Chemical Process Instrumentation
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Lecture - 51
Miscellaneous Measurements: Composition

Welcome to week 11 as of now we have talked about measurement of temperature pressure flow and level. Now, apart from these process variables as chemical engineer we will also required to frequently measuring important variables such as concentration or composition density viscosity etcetera. So, in this week we will talk about miscellaneous measurement such as concentration or compositions density viscosity and pH.

First, we will start our discussion on measurement of concentration of compositions we will briefly discuss two important methods. The first method, we will talk about is based on chromatography and the second method we will talk about is based on spectroscopic. In particular, we will talk about UV visible spectroscopic it is niggles to mention that for a chemical engineer measurement of concentration is extremely important suppose you want to separate a mixture into given purity. So, you need to know the concentration of the separate extremes you are carrying out a reaction in a reactor you would like to know the concentration of the products in the product stream.

So, the concentration measurement is extremely important for all process engineers. So, today we will start our discussion on concentration measurement or composition measurement.

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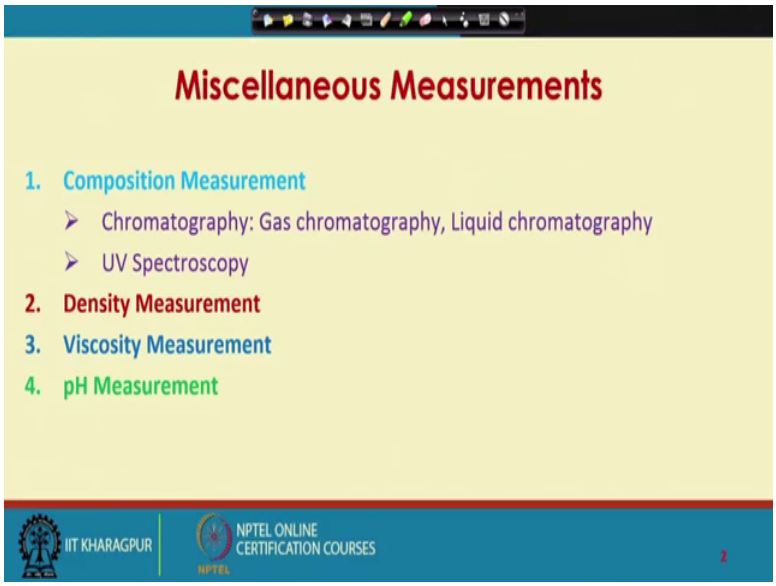

So, these are the miscellaneous measurements, we planned to discuss in this week composition measurement through chromatography and UV spectroscopy density measurement viscosity measurement and pH measurement.

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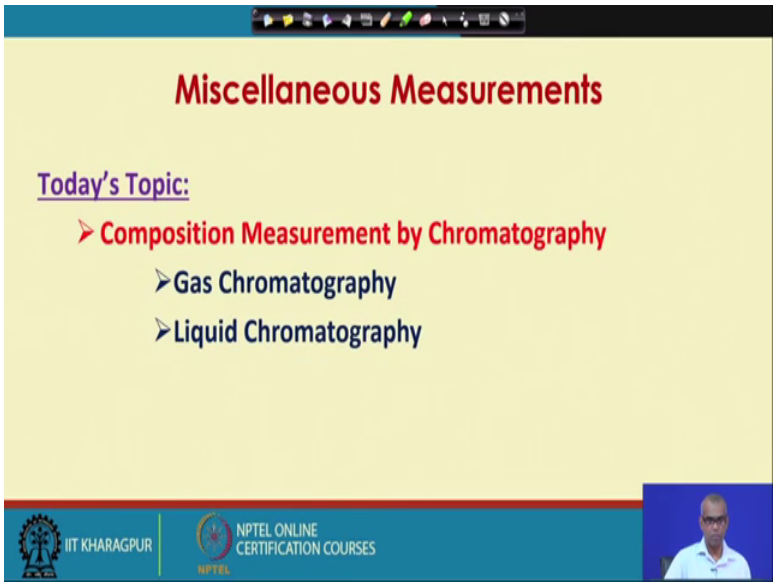

So, today we will start our discussion on composition measurement by chromatography and we will talk about two methods under this category gas chromatography and liquid.

