2.3 Test on cement:

2.3.1 Physical Tests (IS: 4031)

1. Fineness Test
    - Sieve Method:
    - Air Permeability Method
    - Wagner Turbidimeter Method
2. Consistency Test
3. Initial and Final Setting Times:
4. Soundness Test
5. Autoclave Test
6. Strength
    - Compressive Strength
    - Tensile strength
7. Heat of hydration
8. Specific Gravity Test

1. Fineness Test

The degree of fineness of cement is the measure of the mean size of the grains in it. There are three methods for testing fineness: the sieve method—using 90 micron (9 No.) sieve, the air permeability method— Nurse and Blains method and the sedimentation method— Wagner turbidimeter method. The last two methods measure the surface area, whereas the first measures grain size. Since cement grains are finer than 90 micron, the sieve analysis method does not represent true mean size of cement grains. Also, the tiny cement grains tend to conglomerate into lumps resulting in distortion in the final grain size distribution curves. Considering these demerits, fineness is generally expressed in terms of specific area, which is the total surface area of the particles in unit weight of material.

Conditions affecting Fineness: The chemical composition and the degree of calcination influence the hardness of the clinker and consequently the fineness to which the cement is ground. Clinker, high in iron or silica, is apt to be hard and difficult to grind. The same is true with a hard-burned clinker. Fineness is also influenced by the time of grinding and the character of the pulverizing machinery. It has been found that cement becomes finer with age provided it does not absorb too much moisture. This is probably due to the decrepitation of the coarser grains resulting from the hydration of the embedded lime particles.

Importance: Finer the cement more is the strength since surface area for hydration will be large. With increase in fineness, the early development of strength is enhanced but the ultimate strength is not affected. An increase in the fineness of the cement increases the cohesiveness of the concrete mix and thus reduces the amount of water which separates to the top of a lift (bleeding), particularly while compacting with vibrators. However, if the cement is ground beyond a certain limit, its cementative properties are affected due to the prehydration by atmospheric moisture. Finer cement reacts more strongly in alkali reactive aggregate. Also, the water requirement and workability will be more leading to higher drying shrinkage and cracking.

i) Sieve Method: 100 g of cement sample is taken and air-set lumps, if any, in the sample are broken with fingers. The sample is placed on a 90 micron sieve and continuously sieved for 15 minutes. The residue should not exceed the limits specified below:

ii) Air Permeability Method: The fineness of cement is represented by specific surface, i.e. total surface area in cm^2 per gram or m^2 per kilogram of cement and is measured by Lea and Nurse apparatus or by wagner turbidimeter..

The Lea and Nurse apparatus shown in Fig. 28 essentially consists of a permeability test cell—where cement is placed and air pressure is applied, flowmeter—to determine the quantity of air passing per second through its capillary tube per unit difference of pressure, and manometer—to measure the air pressure.

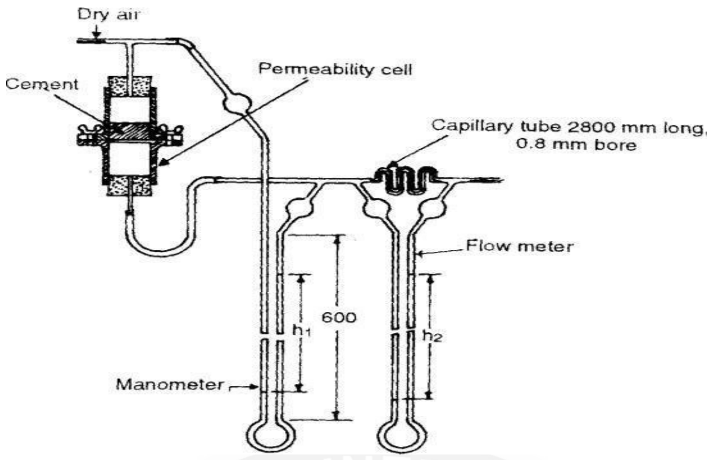

Fig. 9 Permeability Apparatus

To determine the fineness, a cement sample of 20 mm height is placed on a perforated plate (40 micron perforations) and air pressure is applied. The manometer is connected to the top of the permeability cell and the air is turned on. The lower end of the permeability cell is then slowly connected to the other end of the manometer. The rate of flow is so adjusted that the flow meter shows a pressure difference (h_2) of 30-50 cm. The reading (h_1) in the manometer is recorded. The process is repeated till the ratio h_1/h_2 is constant. The specific surface is given by the expression

where L = thickness of cement layerA = area of cement layer

d = density of cement

Y = porosity of cement (0.475)h_2 = flowmeter reading

h_1 = manometer reading

K is the flow meter constant and is obtained by

$$Q = \frac{K h_2}{P} d_1$$

where m = viscosity of air

d_1 = density of kerosene

Q = quantity of air passed per second

The minimum specific surface for various cements should be as specified in Table 3.

