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KING FAISAL UNIVERSITY
College Of Engineering

DEPARTMENT OF CHEMICAL ENGINEERING

CHE 405: Chemical Engineering lab II

"Lab Manual"

Major Topics covered and schedule in weeks:

Topic	Week #	Courses Covered
Introduction to lab safety.	1	3
Experiment 1: Continuous stirred tank reactor	2	3
Experiment 2: Dynamic behavior of CSTR	3	3
Experiment 3 Reaction rate data using batch reactor	4	3
Experiment 4: Reaction rate data using tubular reactor	5	3
Experiment 5: Reaction rate data using plug flow reactor	6	3
Experiment 6 : Batch Enzyme Reactor (chemicals ordered)	7	3
Experiment 7: Pressure measurement and calibration	8	3
Experiment 8: Temperature measurement and calibration	9	3
Experiment 9: Conversion of work to heat	10	3
Experiment 10: Stirling Cycle Hot Air Engine (conversion of heat to work)	11	3
Experiment 11: Bomb calorimeter	12	3
Design of an experiment	13	3
Final Exam	14	

Specific Outcomes of Instruction (Lab Learning Outcomes):

1. Identify the different types of laboratory reactors, and work with them safely. **(6)**
2. Analyze the experimental data to obtain reaction rate expression (reaction order and specific reaction rate constant)**.(1, 6)**
3. Measure the thermodynamics properties such as temperature, pressure, heat, work and calorific value **(6)**
4. Analyze and interpret experimental data with theories learned in previous courses. **(1, 6)**
5. Write organized and cohesive technical reports. **(3)**
6. Work effectively in a team environment**. (5)**

1. Student Outcomes (SO) Addressed by the Lab:

z	Outcome Description General Engineering Student Outcomes	Contribution
1.	an ability to identify, formulate, and solve complex engineering problems by applying principles of engineering, science, and mathematics	H
2.	an ability to apply engineering design to produce solutions that meet specified needs with consideration of public health, safety, and welfare, as well as global, cultural, social, environmental, and economic factors	
3.	an ability to communicate effectively with a range of audiences	M
4.	an ability to recognize ethical and professional responsibilities in engineering situations and make informed judgments, which must consider the impact of engineering solutions in global, economic, environmental, and societal contexts	
5.	an ability to function effectively on a team whose members together provide leadership, create a collaborative and inclusive environment, establish goals, plan tasks, and meet objectives	H
6.	an ability to develop and conduct appropriate experimentation, analyze and interpret data, and use engineering judgment to draw conclusions	H
7.	an ability to acquire and apply new knowledge as needed, using appropriate learning strategies	

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APPENDIX

Experiment # 1: CONTINUOUS STIRRED TANK REACTOR

I. Objectives:

1. To study the practical working of a laboratory scale stirred tank reactor.
2. To demonstrate the mechanism of a chemical reaction in the CSTR.
3. To calculate the specific rate constant *k*.

II. Theory:

The continuous stirred tank reactor in the form of either a single tank or (more often) tanks in series, is used widely and is particularly suitable for liquid phase reactions. It is particularly used in the organic chemicals industry. Advantages include consistent product quality, straightforward automatic control and low manpower requirements.

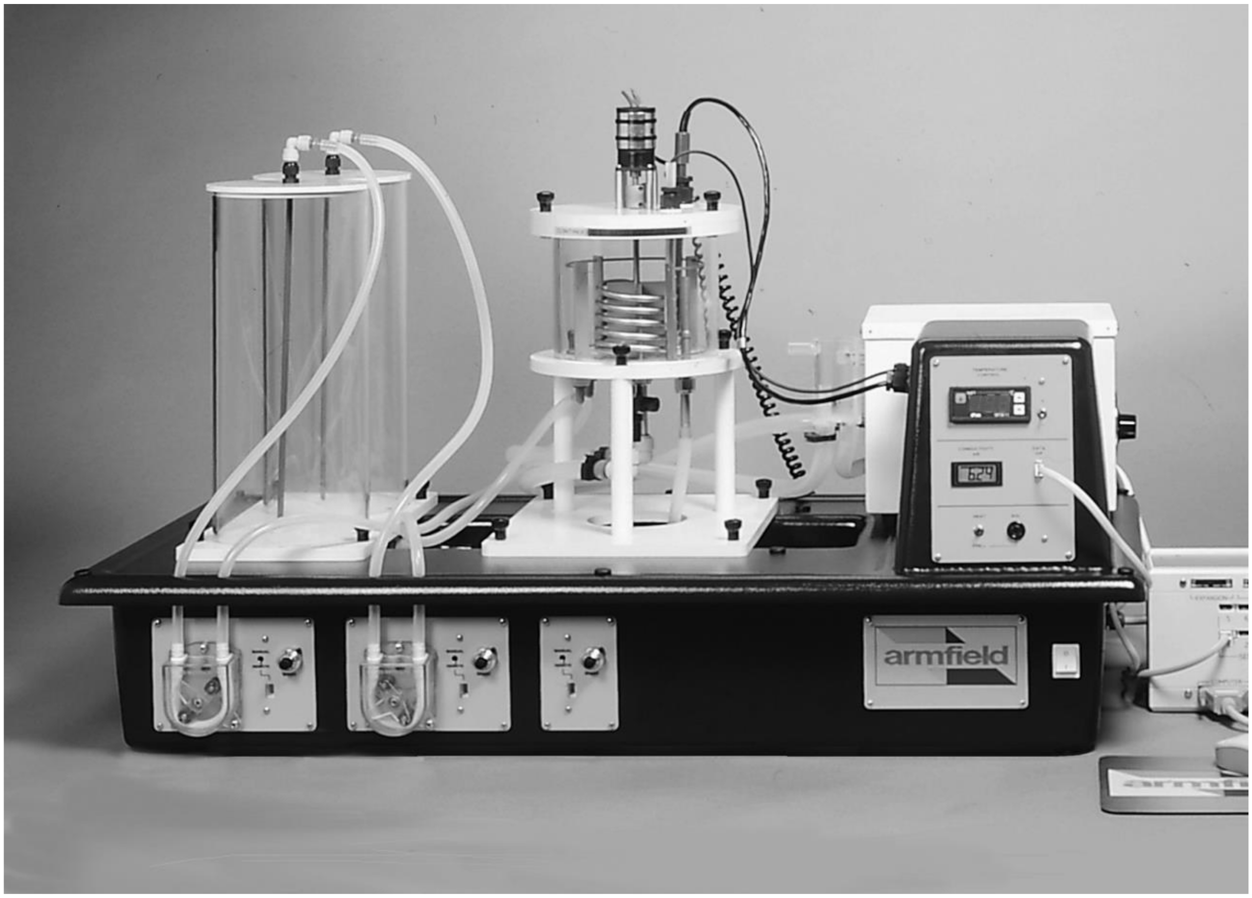


Figure 1: Continues Stirred Tank Reactor

Reactions are monitored by conductivity probe as the conductivity of the solution changes with conversion of the reactants to product. This means that the inaccurate and inconvenient process of titration, which was formally used to monitor the reaction progress, is no longer necessary.

The reaction carried out in this experiment is saponification of Ethyl Acetate by Sodium Hydroxide according to this equation:

$$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$$

$$\text{Sodium Hydroxide} + \text{Ethyl Acetate} \rightarrow \text{Sodium Acetate} + \text{Ethyl Alcohol}$$

In the above reaction, the reactant are sodium hydroxide and ethyl acetate which react and form sodium acetate and ethyl alcohol. The reaction can be considered as equi-molar and first order with respect to both sodium hydroxide and ethyl acetate, i.e. second order overall, within the limits of concentration (0 - 0.1M) and temperature ($20 - 40$°C) studied.

III. Reactor Vessel Description:

The reactor vessel is set on a baseplate which is designed to be located on the four studs of the CEX service unit and then secured by thumbnuts. The reactor is supported by three pillars; position the reactor on the CEX service unit such that a single pillar is to the front.

A stainless steel coil inside the reactor provides the heat transfer surface for either heating or cooling the chemical reactants. The coil is connected either to the hot water circulator or the CW-16 chiller. The coil inlet is at the front of the reactor and the coil return is at the rear of the reactor.

A turbine agitator works in conjunction with a baffle arrangement to provide efficient mixing and heat transfer. The agitator is driven by an electric motor mounted on the lid of the reactor. The motor is driven by a variable speed unit mounted in the front of the service unit. The socket for the motor electrical plug is sited at the rear of the service unit.

Glands in the reactor lid house the conductivity and temperature sensors provided with the service unit. The larger of the two glands is for the conductivity probe. The glands are unscrewed by hand, the probes inserted completely into the reactor until they rest on the reactor base and then the glands re-tightened by hand. Sockets in the side of the console on the service unit are provided to connect each probe. These are of different size so that the probes cannot be wrongly connected.

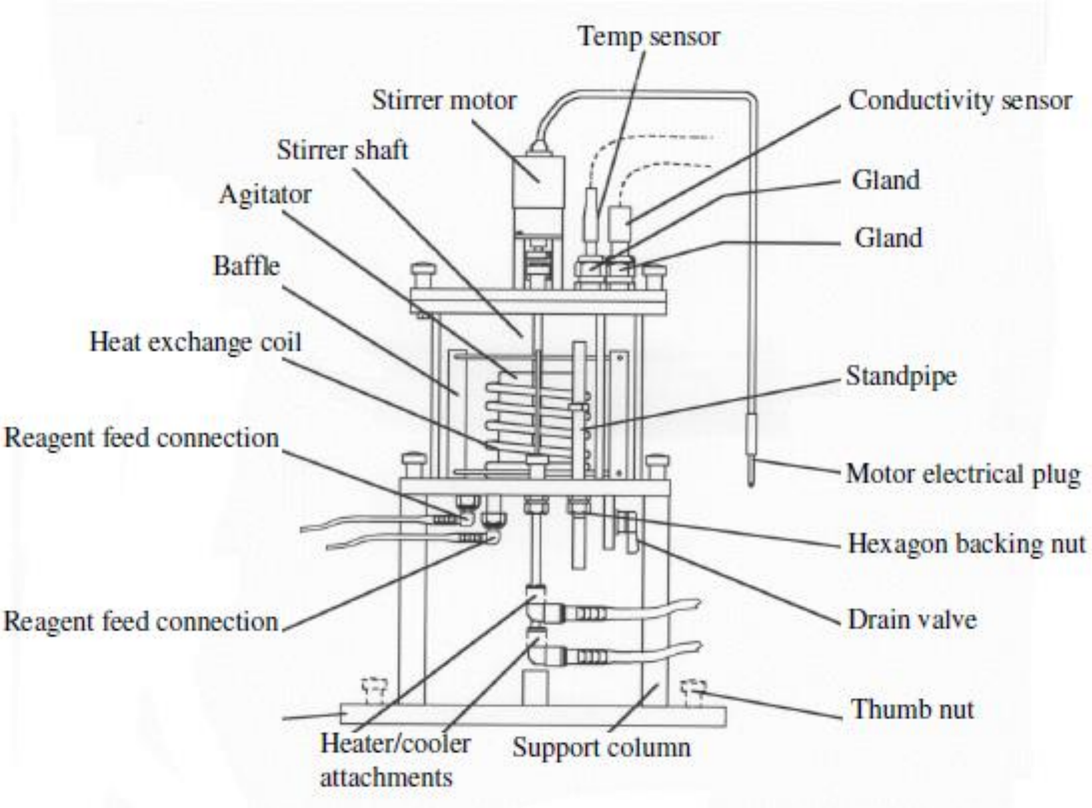


Figure 2: Schematic graph Continues Stirred Tank Reactor components

IV. Procedure:

A. Solutions Preparation :

1- Prepare 0.1 M solution of Ethyl Acetate.
2- Prepare 0.1 M solution of Sodium Hydroxide.

B. Calibrating the feed pumps :

1- Fill both reagent vessels with water.
2- Set the feed pump to 2.0 and determine the flow rate by measuring the volume collected for a defined period.
3- Repeat the procedure for 4.0, 6.0, 8.0 and 10.0.
4- Plot a calibration graph for each pump of speed vs. flowrate (ml/min).

Note: Both pumps have to be calibrated because there might be a slight difference between them.

C. Experimental procedure for CSTR operation :

1- Remove the lids of the reagent vessels and carefully fill with the reagents to a level approximately 50mm from the top. Refit the lids.
2- Set the overflow stand pipe of the reactor vessel to the minimum level, this will ensure that reactor volume is set at 1.0 L.
3- Adjust the set point of the temperature controller to 30°C.
4- Using the calibration graph for each of the feed pumps, set the pump speed control to give 40 ml/min flowrate.
5- Set the agitator speed controller to 8.0.
6- Switch on both feed pumps and the agitator motor.
7- Switch on the hot water circulator when the sensor tip is covered (about 25mm of liquid in reactor).
8- Once the temperature of the fluid reaches 30 °C, start collection of conductivity data until (each one minute) a steady state condition is reached in the reactor and this takes approximately 30 minutes. It is advisable to collect the data for 45 minutes.
9- Now drain off the reactor vessel and repeat the entire experiment (other than Pumps calibration) but with the stirrer switch off.

V. Raw Data:

Time Vs Conductivity							
With Stirring				Without stirring			
Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)
1		24		1		24	
2		25		2		25	
3		26		3		26	
4		27		4		27	
5		28		5		28	
6		29		6		29	
7		30		7		30	
8		31		8		31	
9		32		9		32	
10		33		10		33	
11		34		11		34	
12		35		12		35	
13		36		13		36	
14		37		14		37	
15		38		15		38	
16		39		16		39	
17		40		17		40	
18		41		18		41	
19		42		19		42	
20		43		20		43	
21		44		21		44	
22		45		22		45	
23				23			

VI. Data Interpretation:

The conductivity of the solution is used to determine the conversion achieved within the reactor. While both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst ethyl acetate ethyl alcohol do not. The conductivity of sodium hydroxide solution at a given concentration and temperature however, is not the same as that of sodium acetate solution at the same molarity and temperature, and a relationship has been established allowing conversion to be inferred from conductivity.

You will need the following known constants for your calculations:

F_a	volume feed rate of sodium hydroxide (dm^3/s)
F_b	volume feed rate of ethyl acetate (dm^3/s)
a_μ	sodium hydroxide concentration in Feed vessel ($mol/\ dm^3$)
b_μ	ethyl acetate concentration in Feed vessel ($mol/\ dm^3$)
T	temperature (K)
V	volume of reactor

Now you need to calculate the reactant concentration entering the reactor, which is

$$a_0 = \left(\frac{F_a}{F_a+F_b}\right) * a_\mu \tag{1}$$

$$b_0 = \left(\frac{F_b}{F_a+F_b}\right) * b_\mu \tag{2}$$

$$C_\infty = b_0 \qquad \text{for} \qquad b_0 < a_0 \tag{3}$$

$$C_\infty = a_0 \qquad \text{for} \qquad b_0 \geq a_0 \tag{4}$$

The conductivity associated with the reactants and products are given by:

$$\Lambda_{c\infty} = 0.07[1 + 0.0284(T - 294)]c_\infty \times 1000 \qquad \text{for} \qquad T>294 \tag{5}$$

$$\Lambda_{a_0} = 0.195[1 + 0.0184(T - 294)]a_0 \times 1000 \qquad \text{for} \qquad T>294 \tag{6}$$

Now $a_\infty = 0$ for $a_0 < b_0$ (7)

And $a_\infty = (a_0 - b_0)$ for $a_0 \geq b_0$ (8)

Then we can state that $\Lambda_0 = \Lambda_{c0} + \Lambda_{a0}$, while $\Lambda_{c0} = 0$ (9)

And that $\Lambda_\infty = \Lambda_{c\infty} + \Lambda_{a\infty}$ (10)

For the given information calculate the exit concentration of Sodium Hydroxide (a_1) and Sodium Acetate (c_1) from the following equations :

$$a_1 = (a_\infty - a_0)\left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right] + a_0 \tag{11}$$

$$c_1 = c_\infty \left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right] \tag{12}$$

Calculation set 1:

Using the above equations, calculate the conversion achieved based on the value of the reactant concentration (Sodium Hydroxide). Repeat the calculations using the other setting (without stirring).Plot Conversion Vs time for both cases.

Calculation set 2:

You can also calculate the specific rate constant k of the reaction from the data collected. This can be done by using the steady state concentration of NaOH in the reactor (a_1).

Start with the overall mass balance equation:

Rate of Change within the reactor = Input- Output+ Accumulation

For a reactant a in a reactor of volume V, the overall mass balance at steady-state condition maybe written as:

$$\frac{d(V_{a1})}{dt} = F.a_0 - F.a_1 - V.k.a_1^2$$

Where $F = F_a + F_b$

For the continuous reactor operating at steady-state the volume maybe assumed constant and therefore:

$$k = \frac{F}{V}.\frac{a_0 - a_1}{a_1^2} = \frac{(F_a + F_b)}{V}.\frac{(a_0 - a_1)}{a_1^2} \quad mol/dm^3sec$$

Determine the value of k for the two reactor settings.

VII. Discussion:

Comment on the conversion obtained from the two different cases of stirring. You should pay attention to the conversions achieved, the value of k determined from your data.

Based on your results, what can we conclude about the importance of stirring in a CSTR, and what is the importance of agitation?

Nomenclature:

a_μ	sodium hydroxide concentration in feed vessel	(mol/dm^3)
C_{A0}	Initial sodium hydroxide concentration in mixed feed	(mol/dm^3)
C_A	sodium hydroxide concentration at reactor exit	(mol/dm^3)
b_μ	ethyl acetate concentration in feed vessel	(mol/dm^3)
C_{B0}	ethyl acetate concentration in mixed feed	(mol/dm^3)
C_B	ethyl acetate concentration in reactor at time t	(mol/dm^3)
c_μ	sodium acetate concentration in feed vessel	(mol/dm^3)
v	total volume feed rate	(dm^3/s)
v_a	volumetric feed rate of sodium hydroxide	(dm^3/s)
v_b	volumetric feed rate of ethyl acetate	(dm^3/s)
k	specific rate constant	
L	overall length of tubular reactor	(cm)
r	reaction rate	
τ	Residence time	(s)
V	volume of reactor	(dm^3)
X_A	conversion of sodium hydroxide	
Λ	Conductivity	(mSiemens/cm)
Λ_o	initial conductivity	
Λ_∞	Conductivity after 100% conversion	
Λ_a	sodium hydroxide conductivity	
Λ_c	sodium acetate conductivity	
$\breve{A}$	Arrhenius frequency factor	
E	activation energy	(J/mol)
R	gas constant	(J/mol K)

Experiment # 2: DYNAMIC BEHAVIOUR OF CSTR

I. Objectives :

1. To study the dynamic behavior of stirred tank reactor.
2. To calculate the average residence time t_R.

II. Theory:

Effect of a step input change, calculation of the average residence time.

