University of Jordan
School of Engineering
Department of Chemical Engineering

Reaction Engineering and Control Lab.
(0905564)

August/2025

Version 1

Experiment Number -1-

Continuous Stirred Tank Reactor

Objective

To determine the kinetics and reaction rate constant of the essentially irreversible reaction between ethyl acetate and sodium hydroxide by the capacity flow method and to measure the residence time density function of the effluent stream.

Apparatus

Continuous stirred tank reactor-see Fig.(1)- consisting of reaction vessel, two feed tanks with two pumps, two flow meters and sump tank. Brinkmann pc-600 Probe Colorimeter, continuous chart recorder and glassware for concentration measurements.

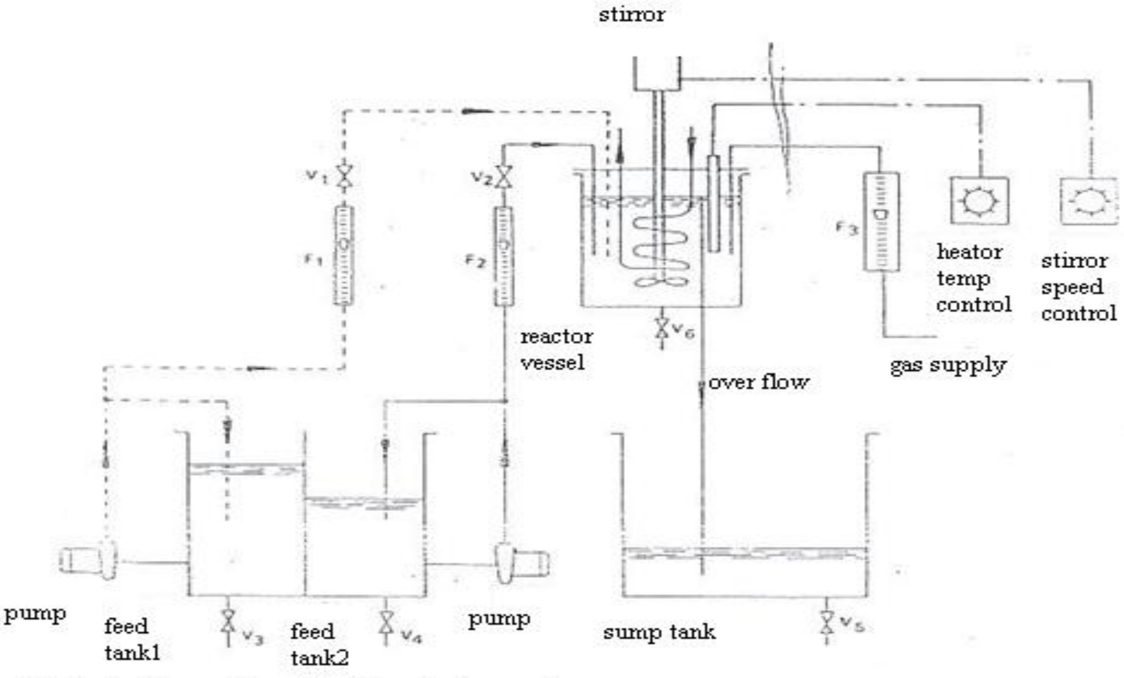


Fig.(1): Continuous Stirred Tank Reactor Apparatus

Procedure

Part A

1. Prepare 40 liters of 0.04 M solution of ethyl acetate and 0.04 M sodium hydroxide , 2 liters of 0.03 M solution of HCl and 0.03 M NaOH (Chemicals prepared should be standardized).
2. Place the 40 liters solutions into the feed tanks.
3. Switch on the pumps, allow the solutions to be delivered to the reactor at equal flow rates and set the stirrer to maximum speed.
4. At steady state find the conversion of NaOH.

5. Repeat the experiment at two different flow rates and at the same temperature.
6. Determine the form of the rate equation between ethyl acetate and sodium hydroxide.

Part B

1. Fill the reactor with tap water.
2. Calibrate the spectrophotometer-recorder combination by plotting a graph of transmittance vs pen deflection.
3. At zero time introduce a pulse of colored tracer (phenol red) into the reactor and record the change of transmittance vs time.

Calculations

1. Determine the reaction rate equation (n, K).
2. Determine E-curve of the effluent stream and determine the reactors flow model.

References

1. Levenspiel, O., “Chemical Reaction Engineering”, Second Edition, John Wiley and Sons, 1972.
2. Denbigh, K.G. and Page, F.H., Disc. Faraday Soc., 17 145 (1954).
3. Kendall, H.B., Chem. Eng. Progr. Symp. Ser. 70 (73), 3 (1976).
4. Wen, C.Y. and Chung, S.F., Can. J. Chem. Eng., 43, 101 (1965).

Continuous Stirred Tank Reactor Data Sheet

Titrants:	
Conc. of HCL	
Conc. of NaOH	

Reactants:	
Volume of NaOH sample	
Volume of HCl needed for titration	
Volume of ethyl acetate sample	
Volume of NaOH added	
Volume of HCl needed for titration	

Item	Run 1	Run 2	Run 3
Flow rate of ethyl acetate			
Flow rate of sodium hydroxide			
Residence time			
Sample 1: Volume of NaOH			
Sample 2: Volume of NaOH			
Sample 3: Volume of NaOH			
Temperature			

Experiment Number -2-

Temperature Measurement

Objectives:

1. To be familiar with many different methods of measuring temperature, highlighting accuracy, calibration and error sensitivity of each in a variety of working conditions.
2. To introduce the students to the international Temperature Scale, and allows investigation of the Platinum resistance temperature measurement method that forms a fundamental part of the standard.

Equipment:

The unit consists of a small bench mounted Console contains a number of different instruments and connection points for sensors. A temperature controlled heater plate is also available as well as a computerized data acquisition system with computer monitor and printer. The sensors available are: Platinum Resistance, Thermistor, & three types of thermocouple. A vacuum flask and stainless steel beaker are also available

Theory:

Temperature can be defined technically as an indication of intensity of molecular activity. The temperature of a body is a measure of the thermal potential of that body and determines whether heat is supplied to or rejected from the body when in contact with a body at different temperature.

Different scales are used in thermometry such as Centigrade scale which is based on the point at which ice melt and pure water boils at standard atmospheric pressure. The Celsius scale indicated that the ice point be substituted by the triple point which in the state of pure water existing as a mixture of ice, liquid and vapor in equilibrium, and equals 0.01°celsius. Another scale is the Kelvin scale, and another one is the international temperature scale.

Procedure:

Several experiments can be performed in the temperature measurement unit:-

Exp. No. 1

The use of liquid in glass thermometer, Vapor pressure & bi-metallic expansion devices for measurement of fixed scale point.

1. Partially fill the vacuum flask with ice and water and place one of the glass tube thermometers in the mixture.
2. Fill 2/3 of the stainless steel beaker with pure water and place rubber disc on top, place the beaker on heater plate and turn on the main switch. Set the heater plate to a temperature of about 200°C.

3. Once the water has reached boiling point, turn the temperature setting down to about 120°C.
4. Record the temperature of the water in the beaker and in the vacuum flask.
5. Repeat using vapor pressure thermometer & the bi-metallic expansion thermometer instead of liquid in glass thermometer.

Exp.No.2

Response of different temperature measuring devices as temperature changes with time.

1. Fill 2/3 of the stainless steel beaker with pure water and place rubber disc on top, place the beaker on heater plate and don’t turn on the main in this stage.
2. Place the Glass thermometer, Platinum resistance, Thermistor and the Thermocouple in the unheated water in the beaker and switch on the heater plate and set the heater plates to temperature 200°C.
3. Start recording the temperature using the computerized unit.
4. Record the temperature of the thermometer each 30 sec.
5. Once the water has reached boiling point, turn the heater off and stop recording.

Exp.No.3

a- The Peltier thermo-electric effect.

1. Partially fill the vacuum flask with ice and water, and fill 2/3 of the stainless steel beaker with pure cold water and place rubber disc on top, place the beaker on heater plate and don’t turn on the main in this stage.
2. Put water in a beaker and measure the ambient temperature by using the glass thermometer.
3. Select one of the shrouded type K thermocouple and do the instructed connection.
4. Now place the thermometer with the thermocouple in the unheated water in the beaker and switch on the heater plate and set the heater plate to temperature of about 200°C.
5. Start recording the temperature and millivolt signal by using the computerized unit.
6. Record the temperature of the thermometer each 30 sec.
7. Once the water has reached 40°C, stop recording and turn the temperature setting down to about 120°C.

b- Seebeck effect.

1. Select two of the shrouded type K thermocouples and connect them as instructed.

2. Place BOTH thermocouples in the vacuum flask and record the millivolt.
3. Place one thermocouple in the stainless steel beaker on the hot plate together with a glass thermometer, and record the millivolt.
4. Place the thermocouple that has been immersed in the vacuum flask into the hot water with the other thermocouple, note the millivolt reading.
5. Now reverse the positions of the thermocouples (Hot beaker thermocouple to vacuum flask), and record the millivolt.

Exp. No. 4

Voltage calibration of different thermometer types using water-ice reference.

1. Select two of the shrouded type K thermocouples.
2. Partially fill the vacuum flask with ice and water, and fill 2/3 of the stainless steel beaker with pure water and place rubber disc on top, place the beaker on heater plate and don’t turn on the main in this stage.
3. Connect the two thermocouples as instructed, and put one of the thermocouple in vacuum flask and the other in the beaker with a glass thermometer.
4. Plug in the Platinum resistance plug and the Thermistor plug to the unit and place the probes in the water in the beaker.
5. Switch on the heater plate and set the temperature to 200°C.
6. Start recording the temperature and the millivolt signal by using the computerized unit at regular intervals until the water boils.

Exp. No. 5

The law of intermediate metals and intermediate temperature associated with thermocouple.

1. Select two of the shrouded type K thermocouples.
2. Partially fill the vacuum flask with ice and water, and fill 2/3 of the stainless steel beaker with pure water and place rubber disc on top, place the beaker on heater plate and don’t turn on the main in this stage.
3. Connect the two thermocouples as instructed by using the two green thermocouple extension leads supplied, and put one of the thermocouple in vacuum flask and the other in the beaker.
4. Switch on the heater plate and set the temperature to 200°C. Once the water has reached boiling point, turn the temperature setting down to about 120°C.
5. Record the millivolt reading.
6. Immerse either junction A, B, or C in the ice-water mixture or the boiling water and record the millivolt.
7. Break the junction B and rejoin this using a RED or BLACK copper connecting lead, and record the millivolt.

8. Immerse the two new junctions either in the ice-water or boiling water, and record the millivolt.
9. Remove the RED or BLACK copper lead and record the millivolt.
10. Take the thermocouple from the ice-water and hold it in your hand until the millivolt meter display is constant and then record the millivolt.
11. Replace the thermocouple in the ice-water and allow the millivolt meter to return to its original reading.
12. Now remove the other thermocouple from the boiling water ALLOW TO COOL SLIGHTLY and then hold in your hand as before until the millivolt meter display is constant and record the millivot reading.

Exp. No. 6

Connection of thermocouple in parallel for averaging of measured temperature and series for signal amplification

1. Partially fill the vacuum flask with cold water, and fill 2/3 of the stainless steel beaker with pure water and place rubber disc on top, place the beaker on heater plate and turn on the main switch. Set the temperature to about 200°C.
2. Once the water has reached 70°C, turn the temperature setting down to about 120°C.
3. **For parallel;** select two of the type K thermocouples, connect the two type K thermocouple to any two of the thermocouple sensor sockets.
4. Place one thermocouple in the vacuum flask and the other in a beaker, full of hot water of about 70°C.
5. Depress the switches INDIVDUALLY and record the temperature. Then depress both switches together and record the temperature.
6. **For Series;** select four type K thermocouples (two shrouded and two exposed tip) and connect them as instructed.
7. The BLACK thermocouples plugs are shown as B, the RED thermocouples plugs are shown as R, the thermocouples junctions are shown as K. The pair of thermocouples shown as H are in the stainless steel beaker and the pair of thermocouples shown as C are shown in the vacuum flask.
8. Once the water in the stainless steel beaker starts boiling, observe and record the millvolt meter display.

Calculation:

Each number indicates to experiment number:

1. Record the temperature of the water by using Glass thermometer, Vapor pressure, Bi-metallic expansion device, Platinum resistance, Thermistor & Thermocouple, and explain why the temperature doesn’t reach 0°C and 100 °C.
2. Plot the temperature readings of the devices used versus time and note the response of each measuring device.
3. Record the reading of millivolt as indicated and explain the negative reading you get in both Peltier thermo-electric effect & Seebeck effect.
4. Refer to the tables in the lab sheet for a type of thermocouple that you used in this experiment showing the millivolt signal and corresponding temperature when the reference junction is held at 0°C and at each temperature reading, compare between the recorded millivot reading and the millivolt from the table that corresponds to that temperature.
5. Record the reading of millivolt as indicated and explain your results.
6. Record the reading of millivolt as indicated and explain your results.

Temperature Measurement Data Sheet

Part 1

Thermometer	Temperature (°C)
Glass thermometer	
Vapor pressure	
Bi-metallic	
Platinum resistance	
Thermistor	
Thermocouple	

Part 2

Time	Temperature (°C) Glass thermometer

Part 3

a- Peltier thermo-electric:

Ambient temperature:

Time	Temperature (°C) Glass thermometer

b- Seebeck effect:

	Voltage
Both thermocouples in cold water	
Both thermocouples in hot water	
Thermocouple (1) in cold and (2) in hot	
Thermocouple (2) in cold and (1) in hot	

Part 5

Intermediate metal:

Voltage for the junction without intermediate metal:

	Voltage
Junction **A** in hot water	
Junction **B** in hot water	
Junction **C** in hot water	
Junction **A** in ice	
Junction **B** in ice	
Junction **C** in ice	

Voltage for the junction with intermediate metal:

	Voltage
Junction **B** in hot water	
Junction **B’** in ice	

Intermediate Temperature:

	Voltage
$E_{hot} - E_{ice}$	
$E_{hot} - E_{hand}$	
$E_{hand} - E_{ice}$	

Part 6

Parallel connection:

Temp by depress switch # 1	
Temp by depress switch # 2	
Temp by depress switch # 3	
Temp by depress switch # 1+2	
Temp by depress switch #	

Series connection:

Voltage =	

Experiment Number -2-

Temperature Measurement (Part 1)

1-THE PELTIER THERMOELECTRIC EFFECT:

Objective: To demonstrate the temperature dependent of electrical potential produced by dissimilar metals in contact.

Equipment: See the attached sheet.

Theory:

The Peltier thermoelectrical effect (1834) can be stated as follows: current flowing across a junction of dissimilar metals causes heat to be absorbed or liberated. The direction of heat flow can be reversed by reversing the current flow. The rate of heat is proportional to temperature versus the voltage reading.

Requirement: plot the measured temperature versus the voltage reading.

2-THE SEEBECK THERMO-ELECTRICAL EFFECT:

Objective: To demonstrate the resultant electromotive force produced in a thermocouple circuit.

Equipment: See the attached sheet.

Theory:

The Seebeck effect or Seebeck principle discovered by T.J Seebeck (1821) states that an electric current flows in a circuit of two dissimilar metals if the two junctions are at different temperatures.

In figure 1: The temperature of the hot junction is the temperature being measured and T_c is the cold junction temperature or the reference temperature. The most common thermocouples are: platinum-rhodium/platinum, chromel/alumel, and copper/constantan. The circuit in figure 1 can be used for temperature measurement as shown as in figure 2. In chemical industries, sometimes it is not possible to maintain the cold junction at 0 °C. Normally, the cold junction is at ambient temperature, with correction is made automatically by temperature-sensitive resister.

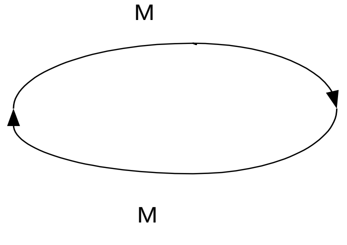


Fig.(1): Electro-thermal circuit of two dissimilar metals.

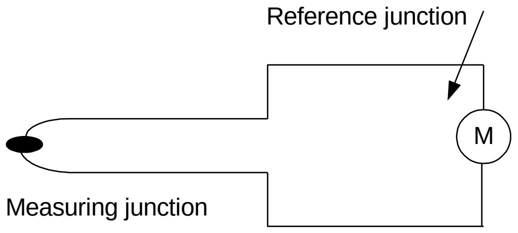


Fig.(2): Thermocouple circuit.

References:

1. Shahian, B. and Hassul, M., Control System Design. Prentice Hall, 1993.
2. Considine, D., M., Process Instruments and Control Handbook. McGraw-Hill, 1974.

Experiment Number -2-

Temperature Measurement (Part 2)

1-THE LAWS OF INTERMEDIATE METALS AND TEMPRETURES:

Objective: To demonstrate the effect of introducing different types of metals in a thermocouple circuit, and the principle of intermediate temperature

Equipment: See the attached sheet.

Theory:

Several thermoelectric laws have been established experimentally by measuring the current resistance, and emf in thermoelectric circuits. These laws are experimentally accepted despite the lack of theoretical development. In fact, we need not to understand the complexities of electron-phonon (the quantum particles of thermal conduction waves) to understand the working principles of thermocouples. We need only to pay attention to the understanding the laws of thermoelectricity.

1-Law of homogeneous materials:

A thermoelectric current cannot be sustained in a circuit of a single homogeneous material by the application of heat alone. A fact could be drawn from this law that two different types of metals are required to construct any thermocouple circuit. Experiments have shown that a measurable thermoelectric current flows in a circuit if a nonsymmetrical temperature gradient is set up in a homogeneous wire. This for no doubt is attributed to non-homogeneous metals.

2- Law of intermediate metals:

The algebraic sum of the thermoelectromotive forces in a circuit composed of any dissimilar metals is zero provided that all of the circuit is at a uniform temperature.

A consequence of this law is that a third homogeneous wire can be added in a thermocouple circuit as long as its extremities are kept at the same temperature. So a device for measuring the thermoelectromotive force can be introduced into a circuit at any point without effecting the net emf if all the connections at the same temperature.

In Figure 1:if $T_1=T_2$, then from the Seebeck principle the net emf is zero.

3- Law of successive or intermediate temperatures:

If two dissimilar homogeneous materials produce a net emf of E_{t_i,t_0} when the two junctions are at T, T_I, the emf generated when the junctions are at T and T_0 is given by:

$$E_{T,T_0} = E_{T_I,T_0} + E_{T,T_I} \qquad (1)$$

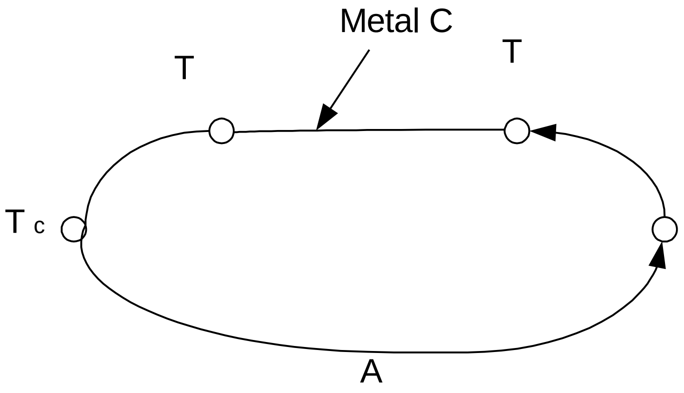


Figure (1): Thermocouple circuit with intermediate metal type C.

