are fixed grating is also fixed detector is also fixed. So, what has to move the slit has to move; that means, I have so many detector fixed. So, I want to collect wavelength grating is also fixed this does not move. So, I have the radiation coming here, detector is fixed, but the slit I can move; that means, I need to have number of slits. I can choose the slit.

The simple moment of the slit itself gives me different wavelengths, is it not? Very nice arrangement, beautiful and the slit moves along the Rowland circle only; that means, with a given slit I can measure put one detector, another slit I can put one more detector another wavelength I can put one more detector with another wavelength; that means, simultaneously I can measure 3 different wavelengths if those 3 different wavelengths correspond to 3 different elements I can do multi element analysis straightaway is it not? Beauty of ICP is you can do with a single sample multi-element analysis if you use Abney mounting.

So, when you buy an ICP the manufacturer will ask you sir would you like to do a single element or multiple elements for example, if you are in a alloy factory producing an alloy making mag wheel you know you must have heard mag wheel. Mag wheel is an alloy used in car wheels. It is a special magnesium aluminium alloy car wheels are fixed with that to tires. So, that is known as mag wheel.

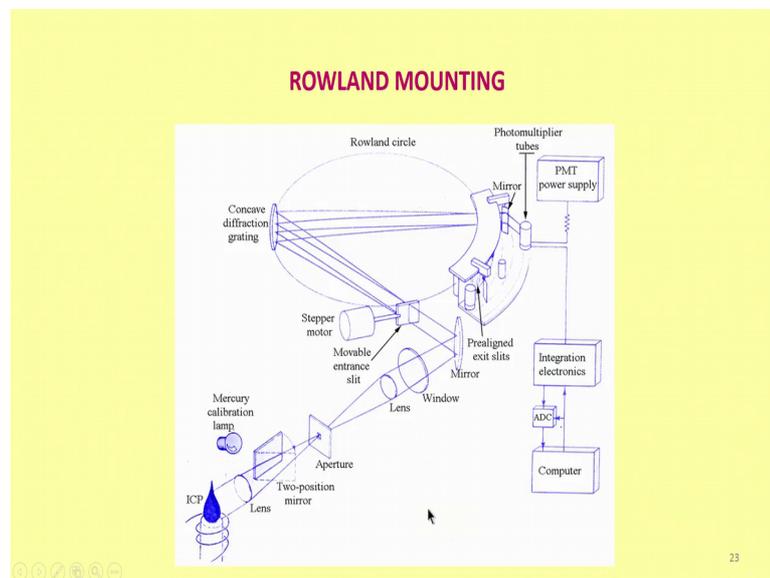
So, that mag wheel if somebody is engaged in the production somebody wants to know how much of magnesium is there how much of aluminium is there how much of many other elements are there. So, the production man in a factory he is not interested in research, but every day he wants to produce mag wheel of the same composition. So, instead of doing one element at a time he can do all the 3 elements he will say give me multi element capability with a ICP I say. So, the manufacturer will suggest, sir, you buy one Abney mounting so that you can do simultaneous analysis without any problem.

But, there is one catch. If your factory is producing mag wheel and you want to analyze the aluminium, chromium and this thing 3 elements may be 5 elements you cannot do any other element because the slit is also fixed. Tomorrow you want to produce some other alloy containing tungsten, he will say, sorry sir I cannot give you that, is it not? But, if you buy Echelle grating you can choose any wavelength corresponding to any

element. So, multi element analysis is done using Abney mounting, but if you want to if you are a research R and D like Indian institute of science my student is working on magnesium, another is working on chromium somebody else is working on tungsten. So, all of them want to use the same instrument. They cannot go for Abney mounting is it not. So, both systems have got advantages as well as disadvantages, yet it depends upon your requirement.

So, in Abney mounting rating and detector are fixed and the slit moves along the axis of the Rowland circle. Rowland circle is most important in all ICP, Inductive Couple Plasma analysis without Rowland circle there is no existence. Only thing is arrangement of slit, arrangement of detector and arrangement of incident angle, these 3 are the most important parameters to be decided when you want to buy a ICP – AES system. So, this is multi simultaneous analysis slightly cumbersome because light source and external optics also need to be repositioned whenever the angle of incidence is changed. So, it is a little tricky, but those who need it will definitely love it.

