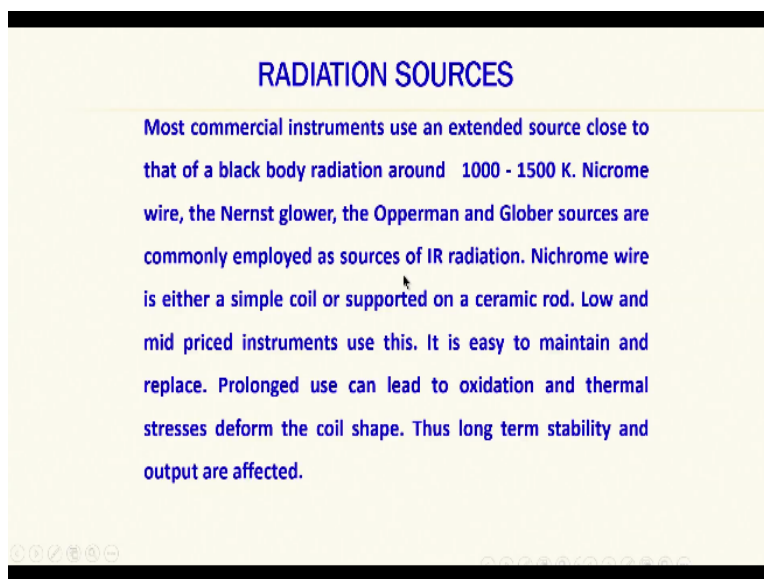


**Infrared Spectroscopy for Pollution Monitoring**  
**Prof. J.R. Mudakavi**  
**Department of Chemical Engineering**  
**Indian Institute of Science-Bangalore**

**Lecture-18**  
**Instrumentation in IR**

So far we have discussed about the optical system and sample handling ok. Now we will continue our discussion about the radiation sources in infrared.

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**RADIATION SOURCES**

Most commercial instruments use an extended source close to that of a black body radiation around 1000 - 1500 K. Nicrome wire, the Nernst glower, the Opperman and Globber sources are commonly employed as sources of IR radiation. Nichrome wire is either a simple coil or supported on a ceramic rod. Low and mid priced instruments use this. It is easy to maintain and replace. Prolonged use can lead to oxidation and thermal stresses deform the coil shape. Thus long term stability and output are affected.

Now most of the commercial instruments used an extended source close to that of a black body radiation, similar close to means similar to black body radiation around 1000 to 1500 Kelvin that means you take any substance black substance material heated 1000 to 1500 degree Kelvin. So that is about 1300 to -make it -273 that makes it around 800 to 1200 degree centigrade that will make the material glow red and any material that is glowing red.

Because of the heat it throws out infrared radiation that is the assumption. So most of the black body radiation materials around 1000 to 1500 give out infrared radiation, I can use any of them. So what are those such materials are nicrome wire that is Nickel chromium Alloy nicrome wire I can uses Nernst glower, I can use an Opperman and a Globber source. These are all technical names Nernst glower, Opperman and Globber sources, but nicrome wire everybody knows that it in the Nickel chromium alloy wire.

They are commonly employed as sources of infrared radiation. So how many of you are seen a nicrome wire, if I ask you the question just like that many of you would say no, but if I ask you how many of you use a heater in a coil at when it is very cold, heater in a room, and how many of you used electric heaters for in the kitchen you know earlier there used to be in the kitchen electric heater with wire round, round, and round in a ceramic base plate.

Even now they are available in the market and you just have to switch it and connect it to a plug the wire will glow red and that is nicrome wire ok. So the nicrome wire I think many of you must have seen it and these nicrome wire is a very simple is used either as a simple coil just like what you see in electric heaters or it can be supported on a ceramic rod I can take a ceramic rod like this a pen and I can wildly nichrome wire around it.

