



**University of Jordan**  
**School of Engineering**  
**Department of Chemical Engineering**

**Chemical Engineering Laboratory (2)**

**0915461**

# Experiment Number -1-

## Vapor-Liquid Equilibrium

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### **Objective:**

In this experiment equilibrium data for the binary system: n-hexane (1) – toluene (2) will be obtained, or for any other system assigned by the lab instructor. Subsequently, the equilibrium data are reduced to obtain the activity coefficients. The determined activity coefficients are fitted to a suitable model such as the two-suffix Margules, Van Laar, NRTL, UNIQUAC, and Wilson equations. The experimental data are to be compared with the predicted activity coefficients using the UNIFAC method, also to be compared with the reported literature values for the system assigned to the group.

### **Equipment:**

The main features of the apparatus are illustrated in figures (1&2). It consists of a boiling chamber (A) in which the mixture under test is vigorously heated to produce a vapor-liquid mixture that passes the glass spiral tube (B) at a high velocity. The rising vapor entrains drops of the liquid which equilibrates with the vapor and this mixture emerges from the tube at equilibrium, striking the thermometer's pocket (C), which contains a thermocouple (1) to measure the boiling temperature. The vapor then rises gently through the central tube (E) while the liquid drops fall back into the chamber (F). The vapor emerging from the tube (E) passes through the annulus (G), its temperature is measured by thermocouple (2), it is then heated electrically in order to prevent condensation and therefore the entire vapor passes to the water cooled condenser (H) where they are totally condensed. The liquid so formed accumulates in the receiver (J) from which a sample of the vapor phase is obtained. When the receiver (J) is full of condensate, the condensate overflows and passes through the tube (P) to mix with the liquid flowing down the tube (K) from the chamber (F). This mixture enters the heater (A) to be re-vaporized and discharged as a mist into the spiral (B) where it is equilibrated on emerging against the thermometer pocket (C).

The apparatus also contains a refractometer, constant temperature water circulating unit and temperature reading device.

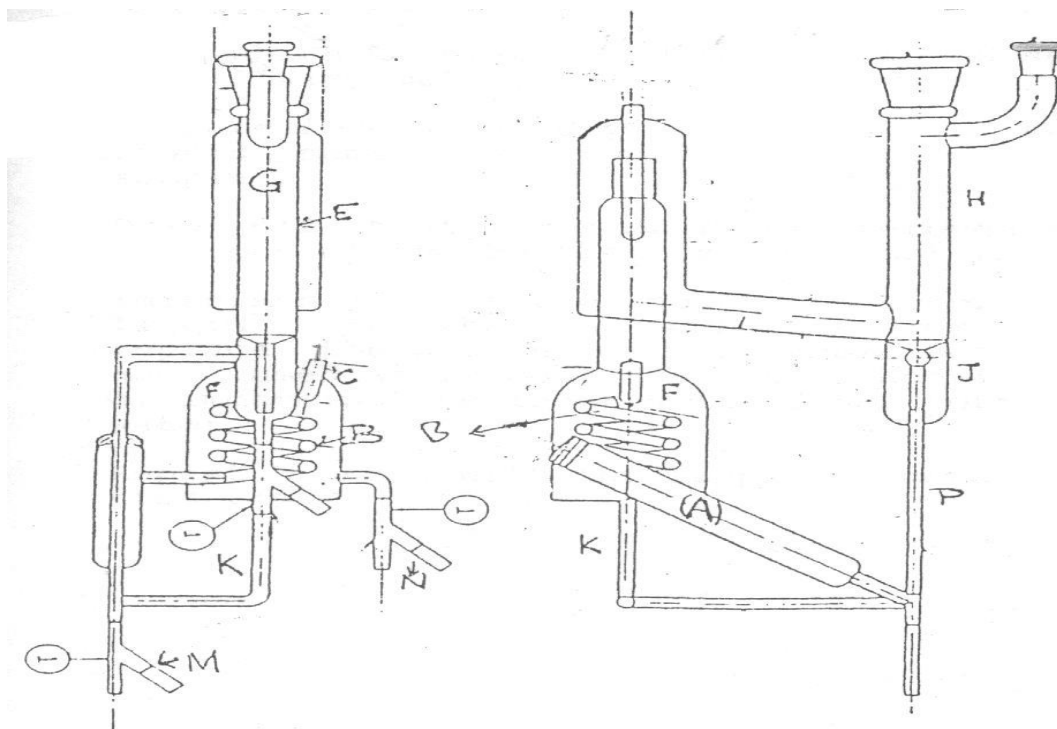


Figure (1): The vapor-liquid equilibria still.



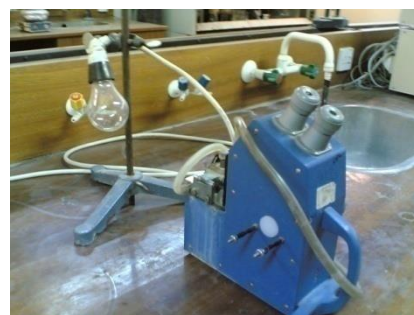
Vapor-Liquid equilibria still



Boiling chamber



Temperature reading system



Refractometer

Photos courtesy of Dalia N. Saleh

Figure (2): Main components and accessories of vapor-liquid apparatus

## **Theory:**

If a liquid mixture of two volatile components (A) and (B) is heated in a closed vessel, the temperature rises and the rate of vaporization of each component increases. As the temperature is raised, the vapor pressure of the liquid mixture, ( $P_T$ ) increases until it is equal to the pressure applied on the system ( $P$ ). At this temperature the liquid mixture starts to bubble and the temperature is called the bubble point ( $T_{bp}$ ) of that mixture at the pressure ( $P$ ). The first bubble of vapor is in equilibrium with the liquid mixture at that temperature ( $T_{bp}$ ).

The partial pressure exerted by each component depends on the composition of the liquid mixture and the temperature. If the liquid mixture is ideal, it obeys "Raoult's Law":

$$P_T = \sum_{i=1}^{C=2} P_i = \sum_{i=1}^{C=2} x_i P_i^{sat}(T) \dots \dots \dots (1)$$

Where ( $x_i$ ) is the mole fraction of component (i) in the liquid phase, ( $P_i = y_i P$ ) is the partial pressure of component (i), ( $y_i$ ) is the mole fraction of component (i) in the vapor phase, and ( $P_i^{sat}$ ) is the saturation pressure of component (i) which is a function of temperature.

If the components form an ideal mixture, the composition of the liquid and the vapor in equilibrium can be predicted from Raoult's Law and Dalton's Law, and this represents the maximum separation (or purification) of the two components that could be achieved by a simple distillation comprising a single vaporization and a single condensation step. In practice most mixtures are not ideal and therefore, equation (1) must be modified by introducing an activity coefficient such that

$$P_T = \sum_{i=1}^{C=2} P_i = \sum_{i=1}^{C=2} \gamma_i(x, P_T, T) x_i P_i^{sat}(T) \dots \dots \dots (2)$$

Equation (2) is termed the modified Raoult's law. Modified Raoult's law is strictly valid at low pressure assuming that the vapor phase forms an ideal gas mixture i.e., the nonideality is accounted for solely by the activity coefficients in the liquid phase, ( $\gamma_i$ ). The activity coefficient of a component in a mixture varies with temperature, pressure and composition, and must therefore be determined experimentally. It is most frequently calculated from the vapor-liquid equilibria data using the modified Raoult's law in the form:

$$\gamma_i(x, P, T) = \frac{y_i \cdot P_T}{x_i \cdot P_i^{sat}(T)} \dots \dots \dots (3)$$

Thus, ( $x_i$ ), ( $y_i$ ), ( $P_T$ ) and ( $P_i^{sat}(T)$ ) should be known before being able to determine the activity coefficients. There are many ways to determine the composition in the vapor and liquid phases including, but not limited to: gas chromatography (GC), HPLC and refractive index. In this lab, you are going to determine the composition in the two phases by measuring the refractive indices of liquid phase and the condensate of the vapor phase in equilibrium with it. The saturation pressures as function of temperature are easily calculated from empirical equations such as the Antoine equation:

$$\log P_i^{sat} = A - \frac{B}{T + C} \dots \dots \dots (4)$$

Where the parameters (A), (B), and (C) are specific to a particular component and can be found from any reference book or simulation package recently.

### Activity Coefficient Models

The activity coefficient of a volatile liquid component in solution can be calculated from many models. Examples of the activity coefficients models written for a binary system are:

#### 1. Two-Suffix Margules Equation

$$\begin{aligned} \ln \gamma_1 &= A_{12} \cdot x_2^2 \\ \ln \gamma_2 &= A_{12} \cdot x_1^2 \dots \dots \dots (5) \end{aligned}$$

Where ( $A_{12}$ ) is the parameter in this equation specific to any binary system. This parameter can be obtained from fitting of the experimental activity coefficients with composition.

#### 2. Van Laar Model

$$\begin{aligned} \ln \gamma_1 &= \frac{A_{12}}{\left[1 + \frac{A_{12}}{A_{21}} \cdot \frac{x_1}{x_2}\right]^2} \\ \ln \gamma_2 &= \frac{A_{21}}{\left[1 + \frac{A_{21}}{A_{12}} \cdot \frac{x_2}{x_1}\right]^2} \dots \dots \dots (6) \end{aligned}$$

Where ( $A_{12}$ ) and ( $A_{21}$ ) are parameters in this model specific to a binary system. These parameters can be obtained from fitting of the experimental activity coefficients with composition.

