PROCESS METALLURGY LABORATORY
(MSE – 314)

Department of Materials Science & Engineering
Indian Institute of Technology, Kanpur

6th Semester
(2013-2014)

Instructor Incharge

Prof. Rajiv Shekhar

GENERAL INSTRUCTIONS

1. Every student should obtain a copy of the MSE-314 laboratory manual.

2. Dress code: Students must come to the laboratory wearing: (1) trousers, (ii) half-sleeve tops and (iii) Leather shoes. Half pants, loosely hanging garments and slippers are not allowed.

3. To avoid any injury, the student must take the permission of the laboratory staffs before handling the machines.

4. Students must ensure that their work areas are clean.

5. At the end of each experiment, the student must take initials from the staff on your data/observations.

6. Laboratory report must be submitted in standard sheet, available at the shopping center in the subsequent lab turn. Reports on ordinary sheets and computer papers will not be accepted.

7. Each member of any group must submit lab report even if the experiment has been performed in a group.

8. The lab report must contain: (1) Title of the experiment, (ii) Three to four lines stating the objectives, (iii) A few lines on background; (iii) Name of all equipments/tools used along with one line description of its use.

9. Student can check their laboratory reports after correction for discussion.

10. **Careless handling of machine will not be tolerated.**

GENERAL INFORMATION

In this laboratory you will be exposed to various aspects of phase diagrams, microstructure, quantitative metallographic techniques and phase transformation behaviour of alloys. You will be doing experiments on different types of materials and thereby understanding the phase formation and microstructure of the materials. You will also carry out experiments on determination of phase diagram using cooling curves of different alloys. You are required to submit a report on each experiment you will be performing. You will be asked questions on various aspects of the experiments. You need to refer to the following text books.

RECOMMENDED READING

1. W.M. Rohsenow and H.T. Choi: Heat, Mass and momentum Transfer, Prentice-Hall, U.K., 1961, Ch. 6 and 7.
2. W.H. McAdams: Heat Transmission, McGraw Hill Book Co.., 3rd Ed.
3. R. Schuhmann, Jr. : Metallurgical Engineering, Vol. 1, Addison Wesley, 1952, PP. 155, 161-68
4. J.H. Perry, Chemical Engineering Handbook McGraw Hill Book Co.., 4rd Ed. , 1963, Sec. 5-5 to 5-14.
5. S. M. Aeron, P.B. Crimes and F.D. Richardson, Trans. IMM, 83, p. C168 (1974).
6. K. Hauffe, The Mechanism of Oxidation of Metals and Alloys at High Temperatures, Progressin Metals Physics, 4 (1953) 71-104.
7. K. Hauffe, Oxidation of Metals, Plenum Press, New York, 1965.
8. Non-Ferrous production Metallurgy-Bray, J.L.
9. Hand book of Non-Ferrous Metallurgy-Liddell.
10. Extractive Metallurgy of non-ferrous metals-W.H. Dennis.
11. J. Om. Boekirs and A.K.N. Reddy, Modern Electrochemistry, VOL. 2, Plenum Press, N.Y.
12. Agglomeration (1962), New York, Welley and Sons, Knepper W.A.
13. J.O.M. Bockris, “Physicochemical measurements at high temperatures”, Butterworths Scientific Publication, 1959
14. Temperature: Its Measurement and control in Science and Industry, Roinhold Publishing Co., New York, Vol. I & II
15. R. Schuhmann, Jr.: Metallurgical Engineering, Vol.-I, Engineering Principles , Addition Wesley Publishing company, U.S.A. 1952, Ch.-4 or any standard book on fuels.

List of experiments FOR Semester 2013 – 14 (II)

PHASE- 1

1. Measurement of heat-transfer Coefficient
2. Flow of gases through beds of solid particles
3. Mass Transfer between Solid and Liquid in gas stirred ladles
4. Gas solid-Equilibrium during decomposition of calcium carbonate
5. Design of an electric resistant furnace and calibration of thermocouple
6. Proximate analysis and calorific value of solid fuel

PHASE II

7. Crushing and Grinding of ore
8. Roasting of Zinc Sulphide
9. Kinetic oxidation of copper
10. Electrolysis of aqueous solution
11. Mineral Beneficiation using floatation

EXPERIMENT- 1

HEAT TRANSFER COEFFICIENT

AIM

The present experiment is aimed at finding an average value of heat transfer coefficient under different fluid flow conditions.

THEORY

Heat exchange between the solid and fluid is said to occur due to convection whenever there is a difference in temperature between two phases and there is motion in the fluid. If the motion of the fluid is caused by the buoyancy forces which arise due to variation in the density of the fluid with temperature, the process is called free or natural convection. If the motion of the fluid is caused by external means such as blower, wind, fan or pump, the process is called as forced convection. Heat exchange due to convection may be described in terms of a heat transfer coefficient ‘h’ by Newton’s law of cooling as:

$$Q = Ah(T_S - T_F)$$

A is the area of the solid-fluid interface, T_S and T_F are the solid and fluid temperatures respectively. The value of heat transfer coefficient could depend upon the following factors:

(a) Type of the fluid motion.
(b) Properties of the fluid used.
(c) Geometry of the solid-fluid interface.
(d) Difference in temperature between the solid and that of the fluid.
(e) Position along the surface of the solid body.

On the assumption that there is no variation in temperature within the solid body, heat balance gives

$$MC_P \frac{dT_S}{dt} = -Ah(T_S - T_F)$$

M is the mass of the solid body and C_P is the specific heat capacity of the solid.
Solving the differential equation by taking the initial temperature of the solid as T_I, one may obtain temperature, T_t as a function of time as follows:

$$\theta_t = \frac{(T_t - T_F)}{(T_I - T_F)} = e^{-\frac{hAt}{MC_P}}$$

The above equation shows that the plot of ‘$\ln\theta_t$’ versus time ‘t’ will be a straight line with a slope (hA/MC_P). Heat exchange area ‘A’ may be obtained from the dimensions of the solid. M is the mass of the solid. Heat capacity data, C_P is made available from the literature. This lumped parameter solution is reported to be valid as long as the dimensionless Biot number is less than 0.1, i.e.

$$B_I = hL / K < 0.1$$

K is the thermal conductivity of the solid and L is the characteristic length of the body, i.e. the volume to interface area ratio. If the Biot number is greater than 0.1, variation in temperature within the solid body may no longer be ignored, and solution of the following differential equation must be considered:

$$\frac{\partial T}{\partial t} = \alpha\left[\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2}\right] \text{or } \frac{\partial T}{\partial t} = \alpha \nabla^2 T$$

A three dimensional numerical solution, even in simple cases, requires help of computer.

In practice we reduce a three dimensional problems to one dimensional one. This is achieved by making the body an infinite (very large length) cylinder or an infinite slab (with finite thickness and very large surface) etc.

The above equation in one dimension becomes

$$\frac{\partial T}{\partial t} = \alpha \frac{\partial^2 T}{\partial x^2}$$

Heisler Chart:

The solution of the equation is given in the chart (attached). The following dimensionless numbers and graphs are frequently used:

Θ (Dimensionless Temperature) = $\frac{(T_t - T_F)}{(T_I - T_F)}$

Biot Number = hL/K (W/m^2 ^{o}C)

L = Characteristic dimensions of the body (m)
= ½ thickness of a plate.
= Radius of a cylinder.
= Radius of a sphere.
h = heat transfer coefficient (W/m^2 /^{o}C)

K = thermal conductivity (W/m ^{o}C)

Fourier number Fo = $\alpha t/L^2$

α = thermal diffusivity = K/ρC_P (m^2/s)
ρ = density (Kg/m^3)

The solution of the equation takes the form

$\Theta = \Psi$ (Bi, Fo, x/L)
x = coordinate of the point in the body at which temperature or time is to be determined.

First determine Fo and Biot number from the physical properties of the body. Read the temperature Θ for these Biot and Fourier numbers.

PROCEDURE

1. Note down the room temperature and the temperature of the water bath.
2. Measure diameter, length and mass of the aluminum cylinder.
3. Attach the junction of the thermo-couple to the aluminum cylinder.
4. Lower the cylinder to the uniform temperature zone of the furnace.
5. Connect the open ends of the thermocouple wires to mili-voltmeter or the X-Y recorder.

6. Select the appropriate range of the measuring instrument as well as the time scale of the recorder.
7. Wait till the temperature of the cylinder has reached a steady value.
8. Take out the cylinder with the hot junction of the thermocouple still attached to it from the furnace and allow it to cool in the selected medium.
9. Note or record the temperature as a function of time till it has cooled to around 200^{o}C or less.
10. Repeat steps 3 to 9 for each of the three medium, i.e. still air, forced air and water bath.

REPORT

1. Plot temperature versus time for all experiments on one graph paper.
2. Plot the dimensionless temperature, i.e. $(T_t – T_F)/(T_I – T_F)$ versus time and $\ln[(T_t – T_F)/(T_I – T_F)]$ versus time in all cases.
3. Determine the slope of the line, .i.e. (hA/MC_P) from the plot in step 2 above.
4. Calculate the heat transfer coefficient ‘h’ and Biot No. i.e. (hL/K) from the slope.
5. Determine the heat transfer coefficient using Heisler’s chart.

