Mechanical Measurements

Module 4

1. Thermo-physical properties
2. Radiation properties of surfaces
3. Gas concentration
4. Force or Acceleration, Torque, Power

Module 4.1

1. Measurement of thermo-physical properties

In engineering applications material properties are required for accurate prediction of their behaviour as well for design of components and systems. The properties we shall be interested in measuring are those that may be referred to generally as transport properties. The properties that we shall be interested in here are:

a. Thermal conductivity
b. Heat capacity
c. Calorific value of fuels
d. Viscosity

a) **Thermal conductivity**
Thermal conductivity may be measured by either steady state methods or unsteady (transient) methods.

(i) **Steady state methods**
   - Guarded hot plate method
     - Solid, Liquid
   - Radial heat flow apparatus
     - Liquid, Gas
   - Thermal conductivity comparator
     - Solid

Steady state methods normally involve very large measurement times since the system should come to the steady state, possibly starting from initial room temperature of all the components that make up the apparatus. Also maintaining the steady state requires expensive controllers and uninterrupted power and water supplies.

(ii) **Unsteady method**
   - Laser flash apparatus
     - Solid

Even though the unsteady methods may be expensive because of stringent instrumentation requirements the heat losses that plague the steady state methods are not present in these. The entire measurement times may be from a few milliseconds to seconds or at the most a few minutes.

Thermal conductivity is defined through Fourier law of conduction. In the case of one-dimensional heat conduction the appropriate relation that defines the thermal conductivity is
\[ k = -\frac{q}{\partial T} = -\frac{Q}{\partial T} \quad \text{(1)} \]

In Equation 1 k is the thermal conductivity in W/m°C, q is the conduction heat flux in W/m² along the x direction given by the ratio of total heat transfer by conduction Q and area normal to the heat flow direction A and T represents the temperature. In practice Equation 1 is replaced by

\[ k = \frac{Q}{A \frac{\Delta T}{\delta}} \quad \text{(2)} \]

Here \(|\Delta T|\) represents the absolute value of the temperature difference across a thickness \(\delta\) of the medium. Several assumptions are made in writing the above:

- Heat conduction is one dimensional
- The temperature variation is linear along the direction of heat flow
- The above assumption presupposes that the thermal conductivity is a weak function of temperature or the temperature difference is very small compared to the mean temperature of the medium

With this background, the following general principles may be enunciated, that are common to all methods of measurement of thermal conductivity:

- Achieve one dimensional temperature field within the medium
- Measure heat flux
- Measure temperature gradient
- Estimate thermal conductivity
- In case of liquids and gases suppress convection
- Parasitic losses are reduced/eliminated/estimated and are accounted for – in all cases
(i) Steady state methods

Guarded hot plate apparatus: solid sample

The guarded hot plate apparatus is considered as the primary method of measurement of thermal conductivity of solid materials that are available in the form of a slab (or plate or blanket forms). The principle of the guard has already been dealt with in the case of heat flux measurement in Module 3. It is a method of reducing or eliminating heat flow in an unwanted direction and making it take place in the desired direction. At once it will be seen that one-dimensional temperature field in the material may be set up using this approach in a slab of material of a specified area and thickness. Schematic of a guarded hot plate apparatus is shown in Figure 1. Two samples of identical size are arranged symmetrically on the two sides of an assembly consisting of main and guard heaters. The two heaters are energized by independent power supplies with suitable controllers. Heat transfer from the lateral edges of the sample is prevented by the guard backed by a thick layer of insulation all along the periphery. The two faces of each of the samples are maintained at different temperatures by heaters on one side and the cooling water circulation on the other side. However identical one-dimensional temperature fields are set up in the two samples.
The details of the main and guard heaters along with the various thermocouples that are used for the measurement and control of the temperatures are shown in the plan view shown in Figure 2. As usual there is a narrow gap of 1 – 2 mm all round the main heater across which the temperature difference is measured and maintained at zero by controlling the main and guard heater inputs. However the sample is monolithic having a surface area the same as the main, guard and gap all put together. Temperatures are averaged using several thermocouples that are fixed on the heater plate and the water cooled plates on the two sides of the samples. The thermal conductivity is then estimated based on Equation 2, where
the heat transfer across any one of the samples is half that supplied to the main heater and the area is the face area of one of the samples.

Typically the sample, in the case of low conductivity materials, is 25 mm thick and the area occupied by the main heater is 200×200 mm. The heat input is adjusted such that the temperature drop across the sample is of the order of 5°C. In order to improve the contact between heater surface and the sample surface a film of high conductivity material may be applied between the two. Many a time an axial force is also applied using a suitable arrangement so that the contact between surfaces is thermally good.
Example 1

A guarded hot plate apparatus is used to measure the thermal conductivity of an insulating material. The specimen thickness is 25 ± 0.5 mm. The heat flux is measured within 1% and is nominally 80 W/m². The temperature drop across the specimen under the steady state is 5 ± 0.2°C. Determine the thermal conductivity of the sample along with its uncertainty.

1. The given data is written down as (all are nominal values)

   \[ q = 80 \text{ W/m}^2, \quad \Delta T = 5^\circ\text{C}, \quad \delta = 25 \text{ mm} = 0.025 \text{ m} \]

2. Using Equation 2, the nominal value of the thermal conductivity is

   \[ k = \frac{q\delta}{\Delta T} = \frac{80 \times 0.025}{5} = 0.4 \text{ W/m}^\circ\text{C} \]

3. The uncertainties in the measured quantities specified in the problem are

   \[ \delta q = \pm \frac{q}{100} = \pm \frac{80}{100} = \pm 0.8 \text{ W/m}^2, \quad \delta (\Delta T) = \pm 0.2^\circ\text{C}, \]

   \[ \delta \delta = \pm 0.5 \text{ mm} = \pm 0.0005 \text{ m} \]

4. Logarithmic differentiation is possible and the error in the thermal conductivity estimate may be written down as
\[ \Delta k = \pm k \sqrt{\left(\frac{\delta q}{q}\right)^2 + \left(\frac{\delta (\Delta T)}{\Delta T}\right)^2 + \left(\frac{\delta \delta}{\delta}\right)^2} \]

\[ = \pm 0.4 \times \sqrt{\left(\frac{0.8}{80}\right)^2 + \left(\frac{0.2}{5}\right)^2 + \left(\frac{0.0005}{0.025}\right)^2} = \pm 0.018 \text{ W} / \text{m}^\circ\text{C} \]

Thus the thermal conductivity is estimated within an error margin of

\[ \pm \frac{0.018}{0.4} \times 100 \approx \pm 4.6\% . \]
**Guarded hot plate apparatus: liquid sample**

Measurement of thermal conductivity of liquids (and gases) is difficult because it is necessary to make sure that the liquid is stationary. In the presence of temperature variations and in the presence of gravity the liquid will start moving around due to natural convection. There are two ways of immobilizing the fluid:

a) Use a thin layer of the fluid in the direction of temperature gradient so that the Grashof number is very small and the regime is conduction dominant

b) Set up the temperature field in the fluid such that the hot part is above the cold part and hence the layer is in the stable configuration. The guarded hot plate apparatus is suitably modified to achieve these two conditions.