If C = concentration in tank at time t after input step change
C_0 = concentration of the input

Then:

$$C = Co\left(1 - e^{-\frac{t}{tc}}\right) \quad where\ tc = time\ constant$$

$$and\ \frac{dC}{dt} = \frac{Co.\ e^{-\frac{t}{tc}}}{tc} = \frac{Co}{tc} \qquad at\ t = 0$$

Hence t_c might found graphically.

III. Procedures:

D. Solutions Preparation :

3- Prepare 0.1 M solution of Sodium Hydroxide.

E. Calibrating the feed pumps :

5- Fill both reagent vessels with water.
6- Set the feed pump to 2.0 and determine the flow rate by measuring the volume collected for a defined period.
7- Repeat the procedure for 4.0, 6.0, 8.0 and 10.0.
8- Plot a calibration graph for each pump of speed vs. flowrate (ml/min).

Note: Both pumps have to be calibrated because there might be a slight difference between them.

F. Experimental Procedure :

1- Make up 5.0 liters of a solution of 0.1 M sodium hydroxide.
2- Fill one vessel with sodium hydroxide and the other one with distilled water.
3- Start the reactor stirrer and set to a speed of "7".

4- The experiment is carried out at room temperature initially.
5- Start the sodium hydroxide feed pump and set the speed control to maximum in order to fill the reactor to the overflow as quickly as possible. When the reactor is full, slow the feed pump to give a flow rate of 20.0 ml/min.
6- At this point start taking readings of conductivity at regular intervals manually.
7- Start the water feed pump and set to 50.0 ml/min.
8- The conductivity of the reactor contents will begin to reduce and, after a period of approximately 1 hour, will approach the conductivity of the feed solution.

IV. Raw Data:

Time Vs Conductivity							
Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)

V. Calculation:

The final concentration in the reactor will be:-

$$C_\infty = \left(\frac{20}{70}\right) * 0.1 = 0.0285\ M$$

Calculate $\ln((C_\infty - C_1)\,/(C_\infty - C_0))$ where c_1 is the concentration at time *t*, and C_0 is the concentration of NaOH at the start, for readings of *t* throughout the experiment. Plot this value against *t* and calculate the slope (straight line graph passing through the origin). (-1/ slope) is the average residence time t_R which should be equal to (V/F) where *V* is the reactor volume and *F* is the total flow rate into the reactor.

Plot a graph representing the change of Sodium Hydroxide concentration versus time.

Experiment # 3: REACTION RATE DATA USING BATCH REACTOR

I. Objectives:

1. To determine the order, rate constant, and activation energy of the reaction between sodium hydroxide and ethyl acetate.

II. Theory:

In a batch reactor the reactants and the catalyst are placed in a container, are well mixed, which is then closed to transport of matter and the reaction is allowed to proceed for a given time. Then the mixture of un-reacted material together with the products is withdrawn. This is an unsteady state operation where composition changes with time; however at any instant the composition throughout the reactor is uniform.

A batch reactor can be used to find the reaction rate constant, activation energy and order of the reaction. The use of a batch reactor for the most part eliminates the effects due to fluid flow on the resulting reaction rates. Consequently, the data reflect the intrinsic kinetics for the reaction being investigated.

A batch reactor can also be used to demonstrate the mechanism of a chemical reaction and the effects of the operative conditions, such as reaction temperature, concentration, and stirring rate on reaction rate in isothermal and adiabatic conditions. Isothermal condition requires a water chiller or cold tap water in case of exothermic reactions.

The reaction chosen is the hydrolysis of ethyl acetate by sodium hydroxide (saponification); this reaction can be carried out under safe conditions of temperature and pressure, and it is well documented in the literature.

$$\mathrm{CH_3COOC_2H_5 + NaOH \rightarrow CH_3COONa + C_2H_5OH}$$

$$EtAc \; + \; NaOH \;\; \rightarrow NaAc + \; EtOH$$

$$\text{Ethyl acetate} + \text{sodium hydroxide} \Leftrightarrow \text{sodium acetate} + \text{ethanol}$$

As the reaction proceeds, hydroxyl ions are consumed and acetate ions are produced. Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution, whereas ethyl acetate and ethyl alcohol do not. Hydroxyl ion has a very much larger specific conductance than acetate ion. Hence, the alkaline hydrolysis of ethyl acetate may be monitored by following the change in the conductance of the reaction mixture with time. By determination of the conductivity and hence concentration as a function of time, order of reaction and hence the rate constant is found. If the reaction is carried out at different temperatures, the activation energy can also be found.

The following correlations allow calculating the concentration of NaOH, using the measured conductivity, and then the conversion.

At infinite time (Assuming 100% conversion)

$$c_{\mathrm{NaAc}}^{\infty} = c_{\mathrm{EtAc}}^{\mathrm{in}} \qquad \text{if } c_{\mathrm{EtAc}}^{\mathrm{in}} < c_{\mathrm{NaOH}}^{\mathrm{in}}$$

$$c_{\mathrm{NaAc}}^{\infty} = c_{\mathrm{NaOH}}^{\mathrm{in}} \qquad \text{if } c_{\mathrm{EtAc}}^{\mathrm{in}} \geq c_{\mathrm{NaOH}}^{\mathrm{in}}$$

$$c_{NaOH}^{\infty} = c_{NaOH}^{in} - c_{EtAc}^{in} \qquad \text{if } c_{EtAc}^{in} \leq c_{NaOH}^{in}\text{, then}$$

The following correlations allow calculating the conversion at time t:

$$c_{NaOH}^{t} = \left(\frac{\Lambda_t - \Lambda_\infty}{\Lambda_0 - \Lambda_\infty}\right)\left(c_{NaOH}^{0}\right)$$

$$X_A = \frac{c_{NaOH}^{0} - c_{NaOH}^{t}}{c_{NaOH}^{0}}$$

Where Λ_t : Conductivity of reaction mixture at time t

Λ_o: Conductivity of reaction mixture at time t=0

Λ_∞ : Conductivity of reaction mixture at time t=∞

(Assuming 100% conversion)

The conductivity (Siemens/cm) is correlated with concentration of the species and the temperature by the following equations:

Conductivity of sodium acetate at given temperature and concentration can be calculated s

Λ_c=0.070*[1 + 0.0284(T – 294)]$C_{CH3COONa}$*1000 for T ≥ 294for

Λ_{NaOH} =0.195*[1 + 0.0184(T – 294)]C_{NaOH}*1000 for T ≥ 294

$$\Lambda_0 = \Lambda_{NaOH} \quad (\text{Assumes } c_{\mathrm{NaAc}}^{0} = 0)$$

$$\Lambda^{\infty} = \Lambda_{\mathrm{NaOH}}^{\infty} + \Lambda_{\mathrm{NaAc}}^{\infty}$$

III. Description of experimental setup

The reactor is provided of a base to be located on the service unit, and it is vacuum insulated on the walls and the bottom.

Inside the reactor, a stainless steel coil provides the heat transfer surface for either heating or cooling the reactant chemicals. If heating is being carried out, the coil connectors are connected to the supply and return flexible tubing of the hot water circulator, which is incorporated in the service unit. If cooling is required, the flexible tubes can be connected with a chiller or tap water.

A turbine agitator works to provide efficient mixing and heat transfer. The agitator is driven by an electric motor mounted on the head of the reactor, and the motor is driven by a variable speed unit in the service bench and is connected electrically to this by plug. The socket for the plug is on the switchboard of the service unit.

The connections in the reactor head are to house the conductivity and temperature probes, which are supplied with the service unit. The larger of the two is for the conductivity probe. The smaller is unscrewed by hand, the temperature probes inserted and then retightened by hand.

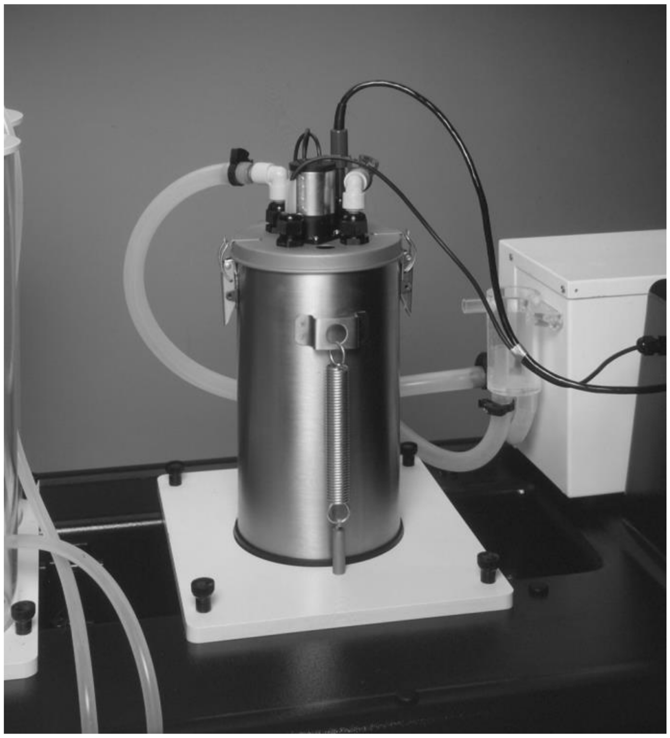


Figure 1: Batch Reactor Unit

IV. Procedures:

A. Solutions Preparation :

1- Prepare 0.05 M solution of Ethyl Acetate.
2- Prepare 0.05 M solution of Sodium Hydroxide.

B. Experimental procedure for Batch operation :

1. Fill the reactor with 0.5 liter of 0.05 M ethyl acetate.
2. Start the hot water circulator and wait till the desired temperature (25^0C) is reached.
3. Pour the 0.05 M sodium hydroxide in the reactor (use the stopper on the lid).
4. Start the stirrer at 50% of the speed and at the same time, start taking the data with a sampling time of 10 sec for 5 minutes.
5. Drain the reactor and repeat the experiment at 30°C and 35°C.

V. Raw Data :

Temperature: 25°C					
Time sec	Λ mS/Cm,	Time Sec	Λ mS/Cm	Time, Sec	Λ mS/Cm□□
10		180		460	
20		200		480	
30		220		500	
40		240		520	
50		260		540	
60		280		560	
70		300		580	
80		320		600	
90		340			
100		360			
110		380			
120		400			
140		420			
160		440			

Temperature: 30°C					
Time sec	Λ mS/Cm,	Time Sec	Λ mS/Cm	Time, Sec	Λ mS/Cm□□
10		180		460	
20		200		480	
30		220		500	
40		240		520	
50		260		540	
60		280		560	
70		300		580	
80		320		600	
90		340			
100		360			
110		380			
120		400			
140		420			
160		440			

Temperature: 35°C					
Time sec	**Λ mS/Cm,**	**Time Sec**	**Λ mS/Cm**	**Time, Sec**	**Λ mS/Cm□□**
10		180		460	
20		200		480	
30		220		500	
40		240		520	
50		260		540	
60		280		560	
70		300		580	
80		320		600	
90		340			
100		360			
110		380			
120		400			
140		420			
160		440			

VI. Calculation:

1. Find concentration of sodium hydroxide using the concentration-conductivity relation. Use excel sheet for calculation and present it in the report.
2. Plot concentration VS time data.
3. Calculate the rate Constant k.
4. Find rate constant of the reaction at 30^0C and 35^0C in the similar manner.
5. Plot lnK Vs I/T and hence find the activation energy and pre exponential factor.

Experiment # 4: REACTION RATE DATA USING TUBULAR REACTOR

I. Objectives

- To determine the rate constant of the reaction between sodium hydroxide and ethyl acetate using a tubular reactor.
- To study the effect of temperature on reaction rate constant and hence to find activation energy and pre exponential factor using Arrhenius law.

II. Introduction

In a tubular reactor, the feed enters at one end of a cylindrical tube and the product stream leaves at the other end. The long tube and the lack of provision for stirring prevent complete mixing of the fluid in the tube.

The tubular reactor also known as plug flow reactor (PFR) is characterized by absence of axial mixing. Hence concentration of reaction mixture moving along the reactor is assumed to vary along the length of reactor only.

Plug flow reactors are relatively easily to maintain (no moving part) and hence find extensive applications in the industry e.g Synthesis of ammonia, oxidation of sulphur dioxide and thermal reforming reactions. A common draw back of this type of reactor is, however, is the difficulty in temperature control and formation of hot spot when the reaction is exothermic.

III. Theory

Consider a differential element of volume dV in the tubular reactor (Figure1).

Mass balance:

Input= output +disappearance by reaction +Accumulation ---- (1)

At steady state, Accumulation = 0

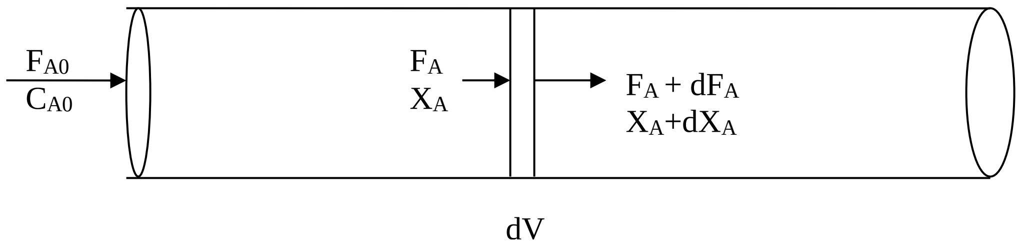


Figure 1- Schematic diagram of Tubular Reactor

Referring to the figure 1

Input of A, (mole/time) = F_A

Output of A, (mole/time) = $F_A + dF_A$

Disappearance of A by reaction, (mole/time) = $(-r_A)dV$

Introducing these three terms in equation (1), we obtain

$$F_A = F_A + dF_A + (-r_A)dV \quad \text{So,} \; -dF_A = (-r_A)dV$$

Noting that, $F_A = F_{A0}(1 - X_A)$ so, $dF_A = -F_{A0}dX_A$

We obtain on replacement

$$-F_{A0}dX_A = -r_A dV \text{ ---------------- (2)}$$

$$\int_0^V \frac{dV}{F_{A0}} = \int_0^{X_A} \frac{dX_A}{-r_A}$$

Thus, $\frac{V}{F_{A0}} = \frac{\tau}{C_{A0}} = \int_0^{X_A} \frac{dX_A}{-r_A}$ ---------------- (3) where τ , is the residence time in the reactor

The equation (3) is called design equation for PFR.

In this experiment kinetics of reaction of sodium hydroxide and ethyl acetate will be investigated to determine the rate constant of reaction.

$$\text{NaOH (A)} + \text{CH}_3\text{COOC}_2\text{H}_5 \text{ (B)} \rightarrow \text{CH}_3\text{COONa (C)} + \text{C}_2\text{H}_5\text{OH (D)}$$

Sodium Hydroxide + Ethyl Acetate → Sodium Acetate + Ethyl Alcohol

The hydrolysis of ethyl acetate can be considered equimolar and first order with respect to sodium hydroxide and ethyl acetate (second order overall), within the limits of concentration (0-0.1 M) and temperature (20-40°C) studied.

Rate expression for the reaction

$$-r_A = kC_A C_B \qquad (1)$$

And for equimolar reactants, $C_A = C_B$, rate expression reduces to

$$-r_A = kC_A^{\,2} \qquad (2)$$

As reaction proceeds concentration changes with conversion,

$$C_A = C_{A0}(1 - X_A) \qquad (3)$$

Reaction rate in terms of conversion becomes

$$-r_A = kC_{A0}^{\,2}(1 - X_A)^2 \qquad (4)$$

The reaction carried out in the CSTR reaches steady state when the concentration of species in the reactor (and then the conductivity) is constant with time.

The concentration of species in the reactor at steady state (and then the conductivity and the final conversion) will vary depending on inlet concentration of reagents, flow rate, volume of reactor, and temperature of reaction.

Concentration as function of conductivity

Sodium hydroxide (reactant) and sodium acetate (product) being ionic compounds contribute conductance to the reaction solution, whereas ethyl acetate and ethyl alcohol do not. The conductivity of a sodium hydroxide solution at a given concentration and temperature, however, is not the same as that of a sodium acetate solution at the same molarity and temperature, and a relationship has been established allowing conversion to be inferred from conductivity.

Conductivity (Siemens/cm) for pure NaOH is given by

$$\Lambda_o = 0.195[1 + 0.0184(T - 294)]\, C_{Ao} \qquad \text{for } T \geq 294$$

And Conductivity (Siemens/cm) of pure CH_3COONa

$$\Lambda_c = 0.070[1 + 0.0284(T - 294)]\, C_c \qquad \text{for } T \geq 294$$

As the reaction proceeds sodium hydroxide concentration will go down and sodium acetate will start forming. Progress of reaction can be monitored by measurement of conductivity contributed by unconsumed sodium hydroxide and formed sodium acetate after reaction. relation between concentration and conductivity can be derived. This phenomenon can be future understood by the plot of conversion Vs conductivity presented in the figure 2.

$$\frac{C_A}{C_{A0}} = \frac{\Lambda - \Lambda_\infty}{\Lambda_0 - \Lambda_\infty}$$

Where Λ : Measured Conductivity (mSiemens/cm)

Λ_o: initial conductivity, i.e conductivity of pure NaOH

Λ_∞ Conductivity of product after 100% conversion,

At 100% conversion

$\mathrm{c}_{\mathrm{NaAc}}^{\infty} = \mathrm{c}_{\mathrm{EtAc}}^{\mathrm{in}}$ if $\mathrm{c}_{\mathrm{EtAc}}^{\mathrm{in}} < \mathrm{c}_{\mathrm{NaOH}}^{\mathrm{in}}$

$\mathrm{c}_{\mathrm{NaAc}}^{\infty} = \mathrm{c}_{\mathrm{NaOH}}^{\mathrm{in}}$ if $\mathrm{c}_{\mathrm{EtAc}}^{\mathrm{in}} \geq \mathrm{c}_{\mathrm{NaOH}}^{\mathrm{in}}$