Equation (1) is evident from Figure 2 and the Seebeck principle:

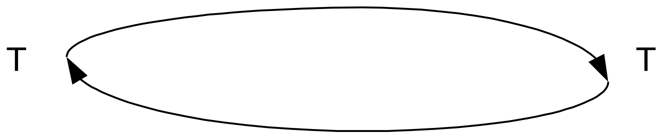


Figure (2): Emfs are additive for materials.

$$E_{T,T_I} = \alpha\left(T - T_I\right)$$

$$E_{T_i,T_0} = \alpha\left(T_I - T_0\right)$$

$$E_{T,T_0} = \alpha\left(T - T_0\right)$$

$$E_{T,T_0} - E_{T_I,T_0} = \alpha\left(T - T_0 - T_I + T_0\right) = \alpha\left(T - T_I\right) = E_{T,T_I}$$

Note that equation 1 follows from the last result, where T_I satisfies: $T_0 < T_I < T$.

Questions:

1. A thermocouple of type J (iron-constantan) has the following input-output data from standard tables:

T (C°):	0	30	300
emf (mV):	0	1.5	15

2. Discuss the validity of the law of intermediate temperatures on the basis of Seebeck principle over a wide temperature range (0 t0 500 °C).

References:

1. Considine, D. M., Process instruments and control handbook. McGraw-Hill, 1974.
2. Benedict, P. E., Fundamentals of temperature, pressure and flow measurements. John Wiley, 1984.
3. Bolton, W., Mechatronics: electronic control systems in mechanical engineering. Longman, 1997.

Experiment Number -2-

Temperature Measurement (Part 3)

Objectives: To understand the static and dynamic characteristics of measuring devices and in particular temperature measuring devices

Equipment: See the attached sheet.

Temperature Sensors:

One of the most important variables in chemical industries is the temperature. It is not only used as a direct control variable, but also as a means to infer about other controlled variables (inferential control). Table 1 below shows the most popular sensors used for temperature measurement.

The term sensor is used to identify an element that produces a signal when subjected an input physical change. Often the term transducer is used interchangeably with the term sensor.

Table 1 Important Sensors (Transducers) for Temperature Measurement.

I. Expansion thermometers
    A. Liquid-in-glass thermometers
    B. Solid-expansion thermometers (bimetallic strip)
    C. Filled-system thermometers (pressure thermometers)
        1. Gas-filled
        2. Liquid-filled
        3. Vapor-filled

II. Resistance-sensitive devices
    A. Resistance thermometers
    B. Thermistors

III. Thermocouples

1. Expansion thermometers:

Liquid-in-glass thermometers: indicate temperature change caused by the difference between the temperature coefficient of expansion for glass and the liquid employed. Mercury and alcohol are the most widely used liquids.

2. Solid Expansion thermometers (Bimetallic strip):

Bimetallic strip thermometer*:* The working principle depends on different expansion coefficients of metals as a function of temperature. Fig(1) shows a typical bimetallic strip thermometer. The temperature-sensitive element is a composite of two different metals fastened together into a strip. A common combination is invar (64% Fe, 36% Ni), which has a low coefficient, and another nickel-iron alloy that has a high coefficient.

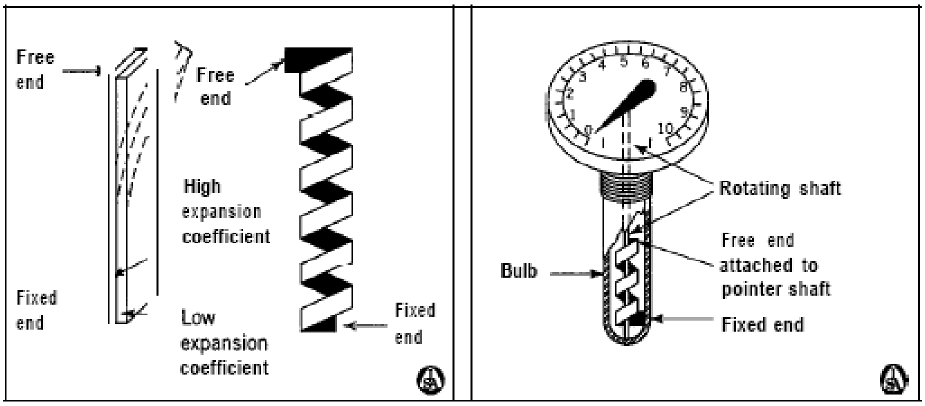


Fig.(1): Details of bimetallic strip thermometer [1].

3. Filled-system thermometers (pressure thermometers):

A typical filled-system thermometer. is shown in Fig.(2) The fluid expands or contracts due to temperature variations which is sensed by the Bourdon spring and transmitted to an indicator or transmitter. These elements are popular in the chemical process industries.due to their design simplicity and relatively low cost.

Resistance thermometers Devices(RTD): Fig.(3) shows RTD which based on the principle that the electrical resistance of pure metals increases with an increase in temperature. This provides an accurate way to measure temperature because measurements of electrical resistance can be made with high precision.

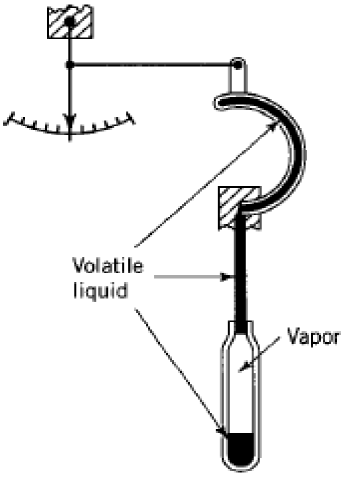


Fig.(2): Filled system thermometer [1,4].

The resisitance of most metals increases linearly with temperature:

$$R_t = R_0(1 + aT) \tag{1}$$

The most commonly used metals are platinum, nickel, tungsten, and copper. A Wheatstone bridge is generally used for the resistance reading and, consequently, for the temperature reading.

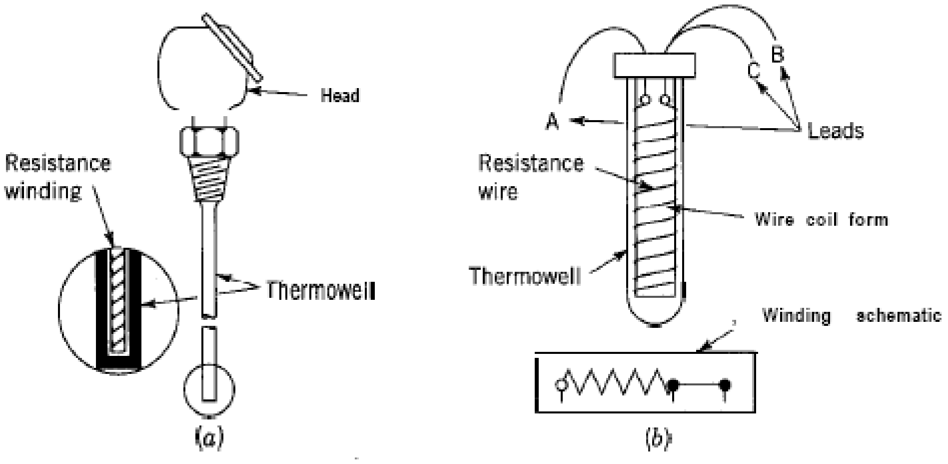


Fig.(3): Resistance thermometer device (RTD): a- Assembly. b- Components [1,4].

1. Thermistors:

Modern thermistors are usually mixtures of oxides such as the oxides of nickel, manganese, iron, copper, cobalt and titanium, other metals and doped ceramics. The material is formed into various forms such as beads discs and rodes as shown in Fig.(4). Thermistors can have either a negative temperature coefficient (NTC), where the resistance decreases with temperature, or a positive temperature coefficient(PTC) depending on the type of materials used. They were originally named from a shortened form of the term *thermally sensitive resistor*. The resistance-temperature relationship can be described by:

$$R = Ke^{b/T} \tag{2}$$

Where K and b are constants.

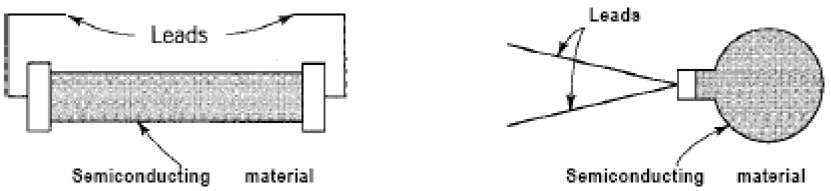


Fig.(4): Thermistor construction [2,4].

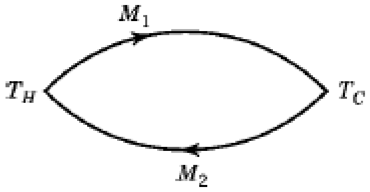


Fig.(5): Thermocouple circuit

2. Thermocouples:

The thermocouple is the best-known industrial temperature sensor. It works on a principle discovered by Seebeck in 1821. The Seebeck effect, or Seebeck principle, states that an electric current flows in a circuit of two dissimilar metals if the two junctions are at different temperatures. Fig.(4) shows a simple circuit in which M1 and M2 are the two metals, TH is the temperature being measured, and TC, is reference temperature. The voltage produced by this thermoelectric effect depends on the temperature difference between the two junctions and on the metals used.

Static Characteristics:

The output values given when steady state condition is attained, after a certain input is received, are known as static characteristics of the sensor. The most important static characteristics are:

1. **Range:** The limits between which the input can vary.
2. **Error:** The difference between the true (standard) value and the result of measurement.
3. **Accuracy:** The extent to which the value indicated by the measuring device is wrong. It is the sum of all the possible expected errors including the calibration accuracy.
4. **Precision:** It describes an instrument’s degree of freedom from random errors. If a large number of readings are taken of the same quantity by a high precision instrument, then the spread of readings will be very small.
5. **Repeatability:** It describes the closeness of output readings form the measuring device when the same input is applied repetitively over a short period of time, with

the same measurement conditions, same instrument and observer, same location and same conditions of use maintained throughout.

6. **Reproducibility:** describes the closeness of output readings for the same input when there are changes in the method of measurement, observer, measuring instrument, location, conditions of use and time of measurement. Fig.(6) compares the accuracy, repeatability and reproducibility.
7. **Sensitivity:** Sensitivity is the ratio of the change in response of an instrument to the change in the stimulus (how much output you get per unit input).
8. **Hysteresis:** The difference between the measuring device readings during a continuously increasing and decreasing change of the input. Fig.(7) shows a typical hysteresis curve.

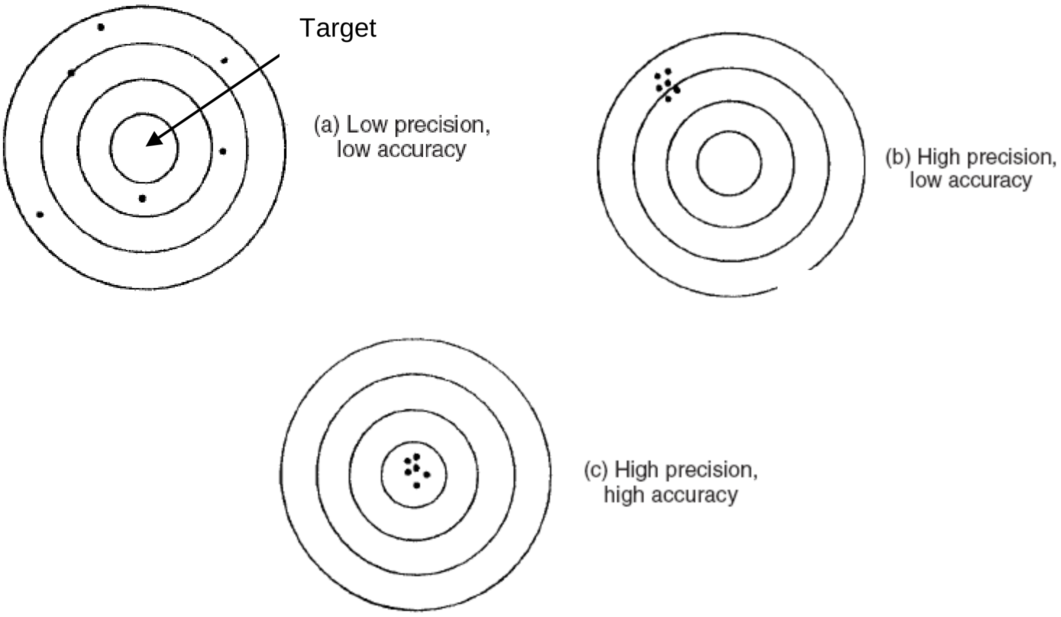


Fig.(6): Comparison of accuracy and precision.

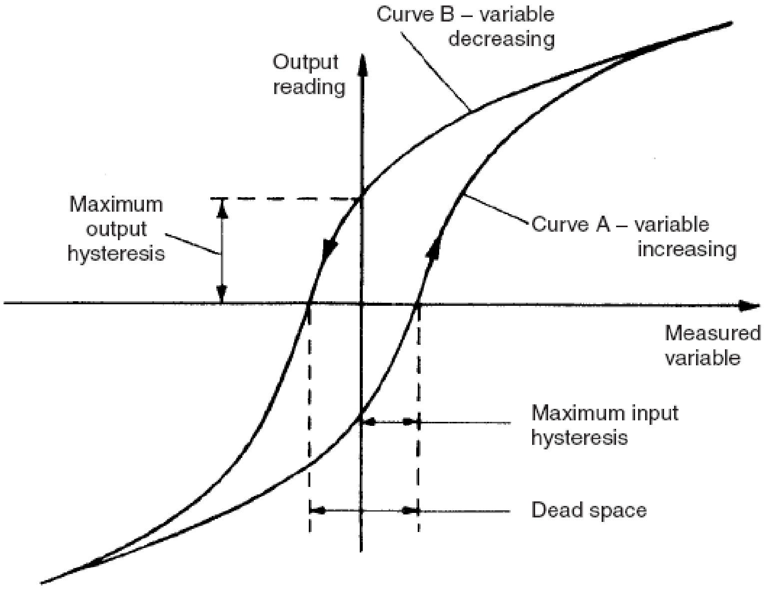


Fig.(7): Measuring device static characteristics with hysteresis.

Dynamic Characteristics:

The dynamic characteristics of a measuring instrument describe its behaviour between the time the input value changes and the time the instrument output attains a steady value in response.

In any linear, time-invariant measuring system, the following general relation can be written between input and output for time t > 0:

$$a_n \frac{d^n y}{dt^n} + a_{n-1} \frac{d^{n-1} y}{dt^{n-1}} + \dots a_1 \frac{dy}{dt} + a_0 y = b_0 u \qquad (3)$$

Where y and u are the output and input respectively, $a_0, a_1, \dots a_n$ and b_0 are constant model parameters.

1. **Zero-order dynamics:** An instrument is said to show zero-order dynamics, if it responds instantaneously to the applied input. Mathematically, this means that: $a_n = a_{n-1} = \dots a_1 = 0$. Consequently, the instrument sensitivity (gain) is given by:

$$K_e = \frac{\Delta y}{\Delta u} = \frac{b_0}{a_0} \qquad (4)$$

Fig.(8) shows the response of such instrument.

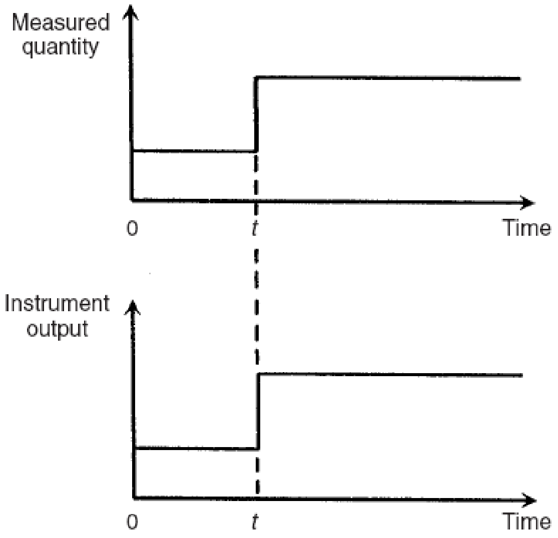


Fig.(8): Zero-order instrument dynamic response.

2. **First-order dynamics:** An instrument is said to show first-order dynamics, if it reaches 63 percent of the final steady state value after a time equals one instrument time constant. First-order dynamic instruments follow a dynamic response given by Eq.(3) after setting $a_n = a_{n-1} = \dots a_2 = 0$ which results in:

$$a_1 \frac{dy}{dt} + a_0 y = b_0 u \tag{5}$$

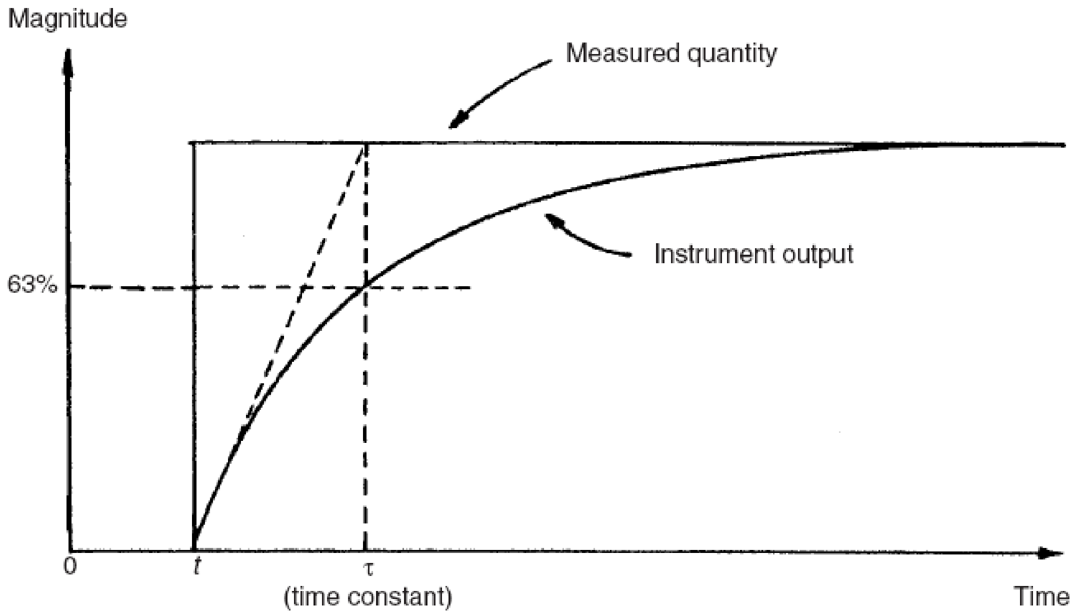


Fig.(9): First-order dynamic characteristics.

The above equation is solved for the output after replacing: d/dt by the D operator to get:

$$y = \frac{(b_0 / a_0)u}{1 + (a_1 / a_0)D} = \frac{K_e u}{1 + t_e D} \qquad (6)$$

Where: K_e and t_e are the instrument sensitivity and time constant respectively. The exact solution of Eq.(6) due to step change in u is shown in Fig.(9). In this figure the output response reaches 99 percent of its final value after five time constants ($5t_e$).