![Diagram of Guarded hot plate apparatus](image)

**Figure 3 Guarded hot plate apparatus for the measurement of thermal conductivity of liquids**

Figure 3 shows schematically how the conductivity of a liquid is measured using a guarded hot plate apparatus. The symmetric sample arrangement in the case of a solid is replaced by a single layer of liquid sample with a guard heater on the top side. Heat flow across the liquid layer is downward and hence the liquid layer
is in a stable configuration. The thickness of the layer is chosen to be very small (of the order of a mm) so that heat transfer is conduction dominant. The guard heat input is so adjusted that there is no temperature difference across the gap between the main and the guard heaters. It is evident that all the heat input to the main heater flows downwards through the liquid layer and is removed by the cooling arrangement. Similarly the heat supplied to the guard heater is removed by the cooling arrangement at the top.

**Radial heat conduction apparatus for liquids and gases:**

Another apparatus suitable for the measurement of thermal conductivity of fluids (liquids and gases) is one that uses radial flow of heat through a very thin layer of liquid or gas. A cross sectional view of such an apparatus is shown in Figure 4. Heater is in the form of a cylinder (referred to as bayonet heater or the plug) and is surrounded by a narrow radial gap that is charged with the liquid or the gas whose thermal conductivity is to be measured. The outer cylinder is actually a jacketed cylinder that is cooled by passing cold water. Heat loss from the bayonet heater except through the annular fluid filled gap is minimized by the use of proper materials. Thermocouples are arranged to measure the temperature across the fluid layer. Since the gap (the thickness of the fluid layer) is very small compared to the diameter of the heater heat conduction across the gap may be very closely approximated by that across a slab. Hence Equation 2 may be used in this case also.
Figure 4 Radial heat flow apparatus for liquids and gases

Typical specifications of an apparatus of this type are given below:

- Diameter of cartridge heater $D = 37$ mm
- Radial clearance = 0.3 mm
- Heat flow area $A = 0.0133$ m$^2$
- Temperature difference across the gap $\Delta T \sim 5^\circ$C
- Heater input $Q = 20 – 30$ W

The diameter is about 75 times the layer thickness. The use of this apparatus requires a calibration experiment using a fluid of known thermal conductivity (usually dry air) filling the gap. Thermal conductivity of air is well known (as a function of temperature) and is available as tabulated data. If an experiment is conducted with dry air, heat transferred across the gap may be determined using Fourier law of heat conduction. The heat input to the bayonet heater is measured and the difference between these two should represent the heat loss. The experiment may be conducted with different amounts of heat input (and hence different temperature difference across the air layer) and the heat loss estimated. This may be represented as a function of the temperature difference.
across the gap. When another fluid is in the annular gap, the heat loss will still be given by the previously measured values. Hence the heat loss may be deducted from the heat input to get the actual heat transfer across the fluid layer. At once Equation 2 will give us the thermal conductivity of the fluid.

Heat loss data has been measured in an apparatus of this kind and is shown as a plot in Figure 5. The data shows mild nonlinear behavior. Hence the heat loss may be represented as a polynomial function of the temperature difference across the gap using regression analysis. The heat loss is a function of 

\[ \theta = T_P - T_J \]

and is given by the polynomial

\[ L = 0.0511 + 0.206\theta + 0.0118\theta^2 - 0.000153\theta^3 \]  \hspace{1cm} (3)

In the above \( T_P \) is the plug temperature and \( T_J \) is the jacket temperature.

![Figure 5 Heat loss calibration data for a radial flow thermal conductivity apparatus](image-url)
Example 2

- A radial heat flow apparatus has the following specifications:
  
  \[ \delta = 0.3 \text{ mm. Heat flow area } A = 0.0133 \text{ m}^2 \]

- The following data corresponds to an experiment performed with unused engine oil (SAE 40):
  
  Heater voltage: \( V = 40 \text{ Volts, Heater resistance: } R = 53.5 \Omega \)
  Plug temperature: \( T_P = 32.9^\circ C, \) Jacket temperature: \( T_J = 28.2^\circ C \)

- What is the thermal conductivity of the oil sample? If the measured parameters have the following uncertainties what will be the uncertainty in the estimated value of the thermal conductivity?
  
  \[ \Delta V = \pm 0.5 \text{ V, } \Delta T = \pm 0.2^\circ C \]

- The heat loss is a function of \( \theta = T_P - T_J \) and is given by the polynomial
  
  \[ L = 0.0511 + 0.206\theta + 0.0118\theta^2 - 0.000153\theta^3 \]

  The heat loss itself is estimated by the above formula with an error bar of \( \pm 0.5\% \).

- First we determine the nominal value of the thermal conductivity using the nominal values of all the measured quantities.
  The electrical heat input to the heaters is given by
  
  \[ Q_e = \frac{V^2}{R} = \frac{40^2}{53.5} = 29.91 \text{ W} \]

- The temperature drop across the sample liquid is
  
  \[ \theta = T_P - T_J = 32.9 - 28.2 = 4.7^\circ C \]

- The heat loss may at once be calculated as
  
  \[ L = 0.0511 + 0.206 \times 4.7 + 0.0118 \times 4.7^2 - 0.000153 \times 4.7^3 = 1.26 \text{ W} \]
(This agrees with the value shown in Figure 5)

- The heat conducted across the liquid layer is then given by

\[ Q_c = Q_e - L = 29.91 - 1.26 = 28.65 \text{ W} \]

- Using Equation 2 the nominal value of the thermal conductivity of oil sample is

\[
k = \frac{Q_c}{A \delta} = \frac{28.65 \times 0.3 \times 10^{-3}}{0.0133 \times 4.7} = 0.138 \text{ W/m°C}
\]