$\mathrm{c}_{\mathrm{NaOH}}^{\infty} = \mathrm{c}_{\mathrm{NaOH}}^{\mathrm{in}} - \mathrm{c}_{\mathrm{EtAc}}^{\mathrm{in}}$ if $\mathrm{c}_{\mathrm{EtAc}}^{\mathrm{in}} \leq \mathrm{c}_{\mathrm{NaOH}}^{\mathrm{in}}$, then

$$\Lambda_\infty = \Lambda_{NaOH}^{\infty} + \Lambda_{NaAc}^{\infty}$$

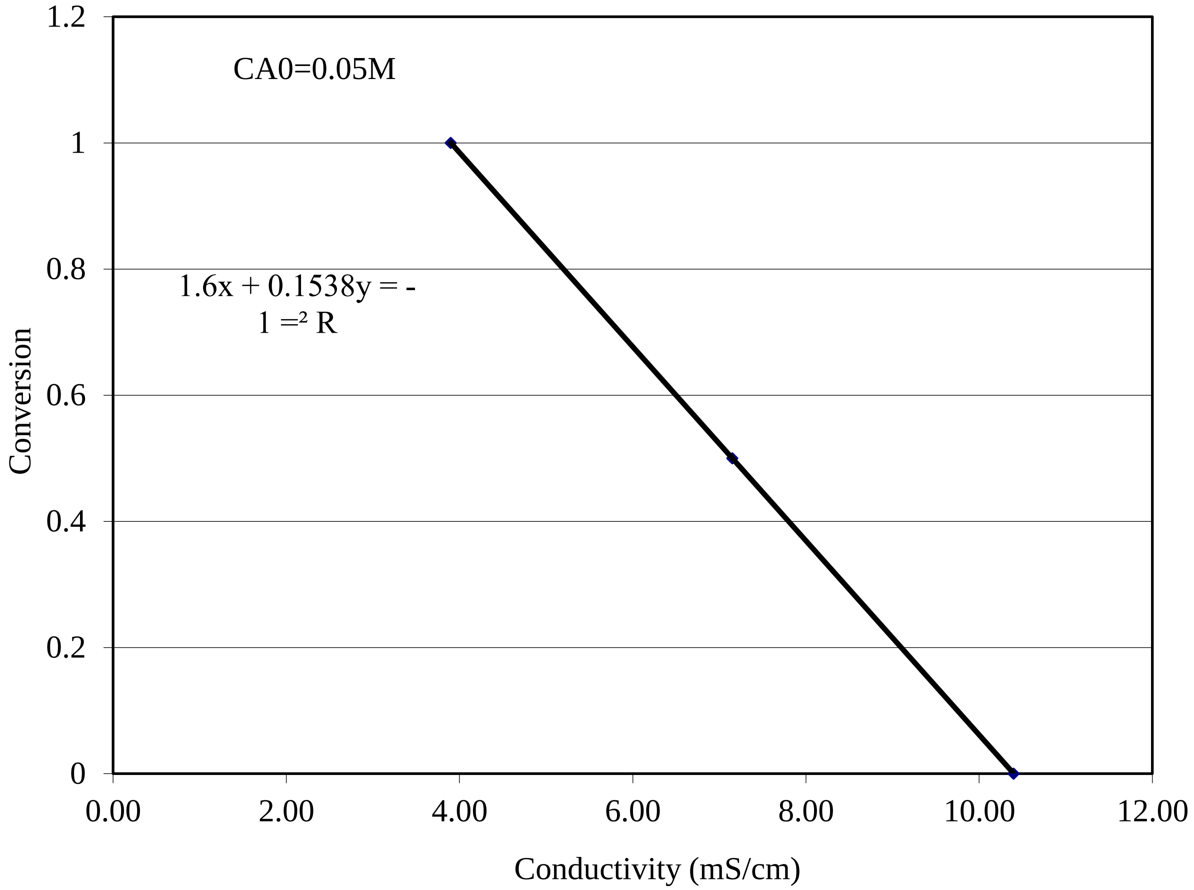


Figure 2. Conversion VS Conductivity

IV. Description of Experimental setup

The reactor vessel (Figure 2) is set on a base plate, which is designed to be located on the four studs of the CEX service unit and then secured by thumbnuts. The reactor is positioned on the service unit with the non-return valve sited on the left and towards the rear.

The tubular reactor in which the chemical reaction takes place is a flexible coil wound around an acrylic former. Total volume of the reactor coil is 0.4 L.

To maintain a constant temperature throughout the reactor coil, the coil bundle is submerged in circulating water, which is automatically maintained at a pre-selected temperature by the temperature controller (TIC) in the console. The actual temperature of the circulating water, and therefore the reactants, is relayed to the controller by a sensing probe (TS), which is held in gland in the lid. Water enters from the circulator at the non-return valve this prevents water draining back through the priming vessel of the circulator when the pump is stopped. Water leaves the reactor at overflow and returns to the circulator.

A filling plug is provided on the top of the reactor vessel to allow the vessel to be filled with clean water. This plug (with sealing washer) must be fitted when the hot water circulator on the CEX service unit is in operation.

Sockets in the side of the console pod of the service unit are provided to connect the conductivity probe and temperature sensor to the instrumentation in the console. These are of a different size so that the probes cannot be wrongly connected. When not in use; the reactor can be drained using valve in the base.

Figure 3 shows the flow diagram. Reactants are pumped from the two feed tanks by the peristaltic pumps and enter the reactor through connectors in the lid of the vessel. Each reactant is preheated by heat transfer coils before being blended together in “T” fitting.

The reactants pass up through the reactor coil and leave the reactor vessel through the conductivity probe housing. This housing allows the conductivity probe (CP) to be held in the stream of reactants emerging from the reactor. Flexible tubing from the hose nozzle is used to guide the reactant to drain.

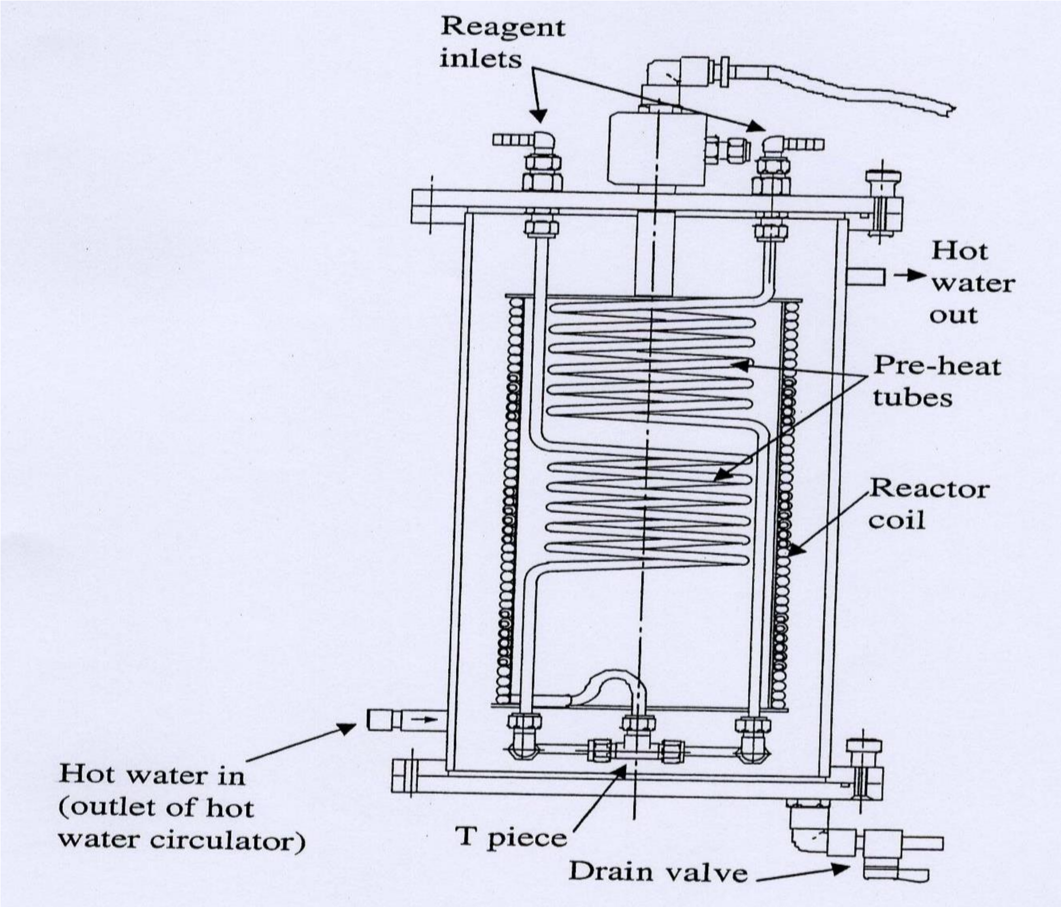


Figure 3: Reactor vessel

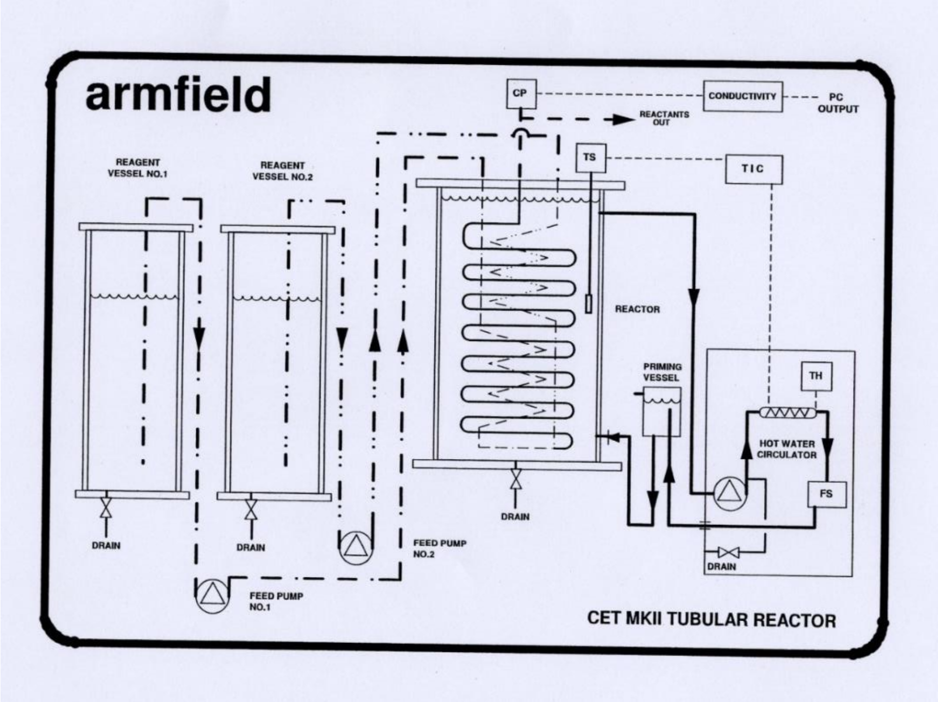


Figure 4: Flow diagram for CET MkII Tubular Reactor

V. Experimental Procedure

- **Preparation of chemicals required for experiments (Reactants)**
- **Preparation of chemicals required for experiments (Reactants)**
- **Dilution of ethyl acetate**

  Armfield recommends the use of a 0.1 M solution of ethyl acetate in the CET MkII reactor. This should be made by diluting concentrated (0.1 M, available in lab) ethyl acetate as follows:

  Volume of concentrate = $Molwt \times 0.1 \times \frac{1}{Density} = \frac{88.11 \times 0.1}{0.90} = 9.8\,\text{ml}$ per liter of solution.

  Total Ethyl acetate solution required for 5 liters = $9.79 \times 5 = 48.95\,\text{ml}$. Add 48.95 ml ethyl acetate in around 1 liter of distilled water in a measuring cylinder. Shake the mixture vigorously until the two liquids have mixed. Add further water to make up the final volume to 2 liter. Add this 2 liter solution to the feed tank. Add 3 more liters of distilled water to the feed tank to make final volume 5 liters.

- **Dilution of sodium hydroxide for use with CET MkII reactor**

Armfield recommends the use of a 0.1 M solution of sodium hydroxide in the CET MkII reactor.

Mass of NaOH required for 0.1 molar NaOH solution=0.1x40=4g,/liter.For experiment 5 liter of 0.1 M NaOH is required. Total amount of NaOH required=20g

This may be prepared by dissolving 20 g of NaOH in around 1 liter of distilled water in a beaker. Add the solution to measuring cylinder and make volume to 2 liter by adding more water. Add this 2 liter solution to the feed tank. Add 3 more liters of distilled water to the feel tank to make final volume 5 liters.

Fill carefully reagent vessels to a level approximately 10 cm from the top.

- Adjust the set point of the temperature controller to 25°C.
- As the experiment involves the collection and storage of conductivity data, the data output port in the console must be connected to the Armfield IFD data logger and the computer. This will enable data logging of the conductivity at selected time intervals over a selected period. If a computer is not available, then the conductivity can be recorded manually at half minute intervals, by reading the value directly from the conductivity meter in the console.
- Ensure the conductivity probe and temperature sensor has been installed properly.
- Using the calibration graph for each of the feed pumps, set the pump speed controls to give 80 ml/min flow rate for each reactant ($F_a = F_b$ = 80 ml/min = 1.33 x 10^{-3} dm^3/sec.)
- Switch on the hot water circulator. The temperature of the water in the reactor vessel will begin to rise and will be automatically maintained at the desired set-point (25°C in this instance).
- Switch on both feed pumps and instigate the data logger program (or begin taking readings if no computer is being used).
- Collection of data will be until a steady-state condition is reached in the reactor.
- Repeat the same steps for 35 ^{0}C temperature .

VI. Raw Data:

Time Vs Conductivity							
25^0C				35^0C			
Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)	Time (min)	Conductivity (Siemens/cm)
1		24		1		24	
2		25		2		25	
3		26		3		26	
4		27		4		27	
5		28		5		28	
6		29		6		29	
7		30		7		30	
8		31		8		31	
9		32		9		32	
10		33		10		33	
11		34		11		34	
12		35		12		35	
13		36		13		36	
14		37		14		37	
15		38		15		38	
16		39		16		39	
17		40		17		40	
18		41		18		41	
19		42		19		42	
20		43		20		43	
21		44		21		44	
22		45		22		45	
23				23			

VII. Report requirement

- Record the following known constants from the experiment using the Nomenclature list below. Ensure use of correct units

v_a =

v_b =

a_μ =

b_μ =

c_μ =

T =

V =

- Calculate steady state conversion by using the relation between concentration and conductivity.
- Find order of reaction from literature and put the rate expression in equation (3) to obtain expression for rate constant. Calculate rate constant by putting the values in derived expression. Compare the value of rate constant with the one you found in literature.
- Find the conversion for other temperature. Plot conversion vs. temperature of reactor. Discuss the trend.
- Using Arrhenius equation, calculate Activation energy

Nomenclature:

A	cross sectional area of tubular reactor	(cm^2)
a_μ	sodium hydroxide concentration in feed vessel	(mol/dm^3)
C_{A0}	Initial sodium hydroxide concentration in mixed feed	(mol/dm^3)
C_A	sodium hydroxide concentration at reactor exit	(mol/dm^3)
b_μ	ethyl acetate concentration in feed vessel	(mol/dm^3)
C_{B0}	ethyl acetate concentration in mixed feed	(mol/dm^3)
C_B	ethyl acetate concentration in reactor at time t	(mol/dm^3)
c_μ	sodium acetate concentration in feed vessel	(mol/dm^3)
v	total volume feed rate	(dm^3/s)
v_a	volumetric feed rate of sodium hydroxide	(dm^3/s)
v_b	volumetric feed rate of ethyl acetate	(dm^3/s)
k	specific rate constant	
L	overall length of tubular reactor	(cm)
r	reaction rate	
τ	Residence time	(s)
V	volume of reactor	(dm^3)
X_A	conversion of sodium hydroxide	
Λ	Conductivity	(mSiemens/cm)
Λ_o	initial conductivity	
Λ_∞	Conductivity after 100% conversion	
Λ_a	sodium hydroxide conductivity	
Λ_c	sodium acetate conductivity	
$\breve{A}$	Arrhenius frequency factor	
E	activation energy	(J/mol)
R	gas constant	(J/mol K)

Part I: Chemical Engineering Thermodynamics

Experiment # 5: PRESSURE MEASUREMENTS AND CALIBRATION

I. Objective:

1. To understand pressure measurement concepts.
2. To identify pressure measurements instruments.
3. To calibrate a pressure gage.

II. Theory:

Introduction

One of the most important concepts in thermodynamics is a thermodynamic state. A thermodynamic state is a set of values of properties of a thermodynamic system that must be specified to reproduce the system. The individual parameters are known as state variables, state parameters or thermodynamic properties. Once a sufficient set of thermodynamic properties have been specified, values of all other properties of the system can be determined. Usually the number of values required to specify the state is two. Pressure and Temperature are two intensive thermodynamic properties that can be measured. All other properties are not measurable. So to analyze any thermodynamic system usually pressure and temperature are measured at different states of the system.

Definitions

Pressure (The symbol: P)

Pressure is the force per unit area applied in a direction perpendicular to the surface of an object.

Mathematically:

$$\boldsymbol{P = \frac{F}{A}} \qquad (1)$$

Where: P is the pressure,

F is the normal force,

A is the area.

Pressure is a scalar quantity. It relates the vector surface element (a vector normal to the surface) with the normal force acting on it.

Pressure is a fundamental parameter in thermodynamics and it is conjugate to volume.

- ***Units***

The SI unit for pressure is the Pascal (Pa), equal to one Newton per square meter (N/m^2 or kg·m^{-1}·s^{-2}).

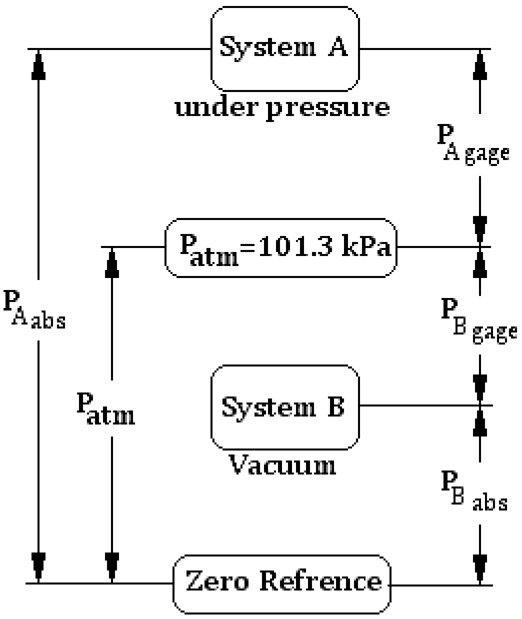
Non-SI measures such as pounds per square inch (psi) and bar are used in some parts of the world, primarily in the United States of America.

- ***The atmospheric pressure*** (The symbol: P_{atm})

Atmospheric pressure is the force per unit area exerted against a surface by the weight of air above that surface in the Earth's atmosphere. In most circumstances atmospheric pressure is closely approximated by the hydrostatic pressure caused by the weight of air above the measurement point.

The standard atmosphere is a unit of pressure and is defined as being equal to 101,325 Pa or 101.325 kPa. The following units are equivalent: 760 mmHg, 29.92 in Hg, 14.696 psi.

- ***Absolute, gauge and differential pressures - zero reference***



  - *Absolute pressure* is zero referenced against a perfect vacuum, so it is equal to gauge pressure plus atmospheric pressure.
  - *Gauge pressure* is zero referenced against ambient air pressure, so it is equal to absolute pressure minus atmospheric pressure. Negative signs are usually omitted.
  - *Differential pressure* is the difference in pressure between two points.

III. Liquid pressure or pressure at depth

Because pressure is commonly measured by its ability to displace a column of liquid in a manometer, pressures are often expressed as a depth of a particular fluid (e.g., centimeters of water or inches of water). The most common choices are mercury (Hg) and water; water is nontoxic and readily available, while mercury's high density allows for a shorter column to measure a given pressure. The pressure exerted by a column of liquid of height h and density ρ is given by:

$$\boldsymbol{P = \rho g h} \qquad (2)$$

Where: P is Pressure
g is gravity acceleration
ρ is density of liquid
h is height of liquid or depth within a substance

IV. Pressure measurement devices

Many techniques have been developed for the measurement of pressure and vacuum. Instruments used to measure pressure are called pressure gauges or vacuum gauges. A manometer could also be referring to a pressure measuring instrument, usually limited to measuring pressures near to atmospheric. The term manometer is often used to refer specifically to liquid column hydrostatic instruments.