DATA

	Units	Aluminum	Air	Water
Density	Kg/m^3	2700	1.18	1000
Specific Heat Capacity	J/Kg^{o}K	896	1.006	4180
Thermal Conductivity	J/ms ^{o}K	204	0.026	0.597

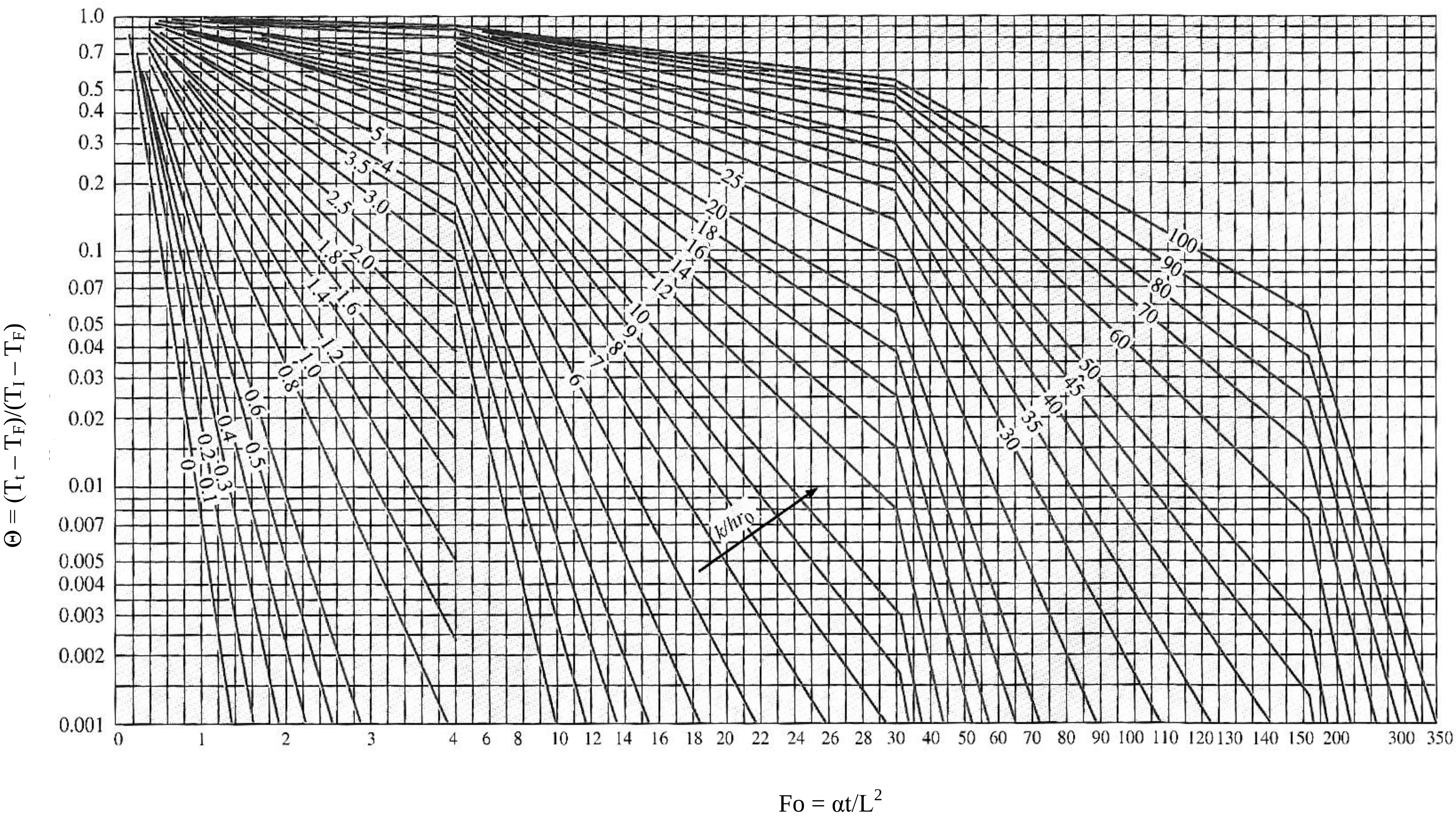
Determination of Heat Transfer Coefficient
DATA SHEET

Date of Experiment: ……………………….

Name Roll No.
(1) ……………………………….. ……………………………….
(2) ……………………………….. ……………………………….

Room Temperature: ………………..^{o}C
Water Bath Temperature: ………………..^{o}C
Length of the cylinder: ……………….. mm
Diameter of the cylinder ……………….. mm
Mass of the cylinder ……………….. g

Air			Forced Air			Water	
Time (min)	Temperature (^{o}C)		Time (min)	Temperature (^{o}C)		Time (sec)	Temperature (^{o}C)
0			0			0	
0.5			0.5			2	
1			1			4	
2			1.5			6	
3			2			8	
4			3			10	
6			4			15	
8			6			20	
10			8			25	
15			10			30	
20			12			40	
25			14			50	
30			16			60	



Transient temperature chart for a long cylinder of radius r=b subjected to convection at the boundary surface r=b (From Heisler). Dimensionless temperature is at the axis of the cylinder.

EXPERIMENT- 2

FLOW OF GAS THROUGH BEDS OF SOLID PARTICLES

AIM

To determine the pressure drop as a function of flow rate of gas passing through beds of solid particles of different sizes and study the critical velocity to cause fluidization.

FLOW RATE MEASUREMENT

Capillary flow meter

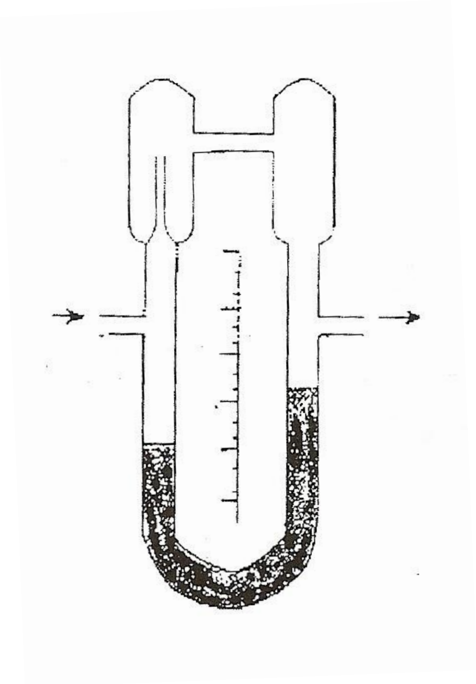
Capillary flow meter is a device consisting of 50 to 100 mm long capillary tube made of glass. Two legs of a manometer are connected across the two ends of the capillary to measure the pressure drop for the flow of gas to occur through the capillary. Schematic of the flow meter is shown in the figure. The flow meter is calibrated for a particular gas against a standard unit such as wet test meter or the soap bubble meter. From theoretical considerations, one may derive the following expression for flow of fluid in a tube under steady and laminar flow conditions:

$$Q = \frac{\pi d^4 \Delta P}{128 \mu_g l}$$

Flow may be taken as laminar of the Reynolds number is less than 2100 i.e.

$$\frac{\rho_g \bar{u} d}{\mu} = \frac{4 \rho_g Q}{\pi d \mu_g} < 2100$$

Pressure drop across the capillary may be expressed in terms of the difference in heights of the fluid such as water or mercury in the two columns of the manometer.



$\Delta P = \rho_f g h$

u =Velocity of fluid

ρ_g = Density of gas, kg/m^3

ρ_f = Density of fluid in the manometer, kg/m^3

μ_g = Viscosity of gas, kg/ms (PI units)

Q = Discharge rate in m^3/s

I = Length of the capillary, m

d = Diameter of the capillary, m

h = Difference in heights in the two legs of the water manometer.

ΔP = Pressure drop in Pascal.

μ_{O2} = $12 \times 10^{-6} \times T^{0.5}$ P, T in ^oK

AERODYNAMICS OF BEDS OF SOLID PARTICLES

Ergun suggested the following equation to describe the flow of gases through packed columns.

$$\frac{\Delta P}{L} = \psi \left(\frac{1-\varepsilon}{\varepsilon^3} \right) \left(\frac{1}{\Phi d_P} \right) \rho_g U_o^2 \tag{1}$$

Where

ε = $1-\rho_S$ (bulk)/ ρ_S (true)
Φ = shape factor =1 for sphere
d_P = dia of sphere or $6/S_o$ any other shape
S_o = surface/volume ratio for solid particle
P = density of gas
U_o = empty tube velocity of the gas
$\Delta P/L$ = pressure drop per unit length of bed
Ψ = friction factor

The friction factor may be found by using empirical correlation as follows:

Ψ = 150/Re Re<0.1 (2)
Ψ = 1.75 + 150/Re 10<Re<2500 (3)
Ψ = $160/\mathrm{Re} + 3.1\mathrm{Re}^{0.1}$ Re>2500 (4)

Re is the Reynolds number for flow of gases through a bed of solid particles i.e.

$$\mathrm{Re} = \frac{\rho_g U_o d_p}{\mu_g (1-\varepsilon)} \tag{5}$$

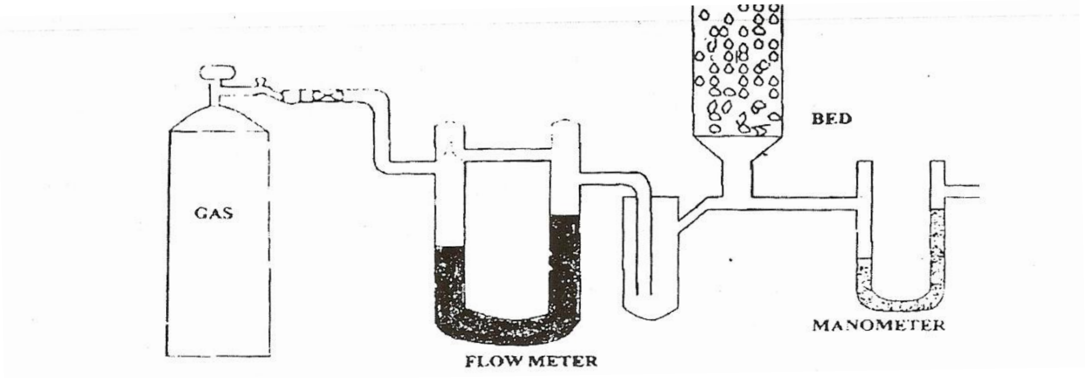
μ_g is viscosity of gas phase. Onset of fluidization would occur when pressure forces excess the gravitational forces i.e.

$$\frac{\Delta P}{L} = (1-\varepsilon)(\rho_s - \rho_g) g \tag{6}$$

Critical empty bed velocity may be determined from equations (1) and (6):

$$U_c = \left\{ \left[\frac{\rho_s - \rho_g}{\rho_g} \right] g \left(\frac{\Phi d_P}{\Psi} \right) \varepsilon^3 \right\}^{\frac{1}{2}} \tag{7}$$

Schematic of the set up to study aerodynamics of beds of solid particles is shown below



STEPS FOR PRESSURE DROP MEASUREMENTS

1. Determine the void ratio of different beds by measuring the bulk and true densities of solid particles.
2. Make connections for the flow of gas to the bed I and pressure drop measurements as shown in the figure above.
3. Make sure that the regulator and needle valves are in closed position.
4. Open the gas cylinder by turning the key.
5. Open the needle valve slowly to obtain certain flow of the gas through the set up.
6. Record the flow rate of the gas and its pressure from the capillary flow meter and the water manometer respectively after near stabilized conditions are attained.
7. Repeat steps (5) and (6) for six different values of the flow rate.
8. Repeat steps (2) to (7) for beds II to IV.
9. Repeat steps (2) and (6) for three different values of flow rate for bed V till fluidization occurs. Record the bed height in bed V for each flow rate.
10. Repeat steps (2) and (6) for three different values of flow rate for bed V beyond the onset of fluidization. Record the bed height in bed V for each flow rate.