Table 3Minimum Specific Surfaces of Cements

Type of cement		Specific surface not less than cm^2/g
Ordinary Portland Cement	(OPC)	2250
Rapid Hardening Cement	(RHC)	3250
Low Heat Cement	(LHC)	3250
Portland Puzzolana Cement	(PPC)	3000
High Alumina Cement	(HAC)	2250
Super Sulphate Cement	(SSC)	4000

iii)Wagner Turbidimeter Method: L.A.Wagner developed a turbidimeter to estimate the surface area of one gram of cement. The cement is dispersed uniformly in a rectangular glass tank filled with kerosene. Then, parallel light rays are passed through the solution which strikes the sensitivity plate of a photoelectric cell. The turbidity of the solution at a given instant is measured by taking readings of the current generated by the cell. By recording the readings at regular intervals while the particles are falling in the solution, it is possible to secure information regarding the grading in surface area and in size of particle. Readings are expressed in sq. cm per gram.

2) Consistency Test

This is a test to estimate the quantity of mixing water to form a paste of normal consistency defined as that percentage water requirement of the cement paste, the viscosity of which will be such that the Vicat's plunger penetrates up to a point 5 to 7 mm from the bottom of the Vicat's mould.

Importance: The water requirement for various tests of cement depends on the normal consistency of the cement, which itself depends upon the compound composition and fineness of the cement.

Test Procedure: 300 g of cement is mixed with 25 per cent water. The paste is filled in the mould of Vicat's apparatus (Fig. 29) and the surface of the filled paste is smoothened and levelled. A square needle 10 mm x 10 mm attached to the plunger is then lowered gently over the cement paste surface and is released quickly. The plunger pierces the cement paste. The reading on the attached scale is recorded. When the reading is 5-7 mm from the bottom of the mould, the amount of water added is considered to be the correct percentage of water for normal consistency.

3) Initial and Final Setting Times:

When water is added to cement, the resulting paste starts to stiffen and gain strength and lose the consistency simultaneously. The term setting implies solidification of the plastic cement paste. Initial and final setting times may be regarded as the two stiffening states of the cement. The beginning of solidification, called the initial set, marks the point in time when the paste has become unworkable. The time taken to solidify completely marks the final set, which should not be too long in order to resume construction activity within a reasonable time after the placement of concrete. Vicat's apparatus used for the purpose is shown in Fig..

The initial setting time may be defined as the time taken by the paste to stiffen to such an extent that the Vicat's needle is not permitted to move down through the paste to within 5 ± 0.5 mm measured from the bottom of the mould. The final setting time is the time after which the paste becomes so hard that the angular attachment to the needle, under standard weight, fails to leave any mark on the hardened concrete. Initial and final setting times are the rheological properties of cement.

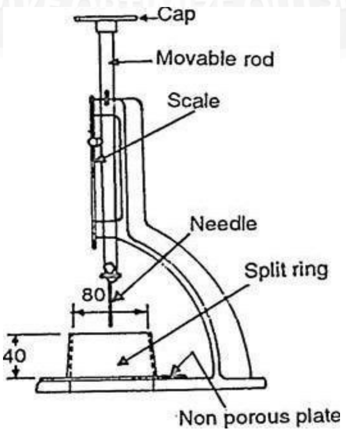

Fig 10 Vicat apparatus

Importance: It is important to know the initial setting time, because of loss of useful properties of cement if the cement mortar or concrete is placed in moulds after this time. The importance of final setting time lies in the fact that the moulds can be removed after this time. The former defines the limit of handling and the latter defines the beginning of development of mechanical strength.

Conditions Affecting Setting Time: The factors influencing the setting properties of cement are its composition, the percentage of retardant, degree of calcination, fineness of grinding, aeration subsequent to grinding clinker, percentage of water used to make cement paste, the temperature of the mixing water, cement and the atmosphere where the cement paste is placed, and the amount of manipulation the paste receives.The effect of lime, silica and alumina in controlling the set have been discussed in Sec. 5.3. The effect of gypsum is to increase the setting time of freshly ground cement. It is usually mixed with the clinker before final grinding, or just after the clinker has received preliminary grinding. The addition of gypsum before calcination causes it to decompose into lime and sulphur trioxide. Since the latter is liberated in the kiln, there is resulting effect on the setting time. Often, an underlimed cement becomes quick setting after seasoning. This can be avoided by adding to the cement 1 or 2 per cent of hydrated lime or the fraction of a per cent of Plaster of Paris. Setting time of cement is rapid with the increase in the fineness of cement. When the mixing water used in testing cement paste is increased by 1 per cent above that required for normal consistency, an increase of about 30 minutes or more is observed in the initial or final set.

