PRACTICAL: 1 PSYCHOMETRIC PROPERTIES

Aim: To study the psychometric properties using psychometric chart.

Requirement: Set of dry bulb thermometer and wet bulb thermometer.

Process:

Take two mercury thermometers. Suspend one in open air. It would give dry bulb temperature. Cover the bulb of thermometer with the help of cotton cloth. Dip the covered bulb in water. It would give wet bulb temperature. Note down the dry bulb and wet bulb temperature. Find out required properties using chart.

Result:

- 1. Enthalpy at saturation = _____
- 2. Relative humidity =_____
- 3. Absolute humidity =_____
- 4. Humid volume = _____

PRACTICAL: 2

JAW CRUSHER

Aim: Crush the given material in Jaw crusher and determine average particle size and reduction ratio.

Requirement: Jaw crusher, Sieves of different mesh no., Mechanical sieve shaker, weighing balance, weight box etc.

Theory:

Crushers are the slow speed machine for course reduction of large quantities of solids. Jaw crusher operated by compression and can break large lumps of very hard materials like ores, rocks etc. It is a primary crusher.

In jaw crusher feed is adjusted between two jaws, set to form V open at the top. One jaw is fixed and nearly vertical and does not move .The other swinging jaw reciprocates in a horizontal plane. The swinging jaw makes an angle of 20 to 30 with fixed jaw. It is driven by an eccentric so that it applies greater compressive force to lumps to caught between the jaws. The jaw faces are flat or slightly blazed.

Large lumps caught between the upper part of the jaws are broken, drop into the narrower space below and are re-crushed next time the jaws close. After sufficient reduction they dropped out the bottom of the machine .The jaws open and close 250-500 time per minute.

Reduction ratio: Size of feed /Size of product.

Reduction Ratio: Reduction ratio of the diameter of largest particle in feed to the diameter of the largest particle in the discharge.

Process:

- > Clean the jaw crusher by running empty and than using air blower.
- > Run the crusher empty for few seconds.
- > Feed the solid material through the hopper slowly.
- Collect the crushed solid material at the bottom of crusher using storage beam.

- Analyze the 0.5 to 1 Kg of crushed material by sieves of different mesh no using sieve shaker, running it for 15 minutes.
- > Weigh the solid material retained on each sieve and residue.
- > Plot a) % weight retained v/s average particle size on semi-log graph paper.
 - b) Cumulative % oversize and under size on semi-log graph paper.

Observation:

- 1. Type of material taken = _____ gms
- 2. Wt. of crushed sample taken = _____ gms
- 3. Time for shaking on mechanical sieve shaker = ____ min.
- 4. Wt. of total material retain (recover) after shaking = _____ gms.
- 5. Wt. of material loss = _____ gms.
- 6. Size of feed taken = _____ microns

Observation table:

| Sr. | Screen | Screen | Average | Weight | % | Cumulative | Cumulative % |
|-----|---------|---------|----------|----------|----------|------------|--------------|
| No. | mesh | opening | particle | of solid | weight | % oversize | undersize |
| | no. | micron | size | retain | of solid | | |
| | | | | | retain | | |
| 1. | 5 | | | | | | |
| 2. | 12 | | | | | | |
| 3. | 25 | | | | | | |
| 4. | 35 | | | | | | |
| 5. | 45 | | | | | | |
| 6. | 50 | | | | | | |
| 7. | 100 | | | | | | |
| 8. | 200 | | | | | | |
| 9. | Residue | | | | | | |

Results:

- 1. Average particle size from graph = _____ microns
- 2. Reduction ratio = _____

PRACTICAL: 3 ROLL CRUSHER

Aim: Crush the given material, in roll crusher and determine average particle size and reduction ratio.

Requirements: Roll crusher, sieves of different mesh no., mechanical sieve shaker, weighing balance, weight box.

Theory:

Two heavy smooth faced metal-rolls running on parallel horizontal axis are the working element of the smooth roll crusher, particles of feed, caught between the rolls are broken in compression and drop out below. The rolls turn toward each other at the same speed. They have relatively narrow faces are large in diameter so that they can hip moderately large lumps. Typical rolls are 24" in diameter with a 12" face to 78" in diameter with 36" face. Roll speed ranges, from 50 - 300 rpm. Smooth roll crushers are the secondary crushers with' feed ½ to 3 inch in size and product ½ inch to about 20 mesh.

Reduction Ratio: Reduction ratio is the ratio of diameter of largest particle in feed to diameter of largest particle in the discharge.

Process:

- > Clean the roll crusher by running it empty and then with air blower.
- > Run the crusher empty for few seconds.
- > Feed the solid material through hopper slowly,
- Collect the crushed solid material at the bottom of the crusher using storage beam.
- Analyze the ½ to1kg crushed material by sieves of different mesh numbers using mechanical sieve shaker, running it for ½ hour.
- > Weigh the solids retained on each sieve and also residue.
- > Plot on semi-log graph paper
 - 1. % wt retained v/s average particle size.
 - 2. Cumulative % oversize & undersize v/s particle size.