And then connected the two ends and then heat it. So the wire will get heated and start giving out infrared radiation on all sides. So put a mirror behind it and everything will be reflected to get a parallel beam ok. So the nicrome wire I am sure most of you must be aware that it is a very simple system and it must be very cheap also, now it is not very costly. So most of the low and mid-price instruments used nicrome wire or not.

And it can be replaced with quite often the nicrome wire gets cut and the IR radiation will not come because of the short circuiting or breaking of the wire. So it is easy to maintain and so long as it works it is good enough so prolonged use can lead to oxidation sometimes wire gets oxidized to oxide and oxides are not good conductors of heat and electricity. So you have to heat it more and more to make it look red.

And the more you heat it the more oxide will form the less heated it will be at some point a breaking point will be reached and then thermal stress deform the coil shape and quite often it breaks into two the connection is gone. So the long term stability and output are affected over prolonged use but quite often these are all very simple, you can buy a huge coil of about one fourth or ten fourth coil.

And whenever it gets cut you just go there replace the wire refix it and then restart reusing that is a nicrome wire for you has an IR radiation source ok. See what I am trying to tell you in most of these instrumentation is none of them are really you know high tech equipment but they do they are made to look Hi-tech whenever an instrument manufacturer comes to sell you an instrument is SSR we got only nicrome wire this thing will give your spare wire I know.

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Nernst glower is composed of a mixture of rare earth oxides that are heated by electric current. This has a negative coefficient of electrical resistance. Its electrical conductivity increases with increasing temperature. Therefore external heating is required to start the flow of electricity and it must be carefully regulated to control a 'run away' situation. The Opperman consists of a ceramic rod filled with rare earth oxides with a coaxial metal wire located inside the centre. The wire is heated first which in turn heats the oxide mixture. This is self regulating and does not require external heating.



So that you can use them like that but in actual fact is you can go to market and buy nicrome wire and use it yourself. So the instrument manufacturers make use of your ignorance whenever they are trying to market their equipment. So there they make money ok that the nicrome wire available everywhere ok, second one is Nernst glower. So Nernst glower is a mixture of rare earth oxide just now I was telling you that if the metal forms oxide it will not glow red, it will take more heat.

And it may break very connectivity. Now the rare earth oxides there oxides only and then made it into some sort of a small pen top molded into a peace like this ok so it is not visible here ok does not matter it can be molded into a peace like this about 2 cm pen portion what I am showing you and it can be pressed hard pressed into a small cylinder which can be connected at both ends. So this rare earth oxide.

These rare earth oxides are heated by electric current, they have supply electricity to make them glow red hot ok. So this has got that means you have to heat them at very high temperature. So it has got negative coefficient of electrical resistance, you should remember this because the anything that has got a negative coefficient of electric resistance means it is electrical conductivity increases with increasing temperature.

Most of the metals have the property other way round you take the metal you start heating it, it is electrical conductivity comes down ok, but oxides rare earth oxides behave the other way around you take the rare earth oxides make it into a small pellet, connect the electrical wires start heating it as you heat more and more the more conductivity becomes better and better unlike metal. So the external heating is required initially ok.

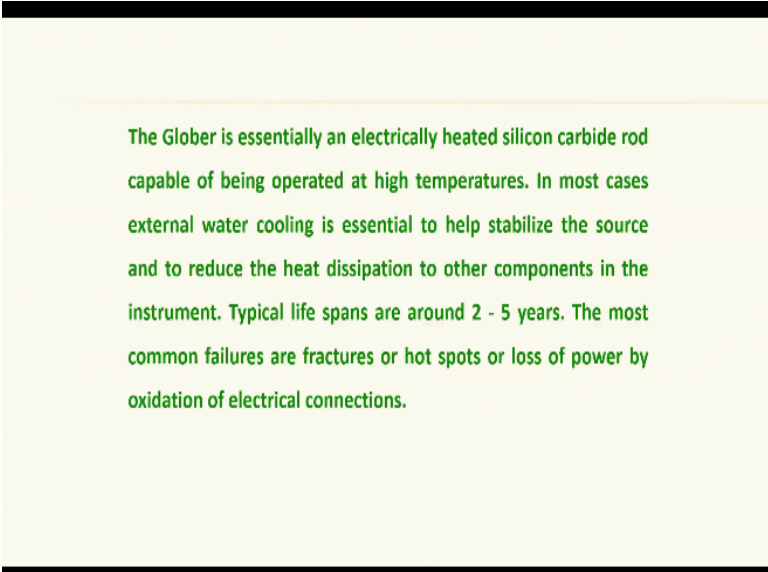
So you cannot heat the electric that rare earth oxide a wire directly by electric current first you have to connect it to an external heater and supply that heat to the Nernst glower and slowly as the temperature increases it will start glowing. So at that point you can cut off the primary heater and then after sometime this is itself will take over as the IR radiation source. So it is electrical conductivity increases with increasing temperature.