## Consistency Tests

Gibbs-Duhem equation states that:

$$(x_1 \cdot d\ln\gamma_1 + x_2 \cdot d\ln\gamma_2)_{T,P} = 0 \dots \dots \dots (7)$$

From this equation, these two equations could be derived:

$$\frac{d\ln\gamma_2}{dx_2} = -\frac{x_1}{1-x_1} \frac{d\ln\gamma_1}{dx_2} \dots \dots \dots (8)$$

$$\int_0^1 \ln\left(\frac{\gamma_1}{\gamma_2}\right) \cdot dx_1 = 0 \dots \dots \dots (9)$$

The consistency of data could be checked using one of these tests:

1. The differential test: This test applies at a specific composition using equation (8). If  $(\ln \gamma_1)$  and  $(\ln \gamma_2)$  are plotted with respect to  $(x_2)$ , the slopes at any given concentration should satisfy this equation.
2. The integral test: The consistency of experimental activity coefficients which are available over the entire concentration range can be checked by plotting  $(\ln (\gamma_1/\gamma_2))$  versus  $(x_1)$ . The net area beneath the curve should equal zero, as indicated by equation (9). The plot must be extrapolated and integrated in the interval  $x_1 \in [0, 1]$ .

## Procedure:

1. Remove the condenser and pour 120 mL of one of the pure components via a clean dry funnel to the still.
2. Replace the condenser; turn on the cooling water through the condenser.
3. Switch on the electricity to the heater and the heating jacket.
4. When boiling commences, reduce the control so that about 20 drops per minute fall from the base of the condenser into the receiver.
5. Note the temperature of the boiling liquid recorded by thermocouple number (1) and continue boiling the mixture at this constant rate until this temperature remains constant.
6. When temperature (1) is constant adjust the jacket heater so that the temperature recorded by the thermocouple number (2) registers about  $(0.5-1.0^\circ\text{C})$  higher than the boiling temperature recorded by thermocouple by thermocouple number (1).
7. Continue boiling for at least one hour. After this period of equilibration take samples of the liquid from tap (N) (at the bottom of the main still) and of condensed vapor from tap (M) (at the bottom of reservoir which receives the drops of condensed vapor), by the following methods:

- a. Prepare four clean dry sample bottles, two for the liquid sample and two for the condensed vapor sample.
  - b. Draw off about 2 mL of liquid through the tap (N) into one of the bottles (this is not the sample).
  - c. Immediately withdraw about 2 mL of the same liquid into the second bottle from tap (N) and replace the stopper of the sample bottle quickly and immediately cool the bottle and its contents by immersing the lower half of the bottle in cold water.
8. Repeat b and c with the sample of condensed vapor from tap (M).
  9. Switch on the water bath, and set the temperature as that recorded on the calibration curve supplied for the system used.
  10. Measure the refractive index of the liquid and vapor by the refractometer
  11. Add a known volume of the other component (B) to the pure liquid (A) and repeat steps (5 – 10) to obtain new results.
  12. Add another volume of component (B) to the liquid in step (11) and also repeat steps (5-10) to obtain new results.
  13. In the second period of experiment, starts with the other liquid (B) and after equilibrium of this run, add increments of the other component (A) to obtain result for many mixture of different composition.
  14. At the end of the other period of experiment, switch off the apparatus, the water bath and the refractometer. Then drain the contents of the still.
  15. Record the operating atmospheric pressure in the Lab.

### **Calculation:**

1. Using the measured refractive indices, find the mole fractions of the volatile component in liquid and vapor phases at equilibrium from the calibration curve of the system.
2. Plot the boiling point-composition diagram ( $T_{xy}$ ) of the system.
3. Calculate the activity coefficients of the components in the liquid phase using equation (3).
4. Fit the two-suffix Margules, van Laar, Wilson, and NRTL parameters to your data and report them. You may want to use the THERMOSOLVER software to carry out the fitting process.
5. At each equilibrium composition, estimate the activity coefficients using UNIFAC method. You may want to use Sandler's modified UNIFAC software.
6. Obtain estimates of the activity coefficients based on literature values. Go to the binary vapor-liquid equilibrium data of the Korean Thermophysical Properties Data Bank ChERIC-KDB for experimental results of the system you have studied. The address is <http://www.cheric.org/research/kdb>.

7. Plot both  $(\ln\gamma_1)$  and  $(\ln\gamma_2)$  versus  $(x_1)$  using your experimental values, predicted values from the four models: two-suffix Margules, Van Laar, Wilson, and NRTL and that of UNIFAC.
8. Check the consistency of the data using the differential and integral tests.

### **References:**

1. Balzhiser, R. E.; Samules, M. R.; and Eliassen, J. D., 'Chemical engineering thermodynamics'. Prentice-Hall, Inc. 1972.
2. Hala, E; pick, Jiri; Fried, Vojtech; and vilim, otakar, 'Vapour-Liquid Equilibrium'. Second edition, Pergamon Press, 1967.
3. Poling, B. E.; Prausnitz, J. M.; and O'connell, J. P., 'The Properties of Gases and Liquids', 5th edition, McGraw-Hill, NY, 2001.
4. Sandler, S. I., 'Chemical, Biochemical and Engineering Thermodynamics', 4th edition, John Wiley and Sons, NY, 2006.
5. Smith, J. M.; Van Ness, H. C.; Abbott, M. M., 'Introduction to Chemical Engineering Thermodynamics', 7th edition, McGraw-Hill, NY, 2006.
6. Winnick, J., 'Chemical Engineering Thermodynamics', John Wiley and Sons, NY, 1997.



## Vapor-Liquid Equilibrium Data Sheet

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Atmospheric pressure: -----

Mixture used	Equilibrium Temperature	RI of vapor	RI for liquid

Instructor signature:
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Date:
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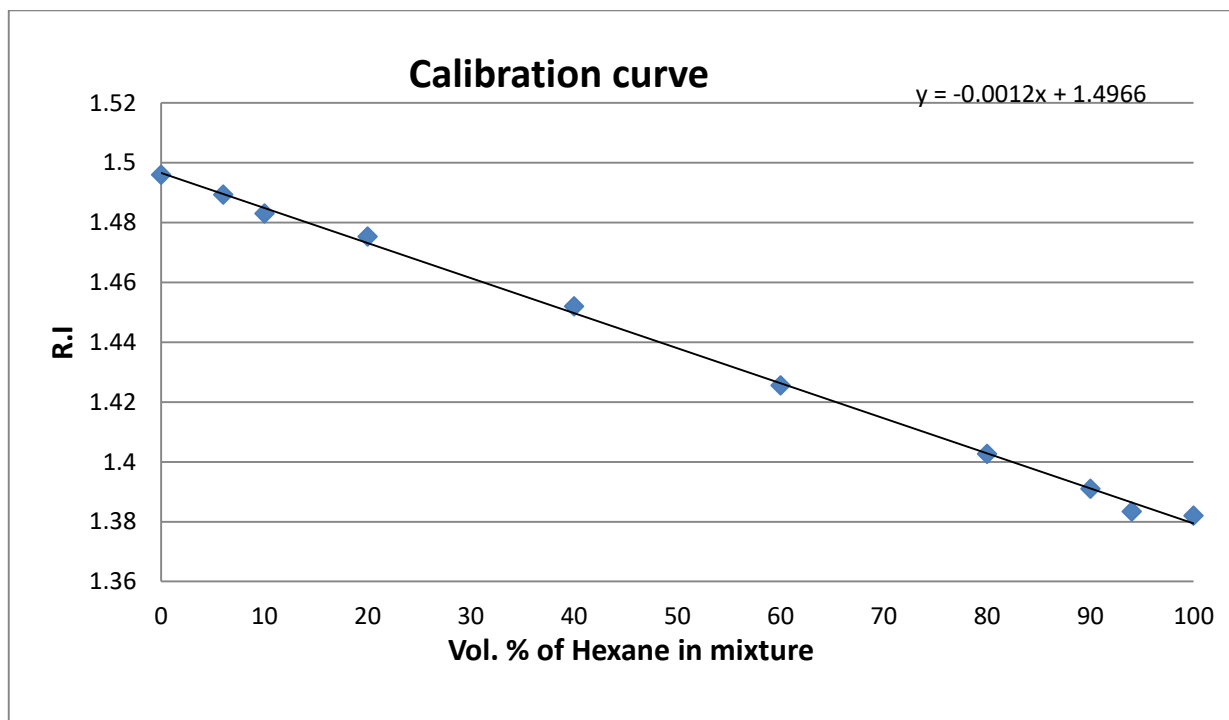


Figure (3): Refractive index Vs volume percentage for (Hexane + Toluene) system

## Experiment Number -2-

### Liquid-Liquid Equilibrium

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#### Objective

To be familiar with mass transfer processes and the phase equilibria involved in liquid-liquid extraction.

#### Equipment

The apparatus consists of three cells. Each cell has a central 100 ml. capacity glass vessel with a side arm near the top for charging the liquids. Each glass vessel is surrounded by a glass jacket through which water is circulated to maintain the cell contents at constant temperature. The cell is provided with an agitator. It also contains a valve for emptying the contents on completion of experiment. The general arrangement of liquid-liquid equilibria apparatus is illustrated in figures (5&6).

