Rajiv Gandhi University of Knowledge Technologies

Department of Chemical Engineering

Chemical Reaction Engineering Lab

(CH3801)

Course Objectives:

- Operate lab equipments like CSTR, Batch, PFR reactors.
- Analyze the concentration versus time data and determine the specific rate constant and the order of the reaction.
- Compare theoretical and experimental conversions in a CSTR and PFR.
- Estimate RTD and model parameters in a CSTR, PFR, packed bed and CSTRin-series.

List of Experiments:

S.No	Name of the experiment
1.	CSTRS in series
2.	Isothermal batch reactor
3.	Isothermal CSTR
4.	RTD studies in CSTR
5.	RTD studies in PFR
6.	Tubular reactor

Course Outcomes:

- Design the experiments to acquire the kinetic and RTD data.
- Analyze the experimental data to obtain the reaction rate expression (reaction order and specific reaction rate constant).
- Attain competency in running the bench scale and pilot scale reactors.

Equipment Diagrams

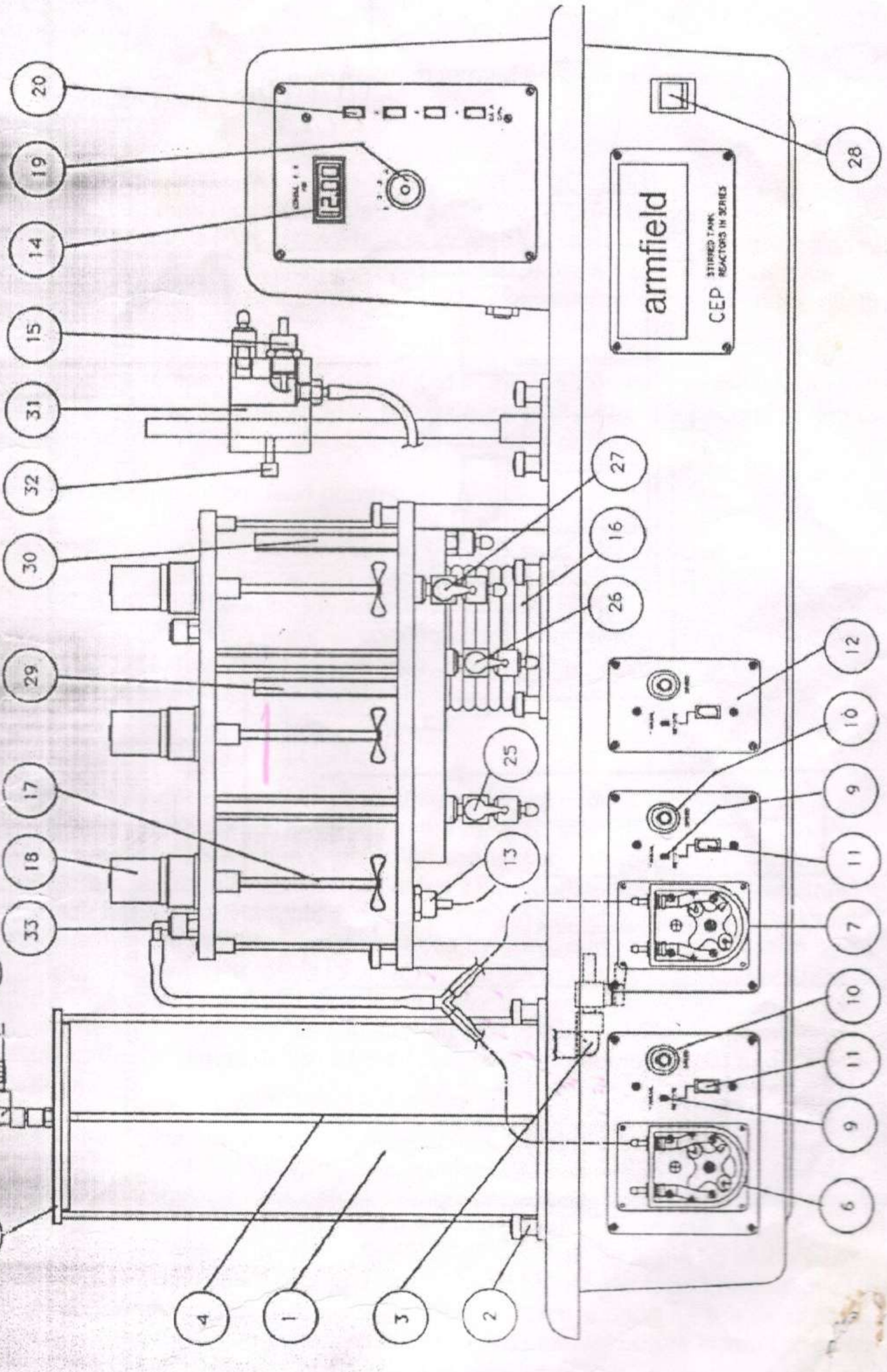

Figure 1: Front View of CEP-MkII Stirred Tank Reactors in Series

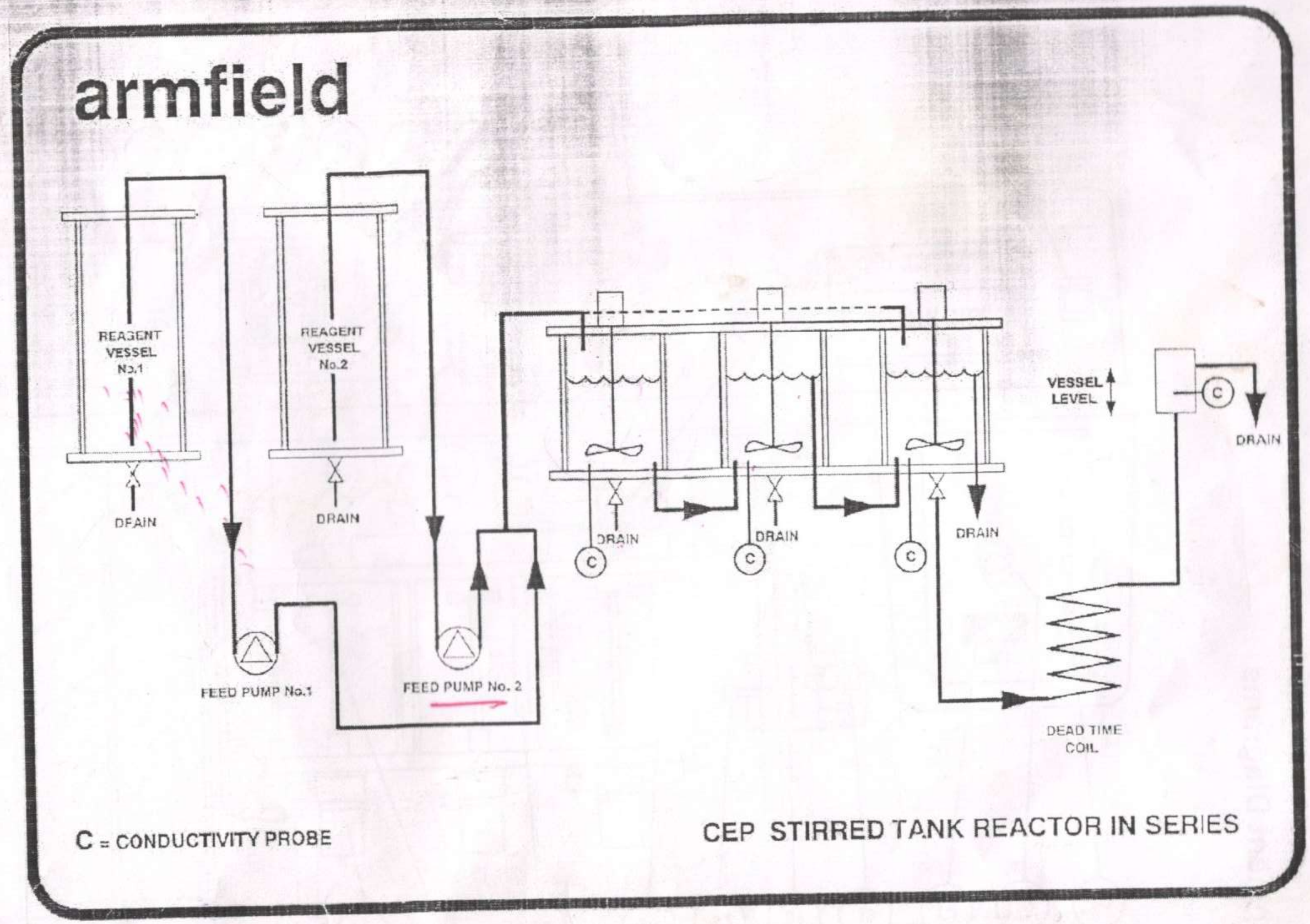

Figure 2: CEP-MkII Stirred Tank Reactors in Series

Description

Where necessary, refer to the drawings in the Equipment Diagrams section.

Overview

The CEP-MkII is constructed on a vacuum formed plinth which is designed for bench mounting and consists of the following:

a. Storage of chemical reagents

A reagent tank module consists of two 5.0 litre capacity glass vessels (1) mounted into a PVC base which is located on studs on the plinth top and held in position by thumbnuts (2). Drain valves (3) are fitted to the underside of each reagent vessel and are accessible from within the trough on the plinth top.

Rigid standpipes (4) are incorporated in the lids (5) of the vessels to provide the suction to the reactor feed pumps (6) and (7). Flexible silicone tubing (8) is used to connect the standpipes to the inlets on the pumps.

b. Variable throughput feed pumps

The peristaltic type pumps (6) and (7) are capable of a throughput range of nominally 0-95ml per minute. Each pump is switched on and off using toggle switch (9) and the speed is adjusted by the 10-turn potentiometer (10). The speed setting of the pump is connected to the 50 way I/O port on the CEP-MKII so that the speed can be logged on a computer using an appropriate interface (not supplied with CEP-MKII).

c. Tank Reactors

Three cylindrical tank reactors with glass walls glass are mounted on a common plastic base. Galleries in the base connect the tanks together as required for operation in series. The connections are shown on the flow diagram on page 8. Liquid from either of the peristaltic pumps enters tank No. 1 (left hand side) via a quick release fitting in the lid of the tank. The liquid flows through the three tanks in series by gravity. In normal operation liquid flows downwards through tank No. 1, leaving at the base and enters tank No. 2 at the base. The liquid then flows upwards through tank No. 2 and leaves via the standpipe (29). The liquid then enters tank No. 3 at the base, flows upwards through tank No. 3 then leaves via the standpipe (30) and flows to drain.

An alternative outlet from the base of tank No. 3 is controlled by a valve (27) that connects to an overflow arrangement (31) via a 'dead time' coil (16). The height of the overflow is adjustable and is locked by tightening the clamping screw (32). When using the 'dead time' coil, liquid enters tank No. 3 directly from the peristaltic pumps via the quick release fitting in the lid of the tank (tank No. 1 and No. 2 are not used). The flexible outlet tube from the overflow arrangement (31) is located inside the cylindrical support for the dead time coil so that the liquid flows into the moulded channel before flowing to drain via the valve at the end of the channel.

d. Variable speed agitators

The CEP-MkII is equipped with a propeller stirrer (17) in each tank reactor. Each stirrer is driven by a separate electric motor (18). All stirrers revolve at the same speed and the speed is adjusted in exactly the same way as the feed pumps using the On/Off switch and the ten turn potentiometer.

e. Continuous conductivity measurement of tank contents.