So external heating is required this adds to cost because I have to provide extra additional heat in the instrument another circuit to be added. So it must be carefully regulated to control a runaway situation. For example if you uncontrollably heat then the rare earth oxide will also get heated to very high temperature and we do not need that, because its life also will go down. So it may start heating it unnecessarily to very high temperature which is not ideal, the Nernst glower has got this property.

That is rare earth oxides, now the Opperman is another type of IR source that consists of a ceramic rod filled with again rare earth oxides but with a coaxial metal wire located inside the center. Now you can understand it will be something like this, I have a rare earth oxide here and I made a small piece a cylindrical piece, but I do have a small metal wire here inside, this is metal wire, this is rare earth oxide cylinder.

This whole thing is about 3 mm, this will be about 5 centimeter, maybe sometimes 2 centimeters also ok. So this is here I have a metal piece to add the heating that means one single instrument will take care of the total arrangement. So you do not need a second circuit unlike uninstaller wire. So with a coaxial metal wire located there inside the center Opperman is design. So the wire is heated first which integrates the oxide mixture.

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The Globber is essentially an electrically heated silicon carbide rod capable of being operated at high temperatures. In most cases external water cooling is essential to help stabilize the source and to reduce the heat dissipation to other components in the instrument. Typical life spans are around 2 - 5 years. The most common failures are fractures or hot spots or loss of power by oxidation of electrical connections.

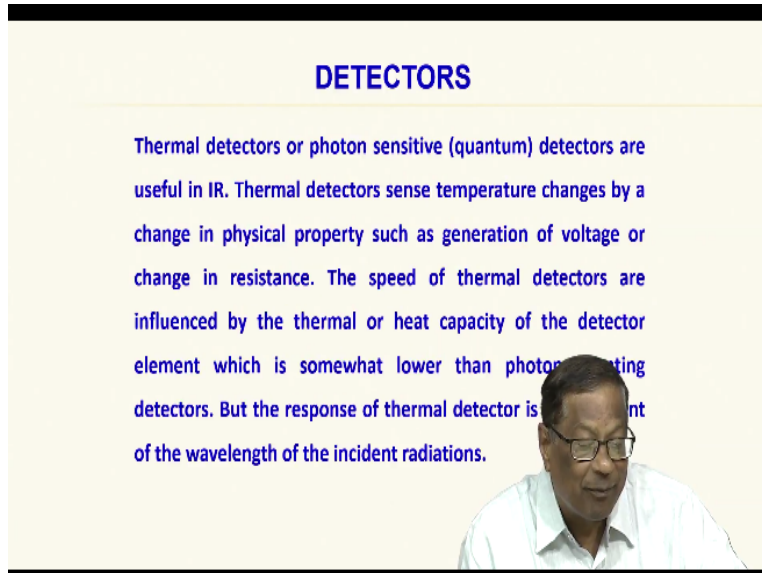
This is self-regulating and does not require any additional external heating. So whenever you buy an instrument you have to really think what instruments you want to what source you would like to have usually you ask the manufacturer what source you are giving for us in the IR. So that if the IR radiation stops coming out I need to replace the source. So the source is it nicrome wire, Nernst glower or Opperman or the Globber.