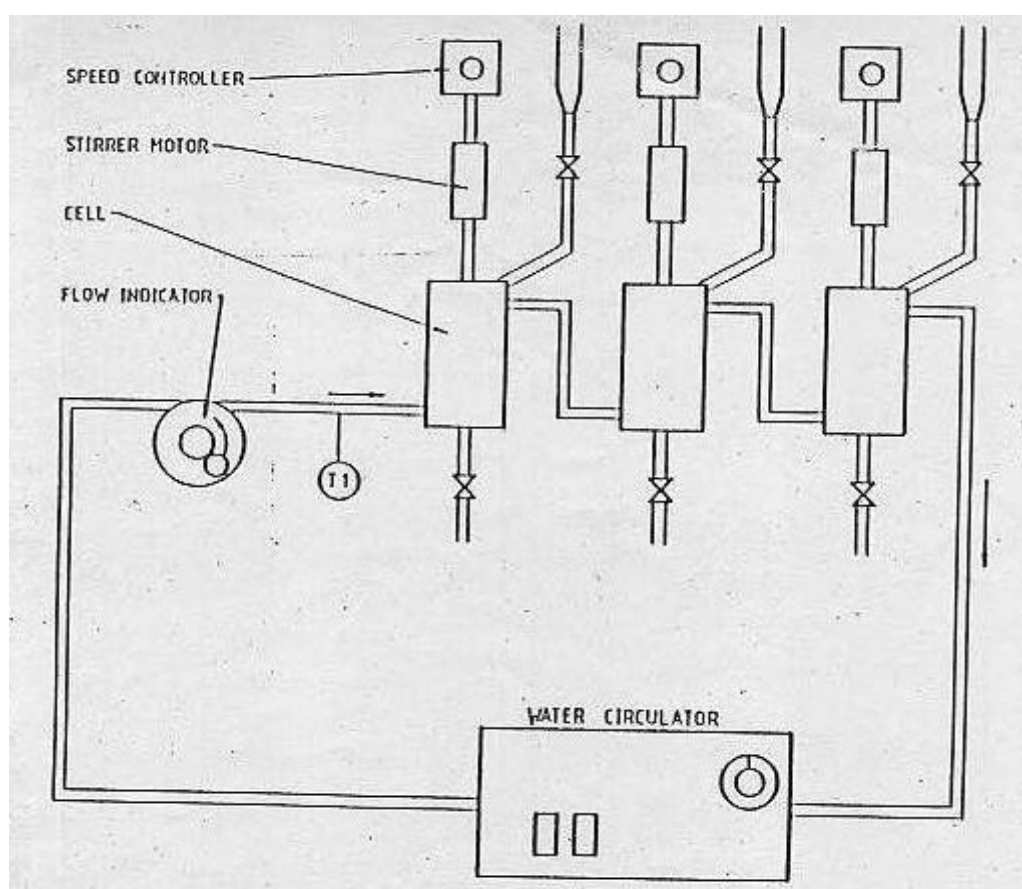


Figure (4): General Arrangement of liquid-liquid Equilibria apparatus

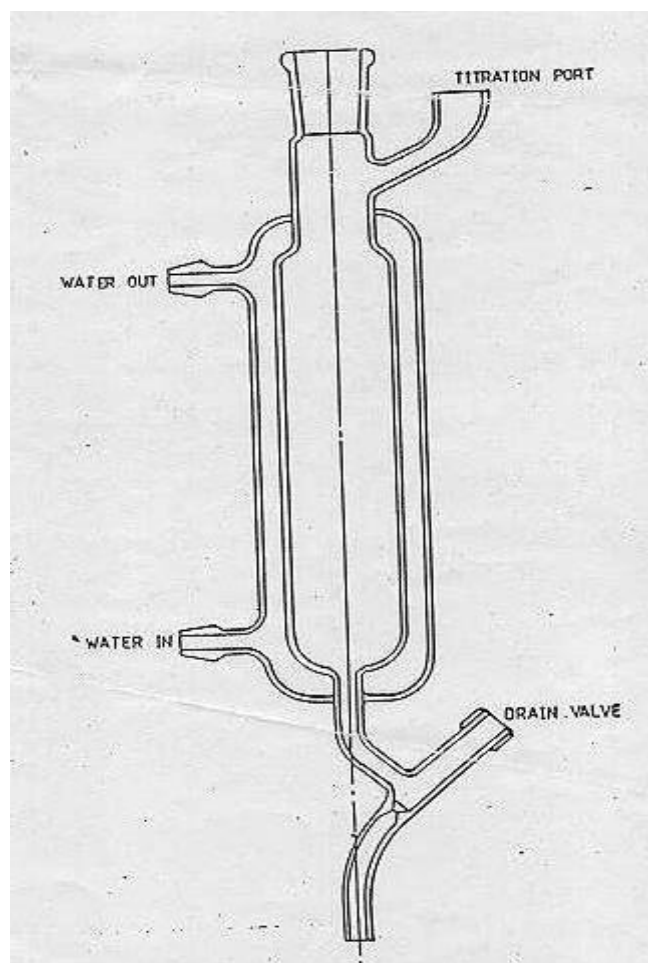


Figure (5): Agitated glass cell for liquid-liquid equilibria determinations

### **Theory:**

Liquid- liquid extraction is the term applied to any operation in which a material dissolved in one liquid phase is transferred to a second liquid phase. The solvent must be insoluble or soluble to a limited extent only, in the solution to be extracted. If the solvent and the solution were completely miscible, there would be no opportunity for transferring the solute from the original solution to a second liquid phase. The degree of solubility of the solvent in the solution, and of the solution and its components in the solvent, are important considerations in the selection of the solvent and in the operation of the extraction process.

Liquid-liquid extraction consists of two basic steps:

1. Intimate mixing or contact of the solvent with the solution to be treated, so as to transfer the solute from the solution to the solvent.
2. Separation of the liquid solution phase from the liquid solvent phase.

The complete extraction process may involve other operations such as the separation and recovery of the solvent from the solute and of the solvent which may be dissolved in the solution, the removal and recovery of the solvent may be more important in

determining the successful application of the extraction process than the degree of extraction and separation accomplished in the two basic steps, particularly when special or costly solvents are employed. The separation and recovery of the solvent may be accomplished by various methods such as distillation or simple heating or cooling to diminish the solubility of the solute or of the solvent.

Liquid-liquid-extraction is widely used for the separation of the components of a solution, particularly when:

1. The components are relatively nonvolatile.
2. The components have substantially the same volatilities from the mixture.
3. The components are sensitive to the temperatures required for the separation by distillation.
4. The desired less-volatile component is present in the solution only in relatively small amounts. In such a case, the less volatile component may be extracted solution from which it may be recovered more economically; or similar economics may be accomplished with a solvent less volatile than the desired component, even if the increase in concentration of the desired component is not particularly significant.

Usually the different distribution of the components of the feed mixture between the two layers at equilibrium is depended upon to effect the desired separation. The layer containing the greater concentration of the solvent and the smaller concentration of the feed liquid is referred to as the "extract" layer. The other layer, containing the greater concentration of the feed liquid and the smaller concentration of solvent is referred to as the "raffinate" layer.

All states of equilibrium between extract and raffinate can be represented in either a right triangle or an equilateral triangle phase diagram as you had learned before. For each state of equilibrium, there is only one specific tie line which represents it.

### **Reliability Using the Othmer-Tobias Correlation**

The reliability of equilibrium data for any system may be tested by applying the Othmer-Tobias correlation. This correlation states that:

$$\log \frac{(1-a)}{a} = n \log \frac{(1-b)}{b} + s \dots \dots (1)$$

which indicates that there is a linear relation between the values of  $(\log \frac{(1-a)}{a})$  and  $(\log \frac{(1-b)}{b})$  where:

a: weight fraction of the solvent in the extract phase.

b: Weight fraction of the carrier liquid in the raffinate phase.

s:the intercept

n:slope

The system to be used in this lab will be in general: water, an organic solvent and a solute.

## Procedure

A. To determine the ternary mutual solubility curve

a. For water rich phase:

1. Fill the cell with a solution consisting of 20 mls of distilled water and 5mls of the solute.
2. Switch on the thermostat unit and check that water is circulating through the jacket of the cell. Then set the temperature as required.
3. Switch on the stirrer and adjust the speed control to the midpoint of the range.
4. Allow 10 minutes for the contents of the cell to reach the set temperature.
5. Slowly titrate the organic solvent into the mixture in the cell until "Cloudiness" appears and persists. Record the volume of the solvent in the mixture. This is the "cloud point" at which the three components in the mixture are in equilibrium. It represents one point on the ternary curve.
6. Add another 5mls of the solute to the mixture and repeat the steps (2-5).
7. Repeat step 6.
8. Clean the cell and fill it with a solution consisting of 10mls of distilled water and 10mls of the solute. Repeat the steps (2-7). Adding 10mls of the solute each time, as shown in this table1.

Table (1): Water Rich Phase

Volume (ml)		Volume (ml)	
Water	Solute	Water	Solute
20	5	10	10
20	10	10	20
20	15	10	30

b. For the organic solvent rich phase:

Repeat the same procedure in (a) using solute-organic solvent solution and titrating with water.

Use the following compositions in table (2):

Table (2): Organic Rich Phase

Volume (ml)		Volume (ml)	
Organic Solvent	Solute	Organic Solvent	Solute
20	5	10	10
20	10	10	20
20	15	10	30

## B. Tie Line determination:

1. Fill the cell with a heterogeneous mixture consisting of 20 mls of water, 15mls of the solute and 15mls of the other solvent.
2. Agitate the mixture using the mid-point speed for 20 minutes at the desired temperature.
3. Stop the agitator, and leave the mixture for about 20 minutes to settle into two layers.
4. Withdraw a sample of each phase in a sample bottle and measure the refractive index of each sample.
5. Using the calibration curves, determine the percentage of the solute in each phase.
6. Repeat the steps (1-5) using the mixtures in Table (3).

Table (3): Tie-lines Determination

Component	mls of component			
Solute	13	8	5	3
Organic solvent	20	17	25	29
Water	18	26	20	19

**Calculation**

## A. For ternary mutual solubility curve:

1. Calculate the composition of each mixture at its cloud point.
2. Plot the mutual solubility curve for the ternary system on an equilateral triangle.

B. For tie-line determination:

1. Calculate the overall composition of each mixture.
2. Locate on the phase diagram the point which represents the overall composition for each mixture.
3. Find on the mutual solubility curve the points which represent the composition of the extract and the raffinate phases for each mixture.
4. Construct the tie line corresponding to each mixture.
5. Check the linearity of the points which represent the overall composition of each mixture, the composition of extract phase, and the composition of raffinate phase.
6. Check the reliability of the equilibrium data.

## **References**

1. Coulson, J.M.; and Richardson, J.F., "Chemical Engineering ", volume two. Pergamon Press Inc. third edition, 1978.
2. Felder, R.M.; and Rousseau R.W., "Elementary Principles of Chemical Processes". John Wily and Sons. second edition, 1986.
3. McCabe, W.L.; and Smith, J.C., "Unit Operations of Chemical Engineering". McGraw-Hill, Inc. third edition, 1976.