Observation:

- 1. Type of material taken = _____ gms
- 2. Wt. of crushed sample taken = _____ gms
- 3. Time for shaking on mechanical sieve shaker = ____ min.
- 4. Wt. of total material retain (recover) after shaking = _____ gms.
- 5. Wt. of material loss = _____ gms.
- 6. Size of feed taken = _____ microns

Observation table:

| Sr. | Screen | Screen | Average | Weight | % | Cumulative | Cumulative % |
|-----|---------|---------|----------|----------|----------|------------|--------------|
| No. | mesh | opening | particle | of solid | weight | % oversize | undersize. |
| | no. | micron | size | retain | of solid | | |
| | | | | | retain | | |
| 1. | 5 | | | | | | |
| 2. | 12 | | | | | | |
| 3. | 25 | | | | | | |
| 4. | 35 | | | | | | |
| 5. | 45 | | | | | | |
| 6. | 50 | | | | | | |
| 7. | 100 | | | | | | |
| 8. | 200 | | | | | | |
| 9. | Residue | | | | | | |

Results:

- 1. Average particle size from graph = _____ microns
- 2. Reduction ratio = _____

PRACTICAL: 4 BALL MILL

Aim: Grind the given material in ball mill using solid metal balls and determine average particle size, reduction ratio and Rittingers constant.

Requirements: Ball mill, metal balls, weighing balance, mechanical sieve shaker, sieves of different mesh nos.

Theory:

Ball mill is a typical revolving mill. A cylindrical shell slowly moving about a horizontal axis and filled to about half of its volume with the solid and "Grinding Media". The shell is usually of steel lines with high carbon steel metal silicate rock or rubber. The grinding media is metal balls in case of ball mill or metal rods, pebbles etc. Revolving mills may be continuous or batch type. In batch type mill measured quantity of the solid to be ground is loaded through an opening in the shell, opening is then closed. Mill is revolved for specified time. It is then stopped and product is discharged. In ball mill most of the reduction is done by impact as the balls drops from top. A ball mill may be leaded with balls of different sizes. Heavy balls break away only large particles end small light particles fall only on small particle.

Action in mill:

Load of balls in mill should be such that never mill is stopped. The balls occupy somewhat half the volume of mill. In operation balls tallow the cyclic path they are picked up by the inside wall of the mill and carried to the top where they break contact with the wall, fall on material and again at the bottom to be picked again and recycled. Centrifugal force keeps the ball in contact with the wall. Balls do some grinding by slipping and rolling over each other, most of the grinding at the zone of impact.

Reduction ratio:

Reduction ratio is the ratio of diameter of largest particle in feed to discharge.

 $H.P. = (E_2 - E_1) \times 1000 = C D_F - D_P$

3600 x 746

(E₂ - E₁) = Energy actually consumed in grinding of material

Df - Dp

c = Rittingers constant

D_F = Diameter of feed particle

D_P = Diameter of product particle

Process:

- > Clear the ball mill using air blower to remove any residue solid.
- Revolve the mill empty for ½ hour and note down energy consumption in KW, KWK.
- Fill up the grinding media (metal balls) in specified number in the mill. Again revolve the mill loaded with media for ½ hour. Note down energy meter reading.
- Introduce the specified amount of solid to be grounded in mill and revolve the mill again for ½ hour. Note drawn energy meter reading.
- Discharge the material and balls from mill, separate them FROM each other. Take 500gms sample OF grounded material and shake them through no. of sieve using mechanical sieve shaker for ½ hr.
- Weigh the solid retained on each sieve. Find out average particle size of product.
- > Plot on semi-log graph paper
 - 1. % wt retained v/s average particle size.
 - 2. Cumulative % oversize & undersize v/s particle size.

Observation:

- 1. Type of material taken = _____ gms
- 2. Wt. of crushed sample taken = _____ gms
- 3. Time for shaking on mechanical sieve shaker = ____ min.
- 4. Wt. of total material retain (recover) after shaking = _____ gms.
- 5. Wt. of material loss = _____ gms.

6. Size of feed taken = _____ microns

Observation table:

| Sr. | Screen | Screen | Average | Weight | % | Cumulative | Cumulative % |
|-----|---------|---------|----------|----------|----------|------------|--------------|
| No. | mesh | opening | particle | of solid | weight | % oversize | undersize. |
| | no. | micron | size | retain | of solid | | |
| | | | | | retain | | |
| 1. | 5 | | | | | | |
| 2. | 12 | | | | | | |
| 3. | 25 | | | | | | |
| 4. | 35 | | | | | | |
| 5. | 45 | | | | | | |
| 6. | 50 | | | | | | |
| 7. | 100 | | | | | | |
| 8. | 200 | | | | | | |
| 9. | Residue | | | | | | |

Results:

- 1. Average particle size = _____
- 2. Reduction ratio = _____
- 3. Rittingers constant = _____

PRACTICAL: 5 CYCLONE SEPARATOR

Aim: Determination of efficiency of cyclone separator and to plot size v/s efficiency.