Each tank reactor has a conductivity probe (13) installed in the base (only probe in tank No. 1 shown). The conductivity is displayed on a digital meter (14) in the control console in units of milliSiemens. Also, a fourth probe (15) is positioned in the adjustable overflow to measure the conductivity of the solution leaving the 'dead time' coil (16) located behind the tanks on the plinth top.

During a chemical reaction, the conductivity of the reacting solution changes as more of the reactants are converted. This is monitored and used to determine the degree of conversion and the rate of conversion. Also, if a conducting salt is introduced into the system, the conductivity can be used to monitor and record the degree of mixing in the reactors.

The readings of all four probes can be separately displayed on the digital meter (14) using the four position selector switch (19). Also, the probe readings can be simultaneously recorded using the data output sockets (20). See the Operation section for details about connection to a suitable computer interface.

f. Mainsplate (at rear of plinth)

The CEP-MkII unit is supplied with a detachable mains lead with a plug to fit the mains inlet socket (21). This allows connection of the unit to an appropriate electrical power supply. An output socket (22) provides a mains output for powering a chart recorder or other low current instrumentation. This outlet is not required when using the optional CEP-MKII-90IFD-USB data logger.

A Residual Current Device (23), sometimes called an RCCB, provides protection for personnel in the event of a short circuit to earth. Electrical circuits for the mains outlet socket and peristaltic feed pumps are protected individually from over-current by miniature circuit breakers (24).

Equipment Specifications

Overall Dimensions

Height - 0.6m

Width - 1.0m

Depth - 0.5m

Equipment Location

The CEP-MkII Stirred Tank Reactors in Series unit is designed for installation on a firm, level bench or table.

An area adjacent to the unit on the left hand side should be available for storing and mixing chemical reactants.

If intended for use with a computer, there should be enough space on the right hand side of the unit for the interface (CEX-306IFD) and the computer.

Environmental Conditions

This equipment has been designed for operation in the following environmental conditions. Operation outside of these conditions may result reduced performance, damage to the equipment or hazard to the operator.

a. Indoor use;

b. Altitude up to 2000m;

c. Temperature 5°C to 40°C;

d. Maximum relative humidity 80% for temperatures up to 31°C, decreasing linearly to 50% relative humidity at 40°C;

e. Mains supply voltage fluctuations up to ±10% of the nominal voltage;

f. Transient over-voltages typically present on the MAINS supply;

   Note: The normal level of transient over-voltages is impulse withstand (over-voltage) category II of IEC 60364-4-443;

g. Pollution degree 2.

   Normally only nonconductive pollution occurs.

   Temporary conductivity caused by condensation is to be expected.

   Typical of an office or laboratory environment.

Exercise E - Demonstration of the progress of a second order chemical reaction through three continuous stirred tank reactors connected in series

Theory

The reaction:

$$NaOH + H_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$$

Sodium Hydroxide + Ethyl Acetate → Sodium Acetate + Ethyl Alcohol

can be considered 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.

The reaction carried out in a Continuous Stirred Tank Reactor eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place.

The steady state conditions will vary depending on concentration of reagents, flowrate, volume of reactor and temperature of reaction.

Method

Make up 5.0 litre batches of 0.05M sodium hydroxide and 0.05M ethyl acetate.

IMPORTANT: It is essential when handling these chemicals to wear protective clothing, gloves and safety spectacles.

Remove the lids of the reagent vessels and carefully fill with the reagents to a level approximately 50mm from the top. Refit the lids.

Collection of conductivity data will be until a steady state condition is reached in the reactor and this takes approximately 45 minutes. It is advisable to set the data collection period to, say, 60 minutes.

Using the calibration graph for each of the feed pumps, set the pump speed controls to give 60 ml/min flowrate.

Set the agitator speed controller to 7.0.

Switch on both feed pumps and agitator motor, and instigate the data logger program (or begin taking readings if no computer is being used).

It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.

Interpretation of Results

Having used the Armfield data logger CEX-306IFD to record the conductivity of the contents of the reactors over the period of the reaction, the conductivity measurements are automatically translated into degree of conversion of the constituents.

This is achieved by filling reactors 1 and 2 with water until water overflows the standpipe in reactor 2. Now empty the contents using drain valve (25) into a measuring cylinder and divide the total volume by 2 (both reactors have identical operating volumes).

Reactor 3 is usually operated at fixed volume with liquid exiting via the standpipe. To check the volume, firstly disconnect the 'dead time' coil from the drain valve at the base of reactor 3. Fill reactor 3 with water until water overflows the standpipe. Measure the height of water in the reactor then empty the contents into a measuring cylinder by opening the drain valve (27). If using reactor 3 with variable volume (outflow via valve 2) to the 'dead time' coil) the actual volume can be determined by measuring the operating height of the liquid in the reactor and proportioning the volume from the previous measurements.

Chemical Reaction in Stirred Tank Reactors in Series

The Armfield CEP-MkII Stirred Tank Reactors in Series unit is designed to demonstrate the mechanism of a chemical reaction in series connected continuous stirred tank reactors as well as the effects of varying the process conditions such as stirring rate, feed rate etc.

The conductivity of the reacting solution in the reactors changes with the degree of conversion and this provides a convenient method for monitoring the progress of the reaction either manually or by computer.

The reaction chosen is the saponification of ethyl acetate by sodium hydroxide as it can be carried out under safe conditions of temperature and pressure and is well documented.

The experiments involve the collection and storage of conductivity data, the 50 way data output port on the console must be connected to the CEP-90IFD-USB data logger (IFD5 interface) and to the computer using a USB lead. Each of the three reactor vessels contains a conductivity probe and a probe is also positioned at the exit of the dead time coil.

This will enable data logging of all four conductivity values and pump / stirrer speeds at selected time intervals over a selected period of time.

Of course, if a computer is not available, the conductivity can be recorded manually at, say, half minute intervals by reading the value directly from the conductivity meter in the console and using the selector switch to select each probe in turn. However, when recording values manually (not using the CEP-90IFD-USB Data Logger) it will be necessary to convert all conductivity readings to readings of concentration by loading the readings into a suitable spreadsheet.

Although it may be possible to carry out demonstrations using other chemicals it is not advisable as the materials of construction of the reactors may not be compatible.

Before carrying out reactions involving any other reagents please refer to Armfield Ltd. for advice.

Exercise E - Demonstration of the progress of a second order chemical reaction through three continuous stirred tank reactors connected in series

Theory

The reaction:

$$NaOH + H_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$$

Sodium Hydroxide + Ethyl Acetate → Sodium Acetate + Ethyl Alcohol

can be considered 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.

The reaction carried out in a Continuous Stirred Tank Reactor eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place.

The steady state conditions will vary depending on concentration of reagents, flowrate, volume of reactor and temperature of reaction.

Method

Make up 5.0 litre batches of 0.05M sodium hydroxide and 0.05M ethyl acetate.

IMPORTANT: It is essential when handling these chemicals to wear protective clothing, gloves and safety spectacles.

Remove the lids of the reagent vessels and carefully fill with the reagents to a level approximately 50mm from the top. Refit the lids.

Collection of conductivity data will be until a steady state condition is reached in the reactor and this takes approximately 45 minutes. It is advisable to set the data collection period to, say, 60 minutes.

Using the calibration graph for each of the feed pumps, set the pump speed controls to give 60 ml/min flowrate.

Set the agitator speed controller to 7.0.

Switch on both feed pumps and agitator motor, and instigate the data logger program (or begin taking readings if no computer is being used).

It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.

Interpretation of Results

Having used the Armfield data logger CEX-306IFD to record the conductivity of the contents of the reactors over the period of the reaction, the conductivity measurements are automatically translated into degree of conversion of the constituents.

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Exercise E

Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst 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.

Comment upon the results obtained.

If readings have been obtained manually then the necessary calculations are best carried out using a spreadsheet such as EXCEL so that the results can be displayed in tabular and graphical form. On conclusion of the experiment the recorded data can be transferred into the spreadsheet. Enter the following known constants from the experiment using the Nomenclature. Ensure use of correct units.

F_a =

F_b =

a_μ =

b_μ =

c_μ =

T =

V =

Using the spreadsheet, calculate the values of a_o, b_o, c_∞, a_∞, $\Lambda_{c\infty}$, $\Lambda_{a\infty}$, $\Lambda_{a\infty}$, Λ_o and Λ_∞ from the following formulae:

$$a_0 = \frac{F_a}{F_a + F_b} . a_\mu$$

$$b_0 = \frac{F_b}{F_a + F_b} . b_\mu$$

$c_\infty = b_0$ for $b_0 < a_0$

$c_\infty = a_0$ for $b_0 \geq a_0$

$$\Lambda_{c\infty} = 0.070[1 + 0.0284(T - 294)c_\infty$$

$$\Lambda_{a_0} = 0.195[1 + 0.0184(T - 294)a_o$$

$\Lambda_0 = \Lambda_{ao}$ assumes $c_o = 0$

$a_\infty = 0$ for $a_o < b_o$

$a_\infty = a_o - b_o$ for $a_o \geq b_o$

$$\Lambda_{a\infty} = 0.195[1 + 0.0184(T - 294)]a_\infty \quad \text{if} \quad a_\infty \neq 0$$

$$\Lambda_\infty = \Lambda_{c\infty} + \Lambda_{a\infty}$$

For the values of each of the above, the spreadsheet can be used to calculate values of sodium hydroxide concentration (a_1) and sodium acetate concentration (c_1) and the degree of conversion *(X_a)* and *(X_c)* for each of the samples of conductivity taken over the period of the experiment.

These can be calculated and listed in columns (use spreadsheet COPY facility) alongside the readings of conductivity using the following equations:

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

$$c_1 = c_\infty\left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right], \quad \text{for} \quad c_0 = 0$$

$$X_a = \frac{a_0 - a_1}{a_0}$$

$$X_c = \frac{c_1}{c_\infty}, \quad \text{for} \quad c_0 - 0$$

To calculate the specific rate constant, k:

The overall mass balance at steady-state condition may be written as:

Input – Output ± Reaction = 0

i.e. for a reactant a in a reactor of volume V

$$\frac{d(Va_1)}{dt} = F.a_0 - F.a_1 - V.k.a_1^{\,2}$$

For the continuous reactor operating at steady state the volume may be assumed constant and

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

The steady-state concentration of NaOH in the reactor (a_1) may be used to calculate the specific rate constant (k).

Comment upon the results obtained.

Engineering Growth Connection

ISOTHERMAL BATCH REACTOR-
Peristaltic Pump System
(With Data Logging Facility)
(CRE-907RH)

Foreword

Welcome to the fast growing family of K.C. product owners. We appreciate your interest in us and thank you for buying our product.

You have chosen the finest quality product in the market which is produced using latest techniques and has underwent strict quality control tests. It is a product that we are proud to build and you are proud to own it.

Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.

However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.

This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.

All information, specifications and illustrations in this manual are those in effect at the time of printing. We reserve the right to change specifications or design at any time without notice.

Customer satisfaction is our primary concern. Feel Free to contact us for any assistance. So what are you waiting for, roll up your sleeves and let us get down to work!