So I have not taught you about the Globber yet, so what is the Globber, the Globber is essentially an electrically heated silicon carbide rod it is basically silicon carbide rod just like this pen ok small piece of pen about 2 mm thickness and 3 cm or 5 cm rod which can be connected at both ends to the electrical wires for heating. So that is silicon carbide wire it is essential to help stabilize the source and reduce the heat dissipation to other components in the instrument.

Because silicon carbide can be heated to very high temperature. So the heat should not be passed on to other components of the instrument which we melt with my deform and it may lead to

shock etc. So heat dissipation has to be taken care of so the typical lifespan of silicon carbide source is about 2-5 years. So most common failures how do they occur in silicon carbide they break, they as usual they develop hotspots or loss of power by oxidation of the electrical connections.

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**DETECTORS**

Thermal detectors or photon sensitive (quantum) detectors are useful in IR. Thermal detectors sense temperature changes by a change in physical property such as generation of voltage or change in resistance. The speed of thermal detectors are influenced by the thermal or heat capacity of the detector element which is somewhat lower than photon counting detectors. But the response of thermal detector is independent of the wavelength of the incident radiations.

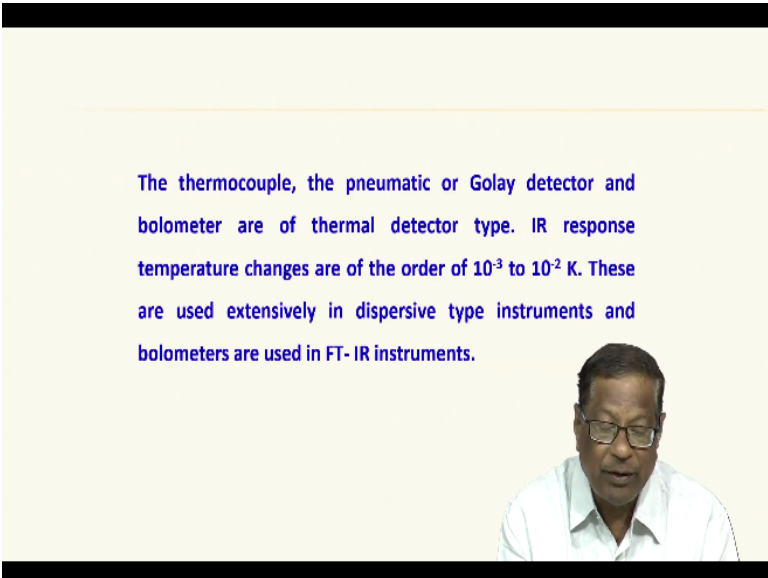
It is not the electrical wires, but it is connector that is happening. So that is about the detectors ok. Now I want to talk to you about the detectors, the most common detector in infrared radiation is thermal detector or photosensitive quantum detector, they are useful in infrared spectroscopy. Thermal detectors normally sense temperature changes by change in the physical property such a generation of voltage or they can change the resistance also.

So the speed we monitor the resistance with the radiation one side and without the radiation other way another time that is whenever I want to take the blank 100% transmittance just like an laser IR I just use the laser without the sample that is 100% just I monitor the resistance and then put the sample monetary resistance. So the speed of the thermal detectors basically are influenced by the thermal capacity or the heat capacity of the detector element which is somewhat lower than photon counting detectors the efficiency.

So the response of the thermal detector is actually independent of the wavelength of the incident radiation it has to be no, so the incident radiation whatever it is but the response of thermal

detector is depends upon how much photons are coming out of the detector. So I can use the thermocouple.

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The thermocouple, the pneumatic or Golay detector and bolometer are of thermal detector type. IR response temperature changes are of the order of  $10^{-3}$  to  $10^{-2}$  K. These are used extensively in dispersive type instruments and bolometers are used in FT-IR instruments.