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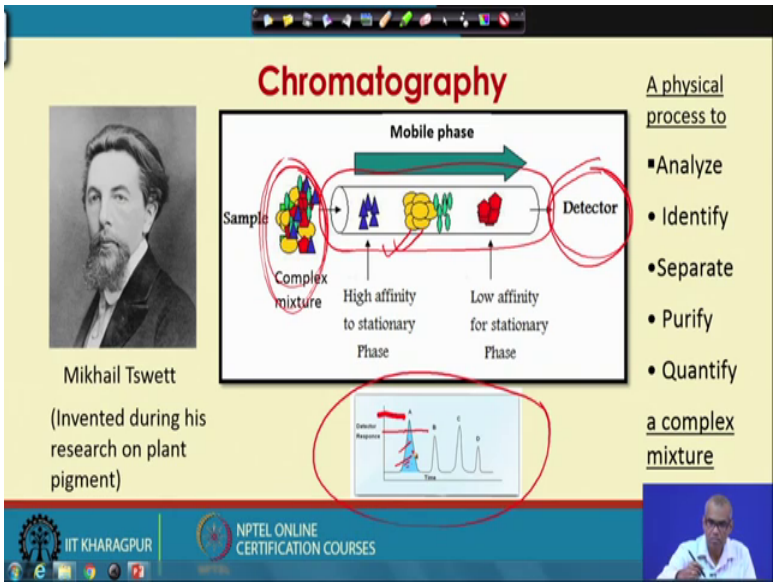

Chromatography: we will see that the principle is same for both chromatography is a physical process to analyze identify separate purify and quantify a complex mixture.

It is a simple and very elegant physical process which you can use to separate a complex mixture and identify and quantify the components Russian scientist Mikhail Tswett invented chromatography during his research on plant pigment, if you look at this diagram this is known as chromatographic column the chromatographic column has say adsorbent particles in it.

This is the complex mixture you want to separate. So, there are more than one component in this mixture. In case of chromatography this mixture is passed through this column and this column contains let us say for the time being adsorbents particles this column containing particles is stationary phase.

And we will use a mobile phase which is a fluid for gas chromatography it is a gas for liquid chromatography it is a liquid in this mobile phase I will inject this complex mixture and same that mobile phase through the stationary adsorbent which is there in the column now the components in the complex mixture will have different affinity for the absorbance or the stationary phase. So, what we will happen is at the other end of column the components will come out at different times because the components will interact differently with the stationary phase.

So, the migration rates of these components through the stationary phase will be different. So, the components different components will come out at different times. So, thereby they are separated. So, the component which is very high affinity to stationary phase will take more time to come out and the components which have low affinity for the stationary phase will come out quickly.

We will have a detector at the other end of column which detects these components that come out. And if I plot the detector response versus time I will get a plot like this which is known as chromatography see this plot has different peaks each peak corresponds to different components.

So, the time at least these different components come out and known as retention time like. So, this is the retention time for a this is retention time for b so and so, forth; looking at this retention time you can identify the component looking at the peak height or the area under the curve you can determine the amount of the component. For example, the amount of a is proportional to the area under this shaded curve.

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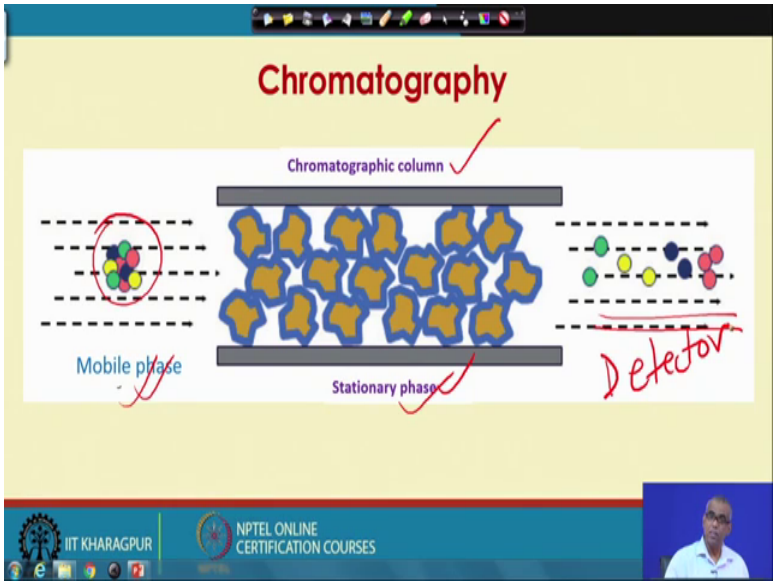

So, the chromatography consists of a column which has stationary phase in it you need a mobile phase the mobile in the mobile phase you inject the sample mixture you want to separate and to identify the mobile phase is force to the stationary phase, because of the different affinity of the components in the mixture with the stationary phase the different

components come out at different times. So, they are separated we will put a detector here to identify this components and quantify this components.