K.C. Engineers Pvt. Ltd.

Important Information About This Manual

Reminder for Safety

Modification on Equipment:

This equipment should not be modified. Modification could affect its performance, safety or disturbance. In addition damage or performance problems resulting from modification may not be covered under warranties.

Precautions and Maintenance:

This is used to indicate the presence of a hazard that could cause minor or moderate personal injury or damage to your equipment. To avoid or reduce the risk, the procedures must be followed carefully.

Contents

ISOTHERMAL BATCH REACTOR (WITH DATA LOGGING FACILITY)

1. OBJECTIVE:

To study of a non-catalytic homogeneous reaction in an isothermal batch reactor.

2. AIM:

- To study the progress of a chemicals reaction and determine the kinetic parameters.
- To determine the effect of temperature on reaction rate constant.

3. INTRODUCTION:

A batch reactor is a closed system with no input and output streams. A batch reactor can operate under the following conditions :

- Isothermal (temperature of reaction mass remains constant).
- Perfectly mixed (composition of the reaction mixture is uniform through out).
- Constant volume (volume of the reaction mixture within the reactor remains constant, there is no appreciable change in the density of reaction mass).

4. THEORY:

REACTION:

$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

(A) (B) (C) (D)

The above reaction is second order reaction (n = 2)

For second order reaction

$$-r_A = \frac{-dC_A}{dt}$$

$$-r_A = KC_A^2 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where $-r_A$ is rate of disappearance of A, C_A is concentration of A at any time t, K is rate constant.

The performance equation for the batch reactor is:

$$t = -\int_{C_{Ao}}^{C_A} \frac{dC_A}{-r_A} \quad \text{..... (2)}$$

Where t is time of reaction, C_{Ao} is initial concentration of A, C_A is concentration of A at any time t.

Substitute the value of $-r_A$ in eq^n (2) from eq^n (1)

$$t = -\frac{1}{K}\int_{C_{Ao}}^{C_A} \frac{dC_A}{C_A{}^2} \quad \text{.....(3)}$$

On solving the above equation we get

$$t = \frac{1}{K}\left[\frac{1}{C_A}\right]_{C_{Ao}}^{C_A}$$

$$t = \frac{1}{K}\left[\frac{C_{Ao} - C_A}{C_A C_{Ao}}\right]$$

Rate constant, $K = \dfrac{C_{Ao} - C_A}{t\, C_{Ao} C_A}$

Degree of conversion, $X_A = \dfrac{(C_{Ao} - C_A)}{C_{Ao}}$

Also, a plot of t vs. $X_A/ (1-X_A)$ shall yield a straight line for an assumed second order reaction with slope = $1/(KC_{Ao})$. From this slope rate constant, K can be calculated.

Effect of temperature on reaction rate constant is expressed in terms of

Arrhenius'law

$$K = K_o\, e^{-E/RT}$$

K_o is the frequency factor and E is called the activation energy of reaction, R is real gas constant, and T is reaction temperature.

A plot of ln K vs 1/T on semilog paper yields a straight line with slope = -E/R

From this slope, activation energy, E can be calculated.

5. DESCRIPTION:

The set up consists of a reactor made up of Borosilicate glass fitted in a constant temperature water bath. One stirrer is fitted for mixing the reactants in reactor and other is fitted in water bath to keep the uniform temperature throughout in the bath. The temperature of bath can be maintained from ambient to 80°C with the help of digital temperature indicator cum controller. Product from the reactor is analyzed by electronic sensor.

The present set-up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed for unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

6. UTILITIES REQUIRED:

6.1 Electricity Supply: Single Phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.

6.2 Water supply (Initial fill).

6.3 Floor Drain Required.

6.4 Computer system : Pentium IV with DVD drive, Windows 7,MS-office 7 version pre loaded, One USB slot required in PC for DATA ACQUISITION CARD.

6.5 Laboratory glass ware:-

Beaker (500 ml)	:	02 Nos.

6.6 Chemicals:-

Distilled water	:	02 Lit
NaOH Pellets	:	10 gm
Ethyl acetate	:	15 ml

7. INSTALLATION PROCEDURE OF SOFTWARE:

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as **"NI-Max"**. Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder "Isothermal Batch Reactor." and then double click on "Setup.exe". Follow the instructions and do the installation.

Go to desktop and double click on "NI-Max". A window opens and looks at left side. Follow the path as "My System" → "Devices and Interfaces" → Click on USB-6008: "Dev1", Right click and rename it as **"049"** and press "enter". Now, it appears as USB-6008: "049" .Close the window. After completion of installation "restart" the computer system.

8. METHOD OF OPERATING SOFTWARE:

Open the EXE of selected condenser by clicking it from program Menu of main Menu bar. Enter Password "kce" for the software and press "enter" or click "OK". Click the "Interface" button to open interface of the software. In the entire software, a "NEXT" button is provided, at left front, which enables to go to next step. Another button named as "BACK" is present. This facilitates to retrieve last step taken. An "EXIT" button, at right bottom end, is present there. It may be used to exit at any session and finally, stopping the software.

Note: In the absence of external hardware (USB-6008), the interfacing section may not able to work.

9. EXPERIMENTAL PROCEDURE:

9.1 STARTING PROCEDURE:

9.1.1 Prepare 1L of N/10 sodium hydroxide solution by dissolving 4 gm of sodium hydroxide in 1L of distilled water.

9.1.2 Prepare 1L of N/10 ethyl acetate solution by mixing 8.8 gm (or 9.8 ml) of ethyl acetate in 1L of distilled water.

9.1.3 Put 400 ml sodium hydroxide and ethyl acetate in two 500 ml beaker.

9.1.4 Close all the valves.

9.1.5 Fill water in the water bath.

9.1.6 Fill ethyl acetate solution in the reactor.

9.1.7 Ensure that ON/OFF switches given on the panel are at OFF position.

9.1.8 Set the reaction temperature (ambient to (50-70) ^{o}C) by increment, decrement and set button of DTC.

9.1.9 Connect electric supply to the set-up.

9.1.10 Start the heater and stirrer of the bath and wait till constant temperature is attained.

9.1.11 Fill sodium hydroxide into the reactor.

9.1.12 Start motor and stirrer of the reactor.

9.1.13 At regular intervals of 3-5 min record the conductivity of reactants in computer interface.

9.1.14 Record the reaction temperature.

9.1.15 Repeat the experiment for different reaction temperature.

9.2 Closing Procedure:

9.2.1 When experiment is over switch OFF the motor and stirrer of reactor.

9.2.2 Switch OFF the heater and stirrer of the water bath.

9.2.3 Drain the reactor and water bath by open their drain valves respectively.

10. Observation & Calculation:

10.1 Data:

Initial concentration of NaOH in feed mixture C_{A0} = 0.1 mole/ltrs

10.2 Observation Table:

S. No.	T (°C)	t (min)	C_A(mole/L)

10.3 Calculations:

$$X_A = \frac{C_{Ao} - C_A}{C_{Ao}}$$

$$K = \frac{C_{Ao} - C_A}{tC_AC_{Ao}} \text{ (L/mole min)}$$

Calculation Table: 1

S. No.	t (min)	X_A	K (L/mole min)

11. Nomenclature:

Nom	Column Heading	Units	Type
C_A	Concentration of unreacted NaOH in the reactor	mole/L	Measured
C_{Ao}	Initial concentration of NaOH in the feed mixture	mole/L	Calculated
K	Rate constant for individual run	L/ mole min	Calculated
N_{NaOH}	Normality of NaOH in feed solution	g eq /L	Given
T	Reaction temperature	°C	Given
t	Time	min	Measured
X_A	Degree of conversion	*	Calculated

* Symbols represent unitless quantity.

12. Precaution & Maintenance Instructions:

12.1 Measure the exact volume of water and weigh the chemicals.

12.2 Always use distilled water, good quality chemicals and standard solution for titration.

12.3 Keep close all the drain valves while filling the reactant in reactor.

12.4 Handle the chemicals carefully.

12.5 Don't ON heater switch before filling water in the water bath.

13. Troubleshooting:

13.1 If there is any leakage tight that part or fix it again after wrapping teflon tape.

13.2 If D.T.C do not display on display board it means sensor connection is not proper (tight computer jacket).

13.3 If switch of the heater is ON but temperature can't rise but panel LED is ON it means bath heater had burned replace that.

14. References:

14.1 Levenspiel, Octave (2001). *Chemical Reaction Engineering*. 3rd Ed. NY: John Wiley & Sons. pp 27-29, 44, 92, 111.

14.2 Fogler H. Scoot (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. pp 148-150, 152-155.

ISOTHERMAL CSTR- Peristaltic Pump System

(With Data Logging Facility)

(CRE-902 RHb)

ISOTHERMAL CONTINUOUS STIRRED TANK REACTOR

1. OBJECTIVE:

To study of a non-catalytic homogeneous reaction in a CSTR under isothermal conditions.

2. AIM:

2.1 To determine the reaction rate constant, (K) for *saponification of ethylacetate with sodium hydroxide at a fixed temperature.*

2.2 To study the effect of temperature on the reaction rate constant, (K) and determine the activation energy, (E) for this reaction.

3. INTRODUCTION:

In an ideal CSTR (that is an ideal steady state flow reactor) the contents in the reactor are well mixed and have uniform composition throughout. Thus the exit stream has the same composition as the fluid within the reactor. These types of reactors are also known as MIXED FLOW REACTOR.

4. THEORY:

REACTION:

$NaOH + CH_3COOC_2H_5 \rightarrow CH_3COONa + C_2H_5OH$

(A) (B) (C) (D)

The above reaction is second order reaction (n = 2)

For second order reaction

$$-r_A = \frac{-dC_A}{dt}$$

$$-r_A = KC_A^2 \quad \text{.......................................(1)}$$

Where $-r_A$ is rate of disappearance of A, C_A is concentration of A at any time t, K is rate constant.

The performance equation for the mixed flow reactor at steady state is:

$$\tau = \frac{V_R}{V_o} \quad \text{.....(2)}$$

In terms of concentration we can write

$$\tau = \frac{C_{Ao} - C_A}{-r_A} \quad \text{.....(3)}$$

Where τ is residence time, V_R is volume of reactor, V_o is volumetric flow rates reactants A and B, C_{Ao} is initial concentration of A.

Substitute the value of $-r_A$ in eq^n (3) from eq^n (1)

We get $\tau = \frac{C_{Ao} - C_A}{kC_A^2}$

Rate constant, $K = \frac{C_{Ao} - C_A}{\tau C_A^2}$

Degree of conversion, $X_A = \frac{(C_{Ao} - C_A)}{C_{Ao}}$

Also, a plot of t vs. $X_A/ (1-X_A)^2$ shall yield a straight line for an assumed second orc reaction with slope=KC_{Ao}. From this slope rate constant, K can be calculated.

Effect of temperature on reaction rate constant is expressed in terms of Arrhenius law:

$$K = K_o e^{-E/RT}$$

K_o is the frequency factor and E is called the activation energy of reaction, R is real g constant, and T is reaction temperature.

A plot of $\ln K$ vs. 1/T on semilog paper yields a straight line with slope = -E/R

From this slope, activation energy, E can be calculated.