- Now we calculate the uncertainty in the nominal value of the thermal conductivity estimated above. Only the heat transferred and the temperatures are susceptible to error. We know that

\[ Q_c = Q_e - L = \frac{V^2}{R} - L. \]

Assuming that \( R \) is not susceptible to any error, we have

\[
\frac{\partial Q_c}{\partial V} = \frac{2V}{R} = \frac{2 \times 40}{53.5} = 1.495 \text{ W/V}, \quad \frac{\partial Q_c}{\partial L} = -1.
\]

Hence the error in the measured value of the heat conducted across the liquid layer is

\[
\delta Q_c = \pm \sqrt{\left(\frac{\partial Q_c}{\partial V} \Delta V\right)^2 + \left(\frac{\partial Q_c}{\partial L} \Delta L\right)^2} = \pm \sqrt{(1.495 \times 0.5)^2 + (-1 \times 0.05 \times 1.26)^2} = \pm 0.75 \text{ W}
\]

- The errors in the measured temperatures are equal and hence the error in the measured temperature difference is

\[
\delta \theta = \pm \sqrt{2} \Delta T = \pm \sqrt{2} \times 0.2 = \pm 0.283 \text{ °C}.
\]

The error propagation formula gives
\[
\delta k = \pm k \sqrt{\left(\frac{\delta \frac{Q}{Q_c}}{Q_c}\right)^2 + \left(\frac{\delta \theta}{\theta}\right)^2}
\]

\[
= \pm 0.138 \times \sqrt{\left(\frac{0.75}{28.65}\right)^2 + \left(\frac{0.28}{4.7}\right)^2} = \pm 0.066 \text{ W/m°C}
\]

Thus the measured value of thermal conductivity of oil sample is 0.138 ± 0.066 W/m°C.

**Thermal conductivity comparator**

Thermal conductivity comparator is a method in which the thermal conductivity of a sample is obtained by comparison with another sample of known thermal conductivity. This method is especially useful for the determination of thermal conductivity of good conductors, such as metals and alloys. The principle of the method may be explained with reference to Figure 6.

![Figure 6 Schematic of a thermal conductivity comparator](image)

A sample of standard reference material (SRM - a material whose thermal conductivity is known and guaranteed by the manufacturer) is placed in series with the material whose thermal conductivity needs to be estimated. Both the materials have identical cross section (usually cylindrical) and heat is allowed to...
flow, under the steady state, as indicated in the figure. Thermocouples are arranged as shown in order to estimate the temperature gradients in each material. Heat loss in the lateral direction is prevented by the provision of insulation as shown. With the nomenclature of Figure 6, we have the following:

\[
\frac{k_A \Delta T_A}{L_A} = \frac{k_B \Delta T_B}{L_B} \quad \text{or} \quad k_B = k_A \frac{L_B}{L_A} \frac{\Delta T_A}{\Delta T_B}
\]  

(4)

The above expression is based on the fact that the conduction heat fluxes through the sample and the SRM are the same. The lengths and temperature differences are the measured quantities and \( k_A \) is the known thermal conductivity of the SRM.
Example 3

- A thermal conductivity comparator uses a standard reference material (SRM) of thermal conductivity $45 \pm 2\% \text{ W/m-K}$. Two thermocouples placed $22 \pm 0.25 \text{ mm}$ apart indicate a temperature difference of $2.5 \pm 0.2^\circ \text{C}$. The material of unknown thermal conductivity is in series with the SRM and indicates a temperature difference of $7.3\pm0.2^\circ \text{C}$ across a length of $20 \pm 0.25 \text{ mm}$. Determine the thermal conductivity of the sample and its uncertainty.

- The given data may be written down using the nomenclature of Figure 6.

\[
k_A = 45 \text{ W/m}^\circ \text{C}, \quad \Delta T_A = 2.5^\circ \text{C}, \quad \Delta T_B = 7.3^\circ \text{C}, \quad L_A = 22 \text{ mm}, \quad L_B = 20 \text{ mm}
\]

- The nominal value of the thermal conductivity of the sample is then given by

\[
k_B = k_A \frac{L_B}{L_A} \frac{\Delta T_A}{\Delta T_B} = 45 \times \frac{20}{22} \times \frac{2.5}{7.3} = 14 \text{ W/m}^\circ \text{C}
\]

- The uncertainties specified are

\[
\delta k_A = \pm 2\%, \quad \delta L_A = \pm \frac{0.25}{22} \times 100 = 1.14\%, \quad \delta L_B = \pm \frac{0.25}{20} \times 100 = 1.25\%,
\]

\[
\delta \Delta T_A = \pm \frac{0.2}{2.5} \times 100 = 8\%, \quad \delta \Delta T_B = \pm \frac{0.2}{7.3} \times 100 = 2.74\%
\]
Since the unknown thermal conductivity depends on the other quantities involving only products of ratios, the percentage error may be directly calculated using percent errors in each of the measured quantities. Thus

\[
\delta k_B = \pm \sqrt{\left(\delta k_A\right)^2 + \left(\delta L_A\right)^2 + \left(\delta L_B\right)^2 + \left(\delta T_A\right)^2 + \left(\delta T_B\right)^2}
\]

\[
= \pm \sqrt{(2)^2 + (1.14)^2 + (1.25)^2 + (8)^2 + (2.74)^2} = \pm 8.85\%
\]

The uncertainty in the thermal conductivity is thus equal to

\[
\delta k_B = \pm 14 \times \frac{8.85}{100} = \pm 1.24 \, W/m^\circ C.
\]

(ii) Unsteady method

Though many methods are available under the unsteady category only one of them, viz. the laser flash method, will be considered as a representative one. Also it is the most commonly used method in laboratory practice and laser flash apparatus are available commercially, though very expensive.