We will see later; what is this different stationary phase? What is the length of the chromatography column? What kind of detector? I will use what are the mobile phases that may be used so and so forth.

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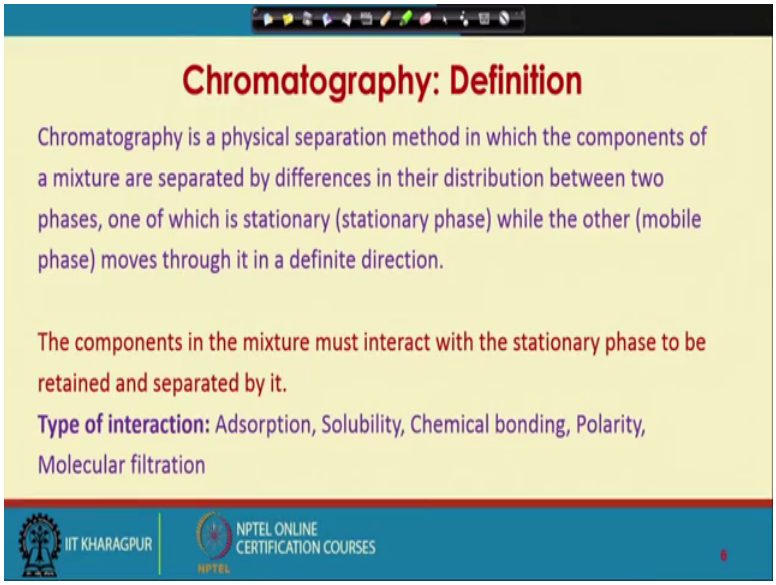

So, chromatography is a physical separation method in which the components of a mixture is separated by differences in their distribution between two phases, one of which is stationary known as stationary phase while the other mobile phase moves through it in a definite direction. Mobile phase also moves with specific velocity the components in the mixture must interact with the stationary phase to be retained and separated by it type of interaction may be based on adsorption solubility chemical bonding polarity and molecular filtration.

So, these are different types of interactions that the components in a mixture may have with the stationary phase. So, depending on that we will have different stationary phases for example, if adsorption is the interaction, that is will use solid adsorbent particles may be granular solids or may be granular solids soak with liquid it may be iron exchange risen. So, there are at different types of stationary phases that are possible we will see this thing in more detail little later for the time being let us consider that the type of

interaction are adsorptions solubility chemical bonding polarity molecular filtration so and so forth.

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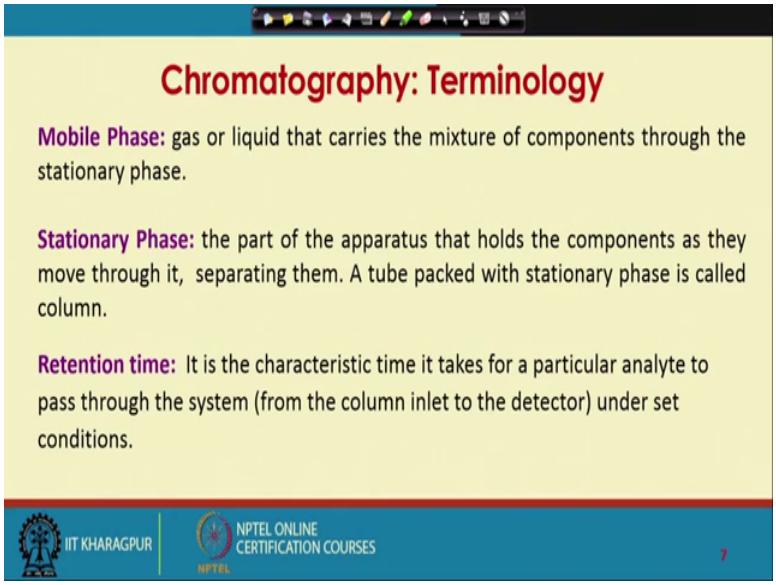

Let us now the familiar with certain terminology used in chromatography mobile phase is the gas or liquid that carries mixture of components through the stationary phase.