REPORT:

1) Calculate and plot the pressure drop versus flow rate of gas for beds I and V on same graph paper and show appropriately the experimental data on these plots for comparison.
2) Determine the minimum velocity of fluidization from the data and compare it with the theoretical value.

DATA

I.D. of glass tubes = 20 mm.
Density of glass beads = 2.3 g/cm^3

Column	I	II	III	IV	V
Material	Iron ore	Iron ore	Iron ore	Glass beads	Iron ore
Size, mm	1.015	1.015 – 2.4	2.4	To measure	0.3
Weight, g	150	150	125	50	20

DATA SHEET

EXPERIMENT -2

Flow of Gases through Beds of Solid Particles

Date of experiment …………………………………

Day and Group …………………………………

Name and Roll No. …………………………………

Sl. N.	Left Col.	Right Col.	Difference	Flow rate	Left Col.	Right Col.	Difference	Pressure
	mm	mm	mm	Cc/s	M	mm	mm	Pa
BED No. 1								
1								
2								
3								
4								
5								
6								
BED No. 2								
1								
2								
3								
4								
5								
6								
BED No. 3								
1								
2								
3								
4								
5								
6								
BED No. 4								
1								
2								
3								
4								
5								
6								
BED No. 5								
1								
2								
3								
4								
5								
6								

EXPERIMENT- 3

MASS TRANSFER BETWEEN SOLID AND LIQUID IN GAS STIRRED LADLES

INTRODUCTION

Mass transfer between solid additions and bulk liquid steel is an integral feature of numerous processing operations carried out in steelmaking ladles. As an example to this, mention can be made of melting and dissolution of deoxidizer elements and alloying additions in steel baths. A situation of pure dissolution (mass transfer) exists when the addition's melting point is greater than that of steel (Note that Fe, Nb or FeW dissolves in steel rather than melt, as their melting points are significantly higher than the liquid steel processing temperature). To enhance the rate of mass transfer from the solid, submerged gas injections have commonly been applied to the contents of the ladle. This is so as the dissolution process is typically boundary layer mass transfer controlled and therefore convection plays important role in influencing the associated rate. It is now well known that the turbulent recirculatory flow fields induced during stirring practices affects the rate of the dissolution or mass transfer processes considerably. Naturally therefore, it is important to know what extent the rate of dissolution is influenced by key operating variables, such as the gas flow rate.

OBJECTIVE:

To determine mass transfer coefficient in the plume region of an axis- symmetric gas stirred ladle as a function of gas flow rate by weight loss technique.

EQUIPMENT:

Water model of cylindrical shaped ladle (Dia = 29cm)
Nitrogen gas cylinder and Rota meter

MATERIAL:

Benzoic acid, cylindrical shaped compacts.

PROCEDURE:

1. Fill water up to 25cm.
2. take benzoic acid cast, dip it in water, squeeze in towel, weigh it on electronic physical balance, measure its length and radius using vernier caliper.
3. Length is to be measured only once, weight and radius has to be measured at the end of each experiment (e.g. 5min, 7.5min, 10 min and 12.5 min)
4. Only one sample is enough for all gas flow rates (2, 4, and 6lit/min)
5. Fit the sample and sample holder such that riveted end lies downwards to avoid dissolution in longitudinal direction.
6. Position the sample at the centre of the plume (10cm below the free surface and 10cm above the bottom).
7. After each experiment switch off the gas.
8. Plot the graph during experiment itself and get it signed.
9. Keep all the instruments back at their old position and return used sample, sample holder and scale.

REPORT:

1. Complete the table from your observations.
Position of the sample in the bath:
Average initial radius and length of the three samples.
(i)
(ii)
(iii)

Gas flow rate, l/min	**Initial weight (g) at various times, min.**				**Final weight (g) at various times, min**				**Corresponding weight at various times, min**				**Estimated average sample radius at various times, min**			
	0	**5**	**12.5**	**22.5**	**5**	**12.5**	**22.5**	**35**	**5**	**12.5**	**22.5**	**35**	**5**	**12.5**	**22.5**	**35**

2. Plot average instantaneous radius (in cm) as a function of time (in secs) for all the three flow rates from the above table in single figure neatly. Try to fit a straight line through each set of four points. <u>*Report the slope of the individual lines in a small table for all three flow rates.*</u>

3. From 2, compute the mass transfer coefficient on the basis of the following relationships:

$$K_{av} = -\frac{dR}{d\,t} \cdot \frac{r_S}{C_s^*}$$

In which K_{av} is the mass transfer coefficient and ρ_s is the density of benzoic acid (1.26 gm/cc) and C_s^* is the solubility of benzoic acid (4.0704 gm/cc). <u>*Report the estimated mass transfer coefficient in a small table for all three flow rates.*</u>

4. The mass transfer coefficient can also be calculated theoretically from the mass transfer correlation given below

$$Sh_d = 0.546\,(Re_d)^{0.57}.(Sc)^{0.33}$$

In this Re_d is the diameter based Reynolds number and is defined as, $(U_P.d)/v$. (Note that U_P is the plume rise velocity and is defined in S.I. units as,

$U_P = 4.4.Q^{0.33}.L^{0.25}.R^{0.33}$, Where L and R being the depth of liquid and vessel radius). The kinematic viscosity of water is 10^{-6} m^2/s and the Schmidt number for the system is about 500. Show the predicted values of mass transfer coefficients for various flow rates in a small table.

5. In a separate plot compare the predicted and experimental values of mass transfer coefficients. What is your observation?

Answer the following question:

1. Define Sh and Sc as they appear in the mass transfer correlation above.
2. Derive the relationship given in three above, applying the principles of material balance. State the assumptions that are implicit in the equation as shown in 3.
3. If the flow rate is maintained constant but the depth of liquid in the system is increased, would the mass transfer coefficient go up or down? Provide quantitative argument to justify your answer.
4. Derive an explicit relationship between mass transfer coefficient and gas flow rate from the given mass transfer correlation.

EXPERIMENT -4
GAS SOLID EQUILIBRIUM DURING DECOMPOSITION OF CALCIUM CARBONATE

AIM

The experiment is aimed at finding the standard free energy, enthalpy and entropy of formation of calcium carbonate through pressure measurement technique.

THEORY

Decomposition of calcium carbonate may be represented as follows:

$CaCO_3(s) = CaO(s) + CO_2$ K = p_{CO_2}

Activity of solid species may be taken as unity as these are present in pure form. The standard free energy for the reaction is related to the equilibrium constant as follows:

$$\Delta G^0 = -RT \ln K = -RT \ln p_{CO_2}$$

Standard enthalpy and entropy changes for the reaction are related to the standard free energy change as described below:

$$\Delta H^0 = \frac{\partial\left(\Delta G^0 / T\right)}{\partial\left(1/T\right)}$$

or

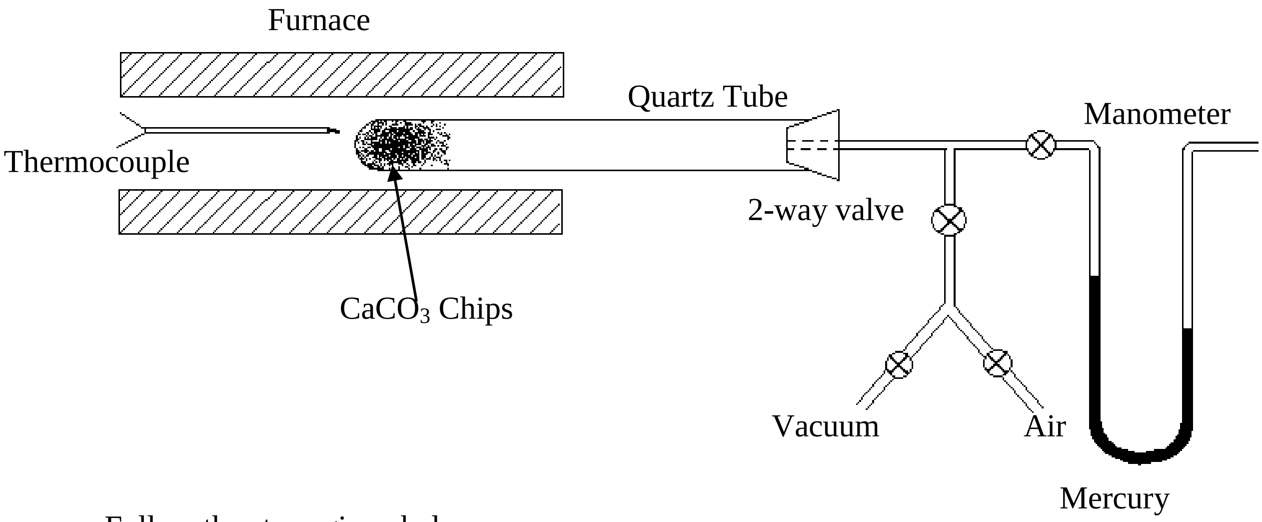
$$\Delta H^0 = -R\frac{\partial\left(\ln p_{CO_2}\right)}{\partial\left(1/T\right)}$$

and

$$\Delta S^0 = \frac{\partial\left(\Delta G^0\right)}{\partial\left(T\right)} = -R\frac{\partial\left(T \ln p_{CO_2}\right)}{\partial\left(T\right)}$$

PROCEDURE

The apparatus for the study on decomposition of Calcium Carbonate is shown below:



Follow the steps given below:

1. Place about 20g of Calcium Carbonate in the silica tubing one end closed.
2. Keep the reaction tube in the furnace and bring it to around 300°C.
3. Evacuate the system using the vacuum pump to around 0.1 mm Hg level.
4. Close the stopcock connection to vacuum pump.
5. Set the temperature controller of the furnace at 760°C
6. Record the changes in the temperature of the furnace and levels of mercury in the two legs of manometer at regular intervals of 2 minutes.
7. Repeat the experiment at 800°C and 840°C

The partial pressure of carbon dioxide in the reaction tube at any instant is related to the difference in the mercury levels in two legs of the manometer as follows:

= [barometer pressure in mm Hg – manometer reading in mm Hg] / 760

Barometer pressure may be obtained from the barometer fixed in the walls of the laboratory.