In this experiment you will be familiar with the following types of instruments:

4.1 manometers

Pressure measuring devices using liquid columns in vertical or inclined tubes are called manometers.

The difference in fluid height in a liquid column manometer is proportional to the pressure difference.

Tube ends are exposed to different pressures. The column will rise or fall until its weight is in equilibrium with the pressure differential between the two ends of the tube.

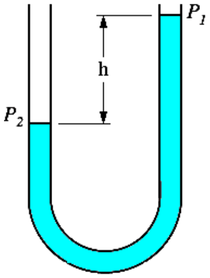
Vertical U tube manometer

$$\boldsymbol{P_2 - P_1 = \rho g h} \quad \text{…………..(3)}$$

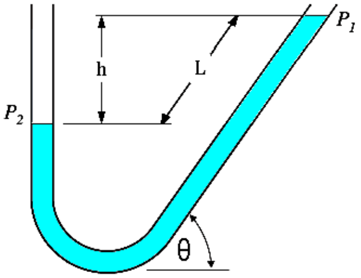
Inclined U tube manometer

$$\boldsymbol{P_2 - P_1 = \rho g h}$$

$$\boldsymbol{P_2 - P_1 = \rho g L \sin\theta} \quad \text{….(4)}$$



Vertical 'U' Tube
Manometer



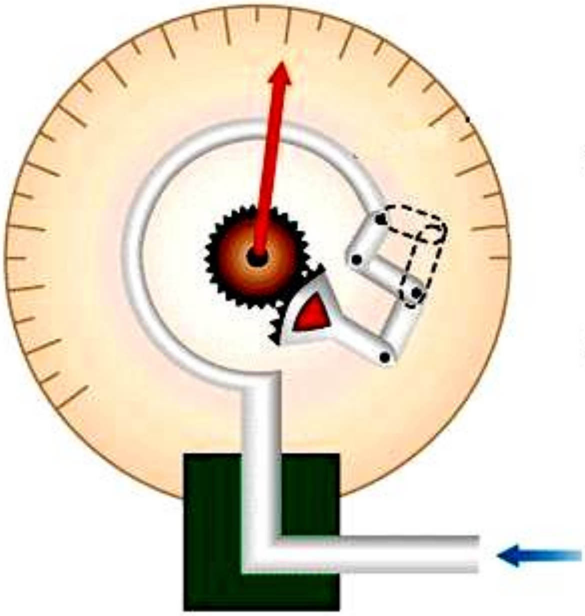
Inclined 'U' Tube
Manometer

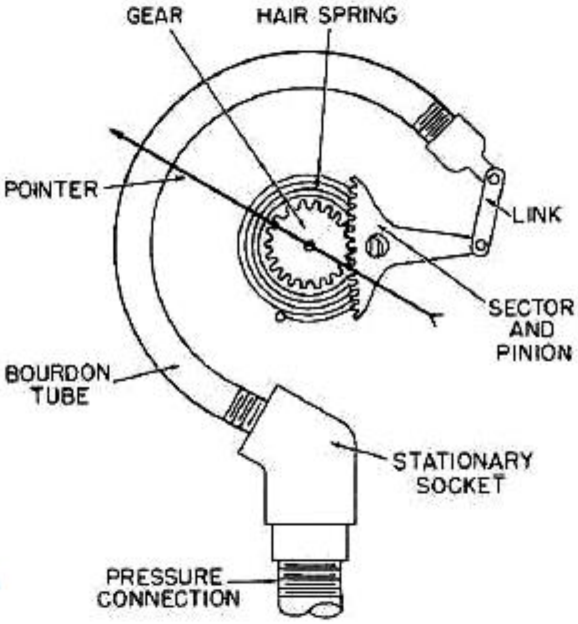
The inclined manometer is more accurate and uses for very small pressure readings

4.2 Bourdon gauge

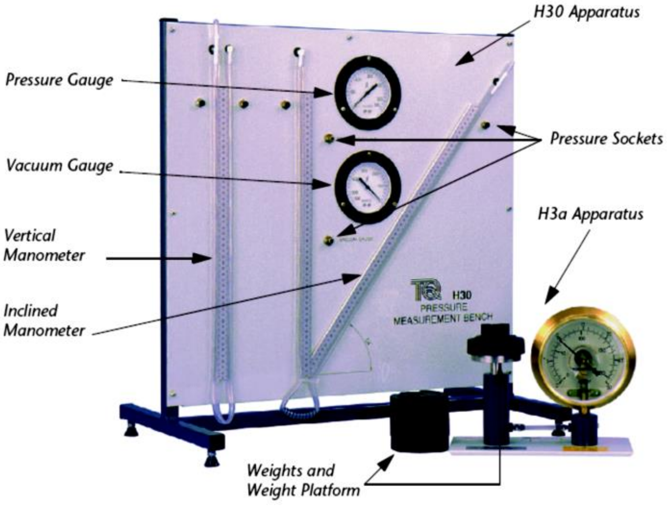
Instrument for measuring pressure, patented by French watchmaker Eugene Bourdon in 1849. The gauge contains a C-shaped tube, closed at one end. When the pressure inside the tube increases, the tube uncurls slightly, causing a small movement at its closed end. A system of levers and gears magnifies this movement and turns a pointer, which indicates the pressure on a circular scale. Bourdon gauges are often fitted to cylinders of compressed gas used in industry and hospitals.

Bourdon gauges are used to measure large pressures that can’t be measured by manometers.





Apparatus:



The setup of the apparatus consists of:

1- *Pressure Measurement Bench*

It is used to compare ways of measuring pressure. Manometry is a fundamental method of measuring low pressure and, together with Bourdon type gauges, is widely used in engineering.

The apparatus includes vertical and inclined manometers, and two Bourdon type gauges. One of the gauges measures pressure, the other measures vacuum. The gauges and manometers each have pressure sockets for direct connection. 'Tee' connectors and tubing are supplied to allow many different connections. A syringe is supplied as a means of creating an adjustable pressure and vacuum

2- *Calibration of a Pressure Gauge Apparatus*

This uses a precision Bourdon type pressure gauge with a clear dial so that its internal workings can be studied. Inside the gauge, the Bourdon tube is rigidly held at one end. When pressure is applied to the gauge, the tube straightens and moves. This movement is transferred through a mechanical system that gives proportional movement of the dial around the graduated scale. The range of the gauge depends on the type and thickness of the material used for the Bourdon tube.

The apparatus also includes a set of weights and a weight platform to apply a known pressure to the Bourdon scale to show its operation and for the calibration experiment

Procedure:

I- *Pressure and Vacuum measurements*

Note; Never apply pressure to the Vacuum Gauge. It will be damaged.

Never apply Vacuum to the pressure Gauge. It will be damaged.

1- Use the tee connect pressure gauge, vertical manometer, and inclined manometer at same time.

2- Connect the nozzle to the syringe.

3- Apply pressure in steps of 50 mm H2O, at each step record the gauge pressure and the two manometers readings.
4- Repeat same procedure for vacuum test.
5- Fill results in table 1

II- Calibration of a pressure gauge.

1- The apparatus should be level.
2- Remove the piston from the cylinder, and then fill the cylinder with water.
3- Insert the piston into the cylinder; allow any air and excess water to discharge through the top hole of the cylinder, allow the piston to settle.
4- Record the cross-sectional area and the mass of the plunger (piston) and the weight platform.
5- Add weights (masses) to the platform in increments as shown in table 2.
6- Record the pressure gauge reading at each mass added.
7- Remove the masses in reverse order as you added them. Record the pressure gauge reading as you remove each mass.
8- Fill the results in table 2

Analysis:

I- Pressure and Vacuum measurements

1. Calculate pressure: for vertical manometer $\boldsymbol{P_{gage} = P_1 - P_2}$ and for inclined manometer $\boldsymbol{P_{gauge} = P_1 - P_2 \sin 54}$

Where:

Pgage = Gauge pressure (in mmH2O);

P_1 = Length of water column at left leg of manometer (in mmH2O)

P_2 = Length of water column at left leg of manometer (in mmH2O)

2. Plot gauge reading against Δh for vertical and inclined manometers.

3. If you want to calibrate the gage to read in Pa units what did you do?

II- Calibration of a gauge pressure

1. Calculate the actual pressure in the system

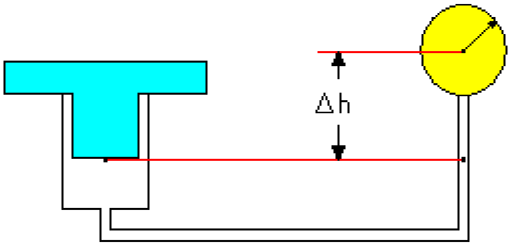
$$\boldsymbol{P = \frac{F}{A}, \; F = Mg \; \rightarrow \; P = \frac{Mg}{A}}$$

Where M is the total mass (kg)

g is the acceleration due to gravity = 9.81 m/s^2

A is the piston area $= \frac{\pi D^2}{4}$

2. Calculate the pressure gauge error for both increasing and decreasing pressures.
3. Plot the calibration curve [actual pressure versus gauge reading].
4. Comment on gauge errors.
5. Why we use the calibration curve.
6. Using same weights what piston dimensions you suggest to calibrate a gauge pressure of full scale reading of 3500 kN/m^2
7. Discuss the error due to the difference in elevation between the piston of dead weight tester and the pressure gauge. If the center of the gauge is Δh higher than the base of the piston what correction should be made.



V. Results:

Pressure							Vacuum						
Pressure gauge	Vertical Manometer			Inclined Manometer			Pressure gauge	Vertical Manometer			Inclined Manometer		
mmH_2O	P_1 mmH_2O	P_2 mmH_2O	Adjusted mmH2O	P_1 mmH_2O	P_2 mmH_2O	Adjusted mmH2O	mmH_2O	P_1 mmH_2O	P_2 mmH_2O	Adjusted mmH2O	P_1 mmH_2O	P_2 mmH_2O	Adjusted mmH_2O
0							**0**						
50							**-50**						
100							**-100**						
150							**-150**						
200							**-200**						
250							**-250**						
300							**-300**						

Table 1: *Pressure and vacuum measurements*

			Increasing Pressure		Decreasing Pressure	
Mass added to piston	Total mass on piston	Actual pressure P	Gauge reading	Gauge error	Gauge reading	Gauge error
Kg	Kg	kN/m^2	kN/m^2	kN/m^2	kN/m^2	kN/m^2
0						
0.5						
1.0						
1.5						
2.0						
2.5						
3.0						
3.5						
4.0						

Table 2: Calibration of a gauge pressure

Experiment # 6: TEMPERATURE MEASUREMENTS AND CALIBRATION

I. Objective:

1. To identify and to be familiar of different types of temperature measuring devices (Thermometers) and to know their principle of work.
2. To show difference between constant current and constant voltage thermometer circuits.
3. To calibrate a thermometer.
4. To study the linearity and principle of work of J & K types thermocouples.

II. Theory:

1. Introduction

Temperature is a physical property of matter that quantitatively expresses the common notions of hot and cold. It is one of the principal properties studied in the field of thermodynamics. Temperature difference between two objects is the driving force for heat transfer. If no heat is transferred between two systems, the systems have the same temperature (Systems are in thermal equilibrium). In the classical thermodynamic approach to temperature, temperature of an object varies with the speed of the particles it contains, raised to the second power. (Kinetic Energy). In the statistical thermodynamic approach, degrees of freedom are used instead of particles.

Much of the world uses the Celsius scale (°C) for most temperature measurements. It has the same incremental scaling as the Kelvin scale (K) used by scientists, where K = C + 273.15 A few countries, like United States, use the Fahrenheit scale (F) for common purposes, a historical scale on which water freezes at 32 °F and boils at 212 °F. Where $F = \frac{9}{5}C + 32$

2. Calibration.

2.1 The ITS-90 Scale

The (ITS-90) is the 1990 revision of the International Temperature Scale. The scale is a calibration standard for Kelvin and Celsius measurements. It uses the freezing point of silver and gold, and the triple point of water, oxygen and hydrogen as reference points. Before ITS-90 standards uses freezing point of pure water to define 0°C not its triple point. This makes a difference of 0.01°C. ITS-90 recommends Platinum Resistance Thermometers (PRTs) as standard in the range between the triple point of hydrogen (-259.2°C) and the freezing point of silver (961.8°C).

2.2 Calibration, Standards and Linearity

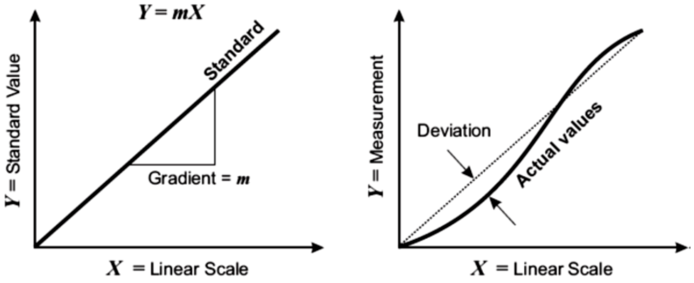


Figure 1: Calibration, Standards and Linearity

To calibrate a device, you must compare it with a known standard. When the standard is linear and you compare results against a linear scale the comparison is easy. You can plot results against the known standard to show errors (or 'deviation') (figure 1). You can then use the calibrated device with confidence, knowing that it will work as you expect. With a non linear standard and device response, you can still calibrate the device, but it is not so easy. Linear devices give a predictable output that you can work with easily. Non linear devices usually need additional parts or circuits that make their output more linear

3. Temperature and Pressure.

Different pressures affect the equilibrium points of some chemical elements. For example, water will boil at less than 100°C at pressures lower than normal atmospheric. This is according to the following relation:

$$\boldsymbol{Boiling\ Temperature\ of\ water = \left(\frac{Ambient\ Pressure - 1013.3}{36.185}\right) + 100\ {}^{o}C}$$

Where 1013.3 is the known standard atmospheric pressure (in millibars) at sea level.

4. Temperature Measuring Devices and their principle of work

4.1 Liquid filled glass thermometer

They work on the principle of a known volume expansion rate of a liquid for a known rise in temperature.

As the liquid expands with a rise in temperature, it moves up or down a thin (capillary) tube behind a calibrated scale.

4.2 Gas thermometer

The gas thermometer works on the principle of a known volume expansion rate of a gas (vapour of a fluid) for a known rise in temperature. As the gas (vapour above a small volume of fluid) expands with a rise in temperature, its pressure increases and pushes against a mechanical mechanism that turns a calibrated dial.

4.3 Bimetal thermometer

The bi-metal thermometer works on the principle of different expansion rates of two different metal strips fixed (laminated) together to form a thicker strip that is a composite of the two metals. As temperature increases, one metal expands more than the other and the composite bends, pushing a calibrated mechanical dial.

4.4 Resistance Thermometers [RT]

(Sometimes called resistance temperature detectors or RTDs)

The resistance thermometers use a known change of electrical resistance in a metal for a known temperature change.

Examples on RT:

- PRT (Platinum Resistance Thermometers): The metal usually thin platinum wire or film. They have a positive temperature coefficient (PTC) because their resistance increases with temperature.
- Thermistor: They have negative temperature coefficient (NTC) because their resistance decreases with temperature.

Figure 2 show how the wires connect to the four wires PRT and what is inside the probe. The four wires are actually two pairs of wires connected together. Two wires usually connect to a measuring device and the other two wires supply the current to the thermometer.

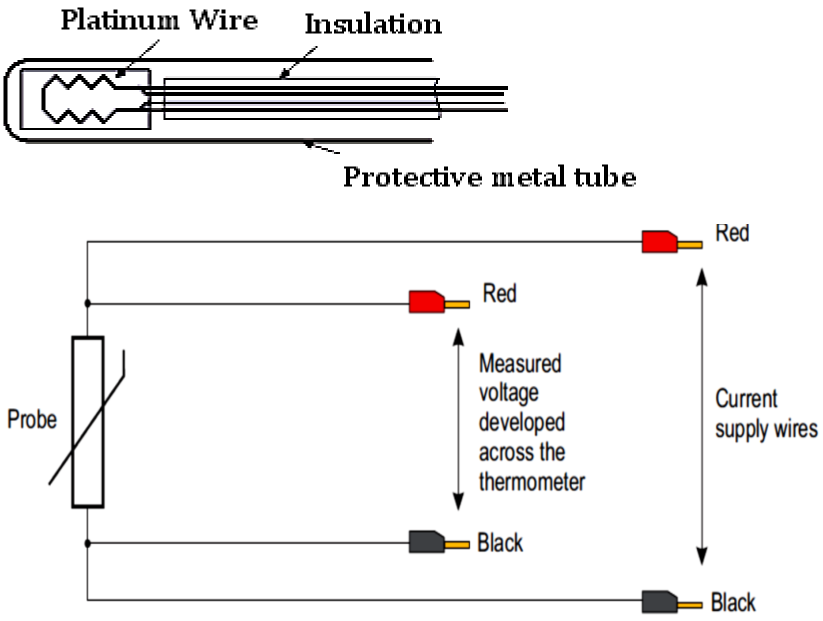


Figure 2: Four wires of PRT

Principle of work: Ohm’s Law

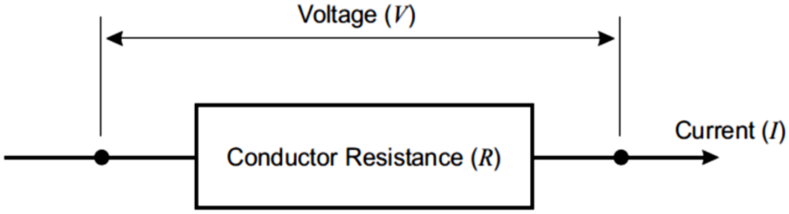


Figure 3: Ohm’s Law

In 1827, a Physicist - George Ohm published his discovery that all electrical circuits obey a simple relationship: $V = IR$ (see figure 3) From this, if you can fix the current in your resistance (make it constant), and your resistance changes, the voltage across the resistance is directly proportional to the resistance. Also, if you can fix the voltage across your resistance (make it constant), and your resistance changes, the current through the resistance is inversely proportional to the resistance.

Figure 4 shows two different ways of supplying a circuit with a resistance thermometer and shows the resistance of the wires that connect the thermometer. In both circuits, a voltage (V1 and V2) ‘drops’ across each of the wires.

In the circuit supplied by a constant voltage, if you extend the wires, their resistance increases, the circuit current becomes lower and more voltage ‘drops’ along the wires. This reduces the current through the thermometer and the measured voltage for a given change of resistance. You would need to increase the voltage source to compensate for the voltage lost in the wires. Also, as the thermometer resistance changes, so does the circuit current and the voltage ‘lost’ along the wires. This gives a slightly non-linear relationship between resistance and voltage change.