Requirements: Cyclone separator, weighing machine, collecting beams etc.

Efficiency = <u>Wt. of the course materials from separator</u> Wt. of the material in feed

Theory:

Cyclones are used primarily for separation of solids from fluids and utilize centrifuge force to effect separation. Such a separation depends not only on particle size but also on particle density, so that cyclones may be used to effect a separation on the basis of particle size or particle density but also both.

Apparatus consists essentially of short vertical cylinder, closed by flat or dished plate on top and by a conical bottom. Air with its load of solid is introduced tangentially at top of cylindrical portion. Centrifugal forces throw solid particles out against the wall and they drop into hopper. The outlet for the air is usually in the center of the top and is also usually provided with a nipple that extends inwardly into the separator to prevent the air short-circuiting directly from the inlet to the outlet. Such separators are widely used for the collecting of wood chips, heavy and coarse dusts and all manner of separations in which the material to be removed is not too fine. They may also be used for separating heavy of course materials from fine dust.

Process:

- > Clean the cyclone separator by running it empty
- > Prepare the difference feed samples using sieves of different mesh number.
- > Run the cyclone separator empty for few second with fully opened path.
- > Feed the sample through hopper

- > Run the air blower simultaneously
- Collect the separated particles of solid in different collectors from two different outlets.
- Analyze the collected material by sieves and find out weight solid retained on each sieve.
- > Repeat the process for number of samples.

Observation table

| Sr. | Wt. of sample | Wt. of course | Wt. of fine solid | % efficiency = | Avg. |
|-----|---------------|---------------|-------------------|----------------------|------------|
| No. | F gms | solid | G gms | <u>wt. of course</u> | efficiency |
| | | c gms | | Wt. of fine | |
| | | | | | |
| | | | | | |
| | | | | | |

Calculation:

% efficiency = <u>wt. of course material from separation</u>

Wt. of fine material in feed

Results:

Average efficiency of cyclone separator as obtained experimentally =

_____%

PRACTICAL: 6 STUDY OF REFRACTOMETER

Aim: To plot % composition v/s refractive index and determine % composition of given unknown mixture containing pure liquids by refractometry method.

Apparatus: Refractometer, air blower, burette, test tubes, cotton etc

Chemicals: CCl₄, Toluene, Acetone etc.

Process:

Open the prism box of refractometer and clean it with the cotton containing acetone. Let prism by dry. Now place few drops of pure liquid on the surface of lower prism. Close the prism box taking care to see that the liquid does not flow away. A film of liquid will thus be enclosed between prism focus. The cross wires of the telescope rotating eye-piece and adjust the mirror so as to get good illumination. Now, turn the prism box slowly backwards and forwards until the field of view becomes partially illuminated and partially dark. The edge of the light band will show a coloured fringe in ease of wide light source. Rotate the compensator until the prism box until this sharp edge is in co-incidence with the intersection of cross wire in telescope. Read directly index of refraction on the scale. Refractive index can be read directly up to the third decimal point and the fourth can be estimated with accuracy of about ± 0.0002 .

Repeat the above process for pure liquid to determine refractive index. Prepare the different mixture using two pure liquids on volumetric basis. Determine the refractive indices of all the prepared samples. On two co-ordinate graph paper plot volumetric composition of prepared mixture v/s refractive indices. Thus, prepare a calibration curve. Also determine refractive indices of given unknown samples and determine composition with help of calibration curve plotted,

Observations table:

- 1. Solution of CCl_4 in decreasing volume order
- 2. Solution of $C_6H_5CH_3$ in increasing volume order

| Sr. No. | Volumetric % | composition | Refractive |
|---------|--------------|-------------|------------|
| | | | index |
| | CCl₄ (%) | Toluene (%) | |
| 1. | - | 100 | |
| 2. | 10 | 90 | |
| 3. | 20 | 80 | |
| 4. | 30 | 70 | |
| 5. | 40 | 60 | |
| 6. | 50 | 50 | |
| 7. | 60 | 40 | |
| 8. | 70 | 30 | |
| 9. | 80 | 20 | |
| 10. | 90 | 10 | |
| 11. | 100 | - | |

Results:

| Sr. No. | Sample | Refractive index | Comp | osition |
|---------|--------|------------------|------|---------|
| | | | CCI4 | Toluene |
| 1. | A | | | |
| 2. | В | | | |
| 3. | С | | | |

PRACTICAL: 7 SIMPLE DISTILLATION

Aim: Study of simple distillation and determination of the relative volatility of the binary system

Apparatus: Distillation flask, condenser, Measuring cylinder, Refractometer, beaker, Heater etc.