You know what is a thermocouple is not, many of you must be knowing thermocouple is a 2 metal strips fused together to give you one single connector and at the juncture it gives you the different current. So the thermocouple is another way of measuring the heat changes that occur in the infrared spectrum of the incident radiation as well as observatory edition. So absorb radiation will have less heat output compared to the unabsorbed 100% transmittance.

So the pneumatic detector and another way of detecting infrared spectrometer spectrum is by a pneumatic detector or a Golay detector it is a name detector and we also use what is known as bolometer different kinds of detectors are used and thermocouple pneumatic Golay detector, bolometer. These are all different kinds of detectors developed over a period of time and most of them are into regular use not that there any in any way better than the existing ones are better than.

They do have certain advantages over others I am not going into the details but I am just going to explain to you the sum of the principal in general. So the response of the IR response of the temperature changes or of the order of about  $10^{-1}$  to  $10^{-2}$  Kelvin, that means the sample difference between the reference sample and the actual sample the temperature of the

incoming radiation generated radiation is actual sample is a generated radiation is actually the unabsorbed.

And once the sample is put I have the lower temperature because part of it is observed. So the temperature the radiation coming out of the sample is much less when part of it is observed. So what is the difference between the observed and unabsorbed radiation  $\Delta T$  we are looking for their typical  $\Delta T$  for Optimum detection is  $10^{-1}$  that is 0.1 degree Kelvin or  $10^{-2}$  degree Kelvin ok.

Now we do not say degree Kelvin we just say  $10^{-2}$  Kelvin but 1 degree 1 kelvin and 1 degree centigrade both are same I think many of you must be aware that what I am saying is the scale is different but the magnitude of the heat is essentially same. So 1 degree Kelvin, 1 degree centigrade both are same. So but only because of highest international standards we use degree Kelvin in such expressions.

So these are used extensively in dispersive type instrument and sometimes we do use bolometers, bolometers are used more in infrared instruments IR. So essentially what are you talking about the Golay detector and thermal detector Golay detector as well as bolometer, they are all thermal and temperature difference is about 0.01 to 0.02 degree centigrade or Kelvin and these are used extensively in dispersive instrument.

That is prism grating rate prism grating or grating rating instruments not in FT-IR, in bolometer are used in FT-IR by I will just give a couple of seconds to think why we use bolometer many of you may not even be knowing what is a bolometer is not it, just go home and check I will give you this is an example in the instrument instrumentation course, you can try to find out what is the answer to this.

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The thermocouple is fabricated with two dissimilar metals such as bismuth and antimony which produce a small voltage proportional to the temperature of junction. Several thermocouples are connected in series for additional output. Half of the junctions are 'hot' containing the active element. Alternate junctions are 'cold' that are thermally bonded to the substrate and remain at a relatively lower temperature. Thin film techniques have miniaturized the thermocouples. The entire assembly is mounted on an evacuated enclosure with IR transmitting window so that conductive heat losses are minimized. The response time is about 80μsec.



So what is a bolometer ok, so the bolometers are used in infrared FT-IR instruments ok. Now the thermocouple we go one by one again some good and bad advantages, good and bad factors as far as thermocouple is concerned it is fabricated with 2 dissimilar metals such as Bismuth and antimony, it can produce many thermocouples with different chromium, Nickel, some hydrolytic platinum, rhodium.