A good example for such instrument (first order instrument) is the liquid-in-glass thermometer. If a thermometer at room temperature is plunged into boiling water, the output does not rise instantaneously to a level indicating 100°C, but instead approaches a reading indicating 100°C in a manner similar to that shown in Fig.(9).

Requirements:

1. Study experimentally the static and dynamic characteristics of the available temperature measuring instruments in the Lab.
2. Investigate the dynamic linearity of the selected temperature sensor using the mirror image method.

References:

1. Smith, C. A. & Corripio, A. B. (1997). Principles and practice of automatic process control, John Wiley & Sons, New York.
2. Morris, A. S. (2001). Measurement and Instrumentation Principles. Butterworth-Heinemann, Oxford.
3. Shahian, B. and Hassul, M., Control System Design. Prentice Hall, 1993.
4. Considine, D., M., Process Instruments and Control Handbook. McGraw-Hill, 1974.

Experiment Number -3-
Batch Reactor

Objectives

1. To find the reaction rate constant for Saponification reaction in a batch reactor.
2. To study the effect of varying process conditions on the reaction rate constant.

Apparatus

The batch reactor vessel is a double skinned glass vessel with one liter and a half of internal working volume, the reactor is mounting on a computer controlled chemical reactors service unit which also provides the ancillary services for the reactor. The reactor is equipped with an agitator driven by an electric motor and controlled using the Armfield software. The speed can be varied using the up/down arrows or by typing in a value between 0-100%-see figures (1) & (2). A clear, acrylic, self-contained hot water circulator is positioned on the unit at the front right hand side-see figure (3)-this vessel incorporates an element for heating water, a thermostat and level detector are incorporated in the vessel to prevent the heater from operating if the water is too hot or the level in the vessel is too low. A thermocouple sensor T2 is supplied with this unit to measure the fluid temperature in the vessel. Flexible tubes are used to connect the circulator to the reactor. Water, heated by an electrical heating element in the circulator, is circulated by a gear pump located at the back of the plinth behind the vessel. Temperature control is achieved by circulation of heated water through the jacket of the batch reactor. T1 is designed to be used individually with reactors in conjunction with the PID controller in the software and T2 is to be used with hot water circulator vessel. A conductivity probe is supplied to be used with the reactor, the conductivity is displayed on the software in units of millisimens/cm. During the chemical reaction, the conductivity of the reacting solution changes as more of the reactants are converted. This data can be logged and used to determine the degree of conversion and the rate of reaction.

figure (1): computor controlled chemical reactor service unit

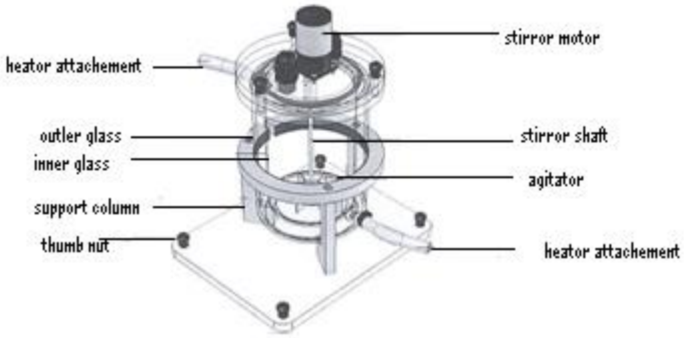


figure (2): Transparent Batch Reactor

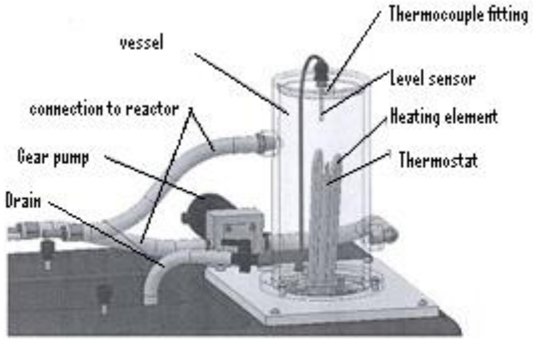


figure (3) : Hot Water Circulator

Theory

The Armfield batch reactor is designed to demonstrate the mechanism of a chemical reaction in a reactor, as well as the effects of varying process conditions such as reaction temperature and reagents concentrations. In this experiment, the Saponification of Ethyl acetate by Sodium hydroxide is the reaction chosen to study these conditions.

$NaOH+CH_3COOC_2H_5 \rightarrow CH_3COONa+C_2H_5OH$

For this second order elementary reaction the rate equation is:

$$-r_A=k.C_A.C_B \qquad (1)$$

Where,

r_A = rate of reaction ($mol.L^{-1}.S^{-1}$)

K = reaction rate constant (S^{-1})

C_A = concentration of NaOH ($mol.L^{-1}$)

C_B = concentration of Ethyl acetate (mol.L^{-1})

If the initial concentrations of reactants are equal, then:

$$-dC_A/dt = k.C_A^2 \quad (2)$$

Integration of this rate law for a single reactant gives;

$$1/C_A = k.t + 1/C_{Ao} \quad (3)$$

t = time (s)

C_{Ao} = initial concentration of NaOH (mol.L^{-1})

Hence, a plot of $1/C_A$ against t gives a straight line of gradient k and an intercept of $1/C_{Ao}$

If the initial concentrations of reactants are not equals, then the rate equation above should be combined with the following equations:

$$\theta = C_{Bo}/C_{Ao} \quad (4)$$

$$X_A = (C_{Ao} - C_A)/ C_{Ao} \quad (5)$$

$$C_B = C_{Bo} - C_{Ao}X_A \quad (6)$$

Where,

C_{Bo} = initial concentration of Ethyl acetate (mol.L^{-1})

X_A = conversion of NaOH

By combining previous equations, the following equation will be obtained:

$$-r_A = dC_A/dt = k.C_{Ao}^2(1-X_A)(\theta - X_A) \quad (7)$$

Separating, integrating and linearizing gives,

$$\mathrm{Ln}\left(\frac{\theta - X_A}{\theta(1-X_A)}\right) = C_{Ao}((\theta - 1).kt \quad (8)$$

This equation in the form Y =mX+ b where, $C_{Ao}((\theta - 1).k$ is the slope of the line.

It can also be determined by using conductivity as follow;

$$C_A = (C_{A\infty} - C_{Ao}\left(\frac{\Lambda_o - \Lambda}{\Lambda_o - \Lambda_\infty}\right) + C_{Ao} \quad (9)$$

Where,

Λ_o = initial conductivity (Siemens/cm)

Λ_∞ = conductivity after ∞ time (Siemens/cm)

Λ = conductivity at time t (Siemens/cm)

$C_{A\infty}$ = concentration of NaOH after ∞ time (mol.L^{-1})

Once CA is determined, conversion can be calculated, then by using equation (8),k can calculated.

Finally: Arrhenius behavior should be checked as follows:

$$K = k_o e^{-E/RT} \tag{10}$$

Where,

k_o = Arrhenius frequency factor

E = activation energy (J.mol^{-1})

R = Gas constant (J. mol^{-1}.K^{-1})

T = absolute temperature (K)

Linearizing the equation

$$\text{Ln k} = -\frac{E}{R}\frac{1}{T} + \ln k_o \tag{11}$$

A plot of lnk against 1/T should give a straight line with a slope of ($-\frac{E}{R}$) and an intercept of ln k_o .

Procedure

1. Make up 0.25 liter batches of 0.08M sodium hydroxide and 0.25 liter of 0.08M of ethyl *acetate.*
2. Open Armfield Software and choose 'Isothermal operation'.
3. Set up the Hot water circulator, adjust the settings on the PID loop and set point to 25C^o .
4. Charge the batch reactor with the 0.25 liter of sodium hydroxide solution. Set the reactor agitator to 100% and press 'power on' to start the agitation. Then press 'Hot Water Circulator' and water will start to recirculate through jacket reactor and vessel.
5. Ensure that the conductivity and temperature sensors have both been installed correctly.
6. Set the sample time interval about 30 second and the data collection period to 30 minutes.

7. Switch on the Hot Water Circulator. The temperature of the sodium hydroxide in the reactor vessel will begin to rise and will be automatically maintained at the desired set-point (25 C^o) by the action of HWC in the jacket.

8. Carefully add to the reactor 0.25 liters of ethyl acetate solution and start the data logger program. Conductivity with respect to time will be collected for about 30 minutes as chosen previously.

9. Repeat the experiment as above at the same concentrations but with the temperature controller set to 35C^o and 45 C^o respectively.

10. Also repeat the experiment at 25 C^o but with 0.08M NaOH and 0.1M ethyl acetate, then with 0.06M NaOH and 0.08M ethyl acetate.`

Calculations

1. Calculate the reaction rate constant for each run.
2. Show that the reaction rate constant is a function of temperature.
3. Find the activation energy (E) and the frequency factor (k_o).

References

1. Octave Levenspiel,’’Chemical Reaction Engineering”, Second Edition, John Wiley and Sons,1972.
2. CEXC Instruction Manual, ISSUE 6, March 2012.
3. CEB-MKIII Instruction Manual, ISSUE 2, December 2011.

Experiment Number -4-

Tubular Reactor

Objectives

A. To determine the rate constant using a tubular reactor.
B. To investigate the effect of throughput on conversion.
C. to demonstrate the temperature dependence of the reaction and the rate constant

Equipment

Overview

The reactor vessel (1) is set on a base plate (14) which is designed to be located on the four studs of the CEXC service unit and then secured by thumbnuts (15). The positioning of the reactor on the service unit is illustrated. 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 (4) wound around an acrylic former. Total volume of the reactor coil is 0.466 L.

In order 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 PID temperature controller which is incorporated in the Armfield Software. The actual temperature of the circulating water and therefore the reactants is relayed to the controller by a k-thermocouple (T1) which is held in gland (8) in the lid. Water enters from the circulator at the non-return valve (11) - this prevents water draining back through the vessel of the circulator when the pump is stopped. Water leaves the reactor at overflow (12) and returns to the circulator.

A filling plug (17) 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 CEXC service unit is in operation. Otherwise air will enter pushing down the water level of the reactor and over filling the HWC vessel.

Sockets at the rear of the service unit are provided to connect the conductivity probe and temperature sensors to the instrumentation.

An additional temperature sensor ‘T2’ is supplied with CEXC that allows the Temperature of the Hot Water Circulator (HWC) to be also monitored. Therefore T2 temperature sensor must be immersed in the HWC vessel through the hole in the lid.

Both temperature values and conductivity values are data logged using a PC in conjunction with the Armfield software interface.

When not in use, the reactor can be drained using the valve (13) in the base.

Flow of materials

Reactants are pumped from the two feed bottles supplied by the peristaltic pumps and enter the reactor through connectors (6) and (7) in the lid of the vessel. Each reactant is pre-heated by heat transfer coils (2) and (3) before being blended together in “T” fitting (5).

The reactants pass up through the reactor coil and leave the reactor vessel through the conductivity probe housing (16). 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 (10) is used to guide the reactants to drain.

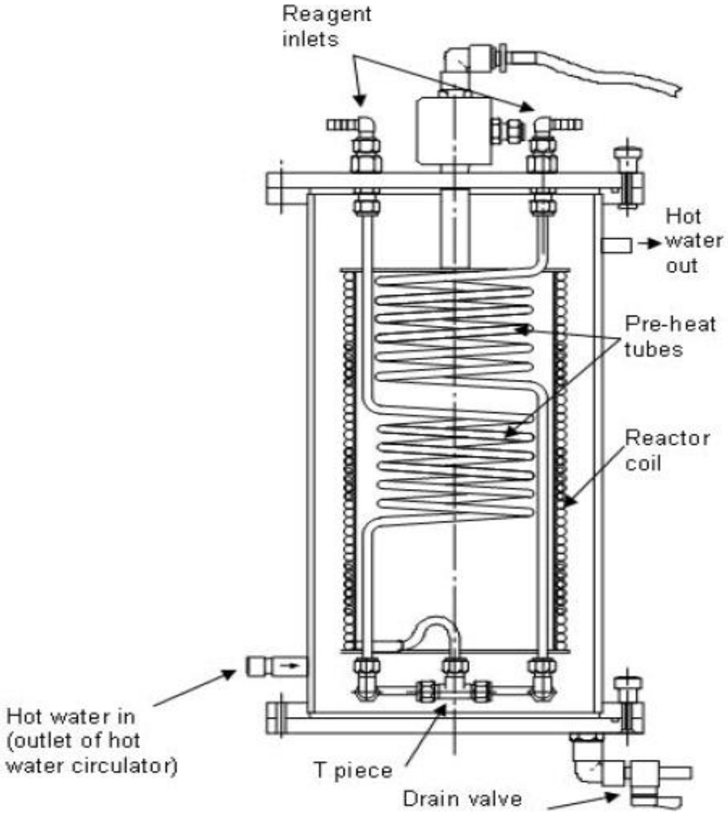


Fig.(1): Tubular Reactor Diagram

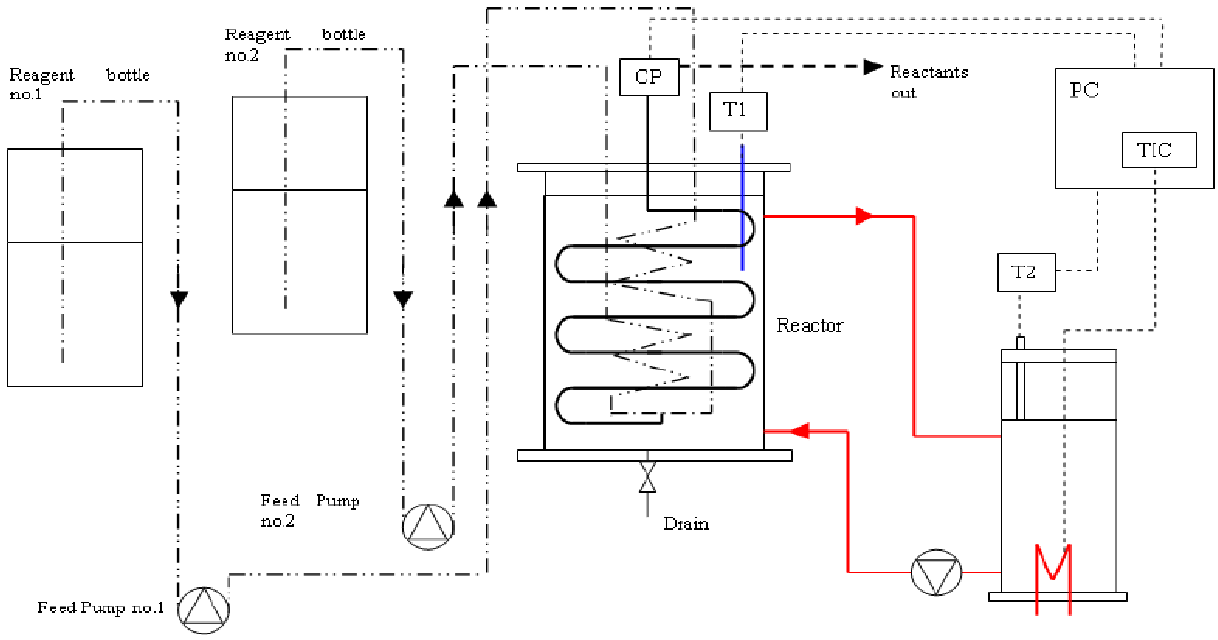


Fig.(2) : Set Up Flow Chart

Theory

A. Determination of the rate constant using a tubular reactor

In this experiment, the Saponification of Ethyl acetate by Sodium hydroxide is the reaction chosen to study these conditions.

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

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.

For this second order elementary reaction the rate equation is:

$$-rA = k.C_A.C_B \text{..................................}(1)$$

If C_A is equal to C_B this simplifies to

$$-rA = k.C^2 \text{.................}(2)$$

The rate constant can be determined by

$$K.t.C_A = \frac{X_a}{1-X_a} \text{..................}(3)$$

$$X_a = \frac{C_{A0}-C_{A1}}{C_{A0}} \text{..................}(4)$$

From equation (3) it can be seen that a plot of $(\frac{X_a}{1-X_a})$ against time (t) will give a straight line of slope kC_A.

Where:

rA = Reaction rate

k = Specific rate constant

C_A = Sodium hydroxide conc. in feed vessel (mol/L)

C_B = Ethyl acetate conc. in feed vessel (mol/L)
t = Elapsed time (s)
Xa = Conversion of sodium hydroxide
C_{A0} = Sodium hydroxide conc. in mixed feed (mol/L)
C_{A1} = Sodium hydroxide conc. in reactor at time t (mol/L)

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.

B. Investigation the effect of throughput on conversion

For a tubular reactor, the mass balance for a reactant is represented by:

$$\int_{CA1}^{CA0} \frac{dC_A}{rC_A} = \frac{AL}{F} \ldots\ldots\ldots\ldots\ldots\ldots..(5)$$

For the reaction between ethyl acetate and sodium hydroxide, it is equi-molar and is second order in nature, thus

$$r = k.a.b$$

where k is the kinetic rate constant.

Arranging for initial concentrations of a and b to be equal r = k.a^2

$$\int_{CA1}^{CA0} \frac{dC_A}{K(C_A)^2} = \frac{AL}{F} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(6)$$

$$\frac{AL}{F} = t_R \ldots\ldots\ldots\ldots\ldots\ldots(7)$$

Integrating

$$\frac{1}{C_{A0}} - \frac{1}{C_{A1}} = Kt_R \ldots\ldots\ldots\ldots\ldots\ldots..(8)$$

Fractional conversion

$$X_a = \frac{C_{A0} - C_{A1}}{C_{A0}} \ldots\ldots\ldots\ldots\ldots\ldots(9)$$

Therefore,

$$K.t_R.C_{A0} = \frac{X_a}{1 - X_a} \ldots\ldots\ldots(10)$$

Thus the conversion factor $\frac{X_a}{1-X_a}$ is directly proportional to t_R the residence time for constant reaction temperatures (T).

Where:

A = Cross sectional area of tubular reactor (cm^2)

L = Overall length of tubular reactor (cm)

t_R = Residence time (s)

F = Total volume feed rate (dm^3/s)

C. Demonstrate the temperature dependence of the reaction and the rate constant

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

$$K = K_0 \; e^{-E/_{RT}} \; \ldots\ldots\ldots\ldots\ldots\ldots\ldots..(11)$$

Linearization of the equation:

$$Ln\;(K) = -\frac{E}{RT} + \ln K_0 \;\ldots\ldots\ldots\ldots\ldots\ldots(12)$$

Where:

E = Activation energy (J/mol)

R = Gas constant (J/mol.K)

K_0 = Frequency factor

T = Reaction temperature (K)

A plot of lnk against 1/T should give a straight line with a slope of (- E) and an intercept of ln k_0 .

Procedure

1. Make up 2.5 liter batches of 0.06M sodium hydroxide and 2.5 L of 0.06M ethyl acetate.
2. Remove the lids of the feed bottles and carefully fill with the reagents. Refit the lids and fit the silicone pipe from the pumps.
3. Ensure the conductivity probe and temperature sensor has been installed in accordance with the Installation section.
4. Start the software using the option of the experiment with heater.
5. 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.