Chemicals: CCl₄, Toluene, Acetone etc

Theory:

In simple distillation the vapour generated by boiling the liquid is withdrawn from contact with the liquid and condensed as fast as it is formed, Since the vapours are richer in the more volatile component than is the liquid left in the still, the liquid residue gets progressively weaker in the component. Hence the composition of this residue and therefore that of the vapour generated from it's continuously changes. This changing relationship is expressed well by Rayleigh's equation

$$\ln F/W = dx/y - x$$

Where, F = no. of moles of feed

W = no. of moles of residue

X = mole fraction of more volatile component in vapour

Simple distillation is resorted to when the components to be separated have widely different in boiling point and where method giving sharp separation is not necessary. The apparatus for simple distillation consists of boiler and a condenser. The relative volatility is calculated by using the relationship as follows

 $\ln F/w = \underline{1} \quad \ln \underline{X_F (1 - X_w)} + \ln \underline{1 - X_w}$

a - 1 X_w (1 - X_F) 1 - X_F

Where, F = total nos. of moles of feed

W = total nos. of moles of residue

 X_F = mole fraction of MVC in feed

 X_w = mole fraction of MVC in residue

Process:

In a clean and dry 250 ml distillation flask add 50ml ccl4 and 50ml toluene. Add few pieces of porcelain in it. Arrange the apparatus in desired manner. Heat the flask at controlled rate to give drop wise distillate from condenser continue the distillation at the system, till 30 to 35 ml distillate is collected. Note the initial and final temperature at an interval of 5ml collection of distillate and then stop the heating. Continue the collection till last drop is received from condenser. Then after transferring distillate and in 100ml cylinder. Note down the volume of distillate in 100ml cylinder. Note down the volume of distillate and residue. Determine refractive index of both. Use calibration curve for the same systematic calibration

| Sr. No. | Volume collected in | Temperature | | |
|---------|---------------------|-------------|----------|--|
| | ml | | | |
| | | Initial °C | Final °C | |
| 1. | 0 | | | |
| 2. | 5 | | | |
| 3. | 10 | | | |
| 4. | 15 | | | |
| 5. | 20 | | | |
| 6. | 25 | | | |
| 7. | 30 | | | |
| 8. | 35 | | | |

Observation table

| Sr. | stream | % volume | | Actual volume | | Total weight | | Mole fraction | |
|-----|---------|----------|---------|---------------|---------|--------------|---------|---------------|---------|
| No. | | | | | | | | | |
| | | CCl₄ | Toluene | CCl₄ | Toluene | CCl₄ | Toluene | CCl₄ | Toluene |
| 1. | Feed | | | | | | | | |
| 2. | Residue | | | | | | | | |

| Feed | Volume | Refractive index |
|------------|--------|------------------|
| Residue | | |
| Distillate | | |

Result:

Relative volatility of the given system as determined experimentally = _____

PRACTICAL: 8 RECTIFICATION

Aim: To study the rectification characteristic of binary system.

Apparatus: Distillation flask, fractionating column, condenser, heating media, refractometer, measuring cylinder, test tubes.

Chemicals: CCl₄, Toluene, Acetone etc

Theory:

Continuous rectification or fractionation is a multistage counter current distillation separation. By this for a given binary system it is possible to separate solution into its component recover}' in state of purify desired. It is frequently used operation method. The rectification unit consist of

(a) A still or re-boiler in which vapour is generated

(b) A rectifying or fractionating column through which this vapour rises in counter current contact with a descending stream of liquid,

(c)A condenser, which condenses all the vapour leaving the top of the column sending part of this condensed liquid back to the column to decant counter to the rising vapours, and delivering the rest of the condensed liquid as product. As the liquid stream descends the column it is progressively enriched with the high-boiling constituent and as the vapour our stream ascends it is progressively enriched with the low-boiling constituent, The column then becomes an apparatus for bringing these stream into intimate contact, so that the vapour stream tends be condense the high-boiling constituent from the vapour. The top of the column is cooler than the bottom, so that the liquid stream becomes progressively hotter as it descends and the vapour stream becomes, progressively cooler as it rises. This heat transfer is accomplished by actual contact of liquid and vapour and for this purpose, likewise, effective contacting is desirable.

Process:

In a clean and dry 250 ml distillation flask add 120ml CCl₄ and 80 ml toluene. Add few pieces of porcelain in it. Arrange the apparatus in desired manner. Heat the flask at controlled rate to give drop-wise distillate from condenser continue the distillation at the system, till 5ml distillate is collected. Note the initial and final temperature. Collect 20 ml of distillate and discard it. Again collect 5ml of distillate note down initial and final temperature and then collect 20 ml of distillate and discard it. After this collect 5ml of distillate note down initial and final temperature and then collect 10 ml of distillate and discard it. Repeat the procedure for last reading. Determine refractive index of all the five sample. Use calibration curve for the same for systematic calibration.