The laser flash method imposes a pulse of heat to a thin sample and monitors the back surface temperature as a function of time. The schematic of the laser flash apparatus is shown in Figure 7.
The sample is in the form of a thin slab and is maintained at the desired temperature by placing it in a furnace. The front face of the slab is heated with a laser or flash pulse and the temperature of the back face is monitored as a function of time. The laser or flash pulse is of such intensity that the temperature will rise by only a few degrees. In other words the temperature rise is very small in comparison with the mean temperature of the sample. If the thermal diffusivity of the material of the sample is $\alpha$, the non-dimensional time is defined as $F_0 = \frac{\alpha t}{L^2}$. The non-dimensional temperature is defined as $\theta = \frac{T}{T_{\text{max}}}$ where $T_{\text{max}}$ is the maximum temperature reached by the back surface of the sample. The following plot (Figure 8) shows the shape of the response using the non-dimensional coordinates. Note that it is not necessary to know the temperatures in absolute terms since only the ratio is involved. It is found that the response is 0.5 at a non-dimensional time is 1.37 as indicated in the figure. The quantity that is estimated from the measurement is, in fact, the thermal diffusivity of the material. Thus if $t_{1/2}$ is the time at which the response is 0.5, we have

$$\alpha = 1.37 \frac{L^2}{t_{1/2}}$$  \hspace{1cm} (5)
If the density $\rho$ and specific heat capacity $c$ of the material are known (they may be measured by other methods, as we shall see later) the thermal conductivity is obtained as

$$k = \rho c \alpha$$ \hspace{1cm} (6)

In practice the temperature signal is amplified and manipulated by computer software to directly give the estimate of the thermal diffusivity of the solid sample.

**Figure 8 Response at the back surface**

**b) Measurement of heat capacity**

Heat capacity is an important thermo-physical property that is routinely measured in the laboratory. In material characterization heat capacity is one of the important properties whose changes with temperature indicate changes in the material itself. The method used for measurement of heat capacity is usually a calorimetric method, where energy balance in a controlled experiment gives a measure of the heat capacity.

**Heat capacity of a solid:**
We consider first the measurement of heat capacity of a solid material. The basic principle of the calorimetric method is the application of the first law of thermodynamics (heat balance) in a carefully conducted experiment. A weighed sample of solid material, in granular or powder form, whose heat capacity is to be measured is heated prior to being transferred quickly into a known mass of water or oil (depending on the temperature level) contained in a jacketed vessel. Let the mass of the solid be \( m \) and let it be at a temperature \( T_3 \) before it is dropped into the calorimeter. Let \( T_1 \) be the initial temperature of the calorimeter and the jacket. Let the mass specific heat product of the calorimeter, stirrer and the liquid in the calorimeter be \( C \). Let \( T_2 \) be the maximum temperature reached by the calorimeter with its contents including the mass dropped in it. Let \( c \) be the specific heat of the solid sample that is being estimated using the experimental data. We recognize that there is some heat loss that would have taken place over a period of time at the end of which the temperature has reached \( T_2 \). Let the heat loss be \( Q_L \).

**Figure 9 Calorimeter for the measurement of specific heat of a solid**

Energy balance requires that
\[ C(T_2 - T_1) + Q_L = mc(T_3 - T_2) \quad (7) \]

Now let us look at how we estimate the heat loss. For this we show the typical temperature-time plot that is obtained in such an experiment.

If there were to be no loss the temperature-time trace should be like that shown by the purple trace made up of two straight lines. After the temperature reaches \( T'_2 \) it should remain at that value! However there is heat loss and the temperature-time trace is a curve that follows the blue line. After the temperature reaches a maximum value of \( T_2 \) it decreases with time. The rate of decrease is, in fact, an indication of how large the heat loss is. If we assume that temperature rise of the calorimeter is very small during the mixing part of the experiment, we may assume the rate of heat loss to be linear function of temperature difference given by \( \dot{Q}_L = K(T - T_1) \). Now consider the state of affairs after the maximum temperature has been passed. The calorimeter may be assumed to be a first order system with

\[ C \frac{dT}{dt} + K(T - T_1) = 0 \quad (8) \]
Here we assume that K is the same as the K in the heating part (mixing part) of the experiment. If we approximate the derivative by finite differences, we may recast Equation 8 as

\[ K = C \left( \frac{\Delta T}{\Delta t} \right) \left( \frac{T - T_1}{T - T_1} \right) \]  

(9)

The numerator on the right hand side of Equation 9 is obtained by taking the slope of the cooling part of the curve at any chosen t after the cooling has started and thus at the corresponding temperature. Now we look at the heat loss term. The heat loss in the mixing part of the experiment may be obtained by integrating the rate of heat loss with respect to time between \( t = 0 \) and the time \( t_{\text{max}} \) at which the maximum temperature is reached. In fact the temperature time curve may be approximated by a triangle and hence the heat loss is

\[ Q_L = K \int_0^{t_{\text{max}}} (T - T_1) \, dt \approx K \frac{(T_2 - T_1)}{2} t_{\text{max}} \]  

(10)

This is substituted in Equation 7 and rearranged to get

\[ c = \frac{C (T_2 - T_1) + Q_L}{m(T_3 - T_2)} = \frac{C (T_2 - T_1) + K (T_2 - T_1) t_{\text{max}}}{2 m(T_3 - T_2)} \]  

(11)
Example 4

In a calorimeter experiment for determination of specific heat of glass beads, 0.15 kg of beads at 80°C are dropped into a calorimeter which is equivalent to 0.5 kg of water, at an initial temperature of 20°C. The maximum temperature of 22.7°C of the mixture occurs by linear increase of the temperature in 10 s after the mixing starts. The subsequent cooling shows that the temperature drops at 0.2°C/min when the temperature is 22.7°C. Estimate the specific heat of glass beads. Assume that the specific heat of water is 4.186 kJ/kg°C.

The given data is written down using the nomenclature introduced in the text:

\[ m = 0.015 \text{ kg}, \ T_1 = 20^\circ C, \ T_2 = 22.7^\circ C \]

\[ T_3 = 80^\circ C, \ C = 0.5 \times 4.186 = 2.093 \text{ kJ/}^\circ \text{C} \]

\[ t_{\text{max}} = 10 \text{ s}, \ \frac{\Delta T}{\Delta t} = 0.2^\circ \text{C/min} = \frac{0.2}{60} = 0.0033^\circ \text{C/s} \]

The cooling constant may be calculated as

\[ K = \frac{C \frac{\Delta T}{\Delta t}}{(T_2 - T_1)} = \frac{2.093 \times 0.0033}{(22.7 - 20)} = 0.0026 \text{ kW/}^\circ \text{C} \]

The heat loss during the mixing process is then approximated as

\[ Q_L \approx K \frac{(T_2 - T_1) t_{\text{max}}}{2} = 0.0026 \times \frac{(22.7 - 20) \times 10}{2} = 0.035 \text{ kW} \]