And the stationary phase is the part of the apparatus that holds the components as they move through it separating them a tube packed to it is stationary phase is called column. So, in case of chromatography separation you have a stationary phase and a mobile phase the stationary phase remains stationary, the mobile phase carries the mixture that you want to separate along with it. And the mobile phase is forced through the stationary phase with the specific velocity the mobile phase is a fluid it may be gas it may be a liquid if I am using gas as mobile phase the terminology, I use is gas chromatography; if I am using liquid as a mobile phase I call it liquid chromatography.

So, a liquid mixture can be separated directly is in liquid chromatography a gas mixture can be directly separated using gas chromatography a liquid mixture can also be separated using gas chromatography, but in that case liquid mixture has first to be vaporized and then pass through the carrier gas. So, is the mobile phase that determines whether I am using gas chromatography or using liquid chromatography in case of gas chromatography the mobile phase is gas. And in the case of liquid chromatography the mobile phase is liquid.

I will see later; what are the different mobile phases that can be used for gas chromatography and liquid chromatography retention time. It is the characteristic it takes for a particular analyte to the pass through the system from the column inlet to the detector under set conditions. So, retention time is the elapsed between the injection and the detection time depends on the component in the mixture. So, looking at the retention time I should be able to identify the component.

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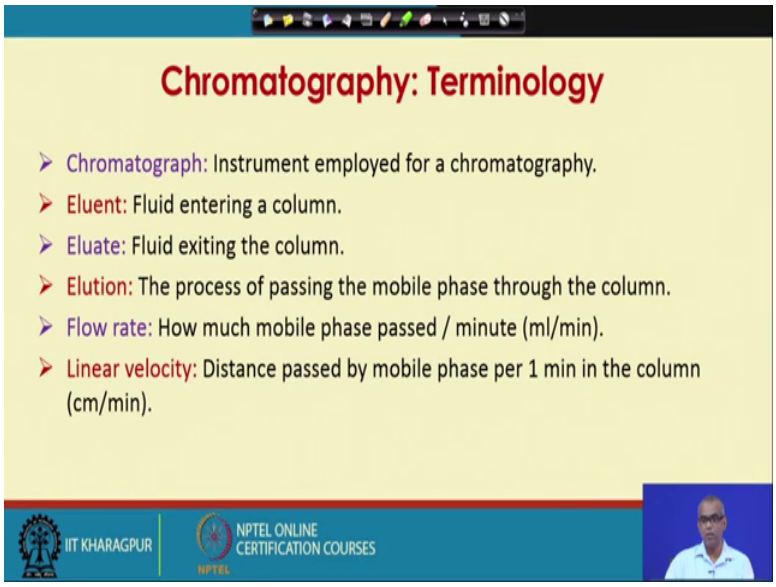

Chromatograph is the instrument employed for a chromatography and chromatogram is the detector versus time signal.

Eluent fluid entering a column eluate fluid exiting the column elution the process of passing the mobile phase through the column flow rate how much mobile phase passed per unit time or I will say per unit per minute. So, milliliter per minute linear velocity distance passed by mobile phase per unit time; let us say per 1 minute in the column I can express it in terms of centimeter per minute.

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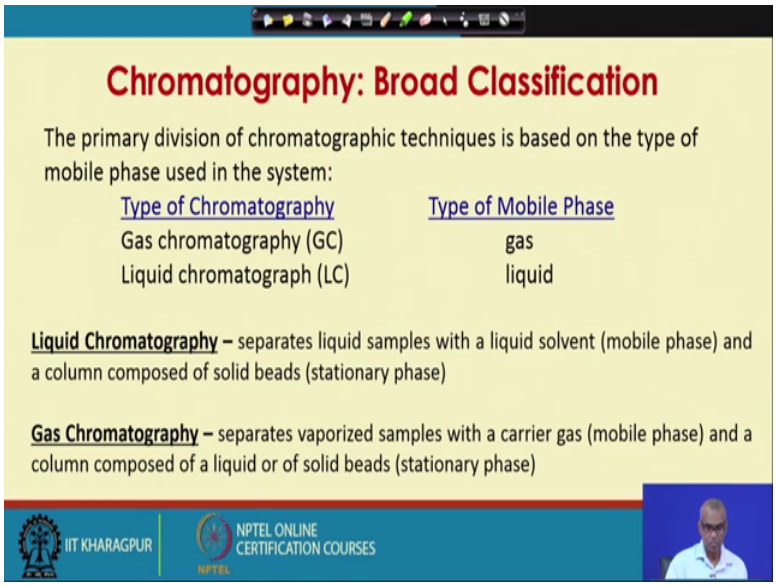

The primary division of chromatographic techniques is based on the type of mobile phase used in the system gas chromatography use gas as mobile phase liquid chromatography use liquid as mobile phase.