Observation table

| Distillate | % v | volume | Vol | ume ml | Wei | ght gm | N | Noles | Moles | fraction |
|------------|------------------|---------|------------------|---------|------|---------|------------------|---------|-------|----------|
| No. | | | | | | | | | | |
| | CCl ₄ | Toluene | CCl ₄ | Toluene | CCl₄ | Toluene | CCI ₄ | Toluene | CCl₄ | Toluene |
| 1. | | | | | | | | | | |
| 2. | | | | | | | | | | |
| 3. | | | | | | | | | | |
| 4. | | | | | | | | | | |
| 5. | | | | | | | | | | |

PRACTICAL: 9 STEAM DISTILLATION

Aim: Determine the vapor efficiency by steam distillation of the given sample.

Requirements: Distillation flask, Boiler, Condenser, 25 cc measuring cylinder.

Process:

Take exactly about 100ml of the sample in distillation flask. Arrange the apparatus as desired. Generate the steam in boiler and pass into distillation flask. Liquid boils condense the vapors and collect the distillate in 25 ml measuring cylinder Note down the distillation temperature. Allow distillate to separate into two layers. Note atmospheric temperature.

Observations table:

| Sr. | Sample | Total | Volume of | Volume of | Vaporization |
|-----|--------|-----------|-----------|-------------|--------------|
| No. | | Volume | upper | lower layer | efficiency |
| | | collected | layer | | |
| 1. | | | | | |
| 2. | | | | | |
| 3. | | | | | |

Result: The vapor efficiency of given liquid sample = _____

PRACTICAL: 10 LIQUID - LIQUID EXTRACTION

Aim: To determine equilibrium solubility of following system at room temperature and to plot triangular diagrams.

- 1. CHCl₃- H₂O CH₃COOH
- 2. $CCI_4 H_2O CH_3COOH$

Apparatus: Burette, pipette, stand, funnels, beaker etc.

Chemicals: Chloroform, CCl₄, CH₃COOH and Acetone.

Theory:

- Liquid-liquid extraction may be used to effect the removal of one or more of the components present in a solution by treatment with a suitable solvent. It may also be thought of as an alternative process of distillation.
- It is to be considered where separation by distillations is difficult to accomplish for any one of several reasons.
 - 1. The boiling points of the constituents may be too close together or
 - 2. Too high for the heating media available or
 - 3. So high that one or more of the constituents may decompose at the temperature involved.
- It has been of particular importance in the petroleum field, where solvent refining of lubricating oil fractions has been used widely to improve their viscosity characteristics. In addition, the recovery of aromatic from paraffinic and naphthenic hydrocarbons and caustic extraction for the removal of sulfur compounds from gasoline are extensively used in the field. Liquid-liquid extraction is used for the refining of vegetable oils, using furfural as a solvent. It is also applied in the manufacture of pharmaceuticals, particularly the antibiotics.
- In liquid-liquid extraction two phases involve are liquid simply solute is removed from one phase (raffinate) by solvent into other phase (extract). Separation of components of liquid mixture by treatment with solvent in which

one or more of desired component is preferentially soluble known as "liquidliquid extraction" Overall extraction process comprising of bringing solvent and solution in intimate contact. Separating the resulting two phases and removed and recovered of solution from each phase.

Compositions of ternary system are conveniently represented by points on triangle diagram covering the entire composition range. Composition may be expressed as mass or mole fraction basis. Such diagrams are used to show phase relationship in the ternary liquid system. When two component B and C are particularly miscible with each other and complete miscibility exist between B and A, C and A solubility relationship all shown as



Curve F.P.G. represents solubility limit and curve is known as "saturation curve": Area under this curve represents two phase region which will split up into layer in equilibrium with each other. These phases are known as "conjugate phases". Line ER connecting conjugate phases is known as "Tie line". Number of tie line may be constructed in two phase region. Tie line disappeared at point 'P' known as 'Plait point' where two conjugate phases become mutually soluble.

Process:

Part: I

10 ml of distilled water is taken in a clean 150 ml conical flask add 1 ml of CHCl₃. Two phases are observed. In the content of the flask now add drop-wise acetic acid with the help of burette till a single phase is observed. Note the amount of acid added. Again adds 1 ml of CHCl₃ in the same flask and add acetic acid till a single phase is observed. Note the amount of acid added. Continue the process upto the addition of 10 ml of CHCl₃.

Part II

10 ml of CHCl₃ is taken in a clean and dry conical flask. Add 1 ml of water. Two phases are obtained in the flask. Add acetic acid drop-wise to get a single phase. Repeat the process for the addition of 10 ml of water. Note the amount of acetic acid added for the addition of each ml of water.

Result: The equilibrium solubility of a ternary systems

- 1. CHCl₃- H₂O CH₃COOH
- 2. $CCI_4 H_2O CH_3COOH$

are shown on triangular graph.

PRACTICAL: 11

STUDY OF CRYSTALLIZATION WITHOUT AND WITH SEEDING

Aim: To determine practical yield of crystallization of hydrated copper sulfate crystals from its saturated solution at 50°C carrying out crystallization.