The specific heat of glass bead sample may now be estimated as

\[ c = \frac{C (T_2 - T_1) + Q_L}{m (T_3 - T_2)} = \frac{2.09 \times (22.7 - 20) + 0.035}{0.15 \times (80 - 22.7)} = 0.661 \text{ kJ/kg}^\circ \text{C} \]
**Heat capacity of liquids**

Essentially the same calorimeter that was used in the case of solid samples may be used for the measurement of specific heat of liquids, with the modifications shown in Figure 11. The sample liquid is taken in the calorimeter vessel and allowed to equilibrate with the temperature of the constant temperature fluid in the jacket and the rest of the works. The heater is energized for a specific time so that a measured amount of thermal energy is added to the sample liquid. Stirrer (not shown) will help the liquid sample to be at a uniform temperature. The temperature increase of the liquid at the end of the heating period is noted down. The liquid specific heat is then estimated using energy balance. Effect of heat loss may be taken into account by essentially the same procedure that was used in the earlier case.

**Figure 11 Apparatus for measurement of specific heat of liquids**
(1 – Lagging, 2 – Constant temperature bath, 3 – Heater, 4 – Sample liquid, 5 – Precision thermometer, 6 – Energy meter, 7 – Switch, 8 – Power supply, 9 – Insulating lid)
c) **Measurement of calorific value of fuels**

Calorific or heating value of a fuel is one of the most important properties of a fuel that needs to be measured before the fuel can be considered for a specific application. The fuel may be in the liquid, gaseous or solid form. The measurement of heating value is normally by calorimetry. While a continuous flow calorimeter is useful for the measurement of heating values of liquid and gaseous fuels, a bomb calorimeter is used for the measurement of heating value of a solid fuel.

**Preliminaries:**

Heating value of a fuel or more precisely, the **standard enthalpy of combustion** is defined as the enthalpy change $\Delta H^\circ$ that accompanies a process in which the given substance (the fuel) undergoes a reaction with oxygen gas to form combustion products, all reactants and products being in their respective standard states at $T = 298.15 K = 25^\circ C$. A bomb calorimeter (to be described later) is used to determine the standard enthalpy of combustion. The process that takes place is at a constant volume. Hence the process that occurs in a calorimeter experiment for the determination of the standard enthalpy of combustion is process I in Figure 12. What is desired is the process shown as III in the figure. We shall see how to get the information for process III from that measured using process I.
Figure 12 Processes in a calorimeter experiment

Let the calorimeter have a heat capacity equal to $C$. The change in the internal energy of the calorimeter when the temperature changes from $T_1$ to $T_2$ is

$$
\int_{T_1}^{T_2} C \, dT
$$

assuming that $C$ is independent of pressure. If we neglect work interaction due to stirring, I law applied to a perfectly insulated calorimeter $(Q = 0)$ shows that $\Delta U_I = 0$ during process I. In the process shown as II the products and the calorimeter are at temperature $T_1$. Thus, for process II we should have

$$
\Delta U_{II} = \Delta U_I - \int_{T_1}^{T_2} C \, dT.
$$

Thus it is clear that $\Delta U_{II} = \Delta U_{I1} = -\int_{T_1}^{T_2} C \, dT = \Delta U_{I1}^0$. The last part results by noting that the internal energy is assumed to be independent of pressure. Note that by definition $\Delta H_{T_1}^0 = \Delta U_{I1}^0 + \Delta(PV)$. Since the products are in the gaseous state, assuming the products to have ideal gas behavior, we have, $\Delta(PV) = p_2 V_2 - p_1 V_1 = (n_2 - n_1) \mathcal{R} T_1$, where the $n_2$ represents the number of moles of the product and $n_1$ the number of moles of the reactant and $\mathcal{R}$ is the
universal gas constant. Thus it is possible to get the desired heat of combustion as

$$\Delta H_{T_1}^0 = (n_2 - n_1) R_g T - \int_{T_1}^{T_2} C \, dT$$

(12)

In a typical experiment the temperature change is limited to a few degrees and hence the specific heat may be assumed to be a constant. Under this assumption Equation 12 may be replaced by

$$\Delta H_{T_1}^0 = (n_2 - n_1) R_g T - C(T_2 - T_1)$$

(13)

The Bomb calorimeter:

Schematic of a bomb calorimeter used for the determination of heating value (standard enthalpy of combustion) of a solid fuel is shown in Figure 13. The bomb is a heavy walled pressure vessel within which the combustion reaction will take place at constant volume. At the bottom of the bomb is placed sufficient
amount of water such that the atmosphere within the bomb remains saturated with water vapor throughout the experiment. This guarantees that the water that may be formed during the combustion reaction will remain in the liquid state. The bomb is immersed within a can of water fitted with a precision thermometer capable of a resolution of 0.01°C. This assembly is placed within an outer water filled jacket. The jacket water temperature remains the same both before and after the combustion within the bomb. There is no heat gain or loss to the bomb from outside and the process may be considered to be adiabatic. The fuel is taken in the form of a pellet (about 1 g) and the combustion is accomplished by initiating it by an electrically heated fuse wire in contact with the pellet. The bomb is filled with oxygen under high pressure (25 atmospheres) such that there is more than enough oxygen to guarantee complete combustion. The heating value is estimated after accounting for the heat generated by the fuse wire consumed to initiate combustion.

Benzoic acid (C₇H₆O₂ - solid) is used as a standard reference material of known heat of reaction \( \Delta H^0 = -3227 \text{ kJ/mol} \). Benzoic acid is taken in the form of a pellet and burnt in a bomb calorimeter to provide the data regarding the heat capacity of calorimeter. A typical example is given below.
Example 5

A pellet of benzoic acid weighing 0.103 g is burnt in a bomb calorimeter and the temperature of the calorimeter increases by 2.17°C. What is the heat capacity of the calorimeter?