REPORT

1. Plot ln p_{CO_2} versus (1/T) to find value of ΔH° for the reaction.
2. Plot (T ln p_{CO_2}) versus T to get value of ΔS° for the reaction.
3. Determine from your data the temperatures at which calcium carbonate would decompose when heated at one atm.

DATA SHEET

Date of Experiment:…………....

Name	Roll No.
1)…………………	………………
2)…………………	………………

Time, min	**Temperature, °C**	**Temperature, K**	**Manometer Left Leg, mm Hg**	**Manometer Right Leg, mm Hg**	**Pressure Difference, mm Hg**

EXPERIMENT NO- 5
DESIGN OF AN ELECTRIC RESISTANCE FURNACE AND CALIBRATION OF THERMOCOUPLE

AIM

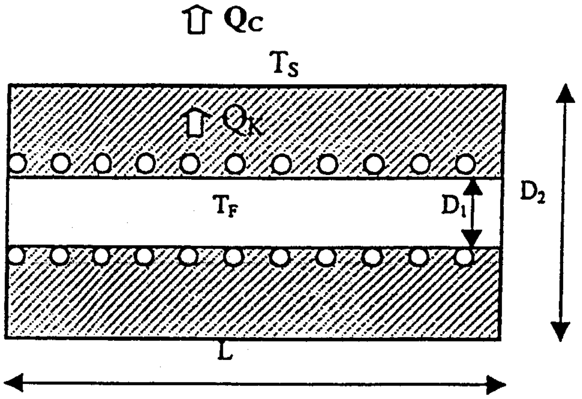
A. To learn construction of an electric resistance furnace.
B. To calibrate a given thermocouple and to determine the temperature profile of a cylindrical tube furnace

PRINCIPLES OF DESIGN:

For a constant temperature of the furnace, the electric power must equal the rate of heat losses to the surroundings by convection and radiation. Heat flows by conduction from inside tube to the outer shell through the refractory thickness. From the outside shell, heat is lost partly by convection and partly by radiation. Heat balance gives the followings:

Q_K = Heat flow by conduction $= 2\pi LK(T_F - T_S)\mathrm{Ln}(\frac{D_2}{D_1})$

Q_C = Heat losses by convection and radiation $= \pi LD_2h(T_S - T_O) + \sigma A(T_S{}^4 - T_O{}^4)$



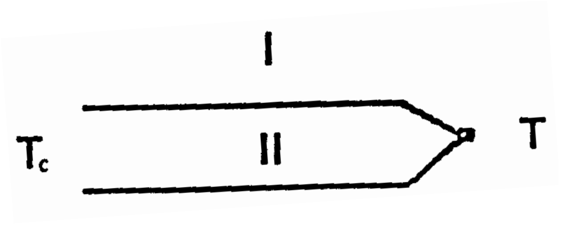
K is thermal conductivity of the refractory. T_F is the furnace temperature, T_S temperature of the outside shell of the furnace and T_O the ambient temperature. D_1 and D_2 are the diameters of the furnace tube and furnace shell respectively. ‘h’ is the overall heat transfer coefficient. Under steady state conditions, Q_K or Q_C must match the product of the voltage and current that passes through the heating element of the furnace i.e.

$$Q_K = Q_C = VI$$

TEMPERATURE MEASUREMENT USING THERMOCOUPLE WIRES

Set of two thermocouple wires is used to measure the temperature of a body in a certain range. It works on the See-back principle according to which and electric current tends to flow in a close loop of two dissimilar metals when two junctions are held at different temperatures. If the two wires are joined at one end only, an electromotive force is developed across the open ends of the wires and it can be measure to know the difference in the temperatures of hot junction and cold junction. The following thermocouples are commonly used to measure the temperature of a body:

(a) Platinum - Platinum + 13% Rhodium	0-1600^{o}C
(b) Platinum - Platinum + 10% Rhodium	0-1600^{o}C
(c) Chromel -Alumel	0-1250^{o}C
(d) Iron – Constantan	0-800^{o}C
(e) Copper - Constantan	0-400^{o}C



Standard tables are available that relate that e.m.f. to the actual temperature of the hot junction provided the cold junction is maintained at 0^{o}C. If the open ends of thermocouple wires are held at a finite temperature, measured e.m.f. must be corrected before using the table i.e.:

$$E_T = E_M + E_O$$

E_T is the corrected value, E_M actual measured value and E_O is the value from the table corresponding to the temperature of the cold junction. Low cost compensating or matching thermocouple wires may be used to increase the length of the thermocouple if necessary. These wires may not withstand high temperature but gives matching e.m.f. in lower temperature ranges when used in correct polarity. It is important that the thermocouple wires or the compensating wires must not touch each other except at the point of the hot junction otherwise the e.m.f. would correspond to the temperature of the point of contact rather than of the hot junction. Thermocouple is calibrated against set or standard temperatures such as melting points of pure metals, boiling point of water etc. The standard thermocouple once calibrated or tested can be used to test and calibrate another thermocouple.

PROCEDURE

Calibration of thermocouple and measurement of temperature profile. Take room temperature as the temperature of the cold end junction of thermocouple.

1. Note down the room temperature. Note the current and voltage of the furnace every 10 minutes until the furnace reaches steady state.
2. Tie a standard and a non-standard thermocouple together.
3. Place both the thermocouple in the centre of the furnace using guides.
4. Measure the e.m.f. at the centre and in steps of 3 cm by pulling the thermocouple to one end of the axis of the tube. Allow sufficient time for the thermocouple reading to reach a steady value for each measurement; this temperature is T_F. Simultaneously measure the corresponding temperatures of the outer surface of the furnace (T_S).

Measurement of cooling profile of furnace

1. Place the thermocouple tip at the centre position of furnace tube again. Allow sufficient time to reach steady value.
2. Put the cold end of thermocouple in ice.
3. Shut off the power supply and note down the temperatures at different intervals of time using thermocouples during cooling till 600^oC is reached.

REPORT

1. Plot the product VI as a function of time.
2. Calculate Q_K and Q_C using the corresponding values of T_F and T_S, as necessary. Calculate the (arithmetic) average flux. Compare the average Q_K, average Q_C, and the steady state value of VI. .
3. Determine the required gauge and length of the wire to construct a furnace of same size as used by you from the data below:
   Number of turns / cm at two ends of the tube (upto 10 cm) = 3
   Number of turns /cm elsewhere = 2
   Maximum voltage available = 240V
   Maximum current permissible = 5A

Kanthal Wire Data

Gauge	Diameter (m)	Ω/cm
16	0.001650	0.011
19	0.001070	0.022
21	0.000813	0.036
23	0.000635	0.057
25	0.000500	0.090

4. Plot the temperature versus distance along the furnace length for both thermocouples.
5. Tabulate your results of the thermocouple calibration. The X and Y axes correspond to the readings of the standard and non-standard thermocouples respectively.
6. Plot temperature versus time for the cooling period for both thermocouples.
7. Comment on the results of your measurements.

Calibration Table for Chromel-Alumel Thermocouples

^oC	0	10	20	30	40	50	60	70	80	90
	mv	mv	mv	mv	mv	mv	mv	mv	mv	mv
0	0.00	0.40	0.80	1.20	1.61	2.02	2.43	2.85	3.26	3.61
100	4.10	4.51	4.92	5.33	5.73	6.13	6.53	6.93	7.33	7.73
200	8.13	8.54	8.94	9.34	9.75	10.16	10.57	10.98	11.39	11.80
300	12.21	12.63	13.04	13.46	13.88	14.29	14.71	15.13	15.55	15.98
400	16.40	16.82	17.24	17.67	18.09	18.51	18.94	19.36	19.79	20.22
500	20.65	21.07	21.50	21.93	22.35	22.78	23.20	23.63	24.06	24.49
600	24.91	25.34	25.76	26.19	26.61	27.03	27.45	27.87	28.29	28.72
700	29.14	29.56	29.97	30.39	30.81	31.23	31.65	32.06	32.48	32.89
800	33.3	33.71	34.12	34.53	34.93	35.34	35.75	36.15	36.55	36.96
900	37.36	37.76	38.16	38.56	38.95	39.35	39.75	40.14	40.53	40.92
1000	41.31	41.70	42.09	42.48	42.87	43.25	43.63	44.02	44.40	44.78
1100	45.16	45.54	45.92	46.29	46.67	47.04	47.41	47.78	48.15	48.52

EXPERIMENT NO- 5

DESIGN OF AN ELECTRIC RESISTANCE FURNACE AND CALIBRATION OF THERMOCOUPLE

DATA SHEET

Date of experiment

Name	**Roll No.**
(1) …………………	……. …………….
(2)…………………	…………………...
(3)…….. ……………	………………….

Furnace Dimensions:

Outside dia = ……..mm; Inside dia = …….. mm; length = ……..mm;
Voltage = ……..V; Current = ……..A; Room Temp = ……..$_o$C;

E.m.f. at 25$_o$C (a)		E.m.f. at 0$_o$C (b)	
Standard	Non standard	Standard	Non standard

Temperature profile when reference is room temperature

Distance (cm)	Measured e.m.f.(mV) (c)		Corrected e.m.f. (mV) d = a + c		Temperature (ºC)	
	Standard	Non standard	Standard	Non standard	Standard	Non standard
0						
3						
6						
9						
12						

15						
18						
21						
24						

Cooling Data when reference is 0$_o$C

Time (min)	Measured e.m.f.(mV) (e)		Corrected e.m.f. (mV) f = b + e		Temperature (ºC)	
	Standard	Non standard	Standard	Non standard	Standard	Non standard
0						
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
12						
14						
16						
18						
20						
22						
24						

EXPERIMENT NO- 6

PROXIMATE ANALYSIS AND CALORIFIC VALUE OF SOLID FUEL

AIM

To determine the proximate analysis and calorific value of coal.

PROXIMATE ANALYSIS

The proximate analysis of coal comprises of determination of the moisture, ash, volatile matter and fixed carbon.