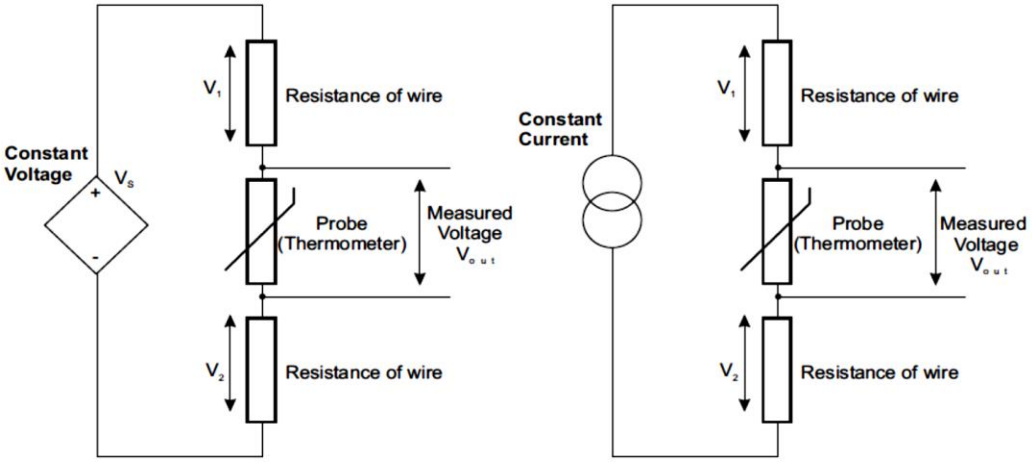


Figure 4: Constant current and constant voltage circuits of a resistance thermometer

In the circuit supplied by the constant current, if you extend the wires, the circuit current does not drop, because the current source automatically compensates. The current through the thermometer remains constant and the measured voltage remains the same. As the thermometer resistance changes, the supply automatically adjust to keep a constant current, so the measured voltage is always directly proportional to the resistance. This gives a very linear relationship between resistance and voltage change

The Wheatstone bridge

The basis of many electrical measuring devices is the Wheatstone Bridge, shown in figure 5. It has four identical resistances (R1, R2, R3 and R4) connected end to end in a diamond shape. A constant input voltage (Vi) connects across two opposite connections. The output voltage is at the other two connections.

The output voltage (Vo) depends on the ratio of the resistors, so that:

$$V_o = V_i\frac{R2}{R1+R2} - V_i\frac{R4}{R3+R4} \text{ or } V_o = V_i\left(\frac{R2}{R1+R2} - \frac{R4}{R3+R4}\right)$$

As the equation shows, when R1, R2, R3 and R4 are all exactly equal, then the output voltage Vo is zero, no matter what happens to the input voltage Vi. However, if one resistance changes (for example - R1), then Vo will change in proportion to the resistance change.

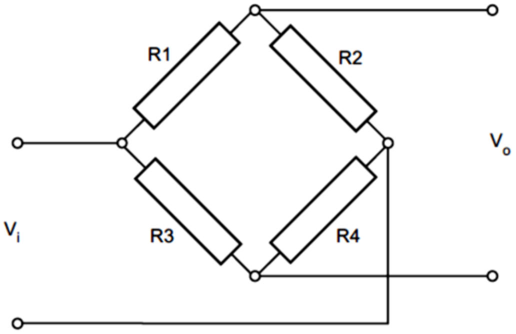


Figure 5: The Wheatstone bridge

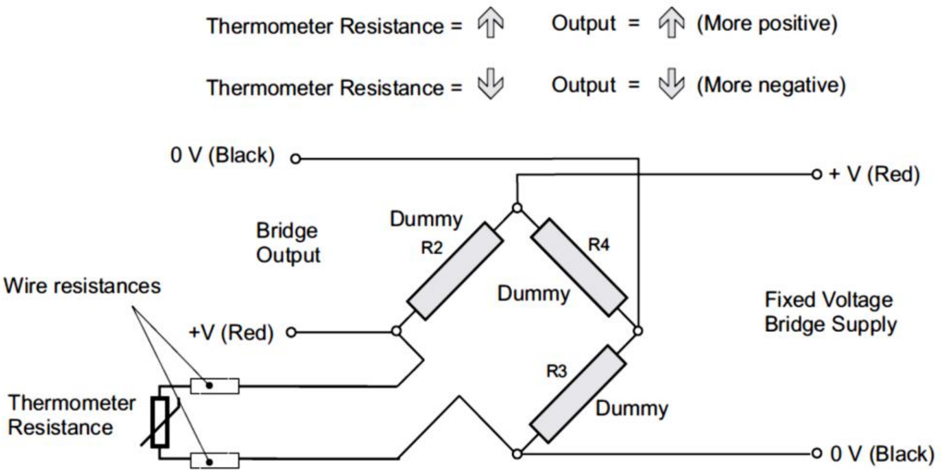


Figure 6:Themometr in Wheatstone bridge circuit

When a single resistance thermometer replaces one of the resistors in a Wheatstone Bridge (Figure 6), the change of the bridge output voltage Vo is proportional to the change in resistance of the thermometer. For a ***positive*** coefficient thermometer resistance, the output from the bridge is not perfectly linear, but for small resistance changes, the output is assumed to be linear. If the wires to the thermometer become long, their resistance affects the linearity of the voltage output. The dummy resistors (R2, R3 and R4) must also change to match the new higher total resistance of the wires and the thermometer.

4.5 Thermocouples

In 1821, a German-Estonian physicist -Thomas Seebeck, discovered that certain conductors (mainly metals) will generate a small voltage when you heat them. This is known as the Seebeck or thermoelectric effect. It works best when you use two different metals and connect them together by a circuit with two junctions. When one junction is at a higher temperature than the other, a current flows around the circuit.

To measure temperature correctly, the thermocouple must measure with respect to a known temperature, so one junction is at a known (reference) temperature (see Figure 7). An artificial cold junction reference using a thermally sensitive device such as a thermistor or diode to measure the temperature of the input connections at the instrument, with special care being taken to minimize any temperature gradient between terminals. Hence, the voltage from a known cold junction can be simulated, and the appropriate correction applied. This is known as cold junction compensation.

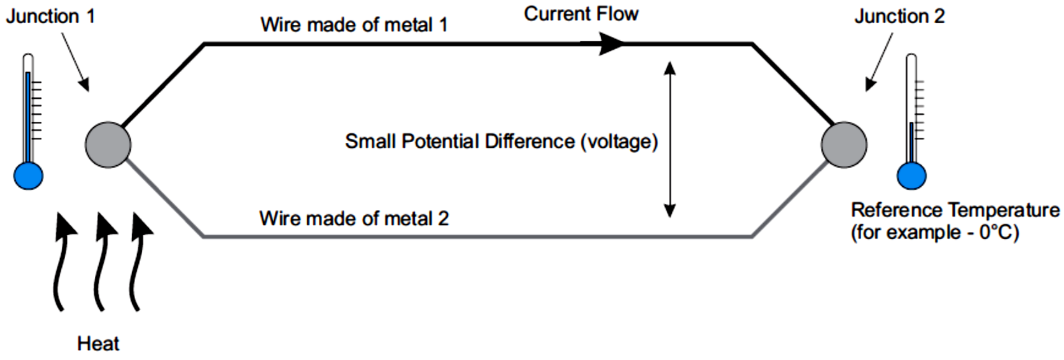


Figure 7: Thermocouple concept

Different metal combinations work better than others for different applications. For example, one pair of metals may work well at lower temperatures, but could melt at higher temperatures, where a different set of metals would work better. Also, some combinations may give more linear results than others.

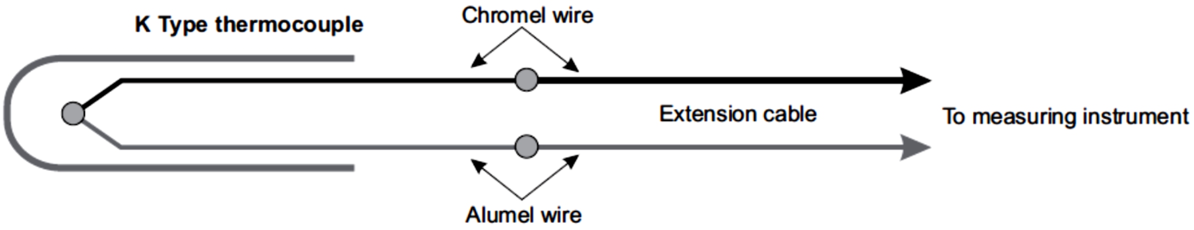


Figure 8: Thermocouple Extension Wires and Circuit Resistance

Because thermocouples use two different materials to produce a small voltage, any cables you use to extend them must be made of the same material as those of the thermocouple wires. If you use different materials, you create additional thermocouple junctions, which can affect the readings.

As with all measuring systems, the electronic circuits that measure the signals from thermocouples must not interfere with the signal levels from the thermocouple. For this reason, the measuring circuits must have a high input resistance, or they will reduce the signal and make it unusable. However, in some applications, the measuring instrument may need a slightly lower input resistance than normal to help reduce any noise picked up on the thermocouple wires.

Because thermocouple circuits use small currents, the resistance of its wires does not have a large affect on the signal, so theoretically they may be very long. However, you should always try to use short cables wherever possible, to reduce any small measurement errors and avoid receiving stray noise signals from other equipment.

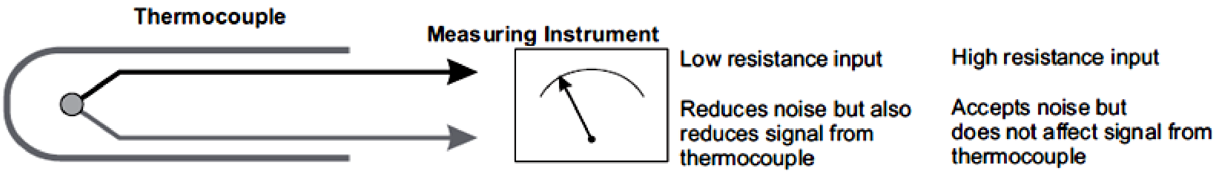


Figure 9: Thermocouple Circuit

III. Apparatus:

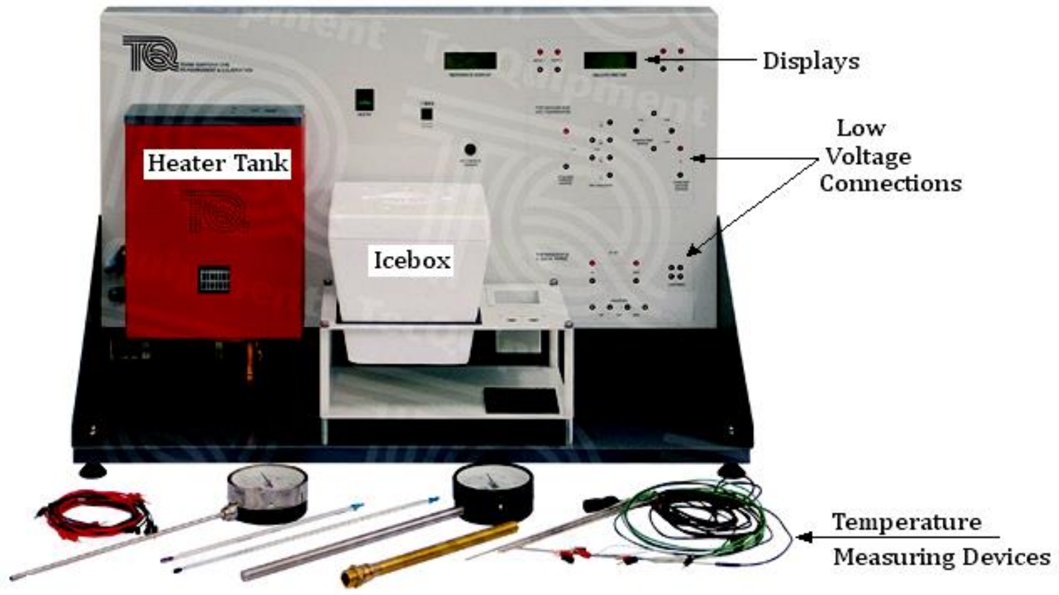


Figure 10: Temperature measurements and calibration apparatus

I. Main Part

- Water heater tank (Red)
- An insulated icebox (White)
- Two Digital Displays 1- Reference display: this display shows the readings of
    - The reference temperature from the reference sensor (PRT).
    - Atmospheric pressure.
    - Boiling temperature of water (at the indicated atmospheric pressure).

    2- Millivoltmeter display: shows readings of four different voltages from each of the four pairs of sockets each side of the display.
- Low Voltage Connections

    PRT and NTC Thermistor Sockets, this includes:
    - A constant current source
    - A constant voltage source
    - PRT simulator.
    - Wheatstone Bridge Circuit

    Thermocouple J and K Type Sockets, this includes:
    - A voltage amplifier
    - Four spare junction sockets
    - Three fixed resistors

II. Temperature Measuring Devices

- Liquid Filled Glass Thermometers
- Gas (Vapour Pressure) and Bi-Metal Thermometers
- Thermocouples (J and K types)
- Resistance Thermometers (RTDs or PRTs)

  PRT: Platinum Resistance Thermometer (Reference temperature)
  RTD: Resistance Thermometer Detector
  PTC: Positive Temperature Coefficient (temperature increase as resistance increase)
- Thermistor (NTC)

  NTC: Negative Temperature Coefficient (temperature decrease as resistance increase)

Procedure:

Part 1 - PRT Simulation, Constant Voltage and Current

1. Connect the simulated PRT directly to the constant voltage and input 1 of the Millivoltmeter (see the solid wires of Figure 11).

2. Switch on the electrical supply and wait a few seconds for readings to stabilize, and then record the input 1 reading of the millivoltmeter.

3. Move the wires to include the resistors (as shown by the dotted wires). This imitates two long lengths of connection wire (as if the wires have been extended).

4. Wait for the readings to stabilize, and then record the Input 1 reading of the millivoltmeter.

5. Repeat same procedure with the connection of constant current source. (Figure 12)

Part 2 - PRT Calibration

1. Disconnect the electrical supply.

2. Shut the drain valve at the back of the heater tank.

3. Unscrew the lid of the heater tank and fill it with approximately 1.5 Litres of clean water, so that it is half full.

4. Refit the lid of the heater tank.

5. Add ice to the icebox and put its lid on.

6. Make sure the heater switch is off and reconnect the electrical supply.

7. Connect the reference sensor to its socket and connect the PRT to the millivoltmeter and the constant current source as shown in figure below. Note that the PRT is connected as a four wire device.

8. Put the reference sensor and the PRT into the icebox (through the holes in its lid). Wait a few minutes for the readings to stabilize and record them (the reference temperature should be between 0°C and 1°C).

9. Now put both devices into the heater tank (through the holes in its lid). Switch on the heater and note the reference temperature.