## Liquid-Liquid Equilibrium Data Sheet

Tie-Lines Determination:

Volume of water (ml)	Volume of Toluene (ml)	Volume of Acetone (ml)	RI of water layer	RI of Toluene layer
20	15	15		
18	20	13		
26	17	8		
20	25	5		
19	29	3		

Solubility curve

A. Water rich phase:

Volume of Acetone (ml)	Volume of water (ml)	Volume of Toluene (ml)
5	20	
10	20	
15	20	
10	10	
20	10	
30	10	

B. Organic solvent rich phase:

Volume of Acetone (ml)	Volume of Toluene (ml)	Volume of water (ml)
5	20	
10	20	
15	20	
10	10	
20	10	
30	10	

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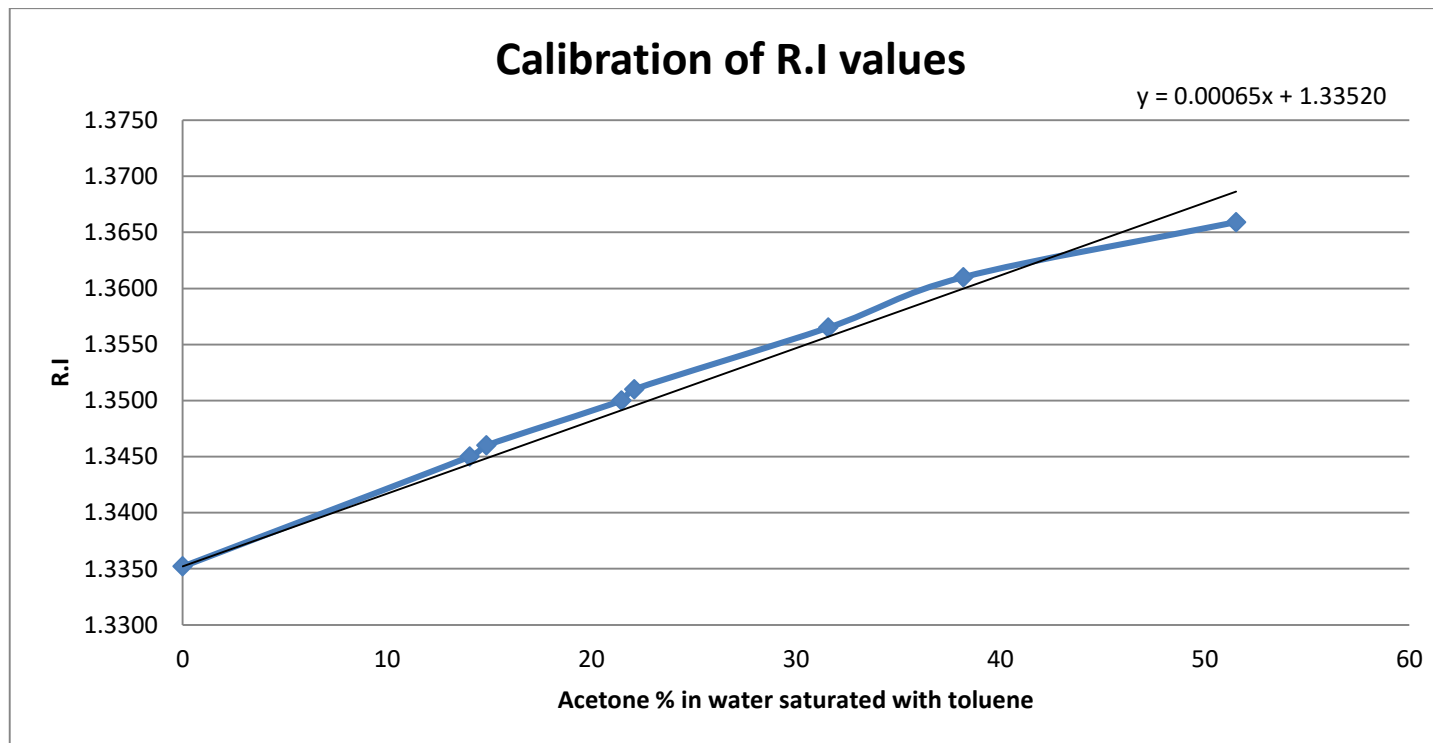


Figure (6): Wt % of Acetone in water saturated with Toluene Vs Refractive Index

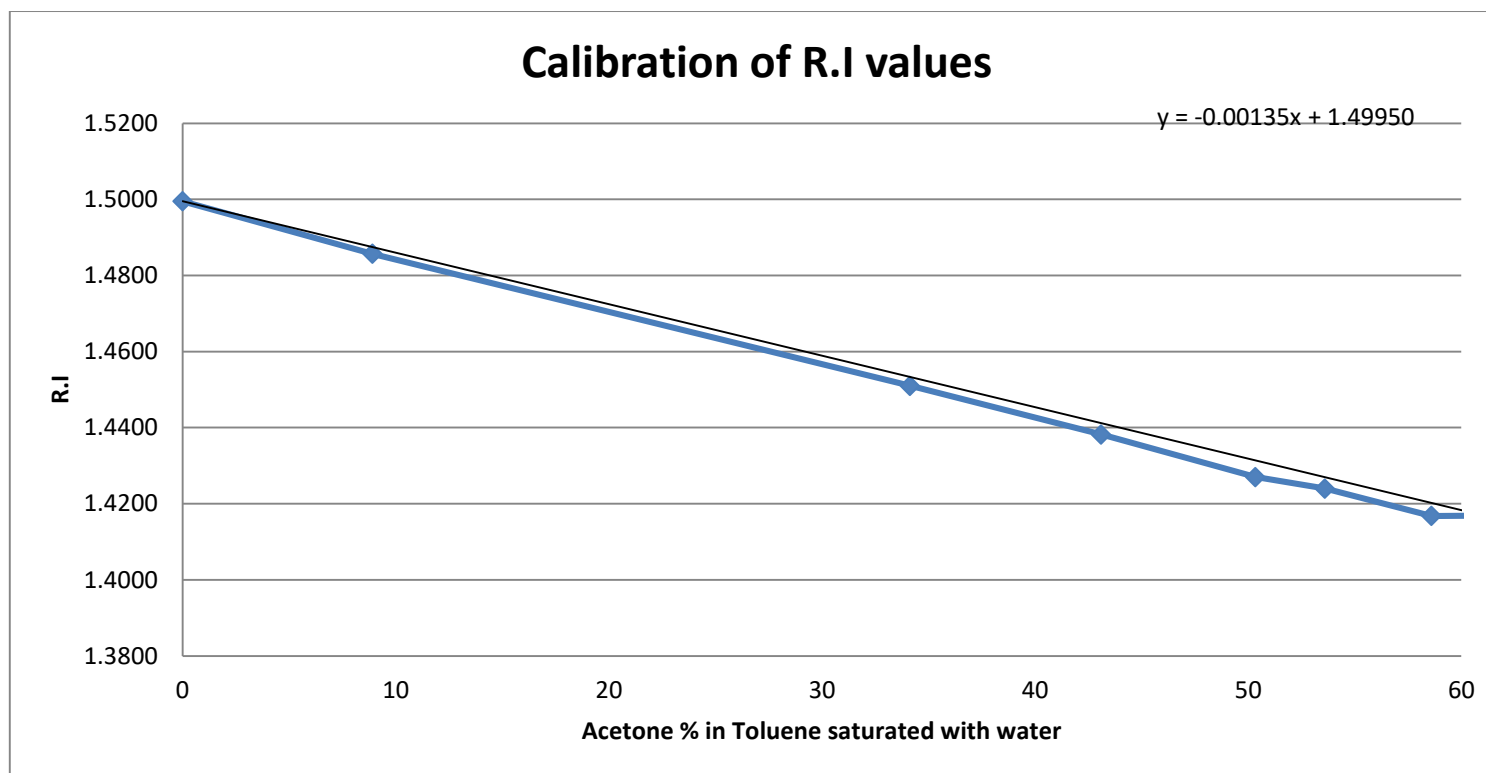


Figure (7): Wt % of Acetone in Toluene saturated with water Vs Refractive Index

## Experiment Number -3-

### Digital Joulemeter

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#### Equipment

The digital Joulemeter measures electrical energy directly in joules and power in watts. It has clear digital display which is most valuable for many demonstrations.

The instrument can be used with both direct and alternating current. It has four internal shunts to give maximum current ranges of 0.7mA, 7mA, 0.7A, and 7A, with a maximum input of 15V for (a.c.) the voltage maximum for (d.c.) is 20V with corresponding maximum current of 1mA, 10mA, 1A, and 10A. A three position "JOULES" switch applies multipliers of x1, x10, and x100 to the four ranges. In the x1 setting the right hand digit of the display will be counting in the unit written above the blue external load socket in use. In the x10 setting the right hand digit of the display will count in units ten times that written above the socket. In the x100 setting the units will be one hundred times that written.

#### Experiment 1

#### Objective

To determine the specific heat capacity of a metal.

#### Requirements

Power supply unit 12V (a.c.), immersion heater (12V, 50A), Aluminum block, calorimeter, thermometer, micrometer and 4mm plug leads.

#### Procedure

1. Connect the circuit as shown in figure (9):

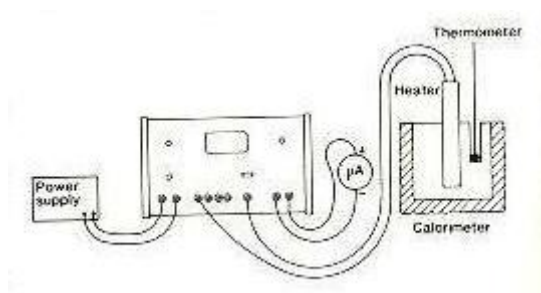


Figure (8): Specific heat capacity of metal circuit.

2. Switch on the heater and once the temperature of the block has risen to about 2°C reset the joulemeter. When the temperature of the block has risen by about 20°C read the thermometer and read the joulemeter.

## Calculations

Refer to the basic definition of specific heat capacity to calculate it for the metal used.

## Experiment 2

### Objective

To determine the specific latent heat of vaporization of liquids.

### Requirements

An isolated beaker, immersion heater, thermometer, power supply unit, and 4mm plug leads.