Moisture

Free water may exist in the coal as adsorbed on the surface, condensed inside fine capillary network and as bound to the coal molecule by chemisorptions and hydrogen bonding.

Volatile Matter

A volatile product obtained by the pyrolysis of coal in the absence of air is known as volatile matter. The product may contain hydrogen, methane, carbon monoxide, carbon dioxide, higher hydrocarbons, tar, water vapors, nitrogen, ammonia, hydrogen sulphide etc. The pyrolysis temperature of coal may lie in the range from 600-800^{o}C. The yield of volatile can be taken as a measure of its rank. Volatile matter will be much less in coke than that of coal as pyrolysis had occurred during coking at around 1000^{o}C. Volatile matter does not contain the moisture of coal but it contains water that is formed from the hydrogen and oxygen of coal during the decomposition.

Ash

Coals are associated with certain mineral or inorganic matter, which gets deposited along with vegetable matter or gets into coal by subsequent infiltration. The ash consists mainly of silica, alumina, iron oxide and lime. When heated, coal as does not melt sharply at any temperature, but begins to soften at much lower temperature than that required melting. The ash content in coke is much higher than in coal.

Fixed carbon

Fixed carbon is obtained by deducting the sum of moisture, ash and volatile matter percentage from 100.

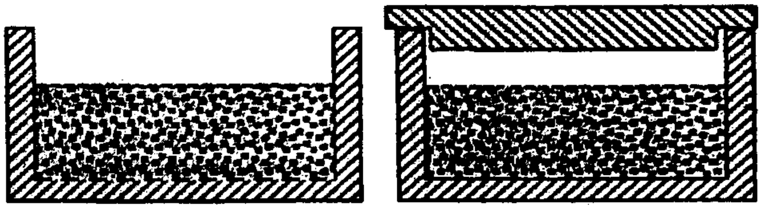
Calorific value

calorific value of the fuel is the quantity of heat liberated when unit quantity of fuel is burned completely. When hydrogen is present in the fuel, it is converted to stream. Thus, higher heating value (also called gross calorific value) is the total amount of heat liberated when one unit of fuel is burnt completely and the combustion products are brought to their standard state (25°C). Lower heating value (also called net calorific value) is
Net calorific value = Gross calorific value – latent heat of condensation

PROCEDURE

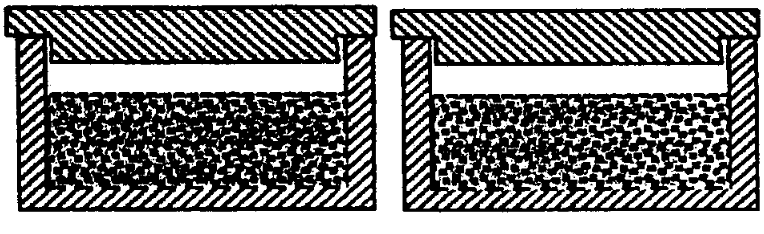
Moisture determination:

1. Dry the silica dish in an oven and weigh.
2. Spread out about 1 gm of 20 mesh coal sample on the dish.
3. Weigh the dish again to find the exact mass of the sample.
4. Heat the dish without any cover in the oven at about 105±5°C for 1 hour.
5. Take out the dish from the oven, cover it with the lid and cool.
6. Weigh the dish to find the loss in weight of coal due to presence of moisture.



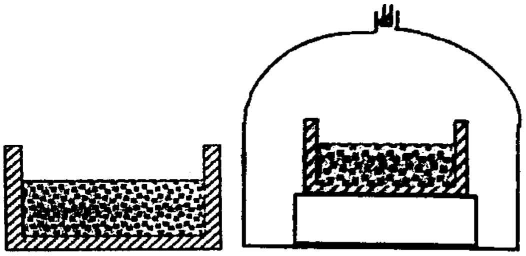
Volatile matter:

1. Heat a clean crucible and its lid at 900±15°C for 7 minutes in muffle furnace.
2. Allow the crucible and lid to cool on a metal plate for a minute and in dessicator for 10 minutes.
3. Weigh the crucible and lid together.
4. Put near 1 gm sample and weigh again to know the exact mass.
5. Insert the crucible with the lid on it into the furnace at 900°C and keep there for a period of 7 minutes.
6. Weigh the crucible with the lid to know the weight loss due to expulsion of volatile matter.



Ash determination:

1. Follow the steps 1 to 3 of moisture determination.
2. Insert the open dish in the furnace at 815°C for an hour.
3. Remove the dish; allow it to cool for 10 minutes on the slab and 15 minutes in the dessicator.
4. Weigh the dish to find the mass left which is the ash content of the coal.



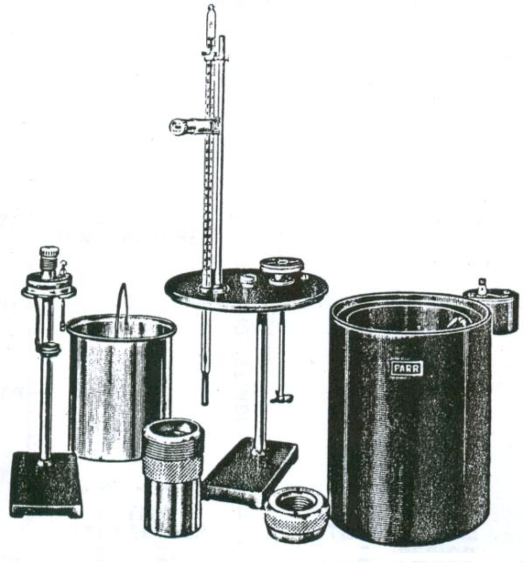
CALORIFIC VALUE OF COAL USING BOMB CALORIMETER

Follow the following procedure:

1. Weigh about 1 g of coal pellet and introduce it into the bomb.
2. Add 1 ml of distilled water and connect the fuse to the lid wires.
3. Pressurize the bomb with oxygen and place it inside the jacket.
4. Fill the jacket with 2000ml of water. Temperature of water must be 2°C less than the room temperature.
5. Assemble the calorimeter and run the motor for 5 minutes.
6. Record the temperature of the water bath accurately using a precision thermometer at an interval of 1 minute for 5 minutes and ignite the charge at the start of 6th minute.
7. Keep recording the temperature every minute till constant temperature is attained.
8. Open the bomb carefully.
9. Wash all the interior surfaces of the bomb with the jet of distilled water and collect the washings in a beaker.
10. Remove all unburned pieces of the fuse wire and measure the length of the pieces.
11. Collect and weigh the residual ash.

Caution: Never approach the top of the bomb due to danger of explosion.

The parts of a bomb calorimeter are shown below:



REPROT

(1) Determine the moisture content (wt.%) in coal samples, as received basis.
(2) Determine the ash content, V.M. and fixed carbon in percentages in both samples, dry basis and put all the results and data in the tabular form.
(3) Draw temperature versus time plot for the bomb calorimeter.
(4) Calculate the calorific value of coal from the data recorded.
(5) Method to calculate gross calorific value of coal (GCV)

$$\text{G.C.V.} = \frac{(t \times W) - [(a \times b) + (c \times d)]}{m}$$

t = Temperature rise in ^{o}C
W = Energy equivalent of the calorimeter in calories per degree centigrade
a = Weight of cotton thread (g)
b = Calorific value of cotton thread (3600 cal/g)
c = Length of nichrome wire used in experiment (cm)
d = Calorific value of nichrome wire (2.3 cal/g)
m = Mass of coal taken (g)

EXPERIMET NO- 6

PROXIMATE ANALYSIS AND CALORIFIC VALUE OF SOLID FUEL

DATA SHEET

Date of experiment

Name **Roll No.**

(1) …………………… …. ……………
(2)…………………… ….. ……………
(3)…………………… ….. ……………

(a) Proximate analysis of coal samples

Experiment	Temperature	Time(min)	Dish Weight	Dish+Sample Weight	Dish+Residue Weight
Moisture					
Volatile Matter					
Ash					

(b) Calorific Value of Coal using Bomb Calorimeter

Weight of Coal Pellet	g
Weight of Thread	g
Length of Wire	cm
Length of unused Wire	cm
Weight of Residual Wire	g
Weight of Residual Ash	g
Amount of Water added to Bomb Calorimeter	g
Weight of Bomb Calorimeter	g

Time (min)	Temp(°C)	Time (min)	Temp(°C)	Time (min)	Temp(°C)

EXPERIMENT- 7

CRUSHING AND GRINDING OF ORE

AIM

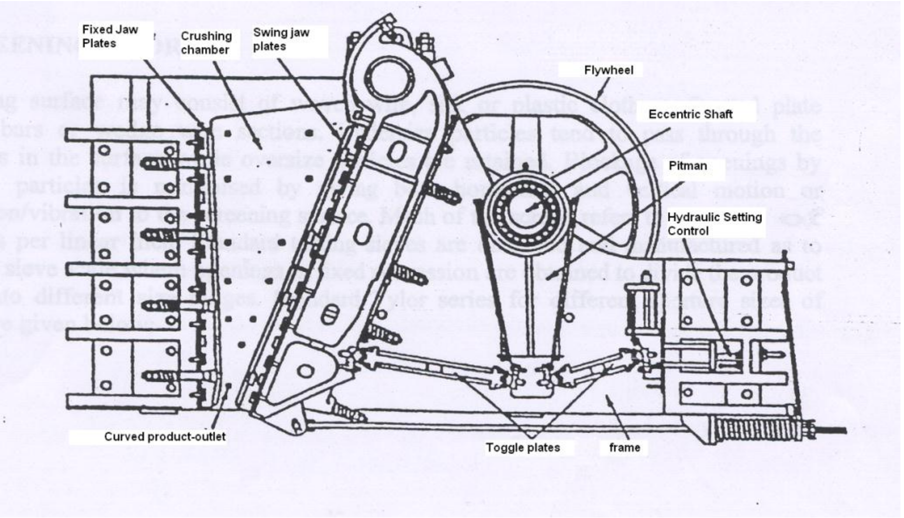
To Study the particle size distribution resulted due to crushing and grinding of the give rock sample.