Consider the combustion reaction. 7.5 moles of oxygen are required for complete combustion of benzoic acid.

\[ C_7H_6O_2 + 7.5O_2 \rightarrow 7CO_2 + 3H_2O \]

The change in the number of moles during the reaction is given by

\[ \Delta n = n_2 - n_1 = \frac{7}{CO_2 \text{ in the product}} - \frac{7.5}{O_2 \text{ in the reactant}} = -0.5 \]

Per mole of benzoic acid basis, we have

\[ \Delta U = \Delta H - \Delta nRT \]

\[ = -3227 - (-0.5) \times 8.3143 \times 298 \]

\[ = -3225.8 \text{ kJ/mol} \]

The molecular weight of benzoic acid is calculated as

Molecular weight = \( 12.011 \times \frac{7}{C} + 1.008 \times \frac{6}{H_2} + 15.9995 \times \frac{2}{O} \)

\[ = 122.12 \text{ g/mol} \]

Thus 0.103 g of benzoic acid corresponds to \( \frac{0.103}{122.12} = 0.000843 \text{ mol} \). The heat released by combustion of this is

\[ Q = 0.000843 \times 3225.8 = 2.7207 \text{ kJ} \]
The temperature increase of the calorimeter has been specified as $\Delta T = 2.17^\circ C$.

Hence the heat capacity of the calorimeter is

$$C = \frac{Q}{\Delta T} = \frac{2.7207}{2.17} = 1.2538 \text{ kJ} / ^\circ C$$

**Figure 14** Bomb calorimeter data for example 6
Example 6

In a subsequent experiment, the calorimeter of Example 5 is used to determine the heating value of sugar. A pellet of sugar weighing 0.303 g is burnt and the corresponding temperature rise indicated by the calorimeter is 3.99°C. What is the heat of combustion of sugar?

Figure 14 shows the variation of temperature of the calorimeter during the experiment. Since heat losses are unavoidable the temperature will start reducing as shown in the figure. By using ΔT as shown in the figure the effect of the heat losses may be accounted for. Note that the work due to stirrer may itself compensate for the heat loss to some extent. The slope of the cooling curve takes this into account naturally.

Molecular formula for sugar is C_{12}H_{22}O_{11}. The molecular weight of sugar is found as

\[
\text{Molecular weight} = 12.011 \times 12 + 1.008 \times 22 + 15.9995 \times 11 = 342.3 \text{ g/mol}
\]

The mass of sugar pellet of 0.303 g corresponds to

\[
\frac{0.303}{342.3} = 0.000885 \text{ mol}
\]

The heat release in the calorimeter is calculated based on \( \Delta T = 3.99°C \) and \( C = 1.2538 \text{ kJ/°C} \) as

\[
Q = -C\Delta T = -1.2538 \times 3.99 = -5.003 \text{ kJ}
\]

The combustion reaction for sugar is given by
Hence there is no change in the number of moles before and after the reaction. Thus the heat of reaction is no different from the heat released and thus

\[ \Delta H^0 = -\frac{5.003}{0.000885} = 5651.9 \text{ kJ/mol} \]

The next case we consider is the measurement of heating value of a gaseous fuel.

**Continuous flow calorimeter:**

Figure 15 shows the schematic of a continuous flow calorimeter used for the determination of heating value of a gaseous fuel. We assume that all the processes that take place on the gas side are at a mean pressure equal to the atmospheric pressure. The gas inlet pressure may be just “a few mm of water column gage” and hence this assumption is a good one. As the name indicates, the processes that take place in the calorimeter are in the steady state with
continuous flow of the gas air mixture (air provides oxygen for combustion) and the coolant (water) through the cooling coils. As indicated temperatures and flow rates are measured using appropriate devices familiar to us from Module 2. The gas air mixture is burnt as it issues through a nozzle that is surrounded by a cooling coil through which a continuous flow of cooling water is maintained. The enthalpy fluxes involved in the apparatus are given below:

1) Gas air mixture \( (m_g) \) entering in at room temperature \((T_{g,entry})\)
   
   Mass flow measured using venturi
   Temperature measured using a liquid in glass thermometer

2) Products of combustion leaving at a higher temperature \((T_{g,exit})\)
   
   Temperature measured using a liquid in glass thermometer

3) Cooling water \((m_w)\) entering at constant temperature \((T_{w,entry})\)
   
   Mass flow measured by rotameter

4) Cooling water stream leaves at a higher temperature \((T_{w,exit})\)
   
   Temperature measured using a liquid in glass thermometer

Energy balance requires that the following hold:

\[
m_gHV = C_{pp}T_{g,exit} - C_{pm}T_{g,entry} + m_wC_{pw}(T_{w,exit} - T_{w,entry}) \tag{14}
\]

In the above equation \(HV\) is the heating value of the fuel, \(C_{pp}\) is the specific heat of products of combustion and \(C_{pm}\) is the specific heat of gas air mixture. The determination of \(C_{pp}\) will certainly require knowledge of the composition of the products formed during the combustion process. The products of combustion have to be obtained by methods that we shall describe later. In the case of
hydrocarbon fuels with complete combustion (possible with enough excess air) the products will be carbon dioxide and water vapor. If the exit temperature of the products is above 100°C the water will be in the form of steam or water vapor. The estimated heating value is referred to as the lower heating value (LHV) as opposed to the higher heating value (HHV) that is obtained if the water vapor is made to condense by recovering its latent heat.

d) Measurement of viscosity of fluids

Viscosity of a fluid is a thermo-physical property that plays a vital role in fluid flow and heat transfer problems. Viscosity relates the shear stress within a flowing fluid to the spatial derivative of the velocity. A Newtonian fluid follows the relation

\[ \tau = \mu \frac{du}{dy} \]  

(15)

In the above \( \tau \) is the shear stress in Pa, \( u \) is the fluid velocity in m/s, \( y \) is the coordinate measured as indicated in Figure 16 and \( \mu \) is the dynamic viscosity of the fluid in Pa s or kg/m s. Viscosity is also referred in terms of Poise which is equal to 0.1 kg/m s. The ratio of dynamic viscosity to the density of the fluid is called the kinematic viscosity. The unit of \( \mu \) is m²/s. An alternate unit is the Stoke that is equal to 0.0001 m²/s.

Figure 16 Newton law of viscosity explained
The shear stress introduced through Equation 15 accounts for the slowing down of a higher velocity layer adjacent to a lower velocity layer. It also accounts for the frictional drag experienced by a body past which a fluid is flowing. Measurement of viscosity thus is based on the measurement of any one of these effects.

Three methods of measuring viscosity of a fluid are discussed here. These are based on:

i. **Laminar flow in a capillary**: The pressure drop in the fully developed part of the flow is related to the wall shear that is related in turn to viscosity.

ii. **Saybolt viscometer**: The time taken to drain a standard volume of liquid through a standard orifice is related to the viscosity.

iii. **Rotating cylinder viscometer**: The torque required to maintain rotation of a drum in contact with the liquid is used as a measure of viscosity.