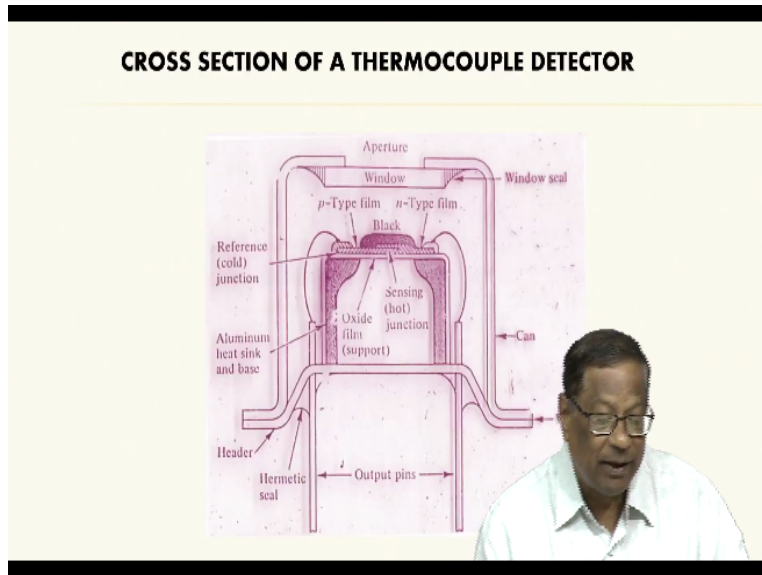
Several types of thermocouples are there which can be used for temperature monitoring in infrared, temperature monitoring of what the transmitted light with the sample once and without the sample another way. So the ratio provides the infrared spectrum with the ration, with the sample and without the sample ok. So a small voltage is produced when the dissimilar metals are used the dissimilar metal piece used together is used for making the IR radiation fall on to that joint where they are fused together.

So it is called a junction, so the junction temperature should be monitored whenever we use infrared spectrum in spectrometer. So several can have sometimes we need several connectors in series for additional output, half of these junctions are hot and containing the active element and alternative junctions are cold ok. So it is hot and cold junctions are thermally bonded to the substrate, the body of the substrate is the detector body ok.

So it may be fixed on the instrument itself. So the re-substrate retains the temperature at a slightly lower rate, then they only outside the instrument ok. So thin film technique miniaturized with thermocouples the entire assembly is mounted on an electric evaluated enclosure with IR transmitting window infrared. So that the conductive heat losses are minimized. The response time is approximately about 2 hours not more than that ok.

And response time is not 2 hours but it is sorry it is 80 microseconds not 2 hours it si 80 micro seconds with so if the IR radiation changes the signal within 80 microseconds it must show on the recorder, then only such detectors are used ok.

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Now here I am showing you the cross section of a thermocouple detector. So you can see I have here the whole size of this detector is about 5 centimeter ok such 2 detectors are used and it uses a p-type film and n-type film and p-type film and here the film is exposed in this area ok, I will show you here. So the film is exposed over a very small portion here and another small portion is here remaining is black painted ok.

Now there is an oxide film here at the bottom of the plating and this is a they are these are the 2 metals ok u can see the 2 white lined areas below this black paint ok and there are 2 different metals one is the sensor sensing junction that is hot and other is the oxide film that is cold. So the oxide film is at the bottom and sensing junction is at the top ok. Now the other parts this is a

output pin ok. So this is how the whole thing is mounted, this whole thing is mounted on this box ok.

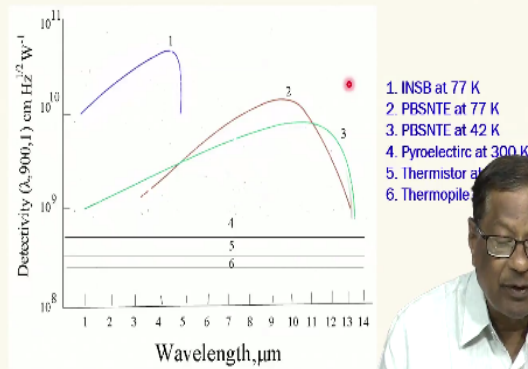
And then there is a header to fix the detector and they are welded and one more support you need and this support is that gives a small gap here in between and I put a small window here this window if it has to allow IR it has to be made from polythene film or a sodium chloride or whatever is the optical quality that is required for our infrared ok. So the aperture is IR transmitter and window is IR transmitter.