10. At intervals of 10°C (shown by the reference temperature), record the input 1 readings of the Millivoltmeter.

11. Stop the experiment and switch off the heater when the reference temperature reaches 100°C.

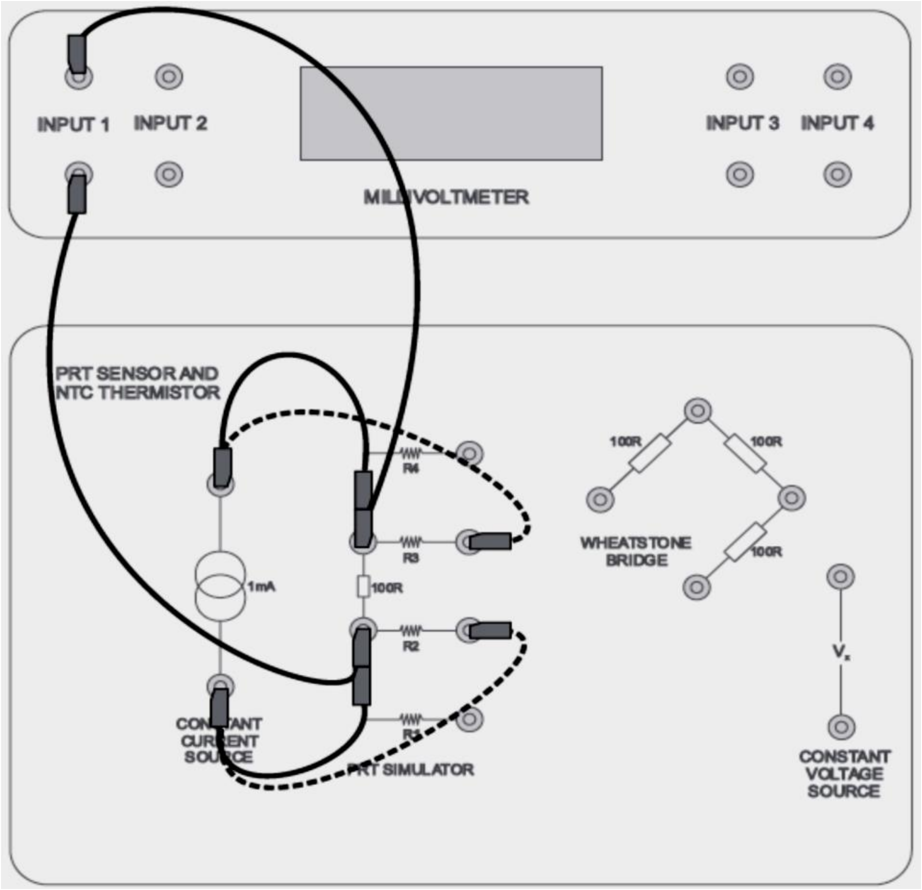


Figure11: Part 1 a- Constant Current

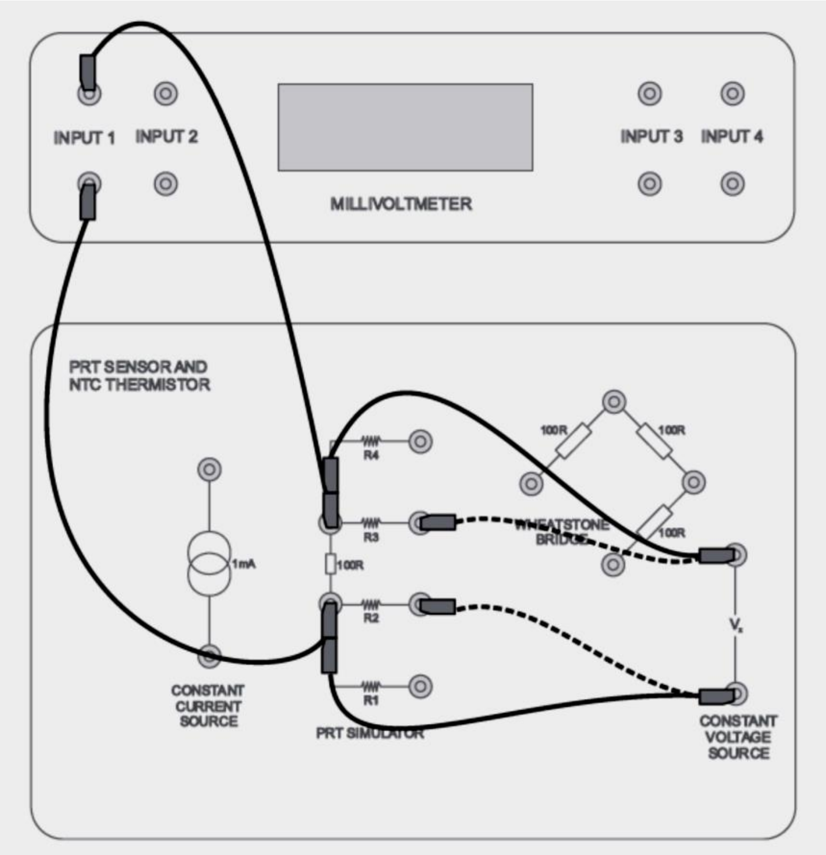


Figure12: Part 1 b- Constant Voltage

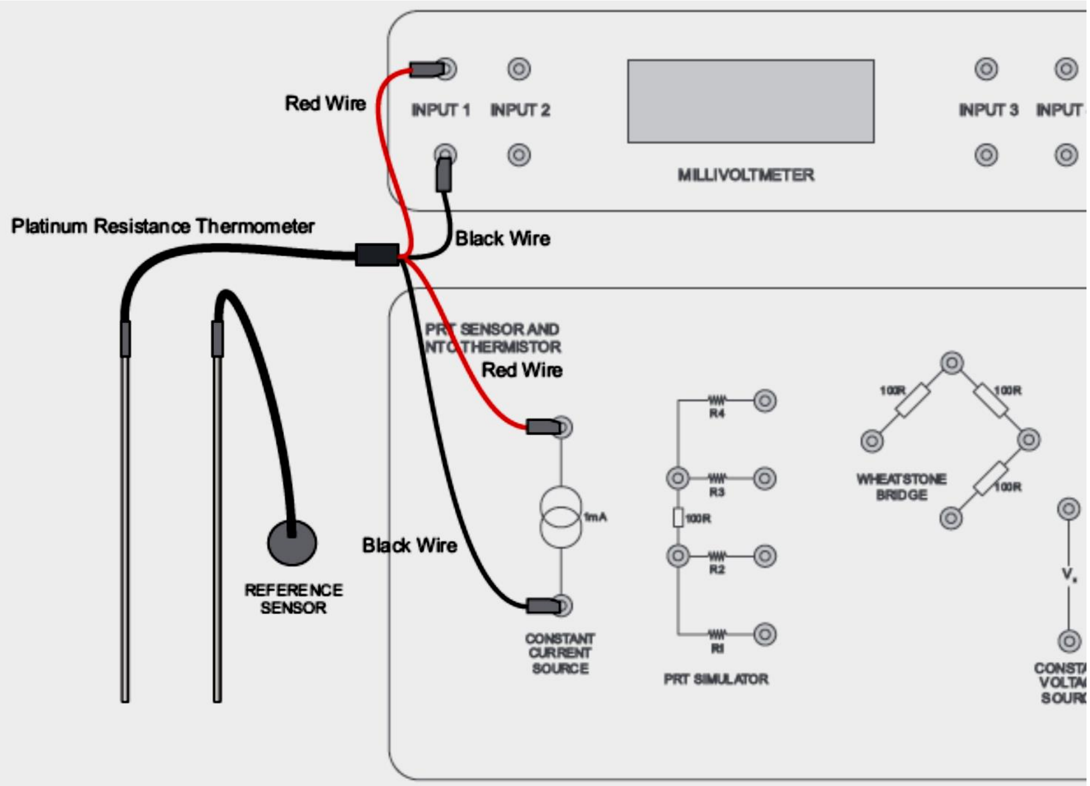


Figure13: Part 2 - PRT Calibration

<u>Part 3 – K Thermocouple Linearity:</u>

1. Connect the reference sensor to its socket and connect the K thermocouple to amplifier and the Thermistor Millivoltmeter as shown in the figure 14. The amplifier amplifies the small voltage from the thermocouple by 20. This makes it suitable for the Millivoltmeter. The actual voltage from the thermocouple will therefore be 1/20 of the reading from the Millivoltmeter.
2. Prepare heater and icebox as in part 2.
3. Put the reference sensor and the Thermocouple into the icebox (through the holes in its lid). Wait a few minutes for the readings to stabilize and record them (the reference temperature should be approximately 0°C.
4. Now put both devices into the heater tank (through the holes in its lid). Switch on the heater and note the reference temperature.
5. At intervals of 10°C (shown by the reference temperature), record the input 1 readings of the Millivoltmeter.
6. Stop the experiment and switch off the heater when the reference temperature reaches 100°C.
7. Repeat the procedure for the other thermocouple.

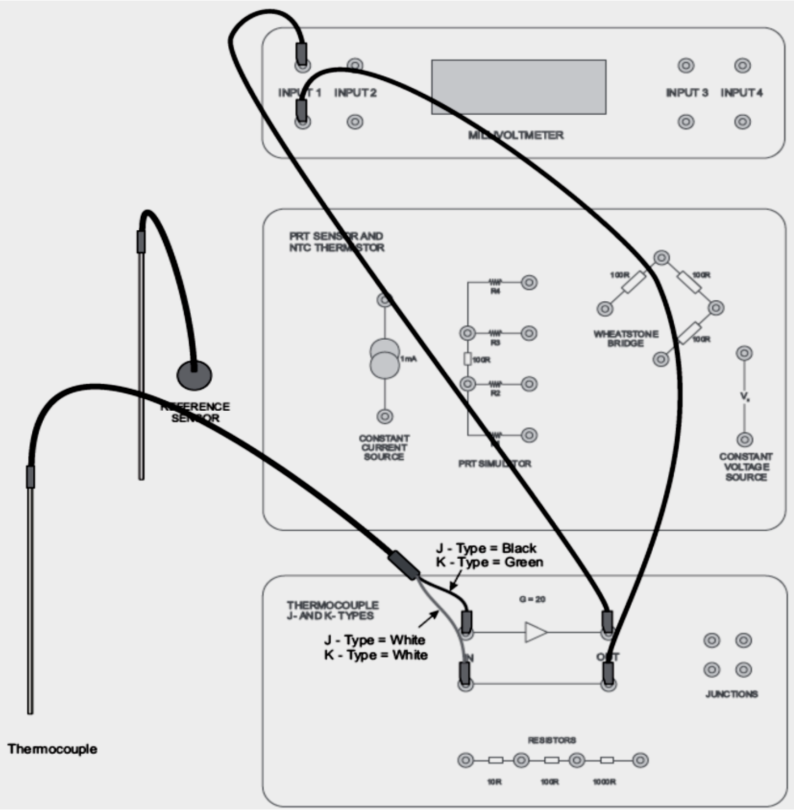


Figure14: Part 3 – K thermocouple linearity circuit

Analysis:

Part 1 - PRT Simulation, Constant Voltage and Current

Compare your results. They should show that when you use the constant voltage, the measured voltage drops when you add the extra wires, but not if you use the constant current supply.

Part 2 - PRT Calibration

1. Given that the constant current is 1 mA, use Ohm’s law to calculate the resistance of the PRT for each row in your table. You should see that is directly proportional to the measured voltage.
2. Plot a chart of resistance (vertical axis) against temperature (horizontal axis). Start the vertical axis at 100 Ohm.
3. Draw a best curve through your results to see how the PRT gives a good linear resistance change over the range 0 to 100°C. Note that resistance increases with temperature (positive temperature coefficient).

4. Add to your results table and chart, the standard resistances given in Table 1 that match the reference temperatures. On your chart, draw a straight line through the standard values. Note any differences between the standard and your measured resistances.
5. On your results table, find the difference between the standard and your results (the deviation) and calculate the percentage error.

Percentage error = (deviation/standard) x 100

6. From your results, can you understand why we can use the PRT as an accurate reference sensor?
7. Can you identify any possible causes of errors?

Part 3 – J and K Thermocouple Linearity:

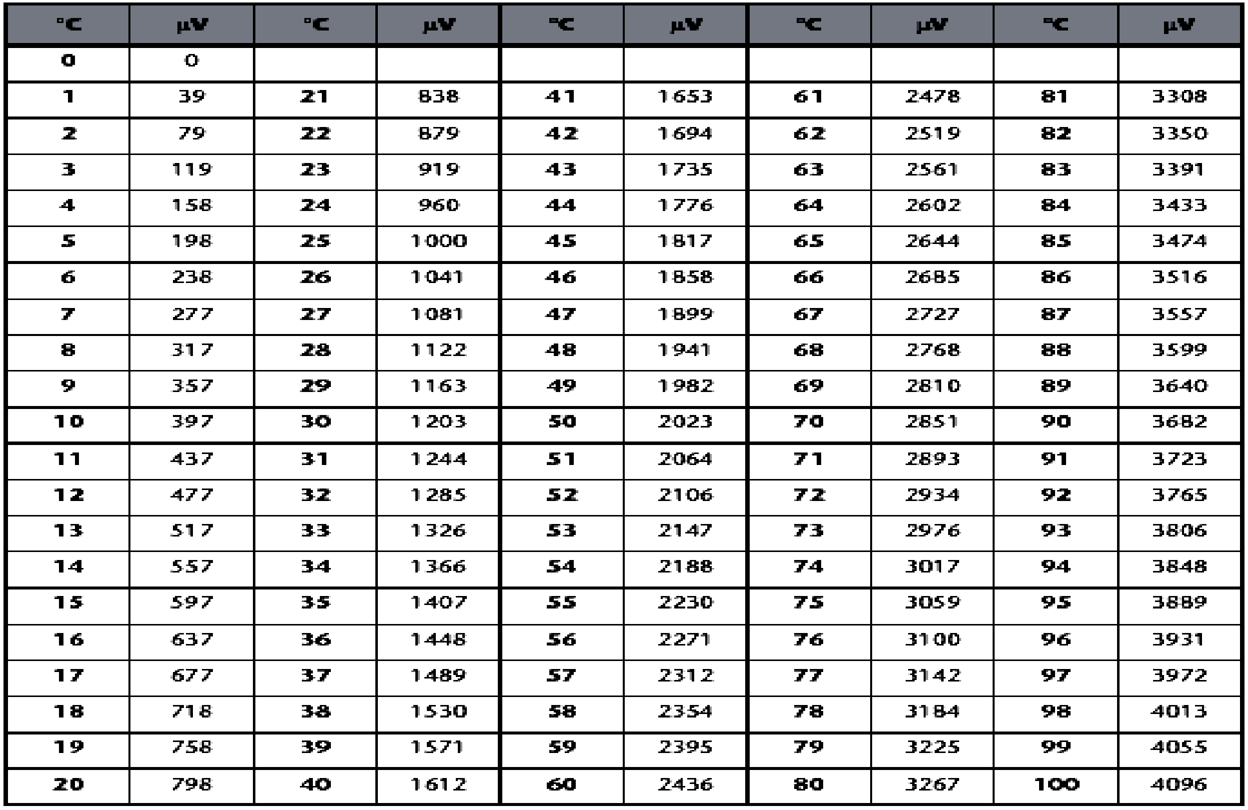
1. Divide your Millivoltmeter readings by 20 to find the actual measured voltage generated by the thermocouples for each row in your table.
2. For each thermocouple, plot a chart of actual measured voltage (vertical axis) against temperature (horizontal axis).
3. Draw a best fit curve through each of your results to see how linear the voltage change is for each thermocouple over the range 0 to 100°C. Note how your results prove that voltage goes up as temperature increases.
4. Compare your results with the standard specifications for the thermocouples (table 2).
5. On your results table, find the difference between the standard and your results (the deviation) and calculate the percentage error.

Percentage error = (deviation/standard) x 100

6. Your results should be reasonably linear, but the deviation may be large and consistent (an offset). Can you identify the causes of any errors? Hint - think about the thermocouples and the connections to the amplifier.
7. Can you understand why thermocouple connections are important, and why you cannot simply connect a thermocouple directly to an ordinary measuring device and expect it to work correctly?

°C	Ω	°C	Ω	°C	Ω	°C	Ω	°C	Ω
0	100.00								
1	100.39	21	108.18	41	115.93	61	123.63	81	131.28
2	100.78	22	108.57	42	116.31	62	124.01	82	131.66
3	101.17	23	108.96	43	116.70	63	124.39	83	132.04
4	101.56	24	109.35	44	117.08	64	124.77	84	132.42
5	101.95	25	109.73	45	117.47	65	125.16	85	132.80
6	102.34	26	110.12	46	117.86	66	125.54	86	133.18
7	102.73	27	110.51	47	118.24	67	125.93	87	133.57
8	103.12	28	110.90	48	118.63	68	126.31	88	133.95
9	103.51	29	111.29	49	119.01	69	126.69	89	134.32
10	103.90	30	111.67	50	119.40	70	127.07	90	134.71
11	104.29	31	111.67	51	119.78	71	127.46	91	135.09
12	104.68	32	112.06	52	120.17	72	127.84	92	135.47
13	105.07	33	112.83	53	120.55	73	128.22	93	135.85
14	105.46	34	113.22	54	120.93	74	128.61	94	136.23
15	105.85	35	113.61	55	121.32	75	128.99	95	136.61
16	106.24	36	114.00	56	121.71	76	129.37	96	136.99
17	106.63	37	114.38	57	122.09	77	129.75	97	137.37
18	107.02	38	114.77	58	122.47	78	130.13	98	137.75
19	107.40	39	115.15	59	122.86	79	130.52	99	138.13
20	107.79	40	115.54	60	123.24	80	130.90	100	138.51

(PRT) Resistance Temperature Detector Standards



°C	µV	°C	µV	°C	µV	°C	µV	°C	µV
0	0								
1	39	21	838	41	1653	61	2478	81	3308
2	79	22	879	42	1694	62	2519	82	3350
3	119	23	919	43	1735	63	2561	83	3391
4	158	24	960	44	1776	64	2602	84	3433
5	198	25	1000	45	1817	65	2644	85	3474
6	238	26	1041	46	1858	66	2685	86	3516
7	277	27	1081	47	1899	67	2727	87	3557
8	317	28	1122	48	1941	68	2768	88	3599
9	357	29	1163	49	1982	69	2810	89	3640
10	397	30	1203	50	2023	70	2851	90	3682
11	437	31	1244	51	2064	71	2893	91	3723
12	477	32	1285	52	2106	72	2934	92	3765
13	517	33	1326	53	2147	73	2976	93	3806
14	557	34	1366	54	2188	74	3017	94	3848
15	597	35	1407	55	2230	75	3059	95	3889
16	637	36	1448	56	2271	76	3100	96	3931
17	677	37	1489	57	2312	77	3142	97	3972
18	718	38	1530	58	2354	78	3184	98	4013
19	758	39	1571	59	2395	79	3225	99	4055
20	798	40	1612	60	2436	80	3267	100	4096

K Type Thermocouple Standards

IV. Results:

Part 1 - PRT Simulation, Constant Voltage and Current

Constant Voltage	
Connection	**Millivoltmeter Reading (mV)**
Direct Connection (Solid Wires)	
Dotted wires connection (Long Wires)	

Constant Current	
Connection	**Millivoltmeter Reading (mV)**
Direct Connection (Solid Wires)	
Dotted wires connection (Long Wires)	

Part 2 - PRT Calibration

PRT Calibration					
Reference Temperature (ºC)	**Measured Voltage (mV)**	**Calculated Resistance (Ω)**	**Standard Resistance from specifications (Ω)**	**Deviation (Ω)**	**Error (%)**

Part 3 – J and K Thermocouple Linearity:

J or K type Thermocouple Calibration					
Reference Temperature (ºC)	**Measured Voltage (mV)**	**Measured Voltage/20 (µV)**	**Standard Voltage (µV)**	**Deviation (mV or µV)**	**Error (%)**

Experiment # 7: CONVERSION OF WORK TO HEAT

I. Objective:

To verify the first law of thermodynamics, and to determine the mechanical equivalent of heat

II. Apparatus:

The apparatus is a bench-type unit, consists of a universal electric motor with variable speed control for driving a water filled brass drum calorimeter, two sets of weights, heavy and light, a set of brake bands to encircle the drum, a spring balance, a thermometer and a counter for recording the revolutions of the drum. All items of equipment are mounted on the steel cabinet which contains the motor control gear.

III. Theory

One of the most fundamental laws of nature is conservation of energy principle (first law of thermodynamics). It simply states that during an interaction, energy can change from one form to another but the total amount remains constant.

For non-flow system (such as the system of this experiment) first law of thermodynamics states that:

$$Q - W = \Delta U \qquad (1)$$

Where, W is work done, Q is heat transferred across system boundary, and ΔU is change in system internal energy.

Applying the first law of thermodynamics to apparatus of this experiment requires a number of definitions to be made and some simplifying assumptions.

System: Let the system be defined as the calorimeter drum, the band brake and the water contained in the drum.

Heat Transfer: Assume the heat transfer to the surroundings is negligible that is $Q = 0$.

Work Done: Work is done on the system by the electric motor which imparts relative movement ‘between the calorimeter drum and the band brake. The band brake grips the drum and the motor has to overcome the frictional force, which is measured by the force that has to be applied to restrain the band brake from rotating.

Restraining Force, $F = W_L - W_s + s$ (2)

The product of the restraining force and the circumference of the drum then gives the work done per revolution.

$$W = \pi DF = \pi D(W_L - W_s + s) \qquad (3)$$

and the total work done during the time period is

$$W = \pi DFN = \pi D(W_L - W_s + s)N \qquad (4)$$

Where N is the number of revolutions.

Change of Internal Energy: The change of internal energy is calculated from the mass of the calorimeter, the mass of the water, their respective specific heats and the change in temperature.

$$\Delta U = (M_c C_c + M_w C_w)\Delta T \qquad \text{……………………….…………………………………….(5)}$$

Mechanical Equivalent of Heat: the mechanical equivalent of heat J is defined as

$$J = \frac{W}{\Delta U} \qquad \text{……………………………………..……..………………………..……….(6)}$$

For same units of W and ΔU, the theoretical value of J is equals to one.

IV. Procedure:

a) Introduce a measured Quantity of cool water into the inside of the drum. Record the ambient temperature.

b) Insert the thermometer at an angle through the central hole in the end of the drum and clamp it.

c) Switch the power on and adjust the speed control to give a speed between 60 and 120 rev/min. The speed can be judged by observing the revolution counter. Place 4 off 50 gram weights on to the light weight carrier and adjust the support for the spring balance so that the band and weights are held with the pointer of the spring balance near the centre of the scale. If necessary add or remove weights from the light weight carrier to achieve this condition.

d) After a few revolutions record the temperature of the water, the number of revolutions shown on the revolution counter and the spring balance reading. Repeat these readings either once per minute or every 100 revolutions to suit your preference. Continue the experiment until the water temperature is 5 ^{o}C higher than the room temperature; again record the ambient temperature.

e) Record the value of the large weights and small weights including the weights of the carriers.