### Procedure

1. Fill the beaker with a certain amount of the liquid to be tested, put its cover, and then insert the heater and thermometer in it.
2. Connect the circuit as shown in figure (10):

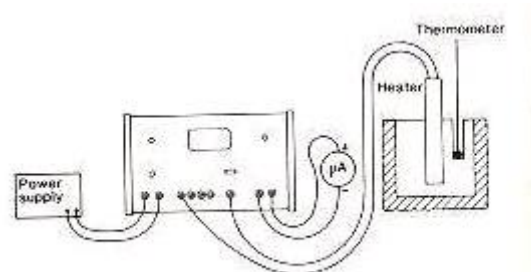


Figure (9): specific heat of vaporization of liquids circuit.

3. Switch on the heater and wait until the liquid boils and record its boiling temperature.
4. Put the beaker on a balance, record the initial weight of the beaker with the liquid in it.
5. Remove the cover of the beaker and let the liquid to vaporize, and then record the final weight of it, and the Joulemeter reading.

## Calculations

Refer to the basic definition of specific latent heat of vaporization to calculate it for the liquid used.

## Experiment 3

### Objective

To investigate the efficiency of a small electrical motor and study its variation with load and applied voltage.

### Requirements

Power supply unit (d.c.), electrical motor unit, rheostat (2A, 50A), line shaft unit, load masses, voltmeter (12V), switch (single pole), and 4mm plug leads.

### Procedure

1. Connect the circuit as shown in figure (11) using the (d.c.) power supply :

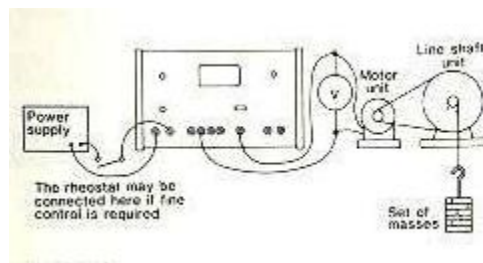


Figure (10): Efficiency of small electrical motor circuit.

2. Adjust the voltage of the power supply until the motor can lift the weight as at a convenient speed.
3. Reset the joulemeter and switch on for as long as it takes the motor to lift the weights through a measured height. Read the voltmeter during the lift.
4. Repeat the experiment at different load masses when the voltage is the same, or at different voltages when the mass lifted remains the same.

### Calculations

Calculate the efficiency of the motor, and study its variation with load and applied voltage.

## Digital Joulemeter Data Sheet

### 1. Specific heat Capacity:

Mass of AL-Block	
Joule meter reading	
$T_1$	
$T_2$	

### 2. Specific latent heat of vaporization:

Initial mass of liquid	
Final mass of liquid	
Temperature of liquid	
Joule meter reading	

### 3. Efficiency of a motor:

Change in height=.....cm

Mass of hanger=.....g

a. At constant Voltage=.....V

Mass lifted (g)	Joule meter reading

a. At constant mass=.....g

Voltage (V)	Joule meter reading

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Date:

## Experiment Number -4-

### Mixing of Powders

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#### **Objective:**

To study the process of mixing and see how the properties of ingredients - the particle size distribution as example - will affect the process. Also investigate the effect of mixing time, and mixing speed on the state of mixing.

#### **Equipment:**

The unit is composed of a double-cone mixer, with a timer, and with a speed controller.

Samples can be analyzed by an ionselective meter, or by titration method.

#### **Theory:**

The mixing index is a measure of how far mixing has proceeded toward equilibrium.

For granular solids, the mixing index is defined as  $\frac{\sigma_e}{S}$  the equilibrium standard deviation for complete mixing over standard deviation

$$I_s = \frac{\sigma_e}{S} = \sqrt{\frac{\mu_p \cdot (1 - \mu_p) \cdot (N - 1)}{n \sum_{i=1}^N (x_i - \bar{x})^2}}$$

where:

$\mu_p$ : is the overall fraction, by number of particles, of sand in the total mix.

N: is the number of spot samples.

n: is the number of particles in the sample.

$x_i$  : Fraction of sand in each spot sample.

$\bar{x}$  : Average fraction of sand in all spot samples.

#### **Procedure:**



3. Weigh certain amount of salt and sand as instructed, and load the material to the mixer.
4. Fix the speed of the mixer and run it.
5. After 5 minutes of running, stop the mixer and take 3-5 samples from different locations of the mixed material and place in sample bottles.
6. Start running the mixer again and repeat taking samples after 10, 15, 20, 30, 45, and 60 minutes.
7. Using the ionselective meter (or any analytical method), the weight of salt as well as sand in each sample taken can be found.
8. Repeat using one of the variables below:
  - a. Speed of rotation.
  - b. Moisture or liquid content of solids.
  - c. Relative amounts of components.

### **Calculation:**

1. Calculate the mixing index for each spot sample.
2. Draw graph of mixing index against the variable selected.

### **References:**

1. Coulson and Richardson, " Chemical Engineering ", Vol.II,Pergamon Press
2. McCabe and Smith," Unit Operation of Chemical Engineering ", 3rd edition.

## Mixing of Powder Data Sheet

Weight of salt: \_\_\_\_\_

Particle size of salt: \_\_\_\_\_

Weight of sand: \_\_\_\_\_

Particle size of sand: \_\_\_\_\_

Time	Sample Location	Sample wt(g)	Vol. of AgNO <sub>3</sub> (ml)
5 min	Top		
	Bottom		
	Right		
	Left		
10 min	Top		
	Bottom		
	Right		
	Left		
15 min	Top		
	Bottom		
	Right		
	Left		
20 min	Top		
	Bottom		
	Right		
	Left		
30 min	Top		
	Bottom		
	Right		
	Left		
45 min	Top		
	Bottom		
	Right		
	Left		
60 min	Top		
	Bottom		
	Right		
	Left		

Instructor signature: \_\_\_\_\_

Date: \_\_\_\_\_

## **Experiment Number -5-**

### **Fluidized Bed Heat Transfer Unit**

---

#### **Objective:**

1. To calculate the heat transfer coefficient in fluidized bed.
2. To show the effect of fluid velocity on pressure drop through the fluidized bed.

#### **Equipment:**

The equipment consists of a glass cylinder through which a controllable flow of air is passed. It is mounted vertically with a diffuser/ filter at the lower end. A further filter at the upper end prevents solid particles suspended in the air stream from escaping. Air from a blower fan is produced controlled by a bleed control valve. This valve is progressively closed causing air to pass through two flow meters in turn and then the chamber at the lower end of the glass cylinder below the diffuser/filter before passing through the solid particles held above the filter.

Two flow meters is used, one having approximately one tenth the range of the other. This ensures that both the initial onset of fluidization can be measured as well as the full range which exceeds the Fluidization velocity. An electric heating element in mounted in such a manner that it can be held at any height within the glass cylinder, the surface area of the heating element is 20 cm<sup>2</sup>. The power input is controlled by a variable transformer and the surface temperature of the element is measured by a thermocouple located close under the surface of the thin copper cylinder which surrounds the heating element. By this means the cooling effect of the air and solid particle can be accurately measured.

Additional thermocouples are placed, one under the diffuser to measure air entry temperature and the other one on a movable probe to enable temperature measurement at any part in the cylinder. The thermocouple marked "X" is located close under the surface of the copper cylinder and is coupled directly to a high temperature cut out.

Pressure drop through the fluidized bed of solid particles is measured by tubular pressure probe which can be adjusted vertically to sense pressure just above the diffuser/filter. See figure (5).

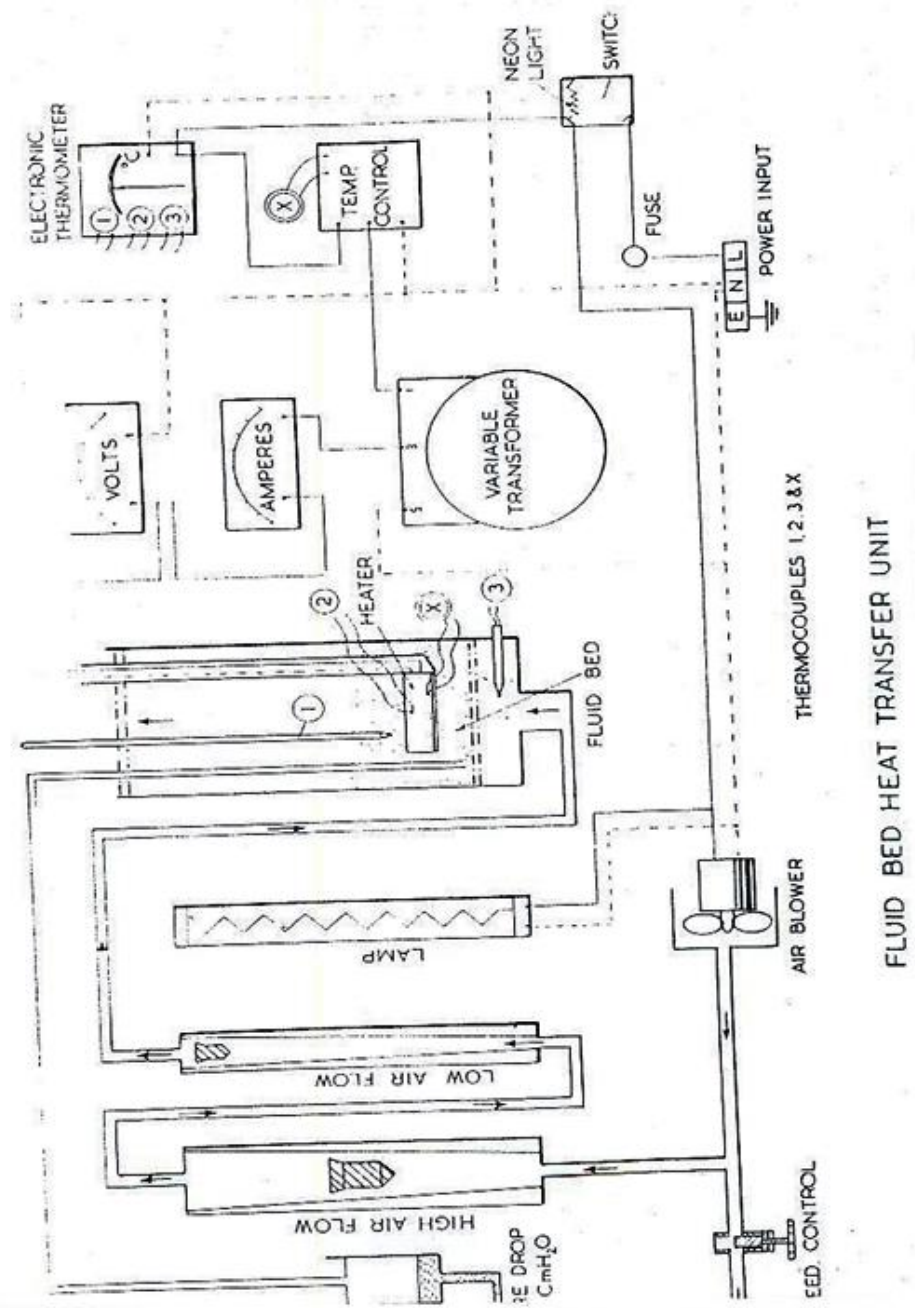


Figure (5): Schematic diagram for fluid bed heat transfer unit.