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So, this is the arrangement of Rowland arrangement. So, here I have an ICP source, that is, a conical arrangement with plasma torch and all that is the end of that torch and then I have a small lens here to make the lines parallel radiation parallel and they may have a 2 position mirror to adjust the angle and then I have a mechanical aperture here, to take out only a small portion of the emission light because plasma has got lot of radiations I do

not want to collect all necessary radiation, only a small portion of that radiation I want to collect and then again that collected radiation must be rendered parallel and lens after that I need a window to quartz window you know they are not glass, quartz.

And then I have a pre aligned mirror so that I can move the radiation towards the diffraction grating because this is one arrangement from there it enters the it has to enter the Rowland circle. So, it has to move towards the grating and to fix it towards entrance sometimes I said entrance slit changes. So, I have a movable entrance slit here. So, from the pre aligned exit slit this is the stepper motor, I can move it 0.01 millimetre, 0.02 millimetre like that I should be able to move the entrance slit and then on to the concave diffraction grating.

Why concave diffraction grating? Because, again the radiation coming out should be parallel. So, we do not want stray radiation stray radiation means loss of intensity, loss of detection limit, loss of signal. So, many things they are not worth it. So, I need a parallel radiation and then here I have an arrangement here you see this portion mirror are these that etcetera and then there is a detector here, photomultiplier tube. I have taught you about photomultiplier tubes detector earlier and nowadays lot of people use photodiode array detectors also diode array detectors. I will teach you more about diode array detectors when I will take a course on infrared spectroscopy.

And, then there is a mirror arrangement the radiation has to come out fall onto the multi photomultiplier tube, what does it do? It generates current depending upon the wavelength that is coming out. So, every electric electromagnetic radiation when it falls on the detector it has to generate current and that current is proportional to the concentration of the element that is present in the sample that is in the ICP plasma here. So, from here to here there is PMT, photomultiplier tube.

Everything is controlled and everything is related to Rowland circle and from this Rowland circle, I have a photomultiplier tube attached to that and then I need a power supply for photomultiplier tube that is also required and then I need to amplify that signal sometimes signal is I have to amplify without amplifying the noise. If the noise also increases signal also increases there is no way I can make out. So, that noise should become less signal should increase.

So, for that I needed an integration electronics and ADC – analog-to-digital converter. This is another important concept in spectrographic of all spectroscopic instruments normally a radiation falling on that will generate current and how much current there that has to be cut into different small portions and converted into some number that is known as the ADC – analog-to-digital converter, totally 100 percent cut into 100 and then count how many of the 100 is coming out, that is, in general ADC – analog-to-digital converter it is available in SP road in Bangalore and any electronic shop you will be able to get ADC ready made available and then that ADC should be connected to the computer as I showed you in the first slide of this session. So, this is how the Rowland mounting arrangement we should understand and it is a beautiful arrangement, lovely.

So, there are other mountings also which is becoming a little popular one is Eagle mounting. So, in Eagle mounting that is another scientist named slit and detector are mounted on the same side of the grating normal to all components of the Rowland circle. Go back, the slit is here and detector is here and the grating is this side on the same side this here it is opposite side the slit and mounting are put behind this, on the same side. You see this grating concave diffraction grating backside of this slit and mount detector are fixed instead of opposite facing it I am putting them behind.

So, if I put them behind the instrument becomes smaller I do not have to have one more equipment multiple this thing this much distance saved now this much of distance equipment space is saved. If I put them detector and it is thing here if I put it this side the instrument becomes compact instead of putting it opposite side. So, Eagle mounting is slightly better than that.

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In Eagle mounting, slit and detector are mounted on the same side of the grating normal to all components on the Rowland circle. This is a compact arrangement.

The Paschen – Runge mounting has slit, grating and detector at fixed positions on the Rowland circle. Most of the commercial direct reading instruments are of this type.