Cements stored in warm rooms will, in general, be quick setting than those stored in cold places. Cold mixing water retards set while warm water accelerates it. Cement exposed to thoroughly saturated atmosphere will set much more slowly than those exposed to a dry atmosphere. If, however, a considerable proportion of moist CO2 is present in the air, the setting time is found to reduce greatly. By lengthening the time of mixing and by prolonged troweling of the surface mortars, it is also possible to considerably delay the setting time.

Test Procedure:

A neat cement paste is prepared by gauging cement with 0.85 times the water required to give a paste of standard consistency. The stop watch is started at the instant water is added to the cement. The mould resting on a nonporous plate is filled completely with cement paste and the surface of filled paste is levelled smooth with the top of the mould. The test is conducted at room temperature of 27± 2°C. The mould with the cement paste is placed in the Vicat's apparatus as shown in Fig. 5.9 and the needle is lowered gently in contact with the test block and is then quickly released. The needle thus penetrates the test block and the reading on the Vicat's apparatus graduated scale is recorded. The procedure is repeated until the needle fails to pierce the block by about 5 mm measured from the bottom of the mould. The stop watch is pushed off and the time is recorded which gives the initial setting time.

The cement is considered to be finally set when upon applying the needle gently to the surface of test block, the needle makes an impression, but the attachment fails to do so.

4)**Soundness Test**

It is essential that the cement concrete does not undergo large change in volume after setting. This is ensured by limiting the quantities of free lime and magnesia which slake slowly causing change in volume of cement (known as unsound). Soundness of cement may be tested by Le-Chatelier method or by autoclave method. For OPC, RHC, LHC and PPC it is limited to 10 mm, whereas for HAC and SSC it should not exceed 5 mm.

Importance: It is a very important test to assure the quality of cement since an unsound cement produces cracks, distortion and disintegration, ultimately leading to failure.

Conditions affecting Soundness: The main cause for unsoundness in Portland cement is the hydration of the uncombined lime encased within the cement particles. Exposed, finely ground, free lime in small percentages, hydrates before the cement sets and produces no injurious effect. The uncombined lime in cement is a result of either underburning the clinker or of excess lime in the raw materials. Freshly ground cement is often unsound due to the presence of uncombined lime. Cement is thus allowed to aerate for two to three weeks, allowing the lime to hydrate, to overcome unsoundness.

Fine grinding of the raw material and clinker help to produce a sound cement. By grinding fine the raw materials, it is possible to produce a homogeneous mixture before burning where the lime is uniformly distributed. The coarse grains of cement may imprison minute particles of uncombined lime which do not hydrate. These lime particles on hydralion produce disintegration.

Le-chatelier Method: The apparatus is shown in Fig. 11.The mould is placed on a glass sheet and is filled with neat cement paste formed by gauging 100 g cement with 0.78 times the water required to give a paste of standard consistency. The mould is covered with a glass sheet and a small weight is placed on the covering glass sheet. The mould is then submerged in the water at temperature of 27°-32°C. After 24 hours, the mould is taken out and the distance separating the indicator points is measured. The mould is again submerged in water. The water is now boiled for 3 hours. The mould is removed from water and is cooled down. The distance between the indicator points is measured again. The difference between the two measurements represents the unsoundness of cement.