**Theory:**

Throughout the whole range of industry there can be found important processes which depend for their success upon good contact between fluid and a particulate solid. A major advantage of fluidized bed operation in heat transfer processes is that the rate of heat transfer is enhanced compared to similar conditions in traditional operations. Applications of fluidization include catalytic petroleum processes, ion exchange water treatment, separation processes and the combustion of solid fuels.

The heat transfer coefficient can be calculated by using the following equation for both (fixed and fluidized bed).

$$Q = h. A. (T_2 - T_1)$$

where:

Q: heat input (Q=V.I).

A: surface area of the heating element.

T<sub>1</sub>: temperature of the bed.

T<sub>2</sub>: Temperature of the heating element.

**Procedure:**

1. With the air bleed control opened fully, switch on the blower.
2. Immerse the heating element fully in the bed.
3. Adjust the variac to maintain the heating element at some predetermined temperature as controlled by the thermostat, and at the same time close the bleed control valve progressively so that the air is induced through the bed material causing the pressure to rise across the bed.
4. Wait for the steady state temperatures to be reached.
5. Record the flow rate, bed pressure drop, air temperature, element temperature, voltage and current at each air flow rate, and also record your observation.

**Calculation:**

1. Plot the bed pressure drop against air flow rate.
2. Plot the heat transfer coefficient against the air flow rate

**References:**

1. J.M. Coulson and J.F. Richardson, "Chemical Engineering", Vol.2, Third Edition, 1978, Pergamon Press.
2. D.Q. kern, "Process Heat Transfer ", McGraw Hill, 1982.



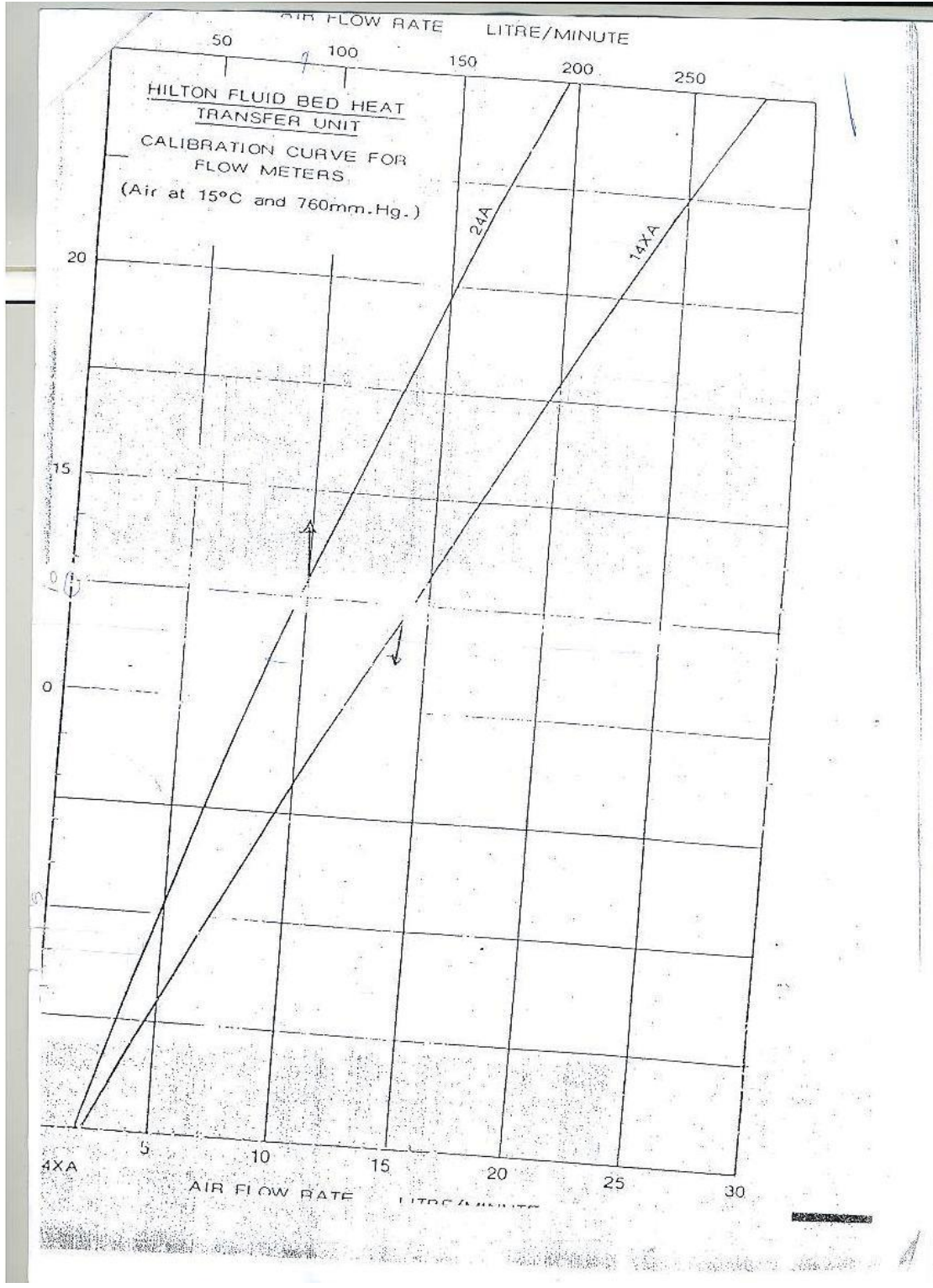


Figure (6): Calibration curve for flow meters for fluid bed heat transfer unit.

## Experiment Number -6-

### Jaw Crusher

---

#### **Objective:**

To study the comminution behavior of different materials, using a primary crusher (Jaw crusher), under various condition; taking in consideration power requirements.

#### **Equipment:**

The unit consists of a laboratory scale Jaw crusher of fixed speed. The gap between the two jaws can be altered using a hand wheel.

A sieve shaker is also available with a set of test sieves.

#### **Theory:**

A number of empirical laws have been put forward to estimate the energy required to effect a size reduction of a given material:

- a. Rittinger's law:

$$\frac{P}{m} = K_R \left( \frac{1}{L_2} - \frac{1}{L_1} \right) \dots \dots \dots (1)$$

- b. Kick's law:

$$\frac{P}{m} = K_c \cdot \ln \frac{L_1}{L_2} \dots \dots \dots (2)$$

- c. Bond's law:

$$\frac{P}{m} = 0.3162 \cdot W_i \cdot \left( \frac{1}{\sqrt{L_2}} - \frac{1}{\sqrt{L_1}} \right) \dots \dots \dots (3)$$

Where:

$L_1, L_2$ : Particles size of product and feed (mm).

P: Power required for crushing and grinding(Kw).

m: Feed rate (tons/hr).

$W_i$ : Bond's work index (Kw.hr/ton).

$K_R, K_c$ : Rittenger's constant and Kick's constant.



## **Procedure:**

1. Prepare the material to be tested and sort according to the size.
2. Choose three samples of different sizes and weigh the required amount as instructed by supervisor of each sample.
3. Adjust the jaw gap setting as required, and tighten the hand wheel.
4. Switch on the jaw crusher.
5. Put the weighed sample into the crusher and immediately start the stop watch.
6. Once the crushing is complete, stop the stop watch and record the time.
7. Arrange the test sieves according to the size of the aperture, noting that the biggest aperture should be at top, and the smallest at the bottom and then the pan.
8. Put the crushed sample on the top sieve, and put the sieves on the sieve shaker.
9. Switch on the shaker and allow the screen process to proceed for about 15 minutes.
10. Weigh the collected solid on each sieve and record the weight.
11. Repeat using the other samples.
12. The process can be repeated using one of the following variable:
  - a. Initial material size.
  - b. Jaw gap setting.
  - c. Type of material

## **Calculation:**

1. Draw graphs of accumulative weight percent of product passed against screen opening and find  $d_{80}$ .
2. Using Bond's law, find the power required for grinding.
3. Find a graphical relation between the power required and the variable selected.

## **References:**

McCab, W.L., and Smith, J.C., Unit Operation of Chemical Engineering, McGraw-Hill, Inc, 3rd ed., (1976).

## Jaw Crusher Data Sheet

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	Coarse size	Intermediate size	Fine size
Dimensions of feed particle			
Weight of sample			
Time need for crushing			

	Mass collected on sieve (g)		
Sieves size	Coarse size	Intermediate size	Fine size

Instructor sign:
Date:

## Experiment Number -7-

### Gravity Sedimentation

---

#### **Objective:**

To demonstrate how the data obtained from batch settling tests can be used in design purposes (e.g. thickener design). This can be achieved through studying the settling behavior of different materials having different properties under various process conditions.