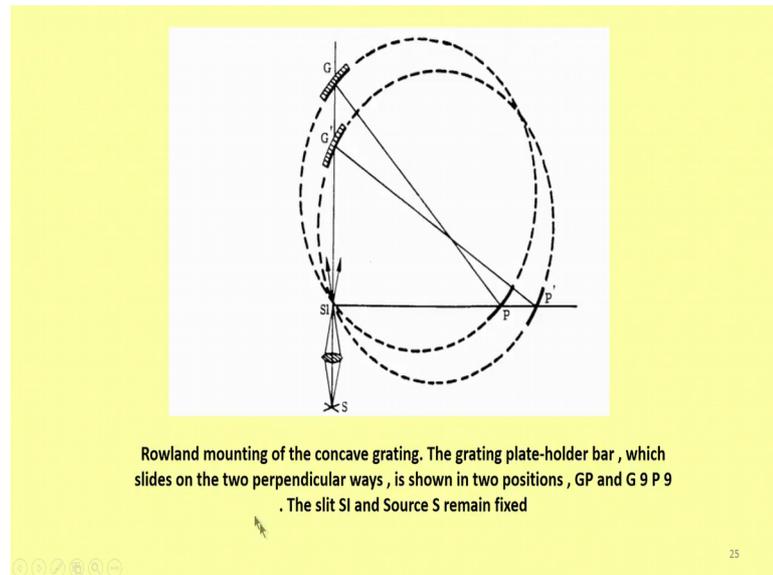
In Wadsworth mounting, a spherical mirror is used to collimate the incident beam which illuminates the grating with a parallel light. When the wavelength region is changed, the detector to grating distance must be changed to maintain proper focus of the diffracted light.

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And, then there is another mounting that is Paschen – Runge mounting. This has slit grating and detector at fixed positions again; that means, simultaneous multi-element analysis, why? Slit is fixed, rating is fixed, detector is fixed you cannot change factory fixed and then useful only for multi element analysis most of the commercial direct trading elements instruments are of this type nowadays.

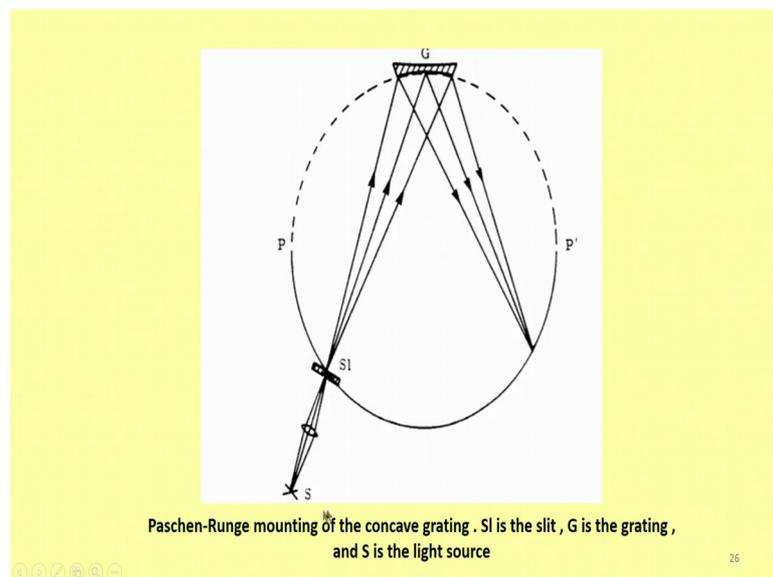
There is another mounting known as Wadsworth mounting. Here, a spherical mirror is used to collimate the incident beam spherical mirror round mirror not concave mirror; very big round mirror, that eliminates the grating with a parallel light. When the wavelength region is changed detector to grating distance must be changed to maintain proper focus. So, the slightly involved arrangement not so popular, but it is another arrangement either way.

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So, this is Rowland mounting of the concave grating schematic. See this the grating plate holder bar, this is the grating plate holder bar and which slides on the 2 perpendicular ways is shown in 2 positions one is here another is here P and P dash GP and G9 the slit si and source s remain fix this one source is fixed this is fixed and then these 2 plates are fixed and then detector should be at P and P dash you get it.