V. Results:

Revolution counter reading									
Water Temperature ⁰C									
Spring Balance									
Ambient Temperature ⁰C									

Large carrier weight (g)	**200**	**Large weight and carrier (kg)**		**Mass of water (g)**	
Small carrier weight (g)	**50**	**Small weight and carrier (g)**		**Mass of calorimeter (g)**	**1.07 kg**
Specific heat of water J/g ⁰C	**4.1855**	**Specific heat of calorimeter J/g ⁰C**	**0.368**	**Drum Diameter (mm)**	**152.4**

Analysis:

1. Select from the table of results those readings, which cover a range of at least 500 revolutions during which time the water temperature rose from below the mean ambient temperature to above the mean ambient temperature.
2. Using a consistent set of units calculate the mechanical work done from equation 4.
3. Calculate the heat generated from equation 5.
4. Calculate the mechanical equivalent of heat J from equation 6.

Calculate the percentage error.

Experiment # 8: Stirling Cycle Hot Air Engine

I. Objective:

- To demonstrate a direct conversion of heat energy into shaft power using a Stirling cycle.
- To investigate the performance of a hot air engine operating on Stirling cycle.
- To investigate the parameters affecting the cycle performance and cycle efficiency.

II. Apparatus:

The setup consists of three main parts:

- Console unit
- Heat Transfer Service Unit
- Stirling engine

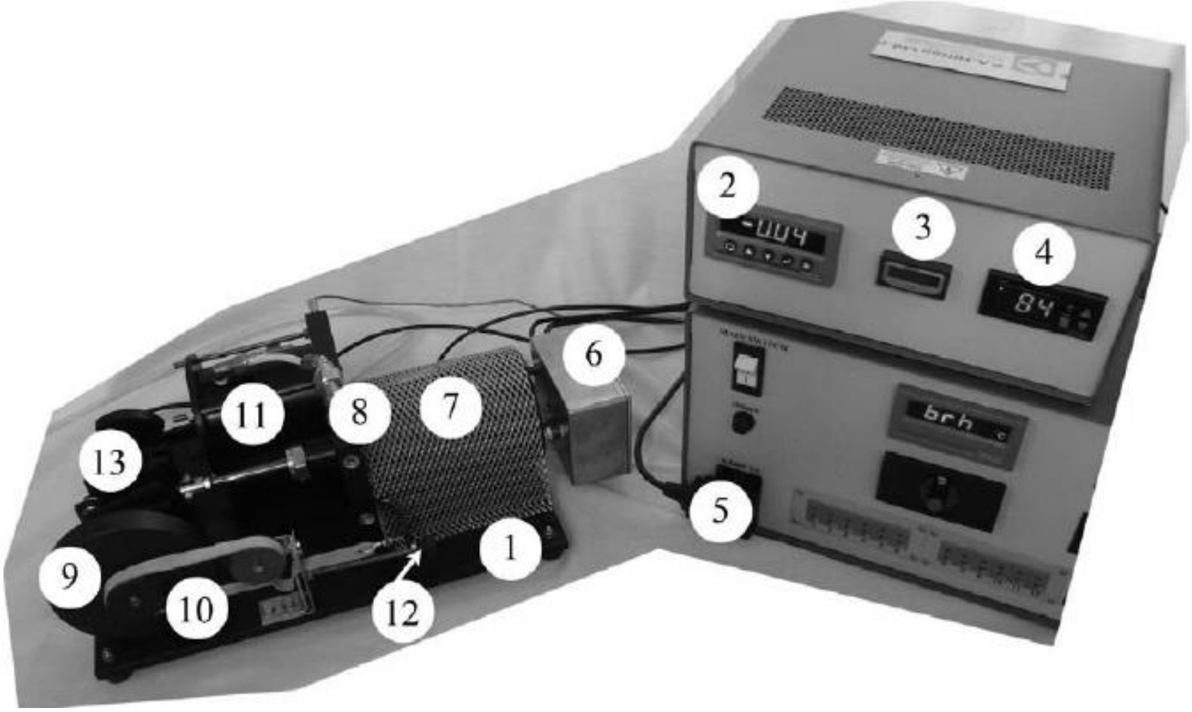


Figure 1: Stirling Engine setup

Diagram Key

1. Stirling cycle engine
2. Load cell display
3. Tachometer indicator
4. Heater temperature display
5. Heater power connection
6. Heater terminal enclosure
7. Heater cylinder
8. Cooling water inlet
9. Flywheel
10. Belt brake
11. Power cylinder
12. Load Adjuster
13. Crankshaft

Stirling Engine Detail and operation

The Stirling Cycle Engine is based upon the Gamma arrangement where there is a parallel power cylinder (11) and heater cylinder (7). The power cylinder is connected to a common power crank

(14) and crankshaft (13). The power crank (14) and crankshaft move the power piston and displacer piston respectively.

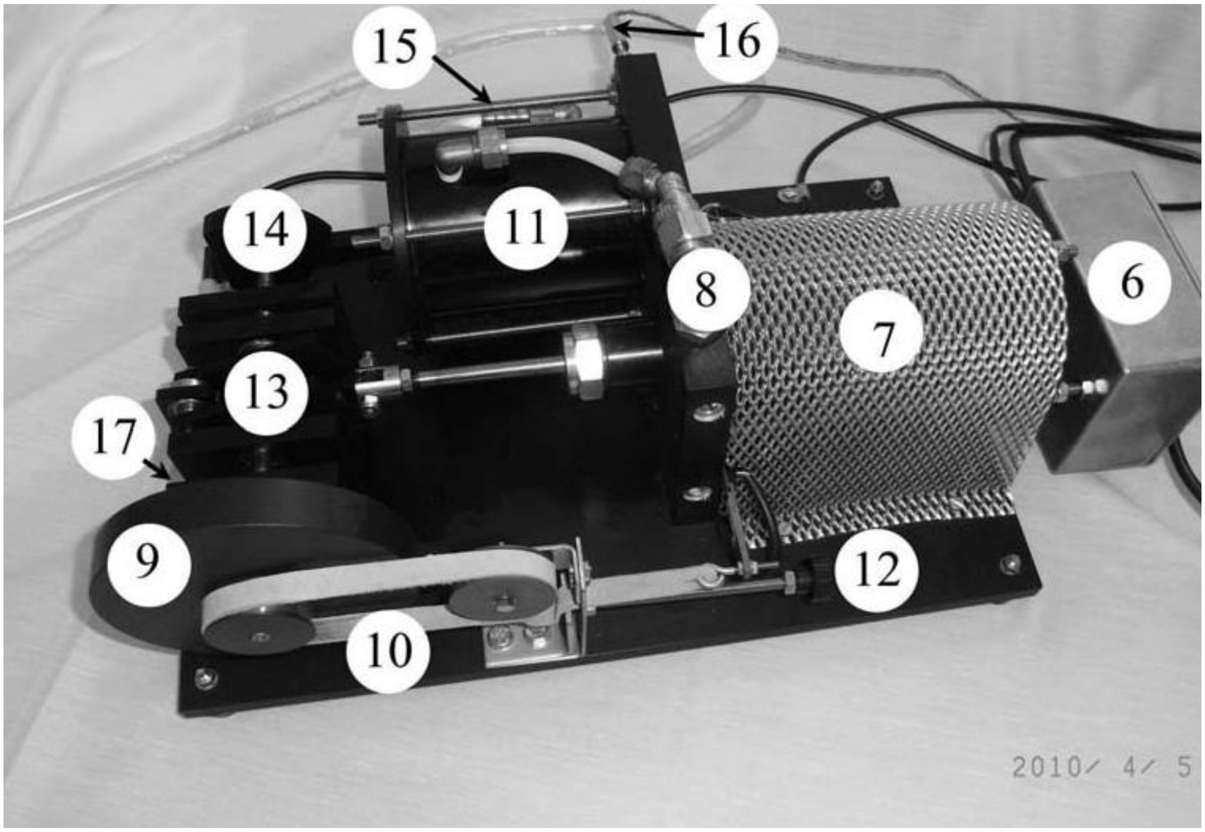


Figure 2: Details of Stirling Engine

A flywheel (9) stores energy from the power stroke to maintain rotation for a complete cycle. The engine must be turned by hand to start and as the displacer piston moves forward hot air around the electric heater is displaced behind the loose fitting displacer piston. Two holes communicate with the power cylinder (11). The hot air, now behind the displacer is being compressed by the forward moving power piston. As the power piston moves over top dead centre the hot air expands to push the power piston out and add energy to the flywheel motion. This energy then carries both displacer and power piston around for a further stroke. The cycle repeats rapidly depending upon the temperature difference between the electric heater and the cold side of the engine which is the power cylinder (11). The power cylinder is water cooled. The setup is fully instrumented to measure all needed data like: hot and cold temperatures, current, voltage, brake force, and engine rpm.

III. Theory:

The Stirling engine was patented in 1816 by Robert Stirling (1790-1878), a minister of the Church of Scotland. It has the potential to be much more efficient than a gasoline or diesel engine. But today, Stirling engines are used only in some very specialized applications, like in submarines or auxiliary power generators for yachts, where quiet operation is important. Also, there are many toy models available, either commercially, or prototypes made by enthusiastic hobbyists. Although there hasn't been a successful mass-market application for the Stirling engine, some very high-power inventors are working on it.

A Stirling engine uses the Stirling cycle, which is unlike the cycles used in internal-combustion engines. No combustion takes place inside the cylinders of the engine.

- The gasses used inside a Stirling engine never leave the engine. There are no exhaust valves that vent high-pressure gasses, as in a gasoline or diesel engine, and there are no explosions taking place. Because of this, Stirling engines are very quiet.
- The Stirling cycle uses an external heat source, so that any heat source is suitable to power a Stirling engine. Thus a Stirling engine can be driven by carbon fuels (coal, natural gas, gasoline, and Diesel oil), hydrogen, solar radiation, nuclear power, waste heat of industrial processes, etc.

Even though it is not a new invention, there have been at least thousands of different designs stemmed from his original patent.

IV. Working principle

The key principle of a Stirling engine is that a fixed amount of a gas is sealed inside the engine. The Stirling cycle involves a series of events that change the pressure of the gas inside the engine, causing it to do work. A simplified Stirling engine uses two cylinders. One cylinder is heated by an external heat source, and the other is cooled by an external cooling source. The gas chambers of the two cylinders are connected by regenerator, and the pistons are connected to each other mechanically by a linkage that determines how they will move in relation to one another. An ideal Stirling cycle consists of 4 processes. Figure 3 shows these processes on P-V and T-S diagrams, and the description of these processes are illustrated in figure 4.

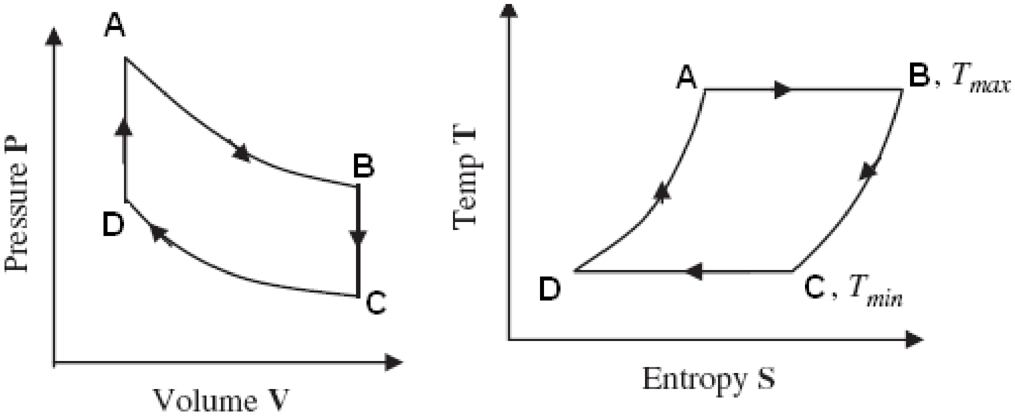


Figure 3 P-V and T-S diagram for Stirling cycle

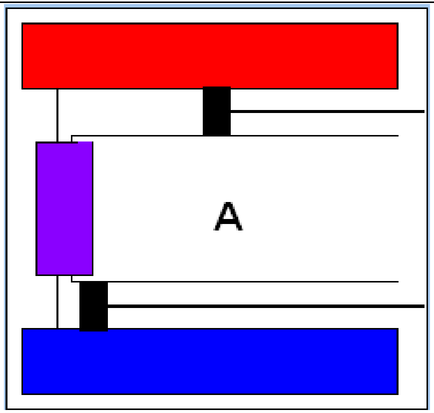
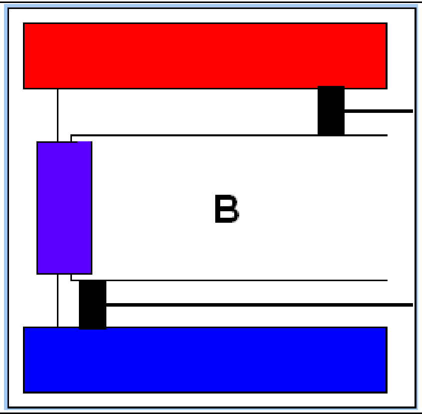
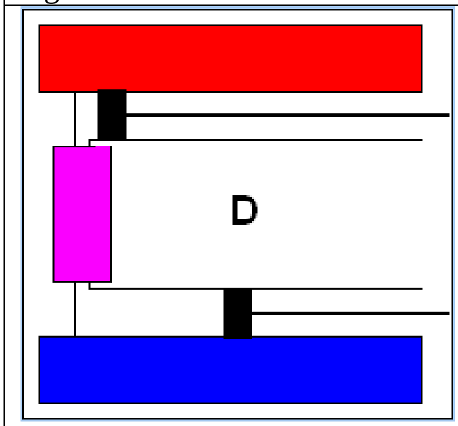
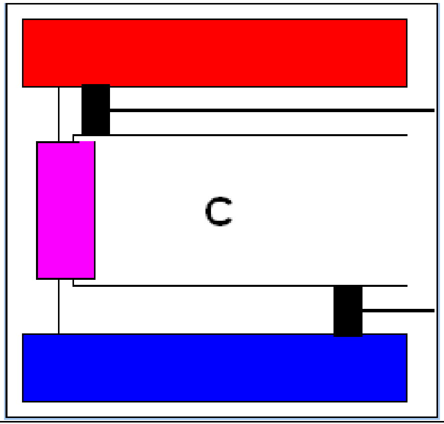
	**Process A-B** Isothermal expansion at temperature T2 in the working cylinder. Heat is supplied from the heat source	
Process D-A Constant volume process whilst temperature increases from T1 to T2. Heat is supplied from the regenerator.		**Process B-C** Constant volume process whilst temperature decreases from T2 to T1. Heat is absorbed by the regenerator.
	**Process C-D** Isothermal compression at temperature T_1, where heat is lost to the surroundings	

Figure 4: Illustration of processes of Stirling cycle

Efficiency of Stirling Engine

➢ Theoretically

❑ Stirling engine efficiency = Carnot efficiency $= 1 - \frac{T_C}{T_H}$

❑ Unfortunately working fluid or gas is not ideal this causes the efficiency to be lower than Carnot efficiency.

➢ In fact, Stirling engine efficiency depends on

❑ Temperature ratio (proportionally)

❑ Pressure ratio (inversely proportional)

❑ Specific heat ratio (inversely proportional

Types of Stirling Engines

Stirling engines are classified to four types with viewpoint of working space. The alpha-type has two power pistons (Figure 5). The beta-type has a displacer and a power piston with the same cylinder (Figure 6). The gamma-type has a displacer and a power piston with independent cylinders (Figure 7). And the double-acting type has four power pistons and uses both the top and bottom sides of the pistons (Figure 8). Also the alpha-type is called the two-piston type. The beta- and gamma-type are called the displacer-piston type.

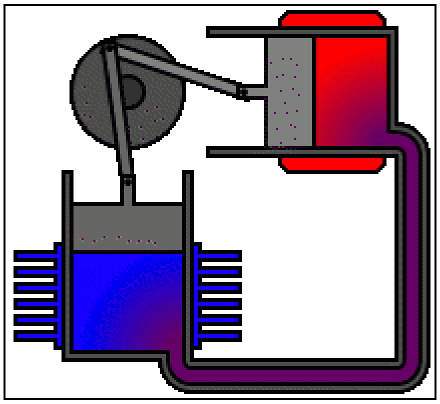
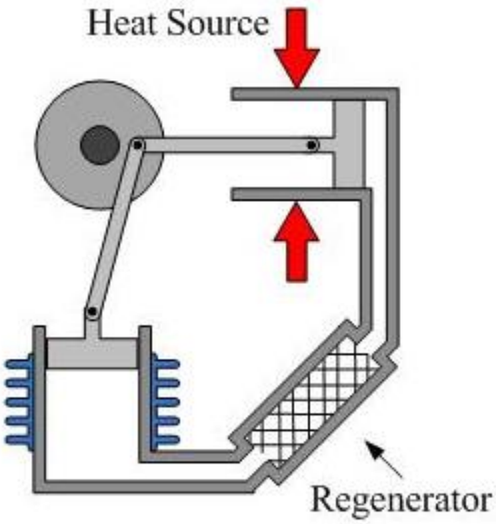


Figure 5: Alpha type Stirling engine



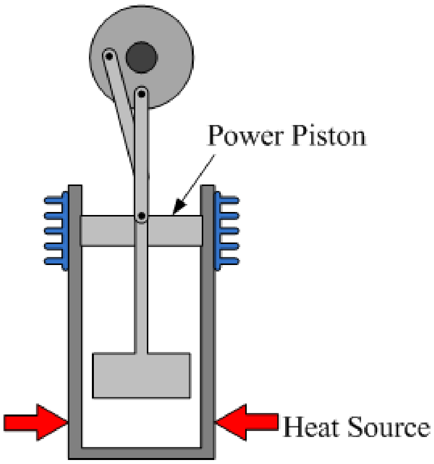
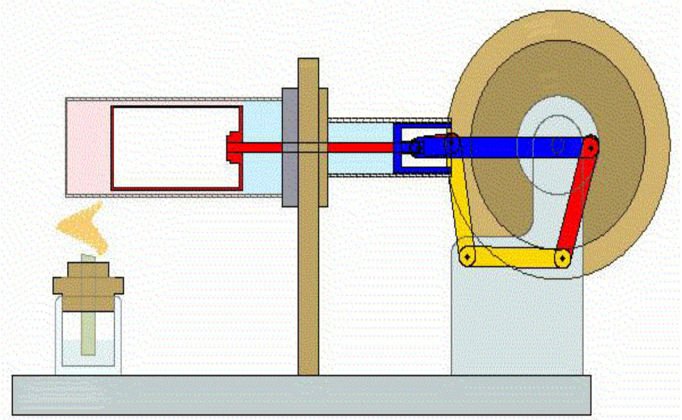