INTRODUCTION

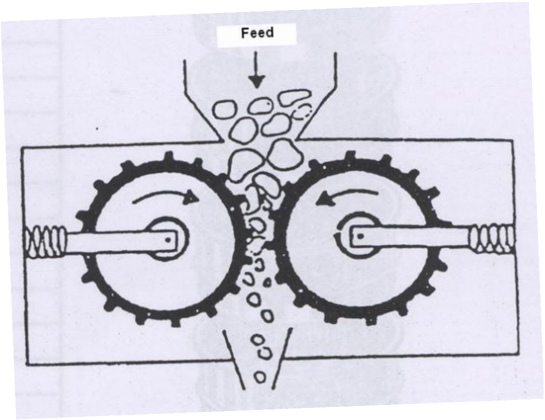
Crushing and grinding refer to bringing about size reduction in the ore so that valuable minerals are liberated from the gangue. It is the first step in any mineral beneficiation by methods such as Tabling, Jigging, Magnetic Separation, Electrostatic Separation, Floatation etc. Crushing devices include jaw crushers, gyratory crushers and roll crusher. Grinding refers size reduction of the material to much finer size of less than 0.1mm. Grinding is normally carried out in tumbling mills which are rotated on horizontal axis. Mills are commonly charged with a grinding medium made of forged steel, cast steel or cast iron having Brinnel hardness value of 300 to 370. The load to the mill may vary from 20 to 50%. The present experiment is aimed at studying the design, construction and action of the laboratory models of jaw crusher, roll crusher and ball mill.

A. **JAW CRUSHER**

1. Note the action of components of Jaw Crusher by turning the fly wheel by hand.
2. Note the movement of different components by idle running of the jaw crusher.
3. Measure the operational dimensions such as gape, mouth, throat, open and closed set.
4. Select large pieces of the rock and feed them singly into the crusher.
5. Study the product and measure the dimensions of the largest particle in the product.
6. Take 2 kg of rock sample in the size range of 25 to 50 mm by hand picking.
7. Feed the material to the crusher without choking the crushing zone of the machine.
8. Carry out the screening of the product by shaking the sieves for about 5 minutes.
9. Retain the product for use as feed to Roll Crusher.
10. Clean the crusher, sieves and the floor with the compressed air hose.



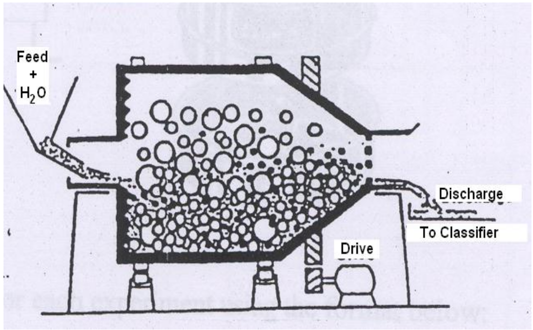
B. ROLL CRUSHER



1. Measure the diameter of the Rolls and the speed of the crusher by idle running
2. Set the crusher to 4 or 6 mm gap between the two rolls
3. Feed about 1 kg of the product of Jaw Crusher starting with the smallest size and note the suitability of treating different sizes of the feed to the machine.
4. Carry out the size analysis of the product of the roll crusher as explained before.
5. Clean the crusher, sieves and the floor with the compressed air hose.

JAW CRUSHER

C. BALL MILL

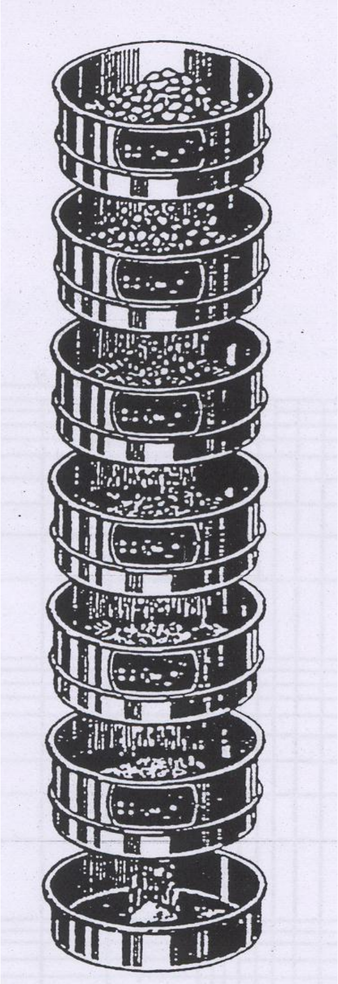


1. Note down the dimension of the grinding mill and find its volume.
2. Estimate the weight of grinding media being used and the volume.
3. Add about 200g of the crushed material in the size range of 3 to 14mm.
4. Run the mill for 10 min.
5. Take out the product completely and weigh.
6. Carry out the size analysis using sieves.
7. Repeat steps 3 to 6 by running the mill for 20 minutes.

D. SCREENING OF ORE

Screening surface may consist of woven wire, silk or plastic cloth, perforated plate grizzly bars of wedge wire sections. Undersize particles tend to pass through the apertures in the surface while oversize particles are retained. Blockage of openings by oversize particles is minimized by giving both horizontal and vertical motion or oscillation/vibration to the screening surface. Mesh of the screens refers to the numbers of openings per linear inch. Standard testing sieves are designed and manufactured as to follow a sieve scale where openings in fixed succession are obtained to divide the product range into different size ranges. Standard Tylor series for different aperture size of sieves are given below:

Mesh No.	Opening, mm	Mesh No.	Opening, mm
3	6.730	35	0.420
4	4.760	42	0.354
5	4.000	48	0.297
6	3.360	60	0.250
7	2.830	65	0.210
8	2.380	80	0.177
9	2.000	100	0.149
10	1.680	115	0.125
12	1.410	150	0.105
14	1.190	170	0.088
16	1.000	200	0.074
20	0.841	250	0.063
24	0.707	270	0.053
28	0.595	325	0.044
32	0.500	400	0.037



SIZE DISTRIBUTION:

The size distribution of particles resulting from either crushing or grinding is described frequently using a lognormal distribution function. An important characteristic of a lognormal distribution is that the mean, median and mode values are equal. Also the spread of particle sizes is readily characterized using geometric standard deviation, designated by σ_g. A plot between particle size (*d*) and their cumulative weight percentage in a probability paper will yield a straight line, if the particle sizes are distributed lognormally. The lognormal probability distribution function, *f*(*d*), is expressed as

$$f(d) = \sqrt{2\pi}.\ln(\sigma_g).\exp\left[-\frac{(\ln d - \overline{\ln d})^2}{2(\ln \sigma_g)^2}\right]$$

A lognormal size distribution can be characterized using two parameters: the mass median size, d_{50} and the geometric standard deviation σ_g.

REPORT:

1. Tabulate the data on Size Analysis for each experiment using the format below:

Mesh No.	Sieve Aperture, (X) mm	Weight Retained, g	% Weight Retained	Cumulative Wt. %, of Passing, (Y)

2. Plot log *Y* versus log *X* for all experiments. *Y* is cumulative wt.% passing and *X* is the size in mm.
3. Determine the value of size distribution modulus *m* as to satisfy the following:

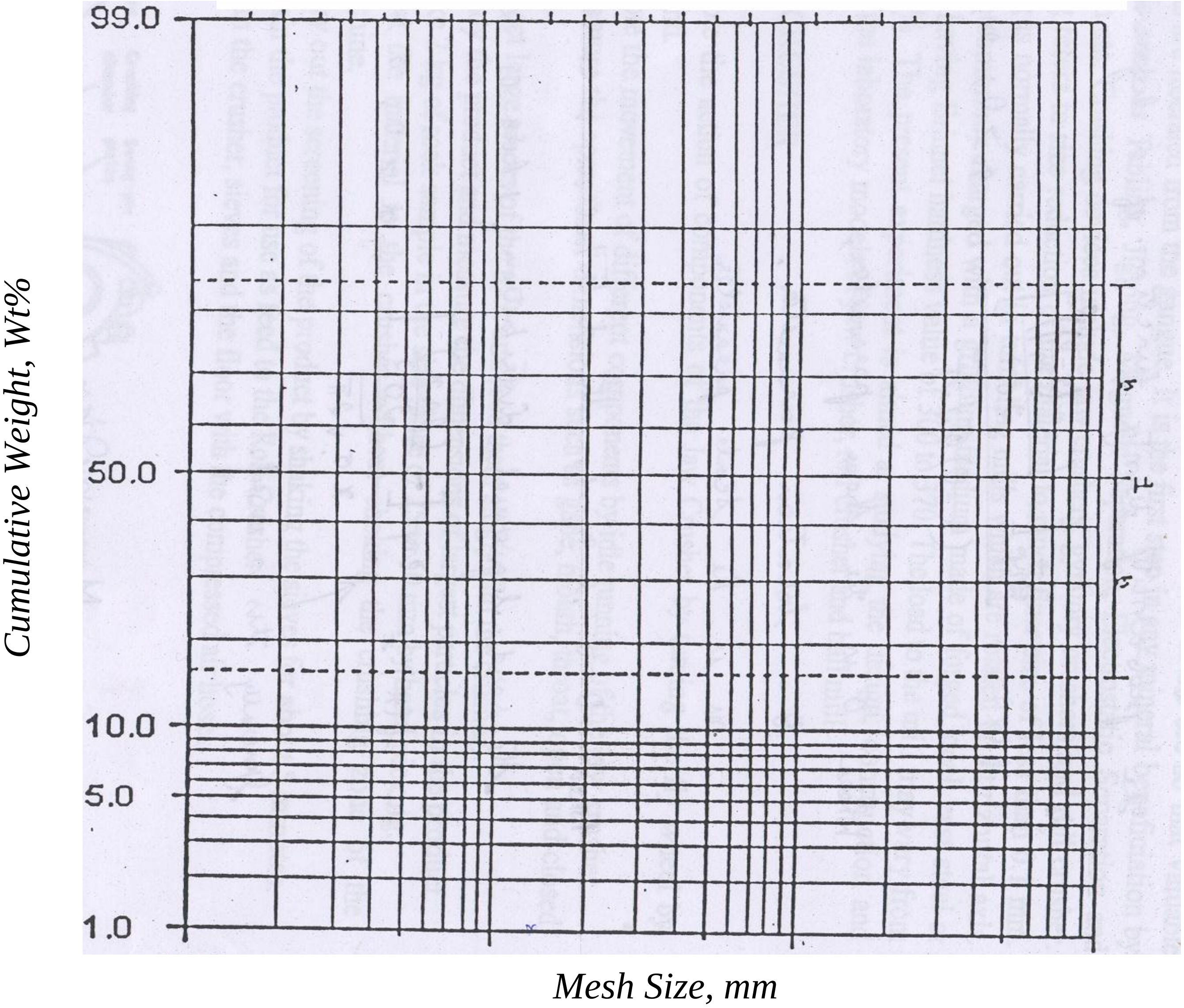
$$Y = 100.(X/K)^m$$

4. Plot the data on probability paper and determine log normal distribution parameters. The mass median size is d_{50}, and the geometric standard deviation is calculated from,

$$\sigma_g = \frac{d_{84}}{d_{50}}$$

where, d_{50} and d_{84} are respectively the particle sizes corresponding to 50% and 84% of cumulative weight percentages that can be referred from the probability paper.