6. Set PID controller loop according to the settings for an Experiment with Heater described in operation section.
7. Adjust the set point of the PID to 30°C.
8. Change PID 'mode of operation' to 'Automatic'.
9. 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^oC in this instance).
10. When temperature reactor is steady type in the value of the concentration of both solutions on the software.
11. 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).
12. 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.
13. 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.
14. Repeat the steps from1-13 but with flow rates of reactants variation to change the residence time of the reactants in the reactor.
15. Calculate the degree of conversion of the reactants at steady state using conductivity readings (from data logger and spreadsheet as described in Exercise) for different values of Fa and Fb. 80ml/min , 40ml/min and 60ml/min are used for this experiment.
16. Repeat the steps from1-13 but with different reactor temperatures. It is suggested that the reaction be carried out at a minimum of three settings, say, 20oC, 25 oC and 30oC in order to plot the graph.
17. Plot 1/T vs. ln k Find E and K_0

Calculations

A. Determination of the rate constant using a tubular reactor

1. The calculations are best carried out using a spreadsheet such as EXCEL so that the results can be displayed in tabular and graphical form.
2. Enter the following known constants from the experiment. Ensure use of correct units.

F_a = Volumetric flow rate of sodium hydroxide (L/s)
F_b = Volumetric flow rate of ethyl acetate (L/s)
C_A = Sodium hydroxide concentration in feed vessel (mol/L)
C_B = Ethyl acetate concentration in feed vessel (mol/L)
C_C = Sodium acetate concentration in feed vessel (mol/L)
C_{A° = Sodium hydroxide concentration in mixed feed (mol/L)
C_{B° = Ethyl acetate concentration in mixed feed (mol/L)
C_{A1} = Sodium hydroxide concentration in reactor at time t (mol/L)
C_{B1} = Ethyl acetate concentration in reactor at time t (mol/L)
T = Reactor temperature (K)
V = Reactor volume (liter)

3. Using the spreadsheet, calculate the values of C_{A0}, C_{B0}, X_a and K from the following formulas:

$$C_{A0} = \frac{F_a}{F_a + F_b} . C_A \dots\dots\dots\dots\dots\dots.. (13)$$

$$C_{B0} = \frac{F_b}{F_a + F_b} . C_B \dots\dots\dots\dots\dots\dots.. (14)$$

$$X_a = \frac{C_{A^\circ} - C_{A1}}{C_{A^\circ}} \dots\dots\dots\dots (15)$$

$$K = \frac{(F_a + F_b\,)}{V} . \frac{(C_{A^\circ} - C_{A1})}{(C_{A1})^2} \dots\dots\dots\dots. (16)$$

B. Investigation the effect of throughput on conversion

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 $\frac{X_a}{1-X_a}$

C. Demonstrate the temperature dependence of the reaction and the rate constant

- Plot 1/T vs. ln k and comment on the graph obtained.
- Find E and K0
- Obtain the reaction rate constant in function of the temperature

References

1. Octave Levenspiel, ’’Chemical Reaction Engineering”, Second Edition, John Wiley and Sons, 1972.
2. Hovorika, R.B.and Kendall, H.B., Eng. Progr., 54(8), 58(1960).
3. Seader, J.D., and Sonthwick, L.M., Chem.Eng.Commun., P 175-183, (1981).

Tubular Reactor /Raw Data Sheet

A. Different concentration of feed materials:

Item	Run(1)	Run(2)	Run(3)
Sodium hydroxide concentration in the feed tank (mole/L)			
Ethyl acetate concentration in the feed tank(mole/liter)			
Flow rate of sodium hydroxide (ml/min)			
Flow rate of Ethyl acetate (ml/min)			
Temperature (°C)			

B. Different Flowrates of feed materials:

Item	Run(1)	Run(2)	Run(3)
Sodium hydroxide concentration in the feed tank (mole/L)			
Ethyl acetate concentration in the feed tank(mole/liter)			
Flow rate of sodium hydroxide (ml/min)			
Flow rate of Ethyl acetate (ml/min)			
Temperature (°C)			

C. Different reaction temperature

Item	Run(1)	Run(2)	Run(3)
Sodium hydroxide concentration in the feed tank (mole/L)			
Ethyl acetate concentration in the feed tank(mole/liter)			
Flow rate of sodium hydroxide (ml/min)			
Flow rate of Ethyl acetate (ml/min)			
Temperature (°C)			

Instructor's Signature:
Date:

Experiment Number -5-
Development of Kinetic Rate Equation from Hydraulic Analog Methods

---

Objectives

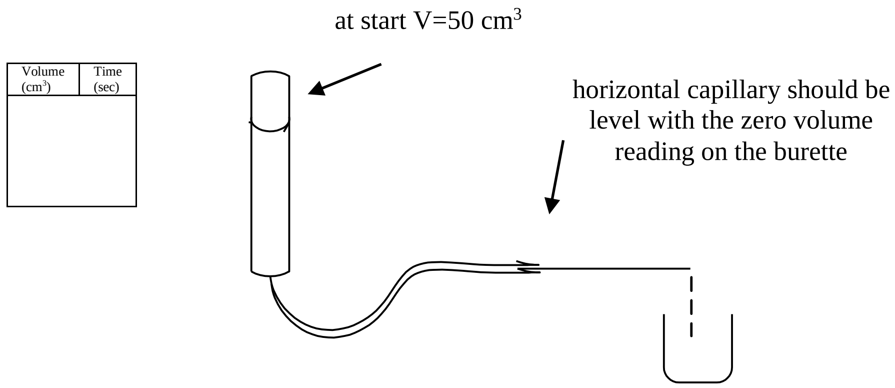
To develop kinetic rate equation of different reaction types and order in batch reactor using hydraulic analog methods.

Procedure

1. First order irreversible reaction

1. Connect a glass capillary to a burette as shown in Figure 1.
2. Fill the burette with water.
3. At time zero, let the water flow out and record the change in volume as time progresses.

This experiment represents first order reaction in a batch reactor in which the volume readings in burette in cm^3 is to be considered as a concentration of reactant in mol/m^3.



at start V=50 cm^3

horizontal capillary should be level with the zero volume reading on the burette

Figure 1. Experimental set up to represent the first order decomposition of reactant A, A→R.

Calculations:

Find the rate equation of the analog reactions

$$\mathbf{A \rightarrow R}$$

$-r_A = k\, C_A^n$, $n \approx 1$.

2. First order series reaction

1. Set up the arrangement shown in Figure 2.
2. Fill burette A with water.
3. At time zero, let the water flow out of both burettes A and R, and record the change in volume in both burettes (A and R) as time progress.

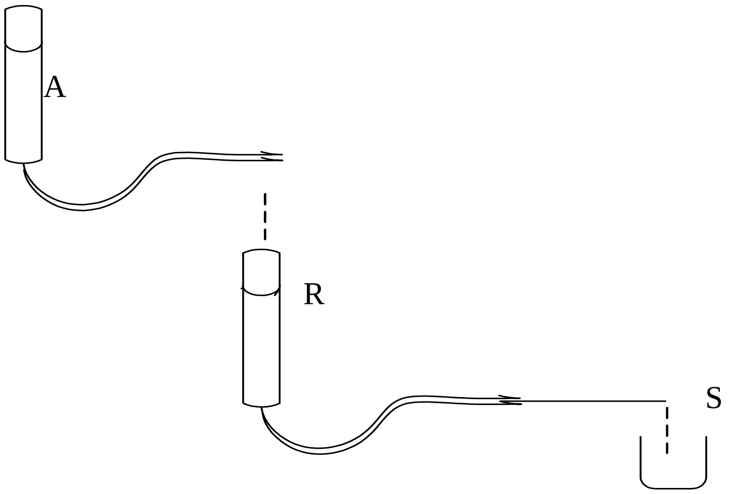


Figure 2. Experimental set up to represent the first order decomposition of reactant A, $A \rightarrow R \rightarrow S$.

Calculations:

The rate of the reaction constants (n,k) should be found for the analog reaction

$$\mathbf{A} \xrightarrow{k_1} \mathbf{R} \xrightarrow{k_2} \mathbf{S}$$

$-r_A = k_1 C_A^m$ and $r_S = k_2 C_R^n$, $m \approx n \approx 1$.

3. First order reversible reaction

1. Set up the arrangement shown in Figure 3 nothing that the level of capillary is above the zero point of the burette.
2. Fill burette A with water.
3. At time zero, let the water flow out and record the change of volume with time.

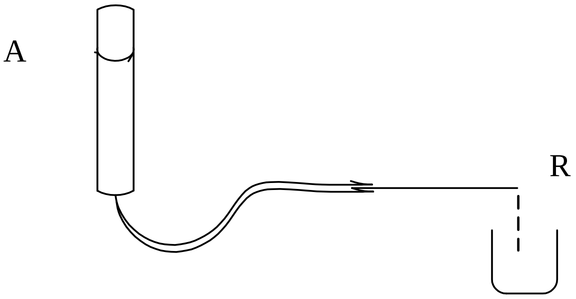


Figure 3. Experimental set up to represent the first order decomposition of reactant A, $A \leftrightarrow R$.

Calculations:

Find the rate equation of the analog reactions

$$A \leftrightarrow R$$

$-r_A = k_1 C_A - k_2 C_R$.

4. ***Reactions with order not equal to one (Non-linear reactions)***

a. ***Reaction order n<1***

1. Connect a glass capillary to a funnel as shown in Figure 4.
2. Fill the funnel with water.
3. At time zero, let the water flow out and record the change of volume with time.

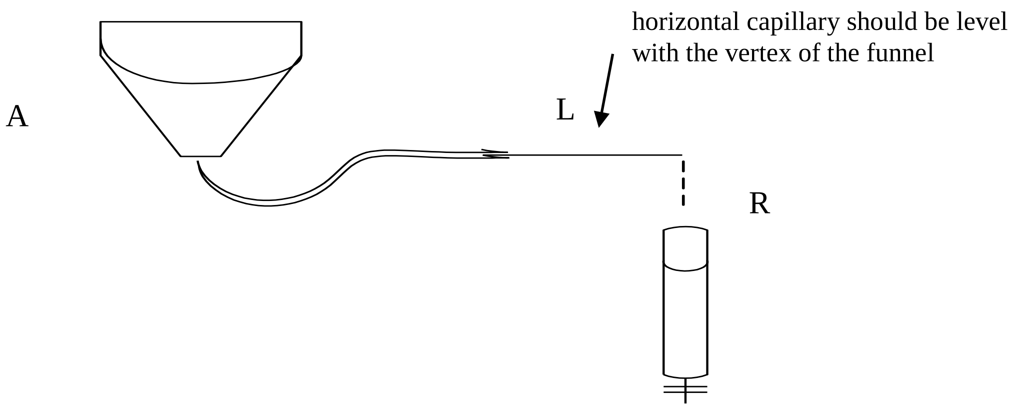


Figure 4. Experimental set up to represent the first order decomposition of reactant A, for non linear rxns n<1.

Calculations:

Find the rate equation of the analog reactions

$-r_A = k\, C_A^n$, $n<1$

b. ***Reaction order n>1***

1. Set up the arrangement shown in Figure 5.
2. Fill the funnel with water.
3. At time zero, let the water flow out and record the change of volume with time.

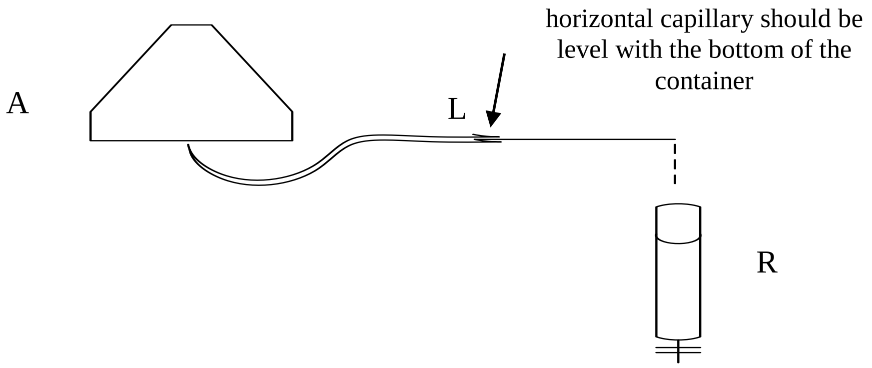


Figure 5. Experimental set up to represent the first order decomposition of reactant A, for non linear rxns n>1.

Calculations:

Find the rate equation of the analog reactions

$-r_A = k\, C_A^n$, n>1

References

Octave Levenspiel, “Chemical Reaction Engineering”, Second Edition, John Wiley and Sons, 1972.

Guo-Tai and Shan-Drag H. , Chem. Eng. Educ. Winter 1984, p. 10. Spring 1984, p. 64.

Development of Kinetic Rate Equation from Hydraulic Analog Methods Data Sheet

Time	Reaction Volume	Reaction Volume	Reaction Volume	Reaction Volume	Reaction Volume

Experiment Number -6-
Level Control

Objectives

1. To become familiar with the coupled tank apparatus.
2. To investigate the steady state and transient performance of the coupled tank apparatus under proportional and proportional plus integral control.

Introduction

Very often a physical system can be represented by several first order processes connected in series. Consider the basic coupled tank apparatus. The equations describing the dynamic behaviour of the system can be derived by considering flow balances about each tank, assuming that the inter-tank holes and drain tap behave like orifices. By taking the Laplace transforms of the equations, the following transfer function is obtained:

$$\frac{h_2(s)}{q_i(s)} = \frac{1lk_2}{(\tau_1 s + 1)(\tau_2 s + 1)} \qquad (1)$$

Where h_1 is the level in tank 2 and q_i is the input flow rate. The time constants τ_1 and τ_2 are obtained by solving the equations:

$$\tau_1 \tau_2 = \frac{A^2}{k_1 k_2} \qquad , \tau_1 + \tau_2 = \frac{A(2k_1 + k_2)}{k_1 k_2} \qquad (2)$$

$$k_1 = \frac{cd_1 a_1 \sqrt{2g}}{2\sqrt{H_{1s} - H_{2s}}} \qquad (3)$$

$$k_2 = \frac{cd_2 a_2 \sqrt{2g}}{2\sqrt{H_{2s} - H_3}} \qquad (3')$$

Where,

A = cross sectional area of either tank 1 or 2.

Cd_1,Cd_2 = discharge coefficient of the orifice (approx.0.6)

a_1 = cross sectional area of orifice 1(the area of the open holes between the two tanks).

a_2 = cross sectional area of orifice 2 (the drain tap).

H_{1s},H_{2s} = steady state height of fluid in tank 1 and tank 2.

H_3 = height of drain tap (3 cm).

The second order transfer function given by eqn.(1) describes the variations in the input flow rate q_i (s). A similar, small signal transfer function can be found which relates h_1(s) -the level of fluid in tank 1- to small variation in the input flow rate q_i(s); It is:

$$\frac{h_1(s)}{q_i(s)} = \frac{\frac{k_1 + k_2}{k_1 k_2} + \frac{A}{k_1 k_2} s}{(\tau_1 s + 1)(\tau_2 s + 1)} \tag{4}$$

The coupled tank apparatus can be converted to a first order system by removing all the bungs from the inter-tank partition. This makes a1 in eqn.(3) relatively large, so that (1/k1) is approximately zero. Using this approximation in Eqn. (1) gives the small signal transfer function for the coupled tank with all bungs removed as:

$$\frac{h_2(s)}{q_i(s)} = \frac{1/k_2}{(\tau s + 1)} \tag{5}$$

Where τ is given by:

$$\tau = \frac{2A}{k_2} \tag{6}$$

Description of Apparatus

The coupled tank apparatus consists of a transparent plexi-glass container measuring approximately 20 cm by 10 cm deep by 30 cm high. A centre partition is used to divide the container into two tanks. Flow between the tanks is by means of a series of holes drilled at the base of the partition. Three holes having diameters of 1.27 cm, 0.95 cm and 0.635 cm are situated 3 cm above the base of the tank. A smaller (bleed) hole of 0.317 cm diameter is situated at a height of 1.5cm. These holes constitute orifice 1 of cross sectional area a1. Water is pumped from a reservoir into the first tank by a variable speed pump which is driven by an electrical motor. The pump motor drive is normally derived from an analogue computer. The actual flowrate is measured by a flow meter which is in the flow line between the pump and tank 1. The depth of the fluid is measured using depth sensors which are stationed in tank 1 and tank 2. This device performs as an electrical resistance which varies with the water level. The change in resistance are detected and provide an electrical signal which is proportional to the height of water. The water which flows into tank 2 is allowed to drain out via an adjustable tap, and the entire assembly is mounted in a large tray which also forms the supply reservoir for the pump. Fully open, the drain tap has a diameter of 0.7cm.

Procedure

1. **Measurement of system characteristics**
   a. **Stead State Characteristic of Final Control Element (Pump)**

   Apply a small positive voltage to the pump drive socket using one of the potentiometers of the analogue computer, and read off the corresponding flow rate from the flowmeter. Increase the voltage in increments of about 0.5 volts and record the corresponding flow rates. Continue until the maximum flowrate is reached.

   Requirement: plot the flowrate versus voltage applied to obtain the pump characteristic (Gp).

   b. **Steady State Characteristic of Measuring Device (Level Sensor)**

   Close the drain tap on tank 2 and run the pump until the tanks are both full. Switch the depth sensor filters in circuit and attach the tank 2 depth output terminal to recorder and record the fluid level in tank 2 and the voltage reading. Open the drain tap and allow the water level to fall one centimeter, record the new fluid level and the voltage reading. Repeat the exercise until the tank is empty.

   Requirement: plot the voltage reading versus the fluid level in tank 2 to obtain the depth sensor characteristics(Gd).

   c. **Dynamic Characteristics**

   Open the holes of 0.95 cm and 0.635 cm diameters and close the bleed hole and hole of 1.27 cm diameter. Open the drain tap fully and run the pump to give a flowrate of 1850 cc/min and record the steady state levels(H1s,H2s). Repeat the experiment with another flowrate(2020 cc/min).

   Requirements: Determine

   $cd_1a_1\sqrt{2g}$, $cd_2a_2\sqrt{2g}$

   Using the following equations:

   $$cd_1a_1\sqrt{2g} = \frac{Oi}{\sqrt{H_{1s-}H_{2s}}} \qquad (7)$$

   $$cd_2a_2\sqrt{2g} = \frac{Qi}{\sqrt{H_{2s-}H_3}} \qquad (8)$$

   Derivable from the relationships for the orifices.

   Calculate k_1 and k_2 using Eqn.(3).

   Calculate $\tau 1$ and $\tau 1$ using Eqn. (2).

   Evaluate the system transfer functions, Eqn.(1) and (4).

   d. **Steady State Operating Levels.**

   Open the drain tap fully and close all except the two holes of 0.635 cm and 0.95 cm diameter. Apply a suitable drive voltage to the pump to obtain a flowrate of

1000cc/min. allow time for the steady state to be reached and measure the tank levels H1s and H2s. Repeat the exercise for flow rates of 1850cc/min and 2020cc/min.

Requirement: compare the steady state level in tank 1 predicted by the equation;

$$H_{1s,th} = H_{2s}(1+(a_2/a_1)^2)-H_3(a_2/a_1)^2 \qquad (9)$$

with the experimental results.