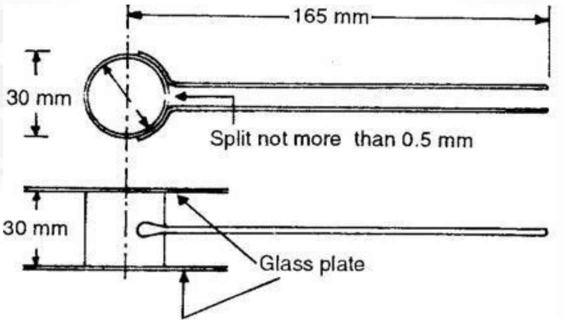

Fig. 11 Le-chatelier Apparatus

5)Autoclave Test: The 25 × 25 × 250 mm specimen is made with neat cement paste. After 24 hours the moulded specimen is removed from the moist atmosphere, measured for length, and so placed in an autoclave at room temperature that the four tides of each specimen are at least exposed to saturated steam. The temperature of the autoclave is raised at such a rate that the gauge pressure of the steam rises to 2.1 N/mm^2 in 1 to 1 ¼ hours from the time the heat is turned on. The pressure is maintained for 3 hours. Then the heat supply is shut off and the autoclave is cooled at such a rate that the pressure is less than 0.1N/mm^2 at the end of the hour. The autoclave is then opened and the test specimens are placed in water at temperature of 90°C.The temperature is gradually brought down to 27±2°C in 15 minutes. The specimens are maintained at this temperature for next 15 minutes and are then taken out. The length of the specimen is measured again. The difference in the two measurements gives the unsoundness of the cement.

6) Determination of Strength

Cement hydrates when water is added to it and cohesion and solidity is exhibited. It binds together the aggregates by adhesion. The strength of mortar and concrete depends upon the type and nature of cement. So, it should develop a minimum specified strength if it is to be used in structures. Cement is tested for compressive and tensile strengths.

Conditions affecting Strength: Cement is very strong at early ages if a high lime or high alumina content is there. Gypsum and Plaster of Paris in small percentages also tend to increase the strength slightly, but when present in quantities larger then 3 per cent, these substances provide variable effects. The effect of the clinker compounds on strength have already been discussed. In addition to the effect of composition, the strength of cement is greatly influenced by the degree of burning, the fineness of grinding, and the aeration it receives subsequent to final grinding. An underburnt cement is likely to be deficient in strength.

i)Compressive Strength: Compressive strength is the basic data required for mix design. By this test, the quality and the quantity of concrete can be cotrolled and the degree of adulteration can be checked.

The test specimens are 70.6 mm cubes having face area of about 5000 sq. mm. Large size specimen cubes cannot be made since cement shrinks and cracks may develop. The temperature of water and test room should be 27°± 2°C. A mixture of cement and standard sand in the proportion 1:3 by weight is mixed dry with a trowel for one minute and then with water until the mixture is of uniform colour. Three specimen cubes are prepared. The material for each cube is mixed separately. The quantities of cement, standard sand and water are 185 g, 555 g and (P/4) + 3.5, respectively where P = percentage of water required to produce a paste of standard consistency. The mould is filled completely with the cement paste and is placed on the vibration table. Vibrations are imparted for about 2 minutes at a speed of 12000±400 per minute.

The cubes are then removed from the moulds and submerged in clean fresh water and are taken out just prior to testing in a compression testing machine. Compressive strength is taken to be the average of the results of the three cubes. The load is applied starting from zero at a rate of 35 N/sq mm/minute. The compressive strength is calculated from the crushing load divided by the average area over which the load is applied. The result is expressed in N/mm^2.

ii) Tensile Strength: The tensile strength may be determined by Briquette test method or by split tensile strength test.

Importance: The tensile strength of cement affords quicker indications of defects in the cement than any other test. Also, the test is more conveniently made than the compressive strength test. Moreover, since the flexural strength, is directly related to the tensile strength this test is ideally fitted to give information both with regard to tensile and compressive strengths when the supply for material testing issmall.

Briquette Method: A mixture of cement and sand is gauged in the proportion of 1:3 by weight. The percentage of water to be used is calculated from the formula (P/5) + 2.5, where P = percentage of water required to produce a paste of standard consistency. The temperature of the water and the test room should be 27° ± 2°C. The mix is filled in the moulds of the shape shown in Fig. 5.11.

After filling the mould, an additional heap of mix is placed on the mould and is pushed down with the standard spatula, until the mixture is level with the top of the mould. This operation is repeated on the other side of the mould also. The briquettes in the mould are finished by smoothing the surface with the blade of a trowel. They are then kept for 24 hours at a temperature of 27° ± 2°C and in an atmosphere having 90 per cent humidity. The briquettes are then kept in clean fresh water and are taken out before testing. Six briquettes are tested and the average tensile strength is calculated. Load is applied steadily and uniformly, starting from zero and increasing at the rate of 0.7 N/sq mm of section in 12 seconds.