5. DESCRIPTION:

The set up consists of two feed tanks through which two reactants are fed to the react Two channel peristaltic pump is provided to measure the individual flow rate chemicals and circulation of feed. The flow rate can be adjusted by operating the spe

variation of respective pump. The CSTR is fitted with stirrer for proper mixing. Constant temperature water bath arrangement is provided to conduct the experiment at various temperatures. Product from the reactor is analyzed by electronic sensor.

The present set up has a facility to interface the system with computer which enables to log the experimental data using computer. The educational software and data-logging package has been developed per unit. This software is capable to tabulate the sample readings according to the requirement of experiment under study and results obtained can be compared.

6. UTILITIES REQUIRED:

6.1 Electricity supply: Single phase, 220 V AC, 50 Hz, 5-15 Amp combined socket with earth connection.

6.2 Water supply (Initial Fill).

6.3 Floor drain required.

6.4 Computer system : Pentium IV with DVD drive, Windows 7,MS-office 7 version pre loaded, One USB slot required in PC for DATA ACQUISITION CARD.

6.5 Laboratory glass ware:-

Measuring cylinder (1000 ml)	:	02 Nos.

6.6 Chemicals:-

Distilled water	:	40 Lit.
NaOH Pellets	:	100 gm
Ethyl acetate	:	200 ml

7. INSTALLATION PROCEDURE OF SOFTWARE:

Two DVDs are provided with the equipment. One DVD is of Device Driver and other DVD of Operating Software. First, place the device driver DVD in DVD-ROM and wait for 5 to 10 seconds. A blue-colored window appears on the screen. Install the software as per directions. When installation is finished, a shortcut would be created on the desktop showing name as **"NI-Max"**. Restart the computer system. Fix USB Card in empty USB slots. This ends the completion of loading of DAQ Card Driver. Now, take Operating Software DVD and place in DVD-ROM. Open DVD and open folder "Isothermal

C.S.T.R." and then double click on "Setup.exe". Follow the instructions and do installation.

Go to desktop and double click on "NI-Max". A window opens and looks at left s Follow the path as "My System" → "Devices and Interfaces" → Click on USB-6 "Dev1", Right click and rename it as "048" and press "enter". Now, it appears as U 6008: "048" .Close the window. After completion of installation "restart" the comp system.

8. METHOD OF OPERATING SOFTWARE:

Open the EXE of selected condenser by clicking it from program Menu of main M bar. Enter Password "kce" for the software and press "enter" or click "OK". Click "Interface" button to open interface of the software. In the entire software, a "NE button is provided, at left front, which enables to go to next step. Another button nar as "BACK" is present. This facilitates to retrieve last step taken. An "EXIT" button right bottom end, is present there. It may be used to exit at any session and fin stopping the software.

Note: In the absence of external hardware (USB-6008), the interfacing section n not able to work.

9. EXPERIMENTAL PROCEDURE:

density of NaOH = 2.13 gm/cms

9.1 STARTING PROCEDURE:

9.1.1 Prepare 20L of N/10 NaOH solution by dissolving 80 gm of sod hydroxide in 20L of distilled water.

9.1.2 Prepare 20L of N/10 ethyl acetate solution by mixing 176 gm (or 196 of ethyl acetate in 20L of distilled water.

9.1.3 Close all the valves.

9.1.4 Open the lid of feed tank A, fill sodium hydroxide solution in feed tank A

9.1.5 Close the lid of feed tank A.

9.1.6 Open the lid of feed B, fill ethyl acetate solution in feed tank B.

9.1.7 Close the lid of feed tank B.

9.1.8 Fill the water in water bath.

9.1.9 Ensure that ON/OFF switches given on the panel are at OFF position.

9.1.10 Connect electric supply to the set-up.

9.1.11 Set the reaction temperature (ambient to (50-70) °C) by increment, decrement and set button of DTC.

9.1.12 Start the heater and stirrer of the bath and wait till constant temperature is attained.

9.1.13 Switch on the peristaltic pump.

9.1.14 By operating Auto/Manual switch and speed variation pot sodium hydroxide and ethyl acetate are passed into the reactor.

9.1.15 Start the stirrer of the reactor.

9.1.16 After about 10 min or time equal to the residence time of the reactor (which ever is greater) record the conductivity of reactants in computer interface.

9.1.17 Note down the reaction temperature.

9.1.18 Repeat the experiment for different RPM of peristaltic pump.

9.1.19 Repeat the experiment for different reaction temperature.

9.2 Closing Procedure:

9.2.1 When experiment is over stop the flow of feed by switching pump off.

9.2.2 Switch OFF the heater and stirrer of the water bath.

9.2.3 Switch OFF the stirrer of the reactor.

9.2.4 Switch OFF the peristaltic pump.

9.2.5 Drain the reactor, feed tanks and water bath by open their drain valves respectively.

10. Observation & Calculation:

10.1 Data:

Working volume of reactor V_R	= 2.2 Lit ✓
Initial Concentration of NaOH in feed solution C_{A0}	= 0.05 g eq/L

S.No.	T (°C)	V_A,LPH	V_B,LPH	C_A (mol/liter)

10.3 Calculations:

$$X_A = \frac{(C_{Ao} - C_A)}{C_{Ao}}$$

$$C_{A0} = \frac{N_{A0}}{2} = \frac{0.5}{2} \rightarrow 0.25$$

$$t = \frac{V_R * 60}{(V_A + V_B)} \text{ (min)}$$

$$K = \frac{C_{Ao} - C_A}{\tau C_A^2} \text{ (L/mole min)}$$

Calculation Table: 1				
S.No.	T (^{0}C)	X_A	t (min)	K (L/mol mi

Q_T = RPM x 2.5 x 0.06 LPH

11. NOMENCLATURE:

Nom	Column Heading	Units	Type
C_A	Concentration of unreacted NaOH in the reactor	mole/L	Measured
C_{Ao}	Initial concentration of NaOH in the feed mixture	mole/L	Calculated
K	Rate constant for individual run	L/ mole min	Calculated
T	Reaction temperature	°C	Measured
V_R	Working volume of reactor	Lit	Given
X_A	Degree of conversion	*	Calculated
t	Time of reaction	min	Measured

* Symbols are unitless

12. PRECAUTION & MAINTENANCE INSTRUCTIONS:

12.1 Measure the exact volume of water and weight of chemicals.

12.2 Always use distilled water, good quality chemicals and standard solution for titration.

12.3 Keep close all the drain valves while filling the reactant in feed tanks.

12.4 Flow should not be disturbed during the experiment.

12.5 Handle the chemicals carefully.

12.6 Do not ON the switch of the heater switch before filling water in the water bath.

13. TROUBLESHOOTING:

13.1 If there is any leakage tight that part or fix it again after wrapping teflon tape.

13.2 If D.T.C do not display temperature on display board it means sensor connection is not proper (tight computer jacket).

13.3 If switch of the heater is ON, but temperature can't rise but panel LED is ON it means heater had burned replace it.

14. REFERENCES:

14.1 Levenspiel, Octave (2001). *Chemical Reaction Engineering*. 3rd Ed. NY: John Wiley & Sons. pp 27-29, 92, 111.

RTD STUDIES IN CSTR

Peristaltic Pump System

(CRE-914 RHb)

Foreword

Welcome to the fast growing family of K.C. product owners. We appreciate your interest in us and thank you for buying our product.

You have chosen the finest quality product in the market which is produced using latest techniques and has underwent strict quality control tests. It is a product that we are proud to build and you are proud to own it.

Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.

However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.

This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.

All information, specifications and illustrations in this manual are those in effect at the time of printing. We reserve the right to change specifications or design at any time without notice.

Customer satisfaction is our primary concern. Feel Free to contact us for any assistance. So what are you waiting for, roll up your sleeves and let us get down to work!

K.C. Engineers Pvt. Ltd.

Important Information About This Manual

Reminder for Safety

Modification on Equipment:

This equipment should not be modified. Modification could affect its performance, safety or disturbance. In addition damage or performance problems resulting from modification may not be covered under warranties.

Precautions and Maintenance:

This is used to indicate the presence of a hazard that could cause minor or moderate personal injury or damage to your equipment. To avoid or reduce the risk, the procedures must be followed carefully.

Contents

RTD STUDIES IN CSTR

1. OBJECTIVE:

RTD studies in a CSTR.

2. AIM:

2.1 To plot the RTD curve for a CSTR using a pulse tracer.

2.2 To determine the dispersion number.

3. INTRODUCTION:

Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation form ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel. To predict the exact behavior of a vessel as a chemical reactor, RTD or stimulus response technique is used.

4. THEORY:

The exit age distribution function of fluid leaving a vessel or RTD of fluid in a vessel is called the E-CURVE. The normalized curve is such that

$$\int_0^\infty E dt = 1 \qquad \text{----------} \quad (1)$$

In stimulus-response experimentation the system is perturbed and then observes how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.

The concentration - time curve for pulse signal at the vessel outlet is called the C-CURVE. Considering steady-state flow of fluid through a closed vessel:

$$C = E \qquad \text{----------} \quad (2)$$

The mean age of the exit stream or mean residence time is:

$$\tau = \int_0^\infty tEdt = \sum tE\Delta t \qquad \text{----------} \quad (3)$$

$$\tau = \frac{\sum (t_i C_i)}{\sum C_i} \qquad \text{----------} \quad (4)$$

And the variance of the E or C distribution is

$$\sigma_t^2 = \int_0^\infty t^2 E dt - \tau^2 = \sum t^2 E \Delta t - \tau^2 \qquad \text{----------} \quad (5)$$

$$\sigma_t^2 = \frac{\sum (t_i^2 C_i)}{\sum C_i} - \left[\frac{\sum (t_i C_i)}{\sum C_i} \right]^2 \qquad \text{----------} \quad (6)$$

Variance, or a measure of the spread of the curve at time θ.

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2} \qquad \text{----------} \quad (7)$$

Models are useful for representing flow in real vessels, for scale up, and for diagnosing poor flow. We have different kind of models depending on whether

Flow is close to plug, mixed, or somewhere in between. For small deviations from plug flow dispersion model is used. Suppose an ideal pulse of tracer is introduced

into the fluid entering a vessel. The pulse spreads as it passes through the vessel,

and to characterize the spreading this model, we assume a diffusion like process superimposed on plug flow. We call this dispersion, the dispersion coefficient D represents the spreading process. $\left(\frac{D}{uL} \right)$ is the dimensionless group characterizing

the spread in the whole vessel.

For closed vessel

$$\sigma_\theta^2 = 2\left(\frac{D}{uL} \right) - 2\left(\frac{D}{uL} \right)^2 \left[1 - e^{-\frac{uL}{D}} \right] \qquad \text{----------} \quad (8)$$

Ignoring the second term of the above equation we get.

$$\frac{D}{uL} = \frac{\sigma_\theta^2}{2} \qquad \text{----------} \quad (9)$$

Defining the reduced time as:

$$\theta = \frac{t}{\tau}$$ ---------- (10)

Exit age distribution at time i

$$E_i = \frac{C_i}{\sum C_i \Delta t}$$ ---------- (11)

Exit age distribution at time θ

$$E_\theta = \tau \times E_i$$ ---------- (12)

Plot a graph between θ vs E_θ.