2. Design of Proportional and Integral Controllers

a. Steady State Errors Using Proportional Control

Open the drain tap fully and plug all except the two holes of 0.635 cm and 0.95 cm diameter. Switch the depth sensor filter in circuit. Use the analogue computer to apply a proportional feedback control loop for the fluid level in tank 2; see Fig.(1). Set the desired level in tank 2 corresponding to sensor signal of approximately 6 volts and record the steady state error for constant reference input and values of proportional gain Kp ranging from 10 to 100.

Requirement: Calculate the theoretical values of the steady state errors using the equation below and compare them with the experimental ones.

Experimental steady state error:

ess,exp= required level- H2s,exp= Yr-H2s,exp *(10)*

Theoretical steady state error is calculated from equation:
ess,th= Yr-H2s,th *(11)*

H2s,th can be evaluated by solving the following equations;

$$cd_1 a_1 \sqrt{2g} = \frac{Oi}{\sqrt{H_{1s,th} - H_{2s,th}}} \qquad (12)$$

$$H_{1s,th} = H_{2s,th}(1+(a_2/a_1)^2)-H_3(a_2/a_1)^2 \qquad (13)$$

b. Steady State Error Using Proportional Plus Integral controllers

Use the analogue computer to apply proportional plus integral feedback control to the fluid level in tank 2; see Fig.(2). Set the proportional gain at a value of 10. Slowly turn up the integral gain at the values of (1, 1.5 and 2) and observe the steady state errors decay to zero.

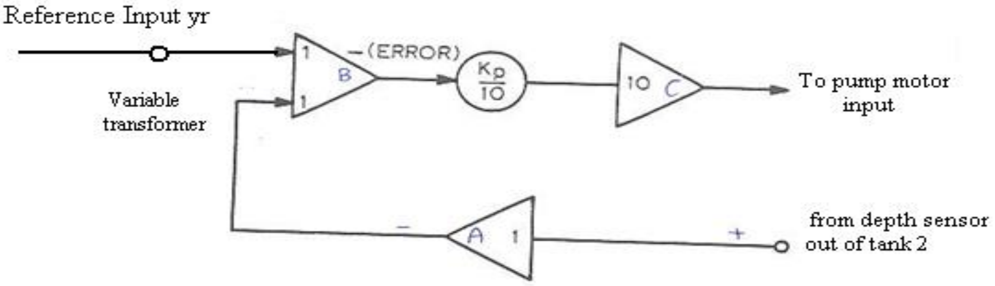


Fig. (1): Patching diagram for Proportional controller

Start from
this point

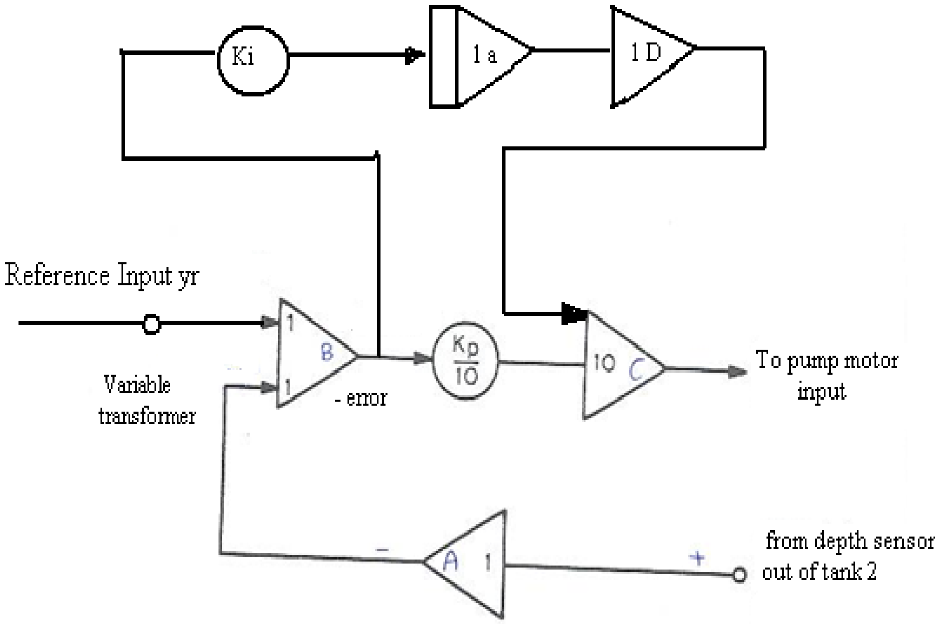


Fig.(2) : Patching diagram for Proportional and Integral controller

Start from
this point

Level Control Data Sheet

Measuring Device calibration:

H_2 (cm)	H_2 (V)

Pump calibration:

Flowrate (ml/min)	Voltage (V)

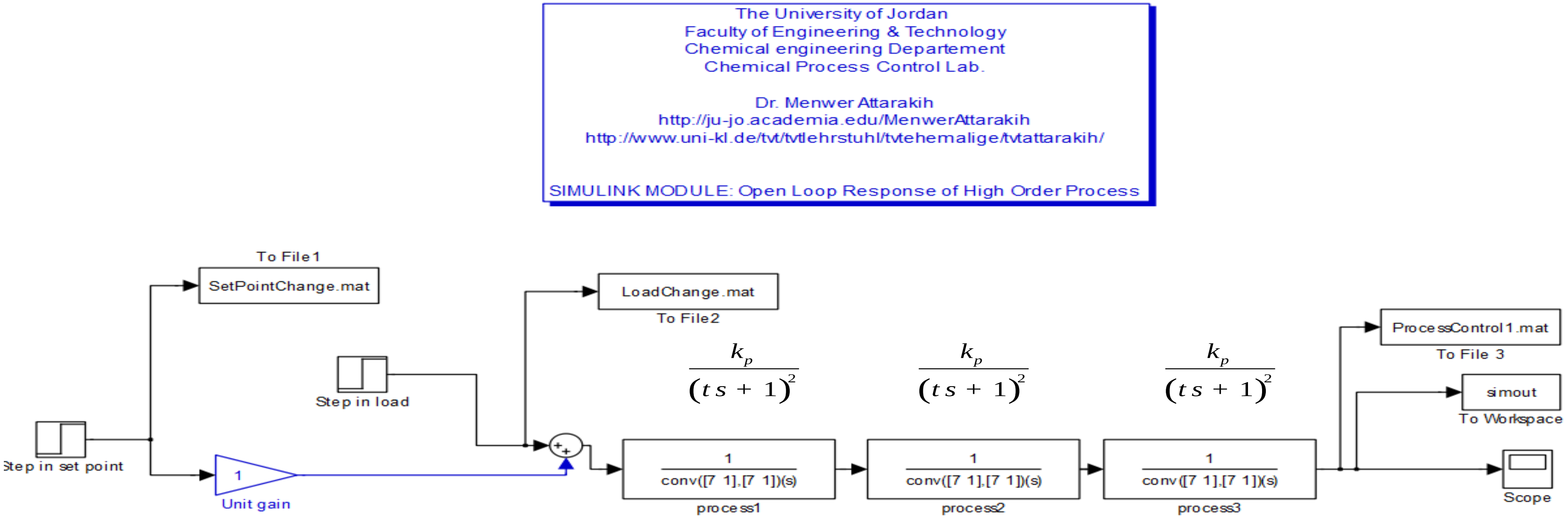
Proportional Controller:

Set point=			
Time	**K_p =**	**K_p =**	**K_p =**

Proportional and Integral Controller:

Set point=			
K_p =			
Time	**τ_i =**	**τ_i =**	**τ_i =**

Experiment Number -7-
Simulink Module



The University of Jordan
Faculty of Engineering & Technology
Chemical engineering Departement
Chemical Process Control Lab.

Dr. Menwer Attarakih
http://ju-jo.academia.edu/MenwerAttarakih
http://www.uni-kl.de/tvt/tvtlehrstuhl/tvtehemalige/tvtattarakih/

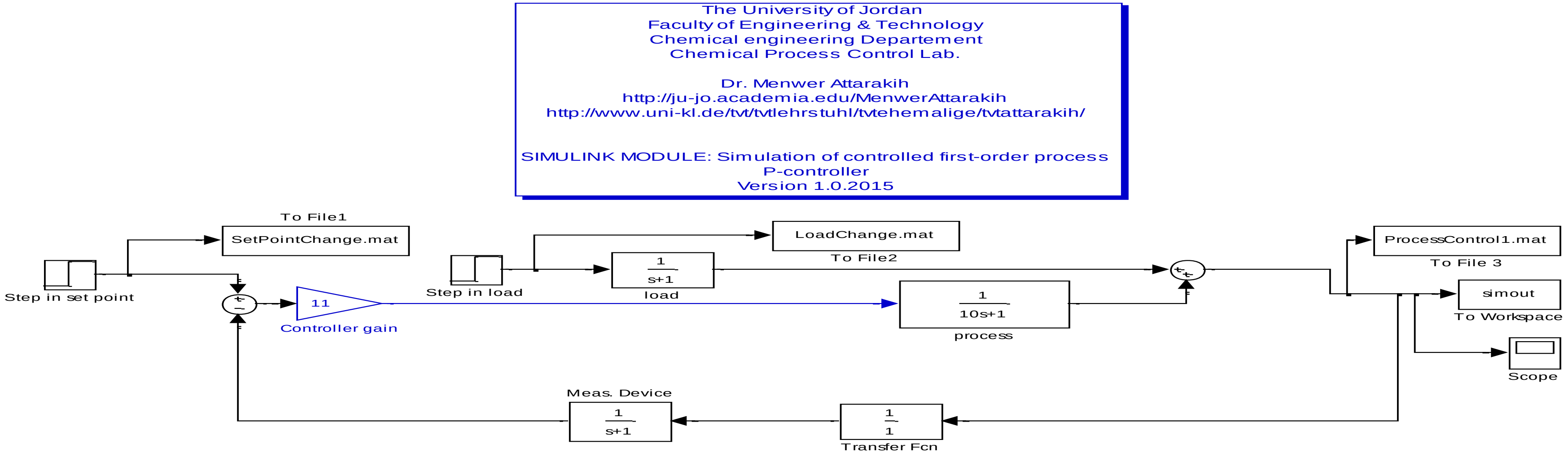
SIMULINK MODULE: Open Loop Response of High Order Process

Procedure:

1. Run the SIMULINK module: **ResponseHighOrderProcessOpenLoop.mdl** to open the above flowsheet.
2. Set $\tau = 7$, $k_p = 1$ & introduce a unit step change in the load (double click the Step in load icon) and run the SIMULINK model using final simulation time: 200 units of time.
3. Double click the scope to see the recorded output response.
4. Run the file: FSIMULINKplot.m to plot the recorded output and save the MATLAB figure in a specified directory.
5. Save the excel file: SIMULINKResults.xls to a specified directory if you wish to plot the recorded output using Microsoft excel.

Required:

1. Fit the recorded response to a First-order plus dead time model (FOPD) and estimate its three parameters (k_p, t_d & τ) .
2. Compare the response of the FOPD to the original response of the high order process on the same plot.
3. Comment on your results.



The University of Jordan
Faculty of Engineering & Technology
Chemical engineering Departement
Chemical Process Control Lab.

Dr. Menwer Attarakih
http://ju-jo.academia.edu/MenwerAttarakih
http://www.uni-kl.de/tvt/tvtlehrstuhl/tvtehemalige/tvtattarakih/

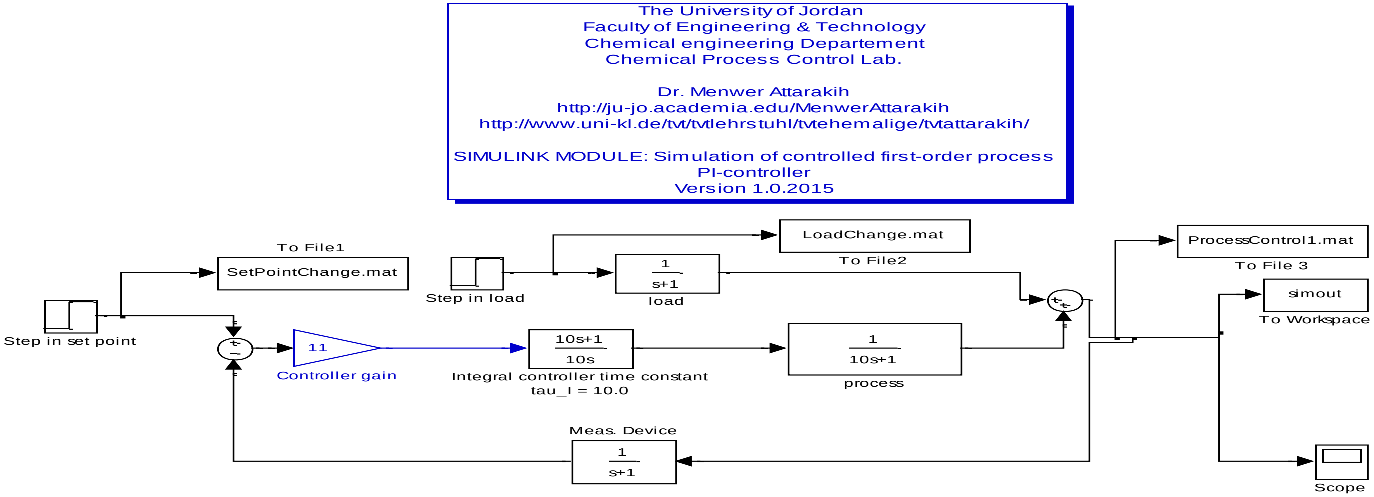
SIMULINK MODULE: Simulation of controlled first-order process
P-controller
Version 1.0.2015

Procedure:

1. Run the SIMULINK module: **ProcessControlFirstOrderPcontroller.mdl** to open the above flowsheet.
2. Set $\tau_p = 10$, $k_p = 1$ and the other transfer functions as shown in the figure & introduce a unit step change in the load (double click the Step in load icon) and run the SIMULINK model using final simulation time: 25 units of time.
3. Double click the scope to see the recorded output response.
4. Run the file: FSIMULINKplot.m to plot the recorded output and save the MATLAB figure in a specified directory.
5. Save the excel file: SIMULINKResults.xls to a specified directory if you wish to plot the recorded output using Microsoft excel.

Required:

1. Increase the controller gain starting with Kc = 1 and increase it by 1 until the offset becomes less than 1%.
2. Calculate the percentage of offset at each value of Kc in step 1 and plot the percentage of offset as function of Kc.
3. Comment on your results.



The University of Jordan
Faculty of Engineering & Technology
Chemical engineering Departement
Chemical Process Control Lab.

Dr. Menwer Attarakih
http://ju-jo.academia.edu/MenwerAttarakih
http://www.uni-kl.de/tvt/tvtlehrstuhl/tvtehemalige/tvtattarakih/

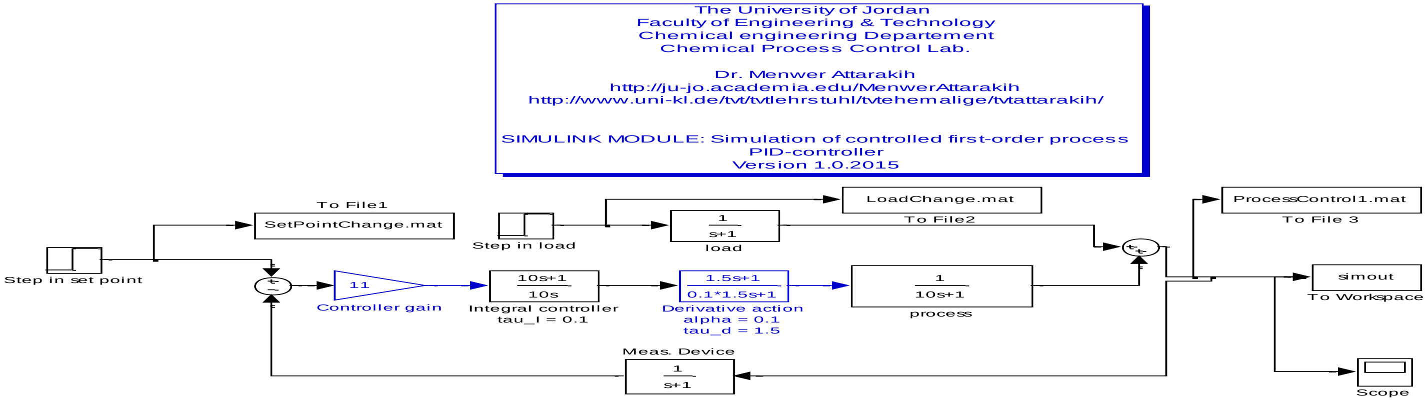
SIMULINK MODULE: Simulation of controlled first-order process
PI-controller
Version 1.0.2015

Procedure:

1. Run the SIMULINK module: **ProcessControlFirstOrderPIcontroller.mdl** to open the above flowsheet.
2. Set $\tau_p = 10$, $k_p = 1$ and the other transfer functions as shown in the figure & introduce a unit step change in the set point(double click the Step in set point icon) and run the SIMULINK model using final simulation time: 200 units of time.
3. Double click the scope to see the recorded output response.
4. Run the file: FSIMULINKplot.m to plot the recorded output and save the MATLAB figure in a specified directory.
5. Save the excel file: SIMULINKResults.xls to a specified directory if you wish to plot the recorded output using Microsoft excel.

Required:

1. Did the Integral control action eliminate the offset? Why?
2. At constant τ_I =10.0 increase the controller gain starting with Kc = 1 until the closed loop response oscillates continuously. What is the physical significance of this controller gain? When the system will become unstable?
3. Repeat step 2 using at least four different integral time constants. Present your results in a table and discuss your results.
4. Repeat step 1 to 3 using only a unit step change in the load.

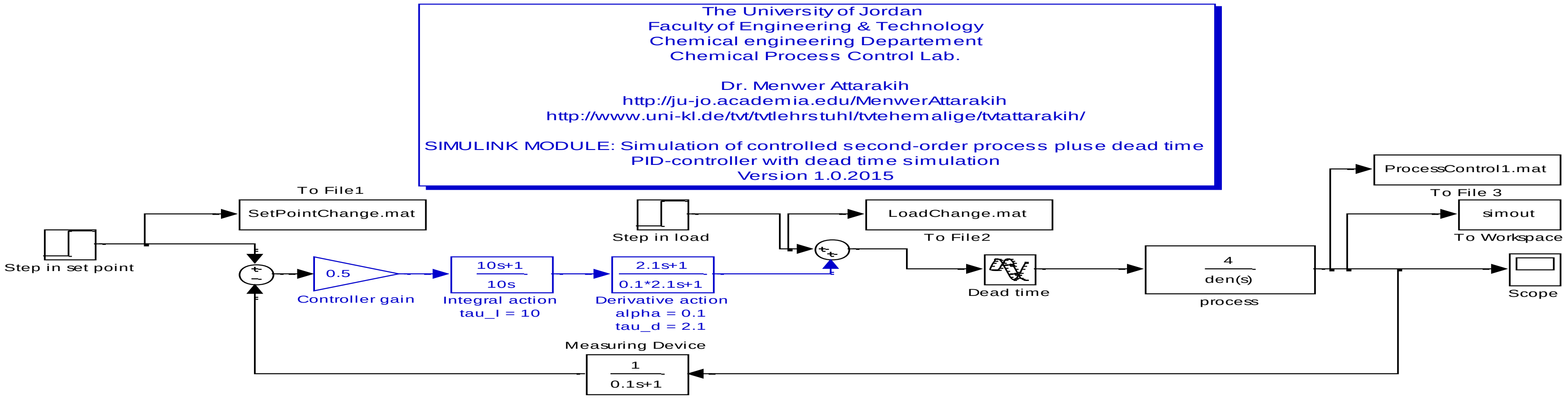


Procedure:

1. Run the SIMULINK module: **ProcessControlFirstOrderPIDcontroller.mdl** to open the above flowsheet.
2. Set τ_p = 10, k_p = 1 and the other transfer functions as shown in the figure & introduce a unit step change in the set point(double click the Step in set point icon) and run the SIMULINK model using final simulation time: 200 units of time.
3. Double click the scope to see the recorded output response.
4. Run the file: FSIMULINKplot.m to plot the recorded output and save the MATLAB figure in a specified directory.
5. Save the excel file: SIMULINKResults.xls to a specified directory if you wish to plot the recorded output using Microsoft excel.

