Hello students, I am Shubhlaxmi Naik

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code is CHD 103 and the course title is

Selected Instrumentation in Chemistry.

I will be discussing about the unit

Analysis of drugs in solid state under

which I will be covering instrumentation

and methodology involved in Differential

Thermal Analysis technique.

The main aim of this module is to get an

insight about the differential thermal

analysis that is detailed instrumentation

and methodology involved in the technique.

By the end of this video you will

become familiar with the technique,

its various components and working.

Okay students to begin with,

let us first understand what

are these thermal methods.

They are those methods in which

changes in physical and/ or chemical

properties of a substance are

measured as a function of temperature.

When we heat a matter,

it undergoes certain physical

or chemical changes,

or both,

which take place over a wide range

of temperatures.

Physical changes like melting

or boiling may occur at wide

temperature ranges depending on

the substance which is involved.

Similarly,

chemical changes like decomposition

may take place at different temperatures,

so these physical

and chemical changes which a substance

undergoes when heated are the

characteristic of that particular

substance which is under study.

By measuring the temperatures at

which these changes occurs and

the heat changes involved,

it is possible to characterize the

compounds present in the sample

and thus identify the sample.

These thermal analysis techniques find

wide applications in the analysis of

solid samples like inorganic precipitates,

glass materials, ceramics,

etc.

There are various thermal methods

of analysis available. To name few

there is a Thermogravimetric

Analysis which is abbreviated as

TGA, Differential Thermal Analysis,

DTA and Differential Scanning Calorimetry,

DSC. Okay so here today we will be

specifically discussing about

differential thermal analysis,

its instrumentation and methodology involved.

A DTA instrument is generally known as

a differential thermal analyzer,

which must consist of following components

that is, a sample assembly holder,

a suitable thermocouple,

a furnace with temperature programmer,

a differential thermal detector, a recorder,

and a atmosphere and coolant controls.

Students this is the schematic

diagram of a DTA system where you

can see sample assembly holder,

thermocouple, temperature program

controller, atmosphere and coolant

controls, amplifier and a recorder.

Now, moving to these each

components one by one.

First we have is a sample holder

assembly which consists of a sample and

a reference containers which are mounted

on a suitable holder with thermocouple.

So this sample holders.

They are usually made by using nickel,

then stainless steel.

We also have platinum alloys. Etc.

Next component is the thermocouple,

so students what is this thermocouple?

It is basically a sensor which

is measuring the temperature.

As we know we are analyzing the

changes in the samples with

respect to temperature right?

So this is what we will give us

the data about the temperature changes.

So here the choice of thermocouple will

depend on its capability to produce a

sufficient enough temperature difference,

its chemical compatibility with the

sample material which is under study and

its ability to give reproducible results.

Depending on temperature,

different materials are used to make

this thermocouple. For temperatures

up to 1100 degrees Celsius

Nickel chromium alloys are used and for

temperatures above 1100 degrees Celsius,

pure platinum and platinum

rhodium wires are used.

These thermocouple wires can be arranged

in different ways in the assembly,

wherein one is brought into contact with

the sample and the reference directly,

and in order to prevent any possible

chemical reaction with the sample they are

covered with the protective metal sheath.

Alternatively, these thermocouples

can also be fitted to the platforms

in which identical cups for the

sample and the reference are used.

So here is the schematic diagram

which shows the arrangement of

sample holders and the thermocouple.

Now. Coming to the next component that

is furnace with temperature programmer.

So this shape of the furnace used

in DTA is usually a tubular shape

which is prepared by winding nichrome

wire on a porcelain support and it can

be used up to 1200 degree Celsius.

For other temperature ranges,

like from 1200 to 1400 degree

Celsius kanthal wire which is wound

on alumino-silicate support are used.

And in case of temperatures up

to 1600 to 1800 degree Celsius,

platinum- rhodium alloy wires which is

wound around an aluminium support are used.

A heating rate of 10 to 20 degrees

Celsius per minute is maintained. This

heating rate greatly influences the DTA

curves, as higher will be the heating rates,

Sharper will be the peaks.

Also, the furnace is provided with

a temperature programmer in order

to maintain a linear temperature rise.

The temperature programmer which

is used should be capable of giving

a wide range of heating rate.

For example 1 degree to 50

degree Celsius per minute.

Next we have is the differential

temperature detector.

As mentioned earlier,

the thermocouple will serve

as the temperature sensor.

The temperature difference that is

delta T between the two junction of

the thermocouple will determine the

strength of the voltage signal produced.

Moving further, we have the next

component that is recorder.

This recording system will mainly

consists of an amplifier which

serves to amplify the signals coming

from the temperature sensor and

differential temperature thermocouple.

So these signals are typically

recorded on a twin pen recorder

on a typical paper.

Also, this recorder should possess

a wide range of sensitivity

and chart speed settings.

Now coming to the last component that

is atmosphere and cooling controls.

In the process of analysis,

the Chamber or the furnace assembly is

required to flush with the selected gas.

So this facility for flushing the sample

and reference assembly with the gas

should be possessed by the instrument.

Also, it should have a cooling facility for

rapid return to the ambient temperature.

In case of low temperature DTA work,

what is done is liquid nitrogen is used.

Also any gas which is sent to

such a low temperature assembly

is completely dried before the analysis

of the sample in order to prevent any

problems due to gas condensation.

So students this was about the components

of differential thermal analysis technique.

Now moving further onto the methodology

of DTA. As you can see in the

schematic diagram of DT analyzer system,

we have the sample and the reference

pans which are kept on the respective

thermocouple placed inside a furnace

covered by heat sheath and also

equipped with a cooling jacket.

The sample under study is placed in

the sample pan marked as ‘S’ and

a reference material is placed in

reference pan which is marked as ‘R’.

both of which are then subjected to

the thermal analysis. So here the

reference material, which is used

should possess certain

characteristics which are as follows.

First is that whatever the

reference material we are using,

it should not undergo any thermal events.

That is, chemical changes,

like decomposition or dehydration

over the entire operating range.

Secondly, it should not react

with the sample holder and the

thermocouple which we are using.

And lastly the thermal conductivity

and the heat capacity of the

reference material should be

Similar to that of the

sample which is under study.

Most widely used reference

materials for DTA are alumina,

magnesium oxide, silicon carbide, etc.

for inorganic samples

And in case of organic samples,

especially polymers,

we have octylphthalate and silicon

oil which are used as references.

One should take note that both the

sample and reference materials

should be used in powdered form.

Also,

the particle size and the packing

conditions of the materials

should be maintained identical

as they influence the results.

A reference material is

also used as a diluent for the sample,

which is a common technique for matching

the thermal properties of the sample

and that of the reference material.

Once the sample and reference material

are subjected to thermal analysis,

the temperature difference will

produce an output voltage which

is then fed to a computer.

The signal received from the sample

thermocouple is converted to temperature,

which is plotted as the X axis

of differential thermogram.

The output across sample and

Reference thermocouples that is,

ΔT is amplified

and consequently serves as Y

axis of the thermogram.

From the graph obtained,

we can have quantitative analysis

and also it helps in determining

the change is whether a physical

change or a chemical change.

So students,

this is how the DTA technique is

used in the thermal analysis

of a particular sample.

These are my references. Thank you.