5. DESCRIPTION:

The setup consists of one feed tank through which water is fed to the borosilicate reactor. A peristaltic pump is provided to measure flow and circulation of feed. The flow rate can be adjusted by operating the speed variation knob of pump. The continuous stirred tank reactor made of borosilicate glass is provided for understanding the RTD characteristics. A pipette is used for dozing the tracer into the C.S.T.R.

6. UTILITIES REQUIRED:

6.1 Electricity supply: Single phase, 220 V AC, 50 Hz, 5-15 Amp. Combined socket with earth connection.

6.2 Water supply (Initial fill)

6.3 Floor drain required.

6.4 Laboratory glassware required:-

Burette (50 ml)	:	01 No.
Conical flasks (250 ml)	:	01 No.
Pipette (20 ml)	:	01 No.
Measuring cylinder (250 ml)	:	01 No.

6.5 Chemicals:-

N/10 NaOH	:	200 ml
Concentrated H_2SO_4	:	100 ml

Phenolphthalein indicator : Few drops

7. EXPERIMENTAL PROCEDURE:

7.1 STARTING PROCEDURE:

7.1.1 Close all the valves.

7.1.2 Open the lid of feed tank and fill the feed tank with water.

7.1.3 Close the lid of feed tank.

7.1.4 Connect electric supply to the set up.

7.1.5 Switch ON the mains ON/OFF switch.

7.1.6 Switch on the peristaltic pump.

7.1.1 By operating Auto/Manual switch and speed variation pot water is passed into the reactor.

7.1.7 Fill N/10 sodium hydroxide in burette.

7.1.8 Switch ON the stirrer and wait till the water comes out from the outlet.

7.1.9 Fill the concentrated H_2SO_4 (10 ml or 20 ml) in the pipette.

7.1.10 Input concentrated H_2SO_4 into the system with the help of pipette.

7.1.11 At regular time intervals (say 30 sec for high water rate, 1 min for low water rate), collect 20 ml sample from the outlet in measuring cylinder.

7.1.12 Transfer the sample solution in conical flask.

7.1.13 Titrate the sample solution, using phenolphthalein as an indicator against N/10 sodium hydroxide (add NaOH from burette).

7.1.14 Repeat the experiment for different RPM (before changing the flow rate, drain the reactor first).

7.2 CLOSING PROCEDURE:

7.2.1 When experiment is over stop the flow of feed by closing the pump.

7.2.1 Switch OFF the stirrer.

7.2.2 Switch OFF the power supply.

7.2.3 Drain the reactor and feed tank by open their drain valves respectively.

8. OBSERVATION & CALCULATION:

8.1 DATA:

Working volume of reactor V_R	= ____ Lit
Volume of sample V_2	= 20 ml
Normality of NaOH used for titration N_1	= 0.1 g eq/L

8.2 OBSERVATION TABLE:

S.No.	t_i (min)	N (RPM)	V_1 (ml)

8.3 CALCULATIONS:

$$N_2 = \frac{V_1 N_1}{V_2} \text{ (g eq/L)}$$

$$C_i = \frac{N_2}{2} \text{ (mole/L)}$$

$$V_\circ = N \times 0.15 \text{ (LPH)}$$

$$\tau_t = \frac{V_R * 60}{V_o} \text{ (min)}$$

$$\Delta t = t_i - t_{i-1} \text{ (min)}$$

CALCULATION TABLE:1

S.No.	t_i (min)	C_i (mol/lit)	$t_i C_i$	$t_i^2 C_i$	Δt (min)	$C_i \Delta t$
		$\sum C_i$	$\Sigma t_i C_i$	$\sum t_i^2 C_i$		

$$\tau = \frac{\sum t_i C_i}{\sum C_i} \text{ (min)}$$

$$\sigma_t^2 = \left(\frac{\sum t_i^2 C_i}{\sum C_i} \right) - \tau^2$$

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2}$$

$$\frac{D}{uL} = \frac{\sigma_\theta^2}{2}$$

$$E_i = \frac{C_i}{\sum C_i \Delta t}$$

$$\theta = \frac{t}{\tau}$$

$$E_\theta = \tau \times E_i$$

CALCULATION TABLE:2

θ	E_i	E_θ

Plot a graph between θ vs E_θ.

9. NOMENCLATURE:

Nom	Column Heading	Units	Type
C_i	Concentration of H_2SO_4 in sample	mole/L	Calculated
D/uL	Dispersion number	*	Calculated
E_i	Exit age distribution at time i	*	Calculated
E_θ	Exit age distribution at time θ	*	Calculated
N_1	Normality of NaOH used for titration	g eq/L	Given
N_2	Normality of H_2SO_4 in sample solution	g eq/L	Calculated
t_i	Time	min	Measured

V_1	Volume of NaOH used for titration	ml	Measured
V_2	Volume of sample	ml	Given
V_o	Volumetric flow rate	LPH	Calculated
V_R	Working volume of reactor	Lit	Given
τ	Experimental mean residence time	min	Calculated
τ_t	Theoretical mean residence time	min	Calculated
θ	Reduced time	*	Calculated
σ_θ	Variance at time θ	*	Calculated
σ_t	Variance at time t	min^2	Calculated
Δt	Average time difference	min	Calculated
N	Speed of peristaltic pump	RPM	Measured

* Symbols are unitless

10. PRECAUTION & MAINTENANCE INSTRUCTIONS:

10.1 Measure the exact volume of water and weight of chemicals.

10.2 Always use distilled water, good quality chemicals and standard solution for titration.

10.3 Keep close all the drain valves while filling the reactant in feed tanks.

10.4 Flow should not be disturbed during the experiment.

10.5 Handle the chemicals carefully.

11. TROUBLESHOOTING:

11.1 If there is any leakage tight that part or fix it again after wrapping Teflon tape.

12. REFERENCES:

12.1 Levenspiel, Octave (2001). *Chemical Reaction Engineering*. 3rd Ed. NY: John Wiley & Sons. pp 293-294, 299-301, 305.

12.2 Fogler H. Scoot (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. pp 871-873, 879, 887-888.

Engineering Growth Connection

RTD STUDIES IN PFR

(STRAIGHT TUBE TYPE–Peristaltic Pump System)

CRE-(904RHb)

Foreword

Welcome to the fast growing family of K.C. product owners. We appreciate your interest in us and thank you for buying our product.

You have chosen the finest quality product in the market which is produced by using latest techniques and has underwent strict quality control tests. It is a product that we are proud to build and you are proud to own it.

Our products are easy to understand and operate. They are excellent for students who are trying to gain practical knowledge through experiments.

However your comfort and safety are important to us, so we want you have an understanding of proper procedure to use the equipment. For the purpose, we urge you to read and follow the step-by-step operating instructions and safety precautions in this manual. It will ensure that your favourite product delivers reliable, superior performance year after year.

This manual includes information for all options available on this model. Therefore, you may find some information that does not apply to your equipment.

All information, specifications and illustrations in this manual are those in effect at the time of printing. K.C. reserves the right to change specifications or design at any time without notice.

Customer satisfaction is our primary concern. Feel Free to contact us for any assistance. So what are you waiting for, roll up your sleeves and let us get down to work!

K.C. Engineers Pvt. Ltd.

Important Information About This Manual

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This is used to indicate the presence of a hazard that could cause minor or moderate personal injury or damage to your equipment. To avoid or reduce the risk, the procedures must be followed carefully.

Contents

RTD STUDIES IN PFR

(STRAIGHT TUBE TYPE)

1. OBJECTIVE:

RTD studies in a Plug flow reactor.

2. AIM:

2.1 To plot the RTD curve for a PFR using a pulse tracer.

2.2 To determine the dispersion number.

3. INTRODUCTION:

In plug flow, the concentration of reactant decreases progressively through the system and in mixed flow the concentration drops immediately to a low value. Because of this fact, a plug flow reactor is more efficient than the mixed flow reactors for reaction whose rates increases with reactants condition.

Real reactors do not satisfy the idealized flow patterns, back mix flow or plug flow deviation from ideality can be due to channeling of fluid through the vessel, recycling of fluid within the vessel or due to the presence of stagnant region or pockets of fluid in the vessel.

To predict the exact behavior of a vessel as a chemical reactor, RTD or stimulus response technique is used.

4. THEORY:

The exit age distribution function of fluid leaving a vessel or RTD of fluid in a vessel is called the E-CURVE. The normalized curve is such that

$$\int_0^\infty E dt = 1 \qquad \text{----------} \quad (1)$$

In stimulus-response experimentation the system is perturbed and then observes how the system reacts or responds to this stimulus. The analysis of the response gives the desired information. A pulse tracer input signal could be used as a stimulus.

The concentration - time curve for pulse signal at the vessel outlet is called the C-CURVE. Considering steady-state flow of fluid through a vessel:

C = E ---------- (2)

The mean age of the exit stream or mean residence time is:

$$\tau = \int_0^\infty tEdt = \sum tE\Delta t \quad \text{---------- (3)}$$

$$\tau = \frac{\sum (t_i\, C_i)}{\sum C_i} \quad \text{---------- (4)}$$

And the variance of the E or C distribution is

$$\sigma_t^2 = \int_0^\infty t^2 Edt - \tau^2 = \sum t^2 E\Delta t - \tau^2 \quad \text{---------- (5)}$$

$$\sigma_t^2 = \frac{\sum (t_i^2\, C_i)}{\sum C_i} - \left[\frac{\sum (t_i\, C_i)}{\sum C_i}\right]^2 \quad \text{---------- (6)}$$

Variance, or a measure of the spread of the curve at time θ.

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2} \quad \text{---------- (7)}$$

Models are useful for representing flow in real vessels, for scale up, and for diagnosing poor flow. We have different kind of models depending on whether

flow is close to plug, mixed, or somewhere in between. For small deviations from plug flow dispersion model is used. Suppose an ideal pulse of tracer is introduced

into the fluid entering a vessel. The pulse spreads as it passes through the vessel,

and to characterize the spreading this model, we assume a diffusion like process superimposed on plug flow. we call this dispersion, the dispersion coefficient D represents the spreading process. $\left(\frac{D}{uL}\right)$ is the dimensionless group characterizing

the spread in the whole vessel.

For open vessel

$$\sigma_\theta^2 = 2\left(\frac{D}{uL}\right) - 8\left(\frac{D}{uL}\right)^2 \qquad \text{---------- (8)}$$

Solving the above equation we get.

$$\frac{D}{uL} = \frac{-1+\sqrt{1+8\sigma_\theta^2}}{8} \qquad \text{---------- (9)}$$

Defining the reduced time as:

$$\theta = \frac{t}{\tau} \qquad \text{---------- (10)}$$

Exit age distribution at time i

$$E_i = \frac{C_i}{\sum C_i \Delta t} \qquad \text{---------- (11)}$$

Exit age distribution at time θ

$$E_\theta = \tau \times E_i \qquad \text{---------- (12)}$$

Plot a graph between θ vs E_θ.

5. DESCRIPTION:

The set up consists of feed tank through which reactant, fed to the reactor.Peristaltic pump is provided to measure the flow and circulation of feed. The flow rate can be adjusted by operating the speed variation knob of pump. The concentration of feed and the product coming out from the reactor are analyzed by chemical titration.

For understanding the RTD characteristics, a special arrangement to inject tracer into the reactor, using a syringe is provided.