Required:

1. What will be the expected effect of the PID controller action?
2. At constant τ_I =10.0 and τ_D =1.5 increase the controller gain starting with Kc = 1 until the closed loop response oscillates continuously. What is the physical significance of this controller gain? When the system will become unstable?
3. Repeat step 2 using at least four different integral and derivative time constants. Present your results in a table and discuss it.
4. Repeat step 1 to 3 using only a unit step change in the load.
5. With the process under the PI control action at τI =10.0 and under continuous oscillation, simulate the closed loop using the PID controller. Record your observations & discuss the results.



The University of Jordan
Faculty of Engineering & Technology
Chemical engineering Departement
Chemical Process Control Lab.

Dr. Menwer Attarakih
http://ju-jo.academia.edu/MenwerAttarakih
http://www.uni-kl.de/tvt/tvtlehrstuhl/tvtehemalige/tvtattarakih/

SIMULINK MODULE: Simulation of controlled second-order process pluse dead time
PID-controller with dead time simulation
Version 1.0.2015

Procedure:

1. Run the SIMULINK module: **ProcessControlPIDcontrollerSecondOrderWithTimeDelay.mdl** to open the above flowsheet.
2. Set $\tau_p = 7$, $k_p = 4$ and the other transfer functions as shown in the figure & introduce a unit step change in the set point(double click the Step in set point icon) and run the SIMULINK model using final simulation time: 300 units of time.
3. Double click the scope to see the recorded output response.
4. Run the file: FSIMULINKplot.m to plot the recorded output and save the MATLAB figure in a specified directory.
5. Save the excel file: SIMULINKResults.xls to a specified directory if you wish to plot the recorded output using Microsoft excel.

Required:

1. What will be the expected effect of the dead time (time delay) on the controlled response?
2. At constant $\tau I = 10.0$ and $\tau D = 2.1$ $Kc = 0.5$ increase the time delay starting with $td = 0.25$ until the closed loop response oscillates continuously. What is the physical significance of this behavior?
3. Repeat step 2 using at least four different integral and derivative time constants. Present your results in a table and discuss it.
4. Repeat steps 1 to 3 using only a unit step change in the load.

Experiment Number -8-
Empirical Dynamic Models

Objective: To model a black box process (e.g. continuous stirred tank heater) using the process step testing method (process reaction curve).

Equipment: See the attached sheet.

1. Theory:

Empirical (*Black box*) models do not describe the physical phenomena of the process, they are based on input/output data and only describe the relationship between the measured input and output data of the process. These models are useful when limited time is available for model development and/or when there is insufficient physical understanding of the process. To generate such empirical models, the step test procedure is carried out as follows:

1. With the loop is opened (the controller is on manual mode), apply a step change in the controller output signal $M(t)$ to the process (see Fig.(1)). The magnitude of the change should be large enough for the consequent change in the transmitter signal to be measurable, but it should not be so large that the response will be distorted by the process nonlinearities. During the step testing there should be no disturbances affect the process.
2. The response of the process $C(t)$ is recorded on a strip chart recorder or equivalent device, making sure that the resolution is adequate in both the amplitude and the time scale. The resulting plot of $C(t)$ versus time has a sigmodal shape and is known as the process reaction curve. The response must cover the entire test period from the introduction of the step test until the system reaches a new steady state.
3. Match the process reaction curve to a FOPDT (First-Order Pluse Dead Time) response.

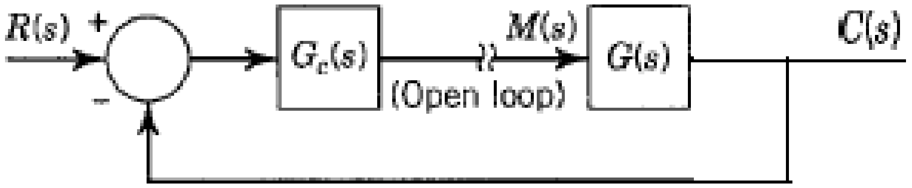


Fig.(1): Block diagram for a typical open loop to generate the process reaction curve.

The FOPDT transfer function is given by:

$$C(s) = \frac{K_p e^{-t_0 s}}{t_p s + 1} . \frac{\text{D} m}{s} \qquad (1)$$

The above equation can be inverted back to the time domain:

$$C(t) = K_p \times \Delta m \times u(t - t_0)\left(1 - e^{-\frac{(t - t_0)}{\tau_p}}\right) \qquad (2)$$

Where $C(t) = c(t) - c_0$ is the output variable in deviation form.

The three model parameters are estimated from the process reaction curve as follows:

$$K_p = \frac{\Delta c}{\Delta m} \text{ at steady state} \qquad (3)$$

The dead time (t_0) and the process time constants (τ_p) are estimated from Fig.(2) below:

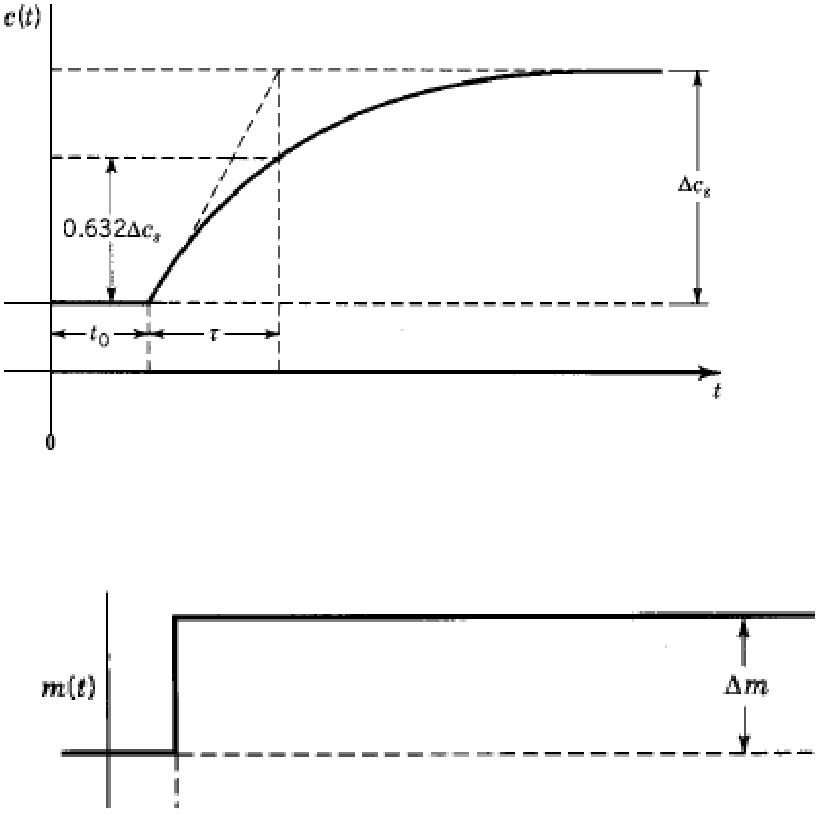


Fig.(2): Method 1 estimation of the dead time and the process time constant from the process reaction curve.

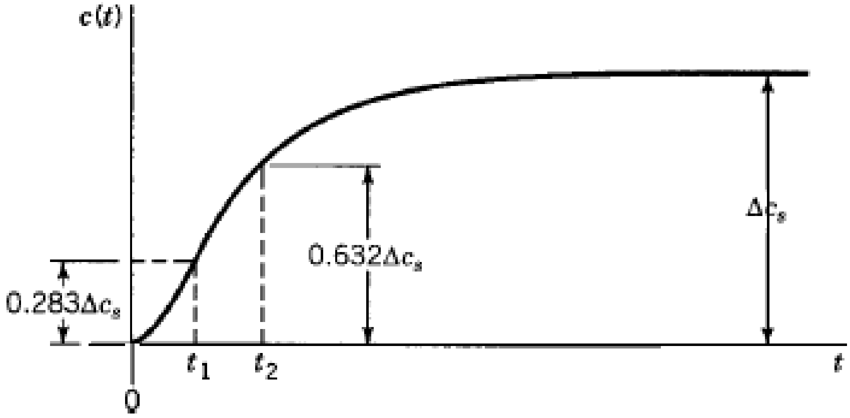


Fig.(3): Method 2 estimation of the dead time and the process time constant from the process reaction curve.

By using method 2 given in Fig.(3) the process time constant and the dead time are estimated as follows:

$$t_p = \frac{3}{2}(t_2 - t_1) \tag{4}$$

$$t_0 = (t_2 - t_p) \tag{5}$$

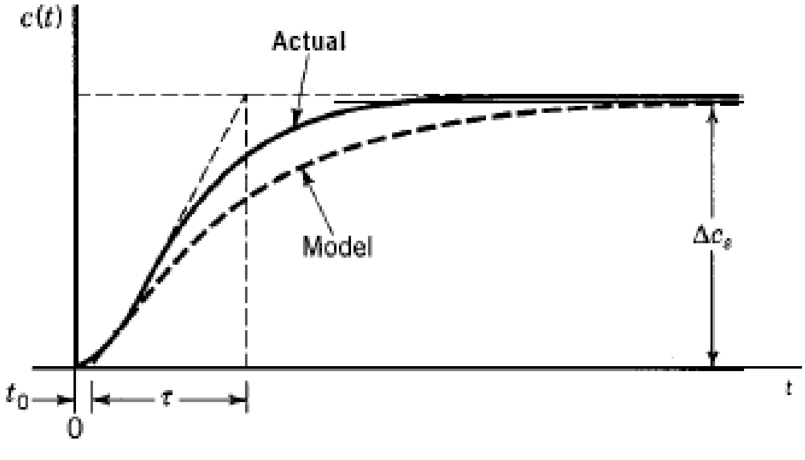


Fig.(4): Method 3 estimation of the dead time and the process time constant by drawing a slope to the process reaction curve passing through the inflection point.

Method 3 uses the inflection point of the process reaction curve to estimate the FOPDT model parameters as shown in Fig (4).

2. Calculations:

1. Use methods 1, 2 and 3 to estimate the three parameters of the FOPDT model and list them as shown in table 1:
2. Plot the resulting models using Eq.(2) along with the experimental data on the same graph.

Table 1: Comparison between different methods for fitting the process reaction curve to FOPDT model.

Method	1	2	3
K_p ( )			
t_0 ( )			
t_p ( )			

3. Comment on the results in (1) and (2).

References:

1. Considine, D. M. (1974). Process instruments and control handbook. McGraw-Hill, New York.
2. Seborg, D. E. & Edgar, T. F. (1989). Process dynamics and control, John Wiley & Sons, New York.

Empirical Dynamic Models Data Sheet

1. Impulse response:

Initial pure water conductivity:

Injection Solution conductivity:

Time(min)	Conductivity (µS/cm)	Time(min)	Conductivity (µS/cm)

2. Step up response:

Initial tank conductivity:

Initial solution tank conductivity:

Time(min)	Conductivity (µS/cm)

3. Step down response:

Initial tank conductivity:

Initial water tank conductivity:

Time(min)	Conductivity (μS/cm)	Time(min)	Conductivity (μS/cm)

Experiment Number -9-
Process Control

Apparatus

The Process Control Trainer is composed of two major sections:

A. Process panel
   - The Process panel consists of a water sump tank, a transparent process tank, different sensors and actuators.
   - The associated front panel shows the process diagram using standard symbols.
   - The Process panel has two cables that connect to the Control panel:
     - Heavy duty cable - with rectangular 10 pin plugs
     - Standard parallel cable - with rectangular 25 pin plugs

B. Control panel
   - It creates the power and information link with the process.
   - It allows designing different control scenarios.
   - The control panel is connected to the power source:
     - Standard Computer Power Cable

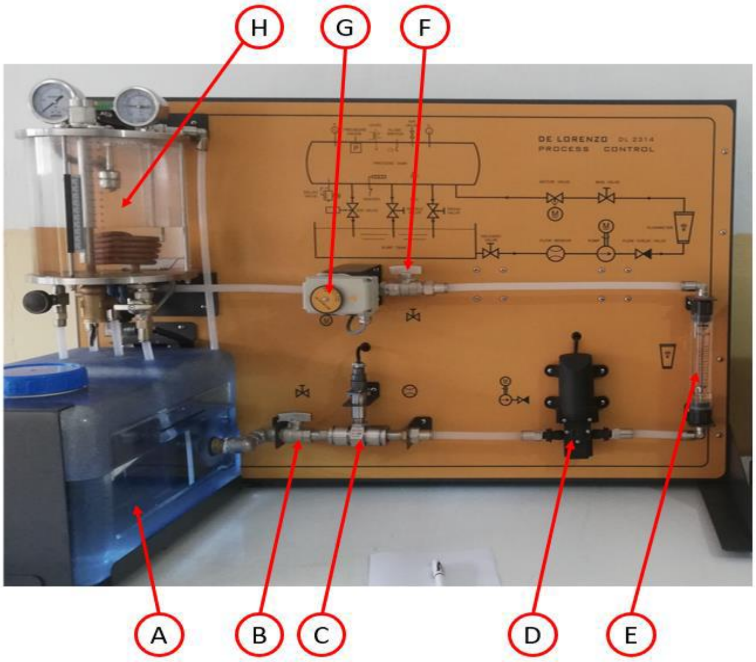


Figure (1): Process Panel

Process unit description

A. **The sump tank**: a non-pressurized reservoir with a big top round lid for water filling. The sump tank has more pipelines fitting in. The metal sided horizontal pipe is for process water supply, while the five top side vertical plastic pipes are for water draining out from the process.

B. **Delivery valve:** when switched off, no water will drain out from the sump tank to the process. We can call it the main water supply valve.
C. **Turbine Flow Meter:** it is a volumetric measuring turbine type. The flowing fluid engages the rotor causing it to rotate at an angular velocity proportional to the fluid flow rate.
D. **Motor pump:** it is a positive displacement pump that utilizes two flexible diaphragms that reciprocate back and forth, creating a temporary chamber, which both draws in and expels fluid through the pump. The pump is powered by a DC motor with 12 V nominal voltage (it may run between 9 and 14V). Note: BBRG, Thermal protected.
E. **Visual Flow meter:** In-line flow meters are indicators for flow rate. This type of flow meters is ideal for measurement of a range of compatible oil, or water-based liquids.
F. **Man valve:** it is a manually powered valve used for reducing the water flow to the transparent tank. It can be used together with the motor valve for controlling the inlet flow.
G. **Motor valve:** it s a 10 V electro-modulated valve used for controlling the water flow to the transparent process tank.
H. **Process tank:** it is a cylindrical transparent high-pressure tank with hermetical enclosure. The process tank is equipped with different analog and visual measuring devices and also releasing valves:
    a) **Sol valve:** solenoid control valves are electromagnetic plunger valves that control the flow rates of liquids or gases. They open with certain stroke positions – dependent on the valve control signal.
    b) **Drain valve:** it is a manually operated valve for draining the water from the transparent process tank.
    c) **Relief valve:** it is a pressure safety valve (PSV) that has a manual lever to activate the valve in case of emergency. It is commonly used to control or limit the pressure in a system.
    d) **Needle valve:** it is a type of valve with a small port and a threaded, needle-shaped plunger. It allows a precise regulation of the flow, although it is generally only capable of relatively low flow rates.

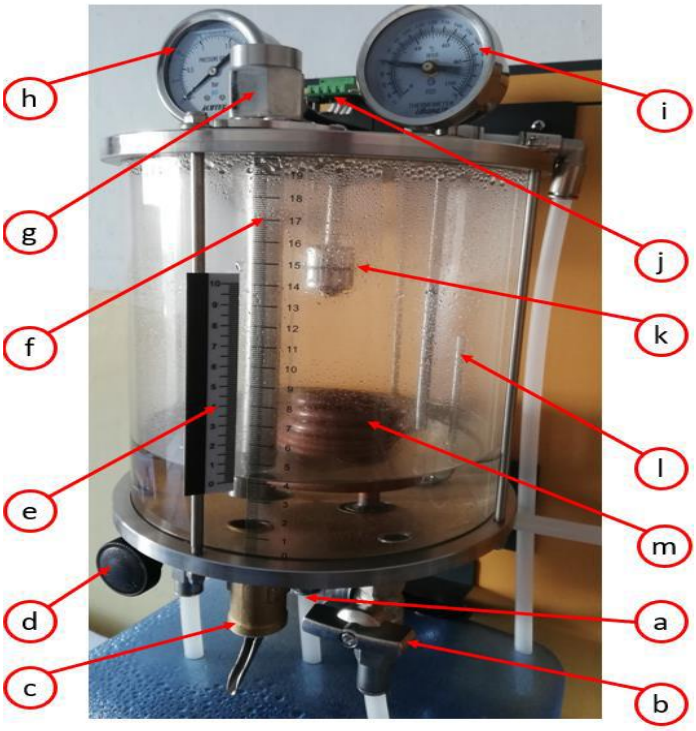


Figure (2): Process tank

e) **Mobile scale:** it is a reference metric scale for measuring the level of the water in centimeters and millimeters. It can be moved up or down on a metallic guideline.
f) **Fixed scale:** it is an engraved metric scale on the transparent tank for measuring the level of the liquid inside.
g) **Level sensor:** it is a capacitive sensor mounted inside the process tank. The increase in water level causes a deeper immersion of the measuring electrode and thereby it increases its capacity. The measured capacity sets the output of the level meter.
h) **Pressure gauge:** it is a device used for measuring the pressure of the compressed air inside the process tank. It can measure the pressure in bar or psi.
i) **Thermometer:** the thermometer is a tool that measures the temperature inside the process tank. Here, it is used to measure the water temperature in Celsius or Fahrenheit degrees.
j) **Pressure sensor:** it is an electronic pressure measurement system. It converts a physical pressure value into an analogue electrical signal by accurately measuring the deflection of a pressure sensitive diaphragm and providing a high-speed proportional output signal that can interface directly with a centralized data monitoring/acquisition system or a local readout or display.
k) **Float switch:** it is a type of level sensor, a device used to detect the level of a liquid within a tank. The switch may be used to control a pump, as an indicator, an alarm, or to control other devices.

l) **Temperature sensor:** it is a Pt100 sensor used to measure the temperature. It is a type of sensor that falls into a group called Resistance Temperature Detectors or RTD's.

m) **Heating element**: it is an immersion copper tubular heater element powered by a 12-48 V source.