All the radiation will fall this black radiation will absorb the radiation matrix of no use but I have a small transparent piece here where the radiation will cause generation of the current one is cold another one is hot. So this is reference cold is reference junction at the other one is aluminium and other is hot junction and this whole thing is mounted on and eliminate this one this portion ok. This red portion is aluminium piece supported required to take out the heat generated by the IR.

So as the infrared radiation move from one frequency to another there will be a peak generated based on the temperature difference. The movement the infrared radiation most another region where there is no infrared radiation or infrared peak the heat generated must be dissipated. So that heat generated is dissipated using this aluminium heat sink that is known as aluminium heat sink. So this is how a thermocouple detector is designed in infrared spectrometer.

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## WAVELENGTH RESPONSE OF SOME IR DETECTORS



That is fairly decent engineering item but we should understand how a detector works. So here is the wavelength response of some IR detectors. For example I have plotted here the detectivity that is detection are at  $\lambda$  around 900  $0.1 \text{ cm}^{-1}$  hertz ok this is  $10^9$ ,  $10^{10}$ ,  $10^{11}$  hertz raise to half and wattage my raise to  $-1$  and here it is wavelength ok. Now I have different curves here 1, 2, 3, 4, 5, 6.

First one is Indium antimony detector, that is the bi metal the thermocouple made of Indium and antimony it keeps on increasing from 1 to 5 micrometers ok and then it falls off what is the wavelength required for us in the IR it is  $4000 \text{ cm}^{-1}$ . Now so this 1 to 5  $\mu\text{m}$ , 1 to 5 micrometer, nanometers basically is quite good, but not that good, so look at number 2, it starts from about 3 micrometer up to 10 micrometer that is good.

And then number 3 is it keeps on increasing from 1 up to almost 11 ok green one and then it falls off at higher wavelengths. So that is lead antimony and tellurium, this both have same 2 and 3 but a little temperature difference makes them worth much better over the entire range. So the whole thing is the research in these bi metal detector is basically concentrated on how far can take the detector make a response up to 10, 11, 12, 13, 14 micrometer.

And then how long it will survive and how many cycles it will survive like that if water suppose pretreatment temperature is 42 degrees or 77 degrees or how is the response lot of research goes

on in IR detector. So look at number 4 it is just a pyroelectric wires at 6 300° number 5 is thermostat at 300 degrees, number 6 is the thermopile at 300 degree Kelvin all of them are Kelvin only I should not say degree centigrade ok.

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A thermistor functions by changing resistance when illuminated with IR. These are made of sintered oxides of manganese, cobalt and nickel and have a high coefficient of resistance (4%/°C). One of these is 'active', which is coated black and mounted to increase its IR absorption whereas reference is optically shielded to prevent IR exposure. Both are mounted on an insulating substrate placed on a heat sink. When connected with a bridge circuit, it compensates the ambient temperature drifts. The response times are a few milliseconds.



So a thermistor functions by changing the resistance when illuminated with infrared. These are made of sintered oxides of manganese, cobalt, Nickel etc. they have a high coefficient of resistance one of these is active which is coated black and another is mounted to increase its IR absorption whereas reference is optical is shielded to prevent the IR exposure. Both are mounted on a insulating substrate placed on a heat sink.

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The Golay pneumatic detector uses the pneumatic expansion of a gas as the measuring device. The unit consists of a small metallic cylinder closed by a rigid blackened metal plate (2 mm square) at one end and a flexible silver diaphragm at the other end. The chamber is filled with xenon. The radiation absorbed by the blackened plate causes the gas to expand and deform the diaphragm which in turn obstructs the light path falling on a phototube. The distortion alters the plate separation and hence the capacity. Response time is 20 msec (similar to TC). It is used among the lot.



So when connected with a bridge circuit it compensates the ambient temperature drift also. So the response time here is about a few milliseconds what was the response time earlier with thermocouple it was 80 microseconds here but now look at it this thermistors have filled few milliseconds. So response of the thermistors are slightly lower than the thermocouples, they change faster immediate response. So they will take up the Golay detector and other instrumentation in our next class, thank you very much.