Log Normal Probability Plot



EXPERIMENT NO- 8

ROASTING OF ZINC SULPHIDE

AIM

The aim of the experiments is to study the kinetics of roasting of ZnS by weight loss method.

THEORY

The sulphide ores are in general difficult to reduce to metal. Therefore, it is desirable to convert the sulphide form of the ore to oxide form, which is suitable for reduction. This conversion is carried out by heating the sulphide ore below its fusion temperature with excess of air.

$$2ZnS + 3O_2 \rightarrow 2ZnO + 2SO_2$$

This is a typical roasting operation. The most general definition of roasting is as follows:
Roasting is a metallurgical process in the extraction circuit wherein the ore is heated below the fusion point of its constituents under such conditions such that the metal component of the ore which is sought to recover will be chemically as well as physically changed from that form in which it occurs in the ore to some other form which will be amenable to same definite subsequent treatment for extraction of the metal.
There are various types of roasting operations possible. The four important ones are (i) oxidizing roasting, (ii) reduction roasting, (iii) sulphate roasting and (iv) chloridizing roasting. As the name suggests oxidizing roasting is carried out to burn out sulphur from sulphides and replace them in whole or in part with oxides. The reaction may be expressed as:

$$MS(s) + \frac{3}{2}O_2(g) \rightarrow MO(s) + SO_2(g)$$

Where M is a bivalent metal.

In industrial practice, roasting operations are carried out in various types of furnaces such as shaft furnace hearth roaster, rotary kilns and fluidized bed reactors. All roasting operations involve heterogeneous chemical reaction, that is to say, gas-solid reactions in which a solid reacts with a gas to produce another solid and gas, and therefore, the kinetics of a roasting operation should conform to the kinetics of gas solid reactions.

MATERIALS AND EXPERIMENTAL PROCEDURE

Materials required for this experiment includes (a) Zinc sulphide, (b) oxygen and (c) nitrogen/ argon.

1. Weigh about 0.60 gm of the dry ZnS pieces, in the wire mesh basket.

2. Flush nitrogen/argon in the furnace tube already heated to give steady state temperature for 5 minutes to drive out air from it.
3. With the help of a thin nichrome wire slowly lower the basket containing zinc sulphide into the furnace. The other end of the wire is attached to the pan of a balance which is used to measure weight loss of the concentrate during the roasting operation with removing it from the furnace. The wire should be freely suspended into the furnace.
4. Note down the weight reading on the balance.
5. Switch off nitrogen/argon and start flushing oxygen at a given slow rate taking care to avoid any carry over of the ZnS particles.
6. Immediately start taking the weight readings at intervals of 2 minutes till almost a constant weight reading is obtained.
7. Note the temperature and oxygen flow rates before and after the experiments. Try to maintain them constant as far as possible throughout the experiment. In case of variation, average values of temperature and oxygen flow rates may be used.
8. Repeat the above for two other temperatures with the same flow rate of oxygen. (Suggested temperature 600°C, 650°C and 700°C)

REPORT

Upon roasting of ZnS, any of the following 3 products are likely to form according to phase equilibria in Zn-S-O system.

Reaction	Log K (K is equilibrium constant)		
	T = 900K	T = 1000K	T = 1100K
$ZnS + \frac{3}{2}O_2 = ZnO + SO_2$	21.77	19.19	17.07
$ZnS + 2O_2 = ZnSO_4$	26.61	22.16	18.61
$3ZnS + \frac{11}{2}O_2 = ZnO_2ZnSO_4 + SO_2$	75.84	64.35	54.97
T. R. Ingraham and H.H. Kellogg: Trans. of Met. Soc. AIME, Vol.227(1963), p.1419			

In addition, this roasting reaction is exothermic leading to significant, rise of sample temperature during oxidation.

Also insitu sintering of sample during oxidation is likely. All these make the kinetics complex. The following procedure for reporting is suggested.

1. Assuming the product to be ZnO, calculate fractional conversion (F) as function of time from your weight loss data.
2. From oxygen flow rate and weight loss data, calculate average value of P_{SO_2} in exit gas at one of the temperature. Then do thermodynamic calculations to predict about product.

EXPERIMENT NO- 8

ROASTING OF ZINC SULPHIDE

DATA SHEET

Date of experiment

Name **Roll No.**

(1) ………………………. …………….
(2)……………………….. …………….

Weight of dry ZnS pieces = g
Weight of ZnS pieces after N_2 purging = g
Initial N_2 flow rate = c.c/min
Initial O_2 flow rate = c.c/min

Initial furnace temperature (^oC)	Time (min)	Weight of zinc sulphide (g)	Corresponding furnace temperature(^oC)	Corresponding oxygen flow rate (c.c./min)
600^oC				
650^oC				
700^oC				

EXPERIMENT- 9

KINETICS OF OXIDATION OF COPPER

AIM

The experiment is aimed at knowing the rates of oxidation of Cu in atmospheric air by the weight gain method.

THEORY

The oxygen must diffuse across the oxide layer to keep the process of oxidation going or the metal atoms must diffuse out of the film and react with the atmospheric oxygen. The flux of metal or oxygen atoms for the reaction can be determined from the Fick's laws of diffusion. For a planer surface, net increase in weight due to the oxidation reaction is given as follows.

$$\left[\frac{\Delta m}{A}\right]^2 = kt$$

Where Δm is increase in weight of sample (g).

A is surface area (cm^2).

t is time (sec).

and k is constant (g^2/cm^4sec^1).

PROCEDURE

1. Take three strips of copper and clean each with an emery paper and then in an acid (dil. HNO_3) solution followed by running water. Dry each strip carefully.
2. Attach the empty bucket with the hanging wire to the balance and insert the bucket into the furnace at 550^oC. Take reading after 3 min.
3. Take out the empty bucket and place one of the strips on it and insert it into the furnace. Take reading after 10, 20 and 30 minutes of time interval.
4. Repeat steps (2) to (4) at two other temperatures i.e. 650^oC and 750^oC.

REPORT

Report tabulated experimental results. From the results plot/calculate the following -

(a) Plot $\frac{\Delta m}{A}$ and $\left[\frac{\Delta m}{A}\right]^2$ versus time and find the value of k in the equation above and compare it for Cu and mild steel.

(b) Plot of log $\left[\frac{\Delta m}{A}\right]$ and $\frac{1}{T}$ for given periods of oxidation (T is temperature in K) and discuss significance of this plot.

(c) Plot log $\left[\frac{\Delta m}{A}\right]$ versus log[t] for all temperatures.

(d) Derive the equation (1) starting with the laws of diffusion and mention the assumptions made.

(e) Calculate the thickness of the oxide film which forms at all temperatures from the above data. Use density of Cu_2O as 6 g/cm^3

EXPERIMENT- 9

KINETICS OF OXIDATION OF COPPER

Weight of the empty bucket (g)	Weight of the bucket + Strip (g)	Difference of the weight (g)	Time (sec)	Initial weight of the metallic strip (g)	Weight gain of the metallic strip (g)	$\frac{\Delta m}{A}$ (g/cm^2)	$\left[\frac{\Delta m}{A}\right]^2$ (g^2/cm^4)	Log(time)
550^{o}C								
			0					
			600					
			1200					
			1800					
650^{o}C								
			0					
			600					
			1200					
			1800					
750^{o}C								
			0					
			600					
			1200					
			1800					

EXPERIMENT- 10

ELECTROLYSIS OF AQUEOUS SOLUTIONS

AIM

(a) To determine the decomposition voltage of aqueous solutions of $ZnSO_4$.

(b) To determine current efficiency by using aqueous solution of $CuSO_4$.

CIRCUIT

The basic circuit to be used as follows.

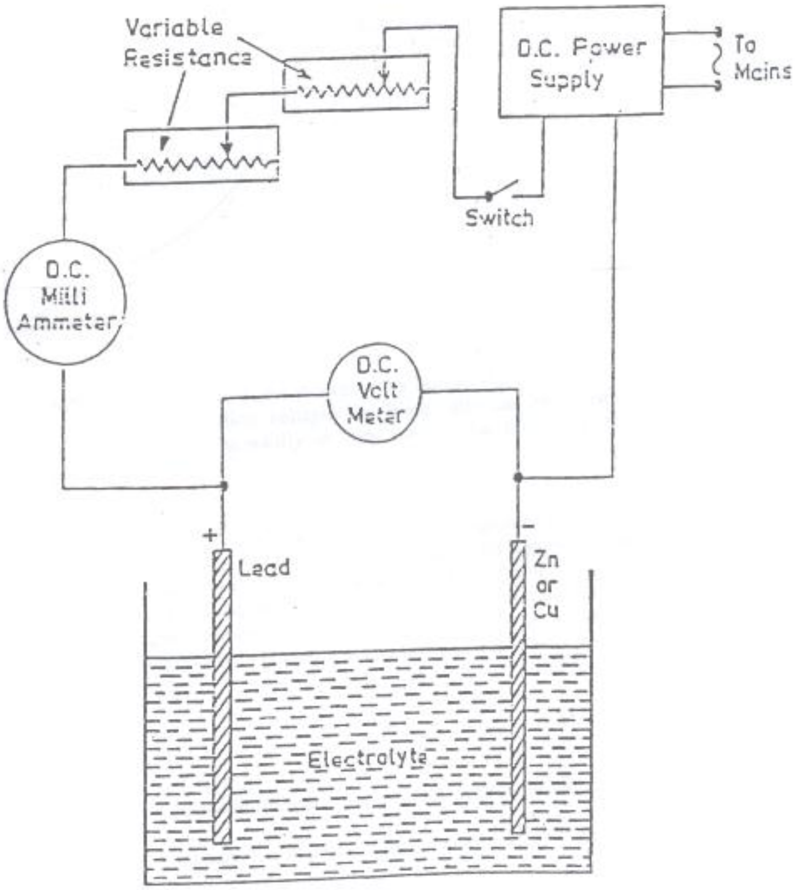


Fig:1

THEORY

The relationship between applied potential and the current flowing in an electrolytic cell usually take the characteristic shape shown in figure 2. The decomposition voltage for the electrolyte is obtained by extending the linear portion of the curve to the voltage axis. The points V^{I} and V^{II} on the figure 2 are the decomposition voltage for aqueous solution and fused salt respectively. As shown in the figure the residual currents (that is current obtained below the decomposition voltage) for fused salts can be significant. For aqueous solution, however, these are generally at very low so that the onset of decomposition is indicated rather sharply.