Figure 6: Beta type Stirling engine



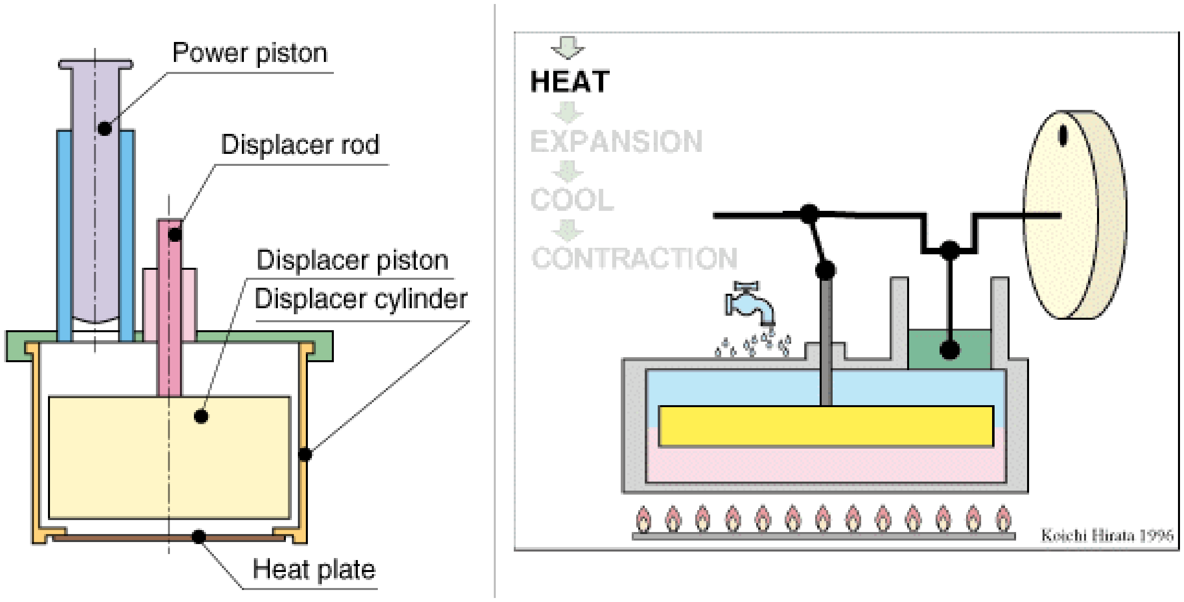
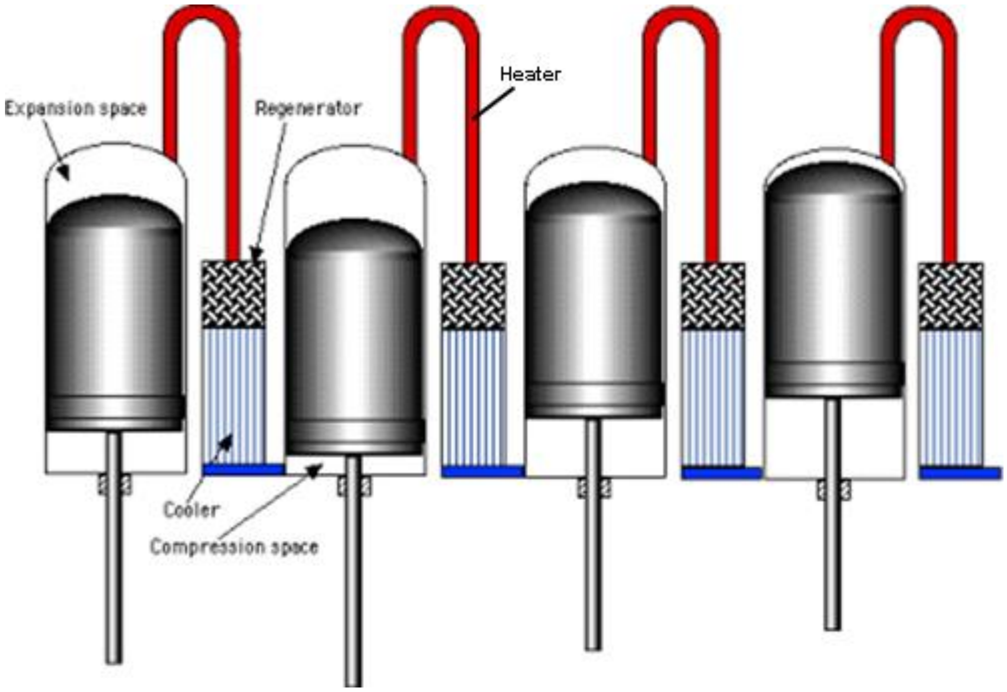


Figure 7: Gamma type Stirling engine



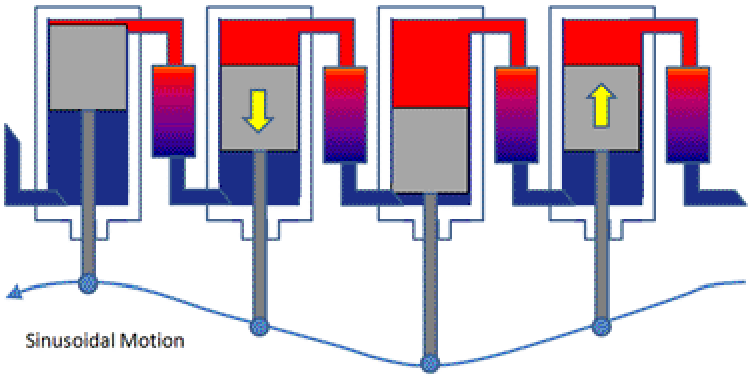


Figure 8: Double acting Stirling Engine – four piston type

V. Procedure:

1. Turn on the main switch of the Heat Transfer Service unit and turn the Heater power control to zero.

2. Ensure cooling water is flowing through the water cooling connections.

3. Zero the Load Cell Display

4. Turn the heater power control on the Transfer Service unit slowly clockwise until a current of approximately 1.3-1.4 amps is shown on the Ammeter.

5. Adjust the power input to keep the temperature of the heater $\mathbf{T_H}$ around 580°C.

6. Now ensure that the load adjuster is set so that the belt brake is loose. Spin the flywheel in a clockwise direction and the engine should start. Allow the speed to stabilize and then record the heater temperature $\mathbf{T_H}$**,** the cold side temperature $\mathbf{T_C}$, the rotational speed **N**, the load cell force **F**, the heater current **I** and the heater voltage **V**.

7. Adjust the load adjuster so that the indicated load increases slightly.

8. Monitor the heater temperature $\mathbf{T_H}$ and adjust the heater control to bring the temperature back to **its** original setting, or as near as possible. Once the system has reached a stable condition again record the parameters as before.

9. Slowly repeat the process increasing the load until the engine stalls, recording the parameters at each load.

Analysis:

1. Torque T [N]

$\boldsymbol{T = F \times r}$ Where $\mathbf{r = 0.02\ m}$ from apparatus data

2. Output Power P_{out} [watt]

$$\boldsymbol{P_{out} = T\omega = \frac{T \times N \times 2\pi}{60}}$$

3. Input Power P_{in} [watt]

$$\boldsymbol{P_{in} = V \times I}$$

4. Engine actual efficiency η_{act}

$$\boldsymbol{\eta_{act} = \frac{P_{out}}{P_{in}} \times 100\%}$$

5. Engine ideal efficiency η_{ideal}

$$\boldsymbol{\eta_{ideal} = 1 - \frac{T_C}{T_H}}$$

6. Plots

- Plot Torque (T) and output power (P_{out}) versus rotational speed (N)
- Plot actual efficiency and ideal efficiency versus rotational speed (N)

7. Comment on all your calculations and plots

VI. Results:

No	T_H °C	TC °C	N rpm	F N	I Amps	V Volts
1						
2						
3						
4						
5						
6						
7						

Experiment # 9: BOMB CALORIMETER

I. Objective:

To determine the heat of combustion (calorific value) of a certain type of fuel.

II. Apparatus:

A bomb calorimeter consists of a stainless-steel vessel into which a sample of the fuel under investigation is introduced, together with high pressure oxygen to facilitate complete combustion. The vessel sits in a container with a known amount of water.

A pair of small electrodes in contact with the fuel is ignited electrically and a complete combustion of the sample takes place. This result in an increase of the temperature of the vessel and the water (collectively referred to as the calorimeter). If the calorimeter and its contents are regarded as the system, no heat flows to the surrounding during this process. A motorized stirrer maintains the temperature distribution of the water as homogeneous as possible.

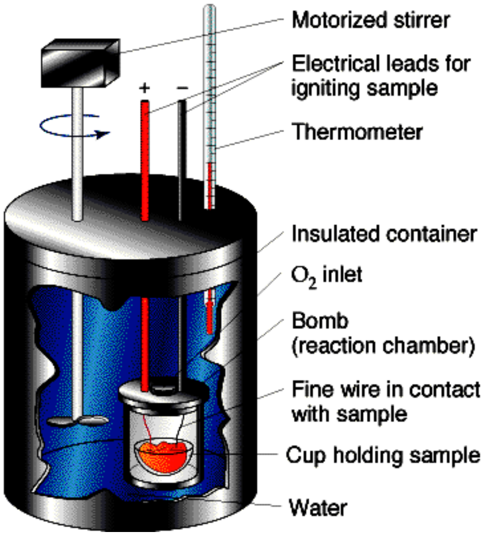


Figure 1: A typical bomb calorimeter

Theory:

Heat of combustion (calorific value) is the quantity of energy released when a unit mass of fuel is burned in a constant volume. The water surrounding the bomb gains the energy of combustion. Heat balance to the bomb calorimeter gives:

Heat liberated from fuel = Heat gained by water and Bomb calorimeter.

$$m_f \times C.V. = m_w.Cp_w.(T_f - T_i)_{corrected} + m_b.Cp_b.(T_f - T_i)_{corrected}$$ ....…………………… … [**1**]

Where,

m_f - mass of fuel burned (kg).

m_w- mass of water (kg).

m_b - mass of bomb (kg).

C_{pw} and C_{pb} are specific heat of water and bomb (kJ/kg K).

T_i - initial temperature (K).

T_f - final temperature (K).

C.V. - Calorific value of fuel (kJ/kg).

Or

$$m_f \times C.V. = m_e.Cp_e.(T_f - T_i)_{corrected}$$ ......………………………… [**2**]

Where,

m_e - equivalent mass of water and bomb calorimeter.

C_{pe}- equivalent specific heat of water and bomb calorimeter.

III. Procedure:

1. Weigh a small quantity of the fuel (about 1/2 gram).
2. Fix the ignition wire and close the bomb.
3. Charge the bomb with oxygen to a pressure of about 30 bars.
4. Insert the bomb in the calorimeter and pour (1.5) liters of water.
5. Insert the thermometer and start the stirrer.
6. Record the temperature of the water every 60 sec up to 5 minutes before ignition.
7. Ignite the fuel and record the temperature every 30 sec until the temperature reaches its maximum value and starts to decrease.
8. Continue recording the temperature during the cooling process every 60 sec for further 5 minutes.
9. Fill the results in the attached data sheet.

IV. Results:

Fuel type = …………………..

Mass of crucible only (g) = …………………..

Mass of crucible + fuel sample (g = …………………..

Mass of fuel (g) = …………………..

Equivalent mass and specific heat $\left(m_e.C_{pe}\right)$ of water and bomb calorimeter (kJ/°C) = 8.4425

Ignition ↓ 300

Time (sec.)	0	60	120	180	240	300	330	360	390	420	450	480	510	540	570
Temp °C															

Time (sec.)	600	630	660	690	720	750	780	810	840	870	900	930	960	990	1020
Temp °C															

Table 1: Time & Temperature Readings

Analysis:

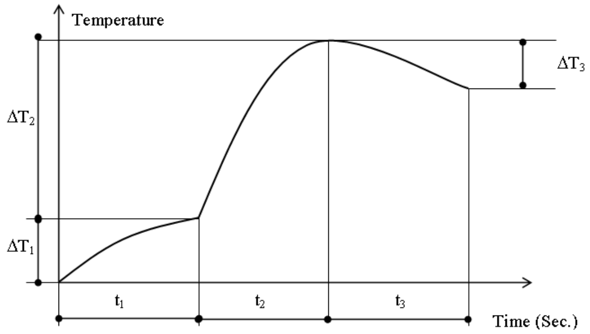
The temperature rise observed will not be the correct value to be used in the above equation since heat may be gained or lost from the apparatus during the experiment. It is necessary to make a correction to the temperature rise as follows:

1. Draw the time temperature curve; it should be like the figure shown below.
2. Find the corrected temperature difference as follows:

   $(Tf - Ti)_{corrected} = \Delta T2 -$ effect of heat added due to stirrer + effect of heat loss to ambient.

   $$(T_f - T_i)_{corrected} = \Delta T_2 - (\frac{\Delta T_1}{t_1} * t_2) + (\left|\frac{\Delta T_3}{t_3}\right| * t_2)$$

3. Calculate the calorific value of the fuel using equation (2).



4. Compare the estimated value of CV with a standard one. What is the percentage error?
5. What are the sources of error?

APPENDIX

General Safety Instructions

The basic purpose of laboratory safety is to protect students, researchers, technicians and teachers from the many hazards encountered during the use of various materials and equipments, so safety in the laboratory must be of vital concern to all those engaged in experimental work. It is therefore the responsibility of everyone to adhere strictly to the basic safety precautions provided and to avoid any acts of carelessness that can endanger his life and that of others around him. It is equally important to always abide by all the instructions for conducting the experimental work during the laboratory sessions. A set of information is presented here to safeguard you while in the laboratory.

1. Smoking is not allowed in the laboratory.
2. Food or beverages are not allowed in the laboratory.
3. Avoid long hair and long sleeved loose clothes and wear lab coat while conducting experiments to minimize the risk of clothing getting caught in the machines.
4. Use appropriate personal protective equipment at all time, like Gloves, Safety glasses, Skin Protection, Hearing Protection, and Foot Protection (don't wear open sandals)
5. No running, playing, bantering, and kidding in the laboratory.
6. Know locations of first aid and all emergency equipments, such as fire alarm, water hoses, fire extinguishers, fire blankets, eyewash stations, and safety showers. Know how to find and use them.
7. Use laboratory equipment for its designed purpose.
8. Always follow instructions and use only machines and equipment that you are authorized and qualified to operate. If you have any question, consult with your supervisor.
9. Know and follow safety rules for specific experiments or tasks.

10. Know potential hazards in your work and ways of working safely to prevent such hazards.

11. Working alone should be avoided. Someone should always be within call when a laboratory procedure is being performed.

12. Avoid mouth contact with any laboratory equipment including pipettes. Use safety filler to fill pipettes.

13. Avoid exposure to gases, vapours, and particulates by using a properly functioning laboratory fume hood.

14. Use ground fault circuit intemipters where there is a risk of an operator coming in contact with water and electrical equipment simultaneously.

15. Follow electrical safety rules and make sure your hands are dry before using electrical equipment, grounding portable electrical tools. Make sure electrical wires are connected properly without short circuit before operating. Wear protective clothing, well-insulated groves and boots, if required.

16. Only trained, qualified personnel may repair or modify electrical or any equipment.

17. Properly support glass wares using stand, clamps, etc. Use proper rings to place round bottom flasks.

18.
    - Reduce fire hazard.
    - Use shower for fire victims.
    - While fire on clothing, do not run or fan flames.
    - Smother flames by wrapping in fire blankets.
    - Spills of flammable solvents can be a source of fire.

19. Upon hearing fire alarm, you should evacuate the area and follow emergency procedure.

20. Report all injuries including minor scratches, cuts, and burns for First Aid treatment. Corrective actions should be taken to prevent future injuries.

21. Report any damage to equipment or instrument and broken glassware to the laboratory instructor as soon as such damage occurs.

22. Wash hands upon completion of laboratory procedures and remove all protective equipment including gloves and lab coats.

Instructions for Preparing Laboratory Reports

The report must be prepared according to the following outline.

1. Title Page

The Title page needs to contain:

a. The title of the experiment.
b. The course name and its code.
c. The names of the writer and his partner, their ID numbers.
d. Name of the instructor to whom the report is submitted
e. The date when the experiment was run and the submission date.

2. Abstract

The abstract should summarize four essential aspects of the report:

a. The purpose of the experiment briefly.
b. Brief description of what was done and how it was done.
c. Selected results (numerical values, if available).
d. Brief assessment of the results e.g. some percentage errors in experimental results in comparison with theoretical values.

* The abstract should be one paragraph of almost 5 lines.

3. Introduction

This should be a very brief and incisive summary that covers the following:

a. Objectives of the experiment.
b. Underlying fundamental chemical and physical principles

*These two points should help you navigate the introduction:

- The experiment is already finished. Use the past tense when talking about the experiment.

"The objective of the experiment was..."

- The report, the theory and permanent equipment still exist; therefore, these get the present tense:

"The purpose of this report is..."

4. Theoretical Background

This section should include the theory behind the experiment. It should also contain all those equations, which are used to acquire a certain result. Theoretical correlations, which are used for comparison with experimental results, should also be included.

5. Procedure

Describe the process in chronological order. Using clear paragraph structure, explain all steps in the order they actually happened, not as they were supposed to happen. It should be written in your own words, e.g. the feed pump was calibrated.

6. Results and Calculations

Summarize the data and present any significant experimental findings. Whenever possible, the results should be presented in graphical or tabular forms because they are much easier to comprehend and interpret. The graphs and tables should be accompanied by a verbal description of the results that draws attention to their most noteworthy characteristics. Graphics need to be clear, easily read, and well labeled.

Show your calculation in details, stating any used equation.

7. Discussion of Results

It is the most important part of your report, because here, you show that you understand the experiment beyond the simple level of completing it. In this section you should discuss your experimental results and observations. If the results are obtained in terms of graphs, then interpret them also. Describe observed trends and possible relationship between parameters e.g. how a change in one variable affects another. Also show how you make comparison with the values obtained theoretically and discuss the deviation of experimental results from theoretical values. The possible source of errors should also be mentioned.

8. Conclusions and Recommendations

Simply state what you know now for sure and what you have learned from the experiment. It should also include main results (numerical values) and the errors between the experimental and theoretical values.

Recommendations are the proposals for future work, e.g. suggested changes in equipment, Study of new variables, or possible experiments in relative fields.

9. Organization and Neatness

The students must organize their reports in accordance with the format described earlier. It is required to use computer packages such as EXCEL, or any other suitable engineering software to draw graphs. The neatness will include how the student has organized his report, accuracy in grammar and spelling, numbering pages, figures, tables and equations.

Report Grading Form

Name of Student: _____________ID #:_______________

Title of Report: _______________________________________

Term:___________________________Date:_______________

		Max. mark	**Actual mark**
1	Title Page	2	
2	Abstract	10	
3	Introduction	5	
4	Procedure	15	
5	Results and calculation	25	
6	Discussion of Results	20	
7	Conclusion and Recommendations	10	
8	Raw Data	5	
9	Organization and Neatness	8	
Total		100	

Comments:

Signed: ___________________________