Liquid chromatography separates liquid samples with a liquid solvent mobile phase and a column composed of solid beads which is stationary phase in case of gas chromatography gas chromatography separates vaporized samples with the carrier gas which is mobile phase and a column composed of a liquid or of solid beads which is stationary phase. So, in case of gas chromatography the stationary phase may be liquid as well as it may consist of solid beads like solid particulate solid adsorbents.

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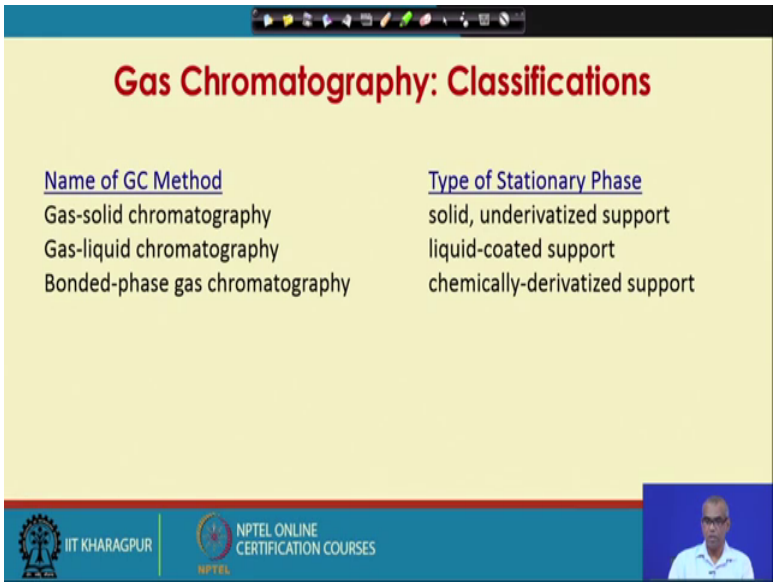

Now, let us look at some classifications these classifications based on types on stationary phase; that are used gas solid chromatography the type of stationary phase used is solid underivatized support gas liquid chromatography.

The stationary phase is liquid coated support bonded phase gas chromatography stationary phase is chemically derivatized support stationary phase is chemically derivatized support.

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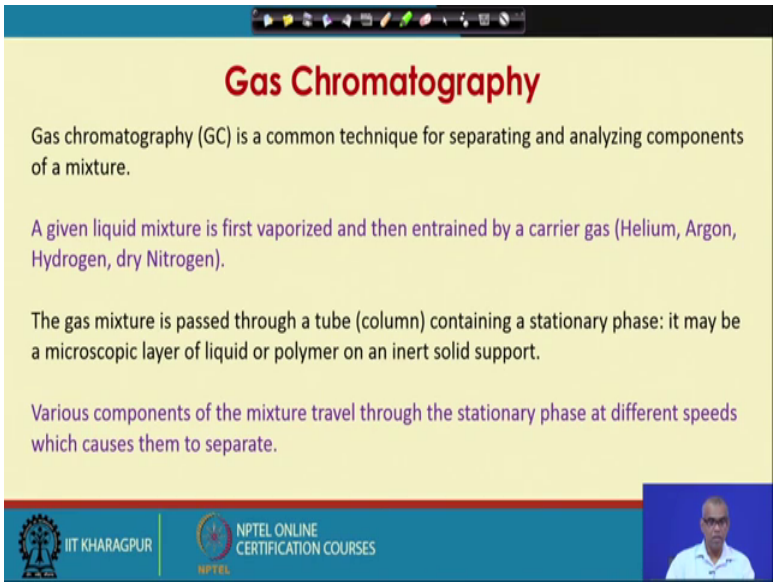

Gas chromatography is common technique for separating and analyzing components of a mixture. A given liquid mixture is first vaporized and then entrained by a carrier gas the carrier gas commonly used a helium, argon, hydrogen, and dry nitrogen. The gas mixture is passed through a tube which is known as column in the terminology chromatography containing a stationary phase: it may be microscopic layer of liquid or polymer on an inert solid support. Various components of the mixture travel through the stationary phase at different speeds which cause them to separate.