- 1. Without seeding
- 2. With seeding

Apparatus: Evaporating dishes, beaker, funnel, burette, thermometer etc.

Chemicals: Hydrated CuSO₄ Crystals.

Theory:

Crystallization process is one in which new phase is created within homogenous mass by formation of solid particles. The drying force for crystallization process is different in the solubility. In the saturated solution the crystal will neither form nor grow but for crystallization system should be supersaturated. Supersaturated may be obtained by

- Simple cooling
- Evaporating a portion of solvent
- > And adding thin substances, which decrease solubility of dissolved solid,

Crystallization process completes in two steps:

Nucleus formation

(1) Crystal growth

Addition of artificial nucleation supports growth and yield of crystals. In initial crystallization seeding is practiced to favour quality and yield.

Process:

Take 2 dry and clean evaporating dishes and label them A and B. Weigh the dishes accurately. Place 10.45 gm CuSO₄.5H₂O crystal and 16.5 ml of water in each dish using the burette. Heat the contents of each dish slowly by stirring using glass rod. Dissolve the crystals in water maintaining the temperature to 50°C to get saturated solution. Remove the dishes from burner cool dish A without addition to room temperature. Cool dish B after immediately adding 0.4 gm CuSO4.5H₂O

crystals. Cool the dishes to room temperature. Determine the weight of dishes with their contents. Filter and dry crystals in air. Determine weight of dried crystals from both dishes separately.

Observation:

| Sr. | Observations | Without seeding | With seeding |
|-----|----------------------------------|-----------------|--------------|
| No. | | | |
| 1. | Weight of dish | | |
| 2. | Weight of dish + CuSO4.5H2O | | |
| 3. | Weight of dish + solution (after | | |
| | cooling) | | |
| 4. | Weight of crystal after cooling | | |
| 5. | Weight of seed added | | |
| 6. | Time for cooling | | |

Calculation:

For theoretical yield of crystals

1. Without seeding

10.45 = C + (3-1-C)25/100

C =

2. With seeding

(10.45 + 0.4) = C + (3-1-C)25/100

C =

3. % yield

Results:

| Sr. | Types of crystallization | Theoretical | Practical yield | % yield |
|-----|--------------------------|-------------|-----------------|---------|
| No. | | yield | | |
| 1. | Without seeding | | | |
| 2. | With seeding | | | |

PRACTICAL: 12 CRYSTALLIZATION (EFFECT OF COMMON ION)

Aim: To study the % yield of crystallization of $Ba(NO_3)_2$ crystal from its saturated solution at 70°C by carrying out crystallization using

- 1. Simple cooling
- 2. Addition of common ion (nitric acid)

Requirement: Ba(NO₃)₂, Beakers, HNO₃ etc.

Process:

In 500ml clean and dry beaker take 200ml of distilled water. Heat it to 70°C. Add 87gms of Ba(NO₃)₂ crystal in it. Stir continuously the content of beaker by maintaining temperature 70°C until all crystal has been dissolved. Immediately divided the solution into two equal parts. Add 10ml conc. HNO₃ to one beaker. Allow both beaker to cool to room temperature. Separate the crystal and dry it. Weight out the crystal exactly. Also measure the volume of mother liquor

Calculation:

Ba(NO₃)₂ in mother liquor

- $X_1 = V_1 X 11.6$ $X_2 = V_2 X 11.6$ 100 100
- % yield = $\underline{C_1 X 100}$ $C_1 + X_1$ % yield = $\underline{C_2 X 100}$ $C_2 + X_2$

Results:

Yield of crystallization

- 1. Simple cooling = ____%
- 2. By adding common ion nitric acid = ____%

PRACTICAL: 13 BATCH SETTLING TEST

Aim: To verify law of settling and to find out the constants in the equation au^b.

Requirements: CaCO₃ powder, Measuring cylinder (1 liter), glass stirrer, stops watch. **Theory:**

Rate of settling decreases with the initial concentration of slurry, owing to measured effective density and viscosity of the media through which the particle settles. The curve plotted shows the effect usually to be expected when slurry of same substances are settling in the column of the same height. Various attempts are made to predict the effect of concentration on the settling rate from the knowledge of curve at one or more concentration. It has been established that settling rate is related to initial concentration

of any type of slurry as

Z/Zi = aw^b Z = Height of slurry at infinite time W= Concentration of slurry a & b= Empirical constants Zi= Height of slurry initially

Process:

Prepare the slurries of CaCO₃ in water of concentration 20 gm/liter, 30 gms/liter and 40gms/liter in different beakers. Now take three measuring cylinders of one liter capacity and fill them with prepared slurry. Initial heights of the slurries are noted. Cylinders are numbered as 1, 2 and 3. Slurries are stirred uniformly using glass rod to have slurry of uniform density. Now allow the slurry to settle without any disturbance. The time corresponding to position of demarkation line between clear liquid and settling slurries is noted in each cylinder. After noting sufficient readings cylinders are stand undisturbed for long time to note the height of infinite time.