Control unit description

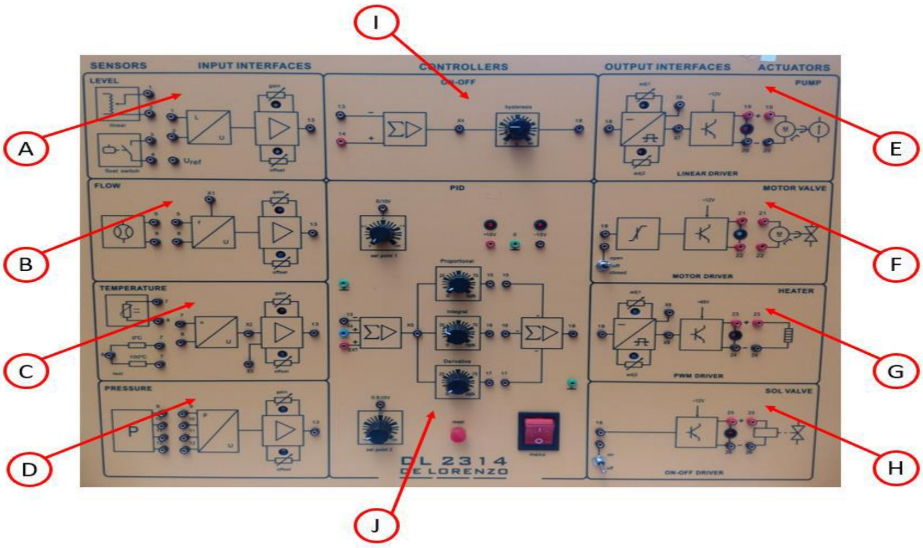


Figure (3): Control Panel

A. Level transducer
B. Flow transducer
C. Temperature transducer
D. Pressure transducer
E. Pump
F. Motor valve
G. Heater
H. Sol valve
I. On/Off control
J. PID control

Experiment 1-Level Control-

Experiment A: Level Sensor Settings

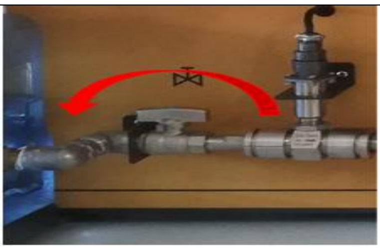
Objectives:

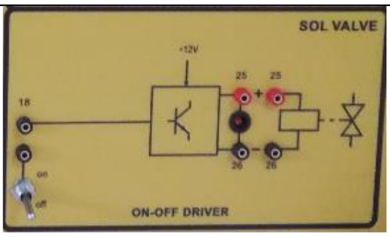
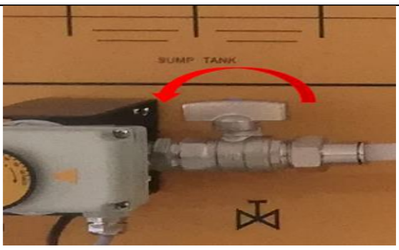
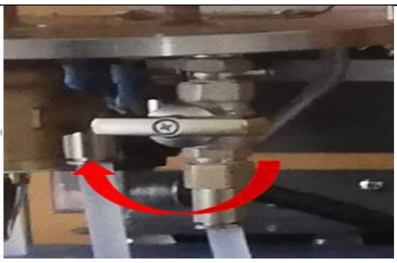
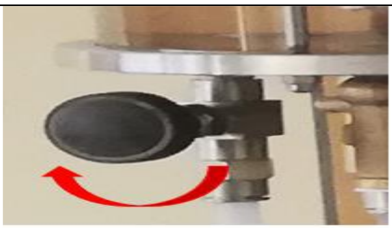
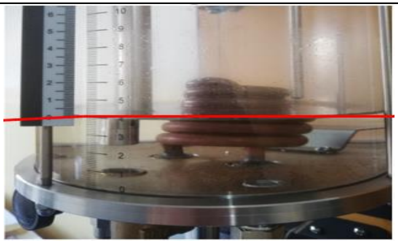
1. To understand the working principle of a capacitive level sensor in order to measure the liquid level in an industrial process tank.
2. To learn how to calibrate the level sensor.
3. To measure the water level in the transparent process tank.
4. To obtain the characteristic curves of the capacitive level sensor submersed in the process tank filled with water.

Procedure:

The following steps should be checked before starting to configure the system:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the Process panel as follows:

CONTROL	FIGURE	STATE	ACTION
DELIVERY VALVE		fully open	Turn the knob counter-clockwise
MOTOR VALVE		fully open	Angular rotation = open

SOL Valve		closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE		fully open	Turn the knob counter-clockwise
DRAIN VALVE		fully closed	Turn the knob clockwise
NEEDLE VALVE		fully closed	Turn the knob clockwise
AIR VALVE		fully open	Turn the knob counter-clockwise
Level in the PROCESS TANK		0 cm of the mobile scale	No water in the transparent process tank

4. Make sure that all the connections of the two DL 2314 PROCESS CONTROL TRAINER units are correct (power cord, heavy duty cable, standard parallel cable).
5. Switch ON the hardware devices from the POWER button on

6. Press the main switch (ON) to power up the experiment
7. Start the pump regulating the voltage on SET POINT 1 and bring the level of the water in the PROCESS TANK to 2 cm of the mobile scale.
8. Once reached the 2 cm level, stop the pump by setting to 0 V the voltage value of SET POINT 1.
9. Gradually decrease the level of the water in the tank by turning counter-clockwise the knob NEEDLE VALVE and/or the knob DRAIN VALVE, back to 0 cm of the mobile scale.
10. Check that the voltage value, read on the voltmeter, corresponds to 0 V, otherwise adjust to obtain the desired value: in this way, you have fixed the minimum working point of the Level Sensor.
11. Start the pump and increase the level of the water to 10 cm of the mobile scale.
12. Stop the pump and check that the voltage value corresponds to 10 V, otherwise regulate the GAIN trimmer in the INPUT INTERFACE until you obtain the desired value.
13. Check that the voltage and the level values are correct by repeating the steps from step 5.
14. Slowly drain the tank until you reach all the level values shown in the table (1) below and write down, for each level value, the corresponding voltage value.
15. Switch OFF the main switch and remove all the connections.
16. Draw the characteristic of the level sensor in the figure below (Voltage vs. Level)

Table (1)

Level (cm)	**10**	**9**	**8**	**7**	**6**	**5**	**4**	**3**
Voltage (V)								

Experiment B: Characteristics of the Pump

Objectives:

1. To understand the working principle of a diaphragm pump.
2. To compute the flow velocity of the pump using the water volume in the process tank and the rising time.
3. To measure the flow velocity of the pump looking at the flowmeter on the DL 2314 Process Panel Unit.
4. To draw the characteristic curve of the flow.

Procedure:

The following steps should be checked before starting to configure the system:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the Process panel as follows:

CONTROL	STATE	ACTION
DELIVERY VALVE	fully open	Turn the knob counter-clockwise
MOTOR VALVE	fully open	Angular rotation = open
SOL Valve	closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE	fully open	Turn the knob counter-clockwise
DRAIN VALVE	fully closed	Turn the knob clockwise
NEEDLE VALVE	fully closed	Turn the knob clockwise
AIR VALVE	fully open	Turn the knob counter-clockwise
Level in the PROCESS TANK	0 cm of the mobile scale	Fill the transparent process tank with water up to 0 cm level on the mobile scale

4. Make sure that all the connections of the two DL 2314 PROCESS CONTROL TRAINER units are correct (power cord, heavy duty cable, standard parallel cable).
5. Switch ON the hardware devices from the POWER button on
6. Press the main switch (ON) to power up the experiment
7. Adjust the voltage on the SET POINT 1 to 3V
8. Start the pump and wait until the level of the water in the tank reaches 5 cm of the mobile scale and then stop the pump.
9. Write down in the table (2)   below the corresponding volume, time, and compute the flow values according to the note below. Use rounded values in order to draw the characteristic more easily.

Tank diameter = 18.5 cm.

Tank volume = area of the base x the height

1 cm^3 = 10^{-3} liters

1 cm^3/sec = 0.06 liters/min.

10. Gradually decrease the level of the water in the tank by turning counter-clockwise the knob NEEDLE VALVE and/or the knob DRAIN VALVE, back to 0 cm on the mobile scale.
11. Close the valves.
12. Repeat steps 4-8 until you reach all the voltage values shown in the table (1) and write down, for each voltage value, the corresponding volume, time, and flow values.
13. Draw the characteristic curve of the flow in the figure below (Flow vs. Voltage)

Table (2)

Voltage (V)	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**
Volume (cm^3)								
Time (Sec)								
Flow(cm^3/sec)								
Flow (liter/min)								

Experiment C: Characteristics of the Static Process

Objectives:

1. To understand how the flow will influence the rising time of the fluid level rate in a level control process.
2. To measure the flow of the pump using the flowmeter.
3. To draw the flow characteristic of the static process.

Procedure:

The following steps must be checked before starting to configure the system:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the Process panel as follows:

CONTROL	STATE	ACTION
DELIVERY VALVE	fully open	Turn the knob counter-clockwise
MOTOR VALVE	fully open	Angular rotation = open
SOL Valve	closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE	fully open	Turn the knob counter-clockwise
DRAIN VALVE	fully closed	Turn the knob clockwise
NEEDLE VALVE	fully closed	Turn the knob clockwise
AIR VALVE	fully open	Turn the knob counter-clockwise
Level in the PROCESS TANK	0 cm of the mobile scale	Fill the transparent process tank with water up to 0 cm level on the mobile scale

4. Switch ON the hardware devices from the POWER button on
5. Press the main switch (ON) to power up the experiment

6. Start the pump by adjusting the voltage on SET POINT 1 at 10V.
7. Wait until the water level has reached 10 cm of the mobile scale.
8. Gradually turn counter-clockwise the knob NEEDLE VALVE and/or the knob DRAIN VALVE to maintain the level of the water in the tank at 10 cm.
9. Wait until the level is stabilized (about 5 minutes). If this will not happen, adjust again the knob NEEDLE VALVE and/or the knob DRAIN VALVE to maintain the level at a constant value of 10 cm.
10. Read the values of the flow on the flowmeter by consulting the liters per minute scale (LPM) and write it down in the table below.
11. Repeat the procedure from step 3 to 7 for all the voltage values listed in Table (3) and write down the results.
12. Draw the characteristic curve of the flow rate (Flow vs. Voltage).

1 cm^3/sec = 0.06 liters/min

Table (3)

Voltage (V)	**10**	**9**	**8**	**7**	**6**	**5**	**4**	**3**
Flow(Liter/min)								
Level (cm)								

Experiment D: Time Constant of the Process

Objectives:

1. To understand the basics of fluid dynamics in order to control a level process.
2. To measure the dynamic response of the level process.
3. To determine the diagram of the dynamic response of the level process.

Procedure:

The following steps must be checked before starting to configure the system:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the Process panel as follows:

CONTROL	STATE	ACTION
DELIVERY VALVE	fully open	Turn the knob counter-clockwise
MOTOR VALVE	fully open	Angular rotation = open
SOL Valve	closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE	fully open	Turn the knob counter-clockwise
DRAIN VALVE	fully closed	Turn the knob clockwise
NEEDLE VALVE	fully closed	Turn the knob clockwise
AIR VALVE	fully open	Turn the knob counter-clockwise
Level in the PROCESS TANK	0 cm of the mobile scale	Fill the transparent process tank with water up to 0 cm level on the mobile scale

4. Switch ON the hardware devices from the POWER button on

5. Press the main switch (ON) to power up the experiment
6. Start the pump by adjusting the voltage on SET POINT 1 at 8V
7. Wait until the water level has reached 3 cm of the mobile scale.
8. Gradually turn counter-clockwise the knob NEEDLE VALVE and/or the knob DRAIN VALVE to maintain the level of the water in the tank at 3 cm
9. Adjust the voltage on SET POINT 1 at 6 V.
10. Wait until the water level is stabilized.
11. If this will not happen, adjust again the knob NEEDLE VALVE and/or the knob DRAIN VALVE to maintain the level at a constant value of 3 cm.
12. Adjust the voltage on SET POINT 1 at 7 V.
13. Measure the level at 10 seconds interval and write down the values in the table (4) below.
14. Switch OFF the mains switch and remove all the connections.
15. Represent the dynamic diagram of the process (Level vs. Time)

Table (4)

Time (Sec)	**0**	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**	**100**	**110**	**120**
Level (cm)													

Experiment E: ON -OFF Control of the Level

Objectives:

1. To understand the operation of a closed loop ON-OFF control system.
2. To learn how to perform the wiring connections of the DL 2314 DC motor pump and level sensor, in order to control the water level in the transparent process tank.
3. To measure the dynamic response of the process.
4. To understand the effects of the hysteresis on the level control.

Theory:

Hysteresis is the measurement of the difference in Y offset of the values generated by the transducer as it measures in a positive going direction, and the same values as the transducer measures back down toward zero (the negative going values).

In many cases, the main portion of this curve does not work out to be a simple straight line offset in each direction. Non-linearity and sampling error tend to make the line less than ideal. Therefore, the general case solution is to have two parallel lines, one passing through the main portion of the positive going values and one through the negative.

The generation of these two lines generally requires some finesse and is not nearly as scientific as some would think. The calibrator must determine where the "main" portion of the curve is and then what line best fits through the positive going points. The second line then needs to be placed through the negative going set, keeping the same slope. The difference in Y intercept of these two lines then becomes the total amount of hysteresis. In a more simplified system, where the nonlinearity is less dramatic, the calculation of the hysteresis becomes much simpler. In this simplified case, the total hysteresis is the difference in Y values compared to the total amount of Y span.

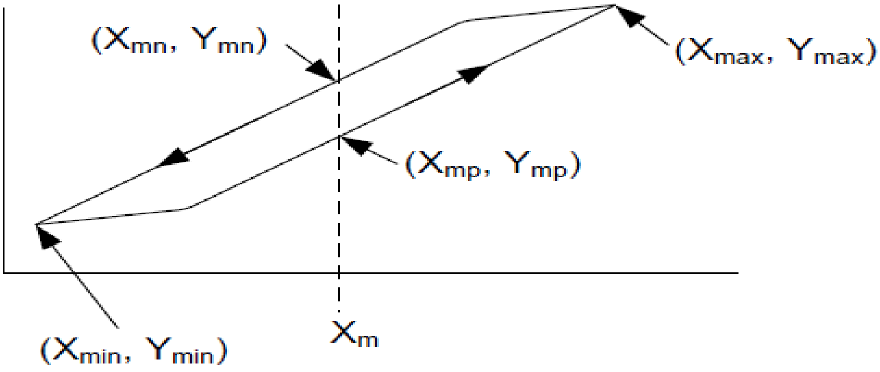


Figure 1. The hysteresis calculation

The calculation of the hysteresis in this simplified condition occurs at the X midpoint of the curve. This point can be located with the following formula:

$$\boldsymbol{X_m = \left(\frac{X_{max} - X_{min}}{2}\right) + X_{min}}$$

Once the midpoint had been located, the two Y values (positive and negative going) can be obtained and the calculation becomes a simple plug and chug.

$$\boldsymbol{Hysteresis}\,\% = \left|\frac{(Y_{mn}-Y_{mp})}{(Y_{max}-Y_{min})}\right| \times \mathbf{100\%}$$

In some complex situations, the curves may be so close that it is nearly impossible to differentiate, or the linearity is so bad that it simply swamps the amount of hysteresis. In these conditions, a hysteresis of 0 is possible. Another unusual condition is where the negative going slope has so much hysteresis that it overlaps the positive going transition. Under these conditions, the slopes of the positive and negative going portions of the curve have Y intercept values that are extremely large. Under these conditions, the calculation, made either graphically or mathematically, using the difference between the slopes, could easily end up with a hysteresis of larger than 100%. Generally, these types of huge hysteresis conditions are intentionally created for fine/course control inputs.

Procedure:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the DL 2314 Process panel as follows:

CONTROL	STATE	ACTION
DELIVERY VALVE	fully open	Turn the knob counter-clockwise
MOTOR VALVE	fully open	Angular rotation = open
SOL Valve	closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE	fully open	Turn the knob counter-clockwise
DRAIN VALVE	fully closed	Turn the knob clockwise

NEEDLE VALVE	fully closed	Turn the knob clockwise
AIR VALVE	fully open	Turn the knob counter-clockwise
Level in the PROCESS TANK	0 cm of the mobile scale	Fill the transparent process tank with water up to 3 cm level on the mobile scale
SET POINT 1	0 V	Turn counter-clockwise the knob from the PID interface

4. Switch ON the hardware devices.
5. Press the main switch (ON) to power up the experiment.
6. Start the pump by adjusting the voltage on SET POINT 1 at 7V
7. As soon as the voltage reaches the SET POINT, the pump will be stopped.
8. Slowly turn counter-clockwise the knob NEEDLE VALVE and/or the knob DRAIN VALVE in order to decrease a little the level: the voltage difference between terminal 14 and terminal 13, caused by the level variation, brings the output of the ON-OFF controller (terminal 18) to a voltage of approx. 10V and the pump will start again.
9. The pump will operate for a short time until the previous level is reached.
10. Adjust the knob NEEDLE VALVE and/or the knob DRAIN VALVE until you determine, approximately, an equal time, for level increase and decrease, between start and stop of the pump: the up and down cycle of the level will repeat itself.
11. The pump will repeat step 9 and will operate for a short time until the previous level is reached .
12. Measure the up and down times of the level between start and stop of the pump with a hysteresis of 0%: write down the results in the table below.

Table (5)

Hysteresis (%)	0	15	30
Set Point (cm)			
Lower limit Set Point (cm)			
Up-raising time of the level (sec)			
Upper lim it Set Point (cm)			
Lowering time of the level (sec)			

Experiment F: Closed Loop P, PI, AND PID Control of the Level

Objectives:

1. To understand the theoretical aspects of the operation of a closed loop control system with proportional action (P), proportional-integral (PI), proportional-integral-derivative (PID).
2. To check the effects of the gain of the loop on the dynamic response of the system.
3. To represent the dynamic response curve of the system.

Theory:

Control systems are classified in two general categories and more precisely:

➢Open Loop Systems

➢Feedback or Closed Loop Systems

An open loop system is characterized by the fact that the control action is independent on the output.
On the contrary, in the closed loop systems the control action depends in some way on the output.

In fact, it is the difference Ɛ between the value of the reference quantity w and the value of the controlled quantity x that gives origin to an action having, as a final objective, the cancellation of such difference.
The block diagram of a general negative feedback control system is shown in Figure 2.