**i) Laminar flow in a capillary:**

![Figure 17 Laminar flow of a fluid in a circular tube](image)
The nature of flow in a straight circular tube is shown in Figure 17. The fluid enters the tube with a uniform velocity. If the Reynolds number is less than about 2000 the flow in the tube is laminar. Because of viscous forces within the fluid and the fact that the fluid satisfies the no slip condition at the wall, the fluid velocity undergoes a change as it moves along the tube, as indicated in the figure. At the end of the so called developing region the fluid velocity profile across the tube has an invariant shape given by a parabolic distribution of \( u \) with \( y \). This region is referred to as the fully developed region. The axial pressure gradient is constant in this part of the flow and is related to the fluid viscosity and the flow velocity. The requisite background is available to you from your course on Fluid Mechanics. We make use of the results of Hagen Poiseuille flow. You may recall that this was used earlier while discussing the transient behavior of pressure sensors.

**Figure 18 Laminar flow in a capillary and viscosity measurement**

A schematic of the apparatus used for the measurement of viscosity of a liquid is shown in Figure 18. The apparatus consists of a long tube of small diameter.
(capillary) in which a steady flow is established. By connecting the capillary to a constant head tank on its upstream the pressure of the liquid is maintained steady at entry to the capillary. The pump – overflow arrangement maintains the level of the liquid steady in the constant head tank. The mean flow velocity is measured using a suitable rotameter just upstream of the capillary. The pressure drop across a length $L$ of the capillary in the developed section of the flow is monitored by a manometer as shown. The reason we choose a capillary (a small diameter tube, a few mm in diameter) is to make sure that the pressure drop is sizable for a reasonable length of the tube (say a few tens of cm) and the corresponding entry length also is not too long. If necessary the liquid may be maintained at a constant temperature by heating the liquid in the tank and by insulating the capillary. The mean temperature of the liquid as it passes through the capillary is monitored by a suitable thermometer.
Example 7

Viscosity of water is to be measured using laminar flow in a tube of circular cross section. Design a suitable set up for this purpose.

We assume that the experiment would be conducted at room temperature of say 20°C. Viscosity of water from a handbook is used as the basis for the design. From table of properties of water the dynamic viscosity at 20°C is roughly 0.001 Pa s and the density of water is approximately 1000 kg/m³. We choose a circular tube of inner diameter $D = 0.003$ m (3 mm). We shall limit the maximum Reynolds number to 500. The maximum value of mean velocity $V_{\text{max}}$ may now be calculated as

$$V_{\text{max}} = \frac{\text{Re}_D \mu}{\rho D} = \frac{500 \times 0.001}{1000 \times 0.003} = 0.167 \text{ m/s}$$

The corresponding volume flow rate is the maximum flow rate for the apparatus and is given by

$$Q_{\text{max}} = \frac{\pi}{4} D^2 V_{\text{max}} = \frac{\pi}{4} \times 0.003^2 \times 0.167 = 1.178 \times 10^{-6} \text{ m}^3 / \text{s}$$

The volume flow rate is thus about 1.2 ml/s. If the experiment is conducted for 100 s, the volume of water that is collected (in a beaker, say) is 120 ml. The volume may be ascertained with a fair degree of accuracy. The development length for the chosen Reynolds number is

$$L_{\text{dev}} = 0.05 D \text{Re}_D = 0.05 \times 0.003 \times 500 = 0.075 \text{ m}$$

If we decide to have a sizable pressure drop of, say, 1 kPa the length of tube may be decided. Using the results known to us from Hagen Poiseuille theory, we have
\[ \frac{-\Delta p}{L} = 32 \frac{vV_{\text{max}}}{D^2} \]

or \[ L = -\frac{\Delta p D^2}{32 vV_{\text{max}}} = \frac{10^3 \times 0.003^2}{32 \times 10^{-6} \times 0.167} = 1.68 \text{ m} \]

In the above the kinematic viscosity is the ratio of dynamic viscosity to the density. The total length of the tube required is \( L_{\text{total}} = L_{\text{dev}} + L = 0.075 + 1.68 \approx 1.76 \text{ m} \). It is also now clear that the head (the level of the water level in the tank with respect to the axis of the tube) should be approximately given by

\[ h = -\frac{\Delta p}{\rho g} = \frac{1000}{1000 \times 9.8} = 0.102 \text{ m} \]

This is a very modest height and may easily be arranged on a table top in the laboratory.

The above type of arrangement may also be used for the measurement of viscosity of a gas. The constant head tank will have to be replaced by a gas cylinder with a pressure regulator so that the inlet pressure can be maintained constant. The gas may have to be allowed to escape through a well ventilated hood. The flow rate may be measured by the use of a rotameter. The pressure drop may be measured using a differential pressure transducer or a U tube manometer or a well type manometer with an inclined tube.
ii). **Saybolt viscometer**

Schematic of a Saybolt viscometer is shown in Figure 19. The method consists in measuring the time to drain a fixed volume of the test liquid (60 ml) through a capillary of specified dimensions (as shown in the figure). The test liquid is surrounded by an outer jacket so that the test fluid can be maintained at the desired temperature throughout the experiment. If the drainage time is $t_s$, then the kinematic viscosity of the test liquid is given by the formula

$$
\nu = \left( \frac{1}{8.65} \right) \times \frac{8.65}{12.5} \times \frac{1}{t_s}
$$

(16)
Example 8

A Saybolt viscometer is used to measure the viscosity of engine oil at 40°C. The time recorded for draining 60 ml is 275±1 s. Calculate the viscosity and its uncertainty.