6. UTILITIES REQUIRED:

6.1 Water supply (Initial fill)

6.2 Floor drain required.

6.3 Laboratory glassware required:-

Burette (50 ml)	:	01 No.
Conical flasks (250 ml)	:	01 No.
Syringe (20 ml)	:	01 No.

Measuring cylinder (250 ml)	:	01 No.

6.4 Chemicals:-

N/10 NaOH	:	200 ml
Concentrated H_2SO_4	:	100 ml
Phenolphthalein indicator	:	Few drops

7. EXPERIMENTAL PROCEDURE:

7.1 STARTING PROCEDURE:

7.1.1 Close all the valves.

7.1.2 Open the lid of feed tank and fill the feed tank with water.

7.1.3 Connect electric supply to the set up.

7.1.4 Switch ON the mains ON/OFF switch.

7.1.5 Switch on the peristaltic pump.

7.1.6 By operating Manual switch and speed variation knob water flows thru the reactor.

7.1.7 Fill N/10 sodium hydroxide in burette. .

7.1.8 Fill the concentrated H_2SO_4 (10 ml or 20 ml) in the syringe.

7.1.9 Place the syringe at the tracer inlet.

7.1.10 Wait till the water comes out from the outlet.

7.1.11 Push the syringe into the system.

7.1.12 At regular time intervals (say 30 sec for high water rate, 1 min for low water rate), collect 20 ml sample from the outlet, in measuring cylinder.

7.1.13 Transfer the sample solution in conical flask.

7.1.14 Titrate the sample solution, using phenolphthalein as an indicator against N/10 sodium hydroxide (add NaOH from burette).

7.1.15 Repeat the experiment for different RPM (before changing the flow rate, drain the reactor first).

7.2 Closing Procedure:

7.2.1 When experiment is over stop the flow of water by closing the pump.

7.2.2 Switch OFF the power supply.

7.2.3 Drain the feed tank and reactor by open their drain valves respectively.

8. Observation & Calculation:

8.1 Data:

Working volume of reactor V_R	= ____ Lit
Volume of sample V_2	= 20 ml
Normality of NaOH used for titration N_1	= 0.1 geq/L

8.2 Observation Table:

S.No.	t_i (min)	V_1 (ml)	N (RPM)

8.3 Calculations:

$$N_2 = \frac{V_1 N_1}{V_2} \text{ (g eq/L)}$$

$$C_i = \frac{N_2}{2} \text{ (mole/L)}$$

$$V_o = N \times 0.15 \text{ (LPH)}$$

$$\tau_t = \frac{V_R * 60}{V_o} \text{ (min)}$$

$$\Delta t = t_i - t_{i-1} \text{ (min)}$$

CALCULATION TABLE: 1						
S.No.	**t_i (min)**	**C_i (mol/lit)**	**$t_i C_i$**	**$t_i^2 C_i$**	**Δt (min)**	**$C_i \Delta t$**
		$\sum C_i$	$\sum t_i C_i$	$\sum t_i^2 C_i$		

$$\tau = \frac{\sum t_i C_i}{\sum C_i} \text{ (min)}$$

$$\sigma_t^2 = \left(\frac{\sum t_i^2 C_i}{\sum C_i} \right) - \tau^2$$

$$\sigma_\theta^2 = \frac{\sigma_t^2}{\tau^2}$$

$$\frac{D}{uL} = \frac{-1 + \sqrt{1 + 8\sigma_\theta^2}}{8}$$

$$E_i = \frac{C_i}{\sum C_i \Delta t}$$

$$\theta = \frac{t}{\tau}$$

$$E_\theta = \tau \times E_i$$

CALCULATION TABLE: 2		
θ	**E_i**	**E_θ**

Plot a graph between θ vs E_θ.

9. NOMENCLATURE:

Nom	Column Heading	Units	Type
C_i	Concentration of H_2SO_4 in sample	mole/L	Calculated
D/uL	Dispersion number	*	Calculated
E_i	Exit age distribution at time i	*	Calculated
E_θ	Exit age distribution at time θ	*	Calculated
N_1	Normality of NaOH used for titration	g eq/L	Given
N_2	Normality of H_2SO_4 in sample solution	g eq/L	Calculated
t_i	Time	min	Measured
V_1	Volume of NaOH used for tritation	ml	Measured
V_2	Volume of sample	ml	Given
V_o	Volumetric flow rate	LPH	Calculated
V_R	Working volume of reactor	Lit	Given
τ	Experimental mean residence time	min	Calculated
τ_t	Theoretical mean residence time	min	Calculated
θ	Reduced time	*	Calculated
σ_θ	Variance at time θ	*	Calculated
σ_t	Variance at time t	min	Calculated
N	Speed of peristaltic pump	RPM	Measured
Δt	Average time difference	min	Calculated

* Symbols are unitless quantity.

10. PRECAUTION & MAINTENANCE INSTRUCTIONS:

10.1 Always use distilled water, good quality chemicals and standard solution for titration.

10.2 Keep close all the drain valves while filling water in the feed tank.

10.3 Flow should not be disturbed during the experiment.

10.4 Handle the chemicals carefully.

11. TROUBLESHOOTING:

11.1 If there is any leakage tight that part or fix that again after wrapping teflon tape.

12. REFERENCES:

12.1 Levenspiel, Octave (2001). *Chemical Reaction Engineering*. 3rd Ed. NY: John Wiley & Sons. pp 267, 293-294, 297, 299-301, 305.

12.2 Fogler H. Scoot (2008). *Elements of Chemical Reaction Engineering*. 4th Ed. ND: Prentice-Hall of India Pvt. Ltd. pp 871-873, 879, 885-886.

Equipment Diagrams

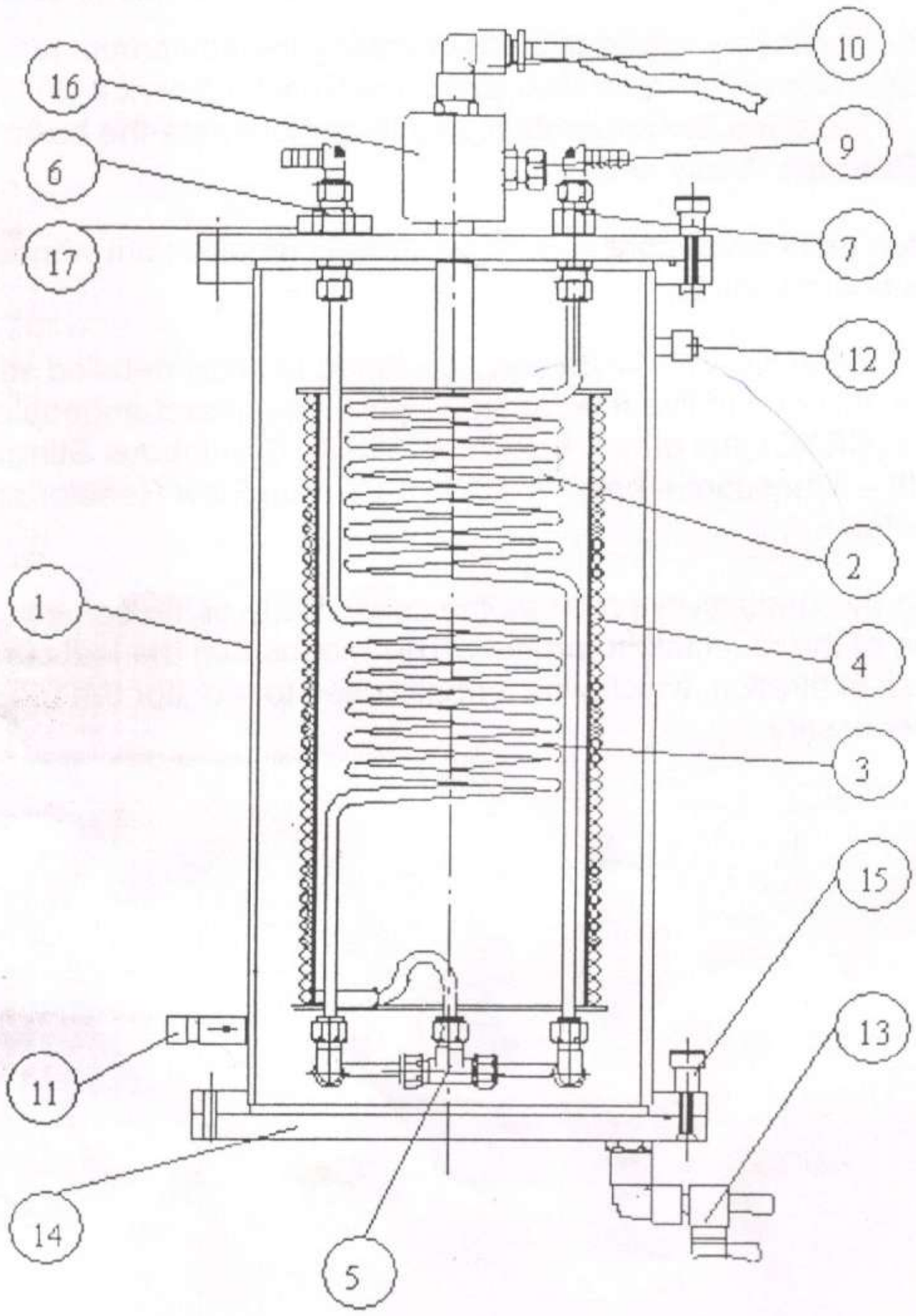
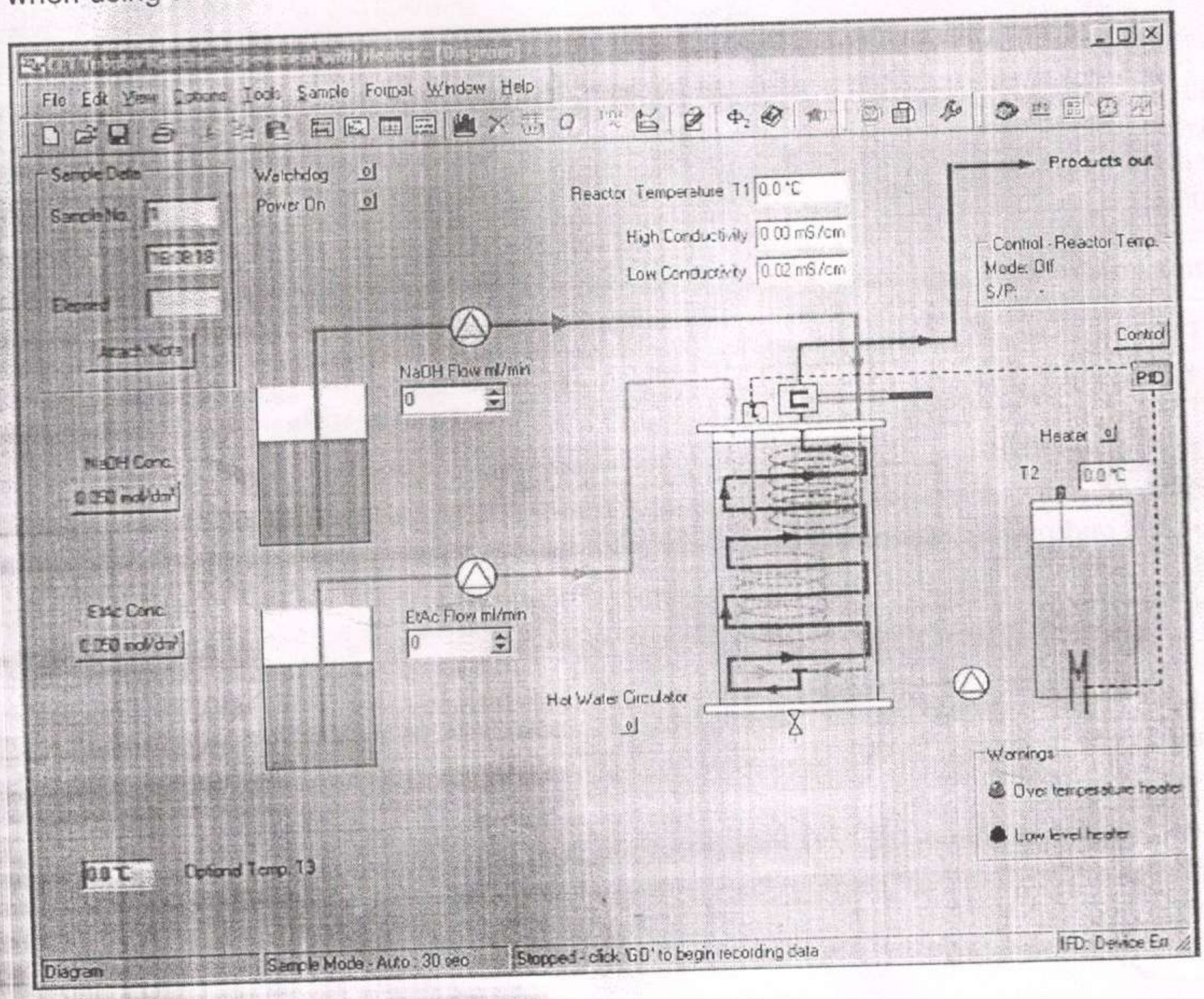