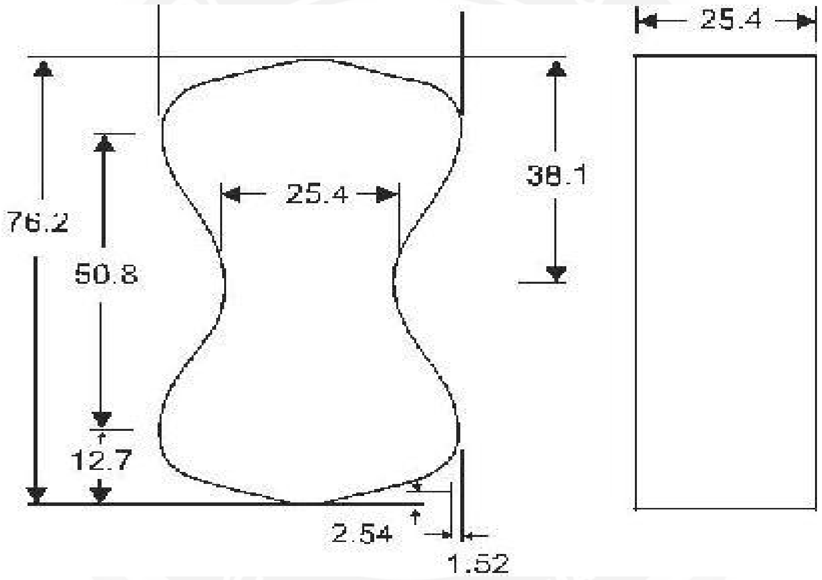

Fig. 12 Dimensions of Standard Briquette

Ordinary Portland cement should have a tensile strength of not less than 2.0 N/mm^2 after 3 days andnot less than 2.5 N/mm^2 after 7 days.

Notes:

(i) In the tension test of cement the load on the briquette should be applied centrally. Since briquettes become brittle with age, the effect of slight eccentricity or any torsional strain is pronounced in long-time tests.

(ii) The strength increases when the loading rate is increased from that specified.

7)Heat of hydration

Heat is evolved during hydration of cement, the amount being dependent on the relative quantities of theclinker compounds.

Importance: The evolution of heat causes an increase in temperature of the concrete, being greatest in mass concreting. Since the cooling of a mass of concrete can only occur from surfaces exposed to atmosphere the temperature of the interior is higher than that at the surface and also there is a rapid increase in strength in the interior than at the surface. Shrinkage cracks may result from stresses, induced by cooling of the surface while the interior of concrete is still at higher temperature. However, in practice, the heat evolution may be taken to its advantage in cold weather provided the concrete is warm at the time of placing and excessive heat loss is prevented by suitable lagging.

Test Procedure: The apparatus used to determine the heat of hydration of cement is known as calorimeter and is shown in Fig. 13. 60 g of cement and 24 ml of distilled water are mixed for 4 minutes at temperature 15°–25°C. Three specimen glass vials 100 × 20 mm are filled with this mixture, corked and sealed with wax. The vials are then stored with the mixture in a vertical position at 27°±2° C. The heat of hydration is obtained by subtracting the respective heat of solution of unhyrated cement calculated nearest to 0.1 calorie.

For determining the heat of solution of unhydrated cement, weigh a sample of about 3 g. At the sametime, weigh out 7.0 g of cement for the loss on ignition.

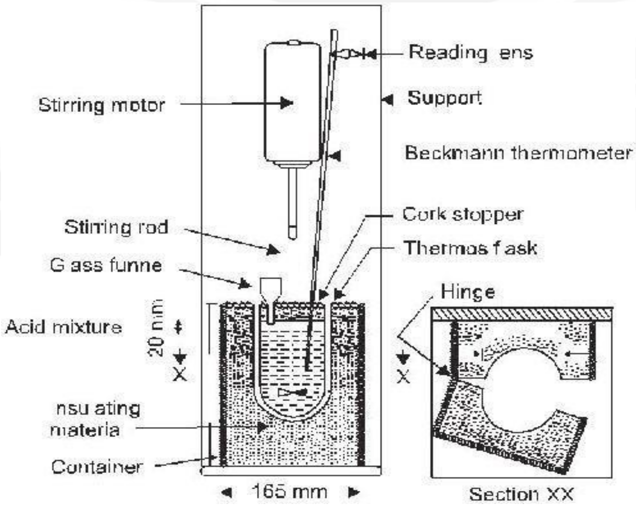

Fig.13 Calorimeter

Heat of solution (Cal/g) of unhydrated cement

where 0.2 is the specific heat of unhydrated cement.

For determining heat of solution of the hydrated cement, one of the glass vials is opened and the adherent wax is removed. The cement is ground rapidly, to avoid carbonation, to pass an 850 micron sieve. From this weigh out 4.2 g and 7.0 g of cement samples for heat of solution and loss on ignition.