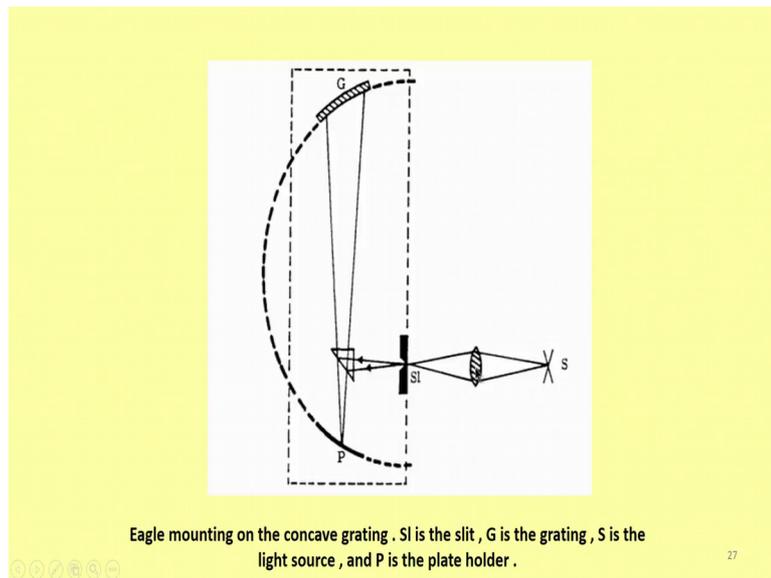
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So, this is a Paschen – Runge source round mirror and then slit and then grating and this thing. You move the grating angle will be different here, different wavelengths you will

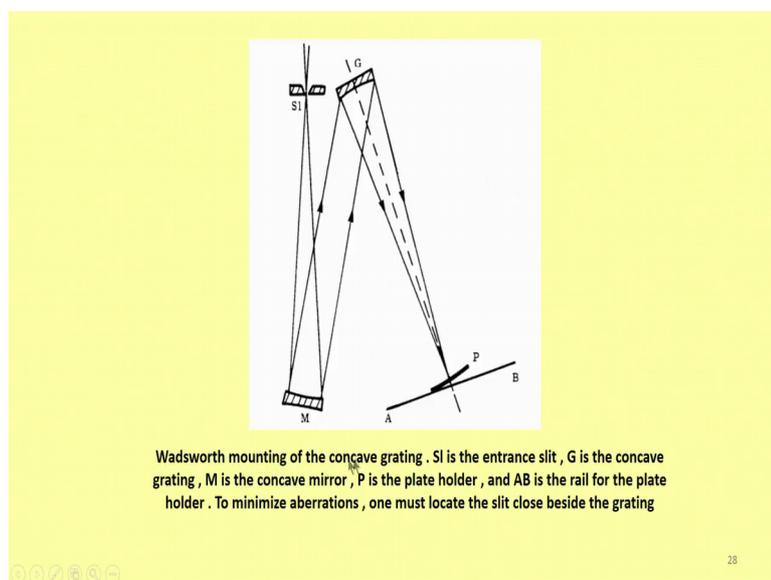
get. Here, it is Eagle mounting source is here S1 slit, this is lens. When you use lengths and when you use mirror these always a question in most of the optical mountings. So, you want all the radiation passing through, use a lens if you use only a small portion use a mirror and take the reflected light. So, it is a very well known physics principle.

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And, then here I have a prism and then gratings are here. So, comes here, goes there and then detector grating finish.

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Here it is Wadsworth mounting, entrance slit where is entrance slit. Here, S1 this is the entrance slit and then G is the concave mirror grating m is the concave mirror from here it comes to the concave mirror, parallel radiation, grating and then detector. P is the plate holder and AB is the rail for plate holder this is the plate holder; that means, you do not have to move the plate holder, if you move it will move this thing. So, that this control of AB gives you a sort of special control to move them small distances.

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PLANE GRATINGS

The Ebert – Fastie and Czerny turner mountings utilize a plane grating in combination with a spherical mirror to collimate the incident beam and to focus the diffracted light on the exit slit or detector. The incident beam passes over the grating and the diffracted light passes under the grating. Since the detector is near normal to the grating plane, the dispersion is approximately linear.

The Czerny –Turner mounting differs from the Ebert – Fastie in that the spectrometer has two spherical mirrors. These are used in combination with array detectors which have replaced photographic emulsions for the measurement of spectra.