Observation table:

| Sr. No. | Height of slurry cm | Time in second | | | | | |
|------------|------------------------|----------------|-------|-----|-------|-----|-------|
| | | 20g | m/lit | 30g | m/lit | 40g | m/lit |
| | | I | II | I | II | I | II |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

Result: The value of constants

b =

PRACTICAL: 14 ADSORPTION OF OXALIC ACID ON CHARCOAL

Aim: To study the adsorption of oxalic acid on charcoal.

Requirement: Conical flask, stopper bottle, funnel, oxalic acid solution, NaOH solution

Process:

Prepare five sets of water and oxalic acid as shown in observation table. Add one gram of activated charcoal. Mix well and stir to obtain equilibrium. Allow the solution to stand for half an hour. Filter the solution and titrate 5ml of it with 0.1N NaOH solution. Add phenolphthalein indicator. Also take blank reading.

Observation table:

| Sr. | 0.5M | Water | Burette | | Conc. of acid solution | | Adsorption | Log | Log |
|-----|--------|-------|------------|----------------|----------------------------|-------------------|-------------|----------------|-----|
| No. | oxalic | | reading | g | | | coefficient | C ₂ | X/M |
| | acid | | | | | | (X) | | |
| | | | Initial | Final | $C_1 = (0.1 \times R_1)/5$ | $C_2=(0.1xR_2)/5$ | | | |
| | | | R 1 | R ₂ | | | | | |
| 1. | | | | | | | | | |
| 2. | | | | | | | | | |
| 3. | | | | | | | | | |
| 4. | | | | | | | | | |
| 5. | | | | | | | | | |

Result: Adsorption of acid on charcoal is = _____

PRACTICAL: 15 PARTITION COEFFICIENT

Aim: To determine partition co-efficient of benzene in benzoic acid

Requirement: Benzene in benzoic acid, 0.2N NaOH solution

Process:

Prepare three solution set as follows

- 1. 50 ml benzoic acid in benzene + 50 ml water
- 2. 35 ml benzoic acid in benzene +15 ml benzene + 50 ml water
- 3. 25 ml benzoic acid in benzene + 25 ml of benzene + 50 ml water.

Shake each glass-stoppard bottle thoroughly for 15 minutes. Allow the mixture to separate into two perfectly clean layers. Separate the two layers with the help of separating funnel.

Pipette out 10ml of aqueous layer and titrate against 0.02 N NaOH Solution Using phenolphthalein indicator. Pipette out 5 ml of organic layer, add 25 ml of water and titrate against 0.1 N NaOH solution. End point is colorless to pink.

Observation table

| Sr. | Volume | Volume | Normality | Normality | Concentration | Concentration | C1/C2 | C_1/C_2 |
|-----|---------|---------|-----------|----------------------|---------------|----------------------|-------|-----------|
| No. | of 0.02 | of | of | of | of benzoic | of benzoic | | |
| | NaOH | 0.1NaOH | benzoic | benzoic | acid in aq. | acid in org. | | |
| | for | for 5ml | acid in | acid in | layer C1 | layer C ₂ | | |
| | 10ml of | of org. | aqueous | organic | | | | |
| | aq. | layer | layer N1 | layer N ₂ | | | | |
| | layer | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

Result: Partition co-efficient of benzoic acid = _____

PRACTICAL: 16 PLATE AND FRAME FILTER PRESS

Aim: Study the variation of rate of filtration with time using a plate and frame filter press, under constant pressure filtration.

Requirements: Plate and frame- filter press, slurry preparing tank, weighing balance.

Theory:

A filter press contains a set of plates and frames designed to provide a series of compartments in which solid may collect. The plates are covered with a filter medium such as canvas. Slurry is admitted to each compartment under pressure, liquid passes through canvas and out a discharge pipe leaving a wet cake of solids behind.

Plates of filter press may be circular or square, vertical or horizontal. Plates and frames of 6" to 56" on a side with 1/4" to 2" thickness set vertically in a metal rack, with the cloth covering the face of each plate and are squeezed tightly together by a screw. Slurry enters at one end of the assembly of plates and frames. It passes through the channel running lengthwise through one corner of assembly. Auxiliary channels "carry slurry from main inlet channel into each frame, solids are deposited on the cloth covered faces of the plates. After assembly of press, slurry is admitted from a pump under press. Filtration is continued until liquor no longer flow out of the discharge. This occurs when frame are fully of solids and no more slurry can enter. The press is then opened, cake of solids scraped of the filter medium and drooped to storage bin.