The heat of solution of hydrated cement (Cal/g ignited weight)

$$\frac{\text{Heat capacity} \times \text{corrected temperature}}{Weight\ of sample\ corrected\ for\ ignition}$$

The ignition loss can be obtained by placing the sample in a cool furnace and raising the temperature of the furnace to 900°C over a period of 1 hour. The sample is kept at 900° ± 50°C for 3-4 hours and then cooled in a desiccator containing anhydrous calcium chloride. Weigh after half an hour. The difference in the two weighings give the loss on ignition.

To determine the heat capacity sufficient quantity of zinc oxide is ignited for one hour at 900°± 50°C. It is cooled in a desiccator containing anhydrous calcium chloride and ground to pass 250 micron sieve. About 7 g of this ignited oxide is reheated to 900° ± 50°C for 5 minutes and then cooled for about 2½ hours (not more than 5 hours). The calorimeter is assembled and temperature reading correct to 0.001°C is recorded to determine the initial heating or cooling correction. The zinc oxide is then introduced. The temperature readings are recorded at one minute intervals until the solution is complete. The recording of readings is continued for next 5 minutes to determine the final heating or cooling correction. The initial and final heating or cooling rates against the corresponding calorimeter temperature are plotted. The two points thus obtained are joined by a straight line. From this graph the corrections are read off for each temperature reading during the solution period. Heat capacity is calculated from the expression.

$$\text{Heat capcity}\left(\frac{\text{Cal}}{°\text{C}}\right) = \frac{\text{weight of ZnO}(256.1 \backslash 0.2 \backslash 0.1)}{\text{corrcted temperature rise}}$$

where, 256.1 is the heat of solution of zinc oxide at 30°C and 0.2 the negative temperature coefficient of the heat of solution, is the final temperature of the calorimeter, 0.1 is the specific heat of zinc oxide and is the room temperature in °C.

8)Specific Gravity Test

The specific gravity of hydraulic cement is obtained using Le-Chatelier flask shown in Fig. 14.

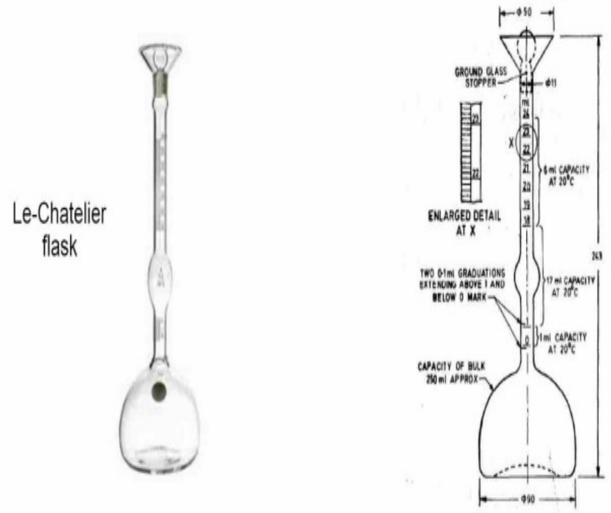

Fig.14 Le-Chatelier Flask for Specific Gravity Test

Conditions affecting Specific Gravity: Long seasonig is the chief cause of a low specific gravity in unadulterated cement. This is because the freshly ground cement when exposed to air rapidly absorbs moisture and carbon dioxide. Cements with high contents of iron oxide have a higher specific gravity. The effect of fineness of grinding upon specific gravity is slight. Very finely ground cements are likely to have lower specific gravities.

Test Procedure: The flask is filled with either kerosene free of water, or naphtha having a specific gravity not less than 0.7313 to a point on the stem between zero and 1-ml mark. The flask is immersed in a constant temperature water bath and the reading is recorded. A weighed quantity of cement (about 64 g of Portland cement) is then introduced in small amounts at the same temperature as that of the liquid. After introducing all the cement, the stopper is placed in the flask and the flask rolled in an inclined position, or gently whirled in a horizontal circle, so as to free the cement from air until no further air bubbles rise to the surface of the liquid. The flask is again immersed in the water-bath and the final reading is recorded. The difference between the first and the final reading represents the volume of liquid displaced by the weight of the cement used in the test.

2.3.2Chemical Tests (IS: 4032)

1. Loss on ignition
2. Silica
3. Combined Ferric Oxide and Alumina
4. Ferric Oxide
5. Alumina
6. Calcium Oxide
7. Magnesia: The filtrate (W3) is acidified with hydrochloric acid and is concentrated to
8. Sulphuric Anhydride
9. Insoluble Residue

1)Loss on ignition: 1.00 g of the sample is heated for 15 minutes in a weighed and covered platinum crucible of 20 to 25 ml capacity by placing it in a muffle furnace at any temperature between 900° and 1000°C. It is then cooled and weighed. Thereafter, the loss in weight is checked by a second heating for 5 minutes and reweighing. The loss in the weight is recorded as the loss on ignition and the percentage of loss on ignition to the nearest 0.1 is calculated (loss in weight × 100). The percentage loss on ignition should not exceed 4 per cent.