Figure 1: Front View of CET-MKII Tubular Reactor

The software also automatically generates a series of 'Watchdog' pulses, required by the PC, ensuring that the hardware shuts down safely in case of a software or communications failure.

CET-MKII Tubular reactor can be used with heater or with Chiller. When the Heater is used settings on the software are required. When using the Chiller CW-17 PID settings are not required. See the installation guide for appropriate controller settings when using the CW-17.



CET-MKII Tubular Reactor with Heater

Exercise A - To determine the rate constant using a tubular reactor

Theory

The rate expression can be shown to be $r = k.a.b$

where if a_μ is equal to b_μ this simplifies to $r = k.a^2$

In the general case the order of reaction n is not known and is shown by $r = k.a^n$

The rate constant can be determined using the CET-MkII Tubular Reactor.

We can show that $kta_\mu = \frac{X_a}{1-X_a}$ (for a second order reaction)

From this equation it can be seen that a plot of $\frac{X_a}{1-X_a}$ against time t will give a straight line of slope ka_μ.

If the inlet concentration a_μ is known, k can be determined.

The reaction:

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

Sodium Hydroxide + Ethyl Acetate → Sodium Acetate + Ethyl Alcohol

can be considered 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.

The reaction carried out in a Tubular Reactor eventually reaches steady state when a certain amount of conversion of the starting reagents has taken place.

The steady state conditions will vary depending on concentration of reagents, flowrate, volume of reactor and temperature of reaction.

Method

Make up 2.5 litre batches of 0.1M sodium hydroxide and 2.5 L of 0.1M ethyl acetate.

IMPORTANT: It is essential when handling these chemicals to wear protective clothing, gloves and safety spectacles.

Remove the lids of the feed bottles and carefully fill with the reagents. Refit the lids and fit the silicone pipe from the pumps.

The experiments involve the collection and storage of conductivity data. The USB port located at the front of the Service Unit must be connected to the computer. This will enable data logging of the conductivity, flow rates and temperature sensors at selected time intervals over a selected period of time.

Ensure the conductivity probe and temperature sensor has been installed in accordance with the Installation section.

Start the software using the option of the experiment with heater.

It has been determined that the degree of conversion of the reagents affects the conductivity of the reactor contents so that recording the conductivity with respect to time using the Armfield data logger can be used to calculate the amount of conversion.

Prior to priming the hot water circulating system, fill the reactor with water. Fill the vessel to a level above the overflow (return to the circulator), just below the reactor lid, using a suitable hose from a domestic supply through the temperature sensor gland (8) in the lid. A non-return valve (11) prevents water flowing out of the reactor via the inlet. Ensure the thermocouple is re-fitted and the gland tightened securely by hand before releasing the outlet tubing.

Set PID controller loop according to the settings for an Experiment with Heater described in operation section.

Adjust the set point of the PID to 30°C.

Change PID 'mode of operation' to 'Automatic'.

Switch on the Hot Water Circulator by clicking 'Hot Water Circulator' and then 'Power On'. The temperature of the water in the reactor vessel will begin to rise and within 10-15 min will be automatically maintained at the desired set-point (30°C in this instance).

When temperature reactor is steady type in the value of the concentration of both solutions on the software.

Switch on the pumps by typing the flow rate in the software and instigate the data logger program (or begin taking readings if no computer is being used).

Reactants will flow from both feed bottles and enter the reactor through the connections in the lid. Each reactant passes through pre-heat coils submerged in the water in which they are individually brought up to the reaction temperature. At the base of the tubular reactor coil, the reactants are mixed together in a "T" connection and begin to pass through the coil. The reacting solution will emerge from the coil through connector (16) in the lid where a probe senses continuously the conductivity which is related to degree of conversion. For an accurate conductivity reading, no bubbles are allowed in the reactant pipe.

Collection of data will be until a steady state condition is reached in the reactor and this takes approximately 30 minutes. It is advisable to set the data collection period to 45 minutes.

Interpretation of Results

The conductivity measurements must now be translated into degree of conversion of the constituents.

Both sodium hydroxide and sodium acetate contribute conductance to the reaction solution whilst 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:

The calculations are best carried out using a spreadsheet such as EXCEL so that the results can be displayed in tabular and graphical form.

On conclusion of the experiment using the Armfield data logger, a set of readings of conductivity with time will be stored in the computer.

At this point, this data can be transferred onto the spreadsheet.

Start the spreadsheet program.

Note that Armfield software performs all the calculations during experiment. However, it is recommended to go through all the procedure and calculations for better understanding.

Now enter the following known constants from the experiment using the Nomenclature. Ensure use of correct units.

F_a =

F_b =

a_μ =

b_μ =

c_μ =

T =

V =

Using the spreadsheet, calculate the values of a_o, b_o, c_∞, a_∞, $\Lambda_{c\infty}$, Λ_{ao}, $\Lambda_{a\infty}$, Λ_o and Λ_∞ from the following formulae:

$$a_0 = \frac{F_a}{F_a + F_b} \cdot a_\mu$$

$$b_0 = \frac{F_b}{F_a + F_b} \cdot b_\mu$$

c_∞	=	b_o	for $b_o < a_o$
c_∞	=	a_o	for $b_o \geq a_o$
$\Lambda_{c\infty}$	=	$0.070[1+ 0.0284(T-294)]\ c_\infty$	for $T \geq 294$
Λ_{ao}	=	$0.195[1+ 0.0184(T-294)]\ a_o$	for $T \geq 294$
Λ_o	=	Λ_{ao}	assumes $c_o = 0$
a_∞	=	0	for $a_o < b_o$

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

$\Lambda_{a\infty} = 0.195[1 + 0.0184(T-294)]\, a_\infty$ if $a_\infty \neq 0$

$\Lambda_\infty = \Lambda_{c\infty} + \Lambda_{a\infty}$

For the values of each of the above, the spreadsheet can be used to calculate values of sodium hydroxide concentration (a_1) and sodium acetate concentration (c_1) and the degree of conversion (X_a) and (X_c) for each of the samples of conductivity taken over the period of the experiment.

These can be calculated and listed in columns (use spreadsheet COPY facility) alongside the readings of conductivity using the following equations:

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

$$c_1 = c_\infty\left[\frac{\Lambda_0 - \Lambda_1}{\Lambda_0 - \Lambda_\infty}\right] \quad \text{for } c_0 = 0$$

$$X_a = \frac{a_0 - a_1}{a_0}$$

$$X_c = \frac{c_1}{c_\infty} \quad \text{for } c_0 = 0$$

To calculate the specific rate constant, k:

The overall mass balance at steady-state condition may be written as:

Input – Output ± Reaction = 0

i.e. for a reactant a in a reactor of volume V

$$\frac{d(Va_1)}{dt} = F \cdot a_0 - .F \cdot a_1 - V \cdot k \cdot a_1^2$$

For the continuous reactor operating at steady state the volume may be assumed constant and

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

The steady state concentration of NaOH in the reactor a_1 may be used to calculate the specific rate constant k.

Comment upon the results obtained.

Exercise B - To determine the kinetic constant of a reaction using an indicator for visually monitoring

The experimental procedure is identical to that of Exercise A with the exception that Ethyl acetate solution will contain 0.01% w/w of Indigo carmine to monitor the change in colour while the reaction takes place.

Method

The ethyl acetate solution has to content Indigo Carmine at 0.01% p/p. This indicator is an acid base non toxic indicator with a change range between 11.5 and 13 of pH going through dark blue to greenish yellow.

Calculate the degree of conversion of the reactants at steady state using conductivity readings (from data logger and spreadsheet as described in Exercise A) for different values of F_a and F_b. Exercise A used flows of 80ml/min so it is suggested that flows of 40ml/min and 60ml/min are used for this experiment. Plot t_R against $\frac{X_a}{1-X_a}$.

Comment on the graph obtained.