#### **Equipment:**

The main items of the apparatus are:

1. Five easily-removable glass tubes, mounted in support frame.
2. Back lighting to illuminate the sedimentation tubes and metric scales mounted alongside.

To help complete the experiments:

1. A stopwatch.
2. Containers to make up slurry solutions in water.
3. Specific gravity (density) bottle.

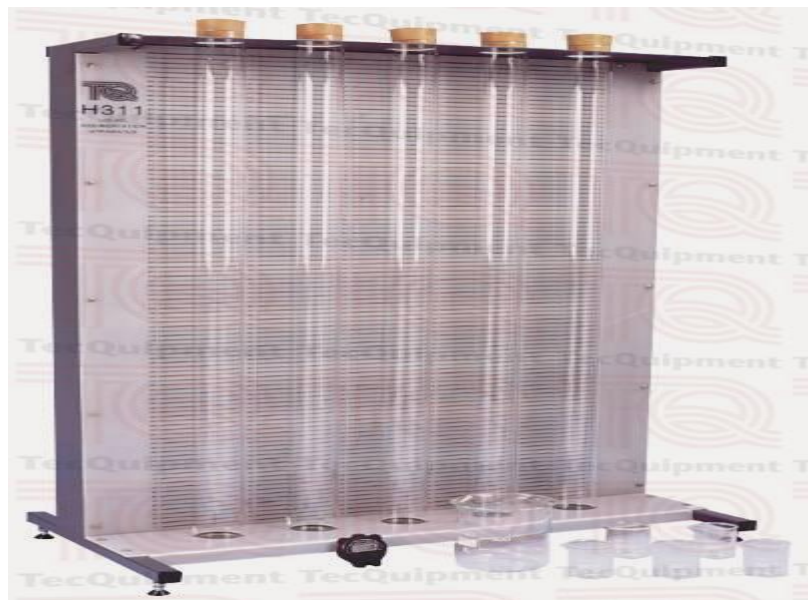


Figure (7): Schematic diagram for Sedimentation unit.

## **Theory:**

The total weight of the solid in the slurry is  $(C_0 \cdot Z_0 \cdot S)$ , where  $(C_0)$  and  $(Z_0)$  represent the initial concentration and height of the suspended solid in a batch-settling test and  $(S)$  is the cross-sectional area of the cylinder in which the test is being performed. The quantity of solid passing through the limiting layer is  $d.C_L \cdot S \cdot \theta_L(V_L + \bar{V}_L)$  where  $C_L$  is concentration of the limiting layer and  $\theta_L$  is the time for this layer to reach the interface,  $V_L$  is the setting velocity and  $\bar{V}_L$  is the upward velocity of the capacity-limiting layer.

$$C_L \cdot S \cdot \theta_L(V_L + \bar{V}_L) = C_0 \cdot Z_0 \cdot S \dots \dots \dots (1)$$

If  $\bar{V}_L$  is assumed to be constant and  $Z_L$  is the height of the interface at  $O_L$ :

then

$$\bar{V}_L = \frac{Z_L}{\theta_L} \dots \dots \dots (2)$$

Substituting into equation (1) gives

$$C_L = \frac{C_0 \cdot Z_0}{Z_L + V_L \cdot \theta_L} \dots \dots \dots (3)$$

The value of  $V_L$  is the slope of the plot of height of interface versus time and is equal to:

$$V_L = \frac{Z_i - Z_L}{\theta_L} \dots \dots \dots (4)$$

$$Z_i = Z_L + V_L \cdot \theta_L \dots \dots \dots (5)$$

Combining equation (3) and (5) gives:

$$C_L \cdot Z_i = C_0 \cdot Z_0 \dots \dots \dots (6)$$

## **Procedure:**

1. Fill one of the graduated cylinders with solution to a certain height.
2. Weigh the required amount of solid and add to the water in the cylinder.
3. Shake the cylinder to make the solution homogenous.
4. Record the height of the interface between the clear liquid and the settling slurry with time.
5. The experiment can be repeated at different :
  - a. Type of material.
  - b. Solid concentration.
  - c. Particle size.

- d. Diameter of the cylinder.
- e. Initial height.

### **Calculation:**

1. Plot the batch settling curves for each run.
2. Calculate the critical height, time, and concentration for each run.
3. Calculate the minimum height for each run.
4. Investigate the effect of the variable studied on the settling behaviour of solid.
5. Plot graphs of the variable studied versus critical height and concentration.
6. Calculate the minimum area of the thickener to handle  $3.785 \times 10^6$  L/day with feed concentration of 200 g/liter solution and thickened sludge concentration of 700 g/ liter solution.

### **References:**

1. Coulosn, J.M., and Richardson, J.F., "Chemical Engineering ", Vol. II, London.
2. Foust, A., et al., "Principle of Unit Operation", John Wiley, New York.
3. Svarovsky , L., ed. "Solid-Liquid Separation" , Butterworhts.

## Gravity Sedimentation Data Sheet

---

	Conc.=	Conc.=	Conc.=
Height	Time	Time	Time

	Conc.=	Conc.=	Conc.=
Height	Time	Time	Time

Instructor signature:
Date:

# Experiment Number -8 - Ball Mill

---

## **1.Objectives:**

1. To grind the given material to a smaller size using a ball mill.
2. To obtain the size distribution of the final mixture by sieving.
3. To analyze the results using the available theories.

## **2.Equipment and materials:**

The unit consists of:

- A laboratory scale rotary ball mill, as shown in Fig (1).
- Metal balls (various sizes).
- A sieve shaker with a set of test sieves.(Appendix A)

### **2.1. Unit description**

Ball Mill is a kind of grinder used for intermediate or fine grinding materials like ores, chemicals, ceramic raw materials and paints. The coarse material charged along with grinding medium, which fall onto the material and mill it by the effects of mechanical forces. Different materials are used as media, including ceramic balls, flint pebbles and stainless steel balls. The size reduction leads to an increase in surface area per unit volume that enhances the rate of the reaction by allowing more sites for the reaction to take place, release valuable substances contained in mixtures for further processing. Moreover improve the packaging, storage and transport conditions of materials.

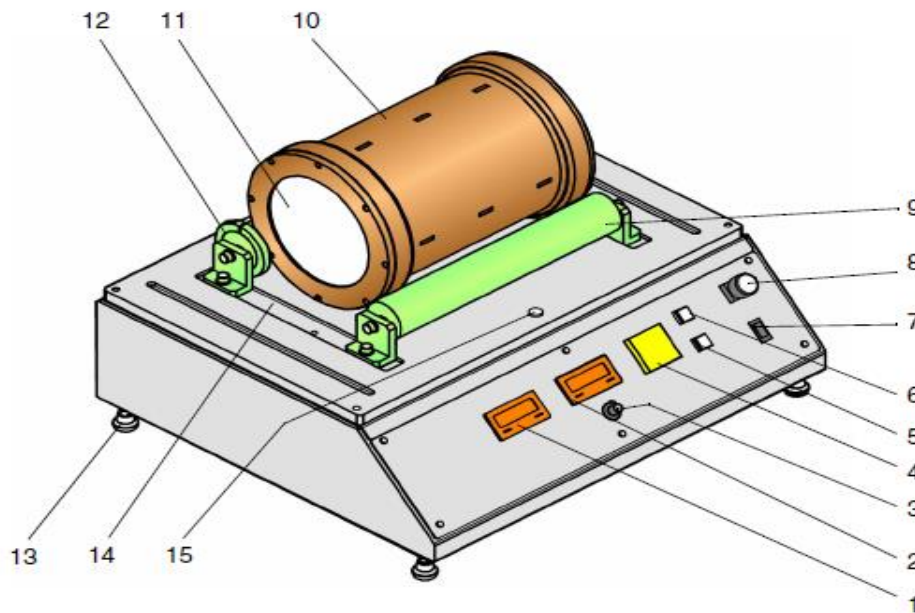
### **2.2. Unit layout**

The unit consists of:

1. Digital display of power consumption in W.
2. Digital display of speed in min-1
3. Speed adjustment of drive roller controller.
4. Timer adjustment of the milling process duration.
5. The OFF button used to stop the drive, resetting the timer to zero.
6. The ON button used to switch on the drive.
7. The master switch used to switch the unit on and its readiness for service is indicated by the lamp.
8. Emergency stop switch.
9. Loose roller.



10. The milling drum, containing 50-70% of milling balls made of stainless steel and the material to be ground .
11. Inspection window.
12. Drive roller.
13. Height-adjustable feet (Align unit to horizontal level)
14. Guide rail
15. Box level



- |   |   |    |                        |
|---|---|----|------------------------|
| 1 | Digital display<br>power consumption in W     | 9  | Loose roller           |
| 2 | Digital display<br>Speed in $\text{min}^{-1}$ | 10 | Milling drum           |
| 3 | Adjuster<br>Speed drive roller                | 11 | Inspection window      |
| 4 | Timer   | 12 | Drive roller           |
| 5 | OFF button                                    | 13 | Height-adjustable feet |
| 6 | ON button                                     | 14 | Guide rail             |
| 7 | Master switch                                 | 15 | Box level              |
| 8 | Emergency stop switch                         |    |                        |

**Fig.(1) : Ball mill layout**

### **3. Theory:**

#### **3.1. Physical principles of milling**

In various processes, solids frequently need to be reduced for subsequent processing. They are

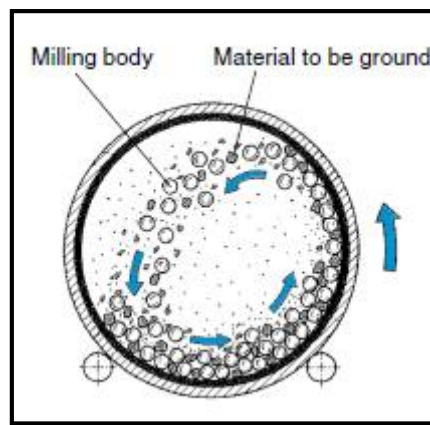
normally reduced by mechanical force. Depending on the mechanical properties of the materials, e.g. hardness, brittleness etc., reduction is carried out using different load types.