2)Silica: 0.5 g of the sample is kept in an evaporating dish, moistened with 10 ml of distilled water at room temperature to prevent lumping. To this 5 to 10 ml of hydrochloric acid is added, and digested with the aid of gentle heat and agitation until solution is complete. Dissolution may be aided by light pressure with the flattened end of a glass rod. The solution is evaporated to dryness on a steam bath. Without heating the residue any further, it is treated with 5 to 10 ml of hydrochloric acid and then with an equal amount of water. The dish is covered and digested for 10 minutes on a water bath. The solution with an equal volume of hot water is diluted and is immediately filtered through an ashless filter paper, and the separated silica (SiO_2) is washed thoroughly with hot water and the residue is reserved.

The filtrate is again evaporated to dryness, baking the residue in an oven for one hour at 105°C to 110°C. Then the residue is added with 10 to 15 ml of hydrochloric acid (1:1) and is heated on a water bath. This solution is then diluted with an equal volume of hot water and the small amount of silica it contains is filtered and washed on another filter paper. The filtrate and washings are reserved for the determination of combined alumina and the ferric oxide.

The papers containing the residues are transferred to a weighed platinum crucible. The papers are dried and ignited, first at a low heat until the carbon of the filter papers is completely consumed without inflaming, and finally at 1100°C to 1200°C until the weight remains constant(say W1).

The ignited residue thus obtained, which will contain small amounts of impurities is treated in the crucible with a few drops of distilled water, about 10 ml of hydrofluoric acid and one drop of sulphuric acid and evaporated cautiously to dryness. Finally, the small residue is heated at 1050°C to 1100°C for a minute or two: cooled and weighed (say W2). The difference between this weight and the weight of the ignited residue represents the amount of silica (W).

Silica (%) = 200 $(W1 - W2)$

3) Combined Ferric Oxide and Alumina: 200 ml of the sample from the filtrate reserved in silica test is heated to a boil. A few drops of bromine water or concentrated nitric acid is added during boiling in order to oxidize any ferrous ion to the ferric condition. It is then treated with ammonium hydroxide (1:1), drop by drop, until the solution smells of ammonia. The solution containing the precipitates of aluminium and ferric hydroxides is boiled for one minute. The precipitate is allowed to settle, filtered through an ashless filter paper and washed with two per cent hot ammonium nitrate solution. The filtrate and washings are set aside.

The precipitate and the filter paper is transferred to the same beaker in which the first precipitation was effected. The precipitate is then dissolved in hydrochloric acid (1:3). The solution is diluted to about 100 ml and the hydroxides are reprecipitated. The solution is filtered and precipitated with two 10 ml portions of hot ammonium nitrate solution. The filtrate and washings are then combined with the filtrate set aside and is reserved for the determination of calcium oxide.

The precipitate is placed in a weighed platinum crucible, heated slowly until the papers are charred, and finally ignited to constant weight at 1050°C to 1100°C with care to prevent reduction, and weighed (W1) as combined alumina and ferric oxide.

If silica is suspected to be carried into the filtrate used for this estimation, the residue in the crucible is treated with a drop of water, about 5 ml of hydrofluoric acid and a drop of sulphuric acid and is evaporated cautiously to dryness. Finally, the crucible is heated at 1050°C to 1100°C for one or two minutes; cooled and weighed (W2). The difference between this weight and the weight (W1), represents the amount of residue silica. This amount is subtracted from the weight of ferric oxide and alumina found as W1 and the same amount is added to the amount of silica (W). The ratio of percentages of alumina to iron oxide should not exceed 0.66.

Combined ferric oxide and alumina (%) = weight of residue × 200

4)Ferric Oxide: 40 ml of cold water is added to 1 g of the sample and while the mixture is stirred vigorously, 50 ml of hydrochloric acid is added. If necessary, the solution is heated and cement is ground with flattened end of a glass rod until it is evident that cement is completely decomposed. The solution is heated to a boil and is treated with stannous chloride solution added drop by drop while stirring, until the solution is dicolourized. A few drops of stannous chloride solution is added in excess and the solution is cooled to room temperature. Then, 15 ml of a saturated solution of mercuric chloride and 25 ml of manganese sulphate solution are added and titrated with standard solution of potassium permanganate until the permanent pink colour is obtained. Iron as ferric oxide is calculated.