Mean speciriv cake resistance = $Kp A^2 (-P)g$

Ρ

Filter cake or filter media resistance (Rm) = $B^{\circ} A^{\circ}$ (-P) g_{c}

p= viscosity of fluid

A = Effective area of filtration

Kp = Constant

(Calculated from graph ploted between V & T/V)

B = Intercept

Kp = Slope

C = Mass of solid deposited per unit volume of filtration

g_c = Gravitational constant

Process

- > Clean the each part of filter press using water as well as slurry preparing tank.
- Prepare the slurry of CaCO₃ powder in water of concentration as specified in slurry preparing tank and arrange the equipment as desirable.
- Stirrer the slurry for few minutes using mechanical agitation to get the slurry of uniform density. Allow the uniform slurry to come in the filter press.
- Slurry will get filtered as a result filtrate will come out through various outlet cooks of plates and solid will be retained in between the filter cloth supported over frame.
- Collect the filtration in a graduate collecting tank noting the time simultaneously for collection of each half liter of filtrate.

PRACTICAL: 17 PUMPS AND VALVES

Aim: Study of Pumps and Valves

Valves:

The value is a device used either to control the flow rate or to shut off the flow of fluid through pipelines and process equipments. Various types of values are used in chemical industry and are constructed out of variety of materials based upon the service requirements. The various type of values in use are:

| 1. Gate valve | 4. Plug valve | 7. Butterfly valve |
|----------------|--------------------|---------------------------|
| 2. Globe valve | 5. Diaphragm valve | 8. Check/non return valve |
| 3. Ball valve | 6. Needle valve | 9. Control valve etc. |

The gate value is commonly used to minimize the pressure drop in open position and to stop the flow rather than controlling it. The globe value is used to control the flow but the pressure drop through globe value is much greater than that for gate value. The needle value is a modification of globe value and generally used for accurate control of flow. The plug value and ball value are used for on-off service and operate through 90°. The non-return value is used when unidirectional flow is desired.

Diaphragm valves are used for fluids such as viscous liquids, slurries or corrosive liquids. They make a use of flexible diaphragm usually of rubber. The butterfly valve is used in large size pipeline and operates on same principle as a damper in a stove pipe. The control valves are used in modern chemical processes for controlling flow automatically. These, valves are operated either electrically or pneumatically. Fig. 7.2 shows various types of valves.



PRACTICAL: 18 HEAT EXCHANGER

Aim: Study of heat exchanger

Double pipe heat exchanger:

It is the simplest type of heat exchanger used in industry. It is used when required heat transfer area is relatively small.

It consists of concentric pipes, connecting tees, return heads and a return bends. The packing glands support inner pipe within the outer pipe. The double pipe heat exchanger arranged in two legs [i.e. when two lengths of inner pipe are connected by return bend.] as shown in Fig. 5.1 is known as a single hair-pin. The tees are provided with nozzles or screwed connections for permitting the entry and exit of the annulus fluid which crosses from one leg to the other through the return head. The return bend connects two legs of inner pipes to each other. This exchanger can be very easily assembled in any pipe-fitting shop as it consists of standard parts and it provides inexpensive heat transfer surface. In this exchanger, one of the fluids flows through inside pipe and other fluid flows through the annular space created between two concentric pipes either in co-current or counter current fashion. It is usually employed for decreasing the temperature of hot fluid with the help of cold fluid when flow rates are low.

These exchangers are usually assembled in effective lengths of 3.65, 4.57, 6 m. The distance in each leg over which the heat transfer occurs is termed as the effective length.



Fig. 5.1 (a) : Double pipe heat exchanger



Fig. 5.1 (b) : Schematic diagram of double pipe heat exchanger (counter current)

The major disadvantages of double pipe heat exchanger are:

- Small heat transfer surface in large floor space as compared to other type (e.g. shell and tube heat exchanger).
- > Dismantling etc. requires large time and
- Maximum leakage points.

Apart from this/the double pipe heat exchanger is very attractive where the total heat transfer surface required is small, 9.29m² to 14m² or less. This is simple in construction, cheap and easy to clean.

PRACTICAL: 19 REYNOLDS EXPERIMENTS

Aim: To study the Reynolds experiments.

Requirements: Reynolds apparatus

Process:

The experimental set up is shown in figure. It consists of horizontal glass tube with flared entrance immersed in a glass walled constant head tank filled with water. The flow of water through glass tube can be adjusted to any desired value by means of valve provided at the out let. The capillary tube connected to small reservoir containing water soluble dye is provided at the center of flared entrance of glass tube for injecting dye solution in the form of fine or thin filament into stream of water.

By introducing water soluble dye into flow of water, the nature of flow could be observed. At low flow rates, the filament of coloured water retained at the axis of tube i.e. It flowed along with the main stream without any lateral mixing. These indicate that flow was in the form of parallel streams which did not interfere with each other. This type of flow patterns is known as laminar or streamlines flow. As the flow rate is increase a velocity is reached which is known as critical velocity, oscillation appears in the coloured filament, it than broken into eddies causing dispersion across die tube section. This type of flow pattern is known as turbulent flow. In between, the laminar and turbulent flow is a transition flow. Reynolds observed that the critical velocity for transition from laminar to turbulent depends upon the diameter of pipe, average velocity of flowing fluid, density of fluid and viscosity of fluid.

Results: Two types of flow were observed.