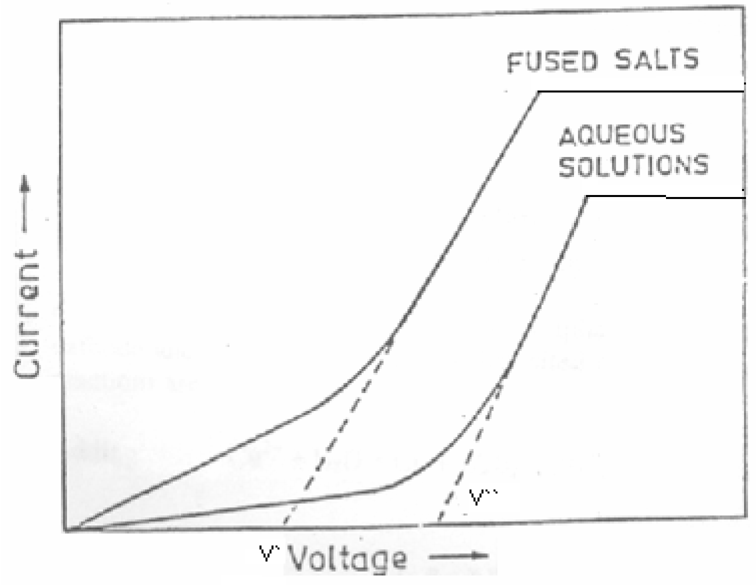


Fig:2

Beyond the decomposition voltage, the variation of current with voltage will be linear. However, current cannot be increased infinitely because, for given electrolyte and a given electrode surface area, there is a limiting current. Thus, beyond a maximum value of voltage, the current should become constant.

$$\Delta G = -ZFE \longrightarrow (1)$$

Where,
ΔG = - Changes in free energy due to the electrolysis reaction, Joules / mole
Z = Valency,
F = Faraday constant (96500 columbs / volt gm equivalent)
E = Reversible E.M.F. of the cell, volts

Again $\Delta G = \Delta G^o + RT \ln J \longrightarrow (2)$
Where, J = activity quotient
Hence $E = E^0 - (RT / ZF) \ln J \longrightarrow (3)$
Where E^0 is reversible e.m.f. of the cell under standard conditions (activities of all species are 1 & Temperature = 298 K).
R = 8.314 J / mole K

$$E^o{}_{cell} = E^o_{anode} + E^o_{cathode} \longrightarrow (4)$$

Where E^o_{anode} and $E^o_{cathode}$ are standard single electrode potentials of anode and cathode respectively (available in standard electro-chemistry texts).

$CuSO_4$: In electrolysis of an acidified aqueous solution of copper sulphate using copper as cathode and lead as anode, copper is deposited on the cathode and oxygen evolves at anode. The reactions are:

Adding: $Cu^{2+} + H_2O = Cu + \frac{1}{2}O_2 + 2H^+$ E^o_{cell} = - 0.892 V

$$E_{cell} = E^o_{cell} - \frac{RT}{2F}\frac{a_{Cu} \times (P_{O_2})^{1/2} \times (a_{H^+})^2}{(a_{Cu^{2+}})} \longrightarrow (5)$$

Since $a_{Cu} = 1 = P_{O_2} = 1\,atm.$

We have $$E_{cell} = -0.892 - \frac{RT}{2F}\frac{\ln\left(a_{H^+}\right)^2}{\left(a^{2+}_{Cu}\right)} \longrightarrow (6)$$

a_{H^+} and a^{2+}_{Cu} can be taken equal to concentration in mole per litre which can be calculated from concentration of Cu SO_4 and H_2SO_4 in the electrolyte.

$ZnSO_4$: Coming to electrolysis of $ZnSO_4$ and H_2SO_4 solution with Zn cathode and Pb anode. The reactions are:

Zn Cathode: $Zn^{2+} + 2e^- = Zn$ E^0 = -0.763 V

Pb anode: $H_2O = \frac{1}{2}O_2 + 2H^+ + 2e^-$ E^0 = -1.229 V

Adding $Zn^{2+} + H_2O = Zn + \frac{1}{2}O_2 + 2H^+$ E^o_{cell} = -1.992 V

$$E_{cell} = -1.992\,V - \frac{RT}{2F}\frac{\ln\left(a_{H^+}\right)^2}{\left(a_{Zn^{2+}}\right)} \longrightarrow (7)$$

Experimentally measured decomposition voltage should match with $/E_{cell}/$ as calculated from Eqs.(6) and (7).

PROCEDURE

A: CURRENT EFFICIENCY

1. Carefully weigh a clean copper cathode.
2. In the cleaned vessel, take a mixture of M/5 $CuSO_4$ and M/5 H_2SO_4 solution.
3. Introduce the Pd anode and Cu cathode into the solution.
4. Pass 40 mA current for ½ an hour.
5. Take out the cathode, clean and dry with distilled water and acetone and weight it.

B: DECOMPOSITION VOLTAGE

1. Set up the circuit as shown in the sketch.
2. Clean anodes and cathodes.
3. In a clean vessel, take one liter of an M/5 $ZnSO_4$ solution: Add 1 ml of M/5 H_2SO_4 to it and stir.

4. Introduce Pb and Zn plates: Pass current for 5 minutes: wait for 10 minutes. On open circuit to bring the electrodes in equilibrium. At 4 ml more of M/5 H_2SO_4 and stir. Wait 5 minutes.
5. Read this open circuit voltages.
6. Set the voltage of the D.C. power supply lower than the above voltage and close the circuit (make sure that there is high resistance in circuit).
7. Note the direction of current. Set the voltage such so as to reach zero current.
8. Wait 5 minutes and take this cell voltage reading. Shake the electrodes once or twice.
9. Increase the voltage of D.C. power supply as well as variable resistance setting in steps to obtain different values of current. At each setting wait for few minutes and also shake electrodes once for stirring.
10. Note current and cell voltage at each setting upto 5 mA Max. (6 points).
11. Decrease current in steps and take a few readings.

REPORT

(a) Plot current vs Cell voltage curve for $ZnSO_4$ solution and find out the decomposition potential.
(b) Calculate the E_{cell} from eqn. (7) and compare its absolute value with the experimentally measured decomposition voltage.
(c) Compare the above values with the open circuit voltage also.
    (i) Calculate current efficiency.
    (ii) Discuss your results.
    (iii) Discuss why hydrogen does not evolve during electrolysis of Cu.

EXPERIMENT- 11

MINERAL BENEFICIATION USING FLOTATION

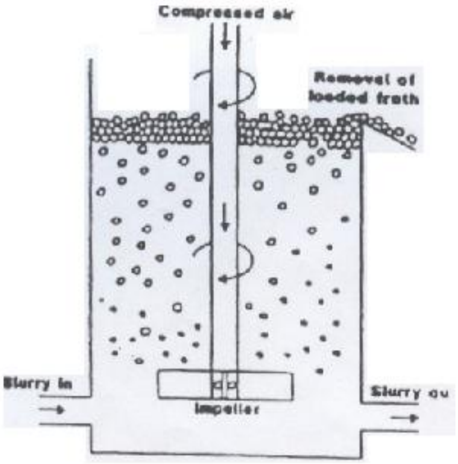
AIM

To study the separation of chalcopyrite by flotation method.

THEORY

Flotation is a mineral beneficiation process in which one particle is separated from another due to the difference in their wetting action rather than gravity. Solid-liquid-air suspension is created in a floating cell by allowing fine bubbles of air to disperse through the solid liquid slurry. Presence of air results in the formation of a froth, which rises to the top. Hydrophilic mineral particles are wetted by water. These particles are, thus, not attached to air bubbles and don’t become a part of the forth. Hydrophobic particles on the other hand are preferentially collected at the air bubble water interface and end up in the froth. Relative wettability of the solid particles in the mixture by water may be altered by adding certain reagents to the slurry. Collectors are added to enhance separation of certain types of minerals as they are selectively absorbed on the minerals and resist wetting action. Frothing agents may be added to enhance stability of the froth and improve the overall efficiency of the process. Depressants have been developed that bar flotation of unwanted minerals regulators are used to maintain proper pH of the solution.

PROCEDURE



1. Determine the density of chalcopyrite powder by water displacement method.
2. Prepare a pulp by mixing 200gm of chalcopyrite powder with 300ml of water.
3. Transfer the pulp into the flotation cell.
4. Add 78mg/Kg powder collector.
5. Stir the pulp for 3 minutes.
6. Add 500ml of water and stir for 1 minute.
7. Add 2 drops of pine oil and stir for 1 minute.
8. Add 500ml of water and stir for 1 minute.
9. Add lime for pH control and stir for 1 minute.
10. Bubble air for 2 minutes and continue stirring.
11. Collect the froth in a tray and weigh it.
12. Add coagulant and let solid particles to settle down and weigh the tailings.
13. Determine the density of the tailing and concentrate by using gravity bottle.

REPORT

1. Draw Schematic of the Flotation Cell and its dimensions.

2. Tabulate your results.
3. Name the common reagents such as collector, frother, activator, regulator, dispersant and depressant those are used in treatment of copper ores in the industry.

FLOTATION DATA SHEET

Chalcopyrite pulp: Volume = cc; Mass =

Amount of ore in the pulp	gm	81.864
Amount of collector added	gm	6.385×10^{-3}
Type of collector added		
Amount of lime added		
Drop of pine oil added		
Time of bubbling air		
Amount of dry froth		
Amount of dry concentrate		

	Ore	Froth	Tailing
W2 (bottle + solid)			
W3 (bottle + solid + water)			
Density			