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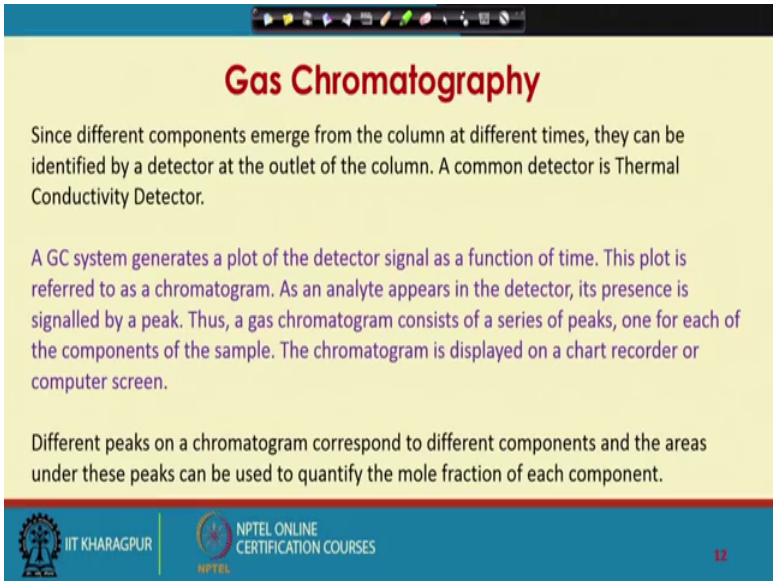

Since different components emerge from the column at different times, they can be identified by a detector at the outlet of the column. A common detector is Thermal Conductivity Detector. There is also flow ionization detector a gas chromatography system generates a plot of the detector signal as a function of time. This plot is refer to as a chromatogram. As an analyte appears in the detector, it is presence is signaled by a peak. Thus, a gas chromatogram consists of series of peaks, one for each of the components of the sample. The chromatogram is displayed on a chart recorder or computer screen.

So, I repeat a gas chromatograph system generates a plot of the detector signal as a function of time this plot is refer to as a chromatogram as an analyte appears in a detector it is presence is signal by a peak thus a gas chromatogram consists of series of peaks one for each of the components of the sample the chromatogram is displayed on a chart

recorder or computer screen different peaks on a chromatogram corresponds to different components and the areas under these peaks can be used to quantify the mole fraction of each component. So, different components come at different time. So, detector detects different components at different times and the detector gives you a response which is plot of detector signal versus time.

So, this plot consist of peaks various peaks there will be three peaks if there are three components in a mixture now you should know what is a retention time for this three different components that you can easily obtain by carrying out an experiment with this three pure components. So, you know that component a retention time is this component b retention time is this so, and so, forth. So, you will be able to identify the components now the area under each of these curves. So, the chromatograph will consists of three peaks. So, there will be three areas under the craft these areas under the craft correspond to the mole fraction of these components in the mixture.

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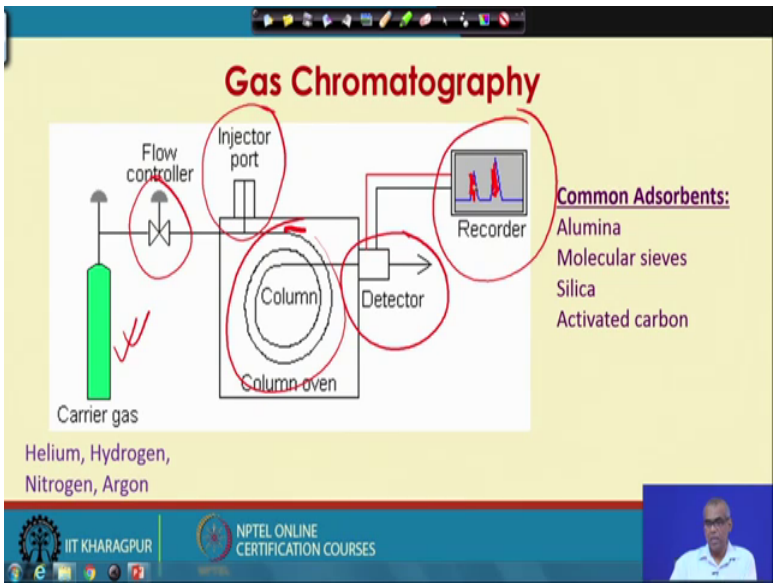