5)Alumina: The calculated weight of ferric oxide and the small amount of silica is subtracted from the total weight of oxides (W1). The remainder is the weight of alumina and of small amounts of other oxides reported as alumina.

6)Calcium Oxide: The combined filtrate reserved in the combined ferric oxide and alumina test is acidified with hydrochloric acid and evaporated to a volume of about 100 ml. 40 ml of saturated bromine water is added to the hot solution and ammonium hydroxide is added until the solution is distinctly alkaline. The solution is boiled for 5 minutes or more, making certain that the solution is at all times distinctly alkaline. Then the precipitate is allowed to settle, filtered and washed with hot water.

The beaker and filter is washed once with nitric acid (1:33) and finally with hot water. Any precipitate (of manganese dioxide) that may be left on the tunnel is discarded. The filtrate is mixed with hydrochloric acid and boiled until all the bromine is expelled. 25 ml of boiling ammonium oxalate solution is added to the boiling solution. The solution is made alkaline with ammonium hydroxide and brought to boiling, the boiling being continued until the precipitated calcium oxalate assumes a well- defined, granular form. The precipitate is allowed to stand for about 20 minutes or until it has settled, filtered and washed moderately with ammonium oxalate solution (one gram per litre). The filtrate and washings (W3) are set aside for estimating magnesia.

The precipitated lime after ignition and heating at 1100°C-1200°C is weighed. The percentage of CaO = weight of residue × 200. Also CaO 0.7,SO_3 2.8, SiO_2 1.2, Al_2O_3 0.65, Fe_2O_3 in percent should not be less than 0.66.

7) Magnesia: The filtrate (W3) is acidified with hydrochloric acid and is concentrated to about 150 ml. To this solution, about 10 ml of ammonium hydrogen phosphate solution (250 g per litre) is added and the solution is cooled by placing in a beaker of ice water. After cooling, ammonium hydroxide is added drop by drop, while stirring constantly, until the crystalline magnesium ammonium phosphate begins to form, and then the reagent is added in moderate excess (5 to 10 per cent of the volume of the solution), the stirring being continued for several minutes. The solution is set aside for at least 16 hours in a cool atmosphere and then filtered. The precipitate is washed with ammonium nitrate wash solution (100 g of ammonium nitrate dissolved in water, 200 ml of ammonium hydroxide added and diluted to one litre). It is then charred slowly and the resulting carbon is burnt carefully. The precipitate is ignited at 1100°C to 1200°C to constant weight, taking care to avoid bringing the pyrophosphate to melting.

From the weight of the magnesium pyrophosphate obtained, the magnesia content of the materialtaken for the test is calculated.

The percentage of MgO = weight of residue × 72.4. Free magnesia in cement should be less than 4 per cent.

8)Sulphuric Anhydride: To one gram of the sample, 25 ml of cold water is added and while the mixture is stirred vigorously 5 ml of hydrochloric acid is added. If necessary, the solution is heated and the material is ground with the flattened end of a glass rod until it is evident that the decomposition of cement is complete. The solution is diluted to 50 ml and digested for 15 minutes. The residue is filtered and washed thoroughly with hot water. The filter paper with the residue (W4) is set aside. The filtrate is diluted to 250 ml and heated to boiling. 10 ml of hot barium chloride (100 g per litre) solution is added drop by drop and the boiling is continued until the precipitate is well formed. The solution is digested on steam bath for 4 hours or preferably overnight. The precipitate is filtered and the precipitate is washed thoroughly. The filter paper and the contents are placed in a weighed platinum or porcelain crucible and slowly the paper is incinerated without inflaming. Then it is ignited at 800°C to 900°C, cooled in a desiccator and the barium sulphate is weighed. From the weight of the barium sulphate obtained, the sulphuric anhydride content of the material taken for the test is calculated. The percentage of SO3 = weight of residue × 34.3. Sulphur in cement should be less than 2.5 per cent.

9)Insoluble Residue: The filter paper containing the residue (W4) is digested in 30 ml of hot water and 30 ml of 2 N sodium carbonate solution maintaining constant volume, the solution being held for 10 minutes at a temperature just short of boiling. It is then filtered and washed with dilute hydrochloric acid (1:99) and finally with hot water till free from chlorides. The residue is ignited in a crucible at 900°C to 1000°C, cooled in a desiccator and weighed. The insoluble residues should not exceed 1.5 per cent.