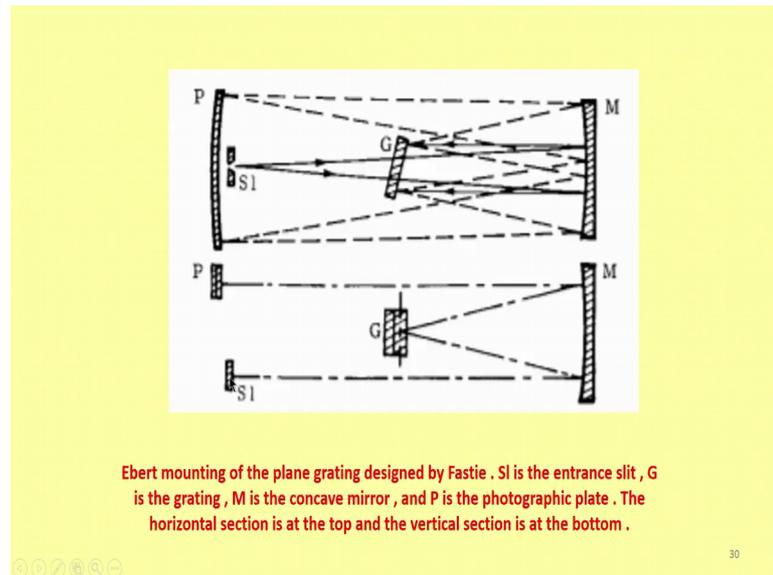
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Sometimes we do use plane gratings also not necessarily concave gratings. Concave gratings have got a way of giving you parallel output; whereas, in plane gratings there are 2 very popular grating arrangement that is Ebert – Fastie and Czerny turner. Nowadays 90 percent of the spectrophotometers or spectrometers they all come with Czerny turner mounting and they utilize a plane grating in combination with a spherical mirror to collimate the incident beam and to focus the diffracted light on the exit slit. If I tell you all this it would not be you will be tired to know what it means, is it not? But, is easy to understand spherical mirror collimation and then incident beam diffracted light and then it has to fall on the slit and detector.

The incident beam passes over the grating and diffracted light passes under the grating, incident beam passes over the grating diffracted beam it need not pass through again I collect it below make that thing compact. So, since the detector is near normal to the grating line the dispersion is approximately linear.

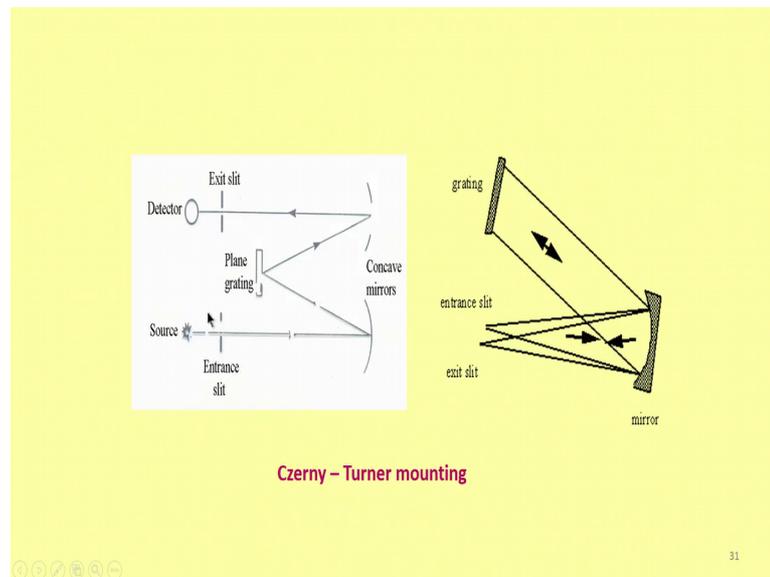
In Czerny turner mounting that differs from slightly in that the spectrometer has 2 spherical mirrors. These are used in combination with array detectors which have replaced photographic emulsion plate you do not need experience in ICP nowadays. You just have to read these from the computer because the photographic emulsion it requires experienced eye you do not need it the computer will do that job.

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So, this is Ebert mounting. S1 concave mirror grating and goes here and then single mirror and this is also essentially same here concave mirror grating comes out on the same side now.

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In Czerny turner mounting source here one mirror plane grating another mirror out. So, I can move this slit anywhere, slit end detector along the Rowland circle.

So, now, that is about the different mountings in optical mountings in ICP I think this information will be very useful if you wish to buy an instrument normally people are not allowed to clean the grating etcetera because that requires special skill, but if you know the arrangement, I am sure it will help you in deciding which optical mounting you should go for ICP instrumentation. So, we will continue our discussion on the detection of electromagnetic radiation in our next class.

Thank you very much.