So, there are by you can separate this components as well as you can quantify these components. So, these are various parts of a gas chromatograph carrier gas which is helium hydrogen dry nitrogen argon etcetera you have a flow controller here so that the carrier gas flow can be regulated. So, the flow rate of the carrier gas must be regulated using the flow controller then you have a sample injection system.

Now, the sample injection system depending on whether you have gas sample or you have liquid sample may be different. So, the gas sample injection and the liquid sample injection may take different form, because when you have this liquid sample the liquid the liquid sample injection will be followed by the vaporization of that liquid this sample injection may be manual it may also be automatic. So, in that case it will be slightly more complex arrangement but.

This sample injection system allows you to inject a known specific amount of sample to the carrier gas these carrier gas. Now flows to the column the carrier gas along with the injected sample in it flows through the column then at the other end of the column when the sample comes out it flows through detector. So, the different components will come out at different time the detector will generate a signal which is known as chromatogram note that they are at two peaks; that means, there are at two components in a mixture their retention time is different.

So, that tells you which component is what and the area under this two curves or the two areas that you see these area as well as these area a measure of the mole fraction of these two components. So, the main parts are the carrier gas flow controller sample injection system the column the detector common adsorbents are alumina molecular sieves silica activated carbon.

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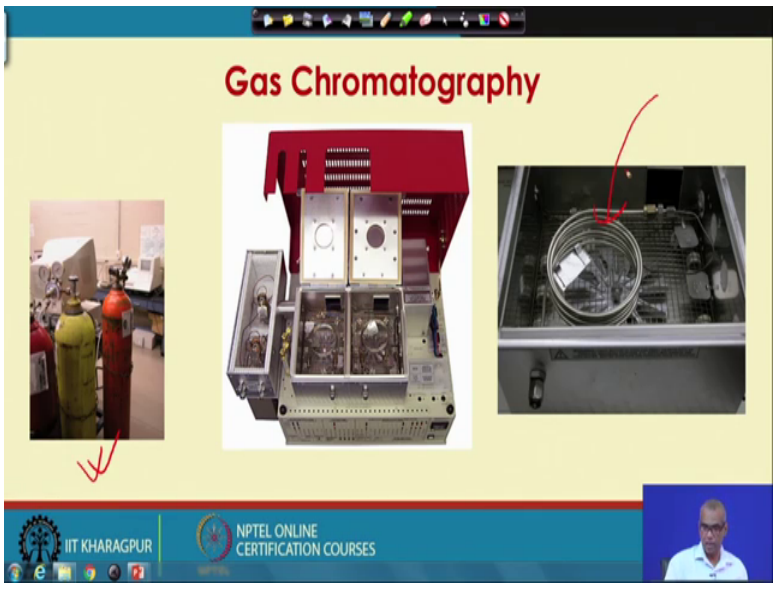

So, this is a photograph these are carrier gas cylinders.

This is the column that you see you have the oven also there. So, that the liquid sample is vaporized and passed through the column along with the carrier gas.

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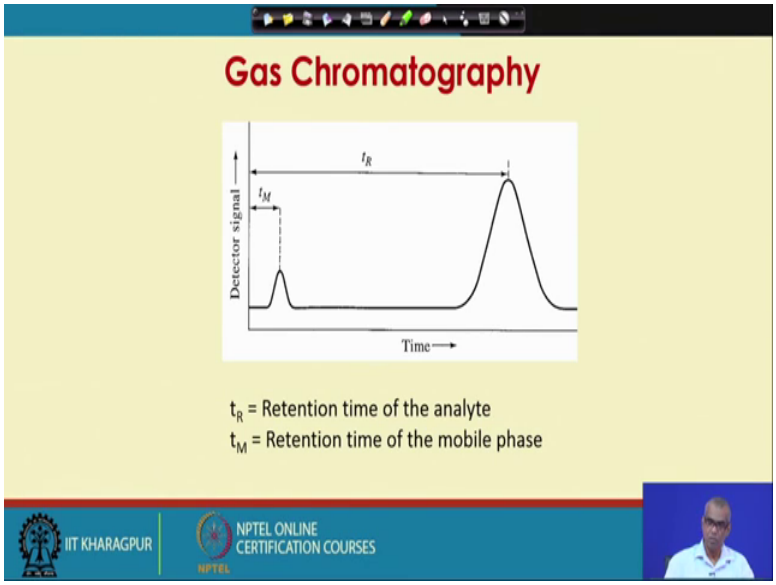

So, this is what retention time means the time elapsed between the sample injection and the point of detection by the detector.