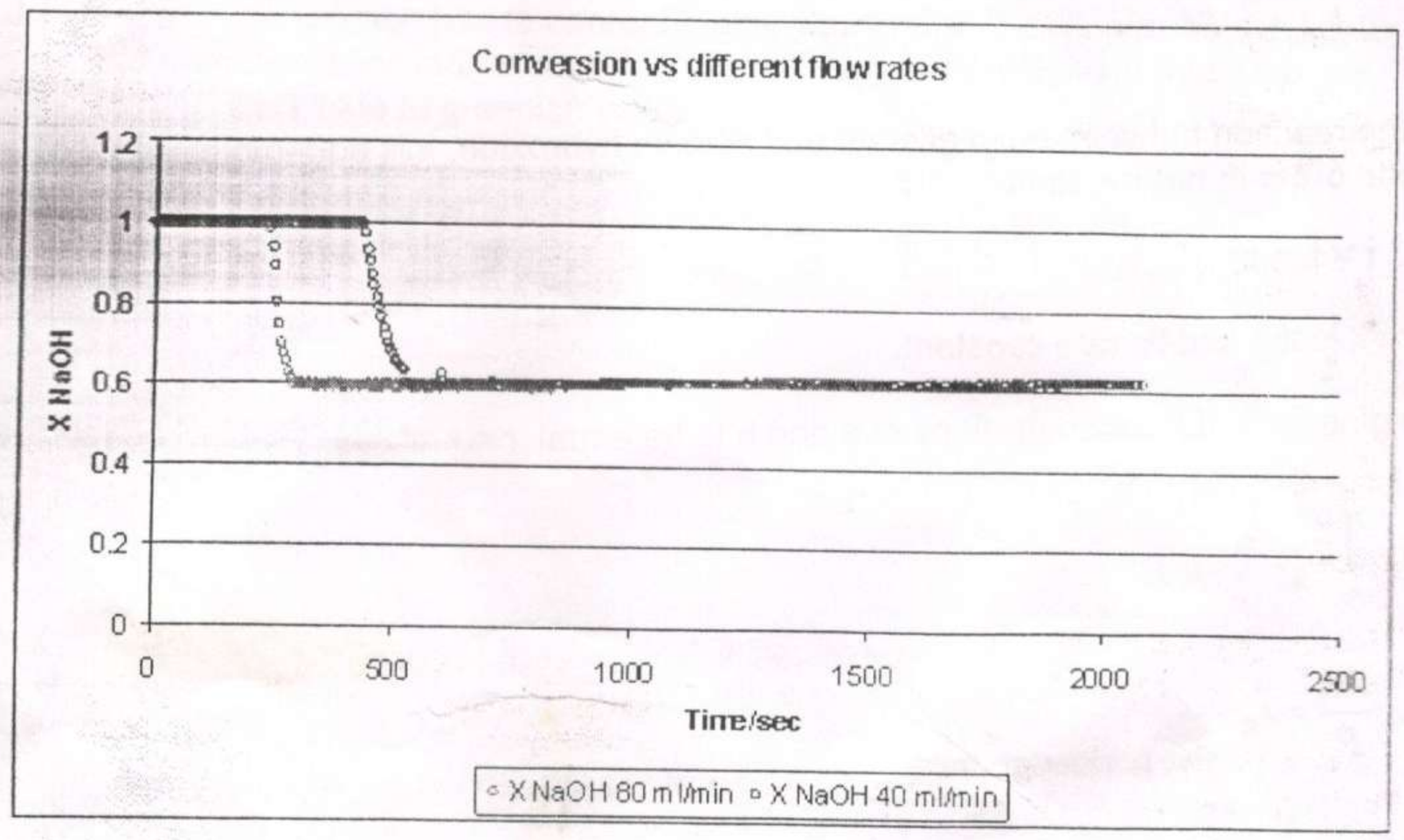


Figure 2: Conversions of NaOH at different flow rates

Exercise D - Demonstrate the temperature dependance of the reaction and the rate constant

Theory

The rate of reaction as characterised by its rate constant *k* is strongly temperature dependent. This is generally expressed as the Arrhenius equation:

$$k = Ae^{-\frac{E}{RT}}$$

where E (activation energy [J/mol]) and R (gas constant [J/mol.K]) are constants, T [K] is the reaction temperature and Ă - frequency factor.

Therefore,

$$\ln k = \ln A - \frac{E}{RT}$$

A logarithmic plot of k vs. 1/T will give a straight line.

Method

The procedure will be identical to that of Exercise A with the excepti... reaction will be carried out at different reactor temperatures.

Exercise A was carried out at 25°C. It is suggested that the reaction be carried out at a minimum of two further settings, say, 20°C and 30°C in order to plot the graph.

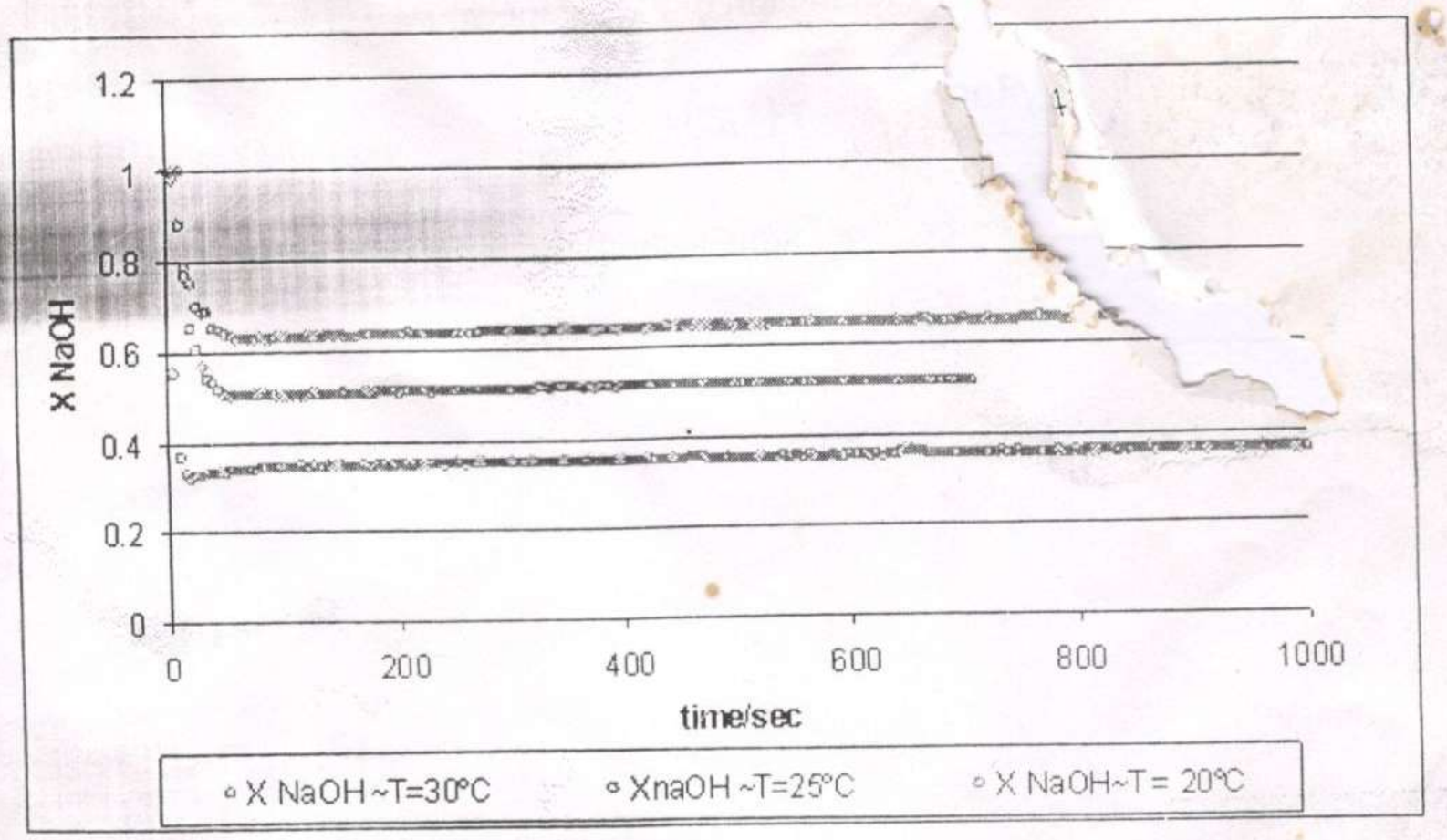


Figure D1: Conversions of NaOH at different temperatures

Plot 1/T vs. ln k and comment on the graph obtained.

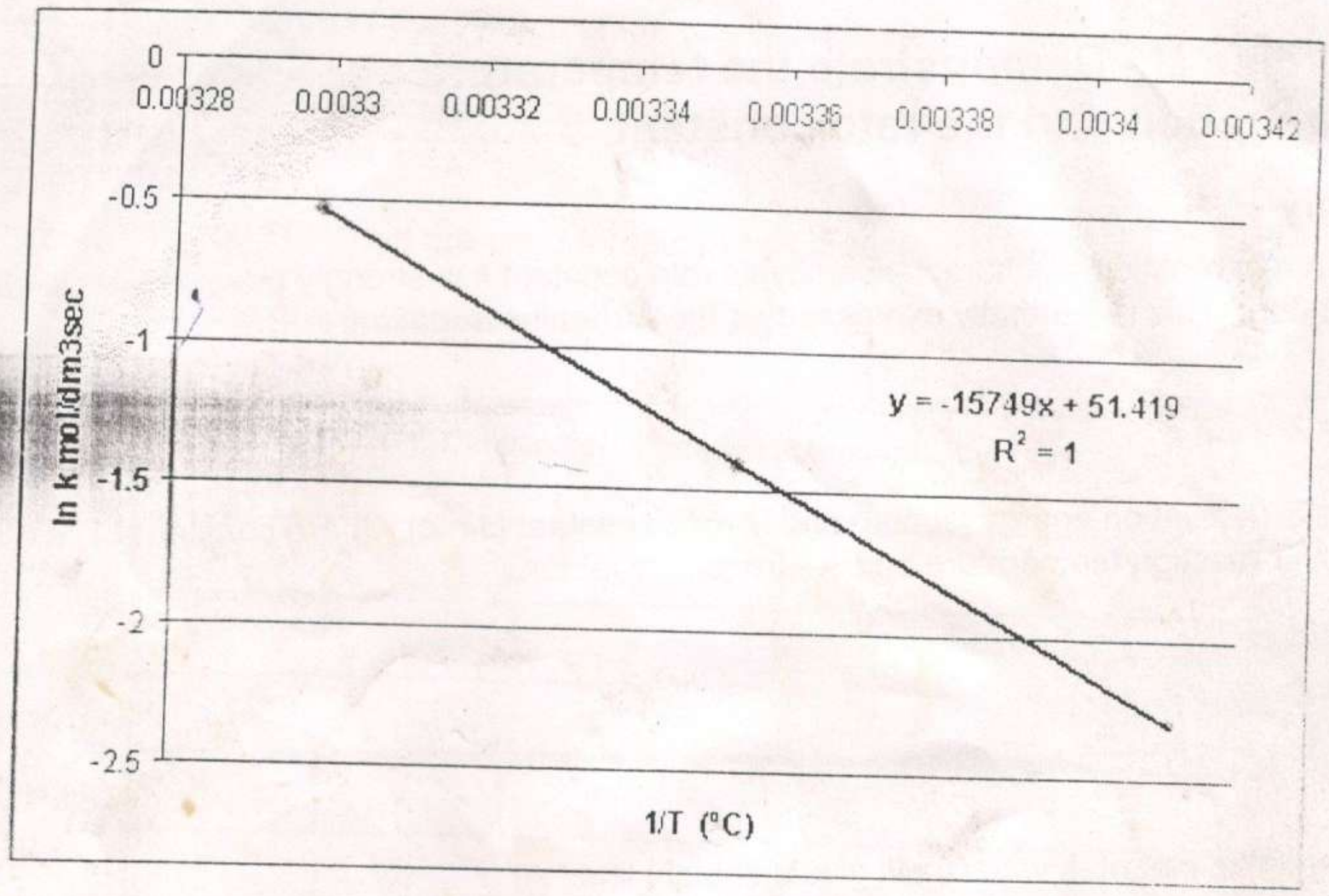


Figure D2: Variation of the Rate constant with Temperature

0 +

ln Q

$$\breve{A} = 2.142 \cdot 10^{+22}\ m^3 / mol\,sec$$

$$\frac{E}{R} = 15749 \qquad E = 130.96\ KJ / mol$$

siendo R = 8.314 J K⁻¹mol⁻¹

Obtaining the reaction rate constant in function of the temperature:

$$\ln k(T) = 51.42 - 157\ldots \qquad T(^\circ K)$$

High activation energy implies a significant sensitivity of the reaction kinetics to the temperature.