Reduction always produces mixtures of coarse and fine material in a range of different grain sizes. Graders are therefore connected downstream to separate this material. The choice of reduction method for a particular material depends particularly on the following properties:

- Particle size of basic material
- Properties of material to be ground (hardness, brittleness, viscosity)
- Required reduction.

### 3.2. Reduction principle

Ball mills are primarily used for fine grinding and pulverizing of dry hard to medium-hard materials. A ball mill consists of a rotating drum, which is filled 50-70% with wear-resistant milling bodies made of steel or hard porcelain and the material to be ground as shown in Fig(2) . When the drum rotates, milling balls are lifted against the inside wall and fall back onto the material to be ground, crushing it through friction, shearing and impact. If the speed is low, there is a wobbling motion, and reduction is achieved mainly through friction.



**Fig. (2): The working principle of ball mill**

Ball mills operate discontinuously via batch-processing. The drum size depends on the product volume. There are wet and dry milling processes. Drums can be cooled or heated and operated under a special atmosphere, e.g. nitrogen.

### 3.3 Empirical laws:

Bonds law has been used to estimate the energy required to effect a size reduction of a given material:

Bonds law:

$$\frac{p}{m} = 0.3162 W_i \left( \frac{1}{\sqrt{L_2}} - \frac{1}{\sqrt{L_1}} \right) \dots\dots\dots (1)$$

Where:

- $L_1, L_2$  : Particles size of product and feed (mm)  
 $p$  : Power required for grinding(kW)  
 $m$  : Feed rate (tons/hr)  
 $W_i$  : Bond's work index (kW.hr/ton).

#### **4. Procedure:**

1. Weigh about 100 g of the material to be tested with particle size (500-1000  $\mu\text{m}$ ).
2. Add the balls and material fraction to the drum. The volume share of material and balls is 50-70% of drum volume.

The volume share of the three balls diameters in the total ball volume is as follows:

- $\varnothing$  5mm = 25% volume share
- $\varnothing$  10mm = 50% volume share
- $\varnothing$  15mm = 25% volume share

3. Set master switch to ON position.
4. Set the required time and speed.
5. Push ON button on the drive roller, the milling process was started for required minutes with the appropriate speed.
6. Switch OFF position.
7. Separate the grounded material from the balls to prepare for the grain size analysis. A sieve with a mesh size of 1.4 mm is useful here.
8. Arrange the test sieves according to the size of the aperture, noting that the biggest aperture should be at top, and the smallest at the bottom and then the pan. (The grain size was made using an analysis sieve with the following mesh size 500,350, 250, 125, and 90 $\mu\text{m}$ ).
9. Put the grinded sample on the top sieve, and put the sieves on the sieve shaker.
10. Switch on the shaker, set the vibrating intensity (H=55), sieving time (T=10).
11. Weigh the products from the sieves, and classify them according to the size range.
12. The process can be repeated using one of the following variable:
  - a. Speed of cylinder.( 150, 250, 350, 450  $\text{min}^{-1}$ )
  - b. Filling level of cylinder.
  - c. Ball size and volume share.
  - d. Duration of milling process. (5, 10, 15, 20 min)

## **5. Calculation:**

1. Draw graphs of accumulative weight percent of product passed against screen opening.
2. Find a graphical relation between the power required and the variable selected.
3. Calculate the bonds work index and compare it with theoretical value.

## **6. References:**

McCab, W.L., and Smith, J.C., Unit Operation of Chemical Engineering, McGraw-Hill, Inc, 3rd ed., (1976).

## Ball Mill Data Sheet

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	Sample #1	Sample #2	Sample #3
Feed particle size ( $\mu\text{m}$ )			
Weight of sample (g)			
Time (min)			
Rotation speed (rpm)			
Power consumption (W)			

	Mass collected on sieve (g)					
Sieves size	Sample #1	Mass fraction %	Sample #2	Mass fraction %	Sample #3	Mass fraction %
Total						

Instructor sign:

Date:

## Appendix (A)

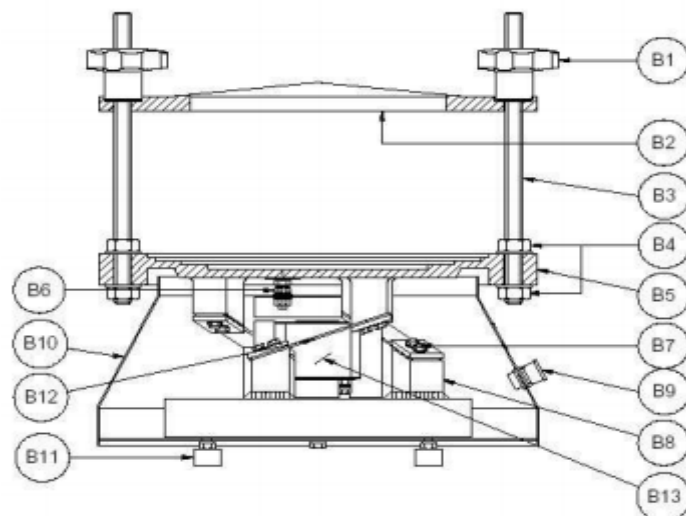
### Sieve shaker

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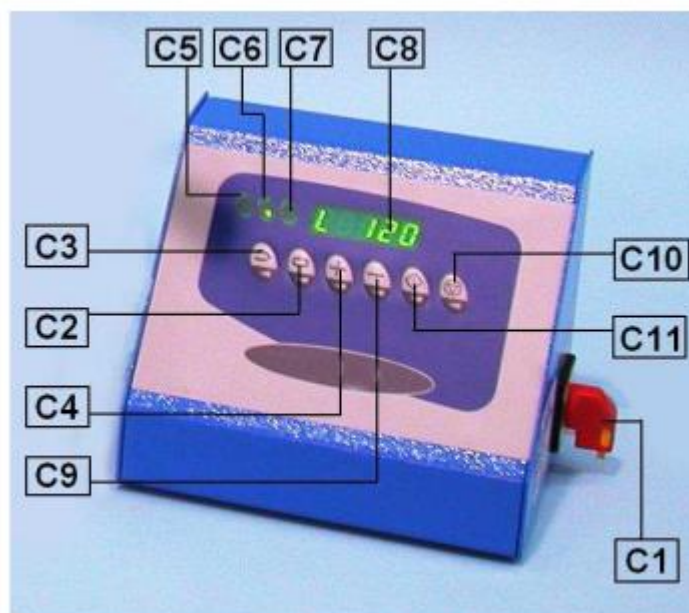
Sieves shakers can be used to sieve all the range of materials with variable intensity. They can handle many sieves at the same time.



#### Unit description



B1	BLOCKING KNOBS	They secure the upper bar (B2) to the sieves, blocking them firmly to the receiver (B5).
B2	BAR	It secures the sieves to the specimen receiver (B5).
B3	THREADED BARS	They allow to regulate the bar height (B2).
B4	FIXING NUTS	They fix the threaded bars (B3) to the specimen receiver. <b>In the shaker A059-01 KIT is present only one pair of data and the bars B2 are screwed into the sieve holder B5</b>
B5	SIEVE HOLDER	It can hold several sieves according to the operator's needs.
B6	ADJUSTMENT SCREWS	They allow to fix and to adjust the specimen receiver (B5) to the electromagnet (B13). In this way, the vibrations produced by the electromagnet are transferred to the sieves placed on the receiver (B5).
B7	FIXING SCREWS	These screws fix the springs (B12) to the support columns (B8).
B8	SUPPORT COLUMN FOR SPRINGS	It allows connecting the base to the specimen receiver (B5) by means of the springs (B12).
B9	CABLE FIXING DEVICE	It fixes the feeling cable to the sieve shaker base, avoiding its incidental pull during the normal working procedures.
B10	GUARD	It allows reaching the inside components of the sieve shaker base. The guard is fixed to the base by some screws.
B11	SUPPORTING FEET	They allow laying the sieve shaker steadily on any surface.
B12	SPRINGS	They transfer the vibrations from the electromagnet (B13) to the specimen receiver (B5).
B13	ELECTROMAGNET	It generates the vibrations, which are necessary for a sieving test. Using the Control Panel (C) it is possible to modify the vibrating intensity according to the user's needs. (See Chapter "USE" of this manual for further details).



C1	<b>MAIN SWITCH</b>	It allows switching on and off the machine.
C2	<b>“SELECTION” BUTTON</b>	By pressing this key it is possible to select one by one the figures on the right of the numeric display ( <b>C8</b> ).
C3	<b>“MODE” BUTTON</b>	By pressing this key it is possible to select the working mode of the sieve shaker. This mode is shown by the first figure of the numeric display ( <b>C8</b> ); See the Chapter <b>“OPERATING MODE”</b> of this manual for further details.
C4	<b>“INCREASE” BUTTON</b>	By pressing this key it is possible to increase the figure shown on the numeric display ( <b>C8</b> ) in that moment.
C5	<b>LED “SIEVING WORKING”</b>	The lighting of this led means that the machine is vibrating.
C6	<b>LED “CONTINUOUS SIEVING”</b>	The lighting of this led means that the sieve shaker is vibrating in the “continuous mode”. For further details, see Charter <b>“SETTING UP”</b> of this manual.
C7	<b>LED “DATA STORING”</b>	The lighting of this led means that the data shown by the display is being modified ( <b>C8</b> ). Once the data is stored, the led stops lighting.
C8	<b>NUMERIC DISPLAY</b>	Using this display it is possible to see all the information referred to the sieve shaker working procedures and also to modify the operating parameters; See the Chapter <b>“USE”</b> of this manual for further details.
C9	<b>“DECREASE” BUTTON</b>	By pressing this key it is possible to decrease the figure shown by the numeric display in that moment ( <b>C8</b> ).
C10	<b>“STOP” BUTTON</b>	By pressing this key, the sieving procedure is stopped.
C11	<b>“START” BUTTON</b>	By pressing this key, the sieving procedure is activated according to the setting up made by the operator.