We use formula (16) with the nominal value of $t$ as 275 s to get the nominal value of kinematic viscosity of engine oil as

$$\nu_{\text{oil}} = 0.22018 \times 10^{-6} \times 275 - \frac{0.1793 \times 10^{-3}}{275}$$

$$= 59.9 \times 10^{-6} \text{ m}^2/\text{s}$$

For uncertainty calculation we first calculate the influence coefficient as

$$I_t = \frac{\text{d} \nu}{\text{d} t} = 0.22018 \times 10^{-6} + \frac{0.1793 \times 10^{-3}}{t^2}$$

$$= 0.22018 \times 10^{-6} + \frac{0.1793 \times 10^{-3}}{275^2} = 2.23 \times 10^{-7}$$

The uncertainty in the estimated (nominal) value of the kinematic viscosity is then given by

$$\Delta \nu_{\text{oil}} = \pm \frac{\text{d} \nu_{\text{oil}}}{\text{d} t} \times \Delta t = \pm 2.24 \times 10^{-7} \times 1 = \pm 2.23 \times 10^{-7} \text{ m}^2/\text{s}$$

The percent uncertainty in the nominal value of the kinematic viscosity is

$$\Delta \nu_{\text{oil}} = \pm \frac{2.23 \times 10^{-7}}{59.9 \times 10^{-6}} \times 100 \text{ m}^2/\text{s} = 0.37\%$$
It is well known that the viscosity of oil varies very significantly with temperature. Hence the drainage time of 60 ml of oil in a Saybolt viscometer varies over a wide range as the temperature of oil is varied. Typically the drainage time varies from around 4000 s at 0°C to about 35 s at 160°C, as shown in Figure 20.

iii) **Rotating cylinder viscometer:**

When a liquid layer between two concentric cylinders is subjected to shear by rotating one of the cylinders with respect to the other the stationary cylinder will experience a torque due to the viscosity of the liquid. The torque is measured by a suitable technique to estimate the viscosity of the liquid. The schematic of a rotating cylinder viscometer is shown in Figure 21. It consists of an inner stationary cylinder and an outer rotating cylinder that is driven by an electric motor. There are narrow gaps a and b where the liquid whose viscosity is to be determined is trapped. The torque experienced by the stationary cylinder is related to the liquid viscosity as the following analysis shows.
Figure 21 Schematic of a rotating cylinder viscometer

The nomenclature used in the following derivation is given in Figure 21. If the gap \( a \) between the two cylinders is very small compared to the radius of any of the cylinders we may approximate the flow between the two cylinders to be coquette flow familiar to us from fluid mechanics. The velocity distribution across the gap \( a \) is linear and the constant velocity gradient is given by \( \frac{du}{dr} = \frac{\omega r_2}{b} \). The uniform shear stress acting on the surface of the inner cylinder all along its length \( L \) is then given by \( \tau = \mu \frac{du}{dr} = \mu \frac{\omega r_2}{b} \). The torque experienced by the inner cylinder due to this is

\[
T_L = \frac{\mu \omega r_2}{b} \times \frac{2 \pi r_1 L \times r_1}{b} = \frac{2 \pi \mu \omega r_1^2 r_2}{b} \tag{17}
\]

There is another contribution to the torque due to the shear stress at the bottom of the cylinder due to the liquid film of thickness \( a \). At a radial distance \( r \) from the center of the stationary cylinder the shear stress is given by \( \tau = \mu \frac{\omega r}{a} \). Consider
an elemental area in the form of a circular strip of area $2\pi r \, dr$. The elemental torque on the stationary cylinder due to this is given by $dT_b = \frac{\mu \omega r}{a} \times 2\pi r \, dr \times \frac{r}{\pi} = \frac{2\pi \mu \omega}{a} r^2 \, dr$. The total torque will be obtained by integrating this expression from $r = 0$ to $r_1$. Thus we have

$$T_b = \frac{2\pi \mu \omega}{a} \int_0^{r_1} r^2 \, dr = \frac{\pi \mu \omega r_1^4}{2a}$$

(18)

The total torque experienced by the stationary cylinder is obtained by adding expressions (17) and (18). Thus

$$T = \mu \pi \omega r_1^2 \left[ \frac{2r_1 L}{b} + \frac{r_1^2}{2a} \right]$$

(19)
Example 9

A rotating cylinder apparatus is run at an angular speed of $1800 \pm 5 \text{ rpm}$. The geometric data is specified as:

\[ r_1 = 37 \pm 0.02 \text{ mm}, \quad r_2 = 38 \pm 0.02 \text{ mm}, \quad L = 100 \pm 0.5 \text{ mm} \quad \text{and} \quad a = 1 \pm 0.01 \text{mm}. \]

What is the torque experienced by the stationary cylinder? What is the power dissipated? Perform an error analysis. The fluid in the viscometer is linseed oil with a viscosity of $\mu = 0.0331 \text{ kg/m-s}$.

First we use the nominal values to estimate the nominal value of the viscosity. We convert the angular speed from rpm to rad/s as

\[ \omega = \frac{1800}{60} \times \frac{2\pi}{\text{Radian per revolution}} = 188.5 \text{ rad/s} \]

The thickness of linseed oil film over the cylindrical portion is

\[ b = r_2 - r_1 = 38 - 37 \text{ mm} = 1 \text{ mm} = 0.001 \text{m} \]

The thickness of oil film at the bottom $a$ is also equal to 0.001 m. We use Equation 19 to estimate the nominal value of the torque as

\[
T = \mu \pi \omega^2 \left[ \frac{2r_2 L}{b} + \frac{r_1^2}{2a} \right]
\]

\[
= 0.0331 \times \pi \times 188.5 \times 0.037^2 \left[ \frac{2 \times 0.038 \times 0.1}{0.001} + \frac{0.037^2}{2 \times 0.001} \right]
\]

\[
= 0.222 \text{ N m}
\]

The influence coefficients are now calculated.
The errors in various measured quantities are taken from the data supplied in the problem. The expected error in the estimated value for the torque is

\[
\Delta T = \pm \sqrt{(I_{r1} \Delta r_i)^2 + (I_{r2} \Delta r_2)^2 + (I_a \Delta a)^2 + (I_b \Delta b)^2 + (I_L \Delta L)^2 + (I_\omega \Delta \omega)^2}
\]

\[
= \pm \sqrt{\left(1.986 \times \frac{0.02}{1000}\right)^2 + \left(5.367 \times \frac{0.02}{1000}\right)^2 + \left(36.736 \times \frac{0.02}{1000}\right)^2 + \left(263.492 \times \frac{0.02}{1000}\right)^2 + \left(2.039 \times \frac{0.04}{1000}\right)^2 + \left(0.00118 \times \frac{5 \times 2 \times \pi}{60}\right)^2}
\]

\[
= \pm 0.005 \text{ N m}
\]

Note that the factor 1000 in the denominator of some of the terms is to convert the error in mm to error in m. The error in the rpm is converted to appropriate error in \(\omega\) using the factor \(2\pi/60\). We have assumed that the viscosity of linseed oil taken from table of properties has zero error!