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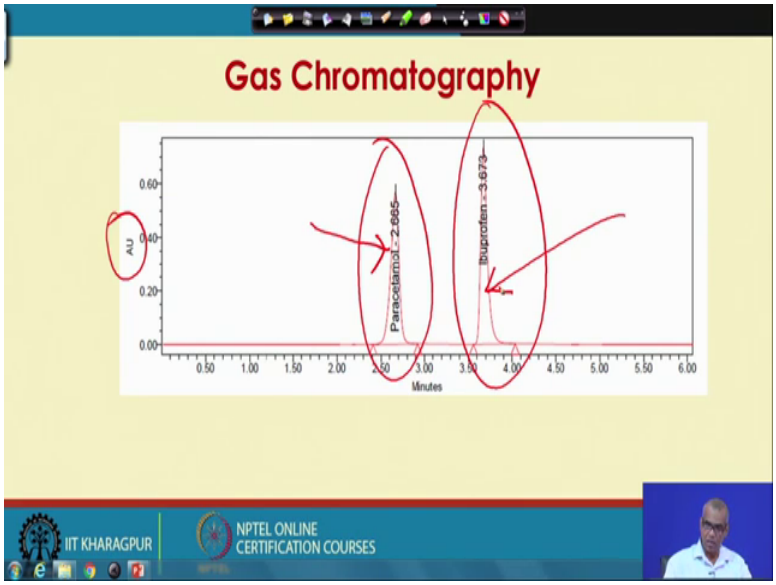

So, this is the typical chromatogram of paracetamol (Refer Time: 24:45) mixture note the retention time of paracetamol is 2.665 minutes.

Whereas retention time of ibuprotein is 3.673 minutes. So, this is the detection detector signal the area under these curve tells you the mole fraction of paracetamol and the area under this gap is a measure of mole fraction of ibuprotein of course, we will first must calibrate to relate the area under the curve with the mole fraction.

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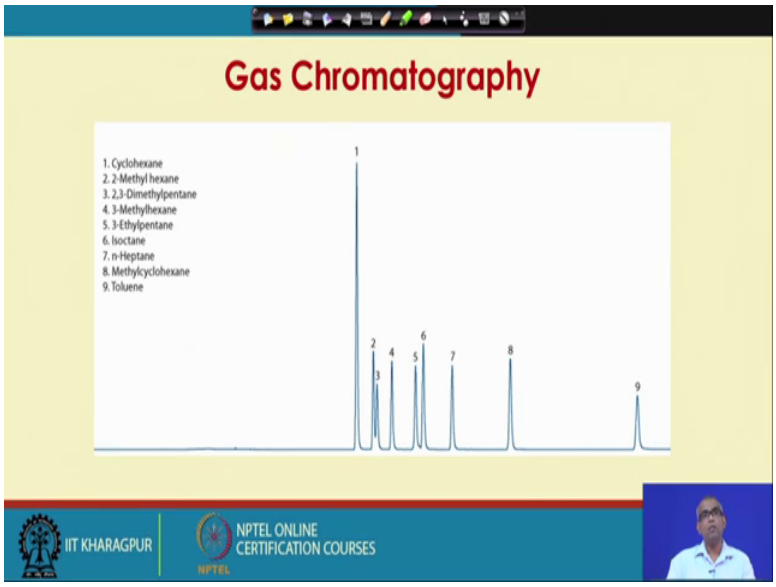

So, this is another chromatogram which has several components.

So, they have different retention times looking at the retention times you can again find out what are the components are. So, retention time tells you about the identification of the components and again area under the curve can be calibrated with the concentration or mole fraction of the component in the mixture.

So, we will stop here. And, in our next lecture we will talk about the more details or the different parts of the chromatograph little bit more details, and I will also talk about liquid chromatographic.