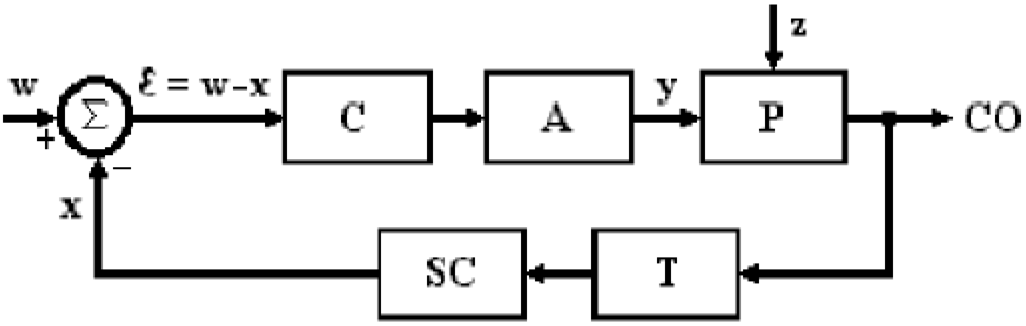


Figure 2. Block diagram of a closed loop control system

Where:
C = controller
A = amplifier
P = process
CO = controlled output or variable (not converted to a signal homogeneous with w)
T = transducer
SC = signal conditioner

w = reference variable (set-point)
x = controlled variable (converted to a signal homogeneous with w)
Ɛ = w - x = error (or difference)
y = regulating variable
z = noise variable

The meaning of the blocks and of the signals is the following:

➢**Controller and Amplifier**: are composed of the set of devices needed to generate the suitable control signal y to be applied to the process.

➢**Transducer and Signal Conditioner**: these are the devices that convert the physical quantity of the controlled output to a quantity homogeneous with the Set-Point.

➢**Error Signal**: it is the signal obtained by the difference between the Set-Point signal and the feedback signal provided by the Signal Conditioner.

➢**Noise**: it is an unwanted (input) signal that changes the output value.

The main advantages of the closed loop control systems with respect to the open loop control systems and that justify the use of the closed loop control can be listed as follows:

➢less sensitivity to the parametric variations

➢less effects of the noise quantities

The importance of the above two advantages is further clarified by the fact that parametric variations and noise are usually random, that is, unpredictable if not in their statistical characteristics.

Analysis and design of the control system

The main objective of the analysis of a feedback control system is the determination of the following characteristics:

➢the response in the transitory phase

➢the response in the permanent regime

➢the degree of stability

Usually, it is not enough to know whether a system is stable; it is necessary to determine the relative stability, that is "how much" the system is stable.
The relative stability is correlated to the transitory response of the system; for this reason, the transitory response takes a fundamental role in the analysis of the global characteristics of the system.
Since in the time domain it is rather difficult to study (that is to solve directly the differential equation) systems of order higher than the second, there are different Graphic Methods that allow the analysis of the feedback control systems.
They are the following:

➢the root locus (study in the s domain)

➢the Bode diagram (study in the w domain)

➢the Nyquist diagram (study in the w domain)

➢the Nichols map (study in the w domain)

Concerning the design, the main objective is that of obtaining the desired behavior specifications in terms of speed of response, accuracy and stability.
The above can be enunciated in two different ways and more precisely:

➢specifications in the frequency domain

➢specifications in the time domain

The specifications in the frequency domain are usually presented in the following terms:

➢gain margin

➢phase margin

➢band width

➢cut-off rate

➢amplitude of the resonance peak

➢resonance frequency

The specifications in the time domain are usually defined in terms of response to the unit step. The latter will have a component at permanent regime and another component at transitory regime.

The performance at permanent regime is an index of the accuracy of the system, while the transitory behavior gives an indication about the speed of response and the relative stability. Typical specifications in the time domain are the following:

➢overshoot

➢delay time

➢rise time

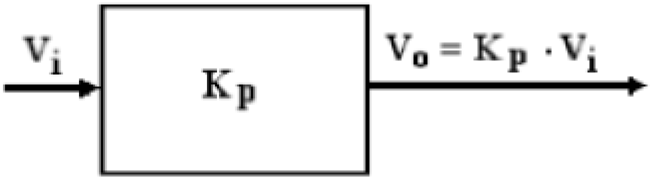
➢adjustment time

➢determinant time constant

In order to obtain the desired specifications and considering that the process has its own transfer function, which is difficult to modify, it is necessary to introduce in the control system a suitable block, the "controller".
The controller can be of the active type (amplifier, integrator, derivator or at two or three positions) and of the passive type (advancing or delaying network).
Through the modification of the characteristics of a standard controller, it is possible to obtain the desired responses from the process (presetting of the controller).
The standard controllers that are normally used in the industry are of the active type and incorporate the Proportional, Integral and Derivative actions, with adjustable parameters, that give rise to the actions that we will described afterwards.
Furthermore, for some applications, it is enough the use of a simpler two position (ON/OFF) controller for the unidirectional systems or a three-position controller for the bidirectional systems.

Proportional (P) action

It is the action introduced by an amplifier-attenuator.
The output, apart from the multiplying coefficient Kp (coefficient of the proportional action or proportional gain), is a perfect copy of the input.
In the figure below an amplifier-attenuator is shown whose **transfer function** is Kp.



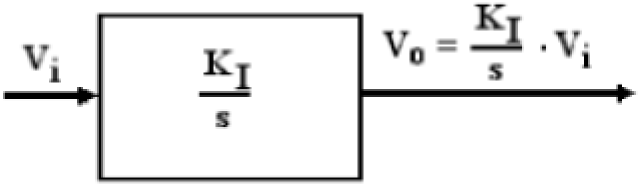
Integral (I) action
This action is introduced by a pure integrator.

The **transfer function** of the block that makes the integral action (figure below) is the following:

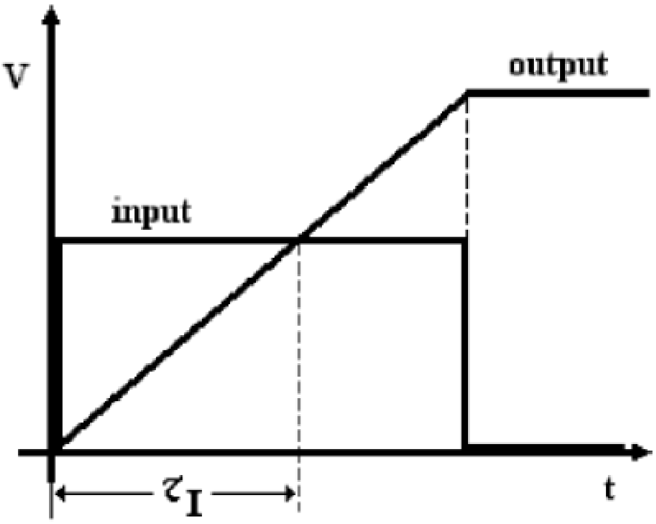
$$\boldsymbol{W}_{(\boldsymbol{S})}=\frac{\boldsymbol{K_I}}{\boldsymbol{S}}=\frac{\boldsymbol{I}}{\boldsymbol{\tau_I}.\boldsymbol{S}}$$

where:

τ_I = time constant of the integral action (s)
K_I= coefficient of the integral action (s^{-1})



The output, relevant to a step input, shows a linear type delay.
After a time, equal to the time constant of the integral action, the output reaches the value of the input (figure below).



Notice that, after having reached the value of the input, the output keeps rising with the same slope, until the input becomes null.

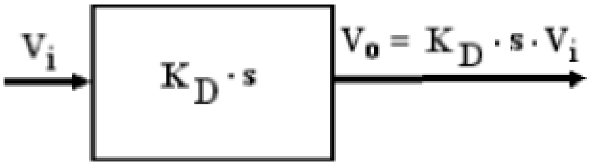
Derivative (D) action

It is the action introduced by a pure derivator.
The **transfer function** of the block performing the derivative action (figure below) is the following:

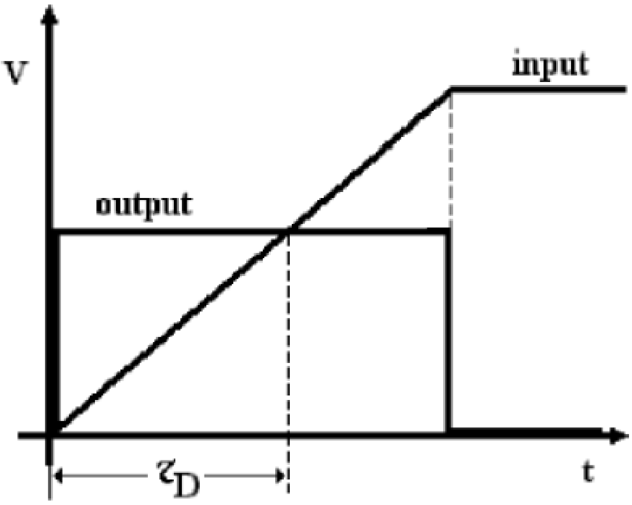
$$\boldsymbol{W}(\boldsymbol{S})=\boldsymbol{K_D}.\boldsymbol{S}=\boldsymbol{\tau_D}.\boldsymbol{S}$$

Where:

τ_D = time constant of the derivative action (s)
K_D= coefficient of the derivative action (s)



The output, relevant to a linear ramp input, has the value that the input will assume after a time equal to the time constant of the derivative action (figure below).



Note that the value of the output, equal to the value that the input assumes after the time τ_D, is kept until the input changes its slope.

Combined PID action

By connecting in parallel the proportional, integral and derivative actions, it is possible to obtain the controller shown in figure bellow, whose **total transfer function** is the following:

$$W(s) = K_p + \frac{K_I}{s} + K_D.s = K_p\left(1 + \frac{K_I}{K_p.s} + \frac{K_D}{K_p}.s\right) = K_p\left(1 + \frac{1}{\tau_I.s} + \tau_D.s\right)$$

where

$$\tau_I = \frac{K_p}{K_I} \qquad \tau_D = \frac{K_D}{K_p}$$

time constant of the integral action *time constant of the derivative action*

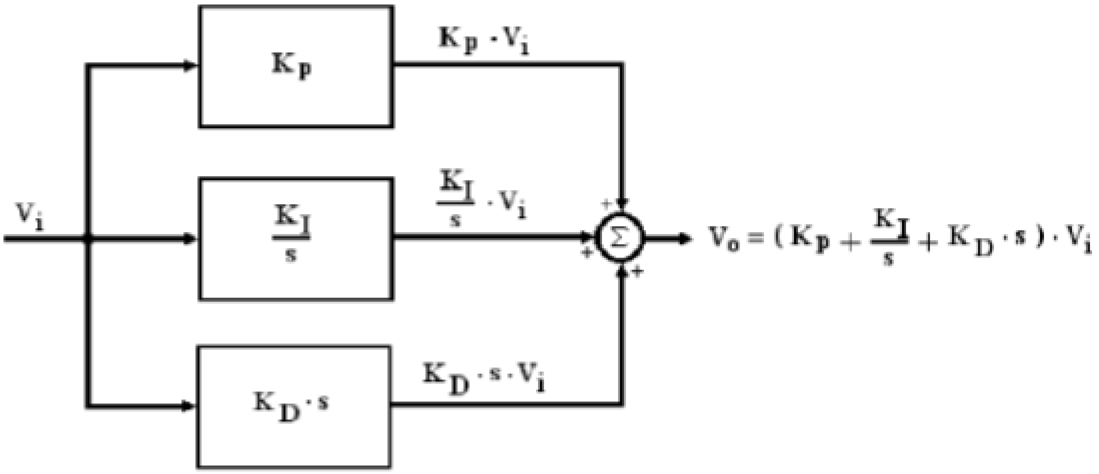


Figure 1. The diagram of Proportional, Integral and Derivative control

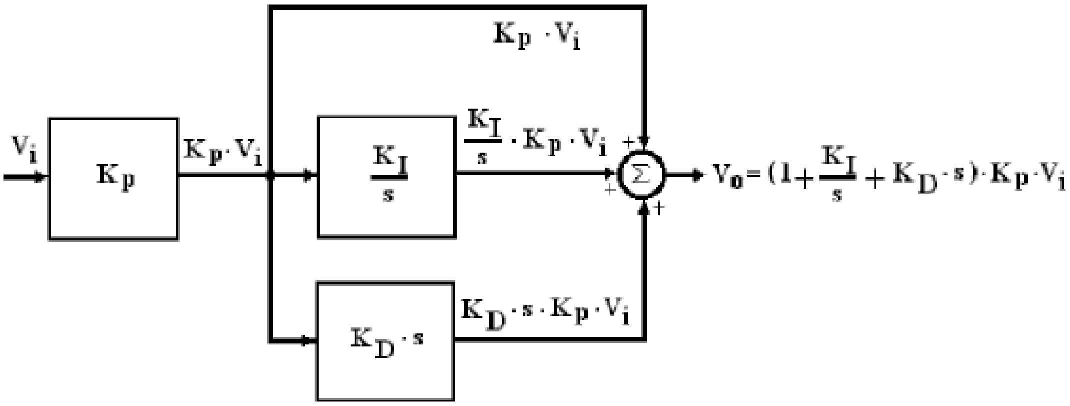
The parallel configuration is intuitive and educationally useful; in the industrial applications, however, the configuration shown in figure bellow is preferred, where the proportional action is placed in series to the other two, which are in turn connected in parallel.
This configuration has the advantage of using the gain of the proportional controller to obtain high signals for the input to the I and D controllers.
For the latter configuration, the total transfer function is the following:

$$W(s) = K_p\left(1 + \frac{K_I}{s} + K_D.s\right) = K_p\left(1 + \frac{1}{\tau_I.s} + \tau_D.s\right)$$

where

$\tau_I = \frac{1}{K_I}$ $\tau_D = K_D$

time constant of the integral action *time constant of the derivative action*



Obviously, if all the other conditions are equal, the regulations of the KI and KD coefficients are not the same for the two configurations of the PID controller.

Very often the KP coefficient, instead of being expressed as the value of the amplification, is expressed as **Proportional Band B_p**, that is:

$$B_p(\%) = \frac{1}{K_p}.100$$

Procedure:

1. Make sure that the sump tank is filled with approximately 15 liters of distilled water. See the LEVEL black line on the right side of the sump tank.
2. Align the 0 cm mark of the mobile scale with the 4 cm mark of the fixed scale engraved on the transparent process tank.
3. Set the DL 2314 Process panel as follows:

CONTROL	**STATE**	**ACTION**
DELIVERY VALVE	fully open	Turn the knob counter-clockwise
MOTOR VALVE	fully open	Angular rotation = open
SOL Valve	closed	SOL VALVE on the Control Panel not connected (Normally Closed) or Off switched
MAN VALVE	fully open	Turn the knob counter-clockwise
DRAIN VALVE	fully closed	Turn the knob clockwise
NEEDLE VALVE	fully closed	Turn the knob clockwise
AIR VALVE	fully open	Turn the knob counter-clockwise
Level in the	3 cm of	Fill the transparent

PROCESS TANK	the mobile scale	process tank with water up to 3 cm level on the mobile scale
SET POINT 1	0V	Turn counter-clockwise the knob from the PID interface
SET POINT 2	0V	Set in the middle position the knob from the PID interface
PROPORTIONAL	0%	Turn counter-clockwise the knob from the PID interface
INTEGRAL	0%	Turn counter-clockwise the knob from the PID interface
DERIVATIVE	0%	Turn counter-clockwise the knob from the PID interface

4. Make sure that all the connections of the two DL 2314 PROCESS CONTROL TRAINER units are correct (power cord, heavy duty cable, standard parallel cable).
5. Adjust the voltage on SET POINT 2 at 10 V.
6. Adjust the PROPORTIONAL knob at 25%
7. Immediately fully open the NEEDLE VALVE by turning its knob counterclockwise.
8. Write down in Table 9.1 the voltage value read every 10 seconds until the system is stabilized

Table 6 (example)

Time (sec)	**0**	**10**	**20**	**30**	**40**	**50**	**60**
K_p = 25 %							
K_p = 50 %							
K_p = 75 %							
K_p = 100 %							

9. Bring the water level in the tank back to 3 cm through the DRAIN VALVE and the NEEDLE VALVE.
10. Adjust the PROPORTIONAL knob at 50%,75% and 100% and repeat the procedure from step 7.
11. Draw the curves of the closed loop dynamic response for all the values of the PROPORTIONAL controller in the same figure (Level vs. Time).
12. For PROPORTIONAL - INTEGRAL (PI-controller) repeat same procedure for P-controller and write down in Table 10.1 the voltage value read every 10 seconds until the system is stabilized (steady level at approximately 8 cm).

Table 7 (example)

Time (sec)	**0**	**10**	**20**	**30**	**40**	**50**	**60**
K_p = 25 % K_I = 25%							
K_p = 50 % K_I = 50%							
K_p = 75 % K_I = 75%							
K_p = 100 % K_I = 100%							

13. For PROPORTIONAL –INTEGRAL-DERIVATIVE (PID-controller) repeat same procedure for P-controller and write down in Table 10.1 the voltage value read every 10 seconds until the system is stabilized.

Table 8 (example)

Time (sec)	**0**	**5**	**10**	**15**	**20**	**25**	**30**	**…**
K_p = 30 % K_I = 25% K_d = 25%								

14. Draw the curves of the closed loop dynamic response in a figure (Level vs. Time).

Appendix A

A. General notions in process control systems

System : system is ”a set of things working together as parts of a mechanism or an interconnecting network; a complex whole”; or, “a group of devices or artificial objects or an organization forming a network especially for distributing something or serving a common purpose”.

Process : The process is a set of physical transformations and/or a set of transfers of matter and/or energy.

Input : "Input" or "Set-Point" is the stimulus applied to the control system. It represents the ideal behavior of the process output. Example: to provide 200 liters per hour.

Output: "Output" of the process is the variable of the process that we want to control (the quantity of delivered water).

B. Process variables

A process variable is a condition of the process fluid that can change the manufacturing process in some way, or it is an output of the process. Process variables such as: Pressure, Flow, Level, Temperature, Density, pH, Mass, Conductivity. They are detected and estimated by sensors or instruments in the field and act as inputs to the controlling entity, or represent the reason of the process.

There are three types of variables:

Controlled variables - these are the variables that quantify the performance or quality of the final product, which are also called output variables (O).

M**anipulated variables** - these input variables are adjusted dynamically to keep the controlled variables at their set-points (I).

D**isturbance variables** - these are also called "load" variables and represent input variables that can cause the controlled variables to deviate from their respective set points (D).

Set-point change - implementing a change in the operating conditions.

Disturbance change - the process transient behavior when a disturbance enters, also called regulatory control or load change. A control system should be able to return each controlled variable back to its set-point.

Control :"Control" is the group of actions taken to direct a process so that it assumes the desired behavior. Or, “a method, a device or mechanism used to regulate or guide the operation of a machine, apparatus, or system”.

Manual control :In a manual control, the action performed by the man continuously changes on the basis of the result provided by the comparison between the information relevant to the value of the controlled quantity and the information relevant to the value preset for such quantity.

Automatic control : On the other hand, in the automatic regulation the system is able to autonomously control the variables of the control action in order to cancel the difference between the value taken by the controlled quantity and the value preset for it.

Functional block diagram: In the study of the control systems it is useful to graphically describe the way the different components interact, with the scope of showing the flow of the transferred information and the actions of each process variable on the others. Such graphic representation technique is termed "Functional blocks diagram".

Transfer function : The best way to do this is to use the "Transfer Function" that, in general, can be written as follows:

$$= \frac{Y}{X}$$

A transfer function represents the relationship between the output signal of a control system and the input signal, for all possible input values. A block diagram is a visualization of the control system which uses blocks to represent the transfer function, and arrows which represent the various input and output signals.

In a Laplace Transform, if the input is represented by R(s) and the output is represented by C(s), then the transfer function will be:

$$G(s) = \frac{C(S)}{R(S)}$$